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Detritus - Multidisciplinary Journal for Waste Resources and Residues - is aimed at extending the “waste” concept by opening up the field to other waste-related disciplines (e.g. earth science, applied microbiology, environmental science, architecture, art, law, etc.) welcoming strategic, review and opinion papers. **Detritus is indexed in Emerging Sources Citation Index (ESCI) Web of Science, Scopus, Elsevier and Google Scholar**. Detritus is an official journal of IWWG (International Waste Working Group), a non-profit organisation established in 2002 to serve as a forum for the scientific and professional community and to respond to a need for the international promotion and dissemination of new developments in the waste management industry.

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Editorial

INDUSTRY OF THE ALGAE PRODUCTION IN EUROPE: THE FUTURE AND POSSIBLE APPLICATION IN A CIRCULAR AND ECO-FRIENDLY BIOECONOMY

The algae production industry in Europe is undergoing a transformative phase, positioning itself as a critical player in the emerging circular and blue bioeconomy. With its versatile applications ranging from environmental remediation to sustainable aquaculture, algae offer immense potential to address some of the most pressing challenges of our time, including climate change, resource depletion, and environmental degradation.

Algae, particularly extremophilic species, are increasingly recognized as valuable assets in promoting sustainable growth across various sectors. Their adaptability to extreme environments, such as high temperatures or salinity, provides unique opportunities to develop eco-friendly solutions. The European algae sector, comprising both macro and microalgae production, has experienced substantial growth, with Spain, France, and Ireland leading in production (Araujo et al. 2021).

These are important parameters that inquired this organism in the objectives of the 2030 Agenda for sustainable cities and communities, responsible consumption and production and climate action.

A Circular Economy Enabler

One of the core strengths of algae production lies in its alignment with the principles of a circular economy. Algae can contribute to carbon sequestration, nutrient recycling, and renewable energy generation. This makes them ideal for integration into industrial processes, such as wastewater treatment and bioremediation, where they help mitigate pollution while producing valuable biomass. Moreover, algae-derived bioplastics and sustainable aquaculture feed are emerging as promising solutions to reduce reliance on fossil-based resources.

While the industry is experiencing significant growth, several barriers still impede its full potential. High production costs, limited scalability, and a fragmented regulatory landscape hinder broader adoption and market penetration. Addressing these challenges will require concerted efforts in innovation, investment, and regulatory harmonization across Europe.

As reported by Araujo et al. (2021), the European algae sector spans 23 countries and includes companies producing both macroalgae, microalgae, and the cyanobacteria Spirulina. The number of these companies, which was approximately 500 in 2021, has been steadily increasing, with a 150% rise in the number of new companies over the last decade. Some of these companies have been operating since before 1980, indicating long-term interest and stability in the sector.

Among macroalgae, some seaweed species collected along the European coasts such as *Laminaria digitata*, *Laminaria hyperborea*, *Ascophyllum nodosum*, and *Gelidium corneum* are used as feedstock for the extraction of food, and the hydrocolloids alginate and agar. Algae biomass finds diverse applications, serving as feed for aquaculture, and has been investigated for enhancing weight gain in cattle while mitigating enteric methane emissions.

Species like *Dunaliella* and *Chlamydomonas* have been extensively studied for their capacity to accumulate cadmium, lead, and mercury, significantly contributing to the reduction of environmental pollution. Others, like *Chlorella* and *Botryococcus*, can degrade hydrocarbons found in oil spills, aiding in the remediation of contaminated aquatic ecosystems. These algae also produce bioactive compounds with antimicrobial, antioxidant or anti-inflammatory properties, useful for pharmaceuticals, supplements and functional foods. Additionally, their enzymes, pigments, and biopolymers have applications in industrial processes, thanks to their ability to function under extreme conditions, making them an innovative resource for biotechnology.

Other algae species that are gaining importance in the global market include *Chlorella vulgaris*, *Arthrospira platensis*, *Haematococcus pluvialis*, which are source of proteins, carbohydrates, vitamins, minerals and fatty acids, furthermore, the last two are also source of astaxanthin and phycocyanin, very important products in the food, medical, nutraceutical and cosmetic world. Phycocyanin, a significant compound derived from microalgal and cyanobacterial cultures, is employed as a fluorescent marker in diagnostic histochemistry and finds applications as a dye in foods, cosmetics, and therapeutic agents. The global phycocyanin market is steadily increasing in value. With consumers gravitating toward natural food-grade substances, the market is expected to continue growing in the coming decades. Factors influencing the market value of microalgae biomass and the valuable extracted compounds include the production system, production costs (energy, labor), geographical origin, certification schemes (e.g., organic production), and the step of the value chain.

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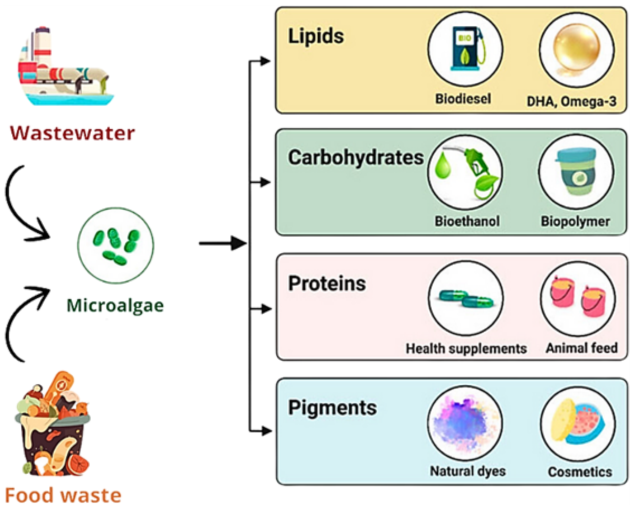

FIGURE 1: Possible applications in industry contexts. Modified from (Srimongkol et al, 2022).

Extremophiles: The Future of Sustainable Production

Extremophilic algae, such as *Galdieria sulphuraria*, are gaining particular attention due to their ability to thrive in harsh environments while providing valuable biochemical compounds. It is a member of the Cyanidiophyceae class which includes unicellular red algae thriving in extreme acidic geothermal settings with high temperatures and low pH. They showcase a unique metabolic versatility allowing for auto-, mixo-, and heterotrophic growth (Ciniglia et al., 2014). Notably, *Galdieria* exhibits remarkable tolerance to toxic metals, enabling its survival in metal-rich environments where other organisms struggle or fail to thrive (Iovinella et al., 2022). Innovative research on these extremophiles highlights their potential to overcome the limitations of conventional algae cultivation. Their resistance to contamination and ability to grow under varied conditions make them excellent candidates for large-scale industrial applications. Additionally, their use in carbon capture and utilization technologies could further solidify their role in the transition to a carbon-neutral Europe.

G. sulphuraria stands out as having commercial potential for remediating challenging wastewaters. Studies (di Cicco et al., 2022) have shown that *G. sulphuraria* is highly efficient in removing nutrients from municipal wastewaters, achieving impressive removal rates for ammoniacal nitrogen (88.3%) and phosphate (95.5%) in large-scale outdoor bioreactors. This extremophile alga emerges as a preferred strain for energy-efficient nutrient removal from urban wastewaters, surpassing other strains in terms of removal efficiencies and rates.

Galdieria cells have shown significant efficacy in recovering various crucial elements, including Rare earth elements (REEs), simplifying the extraction process (Iovinella et al., 2022). REEs, also known as lanthanides, possess unique magnetic and catalytic properties crucial for numerous technologies, including wind turbines, solar panels, batteries, fluorescent lamps, and electronic displays, among others. Traditional methods of extracting lanthanides from ores, such as pyrometallurgy and hydrometallurgy, are not only environmentally damaging but also expensive (Figure 1).

Conclusions

The future of algae production in Europe is undeniably bright. As technological advancements continue to drive efficiency and scalability, the algae industry is ready to become a cornerstone of Europe's sustainable bioeconomy.

However, achieving this vision will require overcoming existing challenges related to production costs, regulatory frameworks, and market acceptance. Collaboration between industry stakeholders, policymakers, and researchers is essential to unlock the full potential of algae in contributing to Europe's ecological and economic goals.

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CONSUMER BEHAVIOUR WITH REGARD TO WASTE SEPARATION IN PUBLIC AND PRIVATE SPACES

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ABSTRACT
Consumer behaviour plays an important part in waste separation. In contrast to private households, consumer behaviour in public spaces, like parks, pedestrian zones and on sidewalks, has hardly been analysed and waste separation in public is scarcely implemented. Recent research on the characterization of public waste indicates that waste from public spaces contains a high proportion of recyclables, which shows that considerations concerning the introduction of separate waste collection are reasonable. This study aims to understand the differences between consumer waste behaviour in private and public spaces. Waste separation behaviour in public spaces is analysed through guided interviews (n=12) and an online survey (n=238) of residents of Vienna (Austria) and is compared to waste separation behaviour in private spaces. The results show that, firstly, social norms regarding waste separation are more established in private households than in public spaces. Secondly, although the total amount of waste generated in public spaces is lower, recyclables (paper, plastic, metal, glass) are relevant waste fractions in public waste and are therefore regarded as important when high resource recovery is pursued. Thirdly, waste separation in public spaces requires more effort on the part of consumers disposing of waste than in private spaces. This is mainly due to the lack of recycling bins. Fourthly, waste separation in public spaces is seen as a lower priority by respondents compared to litter prevention. The results suggest that separation behaviour varies according to the contextual space and cannot be regarded as identical.

1. INTRODUCTION

The rise in packaging materials usages, particularly from online retail and take-out food and drinks, requires a new approach to waste management, embracing the principles of the circular economy (Bilitewski and Härdtle, 2013). To achieve a circular economy, it is crucial to promote waste prevention and reduction, as well as to prioritise separate waste collection over other waste treatments (EC, 2008). Furthermore, the European Union (EU) has mandated that its member states recycle 60% of their municipal waste and 70% of their packaging waste by 2030 (EC, 2018a, 2018b). When it comes to the plastic packaging sector in Austria, there is a significant need for improvement as the recycling rate (currently approx. 25%) needs to increase drastically to achieve a 55% target by 2030 (BMK, 2022; EC, 2008).

Human behaviour and separation habits play a crucial role in waste separation since they greatly impact waste processing and the quality of secondary materials (Kranert, 2017). When analysing waste separation, the literature focuses primarily on household waste (Timlett and Williams, 2008). However, there is a lack of information regarding separation behaviour in other locations, such as public spaces like streets, parks and squares. Research on public waste often focuses only on littering (Al-mosa et al., 2017; Bator et al., 2011; Liu and Sibley, 2004; Schultz et al., 2013) and waste separation behaviour is rarely addressed. However, public waste and household waste exhibit distinct characteristics, indicating a significant potential for recyclables in public waste, particularly packaging waste and disposable products (Kladnik et al., 2024). Public waste is of high quality and quantity for separate collection in highly frequented locations such as pedestrian zones and shopping areas, but is low in green areas and leisure centres (Gangl et al., 2022). As public waste is often collected as mixed waste, its potential has hitherto remained unexploited.

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The aim of the study is to understand the differences in consumer waste separation behaviour in public and private spaces. The results will provide valuable insights into the perception of waste in different contextual spaces and will show how the introduction of waste separation in public should be approached. The study raised the following research questions: What are the motivations behind and obstacles to waste separation in private households and in public spaces? Are there differences in waste separation behaviour in public and private spaces? If so, what are the differences in social norms behind such behaviour, the respective resultant waste fractions, as well as the comparative waste separation difficulties encountered in the public and private spheres? Finally, how is littering as compared to waste separation in public spaces perceived?

The paper is structured as follows: Section 1 presents the factors that influence waste separation behaviour, with a focus on social norms. This is done based on the available literature. Section 2 outlines the methodology and dataset in the study presented, consisting of 12 guided interviews and a questionnaire survey with 238 respondents. Section 3 presents the results of the case study. Finally, section 4 contains the discussion and section 5 consists of the conclusion.

1.1 Factors influencing waste separation behaviour

Public spaces are accessible to everyone, implying that their use precludes the exclusion of others (Corten, 2019). These spaces bring together individuals from diverse backgrounds, often without prior acquaintance. While public spaces evoke a sense of community and togetherness, actual interaction and exchange among individuals tend to remain minimal (Belk, 2017; Jansson, 2011; Kemp, 1999). Even if direct interaction is not particularly pronounced, it still influences behaviour. Goffman (1971) argues that individuals actively adopt different roles. Their behaviour is context-dependent and shaped by norms and expectations, likened to a performance in which individuals present themselves – both verbally and non-verbally – according to the audience and setting (Goffman, 1963a).

Understanding the dynamics of behaviour in public spaces is essential for enhancing waste separation practices. Previous research has mainly focused on separation behaviour in private households. Hence, information on separation behaviour in public spaces is limited. First, the primary factors that influence waste separation in households are summarised. Subsequently, the influencing factors for waste separation in public spaces will be addressed in this section.

According to Knickmeyer (2020), the main social influencing factors in private households are socio-demographic characteristics, psychological factors, economic factors and political background. Psychological factors relate to the perceived comfort and effort of separating waste. The convenience of infrastructure is one of the most important influences to increase household waste separation behaviour and is effective if the following criteria are met (Bernstad, 2014; Knickmeyer, 2020; Miafodzyeva and Brandt, 2013; Zhou et al., 2022): short distances and a strategic location of collection points for recyclables; high frequency of collection; availability of curb-side collection; storage space in households; (smart) visual design of collection points. In simpler terms, the easier it is to use and to access the separation system, the more likely it is to be used (Knickmeyer, 2020). A lack of infrastructure (the availability of bins) presents a barrier for participating in waste separation (Jesson et al., 2014; Timlett and Williams, 2009). Thus, the provision of recycling bins can significantly increase the collection rate (Brothers et al., 1994; McCoy et al., 2018). Furthermore, collecting multiple recyclables, such as plastics and metals, in one recycling bin has been shown to simplify separate waste collection and increase participation rates (Oskamp et al., 1996).

As previously mentioned, there is little knowledge about separation behaviour in public spaces. It is important that the infrastructural factors are kept as low-threshold and simple as possible (Gangl et al., 2022). Leeabai et al. (2019) suggest a threshold distance between 8 and 410 m as relevant for influencing waste disposal behaviour. In the case of residual waste bins in public spaces, individuals use cognitive maps of the environment in order to identify the location of the nearest waste bin (Hartl and Hofmann, 2024). The provision of waste bins and short distances have an impact on reducing littering (Al-mosa et al., 2017; Bator et al., 2011; Schultz et al., 2013). The most frequently discarded items worldwide include cigarette butts, and the impact of takeaway packaging is on the rise (Bator et al., 2011; Castaldi et al., 2021; Schultz et al., 2013). The littering of cigarette butts can be reduced significantly by installing ashtrays and waste bins (Liu and Sibley, 2004). Overall, the studies suggest that simply providing recycling bins, without changing people's attitudes, already leads to better collection rates.

Separation knowledge is also an important factor to consider (Sorkun, 2018; Zhou et al., 2022). Varying separation systems and a lack of knowledge, on the one hand, can act as barriers to separate collection (Jesson et al., 2014). Consistently utilised waste separation systems, on the other hand, have a positive effect over time (Jenkins et al., 2003). Gangl et al. (2022) suggest presenting information in an intuitive manner in order to achieve a subconscious impact. Information should be provided in a simple form: Little text, a large font and photos have a positive effect (Rousta et al., 2015; Sussman et al., 2013). Gamification, for example through waste bins that react with sounds or smileys and reward users, can also promote intuitive behaviour (Berengueres et al., 2013).

This research focuses on social norms. Social norms (synonymously used for `subjective norms´) are the expectations of how to behave and are influenced by significant others, such as family, friends or neighbours (Ajzen, 1991; Miafodzyeva and Brandt, 2013). These norms vary depending on cultural factors (Becker, 2014). There is, as has been mentioned, a lack of research on social norms regarding waste separation in public spaces. The studies which are mentioned in the following focus on private households. It is shown that waste separation behaviour in private households is influenced by social and moral norms (Miafodzyeva and Brandt, 2013; Nguyen et al., 2015; Zhang et al., 2015). In a study by Park and Ha (2014), it can be seen that social norms, together with attitudes, influence participation in separate collection. Past separation behaviour also has a significant influence on current behaviour, which is why habit formation can be seen as promoting separate collection (Xu et al., 2017). If separate collection is socially approved, it will motivate people to take action in that direction (Cialdini, 2003). Role models can be used to guide social norms, which, for example, led to a 42% increase in the separate collection of organic waste in a cafeteria in Canada (Sussman et al., 2013). Sussman et al. (2013) also shows that signage with important information and instructions lead to an increase in the separation behaviour of organic waste of approx. 20%.

While waste separation behaviour in private households is largely shaped by social and moral norms, as well as past behaviour and habit formation, the situation changes when we turn our attention to public spaces. In this context, a key distinction is that public spaces involve the use of common goods, such as waste management systems. The use of common goods creates a social dilemma (Belk, 2017), wherein individual and collective interests can be at odds (Kollock, 1998; Van Lange et al., 2013). As Ostrom and Ahn (2007) note, the challenge of collective action arises from the short-term appeal of free-riding, which can outweigh the incentive to contribute to common goods. Consequently, norms and values shaping behaviour related to public goods reflect a tension between social and personal identity.

The focus of this paper is on waste separation in public spaces. However, since littering is a well-explored topic in the literature, it will be addressed here in order to examine the relevance of waste separation in relation to littering. It should be noted that although littering occurs mostly unconsciously, the perpetrators are aware that it is an antisocial behaviour (Gangl et al., 2022). In clean settings, littering occurs less frequently as it sends a message to refrain from this behaviour (Bator et al., 2011). Based on the study by Sussman et al. (2013), negative role models can promote negative behaviour. With regard to littering, places where littering is already taking place are more likely to encourage further littering (Dur and Vollaard, 2015). Another study by Keizer et al. (2011) deals with the difficulties of a reversal effect with signages: According to the study, if prohibitions signs are located in places that support negative norms, e.g. places heavily affected by littering with an anti-littering sign, fewer people are likely to comply with anti-littering measures. In fact, it triggers a reversal of behaviour, resulting in more littering than in the same situation without a prohibition sign. Therefore, in this case, other measures would be more appropriate than prohibition signs.

The behaviour of consumers regarding littering and waste separation will be explored within this paper, with a focus on the latter. Participation in separate waste collection requires adequate infrastructure, knowledge, and norms. Public waste is often collected as mixed waste, showing that infrastructure is already a barrier to participation. It is worth investigating whether and how consumers perceive this barrier. Furthermore, this study aims to understand the differences in social norms between private and public spaces in order to fill the current knowledge gap.

1.2 Waste collection and infrastructure in the city of Vienna

With a population of nearly 2 million, Vienna is Austria's largest city (StatistikAustria, 2023). It covers an area of 414.87 km² (MA23, 2023) and has a population density of 4,656 inhabitants/km² (MA23, 2022). Vienna attracts over 5 million visitors annually (MA23, 2023). Concerning household waste, separate collection schemes are established. Mixed waste and paper from households are often collected through a kerbside collection system, while glass, lightweight packaging and organic waste are collected at collection points in public areas (about 4.400 public collection points for household waste) (MA48, 2017). The collection, both of private and public waste, is predominately managed by the municipal department MA48 (GEM, 2023). At the time of the study, various municipal solid waste fractions were collected and treated, including mixed waste, paper, glass, lightweight packaging (plastic, metal, beverage cartons) and organic waste. The municipality provides collection services for bulky waste and hazardous waste at disposal centres. However, since bulky and hazardous waste are not relevant to the study presented, these waste fractions will not be further discussed. In total, Viennese households produce 289 kg/cap per year of residual waste (518,500 t) annually, which is significantly higher than the Austrian average of 166 kg/cap (BMK, 2017; MA48, 2017). Examining mixed residual waste characteristics from private households, the proportion of recyclables is as high as 47% (MA48, 2017). Public waste is seldom collected separately and waste amounts are currently largely unknown (Gangl et al., 2022). About 20.800 public waste bins are installed throughout the city (MA48, 2023a), which are manually emptied/collected by street sweepers. In the course of collection, they manually sort PET bottles and drink cans from public bins for mixed waste (MA48, 2017). Public waste is documented separately from other waste streams as street sweepings. According to the city of Vienna (MA48, 2023b), 15,635 t/a of street sweepings (without gravel) were collected in 2020. Keeping the numbers mentioned in mind (relatively high per capita waste production, high share of recyclables in residual waste, high population density and tourism, limited options for separate waste collection in public spaces), the city of Vienna thus constitutes an ideal case study on the need to gain deeper insight into consumer waste separation in public spaces.

2. DATA AND METHODOLOGY

The current research employs a multi method approach to assess consumer attitudes towards waste separation in public, combining both qualitative and quantitative data. This approach helps to balance the limitations of each method (Hammond, 2005). In the following, two studies are presented: First, a qualitative approach was chosen to gain insights via interviews with consumers. Based on these results, a quantitative survey was conducted to test hypotheses regarding the difference between waste separation in private and in public areas.

2.1 Definition of public and private space

Within this study, private spaces are defined as areas that are only accessible to a specific person or group of people, i.e. private households. Households dispose of their waste via kerbside collection and at collection points. As recyclables (paper, plastic, metal, glass) are often collected at public collection points in Austria, the nearest possible collection point for recyclables is considered within this study when private households are addressed. In this study, the term 'public (waste) bins' refers to bins for mixed waste collection, while 'recycling bins' refer to bins for separate waste collection of recyclables. The definitions of public space and public waste are based on Kladnik et al. (2024): Public space is defined as all public areas that are accessible to everyone and that can be cleaned by the local street cleaning services (e.g. pedestrian zones, streets, parks, public squares, etc.). This definition excludes semi-public spaces, such as enclosed spaces (e.g. museums, public institutions, etc.), and public spaces that require payment for use (e.g. cafes, amusement parks, etc.). In this sense, public waste is defined as waste collected from waste bins in public spaces.

2.2 Guided interviews

Semi-standardised interviews were conducted face-to-face in January 2023 with 12 participants (see details in the supplements). The sample size was selected to achieve theoretical saturation, which refers to obtaining the key insights without requiring additional interviews for further information, as recommended in the literature (Przyborski and Wohlrab-Sahr, 2021). The sampling was conducted to approximate the demographic characteristics of Vienna by interviewing people from a diverse range of districts in Vienna. The aim of the semi-structured interviews was to understand behavioural patterns regarding waste separation. The interview guideline covered four themes: personal attitudes towards waste separation, waste separation in private spaces, waste separation in public spaces, and solutions for improvement. The interviews were recorded and transcribed using the program Amberscript. The MAXQDA program was used to analyse the data using a qualitative content analysis according to Mayring (2002), with coding based on Flick (2007) and Clarke and Braun (2017).

2.3 Questionnaire survey

A total of 238 respondents participated in the online and offline questionnaire survey conducted from March to May 2023. The sample was carefully selected to represent the population of Vienna in terms of gender, age, and education level. To be eligible, participants had to have lived in Vienna for at least one year and had to be at least 18 years old. The survey was initially distributed to a wide circle of acquaintances using the snowball principle. Additionally, printouts for the survey (with a QR code) were distributed in residential areas and busy public places, with respondents in the latter areas questioned in person. This was done to ensure accessibility to all population groups, including those who may have difficulty accessing online formats such as older people and those with lower levels of education. Table 1 summarises the characteristics of the sample.

TABLE 1: Demographic information of respondents: questionnaire survey.

Variables	respondents (n=238)		Population in Vienna*
	n	%	%
Gender			
Male	109	45.8	48.9
Female	121	50.8	51.1
Divers	8	3.4	n.d.
Age (year)			
18–29	80	33.6	20.5
30–39	42	17.6	19.6
40–49	30	12.6	16.4
50–59	25	10.5	16.6
>60	61	25.6	26.8
Average age	43.3 years		41.2 years
Level of education			
Compulsory school/ no school education	15	6.3	21.2
Secondary school (no qualification for university attendance)	10	4.2	8.9
Apprenticeship	22	9.2	20.7
AHS/BHS/College (with qualification for university attendance)	53	22.3	19.7
University of Applied Sciences/University	138	58.0	29.5

** Source: MA23, 2023*

The survey consisted of four topic groups containing 30 questions in total (see details in the supplements), followed by an open question for further comments. The topic groups were: social norms, waste separation in private and public spaces, waste separation and difficulties, waste in public spaces.

In the topic group "social norms", participants were asked about their perception of waste separation in society, in private and public spaces, with respect to five items: "*My family thinks I should separate waste*", "*My friends think waste separation is a good thing*", "*My acquaintances believe I should separate my waste*", "*It is socially expected that people separate waste*", and "I *have the feeling that people in Vienna should separate their waste*" (5-point Likert scale from 1 ("I totally disagree") to 5 ("I totally agree")).

In the next group, participants were asked to report the types of waste they had produced and actively separated, in private and public spaces, during the previous week with two items: "*What waste was produced last week?*", and "*What waste did you actively separate last week?*" (Multiple-choice options included "residual waste", "paper", "glass", "metal", organic waste", "plastic" and "other").

In the topic group "waste separation and difficulties", the following five items were included, concerning private and public spaces: "*I don't have time to separate waste*", "*I don't worry about waste separation and dispose of my waste in the residual waste*", "*There are enough separation options to separate waste*", "*The available separation options cover all types of waste that I could separate*", and "*It takes a long time to find the next separation option*" (5-point Likert scale from 1 ("I totally disagree") to 5 ("I totally agree")).

In the last topic group, participants were asked with six items, concerning only public spaces: "*It bothers me when rubbish lies on the ground or in the environment*", "*It is important to me that my surroundings are tidy and clean*", "*If I can't find a rubbish bin, I take my rubbish home with me if necessary*", "*I separate my rubbish as best I can, but I don't force myself to do so*", "*I keep recyclables with me until I find a way to separate them*", and "*If I can't find a separation option in public, I take recyclables home and separate them there*" (5-point Likert scale from 1 ("I totally disagree") to 5 ("I totally agree")).

Demographic variables (sex, age, education, net income, number of household members, number of underage children) were assessed at the end. To avoid any misunderstandings, each thematic group begins with definitions of private and public space.

Based on the results of the guided interviews, the following hypotheses (H) were derived which were tested for significance:

- H1. People feel more obliged to separate their waste in private spaces than in public spaces.
- H2. In public spaces, mainly residual waste accumulates, while in private spaces, other waste fractions are also relevant.
- H3. Separating waste in public spaces requires more effort than in private spaces.
- H4. People in public spaces prioritise not leaving waste in the environment over separating waste.

The software program SPSS was used for statistical data analysis. The hypothesis tests were performed using a Wilcoxon test. Prior to this, the reliability was checked using Cronbach's alpha, which ideally should be above 0.7 (Field, 2009). The Cronbach's alpha values were satisfactory: Cronbach's alpha for social norms with 10 items = 0.827, for waste separation and difficulties with 10 items = 0.794, for waste in public spaces with 5 items = 0.750. The item "*I separate my rubbish as best I can, but I don't force myself to do so*" from the last topic group (waste in public spaces) had to be excluded due to a low Cronbach Alpha. The effect size was calculated according to Field (2009) to determine whether the effect of the results is substantial, which is the case when the effect size exceeds 0.5.

3. RESULTS: CASE STUDY IN THE CITY OF VIENNA

3.1 Guided interviews

When discussing separate waste collection, the interviewees mainly referred to waste separation in private households. The interviewees believed that separate waste collection is important for several reasons. The most commonly mentioned reason is its potential for resource reuse and recovery, which is why material recycling is generally preferred over energy recovery. Interviewees associate energy recovery, such as waste incineration, with the loss of resources and environmental pollution, while material recycling is viewed as a way to conserve resources and keep them in the cycle. Another reason mentioned to support material recycling is the prevention of harmful chemicals/hazardous waste. Most interviewees separate their household waste into four categories in private spaces: residual waste, paper, lightweight packaging (plastic and metal), and glass. However, fewer than half of the interviewees collect organic waste separately at home due to a lack of collection bins available at kerbside or at collection points. The municipality also offers disposal centres for the collection of, e.g. bulky and hazardous waste. However, as these waste streams are not relevant to this paper, they will not be addressed.

Based on the interviews, the waste separation behaviour of consumers can be grouped according to the following four attitudes and practices concerning household waste: First, separate waste collection is often integrated into daily life. People combine the journey to public collection points with their errands, such as the journey to work for saving time. Therefore, the location and distance from households to public collection points for recyclables are important. This leads to the second factor, the time aspect. The interviews reveal an ambivalent attitude towards the time required for separate waste collection in private households. While most interviewees consider the time required to be low, it is also speculated that it may be the reason why some people do not participate in separate collection. Third, separate waste collection in private households can be challenging due to overfilled common household containers, especially for waste paper, and a lack of bins for organic waste collection. Fourth, interviewees report collecting waste fractions that can be disposed of through kerbside collection or at nearby public collection points for recyclables. Thus, consumer understanding of the separate collection system relies heavily on both the presence of different waste collection bins and the corresponding labelling on the bins. This is consistent among interviewees who have changed residence or come from other countries. Kerbside collection is the preferred collection method for organic waste. The collection method for organic waste depends on the respective district, that is, on whether waste can be disposed of at a kerbside or must be brought to collection points. According to some interviewees, they stopped collecting organic waste separately when kerbside collection was no longer available, e.g. after moving. This emphasises how appealing kerbside collection is to consumers. In general, the majority of interviewees expressed satisfaction with their waste separation practices at home. However, they recommended increasing the number of paper collection bins in private households and providing kerbside collection for organic waste.

These findings on waste separation in private households differ strongly from waste separation in public spaces. While the interviewees take separate collection in private households for a given, they seem to be surprised to be asked about their waste separation behaviour in public spaces as they do not practise separate collection there. This is also depicted in the number of codes used for the qualitative content analysis: A total of 410 segments of the interviews were coded for separate waste collection in private spaces. In comparison, only 196 coded segments related to public spaces. Additionally, interviewees were not aware of the differences in their separation behaviour in private and public spaces. Overall, interviewees reported generating minimal waste in public spaces, which they typically disposed of as residual waste, regardless of how meticulously individuals may separate it at home. If at all, waste is typically only separated in public areas where nearby recycling bins are available.

There are several reasons why people fail to separate waste in public. Firstly, interviewees attribute their failure to a lack of recycling bins as well as to time constraints. Consumers find it inconvenient to separate waste in public and they expressed reluctance to expend additional effort in searching for recycling bins to separate their waste due to time limitations. Information about the location or distance to recycling bins is not easily accessible, while mixed waste bins are more visible to consumers. Therefore, the use of mixed waste bins appears more convenient, and interviewees report that these are satisfactory in terms of both quantity and distance. Secondly, preventing littering in public by disposing of waste in (mixed waste) bins is conceived as crucial to interviewees, indicating that separate waste collection is not seriously considered in public areas. Interviewees emphasised that they never leave their waste on the ground or in the environment. They always use public waste bins that are usually for mixed waste. In general, interviewees expressed a wish for more recycling bins in public spaces to facilitate separate waste collection. The idea of signs indicating the location of separate waste collection facilities was viewed positively.

Figure 1 visualises the key findings from the guided interviews. The findings from the guided interviews raise questions and indicate distinctions made between private and public spaces. These differences were further investigated in a quantitative survey that focused on social norms, efforts required to separate waste, and the perception of waste in public, using the hypotheses outlined earlier (see chapter 2.3.).

3.2 Questionnaire survey

The four hypotheses (H) defined were statistically tested for their significance. In order to test H1, whether people feel more obliged to separate their waste in private spaces than in public spaces, a statistical summary of data gathered from the survey was compiled to assess that claim (Table 2). As the data are not normally distributed, a Wilcoxon test was used. Social norms, favouring separate waste collection as a socially accepted behaviour, are significantly higher in private spaces (median = 4.40) than in public spaces (median = 4.00; asymptotic Wilcoxon test: $z = -9.138$, $p < 0.001$, $n = 238$ - T-test: $t(237) = 9.499$, $p < 0.05$). The effect size is 0.592, indicating a strong effect (Field, 2009). Therefore, this research shows that there is a significant difference in social norms regarding waste separation in private and public spaces. The findings suggest that people feel a greater responsibility to separate their waste in private settings than in public ones. Public waste separation norms seem less established than those in private households.

To test H2, respondents had to select the waste fractions they generate and actively separate. This was to test

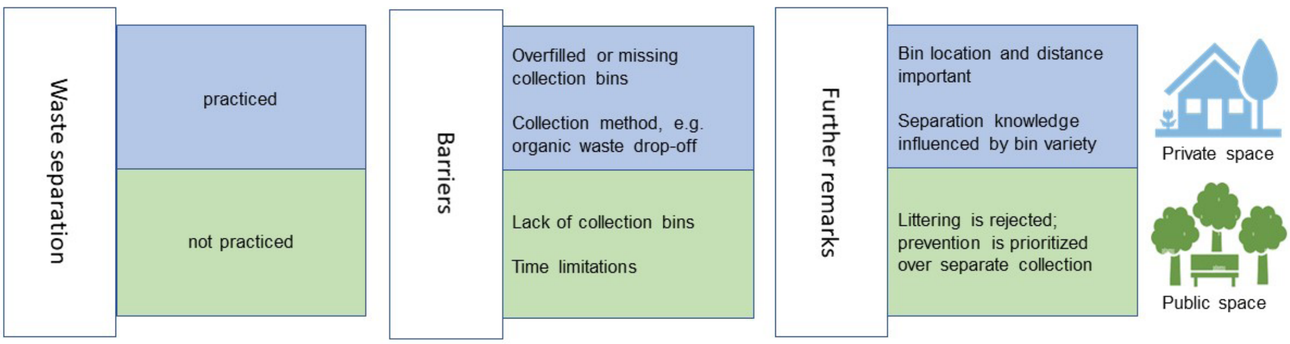

FIGURE 1: Key results from guided interviews for private (blue) and public (green) spaces.

TABLE 2: Descriptive statistics for respondents' ratings for hypothesis H1, H3 and H4. Respondents' ratings are based on a 5-point Likert scale from 1 ("I totally disagree") to 5 ("I totally agree").

Option	Mean	Median	Standard error	Standard deviation
H1: People feel more obliged to separate their waste in private spaces than in public spaces				
Private space	4.32	4.40	0.039	0.601
Public space	3.84	4.00	0.051	0.786
H3: Separating waste in public spaces requires more effort than in private spaces				
Time				
Private space	1.36	1.00	0.036	0.551
Public space	2.37	2.50	0.060	0.920
Waste separation options				
Private space	2.39	2.33	0.061	0.946
Public space	3.41	3.67	0.058	0.901
H4: People in public spaces prioritise not leaving waste in the environment over separating waste				
Littering	4.31	4.47	0.036	0.556
Separation	3.06	3.00	0.070	1.078

whether there was a difference in waste fractions between private and public space. This hypothesis was chosen because many interviewees reported that residual waste was their main waste fraction in public, while in private household's other waste fractions were also relevant. Table 3 indicates that all waste fractions were selected by the respondents. The difference is that fewer respondents chose waste fractions relevant for separate waste collection in public than in private spaces. Figure 2 summarises the self-reported collection rate, reflecting the ratio of waste generated to waste collected. It becomes evident that the collection rate for all waste fractions is higher in private spaces.

The descriptive statistics disprove hypothesis H2. As anticipated, apart from mixed waste, also other waste fractions, such as paper, plastic, organic waste, glass and metal are as relevant in public spaces as they are in private spaces. Therefore, separate waste collection in public spaces can be regarded as potentially reasonable. The difference between private and public spaces also lies in the collection rate (which was here defined as the ratio between reported waste separated and reported waste generated). For household waste, the collection rate was found to be higher than for public waste, meaning that more respondents claimed to separate their waste. In terms of waste generation in private spaces, the order (from highest to lowest) is paper, plastic, organic waste, glass, and metal. The order of separation in private spaces (from highest to lowest) is paper, plastic, glass, metal, and organic waste, which is similar for the generation and separation of waste in public spaces. Organic waste has the lowest collection rate for both private and public space. It is important to note that the collection rates cannot be directly compared to the waste management municipality's data: the survey refers to the number of respondents that selected waste fractions they generated and actively separated, while the other refers to the quantity collected by the municipality.

In order to test H3, whether separating waste in public spaces requires more effort than in private spaces, the items of this construct are categorised into two groups based on the underlying concept: the items "*I don't have time to separate waste*", "*I don't worry about waste separation and dispose of my waste in the residual waste*", "*If I can't find a rubbish bin, I take my rubbish home with me if necessary*" pertain to time, and the items "*The available separation options cover all types of waste that I could sep-*

TABLE 3: Descriptive statistics for respondents' answers for hypothesis H2, presenting the number of answers per waste fraction generated and per waste fraction separated in both private and public spaces.

Option	Paper	Plastic	Organic waste	Glass	Metal
	n*	n	n	n	n
H2: In public spaces, mainly residual waste accumulates, while in private spaces, other waste fractions are relevant.					
Waste fraction generated					
Private space	231	224	207	196	171
Public space	124	125	67	44	42
Waste fraction separated					
Private space	228	195	88	184	153
Public space	68	73	10	31	27

** Number (n) of total respondents = 238*

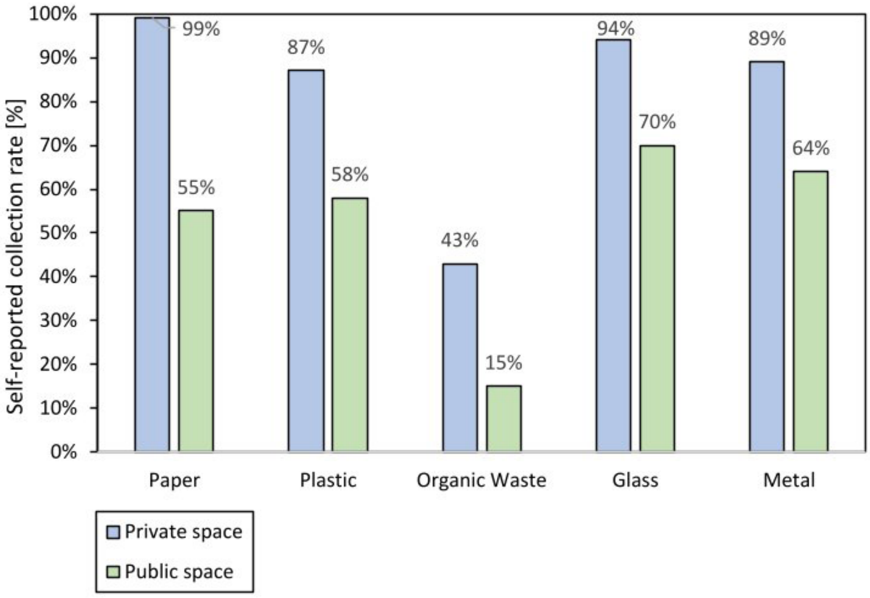

FIGURE 2: Self-reported collection rates in private and public spaces based on the questionnaire survey (n=238). The collection rate reflects the ration of separated waste fractions to the total collected waste fractions.

arate", "*It takes a long time to find the next separation option*" assess the perception of waste separation options. The statistical data is summarised in Table 2.

The items concerning time are significantly lower in private spaces (median = 1.00) than in public spaces (median = 2.50; asymptotic Wilcoxon test: $z = -11.357$, $p < 0.001$, $n = 238$ - T-test: $t\ (237) = -17.657$, $p < 0.05$) indicating that consumers have more time to separate their waste in private households. Using recycling bins for separate waste collection is associated with more effort in public spaces (median = 3.67) than in private spaces (median = 2.33; asymptotic Wilcoxon test: $z = -10.972$, $p < 0.001$, $n = 238$ - T-test: $t\ (237) = -14.930$, $p < 0.05$). The effect sizes indicate a strong impact for both time (0.736) and separation options (0.711) (Field, 2009). This supports the hypothesis that waste separation requires more effort in public areas than in private households. More effort is required in public areas as time is a limiting factor and fewer recycling options are available, both in terms of bin quantity and accessibility.

To test H4, whether people in public spaces prioritise not leaving waste in the environment over separating waste, the items were categorised into two groups: the items "It bothers me when rubbish lies on the ground or in the environment", "It is important to me that my surroundings are tidy and clean", "If I can't find a rubbish bin, I take my rubbish home with me if necessary" deal with littering, while the items "I keep recyclables with me until I find a way to separate them", "If I can't find a separation option in public, I take recyclables home and separate them there" pertain to separation facilities. As mentioned before, one item was excluded to ensure reliability. The statistical data is summarised in Table 2.

The relative importance of preventing littering is significantly higher (median = 4.47) than the separation of waste in public places (median = 3.00; asymptotic Wilcoxon test: $z = -12.356$, $p < 0.001$, $n = 238$ - T-test: $t\ (237) = 19.374$, $p < 0.05$). The effect size indicates a strong effect, with 0.801 (Field, 2009). Individuals consider it more important to avoid leaving their waste in the environment than to separate it. Respondents emphasise that littering is not appreciated and can be disruptive. In public spaces, the main objective is to prevent littering.

4. DISCUSSION

Several implications can be derived from the study presented: First, waste separation is typically associated with private households rather than public spaces. Questions about waste separation behaviour in public may even cause surprise. Although individuals are aware of the importance of waste separation and practice it meticulously at home, this behaviour is not always transferable to public spaces. The reason for this difference may be related to the lack of recycling bins. The availability of recycling bins clearly suggests which types of waste should be separated (Knickmeyer, 2020). The absence of public recycling bins thus suggests that separate collection is not a priority. Therefore, a shift in social norms together with improved bin availability is necessary to demonstrate the equal value of separate collection in both areas. Second, to promote separate waste collection in public, it is important to provide user-friendly waste separation facilities and to increase convenience. The biggest obstacle is the lack of recycling bins in public spaces. Another hindrance is the lack of knowledge about the locations of recycling bins. Offering the necessary infrastructure to participate in waste separation is a key factor in encouraging consumer waste separation behaviour (Bernstad, 2014; Knickmeyer, 2020; Miafodzyeva and Brandt, 2013; Zhou et al., 2022). Thirdly, consumers are often unaware of the waste they generate in public spaces. This study observed that people struggle to consciously recognise and name the different types of waste they accumulate in public. The contributing factors to this lack of awareness are unclear, but one suggestion is that the relatively low amount of waste generated in public spaces may distort the perception of waste types. Fourthly, waste separation is not a priority in public areas. To prevent littering, consumers use the available waste bins, which are often for the collection of mixed waste. Consumers see public waste bins as collection possibilities for any type of waste and not only for residual waste.

Noticeable linguistic anomalies suggest a distinct view on waste in public spaces: The interviewees distinguished between separate collection for recyclables and the collection of residual waste when talking about household waste. For public spaces, they referred to 'normal' waste bins, which are actually waste bins to collect mixed waste/ residual waste. This suggests that throwing waste into residual waste bins is considered the norm in public and that separate collection may not be practiced. Regarding public waste, some respondents stated that they 'simply' dispose of their waste in public waste bins, indicating that this is an intuitive behaviour and that separate collection is perceived as difficult and unusual.

This study focuses solely on the city of Vienna. Therefore, the results are geographically limited and cannot be generalised to other areas, as waste management systems and social norms may differ. However, the city of Vienna encompasses different relevant factors, which make the city an ideal location for such an analysis: There are differences in municipal solid waste generation between urban and rural areas. For instance, Vienna's annual residual waste generation is 74% higher (289 kg/capita) than the Austrian average (166 kg/capita) and household waste is separated less frequently in Vienna compared to other regions in Austria (BMK, 2017; StatistikAustria, 2017). Urban residents generate more waste due to their distinct consumption habits (Secondi et al., 2015). Further, the willingness to engage in pro-environmental behaviour depends on the context, such as living spaces, leisure and recreational activities, and work-related activities (Barr et al., 2011; Miao and Wei, 2013). Within the city a vast number of contextual spaces and activities are present and practiced.

Another limitation of the study is the methodology used to analyse consumer behaviour which relies heavily on self-reported data. For instance, the questionnaire survey may be subject to recall bias. Given the low level of waste generation in public spaces, respondents might find it challenging to accurately remember their waste separation activities from the previous week as it is asked in the questionnaire survey. This can lead to uncertainties in the results. Respondents may as well have difficulty identifying the correct waste fractions and their contribution due to a lack of awareness regarding the waste generated, especially in public spaces.

Moreover, the reliance on guided interviews and questionnaire surveys means that the accuracy of the results is dependent on the honesty of the respondents, without any means of checking the reliability of their answers. The results from self-reported data may be idealised for several reasons. Respondents may select their answers in favour of waste separation due to social desirability or to avoid the potential effects of stigma (Goffman, 1963b). Although, there is no evidence of systematic bias in self-reporting (Kormos and Gifford, 2014), and research has shown that the correlation between social desirability and self-reported pro-environmental attributes is low or non-existent (Kaiser et al., 1999; Milfont, 2009), the validity of self-reported data can vary (Kormos and Gifford, 2014). Despite efforts to minimise bias within this study, such as employing self-administered questionnaires and ensuring participant anonymity to reduce social pressure and social desirability bias (Kormos and Gifford, 2014), self-reported data without triangulation should be interpreted with caution and critically considered within the context. Incorporating observational studies to validate the results would further enhance the robustness of the study. However, due to time and cost constraints, such methods were not feasible for this research.

Additionally, both methods involved using a sample group consisting of individuals with a high level of education, which may introduce a sampling bias as it does not represent the diversity of Vienna`s population. The study would benefit from a larger and more representative sample to ensure comprehensive insights. Considering that the interviews predominantly included individuals who separate their household waste, it would also be beneficial for further studies to incorporate a focus group comprising individuals who do not engage in waste separation at all. Conducting interviews with this group could provide valuable insights into differing attitudes and behaviours, thereby enriching the findings. However, reaching out to this focus group may prove challenging, as their behaviour contrasts with socially desirable norms.

5. CONCLUSIONS

The literature commonly describes waste separation behaviour as uniform. However, this study shows that separation behaviour can vary across locations. The current study analysed consumers' waste separation in private and public spaces using guided interviews and an online survey. The findings indicate significant differences in separation behaviour:

a. Participation in separate waste collection is more established as a social norm in private households than in public areas.
b. Although less waste is generated in public areas, recyclables from such areas are still relevant waste fractions. Therefore, it is reasonable that recyclables in public spaces are collected separately and not as mixed waste.
c. Participating in public separate waste collection can be more complex for consumers in public areas than when in their respective private households, which clearly constitutes an obstacle to separate waste collection.
d. Respondents prioritise preventing littering over separate waste collection in public spaces.

Recovering recyclables is preferable to incineration and can contribute to increasing recycling rates in line with the Circular Economy Package. Recycling bins should not be randomly placed, but strategically placed in public areas where it can be economically and ecologically justified. Although requiring appropriate infrastructure, public squares and areas with high foot traffic are regarded as ideal for introducing separate collection in public, as these areas have a high potential for recyclable materials (Gangl et al., 2022). Besides offering appropriate recycling bins it is clear from the study that initiatives to foster the social norm for waste separation in public spaces are necessary. This could be achieved, e.g., through informational campaigns on the bins themselves. For purposes of future research and public policy, it should be noted that respondents in this study indicated that they were not aware of the location of recycling bins in public spaces, which prevents them from participating in separate collection. It would be worthwhile to investigate whether this also applies to public spaces in the respondents' neighbourhoods. In addition to the lack of information about the location of public recycling bins, it would be worthwhile to investigate the effectiveness of signage and other means of indicating separate collection facilities in public areas. Furthermore, it remains unclear whether consumers are aware that public waste bins are intended merely for residual waste collection. It is unknown whether their separation behaviour in public would change if they possessed such knowledge.

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FOOD WASTE CRISIS IN THE UNITED STATES OF AMERICA: THE KNOWN, THE OVERLOOKED AND THE UNDISCLOSED

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ABSTRACT

With a loss of 1,250 calories per day per person or 140 kg/p/yr, the United States (U.S.) has a large problem with food waste. The later a product is wasted in the supply chain, the greater its environmental cost. Many food waste mitigation efforts focus on the retail and consumer stages, but there is also considerable food waste in the production phase, which includes growing, harvesting, transportation, storage and processing. The study is conducted in two steps: a bibliometric analysis of 218 articles on food waste in the U.S., followed by an investigation of options to meet food waste recovery targets. The study has identified three options to reduce food waste that are partially based on the bibliometric analysis. The options, Secondary Retail Food Stores, Enhanced Tax Credits, and a Task Force, were evaluated based on cost-effectiveness, diversion from landfills, and waste reduction using the Criteria Analysis Matrix method. The study found that establishing a multi-modal, multi-directional non-governmental Task Force to create awareness and spread knowledge among stakeholders, mainly consumers was the preferred policy alternative to tackle food waste issues in the U.S.

1. INTRODUCTION

Food waste is distinct from food loss. Food waste is food produced for human consumption that was not consumed. Food loss is the change in overall edible material originally intended for human consumption. Food waste occurs in the retail and consumer stages; however, food loss occurs in the agriculture, post-harvest, and processing stages (Thyberg & Tonjes, 2016). One estimate was the United States (U.S.) produced the fifth largest amount of household food waste at 19.3 million tonnes (MT), which was less than China (91.6 MT), India (68.8 MT), Nigeria (37.9 MT) and Indonesia (20.9 MT). The U.S. had an additional 80 kg/p/yr of retail and food service waste, which has been estimated to be 40% of all food waste worldwide (UNEP, 2021). That translates to a per capita estimate of U.S. household food waste of 140 kg/p/yr for all food waste, which is smaller than some other estimates of approximately 175 kg/p/yr by EPA (2020a) or 200 kg/p/yr by Natural Resources Defense Council (NRDC) (Gunders & Bloom, 2017). The total amount of food waste and food loss post-harvest, excluding food left in the fields, was estimated to be 60 MT in 2010 (Buzby et al., 2014), which translates to approximately 200 kg/p/yr out of a total food production of 200 MT or 625 kg/p/yr. This suggests that food loss in the U.S. is greater than 30% of all food production; NRDC sets the total overall at 40% of all food production (Gunders & Bloom, 2017). In a global context, food loss is thought to be substantially lower for developed nations like the U.S. (Thyberg & Tonjes, 2016; Gunders & Bloom, 2017). A UNEP report (2017) found that per capita household food waste was greatest for low-income countries. The report found that food harvesting, distribution, and preservation may not be as effective in developing countries as in developed nations. This occurs even if overall food availability is lower. Although food loss and waste are acute problems in the U.S., they are also a worldwide concern of similar or even greater scope.

Food loss and food waste have microeconomic and macroeconomic consequences. At the national scale, food loss impacts the economy because it reflects ineffective production and distribution processes. In contrast, at the individual scale, food waste has a direct effect on household expenses, both through buyer behavior and resource access. Food loss and food waste may account for 1% to 1.5% of the U.S. GDP or approximately $200 billion (Gunders & Bloom, 2017). There are many reasons for food waste, including spillage or breakage, destruction during transportation or handling of product, or waste produced in the distribution phase, and management of food in the retail sector, preparation and serving of food in restaurants and other service venues, and choices made by consumers as they prepare, serve, and manage the food they bought and

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brought home (Schultz, 2017). An especially troublesome source of food waste is that approximately 80% of U.S. citizens dispose of good and edible food products due to their misunderstanding of the "best used by," "sell by," or "use by" labels (EPA, 2020a). There are at least 50 alternate kinds of food date labeling approaches in the U. S. (Kavanaugh & Quinlan, 2020). Except for infant formula, no regulations require food dating (NYSDA&M, n.d.). Instead, a system of open and closed food dating is voluntarily adopted by manufacturers, distributors, and retailers. Essentially, open dating is used for stock rotation and consumer information; closed dating is used for batch identification, to support product recall, and other product management (Newsome et al., 2014). Labels on food items often confuse consumers as they try to avoid illness from consuming "spoiled" food. About 80% of Americans discard food items based on the labels, although about 40% to 50% report they understand the meaning of food labels (Kavanaugh & Quinlan, 2020). This reflects large quantities of food are being incorrectly discarded due to an inaccurate understanding of the label guidelines. Use by, best by, and sell by labels are quality guides; they are not determinants of food safety (Newsome et al., 2014). Hence, discarding food based on these labels disposes of perfectly edible food items.

There is a conundrum, "food insecurity," that affects one in seven American households (REFED, 2016). Food insecurity refers to a situation where households lack access to sufficient and nutritious food or reliable supplies of culturally appropriate food (Barrett, 2010), which underscores the seriousness of food loss rates of 30% or more in the U.S. For those households with food insecurity, food waste is an immediate and troublesome issue. However, given that food waste amounts to 1% to 1.5% of the U.S. GDP, it is a relatively trivial economic issue for most Americans as it only impacts a relatively small amount of the overall household budget. Food waste is thus overlooked as it does not impact most people's daily lives significantly. Therefore, there may be little impetus to reduce food waste or to change habits that lead to the generation of food waste (Gunders & Bloom, 2017).

Food waste has several environmental impacts, such as excessive water and land use change from misapplied agricultural effort in food production, wasted energy in food processing and transportation, and greenhouse gas impacts and methane generation in landfills, among others (Gunders & Bloom, 2017). About 34% of human-related methane emissions in the world accrue from food waste (Buzby et al., 2014). These methane emissions have 21-34 times more impact on global warming than the equivalent amount of carbon dioxide on a 100-year time horizon (Chai et al., 2016). It is estimated that unconsumed food in the U.S. is derived from more than 50 million hectares of agricultural land (16% of U. S. agricultural land), utilizes 20 trillion liters of water, and nearly a billion kilograms of fertilizer, and results in 150 million MT CO_2eq. of greenhouse gas emissions (land emissions excluded) (Read et al., 2020). The ecological impact of food waste is especially concerning since the growth in population and changing consumption patterns are likely to increase over the coming years. This will substantially increase the food demand, further escalating the burden on food systems and the environment.

