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CHEMICAL RECYCLING OF POLYVINYL CHLORIDE CONTAINING PLASTIC WASTE FOR LIGHT-EMITTING DIODES RECYCLING

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ABSTRACT
The recycling of critical metals using chemical recycling of polyvinyl chloride containing plastic waste is a promising way to solve two important problems in waste management. The first problem is the disposal of polyvinyl chloride. The second problem is the recovery of critical metals. This is being transferred to demonstration scale as part of the "CHM-Technologie" project. Different polyvinyl chloride containing plastic wastes were pyrolyzed. This hydrochloric acid rich vapor is used in a second step to chlorinate indium in light-emitting diodes. Indium chloride has a lower boiling and vaporizes. This is cooled and, together with the decomposed volatile substances in the light-emitting diodes, produces a metal concentrate. The products are solid residues, metal concentrate and gas, which can ultimately be used for other purposes. For metal purification, the liquid metal concentrate is filtered, mixed with water, distilled and electrolyzed to obtain metallic indium qualitatively.

1. INTRODUCTION

Plastic products are used extensively in various fields for building and construction, packaging, agriculture, and many other applications, due to their low price, light weight, long life and mouldability (Geyer et al., 2017). Plastic production has continuously increased in the last decade. The annual production exceeded 330 million metric tons (Mt) from 2016 to 2020. Polyvinyl chloride (PVC) accounted for 9.6% of all plastics in 2020, ranking in third place. There are different methods to dispose plastic waste (Geyer et al., 2017; Geyer, 2020). First, incineration decreases the volume of waste, and the resulting energy can be used for heating and/or generating electricity. Nevertheless, incineration of plastic waste emits excessive CO_2 and may release some hazardous substances, such as polychlorinated biphenyls, dioxins, furans, and persistent organic pollutants (POPs) (Shibamoto et al., 2007).

In some countries, such as Germany or Austria, the landfilling of Total Organic Carbon (TOC)-containing waste is prohibited (Bundesministerium der Justiz und für Verbraucherschutz, 2009). In Germany, of the total 861,000 metric tons of PVC waste in 2021, 1% is still disposed of or landfilled, 57% is used for energy recovery, and 42% is mechanically recycled (Conversio Market & Strategy GmbH, 2022).Second, plastic waste can be dumped in sanitary landfills, which is a convenient and cost-effective, but landfill space is limited in many countries and an inadequate landfill can cause serious environmental pollution (Hopewell et al., 2009; Teuten et al., 2009; Wang et al., 2021). Third, mechanical recycling is suitable for various types of plastic waste and results in lower value materials (Braun, 2002). A fourth method called "chemical recycling" summarized the development of alternative techniques for plastics recycling like pyrolysis, gasification and liquefaction (Quicker et al., 2022; Scheirs & Kaminsky, 2006). For PVC-containing plastic waste gasification and pyrolysis are mainly in the focus of research. For example, the company Ecoloop in Germany in Goslar produces synthesis gas from PVC-containing plastic waste, but the chlorine is absorbed with calcium oxide, where calcium chlorine is generated (VinylPlus, 2015). Here hydrogen chloride or chlorine can`t be used for further processes. Therefore, pyrolysis of PVC can be used and the techniques like two-stage pyrolysis, co-pyrolysis or ad-/absorption of halogens in the vapor phase were tested for different purposes like removing chlorine in oil from mixed plastic waste (Bockhorn et al., 1998; Liu et al., 2013; Muhammad et al., 2015; Shen et al., 2016).

Electronic products are another essential part of modern society, but their importance has perhaps never been as palpable as when the COVID-19 pandemic forced almost every aspect of human interaction to go online. How-

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ever, the pandemic also revealed that the supply chains that provide crucial raw materials for manufacturing electronics are increasingly vulnerable to social, geopolitical, and technical disruptions. These vulnerabilities are likely to escalate in the future, due to global health crises, natural disasters, and global political instability, all of which will be magnified by looming climate change impacts almost 40 metals and minerals that provide critical functionality to electronic products (Althaf & Babbitt, 2021). A lot of these metals used in electronic products, especially specialty metals like rare earth elements, gallium or indium have End-of-Life-recycling rates (EoL-RR) of less than 1% (Graedel et al., 2011). In the EU, total indium consumption is estimated at 64 tonnes per year from 2012 to 2016, with 60% of the total indium used in flat panel displays (FPDs), mainly in the form of indium tin oxide (ITO). In addition, 11% and 9% of the consumption is used in solders and photovoltaics, respectively, while the remaining 20% is used in thermal interface materials, batteries, alloys/compounds, and semiconductors & light emitting diodes (LED) (European Commission, 2020). In Germany there are six different collection groups for electronic devices, where most electronic devices with FPDs are collected in collection group 2, LEDs in collection group 3 and photovoltaics in collection group 6 for further treatment (UBA 2019). LEDs are recycled in integrated copper smelters (Adams, 2016). Indium is not recycled in these integrated copper smelters and is lost due to its currently low value (Reuter et al., 2019).