In 2015, the U.S. government announced plans to reduce food waste by 50% by the year 2030 from a 2010 baseline (EPA, 2021). However, according to all estimates (Gunders & Bloom, 2017; USDA, n.d.-a; EPA, 2020b), the amount of food waste in the U.S. keeps rising. There are, however, additional plans by the U.S. government to reduce food waste, such as social marketing toolkits and the Excessive Food Opportunity (EFO) map. Toolkits are designed to guide the government, non-governmental organizations, and society in minimizing food waste and facilitating composting efforts by providing them with targeted planning approaches to improve composting, minimize contamination, and roll out behavior change initiatives (EPA, 2024a). The EFO interactive map facilitates the nationwide diversion of surplus food from landfills by identifying surplus food generators, compost feedstocks, infrastructure gaps and landfill alternatives (EPA, 2024b).

This research was conducted through bibliometrics to determine recent academic trends in food waste research. These findings would then be applied to identify ways to reduce food waste in the U.S. and evaluate which options might be best to implement using the Criteria Analysis Matrix method. Establishing an organized approach to information dissemination across stakeholders best met the evaluation criteria of cost-effectiveness, overall food waste reduction, and food waste source reduction.

2. MATERIAL AND METHODS

This study was accomplished through two primary steps.

Step 1: A bibliometric analysis following Djeki et al. (2022) and Donthu et al. (2021) of the Web of Science database was conducted for food waste in the United States from 2005 to 2023. Analysis and generation maps were retrieved from VOS viewer software. VOS viewer was selected to create a broader and more graphic analysis of the results. Through the VOS viewer, it is possible to construct connections between the bibliometric results. The keywords search used were "Food waste" and "United States." The systematic review of the literature was carried out based on the following steps: criteria establishment for selecting the articles, defining keywords and examining strategies, refining articles and sorting possible exclusions, analyzing titles, keywords, and abstracts of the articles, tabulating and evaluating the data and finalizing the database. The term "United States" was used instead of "US" or "USA" to improve accuracy in the number of relevant studies for the bibliometric analysis. Publications were refined by limiting the articles to subject areas, namely, environmental science, social sciences, engineering, agricultural and biological sciences, energy, economics, business management, decision sciences, etc. The results were then advanced by applying filters to different criteria, such as "final" articles for the publication phase, "journal" and "review" for the source, and the articles' medium of language only in English. A set of 218 articles was generated after QA/QC processes.

Step 2: Key terms from the bibliometric study suggested areas of recent research interest in the food waste field. These helped determine three broad areas to address food waste: 1) direct management by the government of food waste; 2) private sector actions to improve food management by changing consumer behavior; and 3) indirect government activities to change industry and consumer behaviors. An exemplar was selected for each broad area to be evaluated. The evaluation criteria were chosen to be cost-effectiveness, food waste recovery, and source reduction. Data were collected, and a Criteria Analysis Matrix was used to evaluate the three policy options. A Criteria Analysis Matrix ranks policy options over different criteria on a scale of 1-5, with the criteria also being weighted from least to most important (in this case, from 1-3). This enables a quantitative assessment of disparate options.

2.1 Selection of policy alternatives

The bibliometric study suggested that besides the obvious terms of the United States and food waste, the more descriptive terms sustainable consumption, reduction, and management were important concepts in recent food waste research in the U.S. Sustainability implies a long-term horizon for actions and a focus on environmental consumption implied a focus on household food waste, as that is where food consumption predominantly occurs. Reduction implies that solutions other than alternative technologies for the management of food waste would be important so that overall food waste would be less rather than only seeking to reduce the environmental impact of management. Management implied identifying specific actors that were involved in food waste should be a goal. In the U.S., policies that impact the public are usually initiated by the government, even though there are always exceptions. We therefore thought that two of our three alternatives should be government-originating policies. For balance, we thought one initiative should originate from and affect the private sector. Labeling clearly was an element that affected household food waste, so that should be one target for our policies. Labeling would be a waste-reduction activity, as well. Food waste composting is a management technique that has been of growing interest over the past several decades, but it is a technological solution, not a reduction element, so it was of lesser priority. Activities that bridged public decisions with private actions also seemed to have a certain appeal.

Therefore, we sought policies in scientific, technical, and gray literature that might illustrate these goals. One common governmental action is to ban a deleterious activity. There have been nine state food waste disposal bans enacted since 2014 (Anglou et al., 2024); although some of the prohibitions require following a management hierarchy oriented towards food waste minimization, most of the food waste bans see composting and anaerobic digestion as the primary means of managing food waste. The only effective food waste ban has been in Massachusetts, which Anglou et al. (2024) attribute to enforcement actions and a widespread, large network of compost sites. Another common action is to create a tax incentive. There is an existing federal tax incentive for businesses that donate food. This was first passed in 2005 and was enhanced in 2015 (REFED, 2016). It allows businesses donating food to charities to deduct up to 15% of taxable income. The 2015 enhancements allowed businesses to claim more than the cost value of the food. REFED (2016) has proposed specific enhancements to the tax credit (technical tweaks to cover some unavoidable costs). Another expansion could be for states to create their own tax deductions or credits, as some states have done with clean energy projects (Milne, 2017). California, Colorado, Iowa, Maryland, Missouri, New York, and Pennsylvania offer tax credits for donated food; Arizona offers a tax deduction (National Gleaning Project, 2023). Ensuring that the federal tax credit covers all costs associated with food donation and supplementing the federal program with state credits and donations should make food donation more enticing for retail food stores and farms (NRDC, 2021). Donating food to charities (food banks and soup kitchens) would clearly be a step toward reducing food waste at the retail level.

Food waste management requires public engagement since the largest share of food is wasted by individuals in households (UNEP, 2017). Public education for waste management purposes has long been espoused as a necessary element of achieving better management; however, the past 20 years have mainly seen stagnant recycling rates (at below-optimal levels) in the U. S. despite ongoing education efforts (MacBride, 2012). Newer paradigms for engagement seem to be warranted. One is exemplified by the Center for Sustainable Materials Management, a State University of New York Environmental Science and Forestry (SUNY-ESF) research and outreach group with a focus on improving New York State recycling. It is largely funded by New York State (https://www.centerforsmm.org/). It has a somewhat traditional education element, with a website, outreach and education staffers, and a social media presence. However, it is not only looking to reach out to the general public but also to the governments in New York that plan and manage solid waste programs. It not only developed a GIS-based web portal where individuals can determine what recycling rules apply based on their phone's location but also has worked to harmonize those rules across the hundreds of New York jurisdictions that set recycling rules. It has developed a database of suppliers that can provide "green procurement" materials to local governments and supports seminars for purchasing officers to educate them on their legal and regulatory responsibilities under New York State law. It not only works to create videos supporting recycling programs but has also conducted a "needs assessment" in support of a potential Extended Producer Responsibility law for packaging for New York State. This organization, therefore, conducts work in multiple directions for multiple stakeholders in support of a unified overall goal. We are describing these kinds of organizations as "Task Forces," given their potential for acting in multiple directions in support of a single goal, and we think a similar organization at the federal level in support of reducing food waste could have great effectiveness. Communicating and clarifying information related to food waste data and food date labels such as "use-by," "sell-by," and "best-by" could really help decrease the levels of food waste. It is important for consumers to know that "sell-by" and "best-by" dates are meant as indicators for retailers to help them know when their product will become sub-optimal, not inedible. Similarly, past "use-by" dates don't automatically make them suitable for the garbage. In addition, harmonizing the vocabulary and purpose of such labels by manufacturers and packagers could help ensure that the education program had a greater focus. This would be a campaign for behavior change through multiple channels to consumers and the business community, fostered by the government's regulatory power.

There have been a number of private sector initiatives that intend to reduce food waste. Food waste collection programs have been created to manage source-separated food wastes (mostly through associated compost sites). Many of these have been started in cities, as collection is easier to accomplish with greater population density (Krones, 2019). Some of them used alternative means of transportation, such as bicycle collection (Tsang, 2020). However, even in rural areas, certain compost entrepreneurs have pioneered food waste collection programs (Tonjes et al., 2024). This is a management-oriented program. Another initiative attempts to address food waste by reducing it at the retail level. Grocery stores have been established in Denmark and Britain that sell blemished, irregularly shaped, or otherwise slightly off-spec food (Overgaard, 2016; Gunders & Bloom, 2017). These kinds of stores could be encouraged in the U.S. The focus would be food items that are nearing their use by, best by, or sell-by dates alongside the produce, which is aesthetically subpar. Approximately 10% of food waste in the U.S. is generated at the retail level (UNEP, 2017), so the creation of small secondary stores could lead to significant food waste reduction (Overgaard, 2016). Governments could provide support to entrepreneurs with siting and permitting, even perhaps financial incentives, similar to other small business support mechanisms. The secondary stores would need to work together with nearby grocery outlets to collect food that is going to be removed from food racks and have to access farmer distribution networks to receive "ugly vegetables" and similar off-spec items. Minnesota has supported these kinds of efforts for direct shipments to food banks and soup kitchens from farmers; however, it is a relatively small program (120 farmers across the state have donated vegetables and fruit since 2015, and the state purchases milk for these programs at current retail levels, and in 2020 only 150,000 kg of protein were donated) (Second Harvest Heartland, n.d.). Other industrial reuse programs, where businesses could arrange to off-load unwanted or overstock supplies, faced some difficulties in manufacturing reuse, such as selling or donating unwanted materials, because these actions were associated with the perception that the donor was supporting potential rival companies. This was a significant headwind in industrial reuse opportunities in New York City in the 1990s (Hammer et al., 1997) and may be difficult to overcome in the retail food sphere, which usually has small profit margins and is often highly competitive.

The three policy options selected for evaluation were secondary stores, tax incentives, and Task Forces.

2.2 Selection of evaluation criteria

Three criteria were identified as suitable for evaluating the policy choices: cost-effectiveness, recovery, and source reduction.

Cost Effectiveness (Criteria weighting = 3): Cost-effectiveness will provide a demonstration of decision uncertainty linked with an intervention in this scenario policy alternative. The cost-effective assessment will present the possibility that the decision to implement an alternative is suitable. For example, will the policy alternative option save money, will it cost money, is there a determination of savings per unit of food waste repurposed, etc?

Recovery (Criteria weighting = 1): The most significant direct impact of food waste on the environment is the generation of methane in landfills. Despite programs and policies to prevent emissions of landfill gas into the environment, a substantial portion of landfill-generated methane escapes into the atmosphere. Landfill-generated methane is estimated to be 22% of anthropogenic methane in the U.S. (Nesser et al., 2024). Food has long been identified as the material that generates more methane than any other MSW constituent (Wang et al., 1997). Since methane has a relatively short atmospheric lifespan (half-life estimates range from 10-14 years) (Chai et al. 2016), reducing methane emissions may result in significant reductions in greenhouse gas content over relatively short time frames (Bistline et al., 2022). Therefore, reducing food inputs to landfills (which we are characterizing as "recovery") has great potential to have a large environmental benefit from greenhouse gas mitigation.

Source Reduction (Criteria weighting = 2): It is a truism in solid waste management that reducing the amount of waste that needs management is the most effective means of managing waste. Materials that are not managed do not have direct costs, do not require infrastructure, require no decision-making burdens on consumers, etc. (Zhang et al., 2021). Policies that result in source reduction usually have ancillary benefits that are indirectly oriented toward other aspects of the economy and society. Food waste source reductions can include water conservation, reduced land use impacts, hunger reduction, economic diversification, and entrepreneurship (Thyberg & Tonjes, 2016).

3. RESULTS AND DISCUSSION

3.1 Step 1 Bibliometric Study

Food waste research is accelerating. The mean number of publications per year was 12, but over half (115) of the papers were published in the last four years (mean of 29 publications/yr), with 37 published in 2022 (Figure 1).

To create the co-occurrence map, words with a minimum of seven occurrences in the title and abstract were selected across the papers. The co-occurrence analysis revealed various prominent clusters. For example, the green cluster shows the research centered on "behavior" and "consumption," indicating a number of studies focusing on human behavior and consumption patterns with respect to food. The red cluster focused on "food waste," "management," and "greenhouse gas emissions." It provided research on the importance of managing food waste and

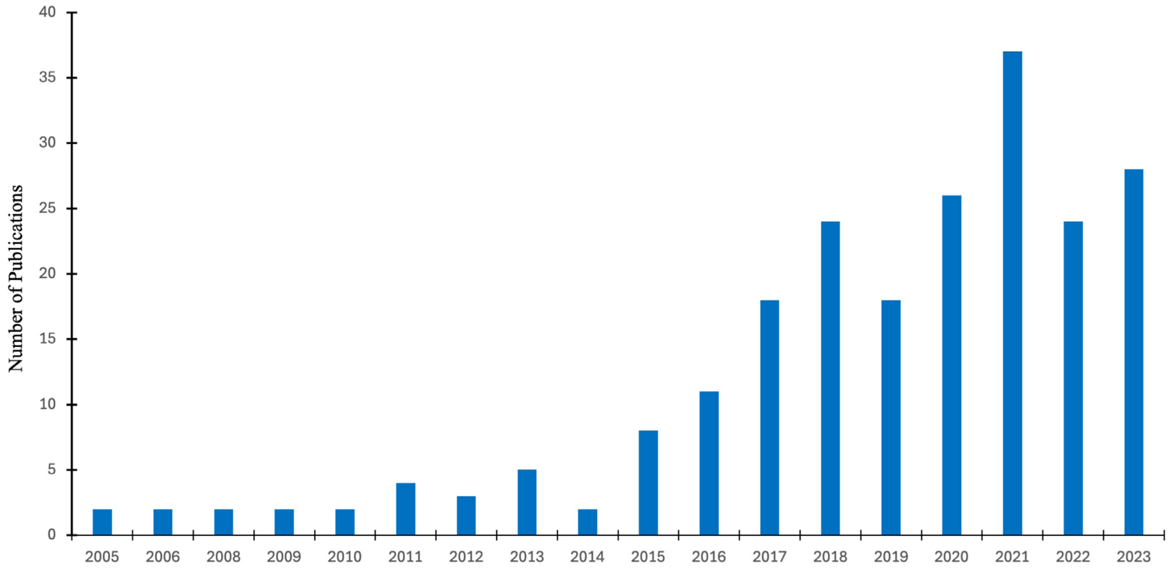

FIGURE 1: Publications per year (2005-2023).

understanding the different environmental impacts of food waste (Figure 2).

3.2 Analysis of Policy Alternatives

3.2.1 Creating Secondary Retail Food Stores

Some retail groceries donate edible food to charities for tax savings because of regulatory requirements or as an ethical choice (Lowrey et al., 2023). Donations can be difficult to manage, as there are transportation and other logistics issues and a loss of potential revenue (offset by tax savings). A model of diversion to alternative retail sales found that this had the potential for greater revenue for the seller, additional entrepreneurial opportunity for the purchasing operation, and perhaps greater diversion from disposal overall (Lee & Tongarlak, 2017). To enhance such operations with off-farm, off-spec fruits and vegetables, as is the model in Denmark and England, it was found that farmers would need expanded processing infrastructure and consistent pricing structures (Johnson et al., 2019). On-site processing of crops, including technical innovations such as dehydration or vacuum-sealing, could provide expanded use for off-spec crops (Birch et al., 2014). Secondary stores selling food items that might otherwise be disposed of might co-opt part of the "good food" perception associated with farmers' markets, where consumers will forgo a degree of convenience and pay more to be part of what is perceived as more sustainable, and healthier food chain that is more supportive of local agriculture

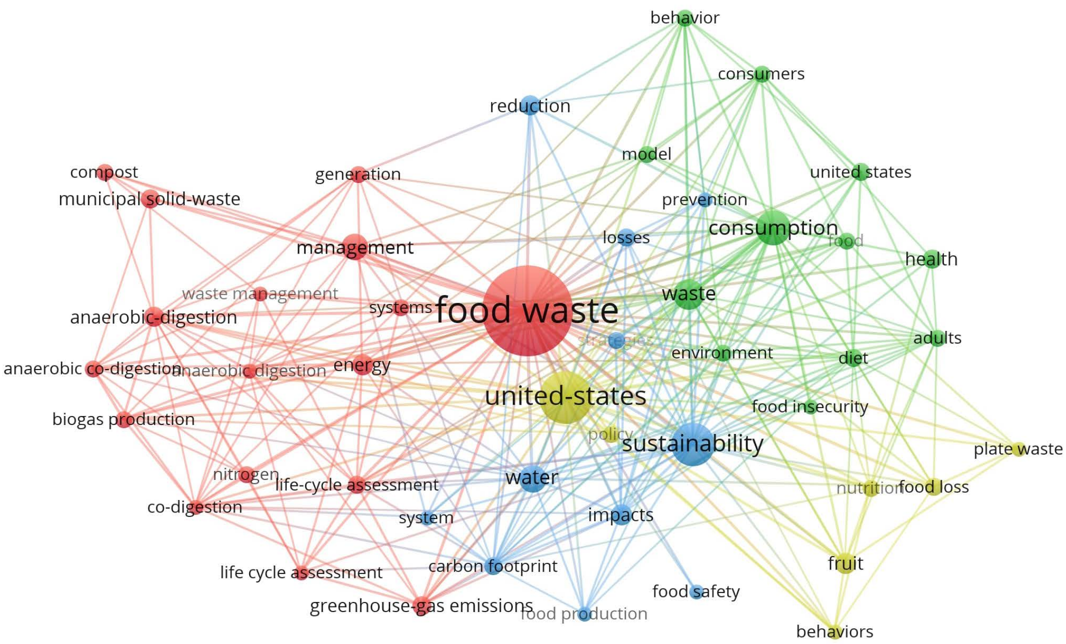

FIGURE 2: Keywords co-occurrence map.

(Connell et al., 2008). Danish (Overgaard, 2016) and English (Gunders & Bloom, 2017) versions of these stores have proven successful, but they have not yet been able to gain a U.S. foothold. This implies some form of government subsidy or other support may be required to begin this initiative in the U. S. In addition, off-spec food will need to overcome the primary consumer selection mechanisms of visual assessment, which means impressions of damaged or irregular food often equate to unhealthy food (Bolos et al., 2019).

- Cost-Effectiveness (Score = 2): Establishing a distribution network from farmers to stores separate from the established network for preferred food quality may have a substantial cost. Diversion of overstock and past-due date materials in established retail stores from disposal pathways is a complication that will probably have some additional costs; transporting food from retailers to these secondary stores will entail certain expenses. Analogies to other materials reuse opportunities imply that the logistics of organized diversion and potential "polishing" of these items to make them re-salable are not trivial (Gelbman & Hammerl, 2015).
- Recovery (Score = 4): The intention of this program is to divert materials from disposal, thereby meeting the goals of "recovery." Recovery may be enhanced at retail stores in establishing this program as once unsuitable materials are being diverted towards other-than-disposal (to the secondary stores), other options can also be considered, such as diversion of edible food to food banks and soup kitchens as well as to the secondary stores, and diversion from landfilling (or waste-to-energy incineration) to composting or anaerobic digestion. Creating a pathway for food losses to secondary stores from farms should actually increase landfilling to some extent; no process is waste-free, so processing and transporting food from farms to secondary stores and losses at the secondary stores should lead to some landfilling of food. This will increase landfilling as food losses on farms in the U.S. are rarely, if ever, landfilled. In addition, if the perception that off-spec food is unhealthy is not substantially changed, then the diversion of these food items into retail outlets may not result in substantial reductions in food waste; rather, since off-spec food on farms is rarely landfilled (Beausang et al., 2017) but retail food wastage is, it may lead to increases in landfilled food waste.
- Source Reduction (Score = 4): Food loss and waste reductions can be brought about in a meaningful manner by creating these secondary markets for excess food (Evans & Nagele, 2017). Preventing farm food loss conserves more resources than retail food waste diversion, probably even so compared to food donations or food waste composting. This alternative is most efficient in terms of source reduction. Again, however, the perception that off-spec food is unhealthy will need to be changed to result in substantial reductions.

3.2.2 Improving Tax Incentives

Tax code changes are common means in the U.S. to change behavior. These tax code changes can be focused on individuals or organizations. Here, there are no easy means to incentivize consumer choices; rather, tax changes are aimed at making farmers and other areas of the food industry change how they behave (NRDC, 2021). Expanding the somewhat limited scope of U.S. tax incentives to include all food diversion expenses is perceived as having benefits (Gunders & Bloom, 2017; NRDC, 2021). NRDC, in particular, believes that state tax incentives can be more effective because of better outreach efforts and the potential to target specific local industries in ways that broader approaches cannot (NRDC, 2021). State tax incentives seem most likely to be effective and result in savings for farms, whereas local extensions are often very effective at spreading information regarding many aspects of farm business practices (Wang, 2014); it seems less relevant for larger grocery chains that are already likely to use professional, comprehensive accounting and tax advisement services.

- Cost-effectiveness (Score = 3): Tax incentives, such as tax credits provided to food rescuers, are often cost-effective as any monetary amount given through such schemes will directly motivate farms, organizations, and businesses to donate food if enough of the costs are covered. Therefore, if tax credits are provided, the expenditures will go exclusively towards their envisioned purpose, i.e., reducing food waste and feeding the needy people. Whenever a business or farm does not donate surplus food, it does not receive any tax benefits. Additionally, by encouraging the donation of healthy food, low-profit margin businesses, like farms, would be supported with these tax incentives by allowing them to recover the cost invested in producing food they could not sell. The issue is whether sufficient credits are available and easily accessible to create the incentive to spend pre-tax money.
- Recovery (Score = 4): The intention of making food donations more affordable for food sources is to divert materials from disposal, thereby meeting the goal of "recovery." The target materials and sources for food loss and waste reduction here are the same as those for the secondary stores, so the impact should be similar if they are equally successful.
- Source Reduction (Score = 4): The source reduction impact of tax incentives will be of the same scope as the secondary store impact.

3.2.3 Task Force

Wastage of food is, to a large extent, a consequence of consumer consumption and buying behaviors. Often, supermarkets tend to order an excessive stock of food items that are more than what is required in order to fulfill the overconsumption needs of the consumer. This puts a heavy strain on the agricultural sector (REFED, 2016). There's an imminent need to relieve this strain, and the same could be achieved through stakeholder collaboration and campaigns. It is imperative to highlight the environmental and social damage caused by overconsumption while promoting wise purchasing habits.

Recent studies have shown that consumers in the

U.S. consider themselves to be well-informed about food waste; however, many do not correctly apply sell-by-use-by-best-by advisories (Bolos et al., 2019). In addition, there are approximately 50 iterations of these kinds of labels, meaning manufacturers and retailers do not have a coherent process for providing good food consumption information (Kavanaugh & Quinlan, 2020). This describes a multi-sector, multi-directional information and guidance opportunity, which would be suitable for a non-governmental organization, has bonafides due to academic or think tank relations and is agile and aggressive in offering work products across many domains. We have described such an organization as a Task Force. To close the gap between information and implementable knowledge, the proposed working group will incorporate educational policies via tailored campaigns, useful resources and public engagement. Some of the ongoing initiatives are updating information on safe storage and developing a "FoodKeeper" App (provides users with information on proper storage, product dating, etc) (USDA, n.d.-b)

Such an organization could conduct work with a relatively small staff on the order of 20 or so and a relatively modest outreach budget focused on social media channels with polished products but no general mass media presence. The group should also have a two-sided, aggressive outreach program: to key regulators such as the U.S. Department of Agriculture and the Federal Trade Commission, plus states with existing food waste support programs, such as those with tax incentives, along with Texas and Florida, as the largest states without existing food waste reduction policies in place. It should have outreach to major food manufacturers and distributors through participation in industry groups and meetings and targeted individual information efforts. An example of this would be the Center for Sustainable Materials Management at SUNY ESF; another could be the Recycling Partnership (https://recyclingpartnership.org/), which has a much larger scope of work and is a much larger organization than envisioned here. Establishing a consistent messaging approach would increase the effectiveness of the labels, educate consumers, and create pathways for "nudges" to improve the use of these tools (Bolos et al., 2019; Thaler & Sunstein, 2008).

- Cost-effectiveness (Score = 5): Amongst the three policy alternatives, setting up a task force and nudging consumers is clearly the least expensive option. It has no direct impact on food waste, however, and will require social change to be effective.
- Recovery (Score = 3): When the consumers are educated about date labels of products and how sell-by, use-by, and best-by dates are indicative of food quality and not its safety, there should be reduced need for stores to aggressively clear shelves of such items. This would minimize the landfilling of food waste.
- Source Reduction (Score = 3): Better understanding of food labels should decrease the overall amount of food waste, similar to the anticipated decrease in landfilled food waste.

3.3 Criteria Analysis Matrix

Food waste remains a significant cause of concern in the United States. The government must adopt advanced policies to reduce the amount of food waste generated in the country and reach its reduction goal. Table 1 presents the analysis of the three selected policy alternatives by applying a Criteria Alternatives Matrix. As depicted in the CAM table, the policy alternative for the Task Force has the highest total score, and so it is the most favorable option of these three for addressing the food waste issue in the U.S.

4. CONCLUSIONS

Consistent with the increase in U.S. food waste, there has been a growth in the number of publications on food waste in recent years. This paper investigated the options to reduce food waste in the United States. Important themes for this research have been sustainability, reduction, manage consumption. Using these key terms as guides, three policy options across the realms of government action, public-private cooperation, and private initiatives were evaluated using the criteria of cost-effectiveness, recovery (as measured by decreases in landfilling of food waste), and source reduction. The first policy alternative is to create Secondary Stores for food that customarily ends up in waste owing to cosmetic concerns or compliance with date labels. In the criteria analysis matrix, creating the Secondary Store scored "4" in terms of both Recovery and Source Reduction. Its Cost-effectiveness was less (scoring a 2). Expanding the scope of federal tax credits and creating similar kinds of state programs was seen as creating similar recovery and source reduction impacts to the Secondary Store policy. It was evaluated as being more cost-effective than the Secondary Stores. Establishing a nimble, multi-target organization (a Task Force) to work with consumers and industry to harmonize the use of date labels for food and to educate consumers on their meaning

TABLE 1: Criteria analysis matrix of policy alternatives.

		Criteria			Total
		Cost-effectiveness	**Recovery**	**Source Reduction**	
(Weights)		**3**	**1**	**2**	
Alternatives	Creation of secondary stores	2 (2 x 3 = 6)	4 (4 x 1= 4)	4 (4 x 2 = 8)	6 + 4 + 8 = 18
	Increased Tax Incentives	3 (3 x 3 = 9)	4 (4 x 1 = 4)	4 (3 x 4 = 8)	9 + 4 + 8 = 21
	Task Force	5 (5 x 3 = 15)	3 (3 x 1 = 4)	3 (3 x 2 = 6)	15 + 4 + 6 = 25

was identified as the preferred option. The government has already run various campaigns in some states to spread awareness regarding food waste. However, the government can enhance the process by providing information on in-store display screens or conducting contests to engage more customers and educate them. When the consumers are educated about the date labels of products and how "sell-by" or "use-by" dates are just suggestions made by the manufacturer for a product's freshness and do not relate to consumer safety, the state could prevent wholesome foods from reaching landfills. Therefore, in terms of recovery and source reduction, it was found to be the most efficient option compared to other alternatives.

4.1 Study Limitations and Future Directions

Step 1 of this study was conducted on the Web of Science (WoS) database by making use of specific keywords to arrive at a holistic analysis. Since WoS Directory was the only platform referred to, other publications platforms comprising scientific documents, such as Scopus, were not part of the current study. The key terms of research that were used to perform the survey were "Food Waste" and "United States," because of which other publications of value may have been overlooked and were not included in the study.

Step 2 could have been enhanced by a more formal evaluation process, using expert opinion elicitation such as a Delphi technique. A wider range of alternatives could have been considered.

In the future, researchers can advance this work by studying other policy alternatives for reducing food waste in the United States. They might explore the effectiveness of policies such as required food waste reporting in companies or programs related to composting, which promotes the diversion of food from landfill sites. A comparative study could be carried out to examine the effectiveness of these options in other nations, offering insights into versatile models for reducing food waste in various regions. Furthermore, future studies could also assess technological innovations, such as smart packaging or food waste tracking systems, and their possible influence on reducing food waste across the distribution chain.

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OPTIMIZATION OF COPPER RECOVERY FROM WEEE BY BIO-METALLURGICAL PROCESS

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ABSTRACT

Bioleaching of metals from Waste Printed Circuit Board (WPCB) is an alternative path for metal mining. Metallic composition of processed PCB at different size were estimated in this study. Acidithiobacillus ferrooxidans was used as a biomining agent to recover copper. Recovery of copper using this organism can be affected with several factors like pH, temperature, pulp density, sample quantity, sample size, inoculum load etc., It is pertinent to screen and optimize the influencing variables to maximize recovery. Four factors viz. particle size, temperature, $FeSO_4$ concentration and sample quantity were screened using Plackett-Burmann design. The process variables, temperature, $FeSO_4$ concentration and sample quantity were optimized using Box-Behnken design at 30°C temperature, 40.08 g/L $FeSO_4$ concentration and sample quantity of 5 g/L with a recovery rate of 80.56%. Using the optimized values, the influence of inoculum load and treatment duration were assessed. Inoculum of 50 ml/L and treatment period of 20 days produced a recovery rate of copper of 86.9%. Redox potential curve and pH variation of the bioleaching system also supports the findings of optimization as well as enhancement.

1. INTRODUCTION

In this electronic era, waste electronic and electrical equipment (WEEE) is increasingly becoming an environmental challenge (Garcia-Valles et al., 2008). According to studies, in South Africa and China, there is a rise of 200-400% in e-waste generated from end-of-life computers in 2020, compared to 2007 (Mmereki et al., 2016). The situation is worse in the case of India with a hike of 500% (Shreyas Madhav et al., 2022). Beyond the case of land utilization, the level of toxicity it causes is very high as it contains, hazardous elements including lead, mercury, arsenic, cadmium, selenium, chromium (VI) and flame retardants more than permissible quantity (Gao et al., 2021).

Conventional methods of metal recovery, including pyrometallurgy (Jung et al., 2017), hydrometallurgy (Pant et al., 2012) and mechanical process (Meng et al., 2017) could cause secondary pollution and therefore microbiological approach of metal recovery, also known as bioleaching becomes ideal with its eco-friendly nature (Xia et al., 2017). This involves the leaching of metals from low-grade ores using suitable microbes by providing them the required growth conditions (Bosecker, 1997). This could also serve as a detoxification technique for heavy metal toxicity in industrial wastes, soil and sewage sludge (Narayan & Sahana, 2009). Besides being served as a tool against environmental contamination, this could act as a secondary source for metals, which could enhance its commercial importance (Awasthi et al., 2019).

The most common elements recovered commercially through bioleaching are copper and uranium using Acidithiobacillus ferrooxidans. This organism leaches copper through the generation of powerful oxidizing agent Fe3+ (equation 1 and 2) (Sugio et al., 1990; Nemati et al., 1998).

$$2Fe^{2+} + ½O_2 + 2H+ \text{ à } 2Fe^{3+} + H_2O \quad (1)$$

$$Cu^0 + 2Fe^{3+} \text{ à } Cu^{2+} + 2Fe^{2+} \quad (2)$$

This process is influenced by several factors like pH, temperature, pulp density, sample quantity, sample size, inoculum load etc., (Deveci et al., 2003). Among these factors, particle size, sample quantity, temperature and FeSO4 concentration were studied in addition to inoculum load and process duration for copper recovery. Copper dissolution rate is found to be highly dependent on temperature as the highest dissolution rate was previously reported as 55-60°C followed by 48°C, 42°C and 30°C (Falagán et al., 2024). In acidic environments, redox potential directly correlates with Fe speciation (Yue et al., 2016). Therefore, redox potential of the system is also analyzed along with pH throughout the bioleaching period to estimate the variation in leaching rate.

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2. METHODOLOGY

2.1 Processing of waste printed circuit board

Printed circuit boards collected from used and disposed computers and televisions has been used for the present study. These were collected in large quantity from Clean Kerala Company, Thiruvananthapuram Unit, Kerala. Additional electronic components such as capacitors, resistors, diodes, sensors, etc. were removed manually. Remaining PCBs were crushed into small powder size particles using a laboratory jaw crusher (Star Trace Private Limited, Tiruvallur, Tamil Nadu). Powdered particles categorized based on size as 300 μm and <180 μm was used for further studies.

2.2 Estimation of metallic composition of PCB

The metallic composition of the samples of different sizes were estimated quantitatively through acid digestion. One gram of PCB sample was taken in an Erlenmeyer flask and dissolved in 100 mL of aqua regia (1:3 conc. HNO_3 and conc. HCl respectively.) (Kim et al., 2018). Contents were digested inside a fume hood for 48 h. After digestion, the solution with dissolved metals was filtered first using glass wool followed by nylon membrane filter (0.45 μm). Filtered solution was diluted 10 times with milliQ water and analysed using Atomic Absorption Spectroscopy (AAS) (Chanda et al., 2014).

2.3 Preparation of bacterial culture

Industrial bacterial leaching requires an organism that could survive extreme physical and geochemical conditions. Acidophiles are such extremophiles that could multiply in higher acidic conditions with pH<3. Among different kind of acidophilic microorganisms, Acidithiobacillus groups are found to be very effective in leaching out Cu, Zn, Fe and As from different mineral sources. Acidithiobacillus ferrooxidans which is a gram-negative, chemolithoautotrophic bacteria involved in microbial leaching was selected as biomining agent (Maluckov, 2017). Acidithiobacillus ferrooxidans (NCIM No:5371, ATCC No:19859) was purchased from National Collection of Industrial Microorganisms, National Chemical Laboratory, Pune, India. The strain was activated in Medium No:54 (Four parts of Solution A: 0.5 g/L $(NH_4)_2SO_4$, 0.5 g/L $MgSO_4.7H_20$, 0.5 g/L K_2HPO_4 and 5 mL of 1N H_2SO_4. One part of Solution B: 167g/L $FeSO_4.7H_2O$, 50 mL of 1N H_2SO_4 in 1 L; static incubation at 30°C for 3-5 days). The organism was adapted to metal by repeated sub-culturing of the strain under increasing concentration of $CuSO_4.5H_2O$ (Bosecker, 1997).

2.4 Bioleaching

Bioleaching of copper from waste PCBs of WEEE was carried out in a 250ml Erlenmeyer flask. The treatments contained 100ml of modified 9K media (Four parts of Solution A: 0.5 g/L $(NH_4)_2SO_4$, 0.5 g/L $MgSO_4.7H_20$, 0.5 g/L K_2HPO_4 and 5 mL of 1N H_2SO_4. One part of Solution B: 167g/L $FeSO_4.7H_2O$, 50 mL of 1N H_2SO_4 in 1 L) inoculated with metal adapted bacterial inoculum at indicated concentrations (Gao et al., 2021). After one day powdered and sterilized PCB was added at a concentration of 10 g/l. The entire content was incubated with constant shaking (160 rpm) at 30°C for 8 to 10 days. For analysis the content was centrifuged at 8000 × g for 8 min and the copper in supernatant was quantified using AAS.

2.5 Screening Analysis

Factorial designs are currently preferred over the traditional one-factor-at-a-time approach (Alfonzetti et al., 2003). The factors responsible for copper leaching were screened using Plackett-Burmann model with four factors and two levels. The factors analyzed were temperature (25°C and 35°C), $FeSO_4$ concentration (26.72 g/L and 40.08 g/L), sample size (<180 μm and 300 μm) and sample quantity (5 g/L and 15 g/L). The treatment duration (10 days), inoculum load (10 ml/L; 8.2×10^9 cells/mL) and initial pH (2.2) were fixed constant. The data on copper content were analyzed using Minitab software (Version 21.3.1.0).

2.6 Optimization of factors

Based on the outcome of screening analysis, three factors namely temperature, $FeSO_4$ concentration and sample quantity were optimized at three levels using Box-Behnken Design(Iqbal et al., 2020).

2.7 Enhancing Recovery

The optimized factors were further explored with inoculum load (10 ml/L, 25 ml/L and 50 ml/L) and incubation time (5,10,15 and 20 days) using two-way ANOVA.

2.8 Variation in pH and Redox Potential

The variation in pH value and redox potential of the bioleaching system were recorded at a regular interval of 24 hours.

3. RESULTS AND DISCUSSIONS

3.1 Metallic Composition of PCB

Metallic composition of the processed PCB samples at different particle sizes are shown in Table 1. Samples of 180 μm size exhibited higher content of Fe (217.70 mg/g) followed by Cu (105.41 mg/g) and Mn (11.05 mg/g) with other metals such as Ni, Mg, Co, Ag in minor quantities and Cr below detection level. Increased sample size of 300 μm exhibited higher Cu content (332.056 mg/g) followed by Fe (121.559 mg/g) and Mn (9.641 mg/g). The Cu content in PCB samples of particle size 300 μm was 33.20% which is significantly higher than 180 μm particle size (10.54%). These values are found to be much higher than the previous reports, which exhibited varied range between 12.5%, 14.6%, 19% and 28% content(Creamer et al., 2006; Feldman, 1993; Hino et al., 2009; Oliveira et al., 2010; Veit et al., 2002). Similar analysis based on mobile phone PCBs, at different particle sizes has reported 15.6% and 29.13% of copper content at 150 μm and 2 mm particle size respectively (Annamalai & Gurumurthy, 2021). Many factors such as the source of PCB, sample processing, characterization methods influence the final content (Bizzo et al., 2014).

The value represents the maximum dissolution of metal when treated with acid, which could be considered as that of 100% recovery value. Dissolution value obtained af-

TABLE 1: Metal composition of PCB sample.

Sample size (μ)	Metal	Quantity (mg/g)
<180	Copper (Cu)	105.41
	Iron (Fe)	217.70
	Silver (Ag)	3.06
	Nickel (Ni)	3.44
	Magnesium (Mg)	2.39
	Manganese (Mn)	11.05
	Cobalt (Co)	0.52
	Chromium	BDL
300	Copper (Cu)	332.056
	Iron (Fe)	121.559
	Silver (Ag)	4.001
	Nickel (Ni)	8.143
	Magnesium (Mg)	2.63
	Manganese (Mn)	9.641
	Cobalt (Co)	1.166
	Chromium	BDL

** BDL - Below Detection Level*

TABLE 3: Optimization of Temperature, $FeSO_4$ concentration and Sample Quantity.

Run	Temperature (°C)	$FeSO_4$ concentration (g/l)	Sample quantity (g/l)	% Cu Recovery
1	30	40.08	15	65.96
2	30	33.4	10	68.99
3	35	26.72	10	52.03
4	35	33.4	5	74.09
5	25	40.08	10	30.34
6	30	40.08	5	80.56
7	35	40.08	10	57.67
8	25	26.72	10	16.86
9	25	33.4	15	15.83
10	35	33.4	15	60.57
11	30	26.72	15	64.22
12	30	33.4	10	68.99
13	30	26.72	5	77.31
14	25	33.4	5	41.13
15	30	33.4	10	68.99

ter microbial leaching is compared with this value to obtain the percentage recovery.

3.2 Factors influencing recovery of copper

Besides particle size, other factors such as temperature, sample quantity and concentration of electron donor play a major role. In order to find the significant factors, a screening design (Plackett-Burmann design) was employed. It is one of the most used screening designs because it needs smaller number of experimental runs and also cost-effective (Fukuda et al., 2018). The combinations of different factors provided for this design and the percentage recovery in each case are listed in Table 2. The factors which possess a probability value (P-value) below 0.05 can be considered to have a significant effect on the process. According to the statistical observations, factors including temperature, $FeSO_4$ concentration and sample quantity are significant and particle size is insignificant. It may be noted that the particle size plays a major role in chemical recovery method, but not in bioleaching. Hence, temperature, $FeSO_4$ concentration and sample quantity were further optimized.

3.3 Optimization of Copper recovery

Optimization of factors using Box-Behnken design of Response Surface Methodology considers three factors (temperature, $FeSO_4$ concentration and sample quantity). The different combinations of factors used and the obtained recovery rate are provided in Table 3. Maximum recovery rate of 80.56% is obtained at 30°C temperature,

TABLE 2: Effect of particle size, temperature, $FeSO_4$ concentration and Sample Quantity on copper recovery.

Run	Particle Ssize (μ)	Temperature (°C)**	$FeSO_4$ conc.* (g/l)	Sample quantity * (g/l)	% Cu Recovery
1	300	35	40.08	5	86.16
2	< 180	35	40.08	5	81.68
3	300	35	26.72	15	70.20
4	< 180	35	26.72	5	75.51
5	300	25	40.08	5	71.93
6	300	25	40.08	15	61.92
7	< 180	25	26.72	5	71.45
8	< 180	25	40.08	15	74.95
9	300	25	26.72	5	69.21
10	300	35	26.72	15	70.20
11	< 180	25	26.72	15	57.29
12	< 180	35	40.08	15	80.58

P values: Particle size = 0.460, Temperature = 0.007, FeSO4 conc.= 0.024, Sample Quantity=0.031

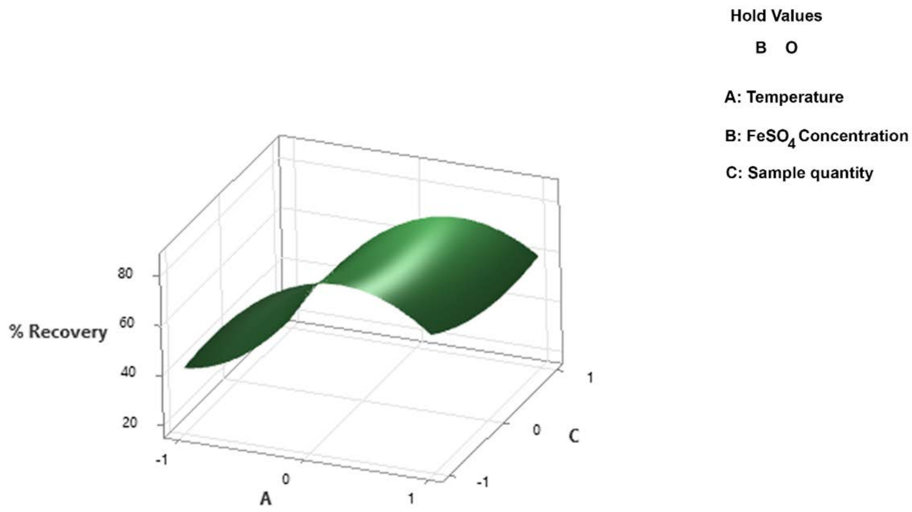

FIGURE 1: Response plot of temperature and sample quantity on % copper recovery.

40.08 g/L $FeSO_4$ concentration and sample quantity of 5 g/L. Statistical analysis of the data obtained through this model has been represented as Table 4. Here, the p-values of temperature (0.000), $FeSO_4$ concentration (0.071) and sample quantity (0.001) indicates the high significance of temperature and sample quantity along with the least significance of $FeSO_4$ concentration in altering the rate of recovery during optimization. The model exhibits a standard deviation(S) of 3.7, which represents how far the data value fall from the fitted value. The R-sq value (99%) and R-sq adjusted value (97%) of the model is found to be higher than R-sq predicted value (82%). This indicates the model is robust. The Variance Inflation Factor (VIF) value of 1.00 in all cases of interactions, shows that the factors are not correlated (Figure 1).

3.4 Enhancing the Recovery

An attempt was made to improve the recovery rate by including microbial factors such as inoculum level and treatment time. Recovery rates of copper using the optimized conditions with three different inoculum load (10, 25 and 50 ml/L) and four different treatment duration (5, 10, 15 and 20 days) are presented in Table 5. While the recovery rate improved till 10th day a lag followed by improvement due to higher inoculum load was observed. This correlates well with the exponential ferrous oxidation rate of copper tolerant strains of A. ferrooxidans (ATCC 19859) between 5 and 9 days (Brahmaprakash et al., 1988). Thus, an inoculum load of 50 ml/L and treatment time of 15-20 days seems to be ideal to achieve a maximum recovery of 86.9%. The considerable elevation in the recovery rate with increased inoculum load was also reported (McSweeney et al., 2011; Wang et al., 2014).

3.5 pH and Redox Potential Curve

Variation in pH and redox potential of the bioleaching system were recorded for 10 days at a specific time interval of 24 hours and the data obtained is represented as Figure 2 a and b. The drop of pH from 2.2 to 1.5 indicates the increase in acidic nature of the system that supports the activity of Acidithiobacillus ferrooxidans. The redox poten-

TABLE 5: Effect of inoculum load and treatment period on copper recovery rate.

Run	Inoculum load (ml/L)	Treatment period (Days)	Cu Recovery rate (%)
1	10	5	25.17
2	10	10	79.89
3	10	15	79.92
4	10	20	79.94
5	25	5	54.09
6	25	10	75.73
7	25	15	76.66
8	25	20	77.41
9	50	5	66.59
10	50	10	70.68
11	50	15	84.24
12	50	20	86.94

p-values: Inoculum load = 0.016, Treatment period =0.023

TABLE 4: Statistical analysis of the optimization results.

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	68.99	2.15	32.09	0.000	
A	17.53	1.32	13.31	0.000	1.00
B	3.01	1.32	2.29	0.071	1.00
C	-8.31	1.32	-6.32	0.001	1.00

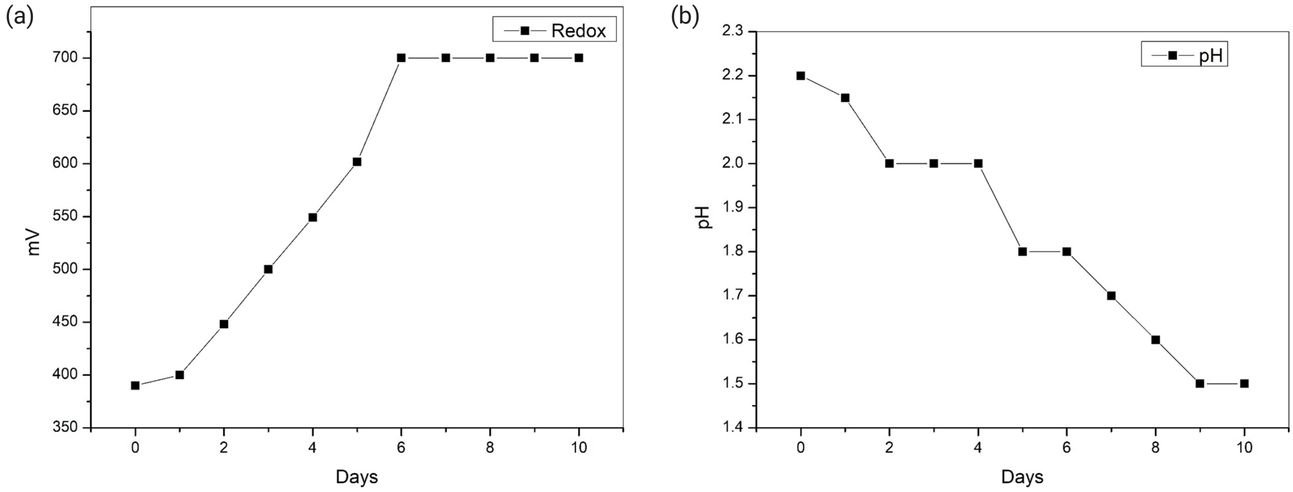

FIGURE 2: Variation in the redox potential of bioleaching system (a); Variation in the pH of bioleaching system(b).

tial data obtained indicates a gradual increase in the value from 390 to 700 mV within the first 6 days and later it is found to be maintained at 700 mV. The gradual upliftment in the redox potential is found to be the indication of a proportional upliftment in the leaching rate. The data obtained is found to be in correlation with the previously reported data (Kinnunen et al., 2020). The constant value obtained through Nernst equation (pe+pH=constant) shows an upliftment from an initial value of 8.79 to 13.63 by 6th day, thereafter a decline upto 13.33 by the 9th day and remains same thereafter.

4. CONCLUSIONS

Bioleaching of copper from used and disposed computer, television PCBs utilizing Acidithiobacillus ferrooxidans can be affected by different physio-chemical parameters among which temperature and sample quantity is found to be independent and significant. Even though $FeSO_4$ conc. is an independent variable essential for the growth of the microorganism, the recovery rate is not found to be altered due its variation during optimization. The optimum condition for bioleaching copper has been estimated as a temperature of 30°C, sample quantity of 5g/L and $FeSO_4$ concentration of 40.08 g/L. The recovery rate of copper is found to be enhanced (86.94%) at a higher inoculum load of 50 ml/L after 15-20 days of treatment under optimized conditions of temperature, $FeSO_4$ conc. and sample quantity. The positive impact of higher inoculum load and increased duration can be further supported by the persistence of redox potential at 700 mV after 6 days of bioleaching at optimized conditions.

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RECYCLING OF PHOTOVOLTAIC PANELS: PROCESS DEVELOPMENT AND TECHNOLOGY TRANSFER DURING THE LAST TEN YEARS

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ABSTRACT
Research and development in photovoltaic panel (PVP) recycling have evolved to maximize recovery, including less valuable but non-renewable materials. Early recycling relied on low-cost physical processes but failed to produce glass pure enough for the glass industry, except for construction. A process involving crushing, sieving, thermal treatment, fine fraction leaching, and wastewater treatment required 75,000 tons/year capacity to achieve a payback time under 6 years. High-value recovery requires advanced methods to separate glass from the encapsulant, ensuring reintegration into glass production. A solvent-based process, tested at the pilot scale, proved technically and economically feasible. Without metal recovery, it remains viable at 30,000 tons/year, producing high-purity glass. Further developments enabled 82% recycling rates, including polymeric and metallic recovery, potential thermal valorization of EVA, and backsheets recycling. Economic analyses highlight challenges in a complete refining process, particularly for Ag and Si recovery. The Ag content in PVP has declined from 0.2% to 0.02% w/w in two decades. At 0.05-0.2% Ag, a 5-year payback is possible for 43,000-18,000 tons/year. Lower Ag levels require higher recycling volumes or supportive measures (e.g., EoL fees) to remain profitable.

1. INTRODUCTION

Photovoltaic systems have expanded significantly over the last decade as an alternative to fossil fuel-based energy production. Technological maturation and the reduction in production costs have positioned photovoltaic panels (PVP) among the best candidates for generating electricity from renewable sources (Jäger-Waldau, 2020).

Given the expansion, both realized and projected, of photovoltaic panel installations (IRENA, 2024; Oñate et al., 2024; Xu et al., 2024), the European Community has included End-of-Life PVP (EoL PVP) among Wastes of Electrical and Electronic Equipment (WEEE), for which proper separate collection and the development of suitable recycling processes are necessary (Directive 2012/19/EU).

PVPs are composed of glass, polymer sealant, backsheet, photovoltaic cells, and a network of metallic filaments and silver ink for electrical contacts (Isherwood et al., 2022).

Recycling processes for PVPs range from simple mechanical treatments to thermal and hydrometallurgical treatments (Padoan et al., 2019).

Conventional mechanical treatments are the most economic, but cannot ensure the production of market-ready products or materials that can be reintroduced into glass production cycles due to the extreme complexity of the photovoltaic composites. Grounded materials obtained by mechanichal operations can be used in building compartment for low value applications (Goh et al., 2024), otherwise require further refinement by innovative technologies such as optical sorting or electrostatic operations (Dias et al., 2018) to reach high quality products.

Innovative technologies have been applied with the aim of liberating components by size reduction such as high voltage pulse crushing (Zhao et al., 2022), microwaves (Pang et al., 2021), and wire explosion (lim et al., 2021), but none of them still reached large scales.

Thermal treatments aim mainly to degrade or soften the polymer sealant (Ethilen Vynil Acetate, EVA) and separate the various layers that make up the panel. Thermal treatments can be performed by conventional methods such as burning (Fiandra et al., 2019) and pyrolysis (Wang et al., 2019) or by innovative technologies such as halogen lamps (Riech et al., 2021) and hydrothermal treatment (Nekouei et al 2024). These treatments are energy-intensive and generate hazardous gases (Deng et al., 2022).

Chemical methods for encapsulant dissolution or swelling (solvent delamination) include the treatment of

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integer or crushed panels with organic solvents (Lee et al., 24; Dias et al., 2021; Li et al., 2022; Prasad et al., 2022), alkaline alcoholic solution (Cai et al., 2022), and more recently supercritical carbon dioxide (Lovato et al., 2021; Briand et al., 2022).

Hydrometallurgical processes for recovering metals like silver, copper, tin and lead are carried out after mechanical treatments (Sanathi et al., 2024). Silver is the mostly addressed metal generally extracted by nitric acid (Oliveira et al. 2020) followed by solvent extraction (Cho et al., 2019) or by innovative deep eutectic solvents (Lemoine et al., 2024). Other investigated metals are Cu, Sn and Pb extracted by mineral acids and then and separated by solvent extraction and precipitation (Chen et al., 2020; Moon et al 2017).

Metal-targeting hydrometallurgical processes often overlook the recovery of the bulk material, which is glass, thus failing to meet the requirements of EU WEEE regulations (Directive 2012/19/EU) on minimum recycling rate.

Silicon cells, which were historically the most valuable component of PVPs, have been the focus of various recycling methods, such as manual dismantling and chemical etching to remove coatings. These techniques were prominent but are now largely obsolete due to the collapse in silicon cell prices driven by Chinese overproduction (Lin et al., 2023). Similarly, recycling processes tailored to CdTe and CIGS panels have fallen out of favor as silicon technology dominates the market, primarily due to its reduced cost and superior efficiency, with no reversal of this trend expected (Di Sabatino et al., 2024). In the same view, the initial focus on high-value materials (metals) should give way to the primary goal of maximizing the recovery of non-renewable resources, including materials typically considered low-value, such as glass (Su et al., 2024).

The primary targets of process development at this time are, on the one hand, ensuring high recycling efficiencies and, on the other hand, recovering high-value products. For the first target, it is essential to focus on recovering glass, which is the most significant component by mass. For the second target, glass must again be considered because if it is too small in size (<0.5-1mm) and contains metal contaminants, its value can only be low. Glassworks require large-sized recycled glass to control its purity. Therefore, the process target must be coarse, clean glass (Vieitez et al., 2011). However, glass alone cannot ensure the economic sustainability of a recycling process, so it could be necessary to implement a waste refinery to recover other valuable components such as Ag, Si, as well as Al from the frame, Cu and Sn from the conductive filaments, and finally potentially recyclable polymeric components.

The economic sustainability of PV recycling faces several key challenges, especially due to the high costs involved in establishing and operating recycling facilities. These costs are notably high when more advanced chemical or thermal processes are required, as these techniques demand substantial investment and maintenance expenditures. IRENA and IEA-PVPS Report (2016) highlights the need for robust infrastructure to support recycling efforts, while Dias et al., 2016 point out that the technical complexity of recovering high-purity materials like silicon and silver from PVPs increases costs and limits profitability when operations are small-scale. Another major factor affecting economic feasibility is the fluctuating market value of recycled materials, which makes it difficult for facilities to achieve consistent revenue. Paiano (2015) and Sica et al. (2018) emphasize that when market prices for these materials are low, it often becomes challenging for recycling operations to remain economically viable. Recovered materials, including silicon, glass, and precious metals, do not always fetch prices that can compensate for the intricate separation and purification processes required.

Additionally, the relatively long lifecycle of PVPs limits the volume of end-of-life materials available annually, which can fall short of the mass required to sustain large-scale recycling plants profitably. Kumar and Chatterjee (2021) observe that this low turnover rate constrains recycling capacity, meaning that without significant volumes, even well-designed facilities may struggle to break even.

For recycling processes themselves, mechanical methods like shredding and magnetic separation offer lower-cost options and can operate at a positive margin with smaller volumes of PVPs. However, their efficiency in recovering high-value components such as pure glass is limited. Mechanical recycling methods are thus more feasible on a smaller scale, generally requiring several thousand tons per year to achieve economic viability (Dias et al., 2016). Integration of mechanical methods and advanced electrostatic separations efficiently separates valuable materials from glass and polymers, offering a simpler, lower-cost alternative to full recovery methods including thermal and chemical treatments. While complete recycling provides the greatest environmental benefits, the integration electrostatic separation in mechanical processes is more viable for smaller waste volumes or where glass recycling markets are limited. Economically and environmentally, it offers a feasible interim solution, with further research needed to optimize and scale the process (Dias et al., 2022).

In contrast, chemical treatments, which use reagents to recover materials at a higher purity level, can effectively retrieve valuable components like silver and high-grade silicon. However, the costs associated with reagents and waste management make chemical recycling economically viable only when facilities treat significant amounts of panels (Choi and Fthenakis, 2010; Kumar and Chatterjee, 2021).

Thermal methods, employing high temperatures to separate materials, are similarly effective at retrieving silicon but require significant energy, pushing operational costs higher (IRENA and IEA-PVPS, 2016; Marwede et al., 2013).

Industrial-scale photovoltaic panel recycling is developing globally, with still limited facilities leveraging advanced technologies to recover valuable materials like silicon, silver, and glass.

In Europe, PV Cycle operates a network of facilities that comply with the WEEE directive (www.pvcycle.org). These facilities combine mechanical and chemical processes to manage several thousand tonnes of panels annually, promoting material recovery while adhering to stringent regulations. Veolia's ReProSolar project in France represents a significant initiative, utilizing thermal and chemical delamination processes to recycle silicon-based panels (www.veolia.com). The plant has a capacity of 5,000 tonnes annually, aiming to recover high-purity materials such as silicon and silver without relying on mechanical shredding. ROSI Solar (www.rosi-solar.com) another French initiative, focuses on the chemical recovery of high-value materials like silicon and silver from EoL-PVP, with an estimated capacity of 3,000 tonnes annually.

Other EU initiatives at pilot scale include Flaxres in Germany (using high intensity light pulse heating the Si wafer for delamination)(https://www.flaxres.com), 9-Tech in Italy using thermo-mechanical treatment (https://www.9tech.it) and addressing silicon valorization (Cerchier et al., 2024), Geltz-Umwelt Technologie in Germany using advanced pyrolysis (https://cordis.europa.eu/article/id/238592-stateoftheart-solar-panel-recycling-plant).

Outside EU the most important initiative is First Solar in the United States and Malaysia for thin-film modules including chemical and thermal treatments that achieve a recycling rate of 90% (semiconductor materials and glass) with 25,000 ton/y potentiality (www.firstsolar.com).

The main obstacles to scaling up end-of-life panel treatment lie in intermediate pilot-scale technology transfer activities, which are essential for assessing the technical feasibility of processes and the quality of products, thus enabling reliable economic analyses to guide scaling decisions.

Below are the main steps of the research activities made in the last 10 years by the research group on Theory of the development of chemical process of the Department of Chemistry of Sapienza University in collaboration with the Sapienza Spin off company Eco Recycling. Research and technology transfer activities addressed the need for further scaling, including pilot demonstrations and economic feasibility assessments.

2. MATERIALS AND METHODS

2.1 Physical treatments

The input waste material were samples from different types of PV devices of different types Monocrystalline, Polycrystalline, and Amorphous Silicon-based panels. Samples were obtained by manually removing external aluminum frames (if present) and cutting a 40 × 40 cm section using a diamond blade and a hammer. Crushing operations were carried out in a two blade rotors crusher (DR120/360, Slovakia) without any controlling sieve and in a hammer crusher (SK 600, Slovakia) using a 5 mm sieve. Thermal treatment experiments were carried out on the coarse fraction (d > 1 mm) in a silite resistance furnace under an air flux of 30 l/h. The samples were placed in ceramic crucibles, heated at a rate of 10°C/min up to 650°C, and maintained at this temperature for 1 hour. After cooling, the samples were sieved, and particle size distribution was analysed (Pagnanelli et al., 2017).

2.2 Solvent treatment

EoL-PVPs were initially disassembled manually to extract aluminum frames. They were then crushed at a pilot scale using a single-shaft shredder (Guidetti Ltd) and classified into three size fractions through sieving: coarse fraction (>3 mm), intermediate fraction (0.5–3 mm), and fine fraction (<0.5 mm). Solvent treatment tests were conducted with a solid-to-liquid ratio of 1:8 at a temperature of 70°C. In the Photolife process, after treatment, the solvent was recovered through filtration and distillation, while solid fractions were separated via gravimetric separation by adding water. This process resulted in glass and metallic filaments settling in the underflow, whereas polymers (EVA and Tedlar) along with cell residues floated in the overflow (Pagnanelli et al., 2019).

The enhanced polymer separation process included sieving after solvent treatment, with a 7 mm cut-off to remove Tedlar from the oversized fraction. This was followed by gravimetric separation, where water was added to the remaining solids suspended in the solvent, creating a biphasic system (water-cyclohexane), leading to the separation of glass and metallic filaments in the underflow (Rubino et al., 2021).

3. RESULTS AND DISCUSSION

3.1 Physical treatments

First activities on photovoltaic recycling focused on simple physical processes. Starting with different types of panels, alternative mechanical treatments and thermal methods were tested with the primary aim of freeing glass and metals from the sealant (Granata et al., 2014; Pagnanelli et al., 2017).

In particular, different combination of mechanical treatment followed by thermal treatment of coarse fraction for EVA removal were compared. Single crushing followed by thermal treatment resulted in an overall glass recovery ranging from 50% to 70%, depending on the type of panel. Introducing hammer crushing after single crushing improved glass recovery to 80–85%, although the recovered products had a significant presence of fine fractions (40–45% by weight, depending on the type of panels). To address the formation of fines, a triple crushing operation was tested, which allowed for a reduction of waste to be thermally treated (only 62% compared to 85% after single crushing). Comparing triple crushing with single crushing plus hammer crushing, the obvious advantage is the use of a single piece of equipment instead of two different ones, thus reducing investment costs. Additionally, glass recovered through triple crushing contained lower amounts of fine fractions compared to the scheme including hammer crushing. As a result, after triple crushing a fraction of pure glass (0.4-1 mm) can be directly recovered by sieving, while coarse fractions can be fed to the furnace for sealant distruction and glass liberation giving coarse glass >1 mm. Thermal treatment resulted in a weight loss of 5-10% due to EVA decomposition. This phase releases various gases and suggests the need for a condensation unit in the recycling plant. The separation of glass from EVA is crucial for the glass's value; it must be free of polymeric materials to be used as cullet in soda-lime glass production. Thermogravimetric analysis confirmed that the recovered glass fractions are clean enough for reuse in manufacturing.

In Figure 1 the scheme of operations and the size distribution of crushed materials and thermally treated materias are reported according to triple crushing and thermal treatment combination.

The minimisation of fine fractions during mechanical operations is advantageous considering that, according to their metal content (Pagnanelli et al., 2017), a metallurgical/hydrometallurgical treatment is necessary to recover these fractions as pure glass. At the same time, it is possible to attempt the recovery of metals present in the panels, which are of different types: metallic contacts (busbars) recovered after thermal treatment of the coarse fraction, and as powder in the fine fraction subjected to leaching.

Metallic contacts containing aluminum (Al), copper (Cu), and silver (Ag) can be directly recovered after thermal treatment and sold as scrap. In the fine waste fractions (<0.08 mm and 0.08–0.4 mm), the most abundant metals are iron (Fe), zinc (Zn), and aluminum (Al), with concentrations below 5 mg/g. The low content and market value of these metals, and low potentiality of photovoltaic panel recycling plants make it less advantageous to include a specific metal recovery section in these plants.

The goal of leaching the 0.08–0.4 mm fractions is to clean the glass to enhance the overall mass recovery of the process. The leach liquor containing Fe, Zn, and Al can be treated in the wastewater section using primary treatments like precipitation and secondary treatments with ion exchange resins, following conventional routes for metal-bearing wastewaters.

For the finest fraction, which represents less than 5% of the total weight of treated panels, higher concentrations of metals are found, with silver (Ag) being of greatest interest due to its higher market value (0.5 €/g) and concentrations in the finest fractions ranging from 0.3 to 2 mg/g. This Ag-bearing fraction could be directly treated in the photovoltaic recycling plant or sold to refiners, improving the overall economic feasibility of the recycling process as further discussed in the following dedicated section.

A preliminary economic analysis, which includes crushing, sieving, thermal treatment of the coarse fraction, leaching of the fine fractions for glass cleaning (not metal recovery), and wastewater treatment, indicates that the process is economically feasible (with a payback time of less than 6 years) with a capacity of 75,000 tons per year. It should be noted that the estimate is conservative and does not account for the costs associated with managing waste panels (recycling fee, that is fee for collection and treatment paied to the smelters by consortia) (Pagnanelli et al., 2017).

3.2 Solvent treatment

The process development triggered by the preliminary data from physical treatment, which highlighted in the laboratory the possibility of using a method different from thermal treatment for the liberation of glass from the sealant (EVA) in the coarse fraction: the use of an organic solvent for swelling the sealant and thereby making it lose adhesion to the glass without dissolving the sealant (Figure 2).

The process, initially optimized in the laboratory, was patented (Toro et al., 2013) and subsequently validated on a pilot scale as part of the Photolife project's demonstration activities (Photolife project). During this project, 3 tons of panels of various types were processed using the same method (referred to as the Photolife process), demonstrating its effectiveness from an environmental and economic point of view (Pagnanelli et al., 2019) and flexibility for different panel types (dos Santos Martin Padoan et al., 2021). The use of the solvent meant that it was not necessary to reduce the initial sizes too much. Therefore, a single crusher, specifically designed to adequately reduce the size of

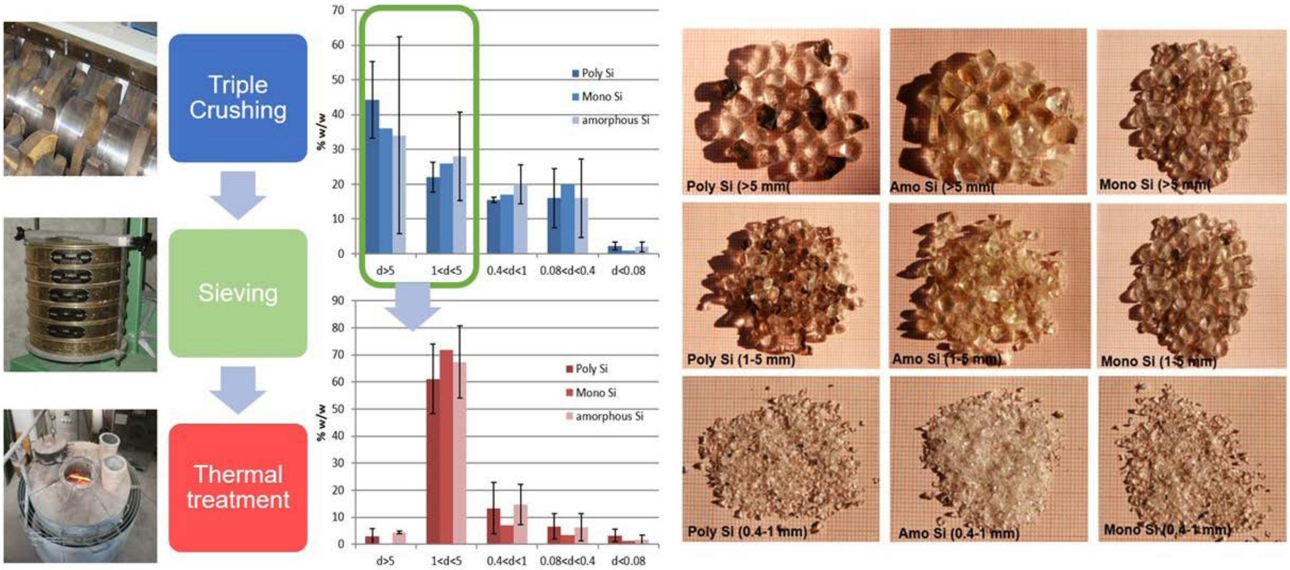

FIGURE 1: Schematic block diagram for optimised physical treatment of EoL PVP including triple crushing, sieving and thermal treatment in a furnace of fractions larger than 1 mm. Size distribution of crushed materials (blue bar graph) and of thermally treated materials (red bar graph). Photos of materials collected after thermal treatment for different kinds of photovoltaic panels (Poly Si: Polycrystalline Silicon; Amo Si: Amorphous Silicon; Mono Si: Monocrystalline Silicon).

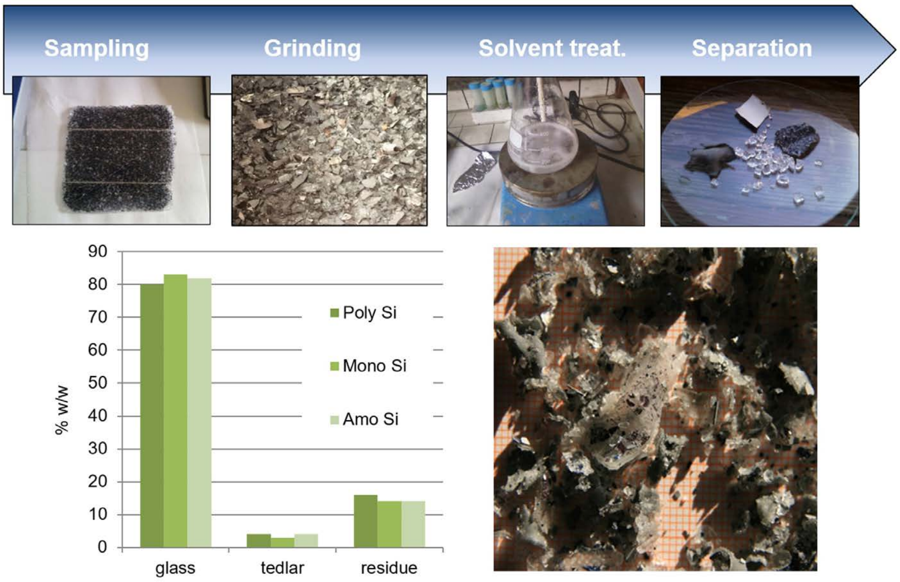

FIGURE 2: Schematic representation of the laboratory scale phase for solvent-base delamination process of EoL PVP fragments; green bar graph reports the weight distribution after solvent treatment of separated components for different types of panels (Poly Si: Polycrystalline Silicon; Amo Si: Amorphous Silicon; Mono Si: Monocrystalline Silicon).

the glass while minimizing the production of low-value fine fractions, was used for the mechanical part.

The integrated mechanical and chemical Photolife process combines automatic panel crushing, solvent treatment of the coarse fraction, and leaching of the fine fractions for glass cleaning from metal impurities. The main products of this process are high-quality glass (>1 mm) obtained from solvent treatment of the coarse fraction and low-quality glass from the fine fractions. The automatic crushing stage was optimized to minimize the production of fine fractions, which ensures the maximum yield of high-quality glass while reducing the amount of low-quality fine glass (<1 mm). The solvent treatment of the coarse fraction allowed for the complete separation of various components within just 1 hour at 50°C with continuous agitation (Pagnanelli et al., 2019). This efficiency in the kinetics of detachment is notably higher than that reported in the literature, where solvents are typically used for cell recovery and often require several days for complete detachment.

The glass recovered through solvent treatment exhibited superior quality standards compared to the glass obtained from thermal treatment. Specific tests designed to assess the quality of recycled glass from solvent treatment confirmed that these samples are suitable as raw materials for producing new PVP. In contrast, glass obtained from thermal treatment had a superficial metal residue, rendering it unsuitable for direct use in foundries.

A preliminary economic analysis of the solvent process, excluding metal recovery, indicated that solvent treatment is economically viable for a processing capacity of 30,000 tons per year. For lower capacities, the End-of-Life (EoL) fee is essential for making the photovoltaic panel treatment economically sustainable. When using the same inputs, solvent treatment presents a lower Payback Time (PBT) compared to thermal treatment. Additionally, a Life Cycle Assessment reveals that the solvent process has positive environmental impacts compared to thermal treatment, largely due to the higher value of the recycled glass (Pagnanelli et al., 2019).

An additional improvement to the process was achieved within the ORiFo project, co-funded by the Italian Ministry of the Environment and Sea Protection. In this project, a laboratory-scale optimization of the solvent treatment operation of the basic Photolife process (already demonstrated on a pilot scale) was proposed, focusing on polymer separation and metal recovery (Rubino et al., 2021), and then a patent application was made (Toro et al., 2020).

Analyzing the Photolife process sequence, the main issue was the lack of separation between polymers: after gravimetric separation, Tedlar (the backsheet) and EVA (the sigillant) residues remained glued together in the light fraction (due to the adhesive properties of EVA), with no possibility of separation through subsequent operations. In response to this need for polymer separation, four different operational sequences were tested. Specifically, the selectivity of the filtration operation (following the solvent treatment) on the degree of polymer separation was evaluated, demonstrating that Tedlar can be effectively separated from EVA residues. Furthermore, compared to the basic Photolife process, an additional thermal treatment of EVA residues (containing the photovoltaic cell and silver from the electrical connections) was introduced. The metal extraction yields highlighted the effectiveness of this strategy compared to direct extraction from uncombusted EVA residues.

By processing 100 kg of crushed material, it is possible to recover 0.03 kg of Ag, 45.5 kg of high-quality glass, 10 kg of Al scraps, and 1.2 kg of metallic filaments.

The Sankey diagram of the process is reported in Figure 3.

Thanks to the optimization, the recycling rate of the implemented process has increased to 82%, while the recov-

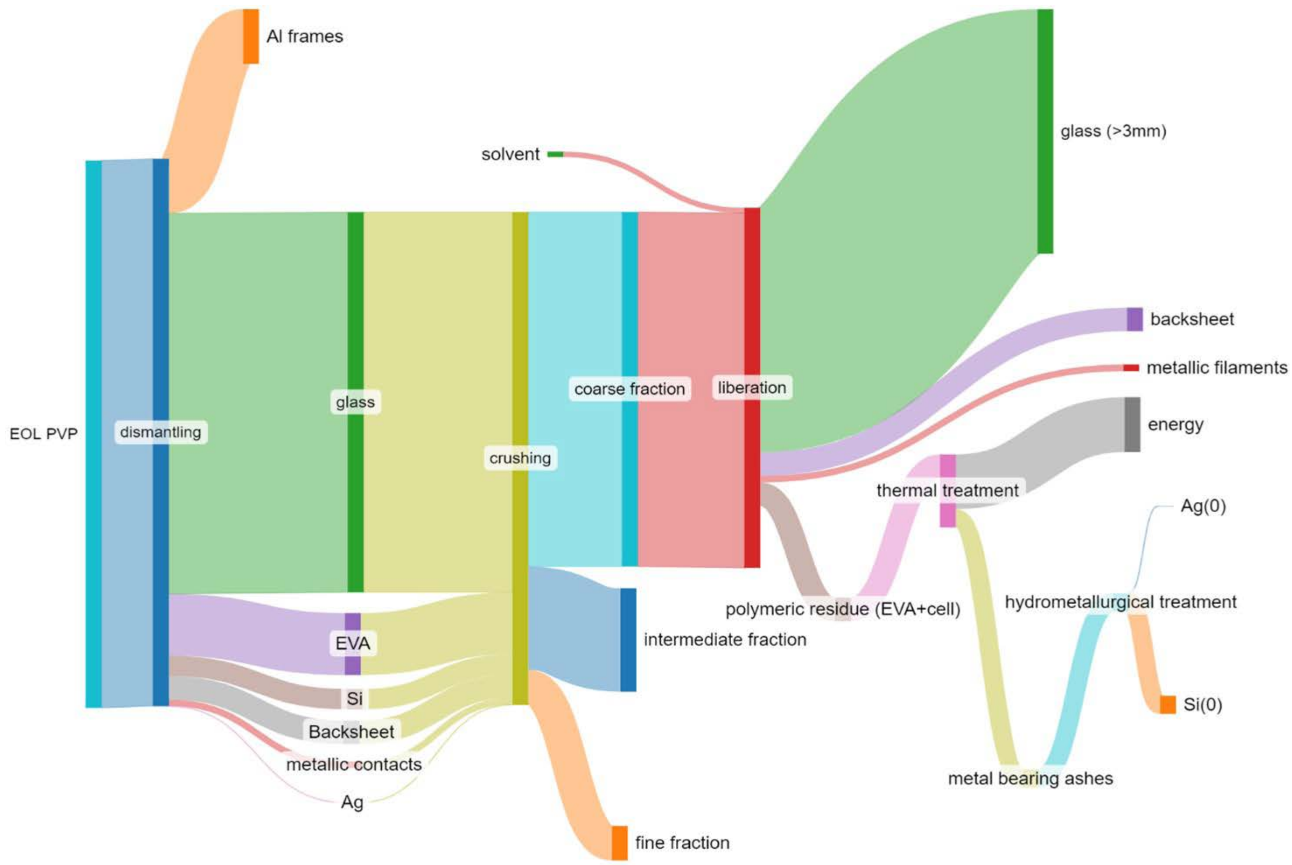

FIGURE 3: Sankey diagram of the process including frame dismantling, panel crushing, sieving, solvent lamination of coarse fraction, thermal treatment of the polymeric residue, and hydrometallurgical treatment for Ag and Si recovery.

ery was estimated at 94%. Remarkably, these figures exceed the EU Directive, which since August 2018 has raised the minimum rates for recovery and reuse and recycling rates to 85% and 80%, respectively.

In addition to improving recycling and recovery rates, the recoverable value of the implemented process has also increased. The total value of EoL-PVP was estimated by considering the composition of the input waste and the market price of the recycled fractions according to scrap prices (https://www.dailymetalprice.com) and metal quotations (https://www.dailymetalprice.com), specific quotations received for high-quality glass recycled by the Photolife process, and literature values. Market prices were fixed at: 0.7 €/kg for Al scraps; 2 €/kg for metallic filaments; 0.2 €/kg for high-quality glass; 0.001 €/kg for recycled Tedlar; 600 €/kg for Ag (https://www.capitalscrapmetal.com/prices/); and 0.5 €/kg for Si. Considering these inputs, it is possible to distribute the value among the different components of EoL-PVP (at this stage, no value was assigned to intermediate glass). According to this analysis, the intrinsic total value of 100 kg of EoL-PVP is 54 €, with Ag representing 53% of this value, high-quality glass 26%, Al scraps 13%, metallic filaments 4.5%, and Si 3.5%. The basic Photolife process, which did not include the metal recycling section, allowed for a recovery of 34% of this intrinsic value. The implemented process, allowing for the recovery of Ag and Si, increases the recoverable value to 73%. Further improvements up to 80% are also possible by optimizing the Ag recovery section.

Additionally, to further increase the recoverable value, the same optimization can be applied to the Si recovery section as residue after Ag leaching from residue ashes.

Economic analysis of the improvement proposed was specifically assessed (Rubino et al., 2020). Simulations indicate that the advanced PhotoLife process is economically feasible at a high recycling volume of 30,000 tons per year. This is largely due to economies of scale, which reduce the plant cost per unit with increased size. However, the hydrometallurgical section's size and cost are critical factors. For smaller volumes, it may be more cost-effective to exclude this section and sell the Ag-Si-EVA material to external processors. Operating costs analysis reveals that labor costs, particularly for manual dismantling of aluminum frames, are a significant constraint. Manual dismantling of frames is time-consuming and costly, and scaling up the process will significantly increase labor hours. Automating this step could improve economic feasibility and scalability. Energy balances show that the thermal treatment of EVA provides significant energy savings. At 30,000 tons per year, the exothermic degradation of EVA could produce about 12.5 MWh per batch, which can be used to heat other parts of the process, such as the leaching reactor and cyclohexane evaporator. This energy credit reduces overall energy consumption and helps meet recovery targets.

3.3 Economic feasibility of metal recovery by hydrometallurgical treatment

Feasibility of recycling process including hydrometallurgical section for metal recovery (Ag, Si) was further investigated (Granata et al., 2022) considering the decreasing concentration of precious metals in PVP due to technology improvements.

In fact, the economic sustainability of photovoltaic (PV) recycling hinges on balancing process costs with revenues from recovered materials, which depends on recycling volumes, recovery rates, and the commercial value of these materials. Silver (Ag), copper (Cu), silicon (Si), and tin (Sn) are the most valuable materials to target, with Ag being particularly significant due to its high value compared to Sn and Cu. Despite Ag's low concentration in PV waste, it often provides higher revenues than Cu and Sn. Thus, investing in Ag recovery is crucial for profitability, especially when Ag concentration is at least 0.1%. For lower concentrations, investments are less impactful at low recycling volumes but become significant at higher volumes.

Selling an Ag-Si rich fraction could be more profitable both short- and long-term, potentially reducing capital and operational expenses. For Ag concentrations around 0.2%, selling the Ag-Si fraction at about 50% of its actual value may be financially advantageous. At lower concentrations (0.05%), hydrometallurgical recovery may offer higher returns but with lower profit margins, unless the material is sold for a significant portion of its value.

The recycling fee also affects profitability. Lower Ag concentrations and PV throughputs may necessitate a recycling fee to make the process economically viable. For concentrations between 0.05% and 0.2%, suitable returns on investment (ROIs) (>20%) can be achieved without a PV fee if recycling volumes are substantial (43,000–18,000 tons/year). With decreasing Ag concentrations, the timing of investments becomes crucial; higher recycling volumes and fees are needed to maintain profitability. For instance, investing in 2025 with current Ag prices may result in positive net present values (NPVs) if recycling volumes are at least 30,000 tons/year.

All these estimates have been done with Ag price ranging from 600 to 750 $/kg (range observed during 2014-2022 period), then they are all conservative considering the current price of Ag which is over 1100 $/kg (https://stonexbullion.com/it/grafici/prezzo-argento/).

4. CONCLUSIONS

EoL-PVP are complex wastes with low intrinsic values, and both of these characteristics hinder the development of economically sustainable recycling processes with respect to other WEEE. While the primary focus in the past might have been on recovering metals (primarily silver), current recycling regulations now highlight an additional need to recover glass, which is the main component of these wastes. The low intrinsic value underscores the necessity for low treatment costs, which are generally associated with physical treatments. However, current technology does not provide products suitable for reintroduction into the glass manufacturing sector. Therefore, it is necessary to support the feasibility of recycling operations with fees for recyclers, who could then invest in dedicated lines for not only high-grade glass recovery but also for recovering valuable (Ag) and critical (Si) metal components. Economic analysis depends on various technical inputs but is also affected by the uncertainty related to the level of support that policy will provide to establish a virtuous cycle of secondary raw material recovery through the introduction of recycling fees, which need to be adjusted over time. In past activities different process options have been developed according to these trends. Solvent lamination seems to be a sustainable solution for effectively recovering high-value materials. In addition process optimisation can also allow for polymer separation improving recycling rates and recovered value significantly. The process is economically feasible at high volumes and could benefit from automation and further optimization to enhance economic viability. Overall, the process shows promise as a more efficient and higher-quality alternative to conventional physical treatments, aligning with both the recycling targets of Directive 2012/19/EU and promoting high-value recycling in accordance with CENELEC EN50625-2-4 guidelines.

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INCORPORATING INDUSTRIAL WASTES INTO MORTAR MIX PRODUCTION: A STEP TOWARDS SUSTAINABLE CONSTRUCTION

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ABSTRACT

The incorporation of calcined sludge (CS) from water treatment plants and nitrophosphogypsum (NPG) into mortar mixtures has been investigated as a potential strategy to reduce the consumption of ordinary Portland cement (OPC) and subsequently lower the carbon footprint of construction materials. Two scenarios were subjected to analysis: in the first, CO_2 emissions resulting from the treatment of NPG and CS were considered; in the second, emissions were considered negligible due to the reduced energy demand of waste treatment processes compared to OPC production. The chemical composition and mechanical properties of the mortars were evaluated through the use of X-ray fluorescence (XRF) and compressive strength testing. The mortar mixtures were prepared with a water-to-binder ratio of 0.485 in accordance with the ASTM standards. The mortar mixture containing 5% NPG and 5% CS exhibited a 6.65% increase in compressive strength, reaching 38.96 MPa after 90 days. Despite a slight reduction in early strength, long-term performance was enhanced by the formation of additional calcium silicate hydrates. Regarding sustainability, a reduction of 62.06 kg CO_2 per cubic meter of mortar was achieved in both scenarios, with more substantial reductions in Scenario 2. These results show that incorporating NPG and CS supports the circular economy by utilizing industrial by-products and provides a sustainable alternative to OPC, significantly reducing CO_2 emissions while maintaining or enhancing mechanical performance, thereby aligning with global efforts towards eco-friendly construction practices.

1. INTRODUCTION

In the Colombian Caribbean region, there are raw materials with high potential as mineral admixtures or partial replacements for Portland cement (OPC). When implemented in mortar mixes, these materials exhibit performance similar to traditional mixes due to their chemical and mechanical properties (Bohórquez González et al., 2020; Palma & Sierra, 2011). The sludges from water treatment plants (WTPS) are residues from the water purification process, studied for their potential (Chang et al., 2020). According to a report by the WTP company of Atlántico, Colombia, an average of 8.6 m^3/s is drawn from the Magdalena River (AAA, 2024; Superintendencia de Servicios Públicos Domiciliarios, 2023). Torres-Lozada et al. (2022) determined that the production of sludge, based on the volume of treated water, can range between 2% and 5%. The cited values are contingent upon the specific materials required by the treatment process at each facility. Based on a sludge production rate of 3%, it can be estimated that approximately 7,681,125 cubic meters of sludge are produced annually in the Atlantico department (Colombia) alone.

Several studies have supported the use of WTPS in the construction sector, either as a supplementary cementitious material (SCM) or in combination with other waste materials (Daza & Medina, 2017; Ruviaro et al., 2021; Daza Márquez, 2022; Mahanna et al., 2024). For example, incorporating calcined sludge in mortar mixes was evaluated by He et al. (2023), and a 10% improvement in compressive strength was achieved by adding 10% sludge ash compared to OPC mixes. Additionally, using alum sludge as a partial cement replacement in mortars was investigated by Liu et al. (2022), and enhanced resistance to high temperatures was observed. Mortar mixes containing 20% alum sludge kept higher compressive strength after exposure to temperatures of up to 800°C, surpassing sludge-free mixes by 15%

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. Similarly, the addition of up to 5% sludge in brick manufacturing was reported by De Carvalho Gomes et al. (2019), resulting in a reduced environmental impact while maintaining satisfactory mechanical properties, demonstrating its viability for use in ceramic materials. These studies indicate the potential of drinking water treatment sludge to improve mechanical properties and durability in construction applications while also contributing to waste reduction and carbon emission mitigation (Z. He et al., 2024).

On the other hand, phosphogypsum (PG), an agro-industrial byproduct from the fertilizer industry, has demonstrated chemical properties that could complement or enhance those found in WTPS for the development of ternary mortar mixes as partial replacements for OPC. Fertilizer production is expected to increase from 47 million tons in 2020 to 49 million tons by 2024, resulting in a rise in the waste generated [2]. The PG-producing industry maintains that its chemical and physical properties make it unsuitable for other industrial sectors, such as construction and agriculture. However, PG has become an essential component of cementitious materials because of its high calcium oxide content (up to 42%) (Rashad, 2017). If converted correctly, the PG byproduct of agro-industrial processes can be used as a substitute raw material for building materials (green technologies). Adding PG to hydraulic mortar mixtures is a promising approach for use. It has been acknowledged as one of the most advantageous ways to dispose of PG because of its low preparation costs and broad use, which align with the global building materials industry's drive toward green and low-carbon growth (Ohji et al., 2017). However, nitrophosphogypsum (NPG), an agro-industrial byproduct of the fertilizer industry, is a kind of phosphogypsum produced in Colombia's Caribbean region and derived from the manufacture of nitrophosphate fertilizer.

In the Atlantico department of Colombia, one fertilizer industry generates an average of 62,000 tons of NPG waste per year (Palma & Sierra, 2011). NPG contains impurities and toxic elements, the majority of which are typically deposited in open landfills for natural degradation, risking potential environmental impact. Due to this method of final disposal, the physical and chemical characteristics of this residue are not fully considered for utilization in the construction sector despite its high content of calcium oxide, a significant compound in the composition of cementitious materials (Rashad, 2017).

The present research evaluates the effectiveness of utilizing calcined residues from water treatment plants (CS) and NPG in mortars for the construction sector, expressed as the percentage of recyclable residue relative to the captured volume at the plant. The primary focus of this research lies in incorporating these residues into a value chain, aiming to contribute to the circular economy by utilizing these materials as sustainable solutions in construction.

1.1 Research significance

Incorporating industrial waste materials, such as WTPS, and agricultural byproducts like NPG into mortar mixtures presents a pivotal step towards sustainable construction. This research highlights that using these materials can significantly reduce CO_2 emissions without compromising the strength or workability of the mortar. These findings motivate the exploration of waste valorization, offering a commercially viable pathway for eco-friendly material development and aligning with global sustainability goals in the construction industry.

2. MATERIALS AND METHODS

This research involved the preparation of cubic mortar samples. The mixing, casting, and curing methods were conducted following the standards outlined in ASTM C305 (2020a), ASTM C109 (2021a), and ASTM C511 (2021b), respectively.

2.1 Mortar mixtures

Four (4) mortar mixtures were formulated, incorporating two (2) types of industrial waste. The first one corresponds to NPG, which, according to the literature, is primarily composed of CaO and SO_3 (Rashad, 2017). On the other hand, the second waste type is associated with CS, with SiO_2, Al_2O_3, and Fe_2O_3 being the primary components (Ruviaro et al., 2021). A fertilizer company from the Colombian Caribbean donated the NPG. It was calcined at 600°C for two hours, crushed, and sieved through a No. 200 sieve (75 microns) (Figure 1). The water treatment sludge was exposed to the sun for 36 hours to eliminate free water. It was then crushed, and the same procedure was followed for the NPG (Figure 2).

The waste materials were considered as a partial replacement for cement at weight-to-weight ratios with replacement proportions of 10%, as estimated according to the existing literature (Calderón-Morales et al., 2021; Chang et al., 2020; Rashad, 2017; Świerczek et al., 2018; Vouk et al., 2017). Both wastes underwent thermal treatment at temperatures of 600°C for 2 hours. The elemental chemical composition of CS and NPG, obtained through XRF analysis, along with their physical properties, is summarized in Table 1. XRF analysis was conducted on the Vanta M Series Handheld instrument. The NPG is primarily composed of SO_3 (52.74%) and CaO (39.8%), with minor amounts of SiO_2, Al_2O_3, Fe_2O_3, and MgO. In contrast, the CS material has a high content of SiO_2 (61.59%) and Al_2O_3 (21.36%), with minor amounts of Fe_2O_3 (10.09%), MgO, and other components.

Scanning Electron Microscopy (SEM) analysis of NPG revealed the presence of elongated, prismatic particles with adhered impurities. These particles were subjected to thermal treatment, resulting in their reduction (Figure 3) (Rashad, 2017; Godoy et al., 2019; Daza-Marquez et al., 2024). In contrast, the SEM images of CS revealed needle-like shapes with porous and inhomogeneous surfaces, particularly in agglomeration areas. The control of particle size resulted in an improvement in the homogeneity of NPG, whereas CS exhibited localized porosity (Figure 4) (Husillos Rodríguez et al., 2011).

The fine aggregate used was natural sand from Santo Tomas, Atlántico, with a fineness modulus of 1.98. The average particle size (D50) was 0.425 mm, while the D10, D30, and D60 were 0.268 mm, 0.335 mm, and 0.460 mm, respectively. The uniformity coefficient (Cu) was 1.72, and the

FIGURE 1: Open-pit NPG quarry.

FIGURE 2: WTP sludges.

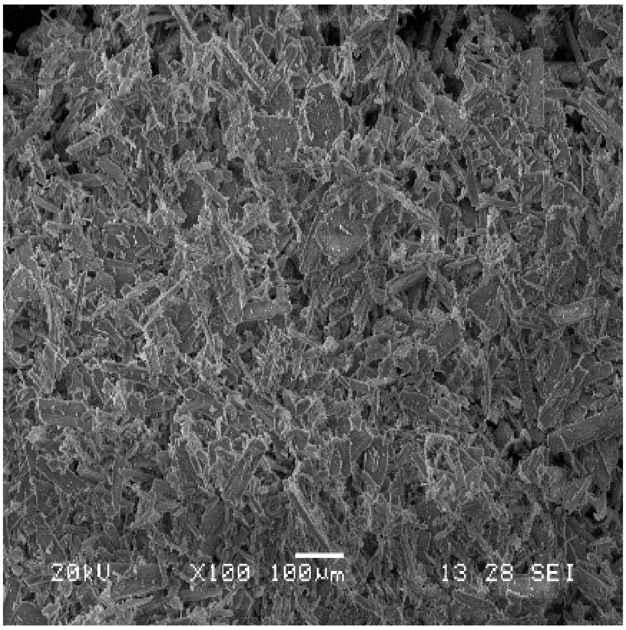

FIGURE 3: SEM images of NPG.

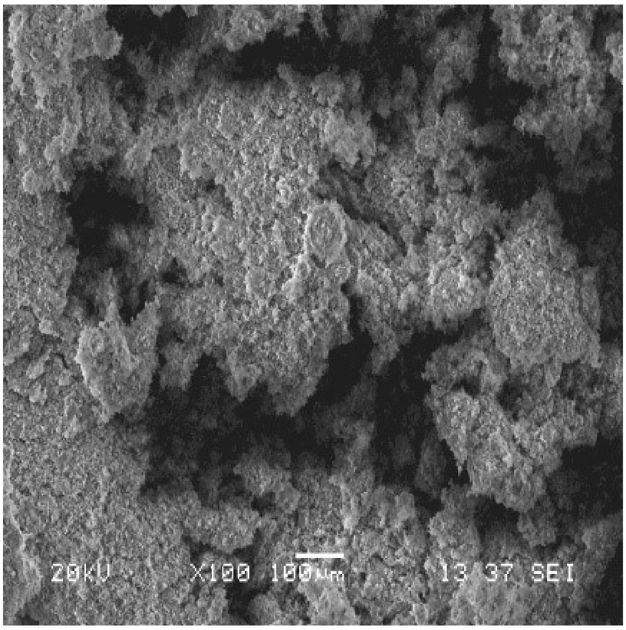

FIGURE 4: SEM images of CS.

gradation coefficient was 0.91. The specific gravity of the sand was 2.61, and its water absorption was 0.78%. These properties comply with the corresponding ASTM standards for sand characterization. The mix was prepared using tap water that met the ASTM C1602M standards (2022).

The quantities of each mortar mixture type are presented in Table 2. These typologies originate from a prior investigation conducted by the researchers (Daza-Marquez et al., 2024; Daza et al., 2023; Daza Márquez, 2022), wherein it was determined that they exhibited optimal mechani-

TABLE 1: Chemical characterization of binders.

Binder	Oxide in %						
	SiO_2	Al_2O_3	SO_3	CaO	Fe_2O_3	MgO	Others
NPG	2.48	0.69	52.74	39.8	0.07	0.18	0.83
CS	61.59	21.36	0.18	1.39	10.09	1.44	3.95

TABLE 2: Quantities of material categorized by type in kg/m^3 with a w/b ratio of 0.485 to produce 1 m^3 of mortar.

Mix	Sand	OPC	NPG	CS	Water
M0	1833.28	666.65	0.00	0.00	325.84
MN10	1833.28	599.98	66.66	0.00	325.84
MN5C5	1833.28	599.98	33.33	33.33	325.84
MC10	1833.28	599.98	0.00	66.66	325.84

cal performance after 90 days of curing according to the guidelines of ASTM C109 (2021a).

2.2 Experimental methods

Once the mixing process was completed, the workability of the fresh mortars was evaluated according to the standard procedure outlined in ASTM C1437 (2020b). This method, commonly known as the flow table test, measures the consistency and flow of the mortar mix (Figure 5). To evaluate the mechanical performance of the mortars, the compressive strength of mortar cubes with dimensions of 50 mm × 50 mm × 50 mm was tested at intervals of 28 and 90 days according to ASTM C109 (2021a). For each mortar type, three cubes were prepared and tested (Figure 6), with the average compressive strength reported.

2.3 Environmental feasibility and system boundaries

This environmental analysis aims to investigate the environmental impact of producing a mortar mixture using different industrial waste materials as a partial replacement for cement. Through a life cycle analysis, an estimation of CO_2 emissions generated from each of the experimentally proposed typologies for producing 1 m^3 of mortar was calculated (functional unit).

In this study, two scenarios were considered. In the first scenario, waste materials (NPG and CS) are commercialized within the construction sector, and their treatment results in an environmental burden due to emissions generated during the drying and calcination processes. In the second scenario, a greater environmental benefit is assumed, where the waste materials are used in a way that results in near-zero emissions due to the lower energy demand of their treatments compared to cement clinker production.

2.3.1 *CO_2 evaluation and boundaries*

The following parameters were considered: the quantity of material to be treated, treatment method, CO_2 emissions, and performance based on the waste's availability. A cradle-to-gate approach is used to construct the examined system (ISO 14040:2006 (2020)). The system boundaries, illustrated in Figure 7, encompass the raw materials and all subsequent processes, culminating in the mixed mortar at the gate.

The CO_2 emissions arising from each material employed in mortar production were determined using the method reported by Yang et al. (2013), using equation Eq. (1). Where Ce is the total carbon emission of the element or material, $CO_{2\text{-}M}$ is the carbon emissions in the material phase (including the treatment), $CO_{2\text{-}P}$ represents the emissions of the production phase. Emissions associated with material transportation were not considered because of their high variability.

$$C_e = CO_{2-M} + CO_{2-P} \quad (1)$$

CO_2 emissions linked to materials ($CO_{2\text{-}M}$) were calculated using Eq. (2), following reference values of the existing literature. W_i represents the volume unit per weight in kg/m^3, while $CO_{2(i)\text{-}LCI}$ is the constant emission in kg per kg of material in $CO_{2\text{-}kg/kg}$.

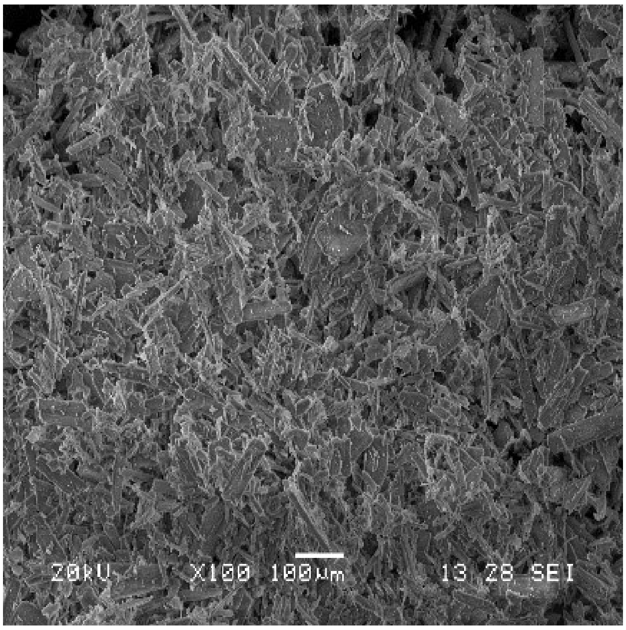

FIGURE 5: Measurement of the slump flow in the mortar.

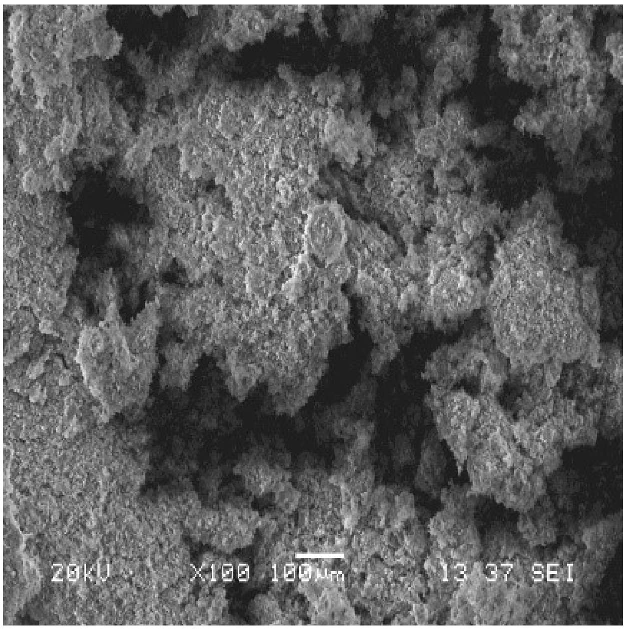

FIGURE 6: Compressive strength assessment setup.

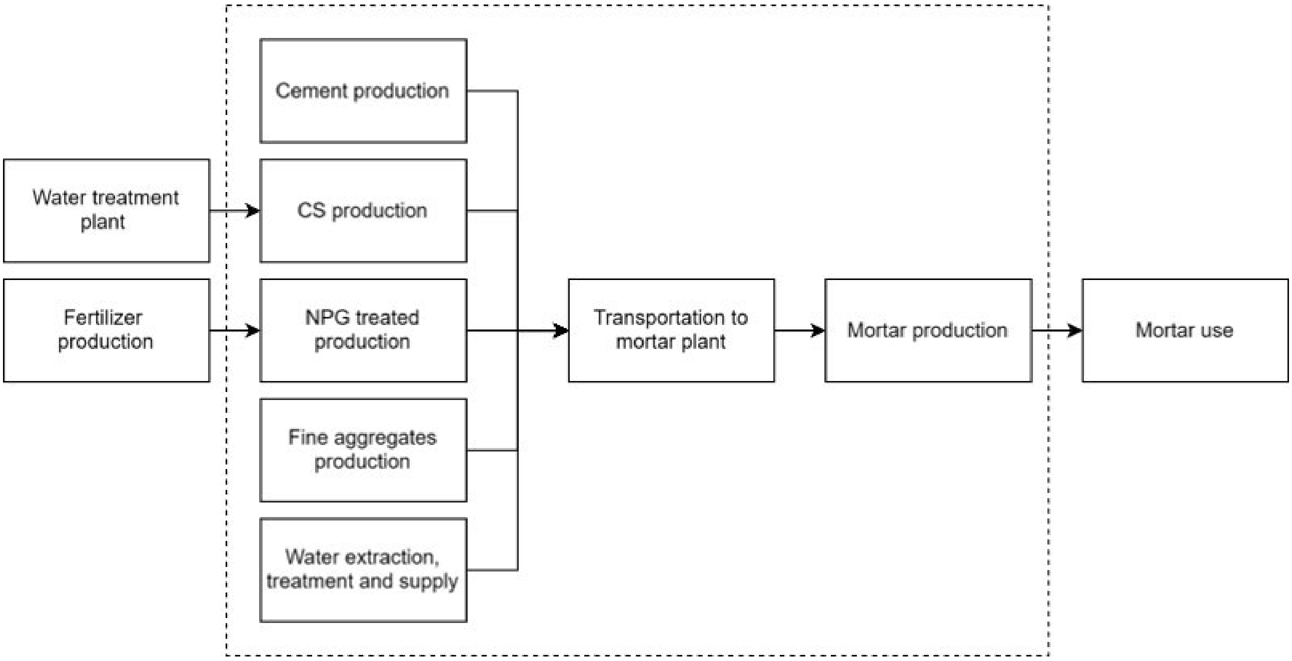

FIGURE 7: System boundaries considered in the environmental analysis.

2-kg/kg.

$$CO_{2-M} = \sum_{i=1}^{n} (W_i \times CO_{2(i)-LCI}) \quad (2)$$

2.3.2 Data collection

The data utilized to formulate the LCA of raw materials was obtained from peer-reviewed scientific literature. The composition of the mortars from previous studies was used as a reference to assess the environmental impact of mortars containing mining residues. Table 3 outlines the CO_2 emissions (in kg per kg) associated with 1 kg of cement, sand, water, NPG, and CS, along with the corresponding data sources.

3. RESULTS AND DISCUSSION

3.1 Workability and compressive strength

Figure 8 summarizes the workability and compressive strength results for all the mixtures. The control mix (M0) showed the highest workability, with a flow percentage of 131.90%. In comparison, the mix with 10% NPG (MN10) had a 3.44% reduction in flowability, achieving a flow of 127.37%. This reduction likely occurs because of NPG absorbent properties, which reduce the free water available for cement hydration. Its porous nature increases the water demand, limiting the mobility of the mortar during mixing (Bhadauria & Thakare, 2006; Rashad, 2017).

The MN5C5 mixture, containing 5% NPG and 5% CS, experienced a more pronounced decrease in flowability, with a value of 115.48%, reflecting a 12.48% drop compared to M0. This significant reduction in workability likely results from the combined effects of both supplementary SCMs. Their porous nature causes them to absorb more water, limiting the free water available for cement particle dispersion and hydration. These findings align with previous studies where highly porous SCMs like NPG and calcined sludge reduced workability by absorbing excess water (Chen & Poon, 2017). On the other hand, the MC10 mixture, with 10% CS, also showed reduced workability, achieving a flow of 117.51%, representing a 10.90% decrease compared to the control sample. As in MN5C5, the porous structure of CS increases the water demand, negatively affecting the mortar's flowability (Krejcirikova et al., 2019).

The M0 achieved a compressive strength of 36.53 MPa. A 9.41% reduction in compressive strength, reaching a value of 33.09 MPa, was observed in the MN10 mix containing 10% NPG. This decrease in strength is attributed to the reduced reactivity of nitrophosphogypsum, delaying the formation of C-S-H during the hydration process (Alaghebandian et al., 2020). Furthermore, the crystalline structure of NPG is believed to limit the development of strong bonds within the cement matrix, negatively affecting the mechanical properties of the mortar. In contrast, the MN5C5 mix, with 5% NPG and 5% calcined sludge, achieved the highest compressive strength, with a value of 38.96 MPa, representing a 6.65% increase compared to the control sample. It is suggested that the combination of NPG and calcined sludge in these proportions enhanc-

TABLE 3: CO_2 emissions per material for mortars.

Material	CO_2-kg/kg	References
Cement	0.9310	(Yang et al., 2015)fly ash (FA
Sand	0.0026	(Yang et al., 2015)fly ash (FA
Water	1.96× 10^{-4}	(Yang et al., 2015)fly ash (FA
NPG	0.1414*	(Nana et al., 2023; Lv et al., 2024)
SC600	0.0356*	(Huang et al., 2024; Lv et al., 2024)

** The corresponding value already includes solar drying, calcination, and crushing emissions.*

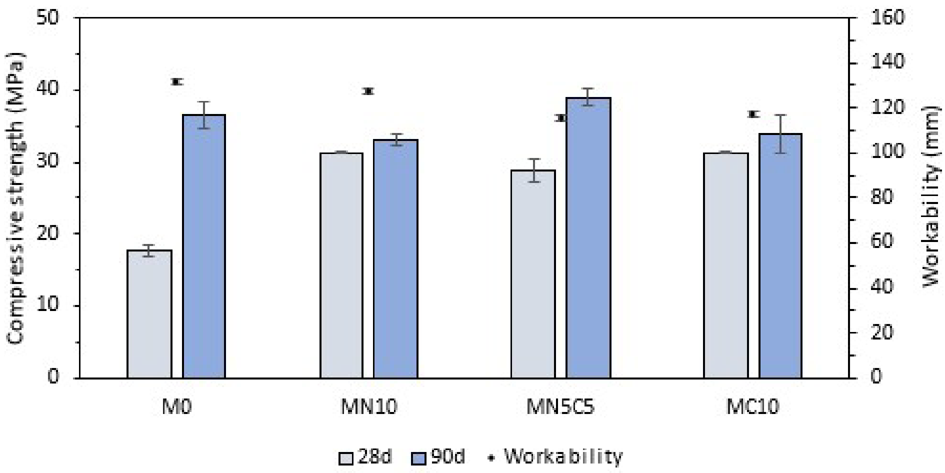

FIGURE 8: Workability and compressive strength in mortar mixtures.

es the long-term mechanical performance of the mortar. The high reactivity of calcined sludge likely accelerated the formation of additional C-S-H, contributing to the improved compressive strength observed at 90 days (Hewlett & Liska, 2019; Lothenbach et al., 2011).