Pyrolysis of PVC was tested to use the generated hydrogen chloride for the recovery of indium from indium-tin-oxide powder and LCD-panels (Park et al., 2009). At the ForCycle II project "Chlorplattform" the utilization of hydrogen chloride from PVC-waste for the recovery of indium from FPDs was successfully tested at small scale. For the recovery of indium from LED bar lights, only an indium enriched metal concentrate was generated. First ecological and economical estimations have shown that CO_2 emissions could be reduced by this new profitable method (Berninger & Peer, 2022). This was inspiring to do further research with PVC-containing plastic waste for the recovery of indium from LED at a bigger scale in the project "CHM-Technologie".

2. MATERIALS AND METHODS

2.1 PVC-containing plastic waste and LED

PVC plastic mixture, a sorting residue from commercial waste, which is similar to the yellow bag in Germany was used from a sorting plant. The PVC-containing plastic waste was sampled for analysis and the rest was pelletized. PVC plastic mixture contains 1.3 wt.-% chlorine and has a calorific value of 25.56 MJ/kg. Industrial PVC containing waste (short: PVC waste from industry) from a manufacturing facility in Germany was used. The PVC waste from industry was sampled for analysis and the rest was pelletized. PVC waste from industry contains 2.9 wt.-% chlorine and has a calorific value of 24.33 MJ/kg. EoL – LED bar lights with a length of 0.8 m or 1.2 m in mixture were shredded to <20 mm for the experiments. A sample from this was analyzed. It contains 1 ppm of indium and the calorific value is 13.52 MJ/kg.

2.2 Energy Dispersive X-Ray Fluorescence Spectroscopy

The element content in the input materials and products was analyzed using the Epsilon 3XLE X-ray spectrometer from PANalytical Kassel. The device was calibrated using the "omnian" standard from PANalytical Kassel and the data was evaluated using the Epsilon Benchtop software from the same manufacturer. The solid samples were crushed to <1 mm for analysis using the Retsch cutting mill at 600 rpm.

2.3 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

ICP measurements were performed using Spectro Arcos ICP-OES in dual side on plasma mode. ICP was equipped with cross flow nebulizer, Scott spray chamber and fixed glass torch with 1.8 mm injector. Measurements conditions were RF power 1350 W, plasma gas flow 14 l/min, nebulizer flow 0.75 l/min and auxiliary gas flow 1.3 l/min. Sample aspiration rate was set to 2.0 ml/min. The wavelength for Cl was 134.724 nm and regression coefficient (r2) of calibration curve was 0.9995.

2.4 Experimental set-up

For each pyrolysis test, the corresponding feed material was placed in the system's collection container. Before starting the pyrolysis treatment, a pressure test was carried out in a cold state to check whether the system was tight. Compressed nitrogen was used to pressurize the system to a level higher than the expected overpressure during the test. The test was not started until the system maintained the overpressure and it could therefore be assumed that there were no leaks in the pyrolysis plant combined with the chlorination reactor (Figure 1).

The system was then flushed with nitrogen (28 l/min) to create an inert atmosphere. Finally, the reaction chamber was heated to the test temperature using an electric jacket heater. The material was then conveyed via a screw conveyor into the heated screw reactor of the system and from there conveyed through a second screw conveyor in the reactor for the specified dwell time. The solid coke produced during the pyrolysis process was continuously discharged into a container. The first discharge was observed after a residence time of approximately 30 min.

The gas phase leaving the pyrolysis reactor was used to chlorinate LED in the chlorination reactor. Here the indium reacts to indium chloride and evaporates. The indium chloride condenses with the liquid oil fraction of the pyrolysis vapor and generates a metal concentrate. The remaining gas was then cleaned from halogens, especially excess HCl in a water scrubber to generate hydrochloric acid and other than the gas was further cleaned in the sodium hydroxide scrubber. Aerosols were removed at the ESP. The gas phase was then analyzed with a gas analyzer from the company pollutek. The measuring principles for the gases CO, CO_2, CH_4, and C_nH_m were a non-dispersive infrared sensor, O_2 was an electrochemical detector and for H_2 it was

FIGURE 1: Picture of the pyrolysis plant combined with the chlorination reactor.

a thermal conductivity detector. The rest is assumed to be nitrogen. The gas was then flared at approx. 950°C using a gas flare for safety reasons.

2.5 Recovery of the critical metal indium from the liquid metal concentrate

The critical metal indium is enriched in the liquid metal concentrate and mixed with organic material from the plastic decomposition of the LED. This must be separated before the indium can be recovered. The metal concentrate with 1 ppm indium was filtered with a 2 µm filter. It was then mixed with water.

The volume ratio was 1:2 (water : metal concentrate). After phase separation, 0.7 kg of water phase with an indium content of 4 ppm was produced. For electrolysis, the indium concentration in the solution must be enriched. The filtrate was distilled to remove the solvent. Electrolysis was carried out at a current of 5.2 volts using platinum coated titanium electrodes and an indium content of 199 ppm indium.