A compressive strength of 33.83 MPa was recorded for the MC10 mix, containing 10% CS, showing a reduction of 7.39% compared to M0. While calcined sludge is highly reactive, it is believed that the partial substitution of cement with 10% calcined sludge reduced the available portlandite (CH) for reaction, thus limiting the formation of C-S-H and negatively affecting the compressive strength of the mortar.

3.2 Environmental feasibility

It is essential to determine the quantity of each material required per cubic meter of mortar, measured in the same units. In total, 666.65 kg/m^3 of OPC is necessary to produce 1 m^3 of traditional mortar with a water-to-cement ratio of 0.485. The relationships between quantities and CO_2 emissions in kg/kg and kg/m^3, based on the specified dosages by type (Table 2), are presented in Table 4.

For scenario 2, it is noteworthy that, because of the thermal treatment applied to mineral admixtures (at temperatures of 105°C and 600°C), which is lower than the clinkerization temperature of cement (approximately 1350°C), and with only 24 hours and 2 hours required for the drying and calcination processes, respectively, the CO_2 emissions for NPG and CS approached zero (Jacoby, 2020). It is important to highlight that, since both waste materials are currently being studied for their potential use in the construction sector and are not yet commercialized, the environmental impact (i.e., CO_2 emissions, renewable energy sources) is attributed to the primary process that generates these wastes (Almusaed et al., 2024).

TABLE 4: CO_2 emissions (kg/kg) of each material used in mortar mixes.

Mix	Sand	Cement	NPG[(a)]	CS[(a)]	Water	Total
M0	4.77	620.65	0.00	0.00	6.39×10^{-2}	625.48
MN10	4.77	558.58	9.43	0.00	6.39×10^{-2}	572.84
MN5C5	4.77	558.58	4.71	1.19	6.39×10^{-2}	569.31
MC10	4.77	558.58	0.00	2.37	6.39×10^{-2}	565.79

[(a)] *The CO_2 emissions generated by waste are close to zero in scenario 2.*

Once the CO_2 emissions per type of material are known based on their dosage, the next step involves calculating the total emissions released to produce one cubic meter of mortar according to the mix typology. The results obtained from both scenarios are presented in Figure 9. Mix typologies formulated with NPG and CS contribute to reducing at least 62.06 kg of CO_2 for each cubic meter of prepared mortar.

In Scenario 1, which incorporates CO_2 emissions from the processing of NPG and CS based on the values presented in the literature, a notable reduction in emissions is observed as the cement content is diminished. The highest emissions were observed in the control mix, designated as M0, with 625.48 kg of CO_2 equivalent. The primary source of these emissions was identified as the cement. A reduction to 572.84 kg CO_2 is achieved in mix MN10, where 10% of the cement is replaced by NPG, a reduction of 8.4%. The MN5C5 mix, which contains 5% NPG and 5% CS, further reduces emissions to 569.31 kg CO_2. The most significant reduction is observed in MC10, where replacing 10% of the cement with CS reduces emissions to 565.79 kg CO_2, a 9.5% reduction compared to the control. Overall, substituting cement with NPG and CS, especially CS because of its lower emission factor, results in significant CO_2 reductions. In Scenario 2, where CO_2 emissions from NPG and CS are considered negligible because of their processing methods, total emissions are further reduced compared to Scenario 1. The control mix (M0) remains unchanged at 625.48 kg of CO_2, as no alternative materials are introduced. However, for MN10, MN5C5, and MC10, the emissions decrease uniformly to 563.41 kg of CO_2, representing an additional reduction compared to Scenario 1. This consistent reduction across the modified mixes highlights the significant environmental benefit of incorporating NPG and CS, especially when their emissions are minimized (Singh et al., 2023).

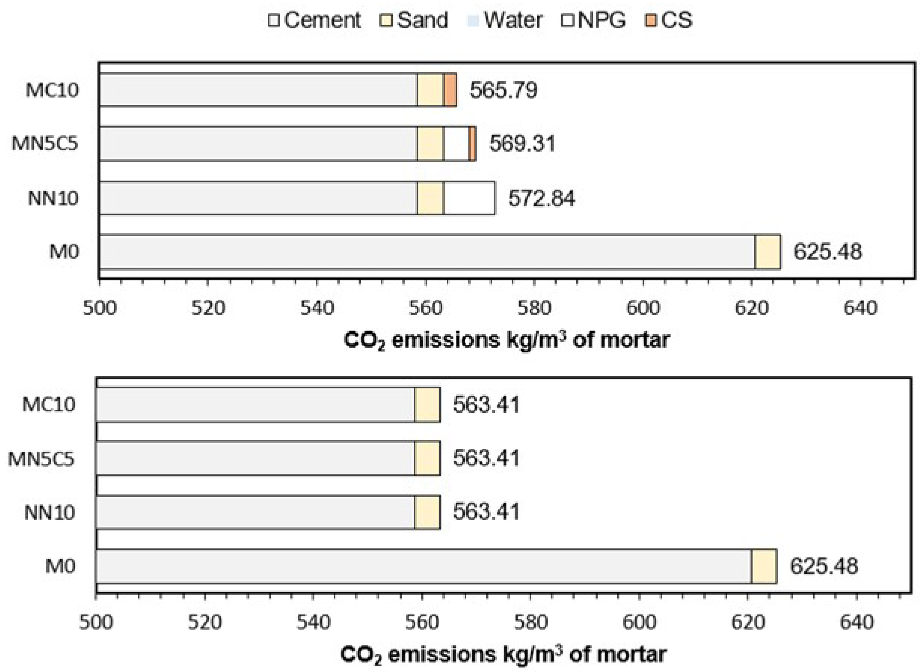

FIGURE 9: CO_2 Emissions based on mixed typology (a) scenario 1 (b) scenario 2.

By correlating the environmental benefits of incorporating each of the proposed mortar samples with the mechanical properties of masonry mortars, it could be established that the simultaneous and individual implementation of NPG and CS does not significantly affect the properties of traditional mortar. Furthermore, it represents an environmental advantage in terms of CO_2 emissions into the atmosphere.

4. CONCLUSIONS

The present study investigates the potential use of CS from water treatment plants and NPG as partial replacements for OPC in mortar mixtures, with a particular emphasis on enhancing environmental sustainability. The following conclusions were derived from the study:

All proposed mortar typologies exhibited comparable compressive strength values to the control sample at 90 days, with an average strength of 35.64 MPa across the different mixes. The compressive strength at early ages exhibited a range of results, with reductions of 7.39% and increases of 6.65% compared to the control sample. Although there are slight variations in mechanical performance, the environmental benefits of reduced CO_2 emissions and waste utilization make these differences worthwhile.

Although there were slight reductions in early strength, the mixture comprising 5% NPG and 5% CS exhibited a 6.65% improvement in compressive strength at 90 days, suggesting long-term performance comparable to traditional OPC mixtures. From an environmental perspective, Scenario 1 (which accounts for CO_2 emissions from NPG and CS processing) resulted in a notable reduction in carbon emissions compared to the control mixture. The environmental impact was further reduced in scenario 2 (where the CO_2 emissions of NPG and CS are considered negligible). This was due to the lower energy demand of processing NPG and CS compared to the production of OPC. This demonstrates that, despite slight mechanical variations, these industrial byproducts maintain the mechanical integrity of the mortar while significantly lowering its carbon footprint. This offers a more sustainable solution in both processing scenarios.

The utilization of CS and NPG contributes to the circular economy by enhancing the value of industrial by-products, minimizing waste, and promoting sustainable construction practices without compromising the mechanical performance of cement mortars.

Overall, the findings highlight that CS and NPG are viable alternatives to OPC in mortar mixtures in the Caribbean region of Colombia, offering both environmental and technical advantages. Further research is required to optimize these materials for maximum sustainability benefits.

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SUSTAINABLE MATERIAL WASTE MANAGEMENT PRACTICES IN THE TEXTILE AND APPAREL SECTOR: A CASE STUDY IN BANGLADESH

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ABSTRACT

This research extensively evaluated the material waste management practices of thirteen manufacturing facilities in Bangladesh's textile and apparel sector, specifically associated with fast fashion production. Employing a multiple case study methodology, both qualitative and quantitative data were collected via concise surveys and subsequent discussions in focused groups. The textile and apparel industry are notorious for producing significant material waste, necessitating urgent and effective management to mitigate its detrimental environmental impacts. Strategically managing waste materials from the outset of textile production presents a substantial research opportunity, aligning with circular economy principles and supporting Bangladesh's pursuit of SDG 12. Additionally, this inquiry involves systematically identifying and classifying waste materials generated across diverse production phases while exploring options for recycling both pre and post-consumer textile waste. From this study it was found out that textile production in the country faces material inefficiency, with 78% of materials used leading to a shortfall of 77.21 USD per 100 kg of clothing. It also focuses on the importance of technology driven managerial and comprehensive strategies for sustainability and addressing forthcoming challenges.

1. INTRODUCTION

The textile and apparel manufacturing industry in Bangladesh has been a significant driver of economic growth, contributing over 84% of the country's export earnings and 12.3% of its GDP, with an annual income of 42.613 billion USD in the Fiscal Year 2021-22 (fibre2fashion, 2023). With more than 7,000 operational industries employing over 4.5 million people, this sector plays a pivotal role in the nation's economy (Shahajada Mia & Masrufa Akter, 2019). However, it is known for its high ecological footprint, particularly in terms of water, energy, and chemical consumption in processes like yarn and fabric manufacturing, dyeing, washing, and finishing. These water-intensive processes contribute to the heavy dependence on groundwater. This consumption leads to a yearly drop in groundwater levels by 2.0-3.0 meters, with a projected depletion rate of 5.10 meters/year by 2030 (Moshfika et al., 2022).

The textile sector in Bangladesh released significant quantities of wastewater in 2011, 2015, and 2020, with discharge volumes reaching 145.0 million m^3, 201.0 million m^3, and 317.0 million m^3, respectively (Miah et al., 2023). The substantial amounts of wastewater have considerable detrimental impacts on the physical, chemical, and biological attributes of aquatic ecosystems, thereby presenting substantial risks to public health, wildlife, and ecological systems (Rafiq et al., 2024c). Approximately 20% of freshwater pollution in Bangladesh can be attributed to the processes of fabric processing and dyeing, which release untreated or partially treated effluents (Gulfam-E-Jannat et al., 2023). Moreover, due to the fast fashion trend driving an upsurge in apparel production, the textile industry produces a significant amount of waste across multiple stages, encompassing spinning, knitting/weaving, dyeing, apparel manufacturing, and finishing (Koszewska, 2019). The global textile industry is one of the most environmentally damaging sectors, resulting in the disposal of more than 1.1 billion kilograms of textile waste into landfills annually (Waste to Fashion, 2022). Recycling efforts are restricted in scope, resulting in a considerable portion being funneled into landfills (Rafiq et al., 2024a) or subjected to incineration, as brought to light by research conducted by Pensupa et al. in 2017 and Niinimamp et al. in 2020. In Bangladesh, approximately 400,000 tonnes of pre-consumer textile waste are generated each year, and only 5% of this waste

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is locally recycled (The Business Standard, 2023). Among this waste is entirely reusable cotton material, which holds an estimated worth of approximately 100 million USD, as reported by Pavarini in 2021. Forecasts indicate that the global volume of textile waste is expected to increase by 60% annually from 2015 to 2030. In the context of Bangladesh, this waste is referred to as 'jhoot' (Patnaik & Tshifularo, 2021), and its handling has evolved into an escalation because of the immediate environmental ramifications. Research in Bangladesh has pinpointed cutting remnants as a major contributor to overall waste generation (Rahman, 2016). Cutting stands out as the primary origin of material waste in the garment industry, with this waste type classified as pre-production waste, encompassing unused fabric, cutting leftovers, surplus materials, and spreading loss (Cao et al., 2022). Importantly, despite these challenges, roughly 87% of the world's textile waste ends up in landfills, contributing to solid waste problems. Notably, as much as 90% of this waste can be transformed into economically valuable products through the application of reuse and recycling methods (Moazzem et al., 2021). Moreover, the integration of post-consumer waste (PCW) in textile production presents a promising solution for mitigating environmental concerns related to waste generation and resource depletion in the industry (Singh & Ordoñez, 2016). Given the textile industry's ongoing efforts to navigate sustainability predicaments, the incorporation of PCW components into manufacturing procedures provides a potential pathway to advance circularity and diminish reliance on new materials. This comprehensive strategy grapples with the diverse issues posed by textile waste and unveils avenues for sustainable techniques and resource preservation in the sector. This research focuses on evaluating how export-oriented factories in Bangladesh adhere to circular economy principles in handling wastewater treatment and material waste throughout the production and post-production stages. It aims to quantify pre-consumer material waste generation in spinning, fabric manufacturing, wet processing, and apparel production. The study proposes a conceptual model for managing both pre-consumer and post-consumer textile waste (Akter et al., 2022), emphasizing recycling within the circular economy framework (Haque et al., 2024).

2. METHODOLOGY

Utilizing an exploratory approach, this study adopted a multi-case methodology. Participant selection hinged on their industry experience, relevance to the research theme, and domain knowledge. Initial outreach to potential case companies was established through both email and direct phone communication. The selection criteria revolved around their location in Dhaka, Bangladesh, involvement in textile and apparel manufacturing geared toward exports, and their openness to sharing information for research purposes. Despite outreach efforts directed at 15 unique companies spanning various sectors of the textile and apparel production chain, only 8 companies ultimately granted their participation to share their information on waste. These selected companies varied in size and product categories, as outlined in Table 1. They were primarily located on the outskirts of Dhaka, the country's major textile industry hub. The research encompassed an in-depth examination of Bangladesh's textile and apparel industry using a semi-structured questionnaire and focus group discussions (Figure 1).

Data collection was carried out by emailing the questionnaire to company contacts, followed by follow-up communications via phone and email and focus group discussions. Data for textile waste was obtained from their data

TABLE 1: Summary of table waste, excess inventory of the textile studied.

Type of manufacturing facility	Annual production capability in kilograms	Final product	All values are in kg calculated for every 100 kg of the final product				
			Raw Material Consumed, kg	Material Wasted, kg	Excess Inventory, kg	Total Conversion Rate, %	Respondent Position
S1: SpinningMills	5,682,000	Finished Yarn: Ring and open-end	122.2	36.5	24.3	78%	Deputy Manager, Production
S2: Spinning Mills	6,852,000	Finished Yarn: Carded and combed yarn					Manager, Quality Assurance
S3: Fabric Manufacturing: Knitting	5,868,789	Knit Fabric: T-shirt, jersey	111.3	3.2	6.8		Manager, Knitting
S4: Fabric Manufacturing: Weaving	5,375,432	Woven Fabric: Woven and denim fabrics, jeans					Manager, Weaving
S5: Wet Processing	6,800,000	Finished fabric	119.2	16.2	13.6		Manager, Dyeing, and Finishing
S6: Wet Processing	5,960,000	Finished fabric					Manager, Dyeing, and Finishing
S7: Apparel Manufacturing: Knit	4,650,000	Apparel: T-shirt, jersey, polo shirts	159.2	11.2	8.2		Production Assistant General Manager
S8: Apparel Manufacturing: Woven	4,570,942	Apparel: Denim Pants, Jeans					Manager Production

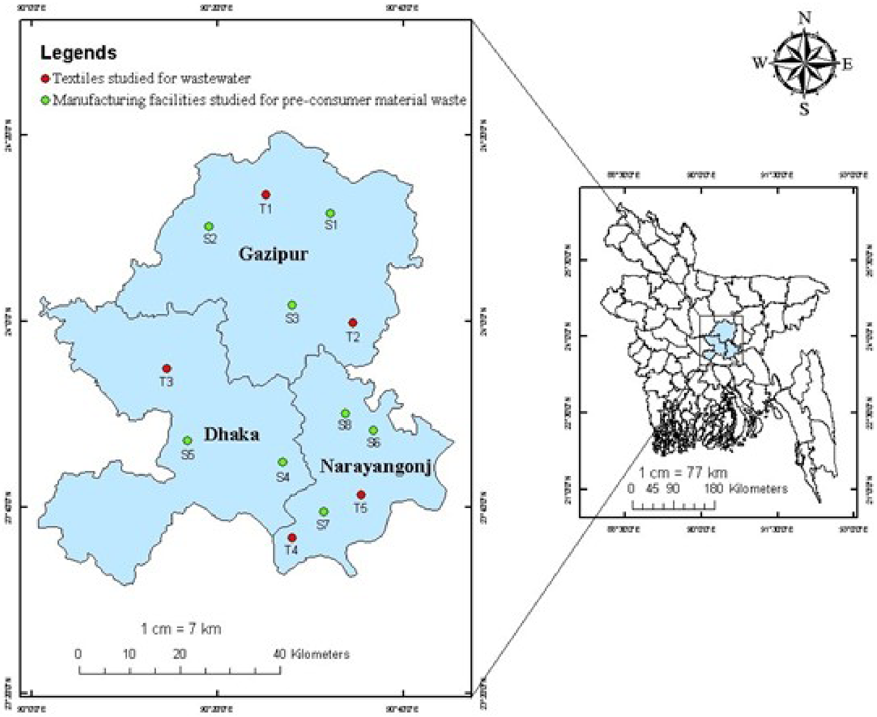

FIGURE 1: Response plot of temperature and sample quantity on % copper recovery.

log over a one-year duration (June 2022- 2023) to derive an average estimate.

The study's goal was to assess economic losses related to waste data and calculate material consumption, waste, and excess inventory in the production of 100 kilograms of apparel. The analysis simplified the final product's representation in kilograms and examined these factors across various production stages. Furthermore, the research explored how production waste could be turned into valuable products, shedding light on the hidden aspects of textile and apparel waste management. Data was collected through interviews and discussions with eight industry experts, covering waste management and local trade. The study also tracked local markets engaged in textile waste trading, identified their destinations, and investigated the processes for converting waste materials into value-added products.

3. AND DISCUSSIONS

3.1 Loss of materials and their associated value during the production processes

The research findings illustrate the material flow mapping (MSM) of textile and apparel manufacturing within the surveyed factories, providing quantitative insights into material usage and wastage at various production stages. The conversion rate reveals the number of materials needed to produce 100 kg of output across all relevant industries in the production chain. According to our calculations, a total of 512 kg of materials were employed to manufacture 400 kg (comprising 100 kg of yarn, 100 kg of greige fabric, 100 kg of finished fabric, and 100 kg of apparel) of end products, equivalent to 78% when expressed as a percentage. Further elaboration of the information in table 1 is provided below.

Spinning: The textile and apparel industry encompasses a range of fibers, including natural ones like cotton, wool, silk, flax, hemp, and synthetic fibers such as polyester, nylon, acrylic, and polypropylene. In Bangladesh, the primary emphasis is on materials based on cotton. Yarn production involves the processing of cotton into yarns using a ring/rotor spinning method, while synthetic fibers are converted into yarns through a melt/dry/wet spinning process. Research findings indicate that, for every 100 kg of yarn produced, 122.2 kg of cotton is consumed, resulting in 36.5 kg of waste and an excess inventory of 24.3 kg. The principal types of waste include cotton lint, damaged yarn, and unfinished cones.

In the spinning process, which typically yields yarn priced at 3.18 USD/kg, the waste generated amounts to 193.34 USD. The substantial disparity between production and delivery raises concerns. Spinning waste exceeds that of other processes, with only a fraction being recycled, while the majority is sold locally. Material usage in this context is influenced by the quality of the fiber. On the other hand, wastage consists of the total process loss (which is often unavoidable) and the amount rejected due to quality concerns.

Fabric manufacturing: The textile and clothing industry primarily centers on the production of knitted and woven textiles, which are essential for making garments. On average, crafting 100 kg of fabric necessitates approximately 110.4 kg of yarn, resulting in 2.5 kg of discarded materials and an additional 7.5 kg stored in inventory. Common waste materials include leftover yarn and defective fabrics like unfinished greigefabric, fly fabric, and scrap yarn. Interestingly, fabric production incurs the least value loss among all processes, with a loss of 31.4 USD. A significant part of the waste comes from surplus yarn, and the remaining is from faulty fabrics. Unused yarn waste is marketed as 'jhut,' while rejected fabrics are sold at reduced local prices.

Wet Processing: Comprising operations such as dyeing, printing, finishing, and washing, the wet processing stage represents a pivotal component. Within this stage, greige fabric is exposed to an array of chemicals, auxiliary substances, and dyes to attain the intended properties and hues. On average, the conversion of 119.2 kg of greige fabric is required to yield 100 kg of finished fabric, consequently leading to an additional stock of 13.6 kg. Fabric dyeing, in particular, results in an average waste of 16.2 kg per 100 kg, often due to issues like uneven dyeing and fabric imperfections. This waste includes defective colored fabric and surplus finished fabric. In the wet processing sector, the loss amounts to 160.92 USD for every 100 kg of processed fabrics. In brief, wet processing is geared toward enhancing greige fabric's properties and colors but generates notable textile waste due to uneven dyeing caused by fabric defects and issues related to GSM (grams per square meters), leading to excess inventory and finished fabric waste.

Apparel Manufacturing: As per our data findings, an average of 159.2 kg of finished fabric finds application within the cutting section for every 100 kg of final products. Additionally, there's a dedication of 8.2 kg of final garments marked as surplus inventory, while the process incurs a loss of 11.2 kg of materials. These losses emanate from fabric cuttings in both the cutting and sewing phases, along with the presence of surplus development samples and excess apparel. Material costs significantly contribute to shaping the overall expense structure in the apparel industry. It's a common industry norm to produce an extra 5–15% of materials to account for potential deficiencies, especially concerning quality concerns in export orders. Furthermore, the magnitude of discarded cut fabric is notably substantial. Our investigation reveals a total shortfall of around 77.21 USD for every 100 kg of clothing manufactured in production facilities. It's crucial to emphasize that roughly 32.64 USD of these deficits result from surplus inventory, with the remaining 44.57 USD linked to the disposal of cut fabric and rejected garments.

The inquiry reveals a substantial clandestine marketplace known as the "stock lot," specializing in surplus inventory apparel and "jhoot." This market deals with both reconditioned and newly crafted garments, often discreetly acquired from garment manufacturers. Local regulations are in place to safeguard the domestic market by prohibiting the sale of excess inventory clothing or fabric produced under a duty-free bonded warehouse license. Stock lot items are traded based on weight, with their pricing varying according to product categories and seasonal trends.

Cut-fabric waste comprises small fabric remnants, typically measuring between 2 to 125 mm. High-quality small fabric scraps are meticulously sorted and processed to create "garment fiber," a pivotal element in the production of recycled-content apparel. Lower-quality cut-fabric waste is shredded and repurposed for various applications, including cushion fillings, mattresses, and upholstery. The cost of cut-fabric waste can vary from approximately USD 0.16 to USD 0.39/kg. In contrast, recycled cotton fiber is valued at around USD 0.45/kg, significantly less expensive than regular cotton fiber. Furthermore, "cut-fabric panels" of diverse sizes are generated during apparel production. Medium-sized panels are transformed into smaller-sized clothing, while larger panels serve purposes such as boutique shop items, bag production, and the creation of cushion and quilt covers, contributing to local garment manufacturing. This recycling process effectively transforms jhoot waste into value-added products.

3.2 Implementing Circular approaches for Recycling Pre- and Post-Consumer Textile Waste to support Sustainable Development Goals

In 2022-2023 period, Bangladesh approximately experienced a 5.6% increase in domestic cotton bale consumption, reaching 9.31 million bales (The Daily Star, 2023). This coincides with major global brands like H&M, Tommy Hilfiger, GAP Inc., Zara, VF Corporation, Hugo Boss, and Ralph Lauren pledging to shift to recycled fibers by 2026 (Harnessing MMF Potential Vital for Bangladesh Textile and Apparel Industry, 2023). This transition offers Bangladesh a valuable opportunity to enhance its production of man-made fibers and strengthen sustainability initiatives. The textile sector plays a substantial part in environmental harm and its impact on climate change. To illustrate, textile waste takes up 5% of landfill space, and the procedures related to textile treatment account for a considerable 20% of freshwater pollution. This underscores the critical need for the adoption of circular economy principles, where recycling is a pivotal and indispensable element. Annually, Bangladesh generates approximately 400,000 tonnes of pre-consumer textile waste, with only 5% being recycled locally (The Business Standard, 2023). The export of post-industrial textile waste, known as "Jhoot," has been on the rise, creating a market of more than 167 million USD (The Story of Waste Fabric (Jhoot), 2020). Despite limited official data on garment manufacturing by-products, records indicate Jhoot exports reached US$64.95 million in FY 2018-19. Bangladesh is home to about 20 recycled fiber factories with a combined capacity of 2,400,000 tonnes, primarily using garment waste as their raw material source (fibre2fashion, 2023). However, the country faces significant challenges, including low local recycling rates (5%), incineration and landfilling of 35% of textile waste, and the export of 60% at higher costs (Juanga-Labayen et al., 2022). Local stakeholders purchase textile waste from textile businesses informally and then export it at a higher price. This business model thus benefits both parties, which explains why local recycling rates are as low as only 5%.This highlights the urgent need to boost domestic recycling efforts and improve textile waste management in the nation. Figure 2 illustrates a recommended closed-loop system aimed at recycling both pre and post-consumer textile waste. This strategy aligns with Sustainable Development Goal 12, fostering responsible production and consumption (Rafiq et al., 2024b), reducing strain on energy and resource-limited economies. Additionally, it facilitates economic growth and

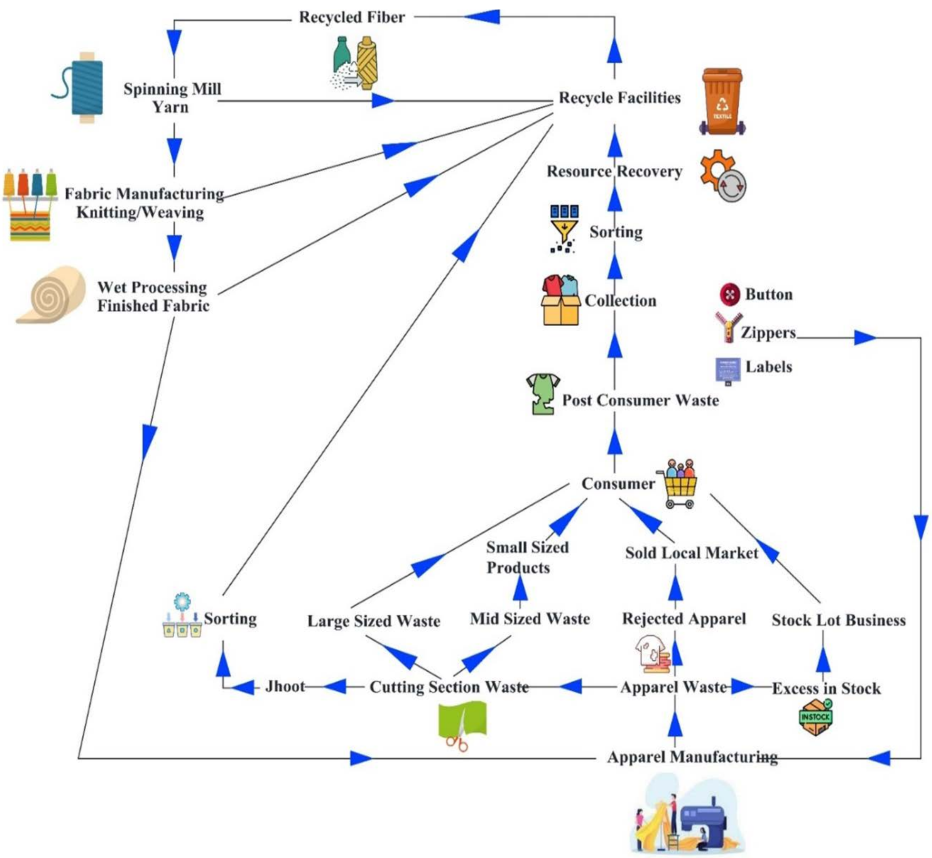

FIGURE 1: Response plot of temperature and sample quantity on % copper recovery.

employment opportunities through the implementation of these circular approaches. This approach is novel and new in context of Bangladesh and requires through study with the stakeholders to overcome the barriers incorpating the circular approach to manage the textile waste.

4. CONCLUSIONS

Promoting a circular approach to managing textile material waste and wastewater, involving both pre- and post-consumer phases, provides significant advantages and guarantees the enduring sustainability of Bangladesh's textile sector. This research findings includes,

- Material utilization inefficiencies in textile production pose significant challenges, with 78% of materials employed resulting in 512 kg of materials to manufacture 400 kg of end products.
- The apparel industry norm of producing an additional 5-15% of materials to address potential deficiencies and the substantial discard of cut fabric contributes to a shortfall of around 77.21 USD for every 100 kg of clothing manufactured.
- This study also suggests a circular economy approach to recycle pre and post textile material waste to foster sustainable development goals in a resource challenged economy.

Therefore, through the recycling of textile waste materials, there is a potential reduction in cotton imports, ultimately lessening the overall water-intensive cotton production. This initiative also introduces a new sector capable of generating revenue and employment opportunities, contrasting with the current practice of landfilling, or exporting these valuable materials. Decreasing landfill use contributes to methane emission reduction. In summary, embracing circular approaches to manage textile waste can bolster resource-limited economies like Bangladesh in the long term while supporting Sustainable Development Goal 12 (responsible production and consumption).

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FULL-SCALE CO-COMPOSTING OF SEWAGE SLUDGE AND WASTE MATERIALS AT VARIOUS MIXING PROPORTIONS AND AERATION CONDITIONS TO PRODUCE STABILIZED COMPOST

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ABSTRACT

Two different sewage sludge (SS) waste materials were commercially co-composted with wood chips (WCh) and grass to determine their conversion potential and elucidate the manner that it is affected by the various process operational parameters. A blend of three different SS streams composted more efficiently in terms of temperature attained during the process than a single SS stream when the materials were co-treated with WCh acting as the bulking agent. WCh facilitated the accomplishment of a temperature over the sanitization limit and concurrently the establishment of a high decomposition rate over a long period. Further increasing the fraction of WCh in the composting mixture had an insignificant effect on the decomposition rate of organics in SS. The presence of grass in a mixture had a negative influence on the composting temperature that retained below the sanitization limits during the entire process. Aeration led to an increment in temperature but in all cases the enhancement remained at levels lower than 20°C throughout the trials. Concentration of heavy metals in all mixtures was sufficiently reduced with composting regardless of the SS type, mixing ratio, the use of a secondary organic substrate and aeration.

1. INTRODUCTION

The contemporary industrialization and urbanization have led to an enormous increase in the generated quantities of sewage sludge (SS), a by-product deriving from the operation of wastewater treatment plants (WWTP). SS entails significant challenges emerging from its costly management and disposal and the fact that its land application encompasses a high ecological risk emerging from a potential accumulation of toxic elements (Sreesai et al., 2013; Romero et al., 2024). Composting is the biological decomposition of an organic material under aerobic conditions through the enzymatic activity of several microorganisms and could be implemented for the treatment of SS prior to disposal mitigating any potential hazard for environment and human health (Temel, 2023). During the process, organic matter is converted into stabilized humic substances whereas any pathogens are eliminated (Muscarella et al., 2023). Legislation defines that a sludge sustained a composting stabilization under certain conditions can be safely disposed on land and implemented as a soil conditioner (Peltre et al., 2015). Nevertheless, certain characteristics of SS such as an unpleasant odour and a heavy metal content among others, are not always disappeared by the process restricting the broad utilization of compost (Smith et al., 2009).

Composting is considered one of the most acceptable and economically feasible technologies for recycling SS (Villaseñor et al., 2011). Nevertheless, due to the low-quality of SS as a composting substrate, co-composting with other types of organic materials is frequently employed for the improvement of its conversion (Ammari et al., 2012; Yousefi et al., 2013). Some physicochemical characteristics of SS may complicate even the co-composting process. For instance, SS contains a high moisture proportion and low porosity that would require the presence of a bulking agents during composting in order to absorb the moisture, enhance aeration and improve the quality of the final product (Yañez et al., 2009). Garden waste, which is commonly characterized by a loose structure, low water content and high C/N ratio is a typical substrate frequently applied to advance the composting of SS (Albrecht et al., 2010; El Fels et al., 2014).

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In the current study, five composting piles with two types of SS were prepared in an industrial environment to recycle the waste into valuable and usable compost. Wood chips (WCh) were the bulking agent that used to prevent compaction and enable oxygenation as well as to balance the moisture content and C/N ratio of the SS. The compost was characterized for its physicochemical properties after 110 days of processing. The objective of the full-scale trials was to: (i) investigate the impact of the SS type, the presence of a bulking agent and of a secondary substrate on the conversion of SS, (ii) determine the effect of aeration of composting pile on the maturity of the produced compost.

2. MATERIALS AND METHODS

2.1 Compostable substrates

Four organic materials, namely a SS waste, a blend of three SS streams of different origin (SSmix), WCh and grass, were selected for the composting trials. SS streams were obtained from four individual municipal WWTPs located in Moravian-Silesian Region (Czech Republic) with mean capacities at 3.000 tonne per month. In all cases, SS waste were received in a dewatered state. SSmix was prepared by mixing in equal proportions three of the SS streams. WCh was obtained from a commercial wood processing unit and were used as the bulking agent to prevent compaction of SS during composting. Grass was obtained from an urban greenery maintenance corporation, received in 1-2 cm long particles due to lawn mower operation and easily mixed with SS and WCh.

2.2 Composting mixtures

Five piles weighted approximately 10 tons were prepared for the composting trials. Each pile contained organic substrates mixed in different proportions. Two mixtures, M1 and M2, prepared by mixing SS and WCh in ratios 1:1 and 1:2, respectively. M3 was made by mixing SSmix and WCh in a ratio of 1:2. To examine the effect of aeration on composting, two mixtures, namely M4 and M5, were prepared so as to have the same composition containing SSmix, WCh and grass at a ratio 1:1:1. Mixtures composition is outlined in Table 1.

2.3 Industrial composting trials

Full-scale experiments were carried out in a composting plant in Moravian-Silesian Region, Czech Republic with capacity close to 2000 t/year. For this study, five piles (5 m × 5 m × 2 m) were prepared within a period of June to September and with the weather being remarkably stable during the entire period. Once the fresh dewatered SS was delivered by a WWTP to the composting plant, it was directly used for the trials.

Composting piles were regularly subjected to turning, which was carried out using a compost turner approximately once per week during the intensive microbiological degradation stage (hydrolytic phase) and on average once per two weeks during the maturity stage. This operation mode allowed the process to be more intensive. Composting trials were carried out for 110 days. Pile temperature was measured using a PT-100 type thermometer in three depths (30, 80 and 110 cm) from the pile surface. Sampling was also conducted at the same depths (30, 80 and 110 cm) after the turning procedure was finalized, gathering a total of 10 kg mixed in a single homogenous sample. To examine the influence of aeration on the composting process, fresh air was pumped into the pile of one mixture (M5) through a perforated plastic tube over the entire composting process. For the analysis of the results, the composting mixture consisted of SSmix and WCh at a ratio 1:2 (M3) was considered as the reference.

2.4 Analytical methods

A WTW 340i pH-meter (SenTix 410 sensor) was used for pH measurement. Total solids (TS) content was measured according to the EN 15934:2012 standard by drying the sample at 105°C until the variation of weight was < 2.0% wt in a KERN DLB 160 3A moisture analyser. VS content was determined by measuring the loss on ignition of the material at 550°C in O_2 atmosphere in a LECO TGA 701 thermogravimetric analyser. Carbon, hydrogen, nitrogen and sulfur concentrations were determined using a LECO Truspec CHN and S628 elemental analyser operated according to the ASTM D5373-16 standard. Concentration of heavy metals including phosphorus and potassium were determined by using a multielement XRF XEPOS (SPECTRO Analytical Instruments GmbH, Germany). To increase results accuracy, measurements of heavy metals in the parent SS and SSmix were triplicated. Escherichia coli and Salmonella spp. were determined in microbiology tests performed following the EN ISO 9308-2:2012 standard method. Samples were analysed in triplicates to accurately determine the moisture, organic matter content, C, H, N, S and heavy metals.

2.5 Ecotoxicological methods

Three ecotoxicological tests were conducted to assess the ecotoxicological properties of the mixtures, namely inhibition of luminescence of marine bacteria Vibrio fischeri, root growth inhibition of Sinapis alba seeds and cultivation experiments with *Hordeum vulgare L.* The tests with V. fischeri and S. alba were performed with aqueous leachates prepared from the composting mixtures in accordance to the EN 12457-4:2002 standard method. Luminescent bacteria tests were carried out with LUMStox luminometer (Hach-Lange GmbH, Germany) in accordance to ISO 11348-1:2007 standard in two parallel determinations and two repetitions with evaluation after 15 and 30 minutes. The inhibitory effect H_t was calculated according to the equation:

$$H_t = [(I_{ct}-I_t)/I_{ct}] \times 100 \qquad (1)$$

where I_{ct} is the corrected luminescence value and I_t is the luminescence intensity of the test sample after 15 min or 30 min exposure in relative units (ISO 11348-1:2007). Root growth inhibition tests with S. alba were conducted as a rapid preliminary screening prior to detailed testing during experiments with *H. vulgare*. The tests were performed in duplicates with 20 S. alba seeds and 5 mL of the leachate per treatment for 72 h. Mean root length was evaluated via ImageJ software. The root growth inhibition *I* was calculated according to the equation:

TABLE 1: Composition of blends used as substrates for composting trials.

Materials	Mixtures M1 (t)	M2	M3	M4	M5
Sewage sludge	5	3.3	-	-	-
Sewage sludge mixture	-	-	3.3	3.3	3.3
Grass		-	-	3.3	3.3
Wood chips	5	6.6	6.6	3.3	3.3

$$I = (L_c - L_t)/L_t \times 100 \qquad (2)$$

where L_t and L_c represent the mean root length in test and control determinations, respectively. Subsequent cultivation experiments with *H. vulgare L.*, variety Bojos involved growth analyses, evaluation of the physiological state of the plants and ecotoxicological tests. The experiments were carried out with stabilized compost samples that were not further diluted with soil. The tests were conducted in 4 replicates (4 pots per compost sample) with 16 seeds in each pot.

3. RESULTS AND DISCUSSION

3.1 Properties of materials and initial mixtures

Table 2 presents the properties of all individual materials used for the formulation of mixtures. TS and carbon concentration in SSmix were comparable to those in the biodegradable waste (grass), however were much higher than those in the similar material (SS). The deviation between the two SS indicates that a different behaviour might be expected during the composting process.

Table 3 presents the initial and final properties of the mixtures formulated for the 5 trials. The moisture content of a mixture is an important parameter influencing microbial activity during composting. Moisture not only supports the metabolic process but also creates a suitable environment for the transport of micromolecules formed from the decomposition of organic matter and dissolution of oxygen. For an efficient process, moisture of the primary mixture should be between 40-60% wt at process beginning (Oshins et al., 2022, Gajalakshmi and Abbasi, 2008). In our case, moisture concentration in the piles M2, M3, M4, M5 was within the optimal range indicating that the specific mixtures had a high potential for a sufficient composting performance. On the other hand, concentration of moisture in M1 was at 64.2% wt which exceeded the upper limit of the optimal range suggesting that M1 conversion during composting might be expected to be inadequate. Comparison of the initial and final TS content of each mixture in Table 3 indicates that composting led to a reduction of the TS concentration in all cases by 5-16% wt. The reduction is considered reasonable and could be expected whereas is attributed to a high temperature attained in piles during the process that causes evaporation and loss of water from the system.

The C/N ratio has been found to affect the composting of organic substrates (Komilis et al., 2011; Ponsá et al., 2010). Carbon and nitrogen are important for the develop-

TABLE 2: Elemental analysis, pH and TS of precursors used in large-scale trials.

Parameter	SS	SSmix*	WCh	Grass
pH	7.57	7.49	5.77	8.16
TS (%wt)	21.63	26.75	57.63	28.50
VS (%wt$_{TS}$)	54.42	58.66	92.42	70.48
Elemental analysis (%wt$_{TS}$)				
C	27.41	31.77	48.55	33.73
H	4.53	5.22	5.56	5.13
N	3.91	4.31	0.51	2.33
S	1.31	1.02	0.05	0.59
P	5.3	3.65	0.17	0.93
K	1.2	1.11	0.87	4.98
C/N (-)	8.18	8.60	111	16.89
Heavy metals (mg/kg)				
As	17.73 ±1.07	7.65±0.80	1.7	1.59
Cd	4.43±0.46	2.00±0.30	0.1	4.50
Cr	97±25.00	100.50±23.60	32.60	16.50
Cu	134±27.80	314±42.80	23.00	36.00
Hg	1.82±0.40	2.06±0.32	ND	ND
Ni	88.8±7.80	26±2.86	9.70	23.40
Pb	55.2±9.98	25.3±6.98	6.90	2.30
Zn	252±32.00	469±39.90	248	115

**Average values from three SS used to prepare the SSmix, ND=Not detected*

TABLE 3: Properties and heavy metal content of primary mixtures and composts.

Parameter		Limit value	M1	M2	M3	M4	M5
pH	Initial	-	6.92	6.44	6.62	7.50	8.01
	Final	-	5.95	6.30	5.82	6.09	6.30
TS (%wt)	Initial	-	50.70	47.06	45.23	51.11	58.32
	Final	-	35. 80	41.99	40.46	40.83.	41.67
VS (%wt_{TS})	Initial	-	77.51	73.31	91.28	85.00	83.07
	Final	-	36.64	51.01	58.34	27.70	31.70
Elemental analysis (%wt_{TS})							
C	Initial	-	39.14	41.62	45.27	42.30	38.01
	Final	-	19.58	25.14	31.38	14.59	15.50
H	Initial	-	5.39	4.93	6.31	4.15	3.32
	Final	-	3.01	3.58	4.02	2.06	2.05
N	Initial	-	2.72	1.95	2.04	2.80	1.72
	Final	-	1.09	1.13	1.66	1.02	1.16
S	Initial	-	0.63	0.42	0.33	0.56	0.38
	Final	-	0.25	0.22	0.28	0.22	0.28
C/N (-)	Initial	-	16.79	24.90	25.89	17.63	25.78
	Final	-	20.96	25.96	22.05	16.69	15.59
Heavy metals (mg/kg)							
As	Initial		7.33	5.59	3.18	5.10	1.33
	Final	20	2.03	1.90	2.28	1.17	4.44
Cd	Initial		0.60	0.57	1.24	2.00	2.00
	Final	2	0.56	0.36	1.61	1.60	1.44
Cr	Initial		90.50	27.80	23.60	95.90	50
	Final	100	30	15	14.50	95	37.90
Cu	Initial		64	51	53	228	190
	Final	100	39	34	50.20	152	72.10
Hg	Initial		0.63	0.53	0.35	0.64	0.70
	Final	1	0.17	0.11	0.20	0.45	0.35
Ni	Initial		61.50	15.70	10.90	20	13.80
	Final	50	16.40	13.20	10	15	10.90
Pb	Initial		40.70	45.80	12.10	31	29
	Final	100	7.60	6.40	27.20	30	28
Zn	Initial		213	195	320	340	320
	Final	300	163	157	300	300	277

ment of microorganisms since bacteria consume nitrogen for their cellular growth and the synthesis of protein whereas use carbon to cover their energy requirements. A microorganism contains 10-15 units of carbon and one unit of nitrogen by weight and hence it is expected to consume 25 times more carbon than nitrogen to cover its needs (Temel, 2023). As a consequence, the C/N ratio of a composting sludge will significantly decrease with process evolution. Given that SS is characterized by a low C/N ratio, mixing the waste with other organic materials e.g. WCh or grass would be a proper practice for increment of its potential for efficient composting. A C/N ratio between 25 and 35 has been found to be optimal for a competent aerobic degradation (Oshins et al., 2022). The initial C/N ratio of the mixtures ranged from 16.79 to 25.89. In particular, the C/N ratio in M2 (24.90), M3 (25.89) and M5 (25.78) were close to the range that ensure a competent aerobic process. In contrast, the C/N ratio in the mixture M1 and M4 was lower than 25 (16.79 and 17.63, respectively) which is the limit above which carbon is rapidly depleted with a concurrent high loss of nitrogen and extended ammonia formation.

The way that the C/N ratio modified by the composting process was not similar in all runs. The ratio in the composts M1 and M2 was higher than that in the initial mixtures because nitrogen loss was greater than the carbon reduction during the process. In contrast, in the composts M3, M4 and M5, the carbon concentration was lower and the nitrogen proportion higher than in the primary mixtures leading to an overall lowered C/N ratio (Raj and Antil, 2011). Although the variation of C/N ratio over the composting time is considered as a stability indicator for the entire process, the parameter could not be reliable factor due to the variability in the composition of the initial mixtures (Muktadirul Bari Chowdhury et al., 2013).

The pH of the starting mixtures is shown in Table 3. The pH affects the biochemical reactions occurring during composting and its stability depends on the activity of various microorganism colonies and the buffering capacity of the system. Theoretically, pH values of a mixture at process start-up that can support the conversion of the material fall within a range of 6.5-8.0 (Temel, 2023). As can be seen from Table 3, the pH of blends (6.4 - 8.0) was precisely within the optimal interval for an efficient aerobic conversion. Moreover, according to the literature (Aycan Dümenci et al., 2021; Singh et al., 2012; Vitinaqailevu and Rajashekhar Rao, 2019; Yılmaz et al., 2022), a pH value of final compost between 5.5-7.2 signifies a successful composting together with a sufficient organic matter conversion. The low pH in compost is mostly attributable to the bioconversion of organic matter and generation of organic acids under aerobic conditions (Gigliotti et al., 2012). In our case, all final compost materials exhibited a pH value between 5.82-6.30 indicating a remarkable conversion of the compostable organic matter contained in the blends.

3.2 Large-scale co-composting of sewage sludge

3.2.1 Composting dependence on sewage sludge type

The effect of SS type on the efficiency of composting was examined by comparing the temperature profiles at three pile depths provided by a mixture of a single SS with WCh (1:2) and those provided by a mixture of an SS blend (SSmix) with WCh at the same ratio. In all cases, the temperature attained at a depth of 30 cm from piles surface was higher than the temperatures at the depths of 80 and 130 cm (Figure 1).

For M3 and at 30 cm from pile surface, the temperature increased from 35°C on the process start up to 65°C after 20 days of process. In contrast, M2 reached a maximum temperature of 53°C on the 24th day, Figure 1. For the M3 mixture, the temperature remained at high level (60-65°C) and slightly dropped after the third turning on the 24th day but kept above 55°C for a total of sixteen days. A temperature between 45 and 55°C allows a material to attain a high biodegradation rate during composting (González et al., 2019). The bulking agent that was supplied in the piles likely assisted the mixtures M2 and M3 not only to attain a high temperature that could ensure sanitization but also to acquire a high decomposition rate for a long period. Interestingly, after a turning 55th day and until the 83rd day the temperature was stable at 43°C for M3 and 38°C for M2. In general, the mixture containing the SSmix performed much better than the mixture containing a single SS in all cases. The differences in the temperature profiles were significant with the SSmix reaching higher temperature than the single SS material. Even though most chemical and physical properties of the two mixtures were similar (Table 3), M3 contained a greater amount of VS (91.28% wt_{TS}) than M2 (73.31% wt_{TS}) which might be the main reason for the better performance for the specific mixture.

3.2.2 Effect of mixing ratio on composting process

The temperature in the composting piles M1 and M2 at depth 80 cm increased for three weeks (Figure 2), which is in agreement with previous studies investigated similar blends (Oviedo-Ocana et, al 2015). The temperature profiles indicate that the active composting phase started even by the first day (Cáceres et al., 2018). The maximum temperatures attained by the piles M1 (55°C) and M2 (54°C) were significantly close whereas appeared only for

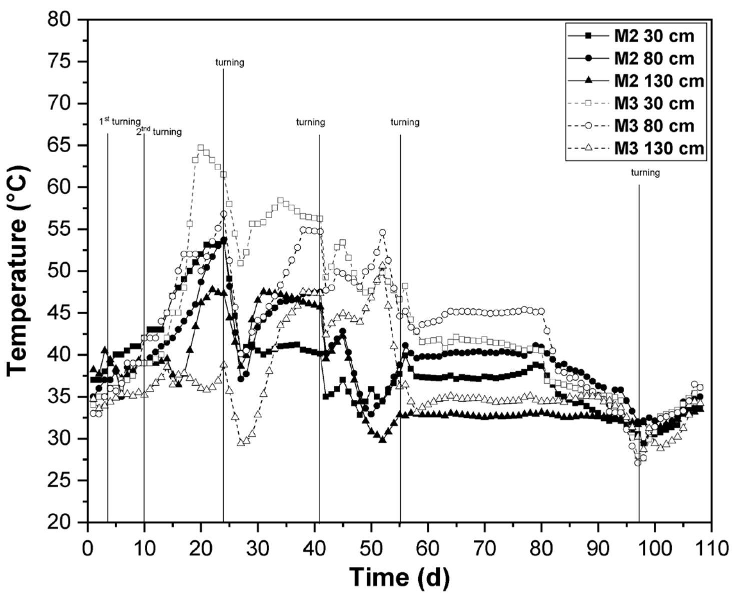

FIGURE 1: Temperature evolution at depths of 30, 80 and 110 cm from pile surface during composting of M2 (SS:WCh 1:2) and M3 (SSmix:WCh 1:2). Vertical lines indicate turning events.

a single day in the third week in both cases. This means that the high fraction of WCh had insignificant effect on the rate of decomposition of the organics in SS. The temperature in both piles dropped at lower levels after the third week. Theoretically, destruction of pathogens and sanitization of the materials in a mixture can be realized when the temperature in pile reaches values greater than 55°C and remains at those levels for at least three days (Oshins et al., 2022). It seems, therefore, that the piles M1 and M2 did not satisfy all the required temperature and time conditions that can ensure an efficient sanitization. On the other hand, according to the theory, thermophilic fungi that are responsible for the decomposition of lignin grow at 50°C and composting is competent when the temperature in the thermophilic phase of the process (>50°C) varies between 52-60°C (Miller, 1992; Tuomela et al., 2000). In our case, the temperatures reached in the piles impose that a sufficient composting could be achieved regardless of the WCh proportion in a mixture.

In spite of the moderate temperatures, microbiological evaluation, shown below, suggested that both final composts were properly sanitized by the process even though WCh did not significantly influence the maximum temperature in the thermophilic phase. In general, WCh contain a low fraction of biodegradable carbon and hence the quantity of the bulking material in the mixture would not be expected to provide any advancement in the temperatures gained during composting (Figure 2) (Tang et al., 2024). Nevertheless, when a low proportion of WCh participated in the mixture, the temperature was slightly higher from the 30th day and throughout the rest of composting process. In more detail, within the cooling phase, the mean temperature difference between the two mixtures was about 5-10°C. In any case, the difference was small enough that cannot be regarded as a consequence of the physicochemical characteristics of the materials or of any other parameters involved in composting. Therefore, it would be reasonable to assume that easily degradable organic compounds like carbohydrates, fats and amino acids that were presumably contained in SS degraded early in composting process, whilst cellulose, hemicellulose and lignin that were enclosed in WCh partially degraded and transformed slowly affecting almost negligibly the process evolution (Haug, 1993).

3.2.3 Effect of a secondary substrate and aeration process on temperature profiles

Figure 3 illustrates temperature profiles for the mixtures M3 and M4 consisted of SS:WCh (1:1) and SS:WCh:Grass (1:1:1). Temperature in M3 rapidly increased from 34 to 54°C within 20 days, which is a typical rise for the early days of composting. The thermophilic phase quickly emerged due to the generation of heat released by microbial degradation of any readily available and highly decomposable organic substances included in the mixture.

Temperature also increased after each turning of M3, but remained below the temperature securing sanitization (55°C). Nevertheless, the temperature remained over 40°C from the 15th to 80th day. Mixture M4 that contained amount of grass exhibited lower temperatures over the entire process. The profile was characterized by an increment from 40 to 45°C within 26 days and then a continuous drop that was not reversed by any turning event. In this case, green waste that replaced part of SS and WCh contained a significant proportion of lignocellulose that likely affected the activity of bacteria leading to a shorter thermophilic and a greater cooling phase. A similar effect of the lignocellulosic fraction of biomass on the composting process has been reported and attributed to the fact that a green waste is slightly more resilient than SS to microbial degradation due to the complicate structure of lignin and cellulose (Bustamante et al., 2012; Zhang 2023). Moreover, the fact that the mixture M3 contained a greater amount of VS (91.28% wt_{TS}) than M4 (85.00% wt_{TS}) (Table 3) may

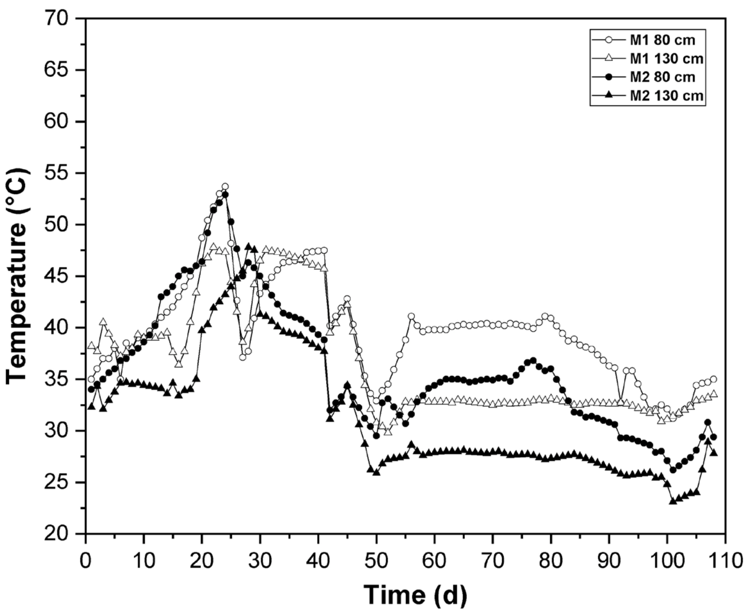

FIGURE 2: Temperature evolution at two depths (80 and 130 cm) during composting of M1 (SS:WCh 1:1) and M2 (SS:WCh 1:2).

also be reason for the superior performance of the mixture without green waste. It should be noted here that none of the mixtures reached a sufficient sanitization temperature level despite the observed advancement of the temperature after every turning. The beneficial impact of the turning procedure on the temperature was also reported by Şevik (2018).

In addition to turning, the aeration of the composting sludge was examined by preparing a pile with the mixture M5, which had the same composition with the mixture M4, and install a direct aeration system in the pile. Temperature in M5 reached a maximum at 54°C within approximately 15 days from the beginning of the trial (Figure 3). Thereafter, the temperature dropped, however, the thermophilic phase (>50°C) (de Bertoldi et al., 1983) started from the 10th and lasted for 18 days. Temperature declined at a constant rate during the mesophilic phase (40-50°C), however, from the 60th day the reduction became acute and temperature dropped from 39 to 18°C within 20 days. From this point onwards, composting process was deemed completed with the temperature maintaining close to the ambient. The fall-off in temperature denoted that most of the organic compounds were consumed during the thermophilic and mesophilic phases and that few easily degradable compounds remained to decompose within the cooling phase (Meng et al., 2017). On the other hand, the non-aeriated mixture M4 showed the highest temperature of 46°C on the 26th day and immediately entered in the cooling phase where remained until the end of the trial. The superior performance of M5 over M4 caused by the aeration, however, the temperature differences were limited, less than 20°C, over the entire process.

3.2.4 Effect of composting on mixtures contamination

Contamination of substrates with E. coli and Salmonella spp. was assessed with a determination of microbial populations before and after the composting process (Table 4). E. coli bacteria are often used as an indicative factor of the overall quality of soil and water. Colony forming units (CFU) per gram of substrate were determined in five replicates per sample with a limit at 50 CFU/g. Salmonella spp. was not detected in any case before and after the composting process. According to legislation (Decree No 474/2000) the determination of E. coli should be performed in five replicates and from the values obtained in them none should exceed 5000 CFU/g and four of them should be lower than 1000 CFU/g.

The values for E. coli before composting were counted at $5*10^4$ (CFU/g) for the mixtures M1, M2 and M3 and at $5*10^3$ (CFU/g) for the mixtures M4 and M5. However, the values considerably decreased to less than 50 CFU/g in all mixtures at the end of the process. The decrease was presumably a consequence of the temperature and the unfavourable conditions for microbial survival established during the thermophilic phase of the trials. According to de Bertoldi et al. (1983) the ideal temperature in thermophilic phase ranges between 40-65°C whereas temperatures above 55°C are necessary to destroy any coliforms. Here, in spite of the fact that the temperature was not at a critical level for an efficient coliform destruction, the concentration of E. coli reduced by at least three orders of magnitude by the composting process in all cases.

3.2.5 Heavy metals fate

Co-composting of SS with other materials can reduce the concentration of heavy metals due to dissolution, adjust the moisture of waste mixture to optimal values and provide a high-quality compost in terms of stability, maturity and agronomic and environmental parameters (Proietti et al., 2016). In particular, co-composting of sewage sludge and WCh can successfully lead to low heavy metal concentrations in the compost compared to the pre-composting

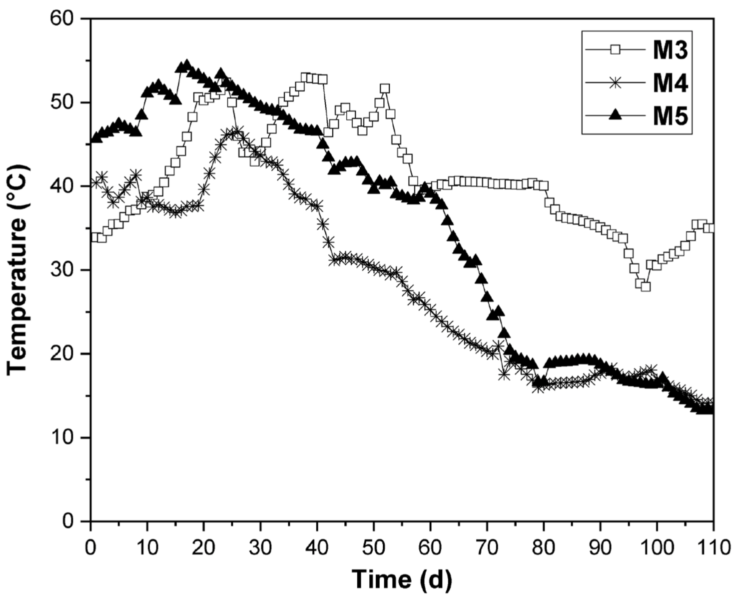

FIGURE 3: Average temperature in three depths (30, 80 and 130 cm) from pile surface during composting of mixtures M3 (SSmix:WCh 1:2), M4 (SSmix:WCh:G 1:1:1 without aeration) and M5 (SSmix:WCh:G 1:1:1 with aeration).

TABLE 4: Contamination of substrates with *E. coli* and *Salmonella spp.* before and after composting.

Mixture		*Escherichia coli*	*Salmonella spp.*
		(CFU/g)	
M1	initial	$5*10^4$	NEGATIVE
	final	<50	NEGATIVE
M2	initial	$5*10^4$	NEGATIVE
	final	<50	NEGATIVE
M3	initial	$5*10^4$	NEGATIVE
	final	<50	NEGATIVE
M4	initial	$5*10^3$	NEGATIVE
	final	<50	NEGATIVE
M5	initial	$5*10^3$	NEGATIVE
	final	<50	NEGATIVE

mixture (Mohee and Soobhany, 2014). This is consistent with the results in Table 3, where the fraction of heavy metals fell with the co-composting of SS and SSmix with WCh and/or grass. The reduction was likely attributable to a dissolution of each individual metal element into the aqueous phase existing in the composting system. It is interesting to note that a decrease in the concentration of all heavy metals in SS and SSmix was primarily occurred along with the preparation of mixtures of the wastes with WCh and grass (Table 2 and 3) and was presumably ascribed to the fact that the lignocellulosic materials contained a low or negligible amount of metal substances. Hence, the composting process itself appears to assist in attaining a low concentration of heavy metals in the solid effluent of a composter.

From Table 3, the concentration of each of the heavy metals was highly variable in the various mixtures. A similar wide variation was described in earlier studies (Ščančar et al., 2000). Table 3 presents the maximum concentrations of the most important heavy metals in a compost that would allow it to be considered appropriate for agricultural use according to the legislation. Zn proportion exceeded the limit value in M3, M4 and M5 before composting (320-340 mg kg^{-1}). This is not surprizing, however, since earlier studies (Gondek et al., 2018; Wang et al., 2005) reported that Zn concentration in municipal SS is usually the highest among the monitored heavy metals and SS frequently contains Zn at proportions approaching a 1600 mg kg^{-1}. Concentrations dropped below boundaries with the treatment. In addition to Zn, Ni proportion in the mixture M1 (61.5 mg kg^{-1}) exceeded the limit of 50 mg kg^{-1}. However, the concentration of the element reduced 3.75-fold with the composting process gaining a value that was far smaller than the upper boundary (Table 3). The proportion of other elements such as Cr and Pb were inside the limits before composting and became even smaller in the final composts. Cd is one of the most mobile heavy metals in the soil and restrictive factor for any land application of SS (Zheng et al., 2020). In all mixtures, Cd ranged in the initial sludge between 0.57-2 mg kg^{-1} whereas varied between 0.36-1.61 mg kg^{-1} in the final compost.

3.3 Ecotoxicity of composts

3.3.1 Vibrio fischeri

A very low luminescence inhibition was observed for the composts M2 and M3 whereas a very low luminescence stimulation (negative values of inhibition) was found for M1, M4 and M5 (Table 5). However, the values of luminescence inhibition oscillate around zero and therefore it can be concluded that none toxic effect existed. The results are consistent with the findings of Roig et al. (2012), who assessed the ecotoxicity of sewage sludge depending on the type of sludge treatment. Therein, composting was identified as the most favourable treatment for attaining a low ecotoxicity.

3.3.2 Sinapis alba

Inhibition of root growth did not exceed a 30% in any of the M1-M5 composts (Table 5). In addition, a root growth stimulation (negative inhibition value) was observed for mixtures M4 and M5. The potential toxicity of composts may be due to the high nitrogen content (Sowinsky et al., 2023), with the source of nitrogen being sewage sludge. However, it should be stressed that the S. alba root growth inhibition values around 20% are still reported as non-toxic.

3.3.3 Hordeum vulgare L.

Cultivation experiments with H. vulgare L. were carried out for all the generated composts. Germination of the seeds was not inhibited and no statistically significant differences were found compared to the control substrate. In addition to the germination, the nitrogen index (NBI) and the chlorophyll content in fully developed leaves were determined for 4 weeks old emerged plants. Both parameters were estimated by using a Dualex (FORCE-A, Germany), whereas the chlorophyll content was assessed using the leaf transmittance ratio at far-red and near-infrared wavelengths, while the accumulation of flavonols was measured using the differential ratio of chlorophyll fluorescence. The Normalized Biomass Index (NBI) was defined as the concentration ratio of chlorophyll to UV-absorbing phenolics, primarily flavonols. The results showed a higher sensitivity of the plants to the substrates used than in the case of germination. Composts M2 and M3 did not show a significant influence on nitrogen index and chlorophyll content compared to the control. However, in the case of the mixture M1, a conclusive decrease of both parameters was observed. Finally, the dry weight of the above-ground biomass was determined and no statistically significant difference was observed for substrates M1-M3 compared to a certified control substrate.

4. CONCLUSIONS

Two different SS streams were subjected to co-composting at industrial scale with a bulking agent (WCh) and grass to determine the effect of process parameters and SS type on the conversion potential and sanitization of the waste material. The mixture that contained a blend of three different SS (SSmix) reached a higher temperature than the mixture contained only a single SS. The presence of

TABLE 5: Ecotoxicity tests with *V. fischeri* and *S. alba*.

Ecotoxicity test	Compost				
	M1	M2	M3	M4	M5
	(%)				
V. fischeri luminescence inhibition (15 min)	-0.07±2.66	2.21±2.58	1.80±5.62	-0.25 ±3.15	-1,32±4.18
V. fischeri luminescence inhibition (30 min)	-0.76±1.99	2.55±1.15	0.84±1.02	-1.12±3.45	-2.58±4.92
S. alba root growth inhibition	23	24	7	-14	-8

a bulking agent assisted the materials not only to attain a high temperature that could ensure sanitization but also to acquire a high decomposition rate for a long period. An increased fraction of WCh, however, had an insignificant effect on the rate of decomposition of the organics in SS. Likewise, the presence of grass in a mixture negatively affected the extent of the thermophilic phase and more promoted the cooling phase presumably due to the complicate lignocellulosic structure of grass that is resilient to microbial degradation. Aeration promoted the composting performance of a mixture although the increment of temperature attained was less than 20°C over the entire process. In any case, aeration appears to be an essential parameter and under conditions a sufficient solution in accomplishing the required temperature conditions for comprehensive sanitization of an SS mixture. Co-composting of SS or SS-mix with WCh and grass successfully reduced the concentration of all heavy metals in the solid fraction likely due to a dissolution in the aqueous phase. All composts provided positive results in ecotoxicological evaluation and cultivation experiments where none significant inhibitory effect on the growth of spring barley was observed.

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ULVAN FROM *ULVA NEMATOIDEA* AS A NOVEL ACTIVE SURFACE MATERIAL FOR TRIBOELECTRIC NANOGENERATORS

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ABSTRACT

Marine algae represent an underutilized biomass resource. Biopolymers can be extracted from different types of algae. Alginates and carrageenans are among the most common biopolymers extracted from brown and red algae. They find applications in the food and biomaterials industries, among others. However, other available marine algae are not commercially exploited. For instance, green algae from the Ulvaceae family remain largely unexploited and have no industrial applications. In particular, Ulva species can serve as a promising source for the extraction of a biopolymer known as ulvan. This work reports the development of triboelectric nanogenerators (TENGs) for energy harvesting applications using ulvan extracted from the green algae *Ulva nematoidea*. Ulvan was extracted via an alkaline method. The extracted ulvan was dissolved in water, poured into petri dishes, and dried to form thin films. TENGs were prepared using Ulvan-Kapton® and Ulvan-Polytetrafluoroethylene (PTFE) triboelectric pairs. The Ulvan-Kapton® TENG showed a maximum voltage of 2.12 V and a short-circuit current of 1.6 μA while the Ulvan-PTFE TENG showed a maximum voltage of 43.60 V and a short-circuit current of 5.6 μA. This performance is similar to the performance of other TENGs fabricated from commercial biopolymers. This suggest that ulvan extracted from *Ulva nematoidea* has potential applications as active surface of TENGs for the development of sustainable energy harvesting devices. This work shows that bio-based materials from green algae can serve as a potential alternative for renewable energy generation. Further research will allow to enhance mechanical properties, electrical performance, and durability of ulvan-based TENGs to improve their practical applicability.

1. INTRODUCTION

Biomass derived from macroalgae is recognized as a source of valuable biopolymers with applications in the biomedical, nutraceutical, cosmeceutical, and pharmaceutical industries, among others. Algae are classified into brown (Phaeophyceae), red (Rhodophyta), and green (Chlorophyta) algae. The difference between them is the presence of different types of chlorophyll and pigments (Osório et al., 2020). Marine algae contain high amounts of biopolymers in the form of carbohydrates (~60%) and proteins (10–47%) (Kraan, 2013). Biomass from the oceans have become an attractive alternative for the extraction of biopolymers. For instance, brown and red algae have been used to extract commercially important biopolymers such as alginate, carrageenan, and agar, among others.

Green algae belonging to genus Ulva (family: *Ulvaceae*) are mainly used as food and remain unexploited for large scale industries. Most research have been focused on their applications as food and pharmaceutical supplements, due to their antioxidant/radical scavenger and anti-inflammatory activity (Le et al., 2019; Matloub et al., 2018). However, Ulva sp. can be a source of biopolymers such as ulvan. Ulvan is a sulfated polysaccharide mainly composed of rhamnose, xylose and uronic acids. The structure of this polysaccharide is dependent to the species, season of collection and processing (Rodríguez-Iglesias et al., 2024). It has been reported that ulvan comprise from 9% to 36% of the dry weight biomass of the cells of Ulva sp. (Kidgell et al., 2019). Ulvan yield ranges from 8% to 29% of the dry weight of algae (Lahaye & Robic, 2007).

The presence of Ulva has been associated to ecological disasters such as green tides and eutrophication. These processes could result in hypoxic conditions (due to algal respiration) and severely compromise the survival of aquatic vertebrates (Wilhelm, 2009). It has been proposed that sustainable harvesting of Ulva sp. could be an alternative to mitigate their negative effects (Torres & De-la-Torre, 2022). Previous work (Torres & De-la-Torre, 2022) showed that green algae can be used for the development for sus-

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tainable energy materials. The biopolymer ulvan, extracted from this family of algae, has been studied for energy-related applications (Gonzales et al., 2024). They can be used as solid and gel electrolytes, and even as electrodes in energy storage applications (Torres et al., 2024).

The use of low-power devices, such as sensors, Wi-Fi modules, and Internet of the things (IoT) devices, have seen significant growth in recent years. These devices require a reliable energy source to operate effectively. Batteries have long been the main energy source for these devices. However, a significant drawback with batteries is the environmental pollution that occurs when they are improperly disposed of, as they can contaminate large water bodies and soil (Melchor-Martínez et al., 2021; Mrozik et al., 2021). Recently, more sustainable solutions have emerged to address this issue, including piezoelectric nanogenerators (Bairagi et al., 2020), triboelectric nanogenerators (Zhu et al., 2024) and bio-based batteries (Liu et al., 2021).

Triboelectric nanogenerators (TENGs) take advantage of the contact electrification phenomenon to harvest mechanical energy. In this process, two surfaces from two different materials are put in contact and separated alternatively. Contact electrification occur when these two surfaces touch, and electrical charges flow from one surface to the other. The active surfaces of most TENGs are made from dielectric synthetic polymers such as polytetrafluoroethylene (PTFE) and polyethylene terephthalate (PET), due to their ability to give and receive electrons by contact electrification. TENGs from synthetic polymers could pose potential ecological problems due to the generation of electronic waste. By contrast, a TENG made from biopolymers could become an alternative energy source of small electronic devices with a limited impact on the environment.

Synthetic polymers have been extensively used as materials for triboelectric surfaces. In recent years, most new TENGs have been constructed using synthetic polymers. For instance, Tung et al. (2025) developed a TENG utilizing a polymer composite film composed of polyhexamethylene guanidine hydrochloride (PHMG), polyvinyl alcohol (PVA), and glutaraldehyde (GA) as a crosslinking agent. The device demonstrated a peak-to-peak open-circuit voltage of 664.5 V and a short-circuit current of 116.8 μA. Zhao et al. (2025) employed a poly(vinylidene fluoride-hexafluoropropylene) / molybdenum disulfide (MoS2) as a triboelectric layer in a TENG; the fabricated device exhibited an open-circuit voltage of 208 V, a short-circuit current of 31 μA, and a peak power density of 1.42 W/m² at 50 MΩ. Yang et al. (2024) developed a TENG using a two-dimensional graphitic carbon nitride incorporated poly(vinylidene fluoride) (g-C3N4/PVDF) as a tribonegative layer and nylon-11 nanofibers as a positive layer. This device achieved an open-circuit voltage of 339 V, a short-circuit current of 14.5 μA, and a power density of 0.94 W/m². You et al. used polyurethanes (PU) on polyethylene terephthalate fabric (PET-F) to create a film for a TENG device. This configuration achieved a peak-to-peak voltage of 352 V and a maximum power density of 18 mW/cm². These examples highlight the widespread use of synthetic polymers in TENG development and their promising electrical performance. However, despite their ability to generate high voltage outputs, the non-biodegradability of synthetic polymers raises environmental concerns, potentially limiting their long-term sustainability.

These environmental concerns have triggered the development of energy harvesting devices using a variety of biopolymers. Small generators such as TENGs have been developed using different biopolymers (Table 1). Chen et al. (2021) developed a chitosan/montmorillonite/lignin (CML) composite film as a tribopositive layer in a TENG. The device featured an open circuit voltage of 262 V and an instantaneous output power of 429 mW/m^2. The authors demonstrated that CML-TENG maintains better performance at high temperatures compared to chitosan-only based devices. Petchnui et al. (2024) developed a TENG using a nanocomposite based on natural rubber (NRL) and chitin nanofibers (ChNF) obtained from shrimp shells biowaste. The TENG with a triboelectric layer composed exclusively of NRL exhibited low output voltages, around 2.56 ± 0.8 V. In contrast, incorporating 0.2% by weight of ChNF significantly enhanced performance, achieving an open-circuit voltage of 106.04 V. Cellulose has also been used as a triboelectric material for TENG. Zhang et al. (2021) fabricated an eco-friendly TENG using cellulose. The device had an open-circuit voltage of 29 V and a short-circuit current of 0.6 μA. Somseemee et al. (2024) developed a TENG using Natural rubber filled with cellulose nanocrystals. The device achieved an open circuit voltage of 80.3 V a short-circuit current of 7.4 μA. Other biopolymer used in TENGs is starch. Ansori et al. (2023) developed a cassava starch/PVA-TiO_2 nanocomposite film as a triboelectric film in a TENG. The device achieved an open circuit voltage of 180 V and a short-circuit current of 13.7 μA.

Most studies on biopolymers for TENG applications have focused on red and brown algae, particularly alginate and carrageenan. While these materials have demonstrated potential for energy harvesting, they exhibit limitations such as complex processing, and low triboelectric performance. For example, Pang et al. (2018) fabricated a TENG using calcium alginate, but its low output voltage (33 V) and short-circuit current (150 nA) suggest that improvements in material properties are needed. Similarly, Li et al. (2023) incorporated silver nanowires into an alginate-based TENG to enhance conductivity, but this increases production costs and environmental impact. The resulting device reached an output voltage of 53 V and a power of 4 μW. Kang et al. (2022) developed a TENG using carrageenan, a biopolymer extracted from red algae. The developed device had a peak-to-peak output voltage of 30 V and an output power of 0.15 mW·m^{-2}. These studies remark the need for alternative biopolymers that balance sustainability, mechanical integrity, and triboelectric efficiency.

Unlike alginate and carrageenan, which have found commercial applications, ulvan remains largely unexploited despite its potential as a sustainable biopolymer. Previous research has primarily investigated it for its bioactive properties, but its role in energy harvesting remains almost completely unexplored. Few studies have investigated the use of green algae as a material for energy devices. For example, Noman et al. used ground Ulva lactuca algae as a triboelectric layer in a TENG. This device achieved a maximum output voltage of 875 V, an output current of 52 μA, and a power density of 272.72 μW/cm² (Noman et al., 2024). However, biopolymers extracted from green algae, such as ulvan, remain largely understudied. Due to its sulfated polysaccharide structure, ulvan may possess favorable triboelectric properties. However, research on ulvan-based TENGs is currently lacking, a gap this work seeks to address.

In this paper, the extraction of Ulvan from *Ulva nematoidea* and its application in the development of a mechanical energy harvesting device (TENG) is reported. Such device can convert low frequency mechanical vibrations into electricity for small portable low-power electronic devices. The extracted ulvan was used to fabricate films to be used as triboelectric surface in this TENG. The films were characterized by Scanning Electron Microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), Differential Scanning Calorimetry (DSC) and tensile tests. TENG's electrical output confirmed the suitability of ulvan as a potential material for energy harvesting devices.

1.1 Highlights

- Ulvan was extracted from *Ulva nematoidea* using an alkaline method.
- Ulvan films were used as a tribopositive triboelectric surface for energy harvesting devices.
- Ulvan-based TENGs achieved an open-circuit voltage of 43.60 V and a short-circuit current of 5.6 μA.

2. MATERIALS AND METHODS

2.1 Materials

Fresh green algae *Ulva nematoidea* were collected in Lima, Peru. All the reagents used in the present study were analytical grade and purchased from Sigma-Aldrich and Himedia Laboratories LLC.

2.2 Extraction of Ulvan

Ulvan was extracted using an alkaline extraction method (Peasura et al., 2015; Tabarsa et al., 2018). The extraction was conducted in two major steps, pre-treatment and extraction (Figure 1).

For the pre-treatment step, green algae were washed with abundant distilled water to remove impurities. After that, they were dried in an oven at 40°C for 72 h. Then, the dried samples were powdered and stored at 4°C. The obtained biomass was treated with ethanol to remove soluble materials, using 34 g of dry algae biomass per 500 mL of ethanol under constant stirring for 24 h. The supernatants were discarded, and the biomass was dried in an oven at 40°C for 48 h.

After the pre-treatment, ulvan was extracted using 0.1 M NaOH (pH 13) under constant stirring at 80°C for 6 h, with a biomass to solvent ratio of 1:35 (w/v). The supernatants were separated by centrifugation (10,000 rpm for 10 min) and discarded. Then the ulvan solution was concentrated by evaporation until 50% of its initial volume and precipitated by slowly adding the concentrated in 2 volumes of cold ethanol. The ulvan precipitate was recovered by centrifugation (10,000 rpm for 10 min). After that, ulvan was repeatedly washed with ethanol to remove impurities or any reagents, and then, dried in an oven at 40°C for 72 h. The dried Ulvan was stored in a desiccator for further use.

2.3 Preparation of triboelectric surfaces and Triboelectric Nanogenerators (TENG)

Ulvan-based films were used as triboelectric surfaces. To prepare ulvan films, a solution of ulvan (1% w/v) in distilled water was stirred at 60°C for 3 h. Then, 10% of glycerol was added to the ulvan solution. Finally, the solutions were poured onto Petri dishes and dried in an oven at 40°C for 48 h.

TABLE 1: Open circuit voltage and short circuit current of different TENGs.

Biopolymer-based TENGs				
Biopolymer	**Open Circuit Voltage (V)**	**Short-circuit current (μA)**	**Power Output**	**Ref.**
Chitosan	262	-	429 mW/m²	(Chen et al., 2024)
Chitin	106.04	-	-	(Petchnui et al., 2024)
Alginate	33	0.15	9.5 μW	(Pang et al., 2018)
Alginate	53	-	4 μW	(Li et al., 2023)
κ-Carrageenan/Agar	-	-	0.15 mW/m²	(Kang et al., 2022)
Bacterial Cellulose	29	0.6	3 μW	(Zhang et al., 2021)
Cellulose	80.3	7.4	1.32 W/m²@ 9 MΩ.	(Somseemee et al.,2024)
Starch	180	13.7	-	(Ansori et al., 2023)
Grounded Ulva algae/ PET	875	52	272.72 μW/cm²	(Noman et al., 2024)
Synthetic Polymer-based TENGs				
Polymer	**Open Circuit Voltage (V)**	**Short-circuit current (μA)**	**Power Output**	**Ref.**
PHMG-GA-PVA	664.5	116.8	-	(Tung et al., 2025)
PVDF-HFP	208	31	1.42 W/m²	(Zhao et al., 2025)
A g-C3N4/PVDF	339	14.5	0.94W/m²	(Yang et al., 2024)
PU-CD4/PET-F	352	-	18 mW/cm²	(You et al., 2024)

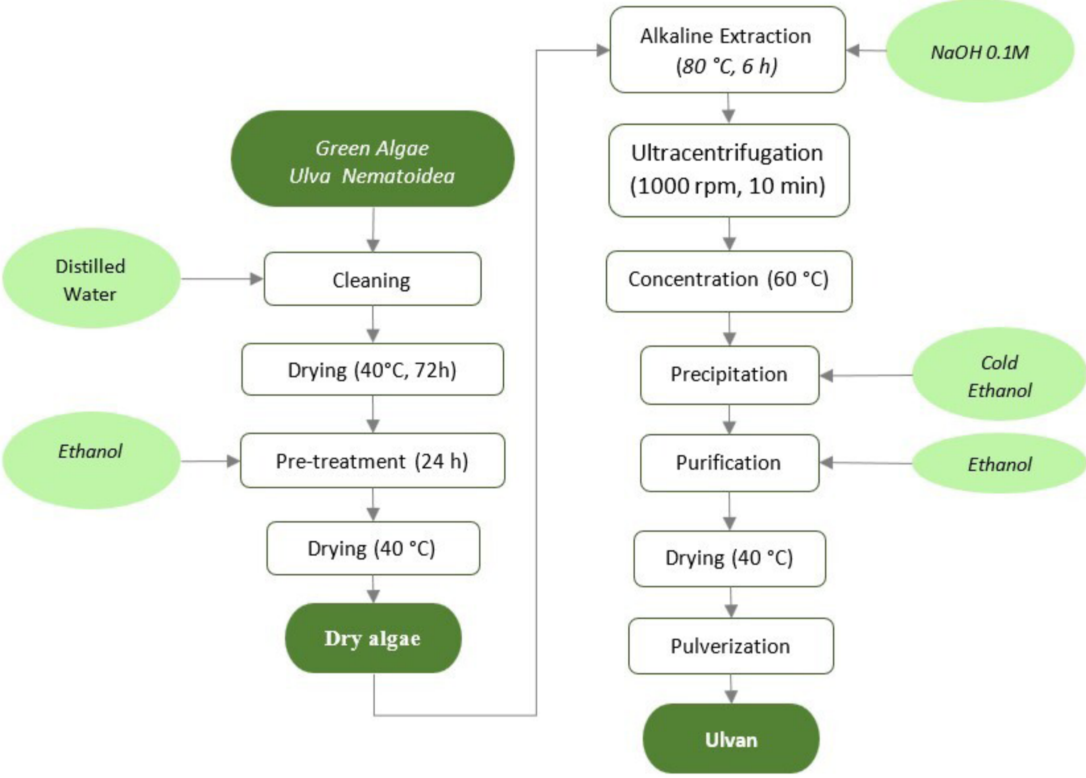

FIGURE 1: Scheme of the alkaline process used in this study to extract ulvan.

TENGs were fabricated using two different triboelectric surfaces, in contact-separation mode. Two types of TENGs were prepared: ulvan-Kapton® and ulvan-PTFE TENGs. Kaptop® feature a tribopositive behavior, i.e. it is prone to give away electrons. On the other hand, PTFE has a tribonegative behavior because is an electron-accepting material. The interaction between the selected triboelectric surfaces and the ulvan film was used to evaluate the triboelectric behavior of the ulvan based active surface. Ethylene-vinyl acetate (EVA) sleeves were used as support for the two surfaces of each TENG. Copper films were used as electrodes (current collectors). These copper films were glued to the polymeric films and attached to the EVA sleeves using a methacrylate adhesive. The Ulvan, Kapton®, PTFE and copper films were cut in 4cm x 4cm squares and mounted in the EVA sleeve as shown in Figure 2a.

2.4 Characterization Techniques

2.4.1 Scanning electron microscopy (SEM)

A Thermo Fisher (Massachusetts, USA) Axia ChemiSEM Scanning Electron Microscopy (SEM) equipped with an energy-dispersive X-ray spectroscopy detector (EDX), was used to observe the morphology of the ulvan films. Tests were carried out in low vacuum with a voltage of 20 kV and a working distance of ~10 mm.

2.4.2 Fourier transform infrared (FTIR) spectroscopy

Ulvan films were characterized using FTIR spectroscopy to identify the functional groups. FTIR spectra was obtained using a Spectrum Two Perking Elmer (Connecticut, USA) spectroscope, equipped with a universal attenuated total reflectance (ATR) accessory. Film samples were cut in 1cm x 1cm squares and inserted in the ATR. Samples were recorded in the 500–4000 cm^{-1} spectral range with a resolution of 4 cm^{-1} and 64 scans. Perkin Elmer Spectrum 10 software was used to acquire the spectra.

2.4.3 Differential Scanning Calorimetry (DSC)

DSC tests were performed using a Perkin Elmer (Connecticut, USA) DSC4000 calorimeter. DSC was calibrated using Indium and Zinc as reference materials. Nitrogen was used as purging gas with a flow rate of 20 mL/min. 10 mg of samples were loaded in aluminum pans and heated from 0°C to 180°C, at a heating rate of 10°C/min. An empty aluminum pan was used as reference. Perkin Elmer Pyris software was used to record the thermograms.

2.4.4 Electric performance of the TENGs

The electrical performance of the TENGs was evaluated using a DPO2022 Tektronix (Oregon, USA) oscilloscope. The instrument was calibrated with a self-calibration function. A 10 MΩ probe was used to perform the measurements. TENGs were manually operated through alternating motions(contact-separation). The generated voltage signal was measured directly with the oscilloscope. Additionally, the rectified voltage was also measured. Voltage data were recorded over a 10-second interval. The short-circuit current was measured using an SR570 Stanford current preamplifier paired with a circuit containing an equivalent resistance. The resistance was selected based on the research of Abid et al. (Abid et al., 2024). According to their study, the optimal load resistance is determined from the

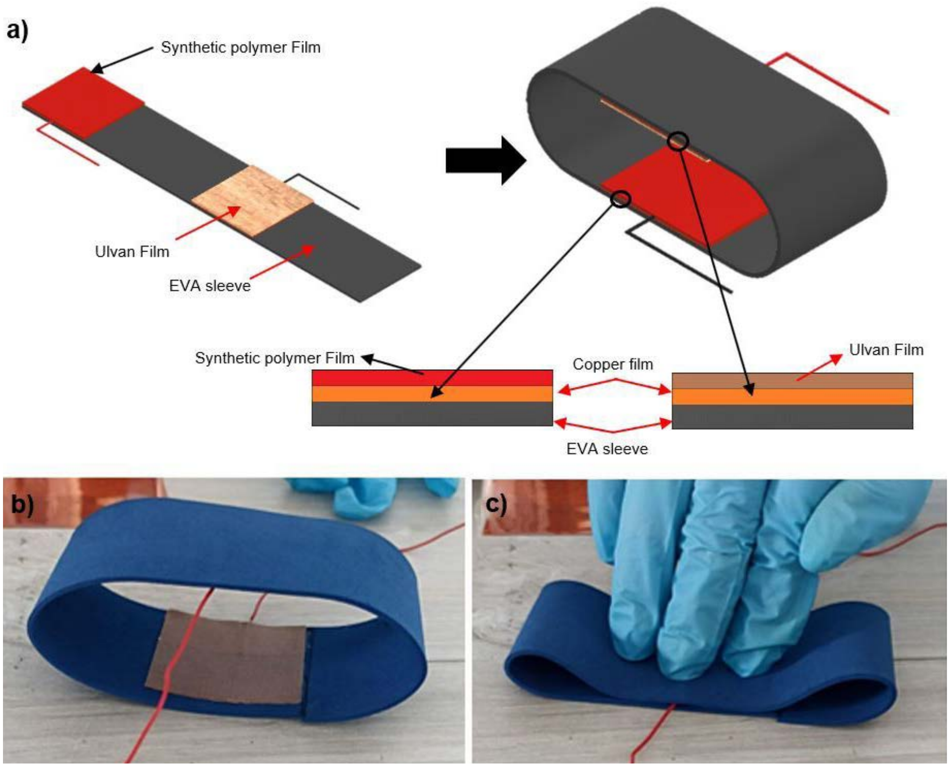

FIGURE 2: (a) Schematic illustration of a TENG fabricated using an EVA sleeve as support. The two polymeric films (ulvan – PTFE / ulvan-Kapton®) are attached to copper films connected by a copper wire. These assemblies are mounted on the EVA sleeves and arranged in opposite positions to form a closed loop. An electrical current is generated when the films are alternately brought into contact (c) and separated (b).

TENG's current curve, which is obtained by testing the device across a range of resistive loads. In order to investigate differences in the maximum value of open circuit voltage and short-circuit current in each TENG (ulvan+PTEF and ulvan+Kaptop®), the results were analyzed using a one-way analysis of variance (ANOVA) followed by Bonferroni post-hoc analysis at a family significance level of 0.05.

2.4.5 Mechanical properties

Mechanical properties were evaluated using a bench top tensile testing machine ESM Mark-10 (New York, USA), equipped with a 10N (±0.5%) load-cell. Films were cut into 1 cm x 7 cm rectangular strips with an average thickness of 72 µm, measured using a micrometer Mitutoyo (Kanagawa, Japan) MDC-25PX, and tested at a grip separation speed of 10 mm/min. 5 repetitions were carried out. Young's modulus, ultimate tensile strength (UTS) and maximum strain were recorded.

3. RESULTS AND DISCUSSION

Ulvan is a sulfated polysaccharide characterized by a backbone composed of α- and β-(1,4)-linked monosaccharides, including rhamnose, xylose, glucuronic acid, and iduronic acid, which form repeating disaccharide units. The primary repeating disaccharides in ulvan are aldo-biuronic acids. These disaccharides are formed from uronic acids (either glucuronic or iduronic acid) that are linked through a (1,4) glycosidic bond to rhamnose units. Disaccharides of the aldo-biuronic type are referred to as ulvanobiuronic acids and are classified into two categories: A and B. The A type, ulvanobiuronic acid 3-sulfate (A3s), consists of β-D-glucuronic acid linked to α-L-rhamnose 3-sulfate via a (1,4) bond, while the B type, ulvanobiuronic acid 3-sulfate (B3s), comprises α-L-iduronic acid linked to α-L-rhamnose 3-sulfate through a (1,4) bond. Additionally, disaccharides of the aldobiose type are present in smaller proportions. These aldobiose-type disaccharides are termed U-type ulvanobiosas. There are two variants of ulvanobiosas: U3s, which consists of β-D-xylose linked to α-L-rhamnose 3-sulfate via a (1,4) bond, and U2's,3s, which consists of β-D-xylose 2-sulfate linked to α-L-rhamnose 3-sulfate through a (1,4) bond (Gonzales et al., 2024; Lahaye & Robic, 2007). Ulvan does not exhibit an ordered conformation. Molecular models indicate that the A3s and B3s disaccharide units adopt stable helix-like conformations when hydrogen bond interactions occur between the oxygen atoms of the sulfate groups on rhamnose and the hydroxyl or carboxylic groups located on the preceding uronic acids (Lahaye & Robic, 2007). Ulvan can be extracted from a variety of green algae species. They have a polysaccharide content in the range of 8.2% to 27.5% (Kidgell et al., 2019). In this study, ulvan was extracted from *Ulva Nematoidea* algae using an alkaline method. The yield of the extracted ulvan was 15.12% based on the dry algae-to-ulvan ratio.

The extracted ulvan was used to prepare films by casting. Pure ulvan films were too rigid and difficult to handle. Glycerol had to be used as a plasticizer. SEM was used to carry out the morphological characterization of the ulvan films. Figure 3a and 3b show a non-smooth texture. White regions (arrows) were identified by EDX as sodium, calcium and potassium oxide. Figure 3c and 3d show the EDX spectra of points 1 and 2 (Figure 3a), respectively. The EDX spectrum of point 1 has no carbon signal as only sodium and sulfur oxide are present. The EDX spectrum of point 2 shows carbon, oxygen, sodium, and sulfur signals. The SEM and EDX results show that the films prepared had a rough, non-homogeneous morphology with the presence of salt inclusions. The sodium signal can be attributed to the extraction process that uses sodium hydroxide. The sulfur signal confirms that ulvan is a sulfated polysaccharide.

The presence of sulfate groups together with other functional groups were studied with FTIR. In order to assess the effect of glycerol, FTIR tests were carried out on pure ulvan and ulvan-glycerol films (Figure 4). The spectra are similar, and the same peaks are observed. The broad absorption band between 3000-3500 cm^{-1}, with a peak around 3355 cm^{-1}, correspond to the stretching vibration of the O-H group (Peasura et al., 2015). The peaks at 2938 cm^{-1} are characteristic of stretching vibrations of C-H bonds in methyl groups. The peaks observed at 1599 cm^{-1} and 1415 cm^{-1} are related to the asymmetric and symmetric stretching vibration of the carboxyl group (-COO-) (Guidara et al., 2020). The peak found at 1030 cm^{-1} is associated with C-O stretching vibrations in C-O-C bonds found in rhamnose and glucuronic units (Chen et al., 2021). The peak near 1225 cm^{-1} is related to the stretching vibration of the ester-sulfate group (S=O) which is characteristic of sulfated polysaccharides (Gonzales et al., 2024).

Figure 5 shows a representative DSC thermogram of an ulvan-glycerol film. An endothermic stepwise change is observed, consistent with a glass transition process. The glass transition temperature (Tg) was measured at the inflection point of the curve. The glass transition temperature had a value of 135.69°C, and the related specific heat capacity (ΔCp) was 0.625 J/g°C. These values agree with previous reports. Morelli and Chiellini (Morelli & Chiellini, 2010) measured the Tg of ulvan extracted from U. armoricana. They reported a Tg of 132.60°C, associated to a ΔCp of 0.496 a J/g°C. Ghazy et al. (Ghazy et al., 2024) carried out a Density Functional Theory (DFT) simulation and calculated a Tg of 165.88°C for ulvan extracted from Ulva lactuca.

The ulvan-glycerol films were then used as triboelectric surfaces. Two different dielectric materials are usually needed to fabricate a contact-separation mode TENG. In this case we have evaluated the electric performance of an ulvan-PTFE and ulvan-Kapton® TENGs. The triboelectric behavior of the ulvan based active surface was assessed evaluating the interaction between these selected triboelectric surfaces and the ulvan film. Kapton® and PTFE were chosen to determine if ulvan featured a tribopositive or tribonegative behavior. The best electric performance of

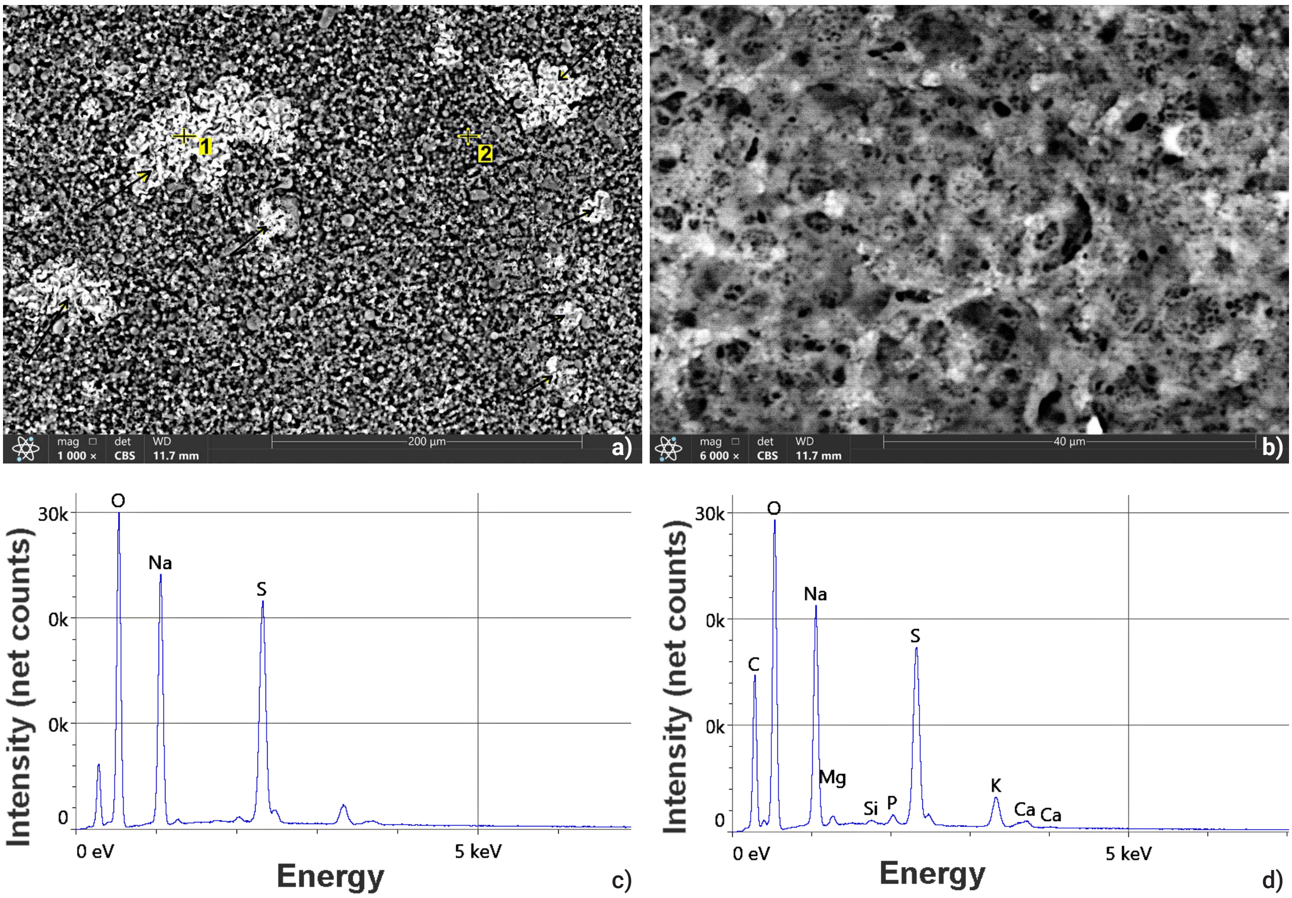

FIGURE 3: SEM micrographs of ulvan-based films at 1000x (a) and 6000x (b). EDX spectra of the point 1 (c) and 2 (d).

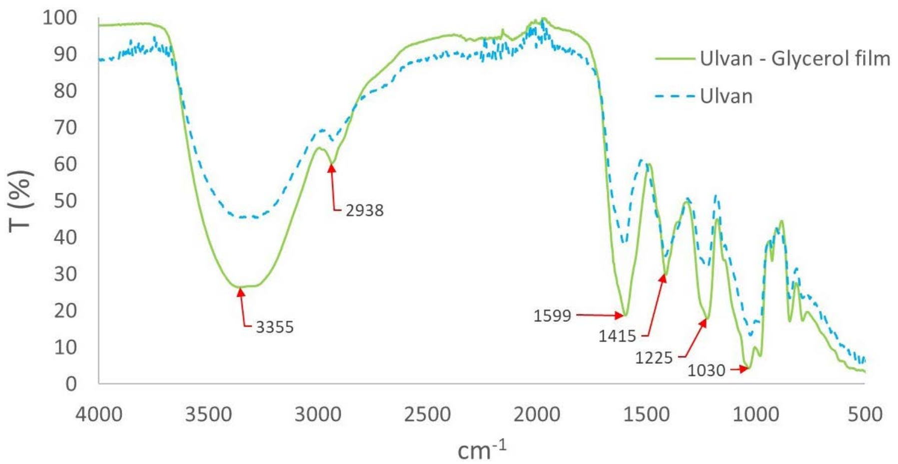

FIGURE 4: Representative FTIR spectra of a pure ulvan and a glycerol-ulvan film.

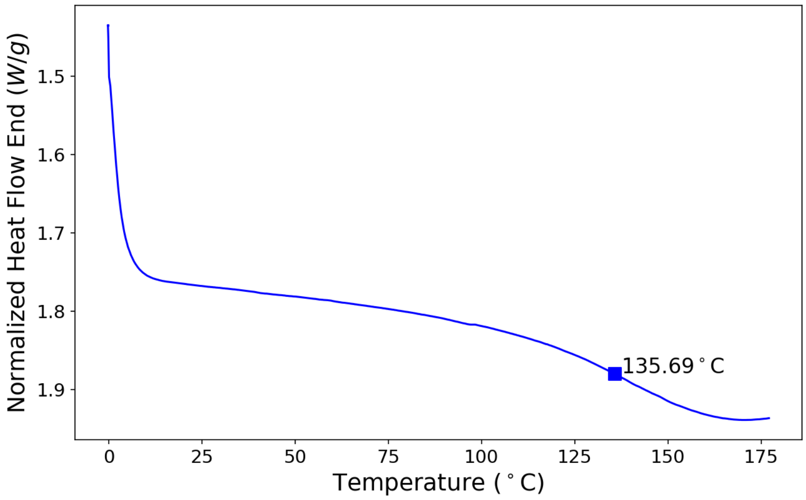

FIGURE 5: Representative DSC thermogram of an ulvan-glycerol film.

a TENG is achieved when a tribopositive material is working with a tribonegative material.

Figure 2 shows how these TENGs work. The triboelectric surfaces are manually pressed together, applying a force of approximately 10 N. Upon contact, free electrons transfer from one material to the other, generating surface charges. When the materials are separated, some electrons return to their original position producing a voltage differential. The voltage signal of the TENGs was analyzed using an oscilloscope. Figure 6 shows representatives signals of ulvan-PTFE and ulvan-Kapton® TENGs. When measuring the open circuit voltage of TENGs, the voltage signal features two peaks (Figure 6a, 6c): one positive and the other negative, corresponding to the contact (positive) and separated (negative) states. This type of mechanical vibration is directly transformed into alternative current (AC). A diode bridge was used to rectify the voltage signal to have a direct current (DC) circuit. The rectified signal (Figure 6b, 6d) shows only positive peaks, with the highest values occurring in the contact state.

The short circuit current was assessed using a preamplifier paired with a field resistance ranging from 3.1 MΩ to 20 MΩ. Figure 7 shows the short circuit current measured across the different resistance used. The maximum short circuit current measured for the ulvan-Kapton® samples was 1.6 μA. The maximum short circuit current measured for the Ulvan-PTFE samples was 5.6 μA. Table 2 lists the maximum open circuit voltage and short circuit current achieved. These results show that the selection of polymeric materials is of utmost importance. Ulvan-Kapton® TENGs featured the lowest voltage value. Ulvan-Kapton® samples featured a maximum voltage of 2.12 V and a short-circuit current of 1.6 μA at 9.3 MΩ and a power density of 0.0281 W/m^2. On the other hand, ulvan-PTFE TENGs showed maximum voltage of 43.60 V, a short-circuit current of 5.6 μA at 9.3 MΩ and a power density of 0.176 W/m^2.

The difference between the fabricated TENGs could be explained in terms of the dielectric properties of the materials. In fact, Kapton® is prone to give away electrons (tribopositive), whereas PTFE is prone to receive electrons (tribonegative). The fact that ulvan-PTFE TENGs feature higher voltages compared to ulvan-Kapton® TENGs, suggests that ulvan is also prone to give electrons (tribopos-

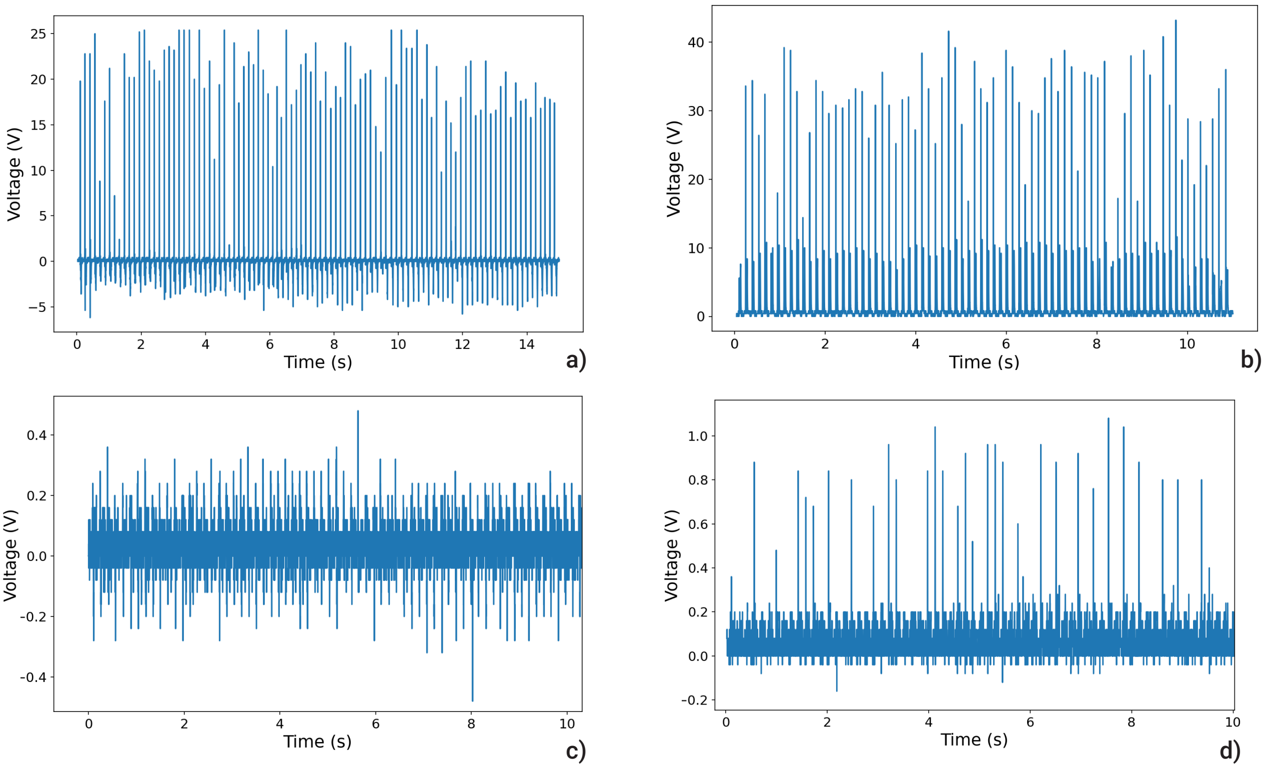

FIGURE 6: Voltage signals from Ulvan-PTFE TENGs in an open circuit mode (a) and a rectified circuit (b). Voltage signals from Ulvan-Kapton® TENGs in an open circuit mode (c) and a rectified circuit (d).

itive). A material that donates electrons works best when paired with a material that accepts electrons to maximize the voltage. For synthetic polymers, there is established knowledge regarding which material combination works better. There are published lists that ranks materials according to their tendency to gain or lose electrons (Khandelwal et al., 2021; Zou et al., 2020a). The currently existing triboelectric series are primarily qualitative, ordered by the polarity of charge production (Zou et al., 2020b). However, this type of information remains scarce for biopolymers.

The voltage and current values that characterize the output performance of both synthetic polymer-based and biopolymer-based TENGs are shown in Table1. Biopolymer-based TENGs exhibit a voltage range of 29 to 262 V and a short-circuit current of 0.15 to 13.7 μA. In contrast, synthetic polymer-based TENGs achieve a voltage output ranging from 208 to 664.5 V and a current output of 14 to 116.8 μA. Ulvan-based TENGs perform similarly to the algae-based TENGs shown in Table 1; however, their performance remains significantly lower than that of synthetic

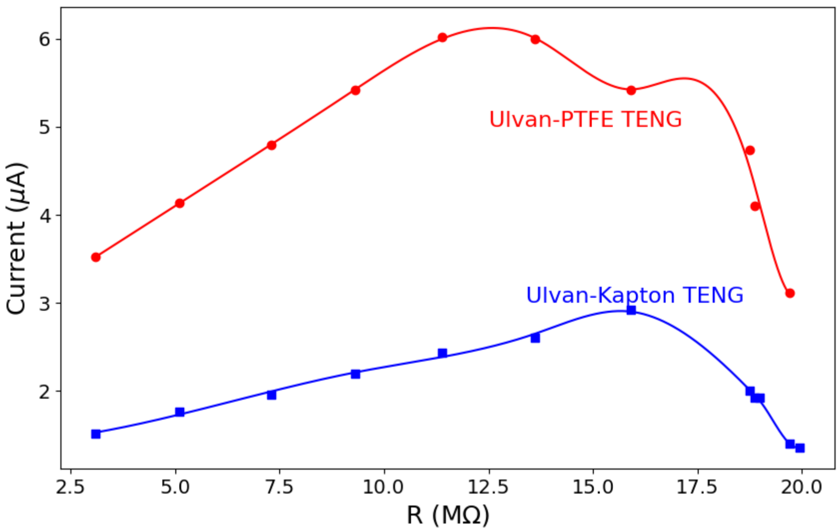

FIGURE 7: Representative curves of the short circuit current values measured across a range of resistive loads (3.1 - 20 MΩ).

TABLE 2: Open circuit voltage and short circuit current of the TENGs prepared in this study (maximum rectified values).