3. RESULTS AND DISCUSSION

In the first test with PVC plastic mixture 21.0 kg PVC plastic mixture, 7.6 kg LED, 10.0 kg water for the first scrubber, and 8.7 kg sodium hydroxide (25%) for the second scrubber were used. The masses of the products produced were 7.7 kg of solid residue from the PVC plastic mixture, 6.4 kg of solid residue from the LED, 1.4 kg of filtered metal concentrate, 12.7 kg of water from the first scrubber, and 8.2 kg from the sodium hydroxide scrubber. ICP-OES/EDXRF analysis of the products show a distribution of chlorine. 88% of the chlorine remains in the solid residue of the PVC plastic mixture, 9% in the solid residue of the LED, and 2% in the filtered metal concentrate. The remaining 1% is in the gas scrubber. The high chlorine content in the solid residues of the PVC plastic mixture from commercial waste can be explained by the formation of salt – probably calcium chloride – due to calcium-containing fillers in plastics. In addition, two circumstances favor salt formation in the pyrolysis process. First, the high temperature due to the removal of plastics from the fillers leads to salt formation. Here, a lower pyrolysis temperature could reduce the amount of chlorine remaining in the solid residue of the PVC plastic mixture.

Secondly, granulation of the material leads to better mixing of the various plastics, as well as of the plastic with fillers and PVC.

A washing step of the solid material from PVC plastic mixture was performed to reduce the chlorine content from 11.00 wt.-% to 2.88 wt.-% and increase the calorific value from 10.2 MJ/kg to 12.5 MJ/kg, but further treatment of the solid material is necessary to use it for cement production. The pyrolysis gas purified in the gas scrubber can be used for energy purposes. The lower heating value is 24.7 MJ/m^3. In addition to indium, the filtered metal concentrate contains organic components that could be used for energy purposes. The lower heating value is 37.6 MJ/kg.

In the second test with PVC waste from industry 7.9 kg PVC waste from industry, 20.0 kg LED, 10.0 kg water for the first scrubber, and 10.0 kg sodium hydroxide (25%) for the second scrubber were used. The masses of the products produced were 1.3 kg of solid residue from PVC waste from industry, 14.7 kg of solid residue from the LED, 0.9 kg of filtered metal concentrate, 11.1 kg of water from the first scrubber, and 9.7 kg from the sodium hydroxide scrubber. ICP-OES/EDXRF analysis of the products shows a distribution of chlorine. 19% of the chlorine remains in the solid residue of the PVC waste from industry, 79% in the solid residue of the LED, and 2% is in the gas scrubber.

A washing step of the solid material from PVC waste from industry was performed to reduce the chlorine content from 7.88 wt.-% to 2.79 wt.-% and decreased the calorific value from 18.0 MJ/kg to 16.4 MJ/kg, but further treatment of the solid material is necessary to use it for cement production. The lower calorific value is not so significant and could be explained by inhomogeneous composition of the solid residue after pyrolysis. The pyrolysis gas purified in the gas scrubber can be used for energy purposes. The lower heating value is 17.1 MJ/m^3. In addition to indium, the filtered metal concentrate contains organic components that could be used for energy purposes. The lower heating value is 31.7 MJ/kg. Indium was recovered from the metal concentrate of the first test with PVC plastic mixture by mixing with water, phase separation, distillation, and electrolysis. The filtered metal concentrate was mixed with water. The indium accumulates in the water phase. 40% of the indium from the LED was found in the water phase. The content of the indium increased from 1 ppm in the 7.6 kg LED bar lights to 20 ppm in the water phase with 153 g. After distillation, the solution contains 0.2 g In/L and a total weight of 15 g. The metal indium was recovered qualitatively from electrolysis.

4. CONCLUSIONS

Products like metal concentrate or gas from the pyrolysis process of PVC plastic mixture or PVC waste from industry can be used for energetic purposes. By washing the solid residue from PVC plastic mixture or PVC waste from industry the chlorine content could be reduced significantly, but the chlorine content is still too high to use for cement production or direct energetic use. Then additional tests like for dioxins/furans or heavy metals for further characterization is needed to find an area of application for this solid residue.

Depending on the chlorine content and especially further filler materials, chlorine could be removed from PVC containing waste. Pyrolysis is a promising recycling route for PVC containing waste in the future, but more research for optimizing process parameters and processing of the products – mainly the solid residue is needed. The yield of indium recovery reached 40% for a 0.2 g/L solution, which resulted in an enrichment factor of 494 compared to the indium content in the LED. The recovery of indium from LED is possible with the combination and further process steps. Here the limits are the heaters and material for the chlorination reactor to handle the heat together with the corrosive environment in an industrial application. For further byproducts from the process steps from the metal concentrate to the metal indium recycling a strong focus should be on reuse in further research activities to keep the cost for an industrial process low and more possible to realize it.

The chemical recycling of PVC-containing plastic waste to recover critical metals is a promising way to solve two important problems in waste management.

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