Triboelectric Layers	Open Circuit Voltage (V)	Short-circuit Current (μA) @ 9.3 MΩ	Power Density (W/m^2)
Ulvan + PTFE	43.60 ± 1.395 [a]	5.6 ± 0.264 [a]	0.176 ± 0.059 [a]
Ulvan + Kapton	1.92 ± 0.171 [b]	1.60 ± .0643 [b]	0.0281 ± 0.0016 [b]

[a, b] *Values are mean ± SD. Letters indicate significantly different groups (Student-Newman-Keuls)*

polymers. This output disparity can be attributed to the intrinsic properties of each polymer type, one of which is the dielectric constant. Synthetic polymers typically possess a higher dielectric constant and lower dielectric loss compared to biopolymers. The dielectric constant is indicative of a material's capacity to store energy; a higher dielectric constant allows for greater energy storage within the material (Urtecho et al., 2024). While synthetic polymers currently outperform biopolymers in TENG applications, sustainable energy harvesting remains a priority. Innovations in material engineering, including chemical modification, surface modification, and the development of high-performance nanocomposites, could lead to significant enhancements in the performance of biopolymers as triboelectric materials, thereby narrowing the performance gap with synthetic polymers.

Additionally, tensile tests were used to assess the flexibility and resistance of the ulvan films to be used in flexible TENGs. A representative strain-stress curve is observed in Figure 8. The test revealed that ulvan-glycerol films exhibit a max strain of 23.67%, with an ultimate tensile strength of 1.85 MPa. The elastic region was observed up to a deformation level of 1.7% strain with a yield stress of 0.28 MPa and a Young's modulus of 0.247 MPa. These films showed low tensile strength, high deformation, and poor shape recovery, with most of the deformation being plastic. This behavior may be associated with the conformation of the ulvan chains and their interactions with glycerol, which was used as a plasticizer. Plasticizers reduce intermolecular forces along polymer chains, enhancing flexibility and chain mobility (Vieira et al., 2011).

The results showed that it is possible to successfully develop a TENG that can generate energy for low power devices using a material obtained from a green alga. Ulvan-based films behaved as a tribopositive material as was prone to give away electrons. Future work will be focused on improving the ulvan triboelectric behavior by preparing composite and nanocomposite films with high dielectric constant. This strategy has been used with other biopolymers and should be explored for ulvan.

4. FUTURE PERSPECTIVES

TENGs present a promising solution for harvesting mechanical energy, addressing the critical demand for renewable and sustainable energy sources. Biopolymer-based TENGs represent a viable pathway for eco-friendly energy harvesting. Although they encounter challenges related to energy generation efficiency and durability when compared to synthetic polymers, ongoing advancements in materials science and engineering hold the potential to improve the properties of biopolymer-based TENGs. It is essential to explore new strategies aimed at enhancing the characteristics of the biopolymers employed as triboelectric layers to develop high-efficiency biodegradable TENGs. Some effective strategies for improving the triboelectric layers include chemical functionalization, surface engineering, and the development of nanocomposites.

Scaling up production represents a significant challenge for biopolymer-based TENGs. To date, only laboratory prototypes have been developed, and transitioning this technology to large-scale production remains a considerable challenge. This process involves several obsta-

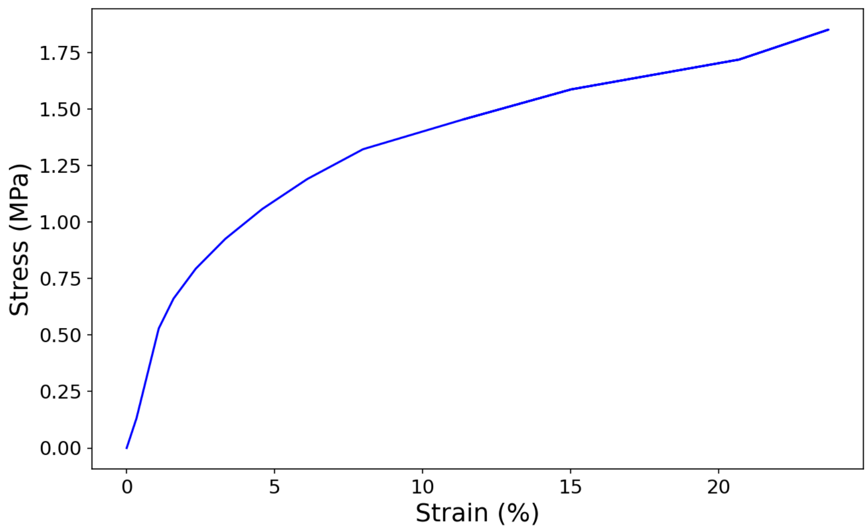

FIGURE 8: Representative Stress-Strain curve of an ulvan film.

cles, including the large-scale extraction of biopolymers, the development of efficient manufacturing techniques for triboelectric surfaces, and the establishment of a scalable production process for biopolymer-based TENGs.

Another significant challenge for future research is evaluating the environmental impact of biopolymer-based TENGs. Utilizing natural materials, such as algae and other biodegradable substances, for biopolymer development presents a sustainable alternative; however, large-scale implementation may lead to unforeseen environmental consequences. A comprehensive life-cycle assessment (LCA) is essential to ascertain the impact of this technology should a large-scale industry emerge from these advancements. Furthermore, the establishment of environmental policies and regulatory frameworks is crucial to ensure the safe, reliable, and scalable integration of these devices as replacements for traditional batteries in Internet of Things (IoT) sensor networks and other applications that require sustainability.

Governments and international organizations could establish funding programs and tax incentives to encourage the development of biopolymer-based energy harvesting devices, such as ulvan-based TENGs. This support can accelerate innovation and facilitate the commercialization of sustainable energy solutions. For instance, the European Commission has proposed actions to unlock the potential of algae in the EU, which could indirectly support ulvan-based technologies. Policies should encourage the incorporation of ulvan-based TENGs into circular economy frameworks. This includes supporting recycling programs and the development of biodegradable electronics to minimize environmental impact. To transition from laboratory research to large-scale production, policies should provide grants and infrastructure support for pilot projects. This assistance can help address challenges related to mass production, cost reduction, and industrial application of ulvan-based TENGs. The ULVA FARM project, funded by the European Maritime and Fisheries Fund, exemplifies how targeted support can lead to successful large-scale cultivation of Ulva species, which could be pivotal for ulvan extraction.

One example of a policy framework related to natural materials is the legislation governing algae. The European Union has established various laws and policies concerning the collection, cultivation, processing, and marketing of algae. This legislation pertains to products intended for human consumption, animal feed, pharmaceuticals, cosmetics, fertilizers, raw materials for the chemical industry, bioremediation, and algae-based fuels (Leinemann & Mabilia, 2019). However, algae possess significant untapped potential for broader applications. Developing new policies and regulatory frameworks could facilitate the expansion of algae-based biopolymers and other sustainable materials, while also promoting the use of natural resources in environmentally friendly technologies.

In summary, although biopolymer-based TENGs encounter several challenges before they can be considered viable alternatives, they possess significant potential. With further research aimed at overcoming these obstacles, this technology could facilitate a transition toward sustainable energy harvesting.

5. CONCLUSIONS

The increasing demand for sustainable energy solutions has driven the exploration of bio-based materials for energy harvesting applications. One significant challenge in this field is the environmental impact of synthetic polymers commonly used in TENGs. This study addressed this issue by investigating ulvan, a biopolymer extracted from *Ulva nematoidea*, as a novel active surface material for TENGs. The research focused on the extraction and processing of ulvan to develop films that were integrated into TENG devices for energy harvesting.

The results demonstrate that ulvan can function as a tribopositive material, particularly when paired with tribonegative materials like PTFE. The ulvan-PTFE TENG exhibited a maximum open-circuit voltage of 43.60 V and a short-circuit current of 5.6 μA, comparable to other biopolymer-based TENGs. In contrast, Ulvan-Kapton® TENG showed a low output compared to the TENGs found in the literature. While the performance remains lower than synthetic polymer-based TENGs, it presents a viable alternative for environmentally friendly energy solutions. Additionally, material characterization techniques, including SEM, FTIR, DSC, and mechanical testing, confirmed the structural integrity, thermal behavior, and flexibility of ulvan films. SEM and FTIR results demonstrated the presence of sulfate groups on ulvan. DSC shown ulvan-based films feature a glass transition temperature of 135.69°C.

The benefits of this research extend beyond energy harvesting applications. Utilizing ulvan from *Ulva nematoidea* provides a sustainable approach to mitigating green tides and eutrophication by promoting the use of these algae. Moreover, the development of ulvan-based TENGs aligns with global sustainability goals. Future research should focus on enhancing the mechanical and electrical properties of ulvan films through chemical modifications, doping, or optimization of the triboelectric layer structure.

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COMPARATIVE ECONOMIC AND ENVIRONMENTAL PERFORMANCE ASSESSMENT OF BIOMASS GASIFICATION PATHWAY FOR GREEN H_2 PRODUCTION

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ABSTRACT

The transition to low-carbon energy sources is essential to limit the temperature rise due to global warming. Biomass is a carbon-neutral source of energy that could aid in the energy transition and decarbonisation goals. This work compares the economic and environmental performances of small-scale (100 kg/h output) green hydrogen [biomass gasification (BG) and water electrolysis (WE)] pathways, using commercial-scale (20 tph output) conventional hydrogen [steam methane reforming (SMR)] as reference. The source of the electricity supply for BG system operations is a major determinant of the life-cycle emissions. It could be sourced externally (grid) or generated in-situ through an auxiliary gasification system. Capture of CO_2 from the syngas stream is feasible through a vacuum pressure swing adsorption (VPSA) unit. The assessment reveals negative specific carbon emissions (up to -19 kg CO_2/kg H_2) when electricity is generated in-situ and carbon capture is carried out in BG systems. The hydrogen costs from the BG pathway are in the range of INR 307-352/kg ($3.71-4.27/kg), competitive with water electrolysis using solar power. The study also suggests the introduction of emission reduction incentive (ERI) as a combined economic-environmental performance indicator. Among the green pathways, BG-H_2 with in-situ electricity generation and carbon capture has the best combined performance, requiring an incentive of $127/tonne CO_2. While commercial-scale SMR has a low hydrogen production cost due to economies of scale, the increasing natural gas prices could make the BG-H_2 pathway lucrative in the future.

1. INTRODUCTION

The increase in greenhouse gas (GHG) emissions over the years have driven the transition from fossil fuels to renewable energy. To achieve climate targets, a 20-50% reduction in fossil fuels is required by 2050, with hydrogen and its derivates playing a big role in the decarbonisation of industries and transportation. About 10-20% of the global final energy consumption is expected to be supplied from low-carbon hydrogen. Hydrogen is an important commodity used in oil refining, steel reduction, ammonia and methanol production, and transportation (BP p.l.c, 2023). It could also be used as an energy carrier to store renewable energy (Narayana Sarma et al., 2024).

The global hydrogen production amounted to 97 million tonnes (MT) in 2023, with low-carbon hydrogen production accounting for only about 1.03%. This comprises mostly of fossil-hydrogen with carbon capture, utilisation, and storage (CCUS) and water electrolysis through renewable energy. Around 15 fossil-hydrogen facilities are equipped with CCUS globally, with a cumulative hydrogen production of 0.6 MT. The electrolyser capacity reached 1.4 GW in 2023, with another 6.5 GW in advanced stages of planning and execution. Green hydrogen is expected to reach 49 MT by 2030, driven mostly by the announced electrolysis projects (International Energy Agency & Clean Energy Ministerial Hydrogen Initiative, 2024). Hydrogen could also be produced from solid biomass through thermochemical, biochemical or electrochemical conversion. Thermochemical processes like gasification could achieve high yields of hydrogen, lower production costs, and high technology readiness levels (TRL). Biomass is a carbon-neutral fuel, which could result in low life-cycle emissions. Carbon capture during hydrogen production could also lead to net CO_2 removal from the atmosphere (International Energy Agency & Clean Energy Ministerial Hydrogen Initiative, 2024).

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The green hydrogen and ammonia policy in India envisages production of 5 MT of hydrogen by 2030 (Ministry of Power Notifies Green Hydrogen/ Green Ammonia Policy, 2022). The abundance of agricultural residues makes the biomass-hydrogen pathway a lucrative option (Technology Information & ICAR - Indian Agricultural Research Institute, 2018). The declining local and industrial demands, uneconomical in-situ management techniques, and meagre returns often prompt the farmers to resort to open field burning, resulting in the loss of agricultural residues (Bhattacharyya et al., 2021). Therefore, diverting these residues for hydrogen production could enhance their value and provide an additional local income source, thereby also reducing air emissions by preventing their open burning and integrating circular economy principles. On the other hand, the increasing renewable power in the Indian electricity system makes water electrolysis a forerunner in fulfilling the green hydrogen demands and the policy objectives (Narayana Sarma et al., 2024). The selection of the appropriate pathways for hydrogen production is influenced by the economic and environmental performance metrics. Hence, the comparison of these pathways on important indicators such as production costs and life-cycle GHG emissions must be investigated.

The performance evaluation of hydrogen pathways is well represented in literature (Arcos & Santos, 2023; Arfan et al., 2023; Ganesh et al., 2024; Garcia G. & Oliva H., 2023; Helf et al., 2022; Lepage et al., 2021; Longden et al., 2022; Materazzi et al., 2023; Narayana Sarma et al., 2024; Salkuyeh et al., 2018). (Longden et al., 2022) estimated that achieving high carbon capture rates from fossil-hydrogen through CCUS requires high CO_2 avoidance costs (> $80/tonne), increasing the hydrogen costs. Water electrolysis could achieve hydrogen costs competitive with steam methane reforming (SMR) at low tariffs. (Salkuyeh et al., 2018) estimated that the direct emissions from biomass-hydrogen pathway could be negative even without carbon capture. Large-scale (18.9 tph output capacity) biomass gasification (BG) systems could compete with natural gas-based hydrogen at biomass costs of $100/tonne, natural gas costs of $5/GJ, or a carbon price of $115/tonne. (Ji & Wang, 2021) reported hydrogen costs of $1.77-2.77/kg from BG, which is much lower compared to water electrolysis from renewable energy ($5.27-5.78/kg). The GWP of renewable electrolysis is 50-70% lower than SMR with carbon capture. (Narayana Sarma et al., 2024) estimated green hydrogen costs of about INR 400/kg ($4.84/kg) through alkaline and PEM electrolysis. (Materazzi et al., 2023) compared the environmental emissions of biomass and waste gasification systems with those of natural gas-to-hydrogen and water electrolysis (WE). They reported that the gasification pathway with carbon capture could achieve negative emissions. (Ganesh et al., 2024) assessed the economics of distributed-scale BG systems and reported that the cost of bio-hydrogen is competitive with that of green hydrogen from electrolysis. The BG pathway could achieve economic parity with fossil-hydrogen pathways for biomass costs of about $30/tonne.

From the brief review of literature, it is clear that SMR is the most inexpensive pathway, as it uses mature technology. BG and WE pathways are comparable in terms of cost, but the BG pathway could achieve much lower emissions compared to WE. It must, however, be noted that most studies in literature have focused on large-scale systems. The unorganised residue supply chain still remains challenging, leading to uncertainties in the biomass availability for conversion. These uncertainties could be minimised through distributed-scale systems focusing on smaller radii of procurement, thereby reducing the costs of transporting the bulky residues (Ganesh et al., 2024; Rosburg et al., 2016). Hence, a performance evaluation of small-scale BG systems and its comparison with other green hydrogen pathways is essential. While the individual economic and environmental performances of various hydrogen pathways are abundantly covered in literature, joint performance assessments and comparison of the distributed-scale green hydrogen pathways are seldom reported.

Considering the above gaps in literature, this study evaluates the costs and emissions associated with hydrogen production from small-scale fixed-bed downdraft gasification systems. Four scenarios are considered in the assessment – BG with purchased electricity (BG1 - with and without carbon capture) and BG with an auxiliary gasification system for in-situ electricity generation (BG2 - with and without carbon capture). The individual economic and environmental performances of these scenarios are compared with the SMR and WE pathways for each kg of hydrogen produced. The Emissions Reduction Incentive (ERI) is introduced as a combined performance assessment indicator and is measured in terms of $/kg of CO_2 emissions saved. The detailed methodology is discussed in the next section.

1.1 Highlights

- Comparison of three hydrogen pathways based on cost and global warming potential;
- Emission reduction incentive introduced as a combined indicator for comparison;
- Negative GHG emissions are feasible with carbon capture in the BG pathway;
- BG pathway displays better combined performance compared to water electrolysis;
- Rise in natural gas prices could make the BG pathway lucrative in the future.

2. METHODOLOGY

2.1 BG pathway description

Biomass is utilised in a fixed-bed downdraft system with dual air entry capabilities, ensuring sufficient oxygen supply for tar cracking within the reactor. This ensures relatively tar-free gas, circumventing the issues faced by other fixed-bed systems. The ash contained in the biomass is extracted from the bottom of the reactor through a screw conveyor. The rate of extraction could be defined based on the biomass properties, making this system suitable for a wide range of biomass. The gas is further cleaned in a series of wet scrubbers to obtain very low impurities, suitable for multiple downstream applications. The system uses pre-processed biomass with a particle size of 50 mm (Ganesh et al., 2024). It could be operated in two modes, depending on the oxidizer used – air gasification for the production of producer gas and oxy-steam gasification for the production of hydrogen-rich syngas (Kumar, 2016). The hydrogen from the gas could be separated through a Vacuum Pressure Swing Adsorption (VPSA) unit with a hydrogen recovery of 70% and purity of over 99.97% (Singh et al., 2023). The off gas from the VPSA unit could be used for the complete fulfilment of the heat demand and partial fulfilment of the electricity demand of the system (Ganesh et al., 2024). Two scenarios are evaluated for the source of the balance electricity demand. The BG1 scenario considers purchased electricity from the grid. The BG2 scenario considers an auxiliary air gasification (AG) unit along with the main gasification unit. This unit consumes additional biomass to generate producer gas, which could be used to produce electricity. The internal electricity requirement for the AG unit operation is assumed to be supplied from the gross power generated by the unit itself. Hence, the system requires no external energy supply and is entirely self-sufficient. BG1 and BG2 are evaluated under two conditions – without carbon capture and with carbon capture – giving rise to four scenarios. The gasification system for hydrogen production and the air gasification unit are depicted in Figure 1 and Figure 2, respectively.

2.2 Economic assessment

The economics of the BG pathway are established by estimating the minimum selling price (MSP) of hydrogen. The MSP is the lowest price at which the project is still profitable. This is evaluated through a discounted cash flow analysis and setting the Net Present Value (NPV) to zero, as described in the below equation (Ganesh et al., 2024).

$$\sum_{i=1}^{N} \frac{(P_i \times MSP)-(CC_i+SOC_i)}{(1+r)^i} = 0 \quad (1)$$

Where P_i, CC_i, SOC_i, r, and N are the annual production, carrying charges, system operating costs in the 'i'th year, the discount rate, and the system lifetime, respectively. The SOC includes the annual expenditure on fuel, utilities, wages, and maintenance. The carrying charges include the Total Capital Recovery (TCR) and cost of capital (interest on debt and returns on equity) (Bejan et al., 1996; Ganesh et al., 2024). The key inputs and assumptions for the economic assessment are provided in Table 1.

2.3 Environmental assessment

While carbon emissions from the direct conversion of biomass are considered biogenic, the other activities such as agriculture, biomass collection, transportation, and pre-processing contribute to the overall emissions. The emissions from hydrogen production depend on the emissions from each stage of the life cycle. The emissions during agriculture phase include N_2O-N emissions from nitrogenous fertilisers and CH_4 emissions from flooded rice fields. The average biomass mix of Karnataka majorly

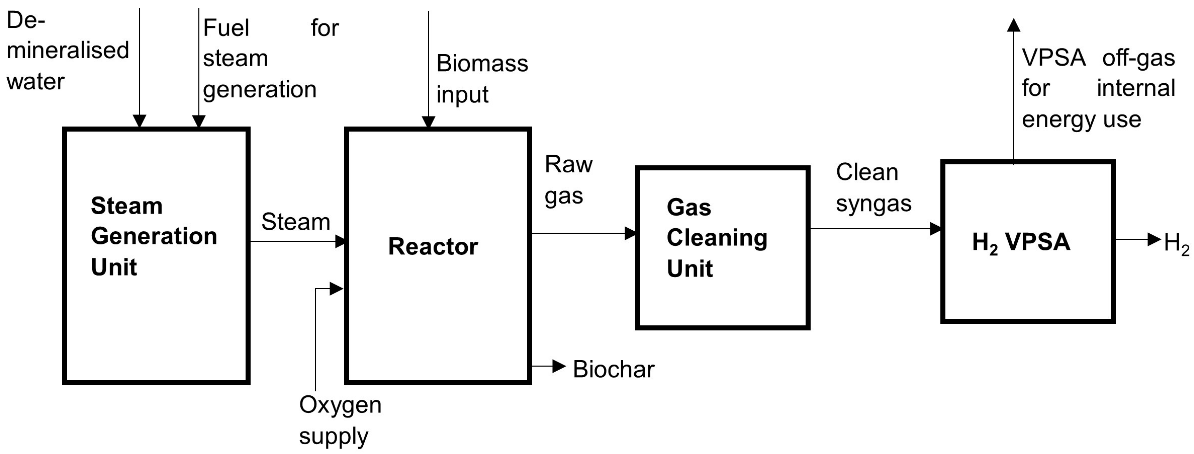

FIGURE 1: Biomass gasification pathway for hydrogen production (Ganesh et al., 2024).

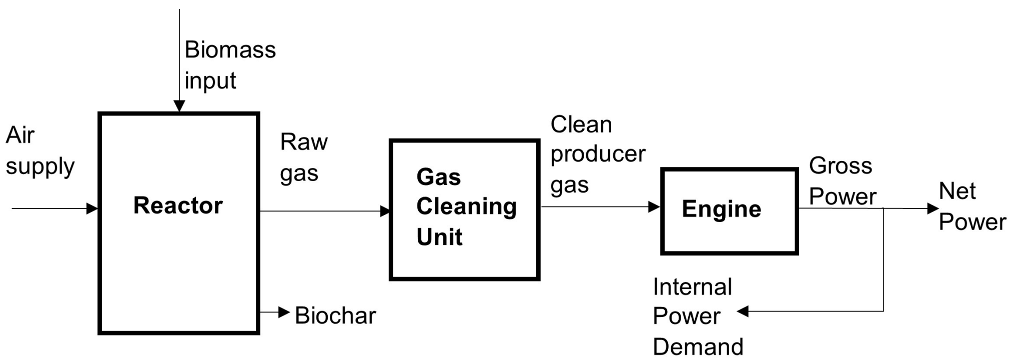

FIGURE 2: Auxiliary air gasification unit (Ganesh et al., 2024).

TABLE 1: Key inputs for economic assessment of BG pathway.

Parameter	Value	Reference
Discount rate	12%	(Union Bank of India, n.d.)
Debt repayment period	10 years	Assumption
System lifetime	25 years	Assumption
Inflation rate	6%	(RI, n.d.)
Annual operation	7500 hours	Assumption
Total capital investment (for 100 kg/h BG-H_2 plant)	INR 863-1177 million ($10.45-14.25 million)	Quotations
Annual maintenance costs	2% of Fixed Capital Investment	(Wang et al., 2019)
Electricity tariff	INR 8/kWh ($0.10/kWh)	(Karnataka Electricity Regulatory Commission, 2024)
Feedstock cost	INR 6/kg ($0.07/kg)	Average cost of biomass as per correspondences with local suppliers
Labour costs	INR 40,000/day ($484/day)	Obtained through expert opinions and considering three 8-hour-shifts per day

includes sugarcane tops and trash (24%), coconut fronds (22%), maize stalk (22%), maize cobs (10%), rice straw (8%), and others (pigeon pea stalk, sunflower stalk, cotton stalk etc.) (Punjab Renewable Energy Systems Pvt. Ltd., 2017). The agricultural emission factors are obtained from (Jain et al., 2016) and (Rathnayake & Mizunoya, 2023). Economic allocation methodology is followed for emission allocation to the crop residues. The life cycle of the BG pathway and the system boundary considered in this assessment are described in Figure 3. The functional unit is 1 kg of hydrogen produced. GREET® 2023 tool is used for the environmental assessment, considering the case of Karnataka State, India.

The prevalence of machinery for the harvest of agricultural biomass influences the biomass collection efficiency. Residue collection without suitable machinery (balers) becomes labour-intensive. The specific diesel consumption for straw baling operations is 7.59 litres/hectare, with a biomass collection efficiency of 74.6% (All India Coordinated Research Project on Farm Implements and Machinery, 2008). The residues are assumed to be procured from up to a radius of 20 km, and the pre-processed biomass is transported by about 100 km before reaching the BG plant. The power demand for the pre-processing plants is adopted from the literature (Tripathi et al., 1998). The indirect emissions from the purchased electricity (for scenario BG1) are estimated through the electricity mix of Karnataka state for 2023 (Central Electricity Authority: Operation Performance Monitoring Division, 2023; Central Electricity Authority: Renewable Energy Project Monitoring Division, 2023). About 2% of the biomass input remains unconverted and comes out from the screw conveyor in the form of biochar, which

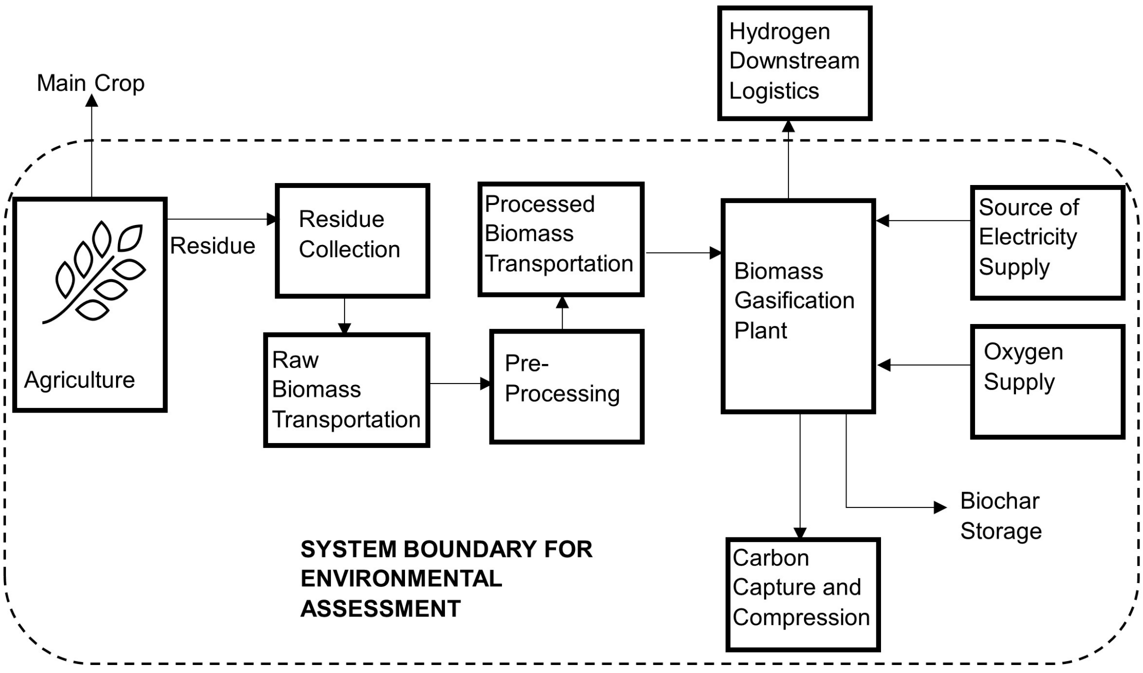

FIGURE 3: Life-cycle of BG-H_2 pathway and system boundary for assessment.

TABLE 2: Key inputs for SMR and WE pathways.

Parameter	Steam methane reforming (Katebah et al., 2022)	Water electrolysis
System output capacity	20830 kg/h	100 kg/h
Total capital investment	\$0.15/kg H_2	\$519/kW (Ahmad Mayyas et al., 2018)
Maintenance costs	7% of fixed capital investment	4.6% of Fixed Capital Investment (T. Ramsden; D. Steward; J. Zuboy, 2009)
Primary fuel demand	3.2-3.4 kg natural gas/kg H_2	12.5 litres (water)/kg H_2 (Ahmad Mayyas et al., 2018)
Electricity demand	Generated internally	55-60 kWh/kg H_2 (Ahmad Mayyas et al., 2018)
Cost of natural gas	\$3.7/GJ	-
Grid electricity tariff	-	INR 8/kWh (\$0.10/kWh) (Karnataka Electricity Regulatory Commission, 2024)
Electricity tariff from remote open-access renewable power plant	-	INR 5.50/kWh (\$0.06/kWh)
Required captive solar plant capacity	-	27 MW (for 100 kg/h H_2 plant)
Solar plant capital costs	-	INR 40 million/MW (\$0.48 million/MW) (Karnataka Electricity Regulatory Commission, 2023)
Levelised cost of electricity from captive plant	-	INR 3.08/kWh (\$0.04/kWh) (estimated)

could be sequestered to reduce the life cycle emissions further. The carbon dioxide in the syngas or off-gas could be separated through a VPSA unit with sodium zeolites (Singh et al., 2022).

2.4 Comparison with other hydrogen pathways and emission reduction incentive

The biomass gasification pathway is compared with steam methane reforming (SMR) and water electrolysis (WE). The costs and emissions of the SMR with and without carbon capture scenarios are obtained from literature (Katebah et al., 2022). Proton Exchange Membrane (PEM) electrolysis is considered in this study, as described by (Ahmad Mayyas et al., 2018). The electricity demand for the electrolysis pathway is assumed to be procured from one of three sources: grid electricity (scenario WE-grid, which is a non-green scenario), captive ground mounted solar PV plant with power banking facilities (WE-Captive), and remote renewable power plants (WE-Remote) at INR 5.50/kWh (\$0.06/kWh - \$1 = INR 82.57, as of December 31, 2023 (Open Financial Exchange (OFX), n.d.), obtained through correspondences with local companies procuring power from open-access remote plants. The key inputs and assumptions for SMR and WE pathways are provided in Table 2. The economic and environmental performances of the four BG scenarios are compared with two SMR scenarios and three WE scenarios.

The captive solar PV plant is evaluated using PVSYST V6.74 to estimate the daily and annual electricity produced. The capacity utilisation factor of a solar plant installed near Bengaluru is assessed to be about 0.193. Wheeling and banking of power is feasible in Karnataka state, which ensures steady availability of solar power (Karnataka Electricity Regulatory Commission, 2023). With the electricity demand for electrolysis at about 55-60 kWh/ kg (Ahmad Mayyas et al., 2018), it is estimated that a 27 MW solar plant (with power banking) could sufficiently provide the annual electricity demand of a 100 kg/h output system.

The economic and environmental dimensions are reconciled by introducing the Emission Reduction Incentive (ERI). It is estimated as the incentive required for each scenario per tonne of CO_2 saved with reference to the scenario with the lowest cost, to achieve economic viability. Scenarios with lower ERIs are considered to have better combined economic and environmental performance.

3. RESULTS AND DISCUSSION

3.1 Minimum selling price of H_2 through BG pathway

The minimum selling price is a function of the fixed operating costs (maintenance), variable operating costs (fuel, labour, and utilities), and carrying charges (capital costs and interest payments). BG2 has higher carrying charges and fuel costs due to the requirement of an auxiliary air gasification unit and additional biomass for in-situ electricity generation. Carbon Capture (CC) from the off-gas or the syngas requires an additional VPSA unit and compressor, increasing the overall hydrogen MSP. Hence, BG1 without CC has the lowest MSP of INR 307/kg (\$3.71/kg). BG2 with CC has the highest hydrogen MSP of INR 352/kg (\$4.26/kg), with close to a half being contributed the fuel costs alone. This is caused due to the additional electricity demand for CO_2 compression, requiring biomass input to the auxiliary gasification unit. The breakdown of the MSP for the four scenarios are provided in Figure 4.

3.2 100-year Global Warming Potential (GWP-100) of BG-H_2 pathway scenarios

Under conditions of no carbon capture, the indirect emissions contribute majorly to the GWP (7.39 kg CO_2-e/kg H_2) due to grid electricity use in the gasification plant. This can be attributed to the high share (54%) of coal in the electricity mix of Karnataka. There is a significant reduction in the GWP with the introduction of in-situ power generation. It is possible to achieve negative emissions with the capture and compression of CO_2 through the VPSA system. The breakdown of the GWP-100 of the four BG scenarios are provided in Figure 5.

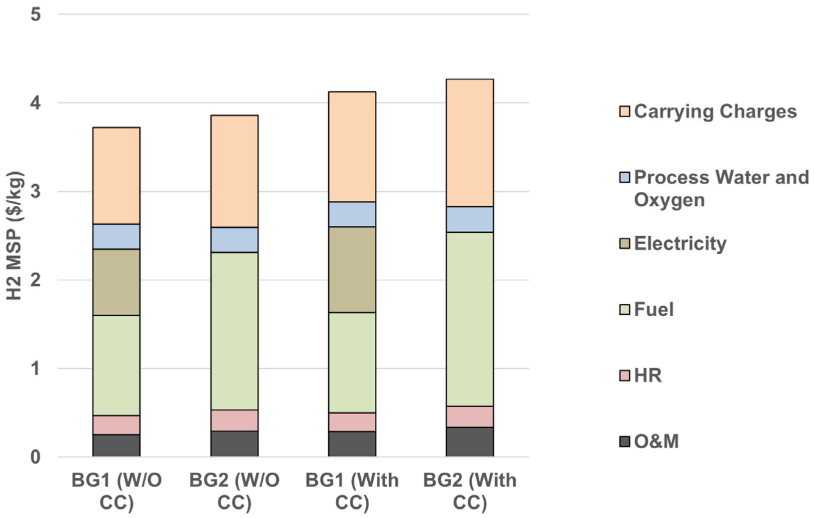

FIGURE 4: Breakdown of hydrogen MSP for BG pathway scenarios.

FIGURE 5: Breakdown of GWP-100 for BG-H_2 scenarios.

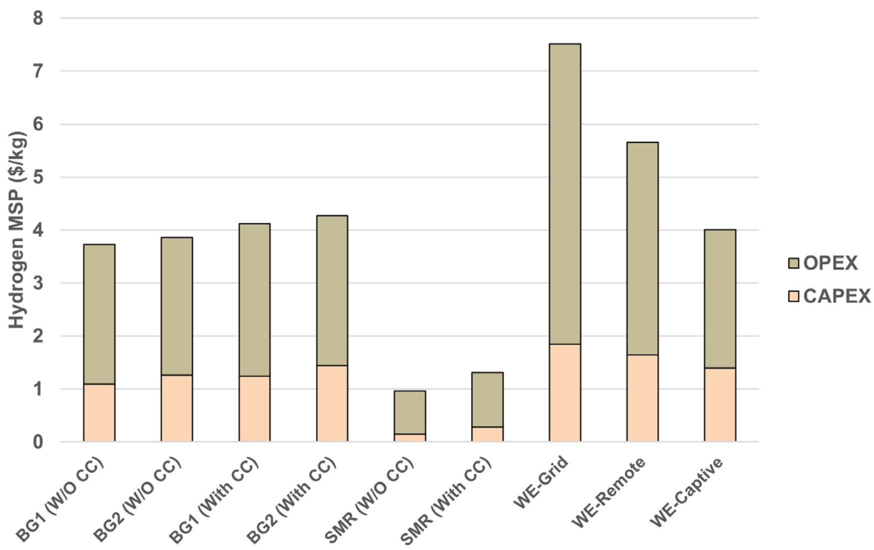

FIGURE 6: Comparison of hydrogen MSPs for BG, SMR, and WE systems.

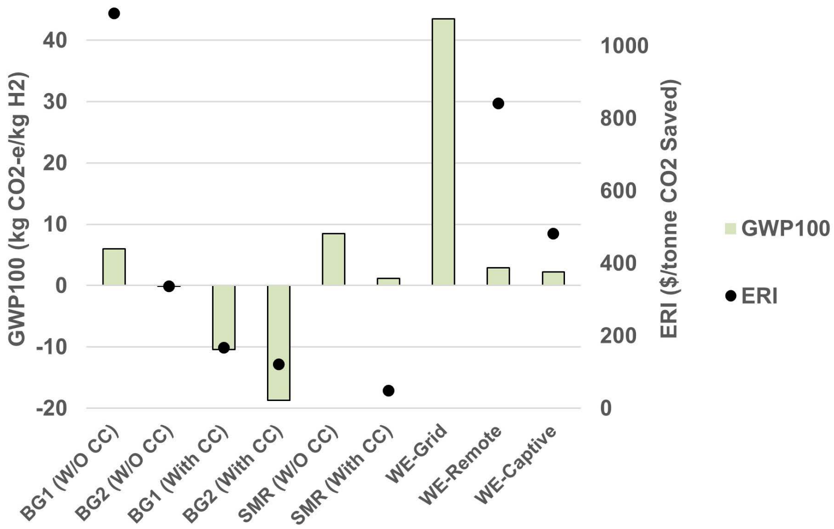

FIGURE 7: Comparison of GWP100 for BG, SMR, and WE systems.

3.3 Scenario comparison and emission reduction incentives

The comparison of BG, SMR, and WE scenarios based on hydrogen cost and GWP100 with the ERIs are provided in Figure 6 and Figure 7, respectively. The lowest hydrogen cost ($0.96/kg) could be obtained through commercial-scale steam methane reforming. However, this pathway has a high GWP of about 8.5 kg CO_2-e/kg H_2. Carbon capture from the SMR process increases the hydrogen cost by 36%, while reducing the emissions by about 86% (Katebah et al., 2022). On the other hand, hydrogen through PEM electrolysis is power-intensive, resulting in very high share of Operational Expenditure (OPEX) in the hydrogen MSP. The highly coal-reliant power grid of Karnataka leads to high emissions in the WE-Grid scenario. Procuring electricity from remote renewable power plants at INR 5.50/kWh ($0.06/kWh) reduces the costs by 25% and emissions by 93%. Installing a captive solar PV plant with a capacity of about 27 MW with power banking facilities improves the economic and environmental performances further. With a 25-35% contribution of the Capital Expenditure (CAPEX) to the hydrogen costs, reduction in the electrolyser stack costs in the future could lead to significant gains. It is evident from Figure 6 that the hydrogen MSP of $3.71-4.26/kg from biomass gasification is comparable to the WE-captive scenario ($4/kg), making it economically attractive compared to the WE pathway. The SMR with carbon capture scenario fares better than the two renewable power scenarios of electrolysis from both cost and emissions perspective. Among all the reviewed scenarios, BG-2 (with CC) displays the best environmental performance.

Individually, SMR without CC has the best economic performance, while BG-2 with CC has the best environmental performance. The combined economic and environmental performance of the hydrogen production pathways are measured through the emissions reduction incentive. Due to its high cost and emissions, BG-1 without CC requires the highest incentives ($1090/tonne of CO_2 saved) to achieve hydrogen cost parity with the reference scenario (SMR without CC). Among the BG scenarios, BG-2 with CC requires the lowest incentive of about $127/tonne of CO_2 saved. Water electrolysis, on the other hand, requires a minimum incentive of $482/tonne of CO_2 saved. Hence, among the green pathways, BG has the best combined performance. The overall best combined performance is displayed by SMR with CC, requiring an incentive of only $48/tonne of CO_2 saved, owing to subsidised feedstock, technology maturity, and economies of scale.

4. CONCLUSIONS AND FUTURE WORK

It is evident from this analysis that the BG pathway to hydrogen has the best environmental potential, while SMR has the best economic potential. The BG pathway is affected by uncertainties in the biomass supply chain. Higher offered biomass prices would increase the participation rates and reduce the procurement radius, leading to higher plant availability and lower feedstock transportation costs. On the other hand, increasing the offered biomass price would increase the variable operating costs. From a purely monetary perspective, the BG system costs could be reduced through suitable policy initiatives. The ERI provides the combined economic-environmental comparison of the alternatives. Among the green hydrogen pathways, the BG pathway has a better combined performance, with an incentive requirement of $127-336/tonne, compared to $482-842/tonne for water electrolysis using solar power. On the other hand, the fossil-hydrogen pathways have better economic metrics due to better technology maturity, feedstock subsidies, and economies of scale. With a heavy reliance on imported natural gas in India (Sukalp Sharma, 2024) and the increasing fuel prices, the extensive development of the biofuel value chain through the BG pathway would provide monetary savings through reduced imports, compared to the SMR pathway. The BG pathway would also pave way for the complete utilisation of the available waste biomass (residues), generating additional income for small and marginal farmers and helping the local economy. The extent of social benefits from the large-scale integration of the biofuel value chain remains to be evaluated. Hence, a rigorous, indicator-based approach for the quantification of the performance of the biofuel value chain is essential in addition to the economic and environmental assessments.

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WHERE DO THEY GO? A REVIEW OF THE WASTEWATER TREATMENT PROCESS AND ITS IMPACT ON THE FATE OF MICROPLASTICS

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ABSTRACT

Wastewater treatment plants (WWTPs) are facilities designed to sanitise wastewater (WW) from different sources. A WW treatment process combines physical, chemical and biological reactions divided into 3 or 4 stages, according to the effluent standards of each site. WWTPs are intended to remove certain pollutants from the water stream, but not microplastics (MPs), plastic particles ranging from 5 mm to 1 µm. WWTPs work as source and destination for MPs. This review provides an insight into the stages included in WW treatment processes according to different technologies employed, alongside a critical evaluation of their influence on MPs. Relevant information and knowledge gaps have been identified. The removal of MPs depends on a variety of factors than just the type of incoming water or WWTP location. The most significant removal of MPs from WW occurs during the preliminary and primary stages. MPs removal efficiency fluctuates depending on the systems operating during the secondary and tertiary phases. As the comparison of information between studies is complicated due to the units and terms used to report and label the collected WW, we recommend that (1) kg/day units are applied if mass balance is of interest, and number of particles per L/day, considering the flow rate data from the sampling dates or annual average flow; (2) to further explain the selection criteria to classify MPs based on their size; and (3) to reach a consensus to name and identify the sampling points, such as the raw WW and effluent from the next stages.

1. INTRODUCTION

WWTPs are facilities that uses physical, chemical, and biological methods to treat wastewater (both sanitary wastewater and urban runoff) step by step, making it substantially cleaner, odourless, and compliant with regulatory criteria for safe release into water bodies (The Council of the European Communities, 1991; Vesilind, 2003). WWTP receives, treats, and stores wastewater from domestic dwellings, commercial and industrial premises, run-off from streets and other surfaces. These treatment works are designed to remove undesirable constituents in a systematic and stepwise fashion; such constituents might include large floating solid items (e.g. litter, vegetation, nappies); fats, oils and grease (FOG); organic matter; nutrients; pathogens (e.g. bacteria and viruses); toxic compounds, etc, and might be in form of suspended or dissolved solids. Some emerging contaminants may not be removed from water stream, such as MPs (Talvitie et al., 2017).

Microplastics are commonly (but not universally) defined as plastic pieces ranging from 5 mm to 1 µm, encompassing a variety of types, colours and shapes (Crawford and Quinn, 2017a; Frias & Nash, 2019). This definition is used for this study. Particles smaller than 1 µm are catalogued as submicron and nanoplastics (Caldwell et al., 2022). There are two categories according to the source of MPs (Cole et al., 2011). Primary MPs are deliberately created to meet specific tasks and products, whilst secondary MPs originate from further fragmentation of larger plastic items, due to mechanical abrasion, UV radiation and/or biological degradation (Crawford and Quinn, 2017a).

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The origins and fate of MPs have been investigated and numerous sources and destinations have been identified (Hasan Anik et al., 2021). As WWTPs receive many waste products from different sources, including large and small plastic particles, these treatment systems could work as an ultimate fate of MPs (Landeros Gonzalez et al., 2022). However, WWTPs are known to be sources of microplastics to water and soil, via the discharge of aqueous effluent into waterbodies and the use of sludge as an agricultural fertiliser and soil conditioner (Weithmann et al., 2018; Cowger et al., 2019).

The route from the raw wastewater (i.e. influent or crude sewage) to the treated water (i.e. final effluent) during WW treatment is a multifaceted procedure. Therefore, it is crucial to identify the different stages included in the WW treatment process and understand the working mechanism behind each step. Likewise, MPs throughout the WW treatment process is complex as there are many factors involved that might change their composition and external features in the final effluent and sewage sludge. However, these factors in some cases are not taken into account when it comes to study the presence of MPs in WWTPs. In this comprehensive review, we describe each stage in the wastewater treatment process according to different technologies applied, alongside a contemporary, critical evaluation of the state of knowledge of the impact of the treatment systems on MPs.

2. METHODS

This paper reviews the treatment process that takes place in wastewater treatment plants, evaluating the technologies implemented at each stage. The first section presents the stages of the treatment activity (preliminary, primary, secondary and tertiary) in relation to their general function and effects. Furthermore, in the second chapter of this study we analyse the impact of the treatment process on the microplastics in wastewater. The literature cited was obtained using “wastewater treatment plants” and “microplastics” as search terms. Published works referred to in the present study focused on: the occurrence and characteristics of microplastics in wastewater and sludge, and how the treatment equipment and other factors influence microplastics. Information such as location, sites for study, population served, average inflow, type of samples taken (WW and/or sludge), sampling collection period, sampling duration, sampling filter pore size, type of polymers, shape and removal efficiency from WW, were essential for the purposes of this review. The research for scientific articles relating to microplastics and WWTPs was completed in April 2022.

3. STAGES OF A WASTEWATER TREATMENT PLANT

3.1 Preliminary stage

The preliminary treatment stage typically consists of the physical exclusion of untreatable material (e.g. large plastic items) from the raw WW by screening and grit/grease removal (Butler et al.,1995; Oakley, 2019). Screening may include coarse and fine bar screens, usually from 25 mm to 6 mm (Butler et al., 1995; Oakley, 2019). The screened WW then continues to aerated grit chambers that facilitate the sinking of heavier solids - particles such as grit, gravel and sand (density of ~2500 kg/m3) (Stuetz and Stephenson, 2009). Fats, oil, grease and other floating materials are skimmed from the tanks (Butler et al., 1995; U.S. EPA., 1999b). Dissolved air flotation (DAF) is a common technique designed to remove insoluble substances such as oils, greases and suspended solids (Englande et al., 2015). During this process, small bubbles are produced by introducing air into WW at high pressure. These bubbles cling to the suspended solids, and consequently these solids rise to the surface and are removed by skimming (Englande et al., 2015; Bui et al., 2020; Chaubey, 2021).

Bar screens are usually cleaned mechanically but also can be cleaned manually. Sludge is collected separately and the effluent is then discharged to the next treatment stage. This preliminary stage of the process is mainly to protect a WWTP’s equipment from any damage or loss of efficiency derived from excessive scum formation, debris, oil films or fat accumulations (Oakley, 2019; Jasim, 2020). The grit removed by the screening process can be further split according to its solid composition. The more fibrous fraction, such as from sanitary products, might be taken out after screening and incinerated, whilst the solid fraction (e.g. grit) goes to landfill or other disposal facility (U.S. EPA., 2003; Michielssen et al., 2016; WWTPs staff, personal communication, October 18, 2021). Figure 1 shows the material discarded from this first stage.

3.2 Primary stage

After preliminary treatment, WW is introduced into a sedimentation tank or clarifier. In the primary clarifiers, the settleable solids and buoyant organic material are removed, which reduces the amount of suspended solids (Oakley, 2019). Chemical coagulants such as iron salts, ferric chloride and aluminium sulphate, are then used to produce dense and rapid-settling flocs (loosely clumped masses of fine particles) in order to increase the removal of suspended solids, phosphorus and heavy metals; and to reduce five-day biological oxygen demand (BOD_5), (Yargeau, 2012; Oakley, 2019; Jasim, 2020). The principal objective is to attain a homogeneous liquid that can subsequently be biologically treated.

The design of these sedimentation tanks falls into three classifications: a) horizontal flow, b) inclined surface and c) circular, radial-flow (Englande et al., 2015; Jasim, 2020) (Figure 2). The horizontal tank is a large rectangular tank where solid settle by gravity whilst the supernatant continues to the next wastewater treatment stage (Englande et al., 2015). The Lamella gravity settler system is an example of the inclined surface sedimentation tank. The inclined plates aid acceleration of deposition of the particles, as the particles agglomerate as soon as they reach the surface of the plate (Tarpagkou and Pantokratoras, 2014; Ma et al., 2019). In a circular tank, the solids that settle at the bottom (primary sludge) are removed continually by a rotary sludge scraper. The sludge is then pumped out and stored prior to treatment (Englande et al., 2015; Oakley,

FIGURE 1: Discarded waste from the preliminary stage. A) Fibrous waste; B) Grit removed.

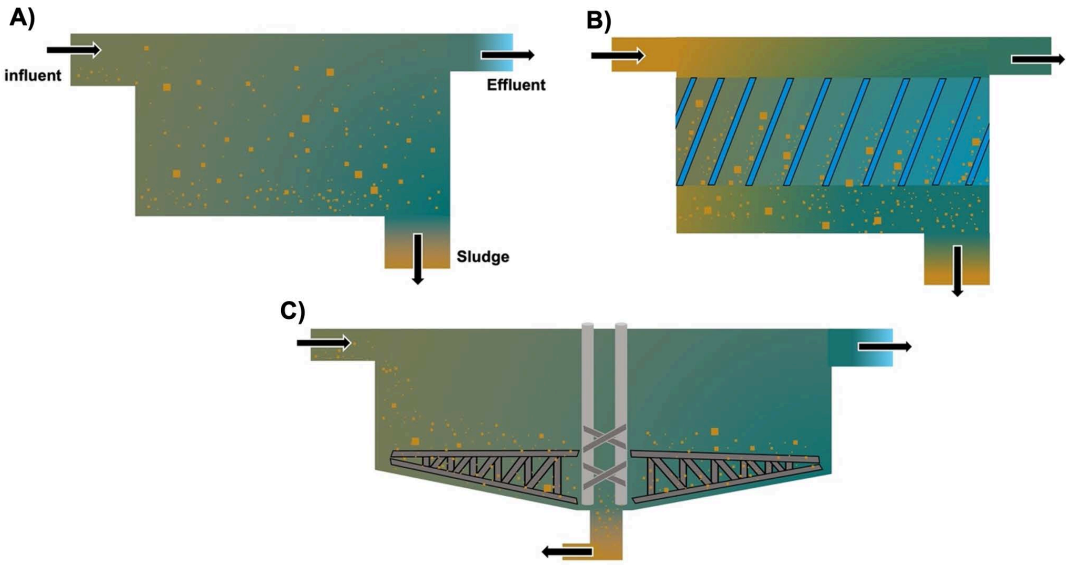

FIGURE 2: Types of Primary Sedimentation Tanks. A) Conventional (horizontal flow); B) Inclined surface (e.g. Lamella gravity settler system); and C) Circular.

2019). Across the preliminary and primary stages, a total of 50-70% of total suspended solids are removed (Qasim, 2017; Jasim, 2020).

3.3 Secondary stage

Secondary treatment applies biological processes to treat the WW (Englande et al., 2015). The purpose of secondary treatment is the removal of OM, suspended solids, reduction of biochemical oxygen demand, and removal of other compounds left from the previous stage (Vesilind, 2003; Jasim, 2020). There are two main categories of biological processes according to the microorganisms' performance mechanism: suspended- and attached-growth processes. In a suspended growth process, microorganisms are maintained in liquid suspension by mixing. In an attached growth (biofilm) process, microorganisms are attached to some type of medium, such as rock, gravel, plastics or other synthetic materials (Cohen, 2001; Metcalf & Eddy Inc., 2004). Both biological treatments sometimes occur in the same process (Metcalf & Eddy Inc., 2004).

Nitrification and denitrification are processed to remove nitrogen compounds from WW. Firstly, the nitrification process occurs when the ammoniacal nitrogen is converted to nitrate under an aerobic environment (Englande et al., 2015). Following this, the nitrate is converted to nitrogen gas, a process called denitrification (Englande et al., 2015; Frankel, 2020). During this process, bacteria degrades nitrogen products from which oxygen is released (Metcalf & Eddy Inc., 2004; Frankel, 2020). In the denitrification stage of treatment, the addition of oxygen is not required. Anaerobic treatment processes are used for treating WW with high concentrations of biodegradable organic materials. These microorganisms do not need oxygen to break down OM from the WW: the OM is decomposed to CH_4, CO_2 and other gases without oxygen (Englande et al., 2015; Ahammad and Sreekrishnan, 2016)

Under aerobic conditions, microorganisms use oxygen in the WW to decompose OM. Surface aerators and aeration diffusers are used to add oxygen to the system (Frankel, 2020). The surface aerators agitate the WW mechanically, so the surface mixes with oxygen from the atmosphere; aeration diffusers introduce air or pure oxygen in the form of bubbles into the WW from the bottom of the treatment vessel (Metcalf & Eddy Inc., 2004). There are many methods to achieve aerobic biological treatment processes, for example, activated sludge, trickling filtration, and fluidised and fixed-bed bioreactors (Yildiz, 2012; Englande et al., 2015; Frankel, 2020). In the following sections, these are explained/evaluated in detail.

3.3.1 Activated sludge

The activated sludge process (ASP) is a biological treatment in the secondary stage that usually includes aeration and clarification tanks (Yildiz, 2012) (Figure 3).This process is a suspended growth biological treatment, thoroughly mixing living microorganisms with organic compounds, using OM as a source of nutrients (Yildiz, 2012; Pennsylvania Department of Environmental Protection, 2014; Jasim, 2020).

As microorganisms are growing whilst breaking down OM, they clump together to form flocs ("flocculation"). Then flocs settle to the bottom of the tank and create an active mass known as activated sludge (Yildiz, 2012; Frankel, 2020). The mixture of activated sludge and WW in the aeration tanks (mixed liquor) flows to the final clarifier tanks where the sludge is removed. The remaining liquid is discharged as effluent. The sludge from this stage can be disposed to be separately treated or reused in the aeration tank (return activated sludge) (Chaubey, 2021). The return of activated sludge helps to maintain an adequate population of microorganisms (Pennsylvania Department of Environmental Protection, 2014; Frankel, 2020).

3.3.2 Trickling filtration

A trickling filter is an attached growth biological treatment process where the organisms are attached to a fixed bed instead of being suspended (Yildiz, 2012) (Figure 4). The effluent from the previous stage flows over a surface to which the microorganisms adhere and grow, creating a biofilm on the surface of the filter medium (Englande et al., 2015). As it is an 'open-air' system, the oxygen can be supplied from the atmosphere, or can be artificially pumped into the system (Englande et al., 2015). One characteristic is the need for continuous recirculation of the treated effluent into the influent, providing a wetting rate sufficient to keep the microorganisms active (Yildiz, 2012).

The filter medium is a bed of inert porous material inside a settling tank usually comprising stone, but it can comprise synthetic material such as glass, PVC and polyurethane foam that may offer a greater surface area and depth for microbial growth (Englande et al., 2015; Ahammad and Sreekrishnan, 2016).

3.3.3 Fluidised and Fixed Bed Bioreactors

Fixed-bed bioreactors consist of a packed bed medium wherein surface microorganisms can settle and grow, whilst the medium components remain immobile (Frankel, 2020). The influent is introduced from the bottom of the tank. For aerobic applications, air is mechanically injected into the chamber or spread across the reactor floor (Yildiz, 2012). In anoxic conditions, these reactors are used for denitrification purposes (Frankel, 2020).

Fluidised bed bioreactors use biocarriers that offer a growing surface to the microorganisms in an aerated environment. They are meant to provide a medium on which the microorganisms can grow and degrade OM (Englande et al., 2015). Biocarriers can be made of polyethylene (PE), polyurethane (PUR), polypropylene (PP), polyvinyl chloride (PVC), and high-density polyethylene (HD-PE), and their sizes range from 7 to 48 mm (di Biase et al., 2019). The hydrophobicity of these polymer carriers increases the microorganism's attachment rates (di Biase et al., 2019).

3.3.4 Membrane Bioreactors

A membrane bioreactor (MBR) is a biological reactor that uses ultra- and micro-filtration membranes to remove high levels of suspended solids (Englande et al., 2015; Frankel, 2020) (Figure 5). This process combines the activated sludge process with membrane filtration. The MBR technology enables a more efficient removal of pollutants, as it involves a relatively long retention time (Englande et al., 2015). The membrane pore size may be as small as 0.1 mm, guaranteeing a high-quality clarified effluent (Wen et al., 2010).

A MBR can be divided into three types according to their configuration: A) Submerged/immersed membrane bioreactor; B) Cross-flow membrane bioreactor; C) Hybrid membrane bioreactor (Figure 5). BOD and suspended solids removal efficiency using MBR technology is typically >90% (Wen et al., 2010).

3.4 Tertiary stage

Some WWTPs include an additional, final, stage of treatment. Tertiary treatment uses advanced techniques to improve the quality of the final effluent. The reasons for additional treatment are to protect water ecosystems and meet the required quality standards of the receiving water (DEFRA, 2012). These techniques include treatment by disinfection (chlorination, UV light, ozonation) and advanced filtration processes (Dhodapkar and Gandhi, 2019; Chaubey, 2021).

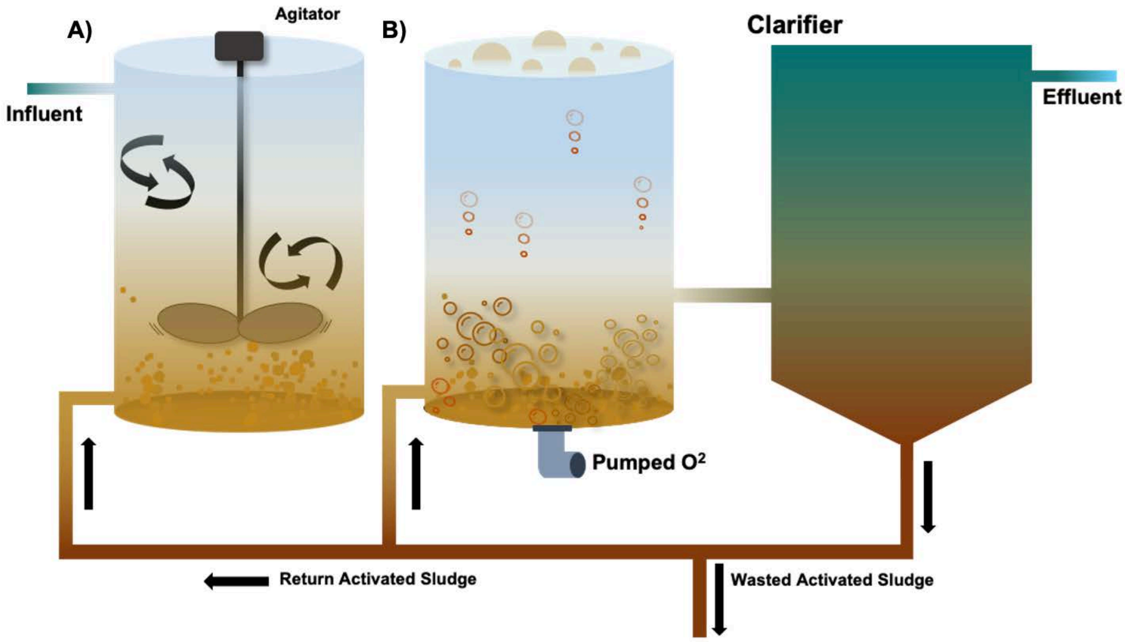

FIGURE 3: Activated sludge process scheme. A) Air mechanically added (surface aerators); B) Pure oxygen pumped into the system (aeration diffusers).

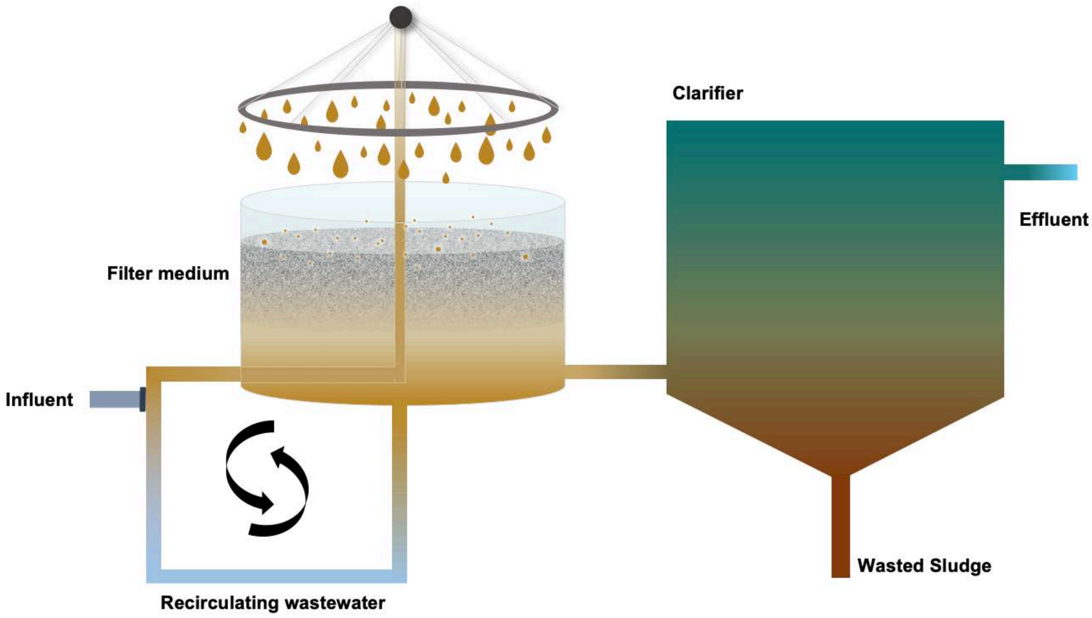

FIGURE 4: Schematic diagram of the trickling filtration process. This system consists of a settling tank, a filter medium, an influent wastewater distribution system, a clarifier and a recirculation pipe.

3.4.1 Disinfection

Chlorination, ozonation and UV light exposure are the basic mechanisms used to further disinfect WW. Chlorine is widely used for its capacity to eliminate many pathogenic microorganisms. Chlorination agents include chlorine gas, chlorine dioxide, calcium hypochlorite ($Ca(OCl)_2$) and sodium hypochlorite (NaOCl) (Stuetz and Stephenson, 2009; Collivignarelli et al., 2017). Their performance is determined by the time of exposure, dosage, and the pH and temperature of the WW (Collivignarelli et al., 2017; Chaubey, 2021). Chlorine residuals need to be removed from the final effluent as they are toxic to the aquatic environment (U.S. EPA., 1999a).

Another alternative for WW disinfection is the application of ozone (O_3). O_3 generation starts by applying a high electrical discharge in a molecule of oxygen (O_2), taken from air or high-purity oxygen, to be dissociated into oxygen atoms. Subsequently, these atoms collide with an oxygen molecule to form this unstable gas (Metcalf & Eddy Inc., 2004; Collivignarelli et al., 2017). Ozone is a strong oxidant capable of disintegrating the cell wall and membrane

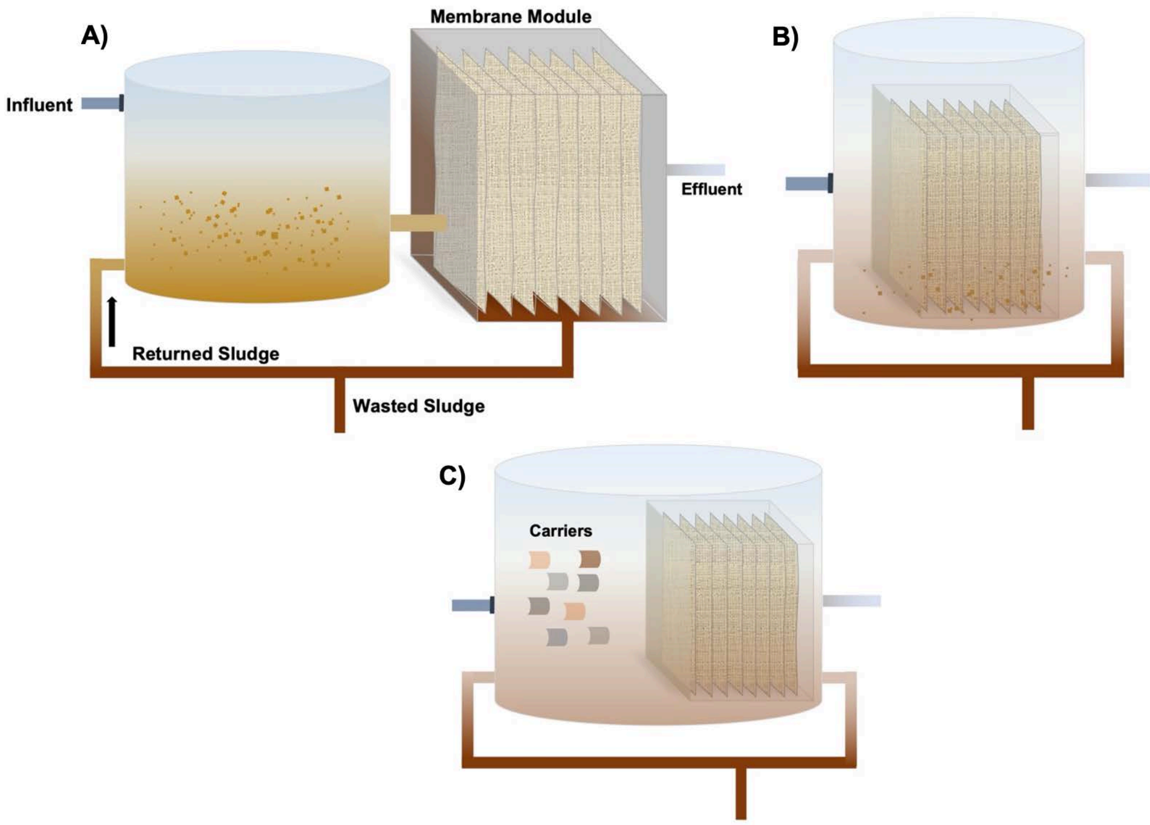

FIGURE 5: Schematic diagrams of the 3 main types of membrane bioreactors. A) Submerged membrane bioreactor; B) Cross flow membrane bioreactor; C) Hybrid membrane bioreactor. The biocarriers are polymers used in the fluidised bed reactors.

of microorganisms. However, it is not suitable for WW containing high levels of BOD and suspended solids due to its high cost (U.S. EPA., 1999b).

Ultraviolet (UV) radiation is a form of electromagnetic energy covering the wavelength range of 100–400 nm, with the optimum germicidal fraction between 220 and 320 nm (USEPA., 1999c; Collivignarelli et al., 2017). Disinfection by UV radiation is a physical process effective against most viruses, cysts and spores as it destroys the genetic material of microorganisms, preventing further reproduction. UV radiation has little effect on nitrogen compounds or ammonia, and some factors such as turbidity and suspended solids can decrease the performance of UV disinfection (USEPA., 1999d).

3.4.2 Advanced filtration systems

Advanced filtration is a process where WW flows through a permeable layer. The materials include a filter paper, a membrane, a sieve or other suitable porous medium (Beverly, 2011). Filtration removes substantial quantities of solids from wastewater, promoting effective disinfection.

Disc-filter technology uses fabric membranes instead of a granular medium to filter the effluent. WW enters thorough a channel and flows through the filter. This filter cloth is made of either polyester (PES) or stainless steel (Metcalf & Eddy Inc., 2004). This technique employs a rotary system that operates in an intermittent or a continuous-backwash mode. The particles retained are thereby removed from the screen (Metcalf & Eddy Inc., 2004).

Granular filtration WW filters WW through materials such as sand, anthracite grains or gravel (Bui et al., 2020). During the flow process, suspended solids are retained, and some of them are biochemically decomposed and removed later (Arndt and Eric, 2004; Beverly, 2005). There are two types of sand filtration: 1) slow (known as gravity) sand- and 2) rapid sand- filtration. Slow sand filtration uses gravity-fed flows, whilst rapid sand filtration uses either pumping or head pressure (Arndt and Eric, 2004).

In order to conduct projects related to WWTPs, it is crucial to acknowledge the design and function of these facilities and understand, in this case, the performance of MPs throughout the treatment system.

4. IMPACTS OF WASTEWATER TREATMENT STAGES ON MICROPLASTICS CONCENTRATIONS

4.1 Effects on microplastics throughout treatment process

The numbers of MPs in the influent and effluent fluctuate in each stage and system, leading to an overall decrease throughout the treatment process. It is during the preliminary and primary stages where most of the MPs are removed via particle skimming and sludge settling processes (Carr et al., 2016; Michielssen et al., 2016; Murphy et al., 2016; Qasim, 2017; Talvitie et al., 2017; Lares et al., 2018; Gies et al., 2018; Magni et al., 2019; Bui et al., 2020). Depending on the plastic size and density (Table 1), mate-

TABLE 1: Density range of some plastics.

Polymer	Density (g/cm^3)	Reference
PE-LD	0.94	Crawford and Quinn, 2017a
PE-HD	0.97	
PLA	1.24	
PS	0.01-1.50	
PUR	0.08-1.03	
CR	0.11-1.25	
PP	0.9-1.27	
ABS	1.00-1.21	
PMMA	1.10-1.25	
PA	1.12-1.38	
PVC	1.15-1.50	
PET	1.30-1.50	
PC	1.40-1.50	
Cellophane	1.40–1.53	Gago et al., 2018
Rayon	1.53	
PTFE (Teflon™)	2.10-2.30	Crawford and Quinn, 2017a

ABS: Acrylonitrile butadiene styrene; CR: Polychloroprene (neoprene) PA: Polyamide; PC: Polycarbonate; PE-LD: Low Density Polyethylene; PE-HD: High Density Polyethylene; PET: Polyethylene Terephthalate; PLA: Polylactic Acid; PMMA: Polymethyl Methacrylate; PP: Polypropylene; PS: Polystyrene; PTFE: Polytetrafluoroethylene; PUR: Polyurethane; PVC: Polyvinyl Chloride

rial attached to the microplastic surface, WW density, and the WW motion produced by the system, the MPs might be retained and removed by skimming of fats, oil and grease if they are found in the surface; or in the grit and solids settled at the bottom of the tanks. For example, the density of the raw wastewater is 1.0 g/cm^3, and for the microbial biomass in a biological reactor can vary from ~1.02 to 1.05 g/cm^3, thus the final density of the WW might be altered by the biomass present in the system (Schuler and Jang, 2007; Xu et al., 2014). Furthermore, this parameter may vary according to the stage at which the composition of the WW is being quantified. The density of the WW and its components, in addition to the density of the particle itself, influence the fate of the MPs. Neutrally buoyant MPs are more likely to escape both skimming and settling processes (Carr et al., 2016).

Bayo et al., (2020) reported that ~74% of MPs were retained during the primary stage in a WWTP located in Spain; similar results were observed by Rasmussen et al., (2021) in Denmark. Gies et al. (2018) found that most of the MPs removed in these stages were fibres. The latter was also observed by Hidayaturrahman and Lee (2019) and Blair et al. (2019), as they found that MP fragments are removed in the next treatment stages. Talvitie et al. (2017) found that ~95% of MPs were eliminated by DAF, although MPs concentrations were relatively low. Fibres in the influent are dominant over other MPs, and usually are retained during the primary stage, possibly because these fibres become easily attached to flocs. However, Tadsuwan and Babel (2022) found a large portion of fibres in the effluent from the secondary stage: in this case the WWTP studied did not have a primary sedimentation stage. From the previous examples, it is demonstrated that the performance and availability of the primary stage is a key factor in the removal of MPs from WW.

Biomass accumulation onto MPs surfaces, i.e. biofouling, can increase the density of the particles, causing them to sink (Kaiser et al., 2017). Biofouling might completely encapsulate these MPs and affect their properties along the treatment process, with small MPs the most affected (Carr et al., 2016; Lee and Kim, 2018). As Yu et al., (2016) explained, the solid retention time (SRT) is a crucial parameter that affects biofouling of a MBR as it can modify biomass behaviour. Therefore, it is expected that the SRT has an influence on the MPs, alongside the hydraulic retention time (HRT). It is important to highlight the differences between the SRT and HRT. The SRT indicates the average period the bacteria last in the reactor, whilst the HRT indicates the time the wastewater remains in the treatment tank or unit (Clara et al., 2005; Eggen and Vogelsang, 2015). According to Tadsuwan and Babel, (2022), sedimentation of larger MPs is influenced by longer SRT during the conventional ASP. Further studies are needed to understand more fully how SRT and HRT may modify the characteristics and fate of the MPs in the WWTPs.

Biological treatments, usually a secondary treatment stage, strongly influence the concentration of MPs in a WWTP (Hidayaturrahman and Lee, 2019; Bui et al., 2020). In secondary settlement tanks, MPs may adhere to suspended solids that settle and become trapped in the sludge (Bui et al., 2020). Murphy et al., (2016) and Blair et al., (2019) reported MPs removal efficiencies from the activated sludge process of approximately 93% and 98%, respectively. Magni et al. (2019) reported a concentration decrease of ~50% in MPs from the secondary to the final effluent, contributing to an overall 84% removal efficiency. Ziajahromi et al., (2017) detected in one of three selected WWTPs a decrease from the primary effluent of 66% after the secondary process, which includes secondary aeration and sedimentation. Hidayaturrahman and Lee, (2019) evaluated three different WWTPs and concluded an average removal of MPs after secondary treatment of ~58%.

The combination of different technologies to achieve a better effluent quality, alongside tertiary processes, has been studied. One study has compared two WWTPs with different treatment systems (Lv et al., 2019). The first system employed aerated grit chambers, oxidation ditch, secondary settling tank and a UV disinfection chamber; the second system used rotary grit chambers and a A^{2}O (Anaerobic-Anoxic-Aerobic)-ASP unit combined with a Membrane Bioreactor. The removal efficiency results were as follows: MBR (~84%), >secondary settling tank (~77%) > oxidation ditch (~17%) > A^{2}O unit (15%). It was found that the A^{2}O-ASP MBR-based unit traps MPs in the sludge and removes them via membrane filtration (Lv et al., 2019). According to Peeva and Livingston (2019), the addition of a membrane module within the ASP reactor increases the clarification and quality of the treated water. Although these results showed efficient removal of MPs from WW, to combine different technologies to further decrease MPs concentration needs further attention as outcomes may not be as expected.

Talvitie et al., (2015) concluded that a biologically active filter as part of a tertiary treatment does not decrease MPs concentration. Likewise, Michielssen et al. (2016), by comparing secondary and tertiary WWTPs, and analysing a novel anaerobic MBR pilot-scale added to the tertiary treatment plant, found that the integration of a second process in the secondary stage does not remove more of the of MPs. However, the addition of a MBR outperformed MP removal rates from both the WWTPs studied Michielssen et al., (2016). The secondary plant applied two secondary treatment processes in series, a trickling filter followed by an ASP; and the tertiary stage included slow sand filtration within its process (Michielssen et al., 2016).

Wastewater treatment facilities implementing MBR technology might promote higher removal of MPs from WW. Lares et al., (2018) found that MBR technology is more efficient in removing MPs compared with ASP technology. Talvitie et al., (2017) compared four different tertiary technologies, rapid sand filtration (with removal efficiency of 97%), DAF (~95), disc filter (40-98%), and MBR. They concluded that MBR is the most efficient technology of those studied as they detected a high removal rate of ~99% from MBR grab and composite samples; the MBR had the smallest pore size (0.4 µm) of these three filtration methods. In contrast, Hu et al., (2022) found that for >40 WWTPs in three different regions in Shaanxi Province, China, MBR does not efficiently remove MPs from WW. The removal efficiency of these WWTPs was more related to differences in the characteristics of the MPs (shape and size) across regions.

Within the tertiary stage, some advanced filtration methods have been studied. Mintenig et al., (2017) studied the effluents from 12 WWTPs located in Germany, and one of them was a tertiary treatment plant with an additional post-filtration system installed. They found that the use of a filter unit provides for significant MP removal. In contrast, a study of 17 WWTPs in the U.S.A. concluded that the presence of an advanced filtration phase was not effective in terms of reducing MPs concentrations (Mason et al., 2016). Although a polymeric identification analysis was not performed, it was deduced that the usage of filters might promote microbial growth and discovered that these microorganisms might be misled as “fibres particles”. The efficiency of the disc filter will depend on the pore size of filters, according to Talvitie et al., (2017), who reported a higher removal efficiency from 20 µm filters (~98%) than 10 µm filters (40%). Although the opposite result was expected, they assumed that this outcome might be due to membrane fouling by excessive flocculant polymers adhered to it. Hidayaturrahman and Lee (2019) stated a 79% MPs removal by a disk-shaped filter with 10-µm pore size. Within the granular filtration field, sand filtration is one of the most studied treatments. Slow sand filtration appears not to be the most suitable method for the removal of MPs, according to Wang et al. (2020), who only detected ~37% removal efficiency. These authors also discovered that beads/pellets and fibres were more efficiently retained than fragment-shaped MPs. A study in the U.S.A. involved a gravity-bed filter simulator which consisted of a column made of anthracite, sand and gravel (Carr et al., 2016). Although the filters contained high concentrations of anthracite fragments, the authors confirmed the efficiency of the gravity sand filtration as it successfully retained microbeads from 300µm to 10µm in size.

Hidayaturrahman and Lee (2019), by comparing different tertiary treatment technologies, determined that rapid sand filtration had the lowest removal rate (74%) compared with membrane disc filter (79%) and ozone (90%) technologies. They also concluded that MPs in WW might get trapped between the grains (medium-sized particles) or adhere to the surface of the grains, whilst the treated water is discharged to the bottom (Hidayaturrahman and Lee, 2019). Another study reported a high removal rate by advanced sand filters, however, was not high enough to compete with the efficiency of the MBR technology (Blair et al., 2019). Mason et al. (2016) stated that, for the removal of microbeads, rapid sand filters are not the most recommended technology to use.

In Spain, a WWTP removed ~93% of MPs from WW during a process that includes coagulation-flocculation tanks, lamellar decanter, rapid sand filter and UV disinfection system, according to Menéndez-Manjón et al., (2022). Ziajahromi et al. (2017) detected a 90% MPs removal efficiency during advanced treatment processes, such as flocculation, disinfection, ultrafiltration, and reverse osmosis. Particles from 1000 µm to 500 µm might be fully removed during ultrafiltration system (Tadsuwan and Babel, 2022).

In summary, the composition and concentration of MPs in effluent involves a variety of factors such as the type of wastewater (i.e. presence of combined sewers), loading, and the general treatment processes on site, particularly if tertiary treatment techniques are included (Mason et al., 2016; Gündoğdu et al., 2018; Blair et al., 2019). After the removal of MPs from WW throughout the treatment process, these are concentrated in the sludge.

Figure 6 summarises the stages of the treatment process, the accumulation removal rates of MPs and the factors involved in the MPs removal. However, it is important to highlight the necessity of a consensus regarding the terms used for the sampling points and avoid misunderstandings. For instance, the WW sampled after the bar screening is sometimes referred as the raw WW, when in fact it is the effluent from the screening stage. The first incoming water, the ‘crude, is the raw WW. The use of a to-scale, fully labelled diagram illustrating exactly where the samples were collected is recommended.

4.2 Contribution of WWTPs to microplastic pollution

WWTPs might influence the release of microplastics or physically degrade the plastic particles into smaller pieces. In a WWTP located in Turkey, an increase in PES particles from the influent (62%) to the effluent (~69%) was detected, denoting a PES source within the treatment process (Gündoğdu et al., 2018). However, some authors found similar results but gave alternative explanations. For example, in Italy, Magni et al. (2019) found epoxy resin, PVC and styrene-isoprene-styrene MPs only in the effluent; concentrations of polyacrylate, polyamide (PA) and polyester

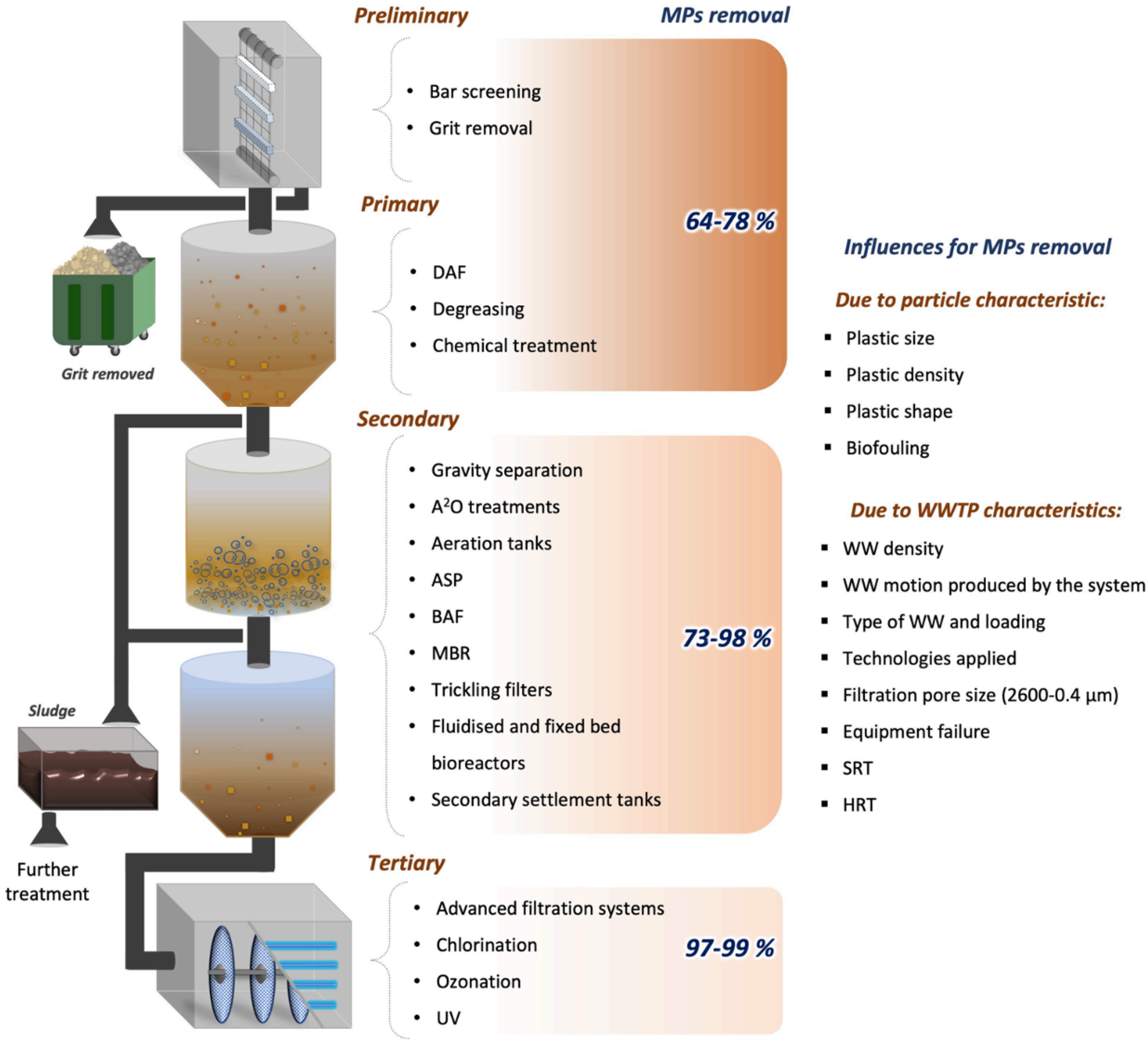

FIGURE 6: Schematic diagram of the processes included in a wastewater treatment plant, alongside the estimated microplastic removal throughout the system and the factors causing such effect.

(PES) particles increased by 7%, 17% and 35%, respectively. These particles may come from paints, adhesive agents and personal care and cosmetics products (PCCPs). Lv et al., (2019) found that the samples from the rotary grit chambers have higher MPs concentration compared with the aerated grit chambers. They assumed that mechanical stirring from the rotary grit chambers further disintegrate the microplastics into more, and smaller fragments. Furthermore, the same authors presumed that air introduction into the aeration systems can influence the MPs to breakdown due to air friction forces, and that the disinfection treatment by UV light, although it enhances MPs removal, it can further decompose the particles to smaller sizes and weakened properties of MPs (Bao et al., 2022).

Within a WWTP, some elements can be a potential direct source of microplastics. The filter medium in trickling filtration is usually made of stone, although they can be replaced by synthetic materials, such as PVC, as it provides a larger surface for growth of microorganisms (Vayenas, 2011). Furthermore, bio-beads, round-shaped particles, sometimes are used as filtering medium in certain treatment facilities, such as for industrial WW (Turner et al., 2019). For the MBR system, Talvitie et al. (2017) reported that MPs might be derived by the occasional breakthrough of the filters, and from the membrane cartridges made of polyvinylidene fluoride (Lv et al. 2019). High numbers of PA particles were detected after filtration from an advanced tertiary treatment system, and it is presumed that they come from the pile fabric itself (Mintenig et al., 2017). The filter materials might vary from PES, PA and PE (Mintenig et al., 2017).

According to Chandrasekaran (2010), rubber is any easy target for ozone, as it can react with the polymer molecules and break the polymer chain, leaving it prone to cracking. However, in a study at a drinking water treatment plant, researchers found that ozonation degrades larger MPs to smaller sizes, which can increase the number of MPs in the effluent (Wang et al., 2020). Fitri et al., (2021) revealed that the ozonation process can impact the chemical structure, in this case of the PE (841 μm in size), influenced by the environment pH, contact time and ozone dosage variation. Li et al., (2022) tested the deterioration of PS plastics by chlorination and ozonation processes, discovering that O_3 is more effective to further breakdown the plastic particles. However, even if the previous study was performed under laboratory-controlled condition using drinking wastewater instead, it can be concluded that disinfection technologies do not eliminate MPs but create smaller particles, including nanoplastics. Further research is needed when it comes to study the disinfection methods in the sewage treatment process with different types and sizes of MPs.

WW treatment facilities can be a source of MPs to effluent, and not only by the purification technologies applied, but also by the degradation of plastic equipment over time (Blair et al., 2019). Therefore, regular maintenance of the treatment facilities might reduce the MPs flow to discharge (Lv et al., 2019). From another point of view, there are other factors that might show an increase of MPs or lead to the detection of new plastic particles from the influent to the effluent, for example, wear and use of synthetics fabrics (e.g. clothing made out PES) when sampling or when experiments are conducted in a laboratory. We emphasise the importance of using blank and control samples to detect any irregularity and correct results. Over- and under-counting the number of particles due to human error (e.g. lack of expertise or fatigue) are other reasons for likely miscalculated values.

4.3 Microplastics in Effluent and Sewage Sludge

The presence of MPs in the final effluent has been reported globally, despite the previous sanitisation process the WW has received. Reported removal efficiencies vary from 73% to 99%, yet a complete removal has not been detected. Information about previous investigations is shown in Table S1 in the supplementary material.

Table S1 also exposes the differences in terms of the sampling points (e.g. raw WW), collection period techniques (grab and composite samples) and duration of sampling campaign, that goes from 1 day to 1 year. Grab sampling consists of one single collection point in a certain time, whilst the composite samples are collected at fixed intervals over an extended period of time. According to Conley et al., (2019), grab sampling is a good technique only for a specific or short period of time, but it may not provide a representative scenario of the wastewater stream. Seasonal variation is a particularly important factor regarding MPs in WWTPs. For example, increases of MPs during warmer periods have been observed, due to either high evaporation of water affecting its dilution, plastic fragmentation due to sunlight radiation or clothing preferences and higher washing frequency (Bayo et al., 2020; Bao et al., 2022; Jiang et al., 2022; Menéndez-Manjón et al., 2022).

Magnusson and Norén (2014), in Sweden, calculated an inflow of 3.25 x10^6 MPs but an outflow of less than 2,000 MPs each per hour based on a single day of sampling, whilst Alavian Petroody et al. (2020), in Iran, reported a total emission of ~ 9 million MPs per day, with a 24-hour composite sampling during a week. A study testing one treatment plant in Spain through a monthly sampling campaign in a year, revealed that ~0.7 MPs/L are released via the effluent (Menéndez-Manjón et al., 2022). However, the minimum size limit they analysed was 100 μm, probably significantly underestimating the total number of MPs in smaller size ranges. The selection criteria to classify MPs in WWTPs according to their sizes must be justified in order to reach an agreement. For example, Lee and Kim (2018) selected 106 μm and 300 μm considering the average value of microzooplankton, plankton and floating waste. Table 2 highlights the different size filters used in the study of MPs in WWTPs.

It is acknowledged that the selection of the different techniques to conduct a study regarding the presence of MPs in WWTPs occurs according to the aims/objectives, limitations and resources available. Nevertheless, the lack of consensus in respect of how the results are reported makes it difficult to compare data across studies. For example, in Thailand, a study testing two WWTPs reported 16.55-77 MPs/L release via the effluent, but in the absence of the outflow data is not possible to modify the units to MPs per day (Tadsuwan and Babel, 2022). Inflow and outflow fluctuations happen hourly and seasonally, therefore, such information must be added to get better quality information about the number of MPs entering the WWTPs and released via the final effluent. Flow variation fluctuates according to the geographical location and the area served, and might determine the pollutant load (Vesilind, 2003). The different sampling techniques are not further discussed in this paper.

The quantity of microplastics are usually published in particles per L/day, however, it can also be calculated as particle mass, as Simon et al., (2018) reported for their final treated outflow, and Rasmussen et al., (2021) for their sampled WW and sludge. The mass measurement can describe the presence of a certain polymer less affected by size ranges and might be more helpful for analytical procedures. However, using particles as a unit might allow the production of information for projects with technological and financial difficulties, as reported by Gimiliani and Izar (2022). We suggest that the number of particles (L/day or kg/day) should be consistently used as units based on the flow rate data from the sampling dates or studies should obtain the annual average flow rate to estimate an annual value.

There are other key factors affecting MPs released to the environment alongside the on-site engineering: the degree of urbanisation surrounding the facilities, economic level, lifestyle and habits of the public, current population served, tourism activities, type of receiving water (e.g. domestic vs. industrial WW), amount of water resources ruled by weather conditions and season, strongly influence the quality and composition of WW (Mason et al., 2016; Murphy et al., 2016; Sutton et al., 2016; Gündoğdu et al., 2018; Lares et al., 2018; Lee and Kim, 2018; Alavian Petroody et al., 2020; Ben-David et al., 2021; Hu et al., 2022; Jiang et al., 2022). In China, even though Hu et al., (2022) did not analyse the type of polymers of the detected MPs, they concluded that the more developed the region, the fewer

TABLE 2: Collection methodology and size categories selected per study.

Number of WWTPs	WW	SS	Type of Sampling	Sampling Duration	Pore filter size (μm)	Reference
3	X		Grab	Monthly, 1 year,	26	Akarsu et al., 2019
1	X		Composite (24 h, every hour)	1 week	500-300-37	Alavian Petroody et al., 2020
1	X	X	Composite (24 h)	2 months, 3 days each	380-180-75-25	Bao et al., 2022
1	X		Grab	5 days within 9 months	2800	Blair et al., 2019
1	X		Composite (24 h)	5 months	5000-1000-355-125	Dyachenko et al., 2017
2	X		Composite (24 h)	5 days	55	Gündoğdu et al., 2018
3	X		Grab	3 days within 2 months	-	Hidayaturrahman & Lee, 2019
3	X	X	Grab	3 months	4750-330	Hongprasith et al., 2020
48	X		Grab	3 days	75	Hu et al., 2022
5	X		Composite (24 h)	2 months	-	Jiang et al., 2022
3	X	X	Grab	2 days within 3 months	300-106	Lee & Kim, 2018
1	X	X	Composite (24 h)	7 days	1000	Lofty et al., 2022
1	X	X	Grab	Once	500-250-125-62.5-25	Lv et al., 2019
1	X	X	Grab	Monthly, 1 year,	500-250-100-20	Menendez-Manjon et al., 2022
2	X		Grab	4 days within 3 months	4750-850-300-106-20	Michielssen et al., 2016
1	X	X	Composite (24 h)	5 days within 5 months	500-20-10	Rasmussen et al., 2021
2	X		Grab	Monthly, 1 year	500-10	Roscher et al., 2022
2	X	X	Grab	1 day	1000-500-50	Tadsuwan & Babel ,2022

The X marks if wastewater (WW) or solid/sludge (SS) samples were collected. The sorting of microplastics by sizes were performed on site or during the laboratory procedures. Information about the WW and SS sampling points is shown in Table S1.

fibres, more foams and more microbeads are found. They concluded that the higher the economic level in the region, alongside the shortage of water resources, a higher number of MPs arrive to the WWTPs. Conversely, Mason et al. (2016) suggested that the number of MP fragments in WW are not associated with the population served by the treatment plant, but there is a direct relationship between the population and the number of fibres. The latter is based on the usage of washing machines (Blair et al., 2019). These fibres might escape into the effluent due to their morphology, which allows them to pass longitudinally through filtration treatments (Ziajahromi et al., 2017).

Alavian Petroody et al., (2020) discovered the presence of microbeads made of PE that are likely to come from PCCPs. This study was in Iran, a country with no current regulations to control or avoid microbeads from PCCPs. Michielssen et al. (2016) found that microbeads were effectively removed during preliminary, primary and the secondary treatment processes. However, some authors did not observe any microbeads in the final effluent but only in the sludge from the 'grit and grease' stage (Magnusson and Norén, 2014; Murphy et al., 2016), or in any other stage (Lv et al., 2019). For tyre particles, it is predicted that ~85% are removed during the primary stage; however, this depends on the load of particles arriving at the WWTP (Sundt et. al., 2014).

The final treated effluent can be used for groundwater recharge and, most commonly, as an indirect water supply through discharge into rivers, lakes and estuaries (Qasim, 2017). It can be reused for irrigation of highways, city parks and farmlands. In some countries treated WW is used for agriculture and forestry irrigation (Table 3) (FAO, 2017). However, the outflows from WWTPs cause concern because of the likely content of microorganisms, nutrients and toxic components, such as MPs, and their consequent effects on the environment. Therefore, the effluent has been studied as a source of MPs to rivers, lakes and oceans.

Sewage sludge is the biosolid fraction generated throughout the WW treatment process. The sludge is further processed in a sewage sludge treatment plant, following these steps: 1) preliminary treatment, 2) primary

TABLE 3: Direct use of treated WW for irrigation purposes.

Country	Irrigated water (x10^{9}m^3/annual)
Australia	0.14
Brazil	0.008
Chile	0.138
China	1.26
Egypt	0.29
Greece	0.069
Iran	0.328
Italy	0.087
Japan	0.0116
Mexico	0.401
Morocco	0.002
Peru	0.114
South Africa	0.006

Source: FAO, 2017

thickening, 3) liquid sludge stabilization, 4) secondary thickening, 5) conditioning, 6) dewatering, 7) final treatment (Kacprzak et al., 2017). This by-product is a mixture of inorganic and organic matter that contains nutrients and helps to improve soil properties and enhance agricultural crops (Singh and Agrawal, 2008; Abdul Khaliq et al., 2017; Fijalkowski et al., 2017). This material is also used for energy recovery purposes from the anaerobic digestion, pyrolysis and gasification processes (Đurđević et al., 2019). Landfill is another option to discard the sludge coming from the treatment plants (Yang et al., 2015).

However, the incineration of dewatered sludge and landfilling are of great concern due to their potential for air and groundwater pollution (Qasim, 2017). Furthermore, as most MPs from WW are partitioned to the sewage sludge, this product might represent a direct source of high concentrations of MPs to soil if it is applied as fertiliser (Fijalkowski et al., 2017; Hurley and Nizzetto, 2018). In Denmark, 70% of dewatered sludge is used for agricultural purposes and the rest is destined for energy recovery purposes (Simon et al., 2018). In North America and Europe, it is estimated that there is an annual input of 63,000–430,000 and 44,000–300,000 tonnes of MPs from the sludge to farmlands, respectively (Nizzetto et al., 2016). South Korea and Switzerland are examples of countries that have banned biosolids usage for agricultural fields, diverting the by-product instead to incineration for energy recovery (Outotec, 2016; Rolsky et al., 2020).

Mahon et al., (2017) analysed sludge samples from anaerobic digestion, thermal drying and lime stabilization processes for seven WWTPs, reporting an average abundance of MPs of 10,000 particles per kg of sludge. They discovered that a lime stabilisation process breaks the MPs into smaller pieces. A study evaluating 12 WWTPs in China concluded that the sludge retained was ~ 23,000 particles per kg dry sludge (Li et al., 2018). Leslie et al., (2017) selected two WWTPs to assess the content of MPs from the influent, effluent and sludge, and they found that the content of MPs in the sludge was ~ 630 particles per kg wet weight. Magni et al. (2019) concluded that 1 tonne dry weight of sludge from an Italian WWTP can transfer ~113 million plastic microparticles to the soil. Nevertheless, the sludge pointed to as a possible source of MPs to soil is sometimes the pre-treated solid that, depending on prevailing legislation, still has to undergo additional treatment to be used in the environment. MPs in this biosolid might be further modified or removed. More studies are needed in the treated sludge used as fertiliser and not from WWTPs directly.

Various reports concurred that fibres are the dominant form of MPs detected in the WWTP and tend to be more fully retained in the sewage sludge (Magnusson and Norén, 2014; Gewert et al., 2017; Ziajahromi et al., 2017; Gies et al., 2018; Lee and Kim, 2018; Corradini et al., 2019; Lares et al., 2019; Yang et al., 2019). Some of the most common polymer found are PES, PP and polyethylene terephthalate, which arise predominately from washing, laundry and industrial processes (Browne et al., 2011; Murphy et al., 2016; Magni et al., 2019). The presence of MPs has become an indicator of previous sewage sludge applications on land (Zubris and Richards, 2005; Corradini et al., 2019). Sometimes, microplastics might never leave the treatment facilities, as they could sink and be retained during the process. For example, Rasmussen et al., (2021) detected that ~ 12% of MPs was missing from the plastic (based on a mass balance calculation), due to sampling and measuring uncertainties. However, these MPs also might get trapped in the digested and activated sludge tanks and remain unaccounted. Many factors are thus involved with the behaviour and release of MPs to either effluent or sludge.

5. CONCLUSIONS

The impact of WW treatment stages and the existing technologies applied, have been critically discussed and reviewed in terms of removal of microplastics. Every facility has a specific design, performance, characteristics, and thus should be assessed accordingly. The removal of MPs from different stages in WWTPs depends upon a range of factors, for example, the STR and HRT, which might interfere with the integrity of the plastic particles. In general, most MPs (up to 78%) are removed in the preliminary and primary stages of WWTPs. The reasons for this are that the particles found in oil and grease on the surface are skimmed out, whilst the ones located in the grit and solids settle at the bottom of the tanks and get removed. The secondary stage is the second most important in terms of MP removal at WWTPs as when the process is complete it eliminates up to 99% of MPs found in the raw WW, with variations in removal rate according to technology. MBR in particular has been shown as an effective system to remove MPs from WW. The combination of different technologies during advanced treatment process, i.e. tertiary stage, has revealed better removal performance, however, it has been suggested that sand filtration technologies alone are the less efficient to eliminate MPs. Additional research to evaluate the function of the systems applied during the tertiary stages is required.

Only a small fraction of the MPs entering a WWTP leave via the final effluent, down to 1% from total in the raw WW, therefore, MPs typically end up being concentrated in the sludge. Their environmental fate depends upon whether the effluent is discharged back into fresh/marine watercourses or used for e.g. irrigation purposes, or they are spread on land, incinerated or landfilled if MPs are found in sludge. MPs may create a great impact in the environment, although their concentration levels of concern are still unclear. The sewage sludge must be treated to be safely used on grassland, therefore, the investigations aimed to the sludge directly from the WW treatment process might not report a proper estimation of the occurrence and appearance of MPs found in the biosolid that is spread in agricultural soil.

Although it is confirmed the presence of MPs in wastewater and sewage sludge, the comparison of data from global studies is complicated as researchers use a variety of means to report the quantity of MPs and to denominate the different WW treatment stages. If mass balance and number of particles are considered, kg/day and L/day are the units we recommend for reporting purposes, according to the flow rate data from the time of the sampling campaign. If MPs are classified based on their size, the size selection criteria must be justified to ease the comparison data to other studies and work on a consensus. We recommend that the incoming water to the WWTPs should be referred as raw WW, crude sewage, or influent, and to avoid calling "influent" the WW that already has started the treatment process, for instance, the bar screening/grit chamber effluent. Furthermore, if the WW of interest is coming from a certain system, the sampling point label should add the term "effluent", not considering the following step. A to-scale, fully labelled diagram illustrating exactly where the samples were collected is advised. Overall, this work aims to provide an insight in the field of MPs and WWTPs together, highlighting some gaps where further investigation is required.

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BIODEGRADATION OF EXPANDED POLYSTYRENE USING *PSEUDOMONAS AERUGINOSA* VITARK5

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ABSTRACT

Polystyrene, a widely used thermoplastic, polluting the environment in the form of micro/nanoplastics. Though traditional methods are commonly employed for plastic management, microbial degradation remains a more promising and eco-friendly approach. The present study focused on the biodegradation of expanded polystyrene (EPS) using microbes isolated from plastic-contaminated sites and assessing the degradants for their industrial importance. An isolate, VITARK5, was able to grow well in Bushnell Haas agar containing EPS as the only carbon source and was chosen for biodegradation studies. With robust glycolipid biosurfactant synthesis and biofilm formation, VITARK5 was identified to be *Pseudomonas aeruginosa*. In order to study biodegradation, VITARK5 was inoculated in Bushnell Haas broth containing thin EPS film and incubated at 37°C and 120 rpm for two weeks. After biodegradation, gravimetric analysis showed a 14.53% weight reduction of EPS film compared to the control. Formation of cracks and rough surfaces was observed on the film under scanning electron microscopy. GCMS analysis showed the presence of industrially important chemicals such as valerenol, 3-hydroxyl, 4-methoxy benzaldehyde, oxalic acid, dodecane, azacyclododecane and hexadecane. FTIR spectroscopy confirmed the presence of functional groups associated with the EPS and its additives. Biofilm formation and biosurfactant production synergistically would have promoted biodegradation. The findings of the present study suggest that the isolate *P. aeruginosa* sp.VITARK5 may be used in bioremediation for polystyrene degradation and valorisation to achieve circular bio-economy.

1. INTRODUCTION

Polystyrene (PS), consists of styrene monomer, are rigid and amorphous thermoplastic polymer containing aromatic rings. It has a cross-linked carbon structure that is highly recalcitrant, and hydrophobic, making it difficult to breakdown (Wang et al. 2024). PS, one of the most commonly used synthetic plastics, is a pertinent packaging and insulation material due to its lightweight and thermo stability (Ho et al. 2018). Expanded polystyrene (thermocol) is used for making food containers and disposable cups that turn out to be single-use plastic wastes contributing to 'white pollution' (Savoldelli et al. 2017). The toxicity and extension of pollution increase with decrease in the size and increase in the concentration of plastic particles produced by several environmental factors. Micro and nano polystyrene particles are more toxic to the aquatic and terrestrial organisms than their parent compounds present in macro polystyrene plastics. Various hazards of micro/nano polystyrene reported include morphological abnormalities, decreased photosynthetic activity and growth inhibition in microalgae; stress, cytotoxicity and histological changes in both aquatic and terrestrial invertebrates; neurotoxicity, genotoxicity, immunotoxicity, developmental toxicity, reproduction toxicity in fish; damage on leaves and roots, alteration in metabolites, changes in chlorophyll content and genotoxicity in plants. Furthermore, additives of PS such as bisphenol A, phthalate plasticizers and brominated flame-retardants are potential endocrine disruptors (Qiao et al. 2022). Furthermore, micro/nano polystyrene enter into food chain of aquatic and terrestrial ecosystem causing biomagnification and affect the public health (Siddiqui et al. 2023).

Several efforts were made to degrade plastics in an eco-friendly method using microorganisms and their associated products such as biofilm, biosurfactants and enzymes. *Pseudomonas* sp. is the most commonly searched bacterium for environmental clean-ups, involved in biodegradation of recalcitrant polymers (Gambarini et al. 2021). *Pseudomonas* sp. AKS2 and *Pseudomonas* sp.

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E4 were reported for degrading polyethylene (PE) using hydrolase and alkane hydroxylase respectively (Wilkes and Aristilde 2017). Polyurethane (PU) was found to be degraded by *P. aeruginosa*, *P. protegens*, *P. cepacia*, and *P. chlororaphis* and *P. fluorescens* (Cregut et al. 2013). There are only few reports on degradation of polypropylene (PP) and polyvinyl chloride (PVC) using *Pseudomonas* sp. because of their high hydrophobicity. However, *P. azotoformans* and *P. stutzeri* could use polypropylene as a sole carbon source after treated with ultraviolet radiation (Arkatkar et al. 2010). Gosh et al. 2013 mentioned *Pseudomonas putida* AJ and *Pseudomonas fluorescens B* – 22 as PVC degrading microbes. Though there are many reports on microbial degradation of PET, *Pseudomonas* sp. was not found to be effective candidates for PET degradation (Wilkes and Aristilde 2017).

Among the petroleum-derived plastics, PS is a popular choice for food packaging industries, despite their biodegradability issues and effect on the environment. However, various research findings showed that PS can be biodegraded by several microbes using their enzymes and biofilms. For instance, *Pseudomonas* sp. isolated from plastic dump yard was able to degrade PS more than 10% after 30 days of incubation (Mohan et al. 2016). Similarly, *Rhodococcus ruber* and *Bacillus paralicheniformis* G1 were able to reduce weight of PS about 0.8% after 56 days and 34% after two months of incubation respectively (Roy and Chakraborty, 2024; Choi et al. 2024). Recently, PS biodegradation by the microbes associated with guts of insects such as *Tenebrio molitor* (Machano et al. 2022) and *Plesiophthalmus davidis* (Woo et al., 2020) is paid more attention. In particular, *Enterobacter hormaechei* LG3, *Klebsiella* sp. WJ2020 and *Cellulosimicrobium* sp. WJ2025 were some potential PS degraders isolated from *Tenebrio molitor* (Lin et al. 2024; Kang et al. 2023). The present study aimed to isolate and identify PS degrading microorganisms from plastic contaminated sites, examine their ability to produce biofilm and biosurfactants that facilitate biodegradation, assess their potential for PS biodegradation, and identify biodegraded metabolites and assess their industrial relevance for valorisation.

1.1 Nomenclature

- FTIR (fourier transform infrared spectroscopy)
- GCMS (gas chromatography mass spectrometry)
- PS (polystyrene)
- PE (polyethylene)
- PU (polyurethane)
- PP (polypropylene)
- PVC (polyvinyl chloride)
- PET (polyethylene trepthalate)
- CV (crystal violet)
- CTAB (cetyl trimethyl ammonium bromide)
- MB (methylene blue)
- THF (tetrahydrofuran)
- PCR (polymerase chain reaction)
- BLAST (basic local alignment search tool)
- NCBI (National Centre of Biotechnology Information)
- MEGA (molecular evolutionary genetics analysis)
- OD (optical density)
- LDPE (low density polyethylene)
- BHET (bis (2-hydroxyethyl) terephthalic acid)

2. MATERIALS AND METHODS

2.1 Polymer sample

Polystyrene samples in the form of Styrofoam (also known as expanded polystyrene or thermocol) used for packaging and insulation were used in this study.

2.2 Chemicals

Agar agar, phosphate buffer saline, crystal violet (CV), K_2HPO_4, KH_2PO_4, $(NH_4)_2SO_4$, $MgSO_4$, cetyl trimethyl ammonium bromide (CTAB), methylene blue (MB), and sodium dodecyl sulphate were obtained from Himedia Laboratories Pvt. Ltd., Mumbai. India. Arginine was obtained Loba Chemie Pvt. Ltd., Mumbai, India. Tetrahydrofuran (THF) was obtained from Avra Synthesis Pvt. Ltd., Hyderabad, India. Triton X-100 was obtained from Sisco Research Laboratories Pvt. Ltd., Mumbai, India. Nutrient broth (peptic digest of animal tissue 5 g/L; sodium chloride 5 g/L; beef extract 1.5 g/L; yeast extract 1.5 g/L) and Bushnell Has medium (K_2HPO_4 1 g/L, KH_2PO_4 1 g/L, $MgSO_4$ 0.2 g/L, $CaCl_2$ 0.02 g/L, NH_4NO_3 1 g/L and $FeCl_3$ 0.05 g/L) were obtained from Hi Media, Mumbai, India. PS agar medium (modified Charnock, 2021) contained two layers. The bottom layer was 50% nutrient strength of nutrient agar. The top layer was PS infused Bushnell Has agar medium. M63 minimal medium (O'Toole, 2011) used was with the composition of K_2HPO_4 7 g/L, KH_2PO_4 3 g/L, $(NH_4)_2SO_4$ 2 g/L, $MgSO_4$ 1 mM and arginine 0.4 g/L. CTAB-MB agar (modified Liu et al. 2013 and Hamzah et al. 2020) was nutrient agar containing 0.2 g/L CTAB and 0.005 g/L MB.

2.3 Sample collection

The soil samples and polystyrene samples that were either partially or completely buried were collected from plastic contaminated area in Villupuram, Tamil Nadu, India. They were collected in sterile sample container, transferred to laboratory within 24 hours and refrigerated at 4°C until further use.

2.4 Enrichment of PS degrading microbes

The collected polystyrene samples were cut and washed gently with distilled water thrice. One gram each of polystyrene and soil sample was added to Erlenmeyer flask (250 mL) containing 100 mL nutrient broth and the flask was incubated at 37°C and 120 rpm for 48 hours. After incubation, the nutrient broth served as the inoculum for the enrichment culture. A volume of 5 mL nutrient broth culture was inoculated into Erlenmeyer flask (250 mL) containing 100 mL Bushnell Haas medium with 1 g of PS samples and the flask was incubated at 37°C and 120 rpm for 14 days. After 14 days, a volume of 5 mL from the enrichment culture was used as inoculum for the successive culture. The procedure was repeated thrice. The culture was observed for turbidity throughout the enrichment period.

2.5 Isolation of PS degrading microbes

2.5.1 PS agar media preparation

The media contain two layers such as nutrient agar layer in the bottom and PS containing agar layer on the top. Nutrient agar of 50% nutrient strength was autoclaved and poured on petri plates covering quarter volume and allowed to solidify. PS solution was prepared by dissolving 0.1 g of PS in 25 mL THF. In 100 mL 1X PBS, 2.5% agar and 15 µL of Triton X-100 was added and autoclaved. Then, it was stirred for 5 to 10 minutes using magnetic stirrer and kept in sonicator wherein PS solution was added into the agar gradually using Pasteur pipette. The agar was poured on previously casted nutrient agar layer and allowed to solidify (Patil and Bagde, 2012 and Charnock, 2021).

2.5.2 Isolation and screening

The culture after triple enrichment was used for isolating PS degrading microbes. An appropriate volume of the culture was serially diluted upto 10^{-6} and spread on the petriplates containing PS agar medium and incubated at 37^{o}C for a week. The plates were observed for growth of microbial colonies. The distinct colonies were subcultured on nutrient agar until pure single colonies were obtained. The isolates were again streaked on the PS agar media, incubated and observed for the duration of microbial growth. An isolate coded as VITARK5 grew well before 48 hours was chosen as the potential isolate.

2.6 Phylogenetic analysis of VITARK5

The isolate VITARK5 was sub-cultured, centrifuged and the pellet obtained was used for DNA isolation using High pure PCR template preparation kit, Roche. A set of universal primers 27F and 1492R were used for PCR and 518F and 800R were used for sequencing. The reaction mixture (25 µL) for PCR contained 0.5 µL (10 µM each) of forward and reverse primers, 0.5 µL template DNA, 2.5 µL of dNTP mix (2 mM) and *taq* DNA polymerase buffer (10x) and 0.2 µL *taq* DNA polymerase enzyme (5 U/µL). PCR thermal cycler was programmed for initial denaturation (94°C for 5 min), cycle denaturation (94°C for 45 s), annealing (55°C for 45 s), extension (72°C for 1 min) followed by 34 cycles and a final extension (72°C for 5 min). The PCR products were resolved onto 1% agarose gel, purified and sequenced. The retrieved sequences were aligned using basic local alignment search tool (BLAST) for similarity search and were submitted to National Centre of Biotechnology Information (NCBI) GenBank database. A phylogenetic tree was constructed using molecular evolutionary genetics analysis (MEGA) software to identify the closely related microorganisms.

2.7 Biosurfactant production

2.7.1 Oil displacement test

It is one of the qualitative tests for detecting the presence of biosurfactants. An overnight culture of VITARK5 in nutrient broth was centrifuged and the supernatant was tested for the presence of biosurfactant. The test was performed on a petri dish containing 20 µL of crude oil forming film on the surface of 20 mL distilled water. A volume of 10 µL supernatant, distilled water and tritonX100 was added on the oil surface of test, negative control and positive control plates respectively. They were observed for formation of zone of oil clearance (Hamzah et al. 2020).

2.7.2 CTAB-MB agar test

It is a semi-quantitative test to identify anionic biosurfactants such as glycolipids (Thavasi et al. 2011). The assay was performed by modifying the medium composition in Liu et al. 2013 and Hamzah et al. 2020. Nutrient agar containing 0.2 g/L CTAB and 0.005 g/L methylene blue was used as the medium for the test. Wells were made in the agar medium into which a volume of 50 µL supernatant, obtained for previous test, was inoculated and incubated at 37°C. The dark blue halo formation around the wells would be observed for anionic biosurfactants (Hamzah et al. 2020).

2.8 Biofilm formation

Microtiter plate method was used to test the biofilm forming ability of the isolate VITARK5. It was grown overnight in nutrient broth and diluted to 1:100 with fresh M63 minimal medium containing arginine. A volume of 100 µl was added to 8 wells (D1 to D8) of 96 well plate and incubated at 37°C overnight. The culture was removed carefully and the wells were washed with phosphate buffer. A volume of 125 µl of a 0.1% CV solution was added and kept at room temperature for 15 min followed by washing the excess stain with phosphate buffer. The plate was turned upside down on tissue paper and allowed to dry overnight. Then, 125 µl of 30% acetic acid solution was added to each well to solubilize the CV and incubated at room temperature for 15 min. All the solutions were transferred to new wells and absorbance (OD_{550}) was measured in a plate reader using 30% acetic acid as a blank (O'Toole, 2011). Based on the average and standard deviation (SD) of OD values of control and test, the cut-off OD (ODc) and final OD values were calculated by the formula given below. The isolate was categorised as weak, moderate and strong for biofilm production by comparing the OD values as shown in Table 1 (Stepanovic et al. 2007).

$$Cut\ off\ value\ (ODc) = (Average\ OD\ of\ control) + (3*SD)$$
$$Final\ OD = Average\ OD\ of\ test - ODc$$

2.9 PS film biodegradation

2.9.1 PS film preparation

PS solution (0.1 g/mL) was prepared by dissolving 5 g of PS sample in 50 mL of THF. PS film was prepared from PS solution. A volume of 10 mL PS solution was poured and casted onto petri plates and kept under fume hood for two days at room temperature. The film thus obtained was cut into appropriate pieces, washed with 75% ethanol, dried with tissue paper and weighed (Patil and Bagde, 2012).

2.9.2 PS film biodegradation assay

Bushnell Has broth supplemented with 0.01% yeast extract was used as the medium for biodegradation

TABLE 1: Biofilm forming ability of VITARK5 based on absorbance.

S. No	OD value	Biofilm production
1.	OD ≤ ODc	Nil
2.	ODc < OD ≤ 2*ODc	Weak
3.	2*ODc < OD ≤ 4*ODc	Moderate
4.	4*ODc < OD	Strong

test. Erlenmeyer flask (250 mL) containing 100 mL of aforementioned medium and pre-weighed PS film inoculated with 1% VITARK5 was used as test whereas uninoculated culture medium served as control. Seed culture of VITARK5 showing OD_{600} range from 0.1 to 0.6 was used for inoculation (Ganesh Kumar et al. 2021). The test and control flasks in triplicates were incubated at 37°C and 120 rpm for two weeks. Plastic films were sampled using sterile tweezers on the last day (Day 14) of incubation in order to assess their biodegradation.

2.10 Assessment of PS film biodegradation

2.10.1 Gravimetric analysis

Plastic films in the test and control culture were washed once with 2% sodium dodecyl sulphate solution to remove bacterial colonies and twice with distilled water on the day of sampling (Khandare et al. 2021). The films were dried gently with tissue paper followed by drying in hot air oven overnight at 50°C. The films were weighed and checked for weight loss. Degradation (%) of plastic samples in the control and test was calculated as weight reduction (%). Biodegradation (%) was calculated by correcting degradation (%) of the test with control. The formulae for weight reduction (%) and biodegradation (%) are given below.

*Weight reduction % = [(Initial weight – Final weight)/ Initial weight]*100*

Biodegradation % = Degradation % of test – Degradation % of control

2.10.2 SEM analysis

The plastic films in the control and test culture were analysed using scanning electron microscope (ZEISS EVO 18). Their surfaces were observed for superficial changes such as roughness and cracks formation due to biodegradation.

2.10.3 FTIR analysis

The chemical modifications of plastic films due to biodegradation were detected by measuring the changes in functional groups on the surface of PS films through FTIR (IRA Shimadzu AVATAR 330, Japan) analysis in ATR mode. OMNIC Software was used for IR spectra analysis and OriginPro was used for constructing graph of the spectral values.

2.10.4 GCMS analysis

The biodegradation products of PS were analysed using Gas Chromatography Mass Spectrometry (GC-MS, Shimadzu, GC-2030, MS-QP2020). The major peaks of the chromatograms were identified using a library search.

2.11 Statistical analysis

MS Excel was used to perform statistical analysis. F-test and Welch's t-test was performed to determine statistical significance between control and treated samples of biofilm formation assay and biodegradation test. A paired t-test was performed to determine stastical difference between the data before and after biodegradation in control and treated samples. A significance level of $p < 0.05$ was considered statistically significant.

3. RESULTS

3.1 Isolation and identification of VITARK5

The triple enrichment culture used for isolation of PS degrading microorganisms was turbid implying microbial growth. On isolation, six colonies showed distinguished morphology on PS agar plates and were named as VITARK1, VITARK2, VITARK3, VITARK4, VITARK5 and VITARK6. The isolate VITARK5 grew well among the other isolates within 48 hours and was chosen for further studies. It was subjected to molecular taxonomic identification and phylogenetic analysis. A BLAST search and phylogenetic tree of 16S rRNA sequence of VITARK5 showed the closest similarity to *Pseudomonas aeruginosa* (Figure 1) and therefore it was designated as *Pseudomonas aeruginosa* sp.VITARK5. The 16S rRNA sequence of the isolate was submitted to the GenBank under the accession number OQ875739.

3.2 Biosurfactant detection and identification

VITARK5 was able to develop a zone of clearance on the oil surface confirming the presence of biosurfactant (Figure 2) and also form a blue halo on CTAB-MB agar suggesting that the biosurfactant produced was anionic and glycolipid in nature (Figure 3).

3.3 Biofilm formation test

The biofilm produced by VITARK5 was stained with crystal violet and OD_{550} was measured (Figure 4). The cut off OD value (ODc) from the control was calculated as 0.044 and final OD value from the test was calculated as 0.202. The calculated values were compared with the standard table (Table 1) and VITARK5 was found to be strong biofilm producer (i.e) 4*ODc < OD.

3.4 PS film biodegradation

3.4.1 Gravimetric analysis

PS films in the control and test cultures were weighed before and after biodegradation. The weight of PS film in control and test culture was 0.167 g and 0.178 g before biodegradation and 0.165 g and 0.150 g after biodegradation respectively. The degradation of PS based on weight reduction was calculated as 1.2% and 15.73% for control and test respectively. The weight reduction in control might be due to leaching out of additives. Therefore, the degradation (%) of the test was corrected with degradation (%) of the control and calculated as 14.53%.

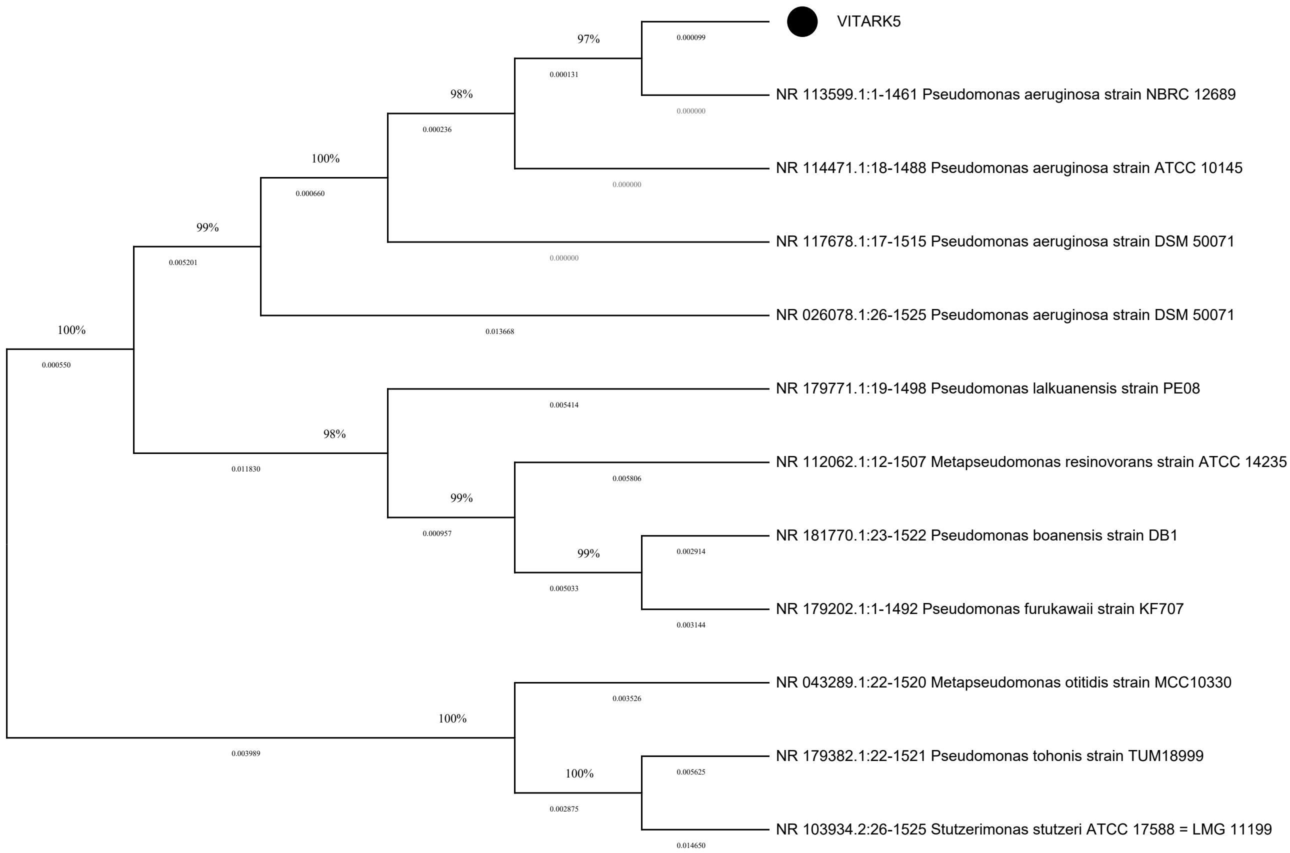

FIGURE 1: Phylogenetic analysis of VITARK5 showing closest relationship with *Pseudomonas aeruginosa*.

3.4.2 SEM analysis

Surface of PS films in the control and test cultures were observed for morphological changes after biodegradation. The surface was observed to be rough and uneven in nature. SEM analysis revealed the crack, pit formation, rupture, and rough surfaces on PS film due to biodegradation by VITARK5 (Figure 5 and Figure 6).

3.4.3 FTIR analysis

FTIR results confirmed the surface changes on the PS film of control and test cultures (Figure 7 and Figure 8). FTIR analysis of PS was well studied by Smith, 2021 and our results were compared with it for identifying significant peaks. The peak at 537.07 cm^{-1} in control and test indicated the presence of halogen compounds that would possibly

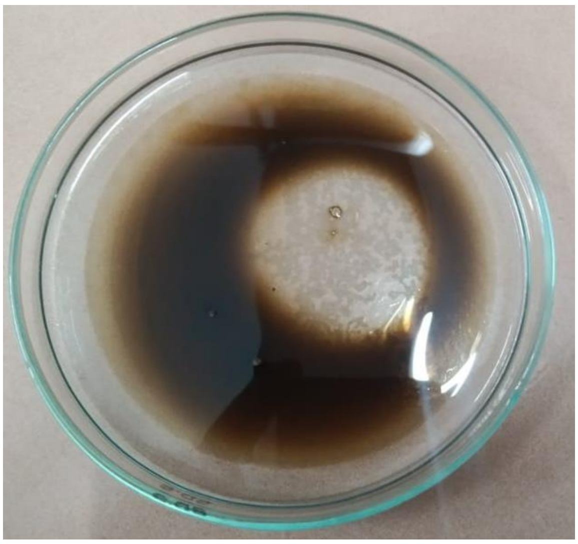

FIGURE 2: Zone of oil clearance by the supernatant of VITARK5 indicating the presence of biosurfactant.

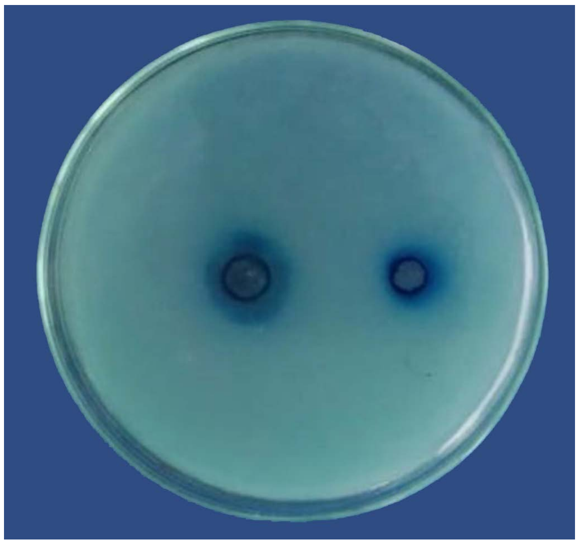

FIGURE 3: Blue halo formation by the supernatant of VITARK5 indicating the presence of glycolipids biosurfactant.

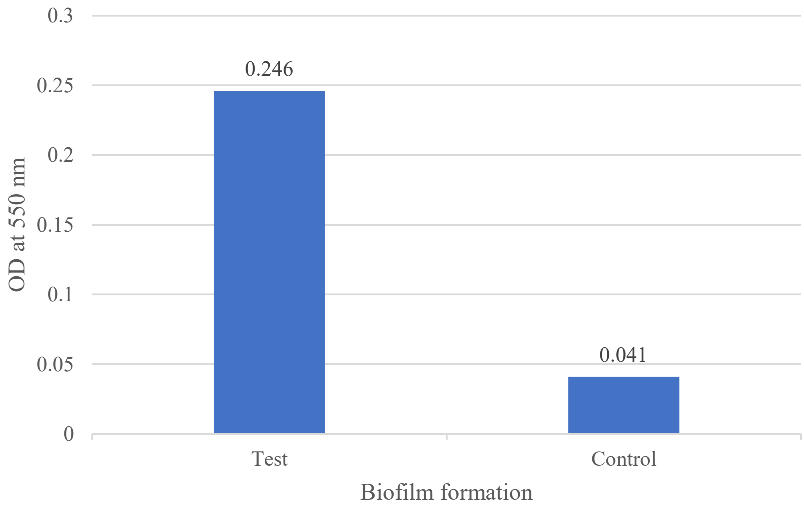

FIGURE 4: A graph showing the biofilm forming ability of VITARK5 compared to control.

be from the presence of brominated hydrocarbons such as hexabromocyclododecane and hexabromocyclohexane used as flame retardants. The absorption peaks ranging from 1620 to 1400 cm^{-1} could identify the ring formation due to C-C stretching in benzene (Smith, 2021). Likewise, peaks at 1451.17, 1492.63 and 1600.62 cm^{-1} were observed in test while at 1452.13 and 1492.63 cm^{-1} in control. The ring bending representing mono-substituted benzene was confirmed by peaks at 696.17 and 695.21 cm^{-1} in control and test respectively. Also, C-H wagging corresponding to the benzene was showed by peak at 751.13 cm^{-1} in both control and test. Furthermore, unsaturated C-H stretching of benzene was observed at 3024.80 cm^{-1} in test. The methylene group in the styrene showed absorption peaks at 2921.9 and 2848.6 cm^{-1} (Fang et al, 2016). Accordingly, there were peaks at 2850.27 and 2923.55 cm^{-1} in test and at 2923.59 cm^{-1} in control. The peaks at 1027.87 and 1069.33 cm^{-1} in test represent the functional group of vinyl ether and primary alcohol respectively. These peaks were seen only in the test implying that they were formed from the biochemical reactions of VITARK5 with PS film.

3.4.4 GCMS analysis

The control and test GCMS spectrum (not shown) was compared to identify the compounds that were degraded, converted and newly formed during biodegradation process. The compounds that were found commonly in test and control such as benzene, oxetane, octadecane, cyclododecane, disiloxane, butenedioic acid, diethyl mercaptal, pentamethyl benzaldehyde and bis(2-ethyl hexyl) phthalate were considered as non-degraded compounds. The compounds that were found only in control such as cyclopentane, 2,5, dimethylbenzophenone, o-cymene, triacontane, eicosane, octane, pentadecanol and furan were considered as degradable or convertible compounds. These compounds would have also leached out from PS film and converted into new compounds. The compounds that were found only in test samples such as benzene propanoyl bromide, dodecane, hexadecane, azacyclododecane, octanal, tridecanal, oxalic acid, valerenol, 3-hydroxyl,4-methoxy benzaldehyde and n-heptyl isocyanate were considered as newly formed or derived compounds.

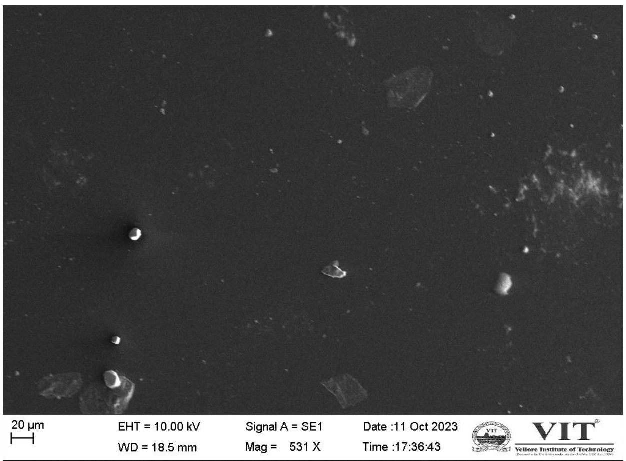

FIGURE 5: SEM image of PS film in control group with no pits or cracks.

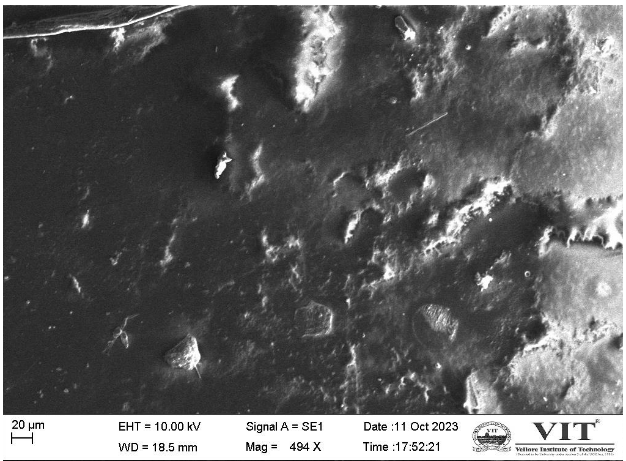

FIGURE 3: SEM image of PS film in test group with rough and cracked surface.

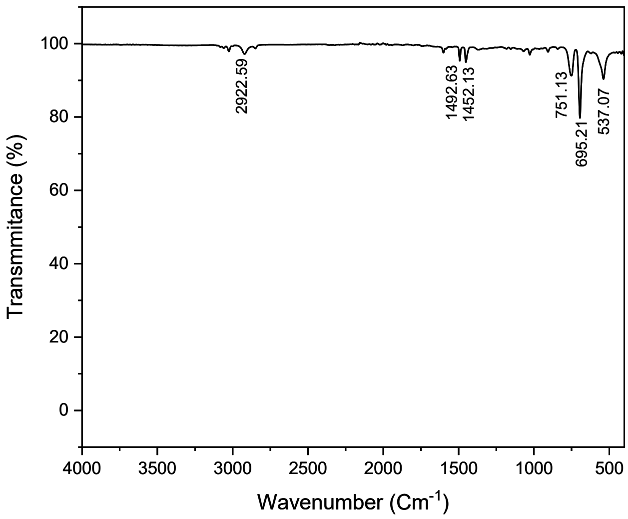

FIGURE 7: FTIR peaks of PS film in control group.

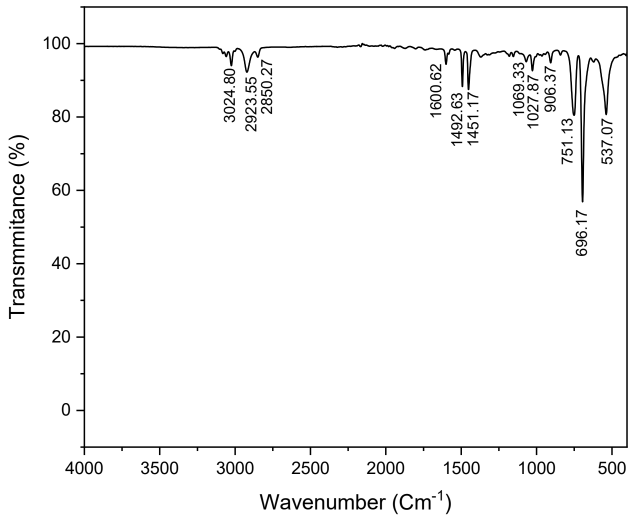

FIGURE 8: FTIR peaks of PS film in test group.

3.5 Statistical analysis

An F-test was performed with the values of control and test groups of biodegradation test and biofilm formation test in order to determine the significance of variance. The observed results showed that unequal variance ($p < 0.05$). The results of Welch's t-test showed that the biodegradation and biofilm formation was statistically significant ($p < 0.05$). The paired t-test was found to be non-significant ($p > 0.05$) for control and significant ($p < 0.05$) for test. These results showed that the degradation in the control was negligible and it was significant in the test.

4. DISCUSSION

Plastic contamination increases the environmental stress by limiting nutrient availability for microbial communities other than plastics. In order to survive, microbes adapt to utilise the plastics as carbon source and thus they are plastic degraders. Therefore, in this study, a plastic contaminated site was chosen for isolating PS degrading microbes. The major challenge in PS biodegradation is the surface properties of PS that hinder microbial interactions. Microbes with the ability to produce biosurfactants, amphiphilic molecules that bridge between hydrophobic and hydrophilic surfaces, and biofilm, an assemblage embedded in an extracellular matrix, are crucial to overcome these hindrances. Therefore, PS degrading isolate VITARK5 was tested for biosurfactant production and biofilm formation.

The inherent hydrophilic character of cell surfaces hinder their interaction with PS necessitating the attachment of hydrophilic functional group on PS surface. Such modifications enable improved adhesion of bacterial colonies and increased accessibility of secreted extracellular enzymes to the polymer surface (Wilkes and Aristilde 2017). These modifications can be carried out by the bio-surfactants that contain polar head attaching to hydrophilic surface while non-polar tail attaching to PS region. Ghosh et al. (2019) reported that biosurfactants secreted by *P. azotoformans* and *P. stutzeri* were able to make hydrophobic polypropylene films into slightly hydrophilic. It is well-known that *Pseudomonas* sp. commonly produce rhamnolipids that are anionic and glycolipid biosurfactants. Noordman and Janssen, (2002) found that rhamnolipids produced by *P. aeruginosa* were able to stimulate assimilation of hydrophobic compounds. Evidently, Taghavi et al. (2022) investigated the effect of rhamnolipid on microbial colonisation and biodegradation of thermoplastics and showed the feasibility of increasing the biodegradation efficiency of PS in the presence of rhamnolipid. Therefore, biosurfactant activity shown by VITARK5 during PS film biodegradation would be because of rhamnolipid production. On the other hand, rhamnolipids were reported for having various role in biofilm development especially microcolony formation (Pamp and Tolker-Nielsen, 2007).

Biofilm is one of the key strategies for the survival of species against unexpected changes of living conditions such as temperature and nutrient availability (Thi et al., 2020). It allows for better microbial and enzymatic interactions through direct contact with the polymer, which in turn, speeds up the biodegradation (McFall et al. 2024, Plakunov et al. 2020). In the study *Pseudomonas aeruginosa* sp.VITARK5 isolated from plastic contaminated site was capable of producing biofilm. *Pseudomonas aeruginosa* is indeed a good biofilm producer and a model organism for biofilm formation (Thi et al., 2020). Thus, VITARK5 could attach on the hydrophobic surface and would have aided in PS biodegradation. Consequently, *Pseudomonas* spp. AKS2 showed increased cell surface hydrophobicity and LDPE biodegradation when they were allowed to form biofilm. It was also found that exopolysaccharides produced by biofilm facilitated microbial adherence to the LDPE surface (Wilkes and Aristilde 2017). It is attested by previous studies on biofilm forming *Pseudomonas* species capable of degrading plastics such as polyethylene, polypropylene and polyvinyl chloride (Ghosh et al. 2019; Giacomucci et al. 2019). Moreover, *P. aeruginosa* employs a complex network of interconnected signal transduction pathways, referred to as quorum sensing which facilitates communication among individual bacterial cells to form self-aggregated biofilm matrix effectively. Beyond the development of biofilms, quorum sensing has been associated with the regulation of various physiological processes essentially include tolerance to stress and adjustments in metabolism (Thi et al., 2020). This in turn makes *P. aeruginosa* to cope up with utilisation of hydrophobic substrate such as plastics for survival. Furthermore, it facilitates befriending other microorganisms of its kind to develop symbiotic consortium that are effective in degrading plastics. For instance, a co-culture of *Bacillus subtilis* MZA-75 and *P. aeruginosa* MZA-85, yielded the highest weight reduction of PU film, in addition to demonstrating the superior esterase activity when compared to the respective strains individually. (Shah et al. 2016). Also, Five distinct strains of *Pseudomonas* and *Bacillus* species as consortium isolated from petroleum contaminated site exhibited synergistic growth utilising PET and bis (2-hydroxyethyl) terephthalic acid (BHET) as their sole carbon sources (Roberts et al. 2020).

PS is generally poly(1-phenylethene-1,2-diyl) meaning that it can be converted to mono-, di- and oligomers of aliphatic or aromatic compounds with the help of enzymes such as oxidoreductases and hydrolases produced by VITARK5. Accordingly, valerenol, 3-hydroxyl,4-methoxy benzaldehyde, Azacyclodecan-2-one and oxalic acid were produced after biodegradation. The major biochemical reactions of reported PS degrading enzymes are PS hydroxylation (Alkane-1 monooxygenase (alkB) and Serine hydrolase), PS depolymerisation by free radicals (Laccase AbiLac1) and hydrolysis of phenyl acetic acid (Arylesterase) (Kim et al. 2024). Alkane hydroxylases and monooxygenases are capable of breaking C-C bonds whereas ring-hydroxylating dioxygenases are capable of breaking PS side chains producing aromatic ring compounds leading to PS depolymerisation (Hou et al. 2021). Choi et al. 2023 reported that styrene monooxygenase, styrene oxide isomerase, phenylacetaldehyde dehydrogenase, and phenylacetyl coenzyme A ligase are some of the key enzymes involved in PS degradation. Though various reports on PS degrading enzymes are available, it is quite difficult to find out the enzymes that degrade expanded PS completely because of its complex structure and composition.

Plastics are not only made up of its respective monomers but also of additives that make them super strong to withstand extreme environmental conditions and resist biodegradation. According to Rijkse and Metsaars 2008, the composition of expanded polystyrene may include but not limited to filler, styrene, blowing agents, flame retardants, nucleating agents, plasticisers, demoulding agents, free-radical polymer initiator, suspension stabiliser, chain-transfer agents and cross-linking agents. Plastics degrading microbes thus face dual challenge of degrading hydrophobic plastic polymers as well as their toxic additives. From GCMS results, it was apparent that VITARK5 degraded PS and additives present in the expanded PS. The degradants produced after biodegradation by VITARK5 were matched with their possible parent compounds for better understanding of PS biodegradation. For instance, butadiene, α-methyl styrene and divinyl benzene are some of the cross-linking agents from which butadienoic acid, pentamethyl benzaldehyde, cymene, dimethyl benzophenone, benzene would have derived. Flame-retardants such as hexabromocyclododecane and hexabromocyclohexane would have broken down to form cyclodecane and benzene propanoyl bromide. Mineral oil and paraffin waxes are used as plasticisers that might be responsible for production of several alkane compounds include octadecane, octane, triacontane, eicosane, hexadecane and oxetane. However, some of the compounds produced such as oxalic acids were the by-products of central metabolic pathway and found to enhance the biodegradation. For instance, oxalic acid produced by P. aeruginosa NY3 during n-hexadecane biodegradation was found to increase the degradation efficiency by acting as promoter (Nie et al. 2017).

Pseudomonas aeruginosa VITARK5 could not only degrade PS but also produce biosurfactants, showing that PS can be exploited as a substrate for biosurfactant synthesis. The PS biodegraded products such as hexadecane, dodecane, 3-hydroxyl,4-methoxy benzaldehyde and oxalic acid find their applications in fuel, pharmaceutical, textile and food sectors. For instance, oxalic acid was used for metal removal (Shathi et al. 2024) and for postharvest quality preservation of fresh fruit and vegetables (Hasan et al. 2023). Therefore, *P. aeruginosa* VITARK5 can be used for PS biodegradation and bio-valorisation that not only mitigates PS waste but also useful for producing economically significant products. However, the entire potential of this strategy will depend on future developments in biotechnology and sustainability regulations.

5. CONCLUSIONS

This study suggests that plastic contaminated soil samples can be used to isolate potential plastic degrading microorganisms. *Pseudomonas aeruginosa* sp.VITARK5 is a potential PS degrading microbe that produces glycolipid biosurfactants and strong biofilm facilitating microbial adherence and colonisation on PS surface. Thus, VITARK5 degrade PS with the help of biofilm, biosurfactant and enzymes. Further research on medium optimisation for biofilm and biosurfactant production using PS as substrate would enhance biodegradation. The degradants of biodegradation were found to be commercially important and therefore biovalorisation can be achieved.

Though various reports are available on microbial degradation PS, most of the reported microbes such as *Bacillus* sp. and *Pseudomonas* sp. are pathogenic. It obstructs their direct application on contaminated environment due to horizontal gene transfer of multi-drug resistance and other toxic traits. Therefore, PS needs to be segregated from contaminated area and treated under controlled environment using these microbes. However, segregation of plastics is a cumbersome and long-drawn-out process. Development of sensors to detect plastics would make the process easier. Though microbial degradation produce less toxic metabolites, their slow degradation rate necessitate effective approaches that enhance rate of the reaction. Tandem approaches like combining biological degradation with physical or chemical pretreatment methods would enhance the degradation and can also be used for production of value added products. For istance, PS is converted to polyhydroxyalkanoate (PHA) by *Pseudomonas putida* CA-3 using a hybrid chemo-biological method (Ward et al., 2006). Savoldelli et al. (2017) used thermal degradation followed by biological degradation for the production of useful by-products such as benzene and naphthalene from PS using different microbes among which *P. putida* was found to be more productive. Furthermore, various tools and techniques of biotechnology such as genetic engineering, metabolic engineering, protein engineering and multi-omics would not only help in the discovery and engineering of many PS degrading microbes and enzymes but also facilitate optimisation of bioprocesses required for valorisation. Future research directions of the present work include increasing the degradation efficiency of *Pseudomonas aeruginosa* sp. VITARK5 by genetic modifications and medium optimisation, identification and characterisation of key enzymes involved in PS biodegradation for enzyme-mediated PS bioremediation, and scaling up the experiments for assessing the feasibility and integration of PS biodegradation for real time applications.

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Environmental Forensic

LEVERAGING ENVIRONMENTAL FORENSIC EXPERTISE FOR EFFICIENT POLLUTION LIABILITY ALLOCATION

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1. INTRODUCTION

The pollution of a site necessitates investigations to identify the polluters responsible and a fair liability allocation process that makes the polluters pay for the damage they have caused to the environment. The role of an environmental forensic expert in the investigative phase – determining the source of pollution and assigning responsibility – is well established. However, the possibility of involving them in the liability allocation process that relies on investigation findings remains largely unexplored. This situation perhaps stems from the perceived risks involved in such a practice, where the investigating agency itself handles liability allocation. One major concern is the potential for a conflict of interest. If the forensic expert(s) have a stake in the outcome, they may be tempted to protect their reputation or shield certain individuals from blame, leading to biased conclusions. These situations can also result in a lack of objectivity, where investigators unconsciously or deliberately favor specific parties due to prior relationships or other influences. Moreover, public trust in the investigation process may erode if the experts are seen as biased or self-serving. A lack of external oversight can also lead to cover-ups, where the investigators downplay their own mistakes or those of related entities to avoid accountability. Such practices can create legal challenges, as affected parties may dispute the findings in court, leading to prolonged legal battles and potential invalidation of the investigation's conclusions. In addition to acceptability and integrity issues, the experts may lack the necessary expertise – mainly the legal – to fairly allocate liability, leading to errors and inefficiencies in the process.

In spite of these risks, there are many advantages that prompt one to explore how the expertise of a forensic scientist can be used for liability allocation. One of the main advantages of allowing the experts to allocate liability is efficiency and speed. Since the investigator already possesses relevant evidence and knowledge of the case, it can make quicker decisions without the delays that often arise when transferring responsibility to another entity. The specialized expertise in investigations ensures a more accurate and informed approach to liability allocation. This method is also cost-effective, as it reduces the need for external consultants or legal bodies, saving both time and resources.

Furthermore, consistency in findings is another significant benefit. When the same experts investigate and assign liability, there is a streamlined approach, minimizing discrepancies or conflicting conclusions that might arise from involving multiple entities. The experts' comprehensive understanding of the case reduces the chances of misinterpretation, ensuring a more contextually informed decision. In addition, keeping the process within one agency eliminates bureaucratic delays that could arise from transferring liability allocation to a different organization. Lastly, direct accountability can serve as a positive factor—when an agency is responsible for both investigation and liability allocation, it may be more committed to ensuring a thorough and fair process. However, leveraging these advantages effectively poses a challenge, as it must be done without compromising the integrity of the liability allocation process. Here, we suggest ways to get the task accomplished.

2. LIABILITY ALLOCATION AS A TWO-STAGE PROCESS TO MAKE USE OF FORENSIC EXPERTISE

Along with the integrity issues, a major challenge in forensic experts undertaking liability allocation is their possible lack of legal expertise required in the process. Yet another key issue is the inability to ensure consistency in the allocation process due to a lack of standard procedures. Priya et al. (2023[a]) had considered these issues and came up with a two-stage liability allocation process to address them.

Priya et al. (2020), after analyzing various court cases, research literature, and statutory documents on pollution liability allocation, identified a set of factors relevant to determining liability. These factors are pollutant characteristics, its volume, toxicity, environmental impact, the migratory potential of pollutants, the extent of approved deviations from standards, each party's level of culpability, the degree to which a party benefited from disposal, financial capa-

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bility, period of occupancy, scale of operations, economic benefits, cooperation with remediation agencies, knowledge of contamination, strength of evidence, agreements between parties, mindset, and economic status. After identifying these factors, they categorized them as technical and non-technical. This classification aimed to facilitate a two-stage liability allocation process: the first stage of technical liability allocation, followed by the final stage of legal liability allocation, which incorporated additional non-technical factors relevant to the case (Figure 1).

To ensure an objective distinction between technical liability and legal liability, they provided formal definitions for these. Technical liability was defined as 'the liability that is proportional to the quantified share of each party in those attributes of pollution/pollutant(s) which can be measured using scientific techniques'. Legal liability, on the other hand, was defined as 'the liability of each party for pollution as decided by an authority authorized to carry out such responsibility allocation in the obeisance of any rules and regulations relevant to the case and applicable to the jurisdiction where the decision is made'. Technical liability is determined solely based on technical factors.

To make the distinction between technical liability and legal liability clear, consider a case where forensic investigations reveal that parties A and B contributed to contamination at a site with pollutant P. Technical liability allocation would involve distributing remediation liability in proportion to each party's quantified share of P. This task is best performed by a forensic expert, who possesses the technical expertise needed for accurate assessment. However, if a contract exists between A and B – one that A claims absolves it of liability but is disputed by B – technical liability allocation alone would not be sufficient to finalize the process. In such cases, a judicial body or other authorized entity must evaluate the validity and applicability of the agreement. Nevertheless, the forensic expert's technical liability assessment would serve as the foundation for the final allocation.

The technical factors suggested to be used for allocating the clean-up liability are:

1. **Spread of chemicals in the environmental medium**: The chemicals get dispersed in the environmental medium once it is released. The more the spread of chemicals in the medium, the more will be the difficulty in remediating the site. Mobility of chemicals were used as a determinant for liability allocation by U.S courts (Graves et al. 2000). Duration of discharge and the time of release also affects the spread of contaminants in the medium. Direct quantification of the spread of contaminants can be done by calculating the volume of the contaminated medium. Indirect methods of quantification include using different coefficients like octanol-water partition coefficient (K_{ow}), the soil absorption coefficient (K_{oc}), etc.
2. **Quantity of pollutants**: Quantity of chemicals was used as a principal criteria for liability allocation by many countries of the world. Other things being similar, remediation cost is generally proportional to the quantity of pollutants. In cases where the volume of contaminated medium is the same, the cost for clean-up would differ based on the quantity of chemicals. Different environmental forensic techniques, like chemical fingerprinting, transport modeling, etc., can be used for determining the quantity of pollutants.
3. **Remediability of chemicals**: The term refers to the ease with which a contaminant can be separated from an environmental medium as a part of remediation efforts. The remediability of chemicals decides the remediation expenses. Remediability of chemicals depends on the properties of chemicals as well as the contaminated medium. Remediability of chemicals also decides the remediation technology that can be adopted at a site for clean-up. The chemicals released combine with the environmental medium to form a matrix. The stronger the matrix, the more difficult the remediation.
   Priya et al. (2023[b]) suggested using 'Remediability Score (RS)' for quantifying remediability. RS is determined on a scale of 0-100. The lower the RS, the greater will be the remediability of the site. The factors determining RS were decided based on the factors that the United States Environmental Protection Agency (USEPA) uses for soil screening and groundwater remediation technologies (FRTR 2025). These factors are:
   - <u>Complexity of technology</u>: The complexity of the technology influences the selection of remediation technology. When many technologies are available for a site, the one with lesser complexity is always preferred.
   - <u>Resource requirement of the technology</u>: When two technologies are found suitable for a site, the one that requires fewer resources and efforts will be selected for remediation.
   - <u>Stage of development in the technology</u>: Remediation technologies with above-average developmental status are always preferred for remediation.
   - <u>Confidence in the technology</u>: When multiple remediation technologies are available for a site, the technologies that are most reliable are considered for the remediation of the site.
   - <u>Efficiency of the technology in terms of contaminant reduction in the environmental matrix</u>: The technology that reduces the contamination level to

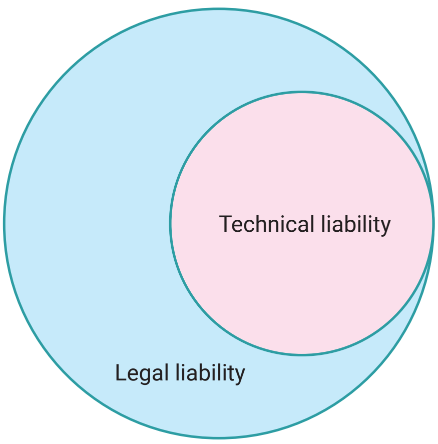

FIGURE 1: Technical vs. legal liability of pollution.

meet the regulatory objectives is always preferred over other technologies.

- Efficiency of the technology in terms of long-term effectiveness: The long-term effectiveness of the technology is considered while selecting the most suitable technology.
- Disturbance caused to the soil: The technology which causes the least disturbance to soil is selected as the most suitable technology, mainly in cases when the site is undergoing any development.
- Time requirement: The technology which requires less time is always preferred over other technologies. Contaminated sites need to be remediated quickly to avoid further spreading of contaminants.

4. **Persistence of chemicals**: The duration for which a chemical remains in the medium is given by persistence. Persistence of chemicals is expressed in terms of the half-life of chemicals. The higher the persistence of the chemicals, the higher will be the costs associated with clean-up. The half-lives of chemicals are available in different databases like National Pesticide Information Centre, European Chemicals Agency (ECHA), etc.

Since technical factors are independent of the specific case, standardized guidelines for technical liability allocation can be developed. Existing guidelines, such as those provided by INTERPOL, focus on investigative procedures and the enforcement aspects of environmental crimes (INTERPOL, 2014). However, they do not offer guidance on determining liability, as legal liability falls under the jurisdiction of individual countries' legal systems. Separating technical liability from legal liability would allow organizations like INTERPOL to establish guidelines specifically for technical liability allocation.

Priya et al. (2023[a]) have proposed an objective procedure for technical liability allocation based on the factors presented above and demonstrated its application with an example. Even if the suggested factors require refinement, their proposal provides a foundational model for technical liability allocation guidelines and presents a viable approach to integrating forensic expertise into pollution liability allocation.

3. ENSURING INTEGRITY OF THE ALLOCATION PROCESS

Ensuring integrity in pollution liability allocation is as crucial as in any other liability allocation process. A reliable and transparent system prevents wrongful attribution of liability and ensures that responsibility is assigned accurately and equitably. It also aligns with legal and regulatory requirements, reducing the risk of disputes, appeals, or reversals. Maintaining integrity enhances credibility and trust among stakeholders, including the involved parties and the general public. Additionally, it minimizes the risk of bias, manipulation, or undue influence from personal, corporate, or political interests. Proper liability allocation ensures that remediation costs are fairly distributed, promoting responsible environmental practices and preventing long-term economic and ecological harm.

To ensure fairness in the allocation process, liability allocation (the technical liability part) by investigating forensic expert(s) should be permitted only when the investigation is done by independent forensic experts. These experts must operate without any obligation to the parties involved in the case, ensuring impartiality in their assessments. However, to maintain the integrity of the process, the cost of such investigations should be covered by a public fund. This fund can later be recuperated through contributions from the guilty parties, with a portion of the damages collected being allocated to replenish the fund. This approach guarantees that financial constraints do not hinder an impartial investigation, and liability is assigned based on scientific evidence rather than the financial capacity of the parties involved. For example, in the United States, the EPA's National Enforcement Investigations Center (NEIC) conducts federally funded environmental forensic analyses to support enforcement actions. When violations are identified, the EPA may seek to recover the costs of these investigations from the responsible parties, including companies found to be non-compliant with environmental laws. Involving such independent agencies in liability allocation improves efficiency while preserving the integrity of the process.

In cases where forensic experts are hired by the disputing parties, they should not be involved in liability allocation. Instead, another independent authority – similar to the National Green Tribunal (NGT) of India, which comprises both forensic and legal experts – should be responsible for allocating liability. The advantage of this approach is that the cost of the investigation is borne by the involved parties rather than relying on public funds, even if for a limited period. However, this method presents the risk that affluent parties may engage highly paid experts to manipulate the technical evidence and escape accountability. To counter this, it is essential to have a competent panel of forensic experts within the allocating authority. A well-qualified and impartial team can ensure that scientific and legal evaluations are conducted with the highest level of accuracy and fairness, preventing financial disparities from influencing the outcome. Table 1 summarizes the options for involving environmental forensic experts in pollution liability allocation.

In the era of artificial intelligence, advanced forensic techniques like machine learning models can be used to reduce the influence of human bias and enhance integrity. For instance, AI-assisted satellite imagery tools were used independently to verify the destruction in the Amazon forest, where the corporate companies had provided misinformation. AI-driven validation is key to preventing bias in environmental liability cases.

4. CONCLUDING REMARKS

Involving environmental forensic experts in pollution liability allocation can enhance the efficiency and accuracy of the process. However, challenges such as their limited legal knowledge, the absence of objective guidelines, and potential integrity concerns must be addressed. A structured, two-stage approach – beginning with technical liability allocation by independent forensic experts, followed

TABLE 1: Options of involving forensic experts in liability allocation.

Case	Determinants of liability	Liability allocation	Role of forensic experts
Independent forensic expert(s) carry out investigation at the contaminated site and carry out liability allocation based on the criteria for technical liability allocation	No factors other than technical	Complete	Investigation and liability allocation as independent experts
	No factors other than technical, but party/parties challenge investigation/allocation	Liability allocation by judicial authority, taking inputs from the independent forensic experts	
	Presence of decisive non-technical factors		
Forensic experts hired by parties carry out investigations at site and investigation results are presented by experts before allocating authority	Technical factors and/or non-technical factors	Authority consisting of independent forensic experts and legal experts carry out allocation based on the investigation results/additional details sought/collected	Different groups of forensic experts involved at investigation stage and allocation stage

by final allocation considering legal and non-technical factors – can provide a balanced solution. Establishing clear, standardized guidelines for technical liability allocation will further ensure consistency and reliability. By adopting this approach, forensic expertise can be effectively leveraged while maintaining fairness and transparency in pollution liability allocation.

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DETRITUS & ART / A personal point of view on Environment and Art

by Rainer Stegmann

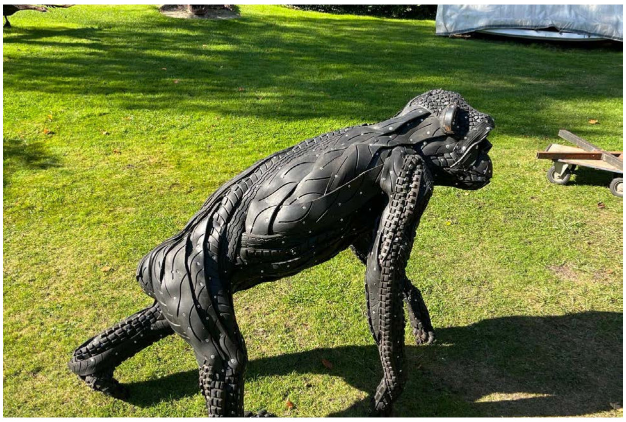
An Afternoon in Varel, Germany

I reported on my DETRITUS Art Page in the edition March/2023 of the fantastic art that Sayaka Ganz creates using daily used plastic items. Sayaka was born in Japan and now lives in the USA. Last summer, she told me she would be staying with two other artists at the beautiful large garden property of Diedel Klöver in Varel, a small city northwest of Germany. Diedel Klöver – the fourth artist – hosted the group. They came together because each artist created an artwork and presented it in the final exhibition in this garden to the public. The exhibition theme was "The Guardians of Sustainability," the guardians were not humans but – very meaningful – four different animals.

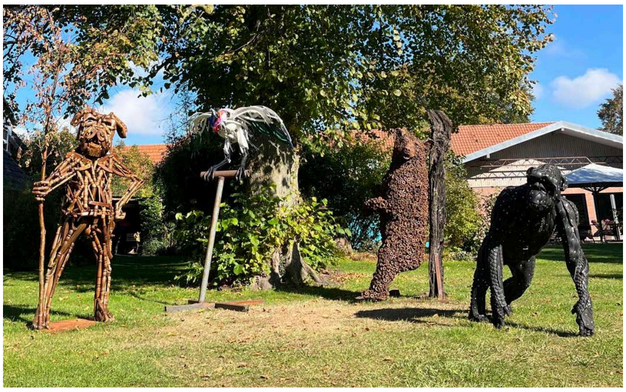
This was an interesting group with Luigi Stinga from Teneriffe, Spain, who used the wooden legs of discarded chairs to create a human body with a dog head holding a more extended branch. The other guardian is a monkey with fur made of rubber of larger rubber pieces from used tires. It was created by Ernest Nkwocha from Nigeria. Diedel Klöver created a big brown bear leaning on a tree. He used a large quantity of rusted iron nuts provided by a local company. The last guardian in this group was an American vulture created by Sayaka Ganz using plastic items like used plastic toys, hangars, plastic dishes and others. All sculptures show expression and movement; they look lively and showed determination to fight for sustainability.

The artists warmly welcomed me, and I had a great chance to gain insight into their work. I witnessed the kinds of materials they use and how they process them to create these incredible sculptures. They had breaks and meals together, and they had a lot of fun. They lived under straightforward conditions and didn't do it for money. Maybe they had sponsors who financed their travel. This situation impressed me a lot because they are devoted to their work. It made me pensive because, in our modern world, many people may have other preferences that are more materialistic. The artists delight us with their outstanding art, clear message of more sustainability, and their ability to make us think about whether our lives are always on the right track. I may return to these artists in more detail in future DETRITUS editions.

Vulture created by Sayaka Ganz.



Gorilla by Ernest Nkwocha.



The Guardians of Sustainability.

In the next edition I will present photos reflecting the art in the transience of nature: fleeting beauty during a perfect circular economy.

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