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RECYCLING OF POTENTIAL HAZARDOUS STONE WOOL INTO A NON-HAZARDOUS NEW STONE WOOL

Theresa Sattler [1,*], Klaus Doschek-Held [2], Anna Krammer [2], Roland Pomberger [1] and Daniel Vollprecht [3]

[1] *Department of Environmental and Energy Process Engineering, Montanuniversitaet Leoben, Franz-Josef-Straße 18, A-8700, Leoben, Austria*
[2] *Department of Environmental and Energy Process Engineering, Montanuniversitaet Leoben, Chair of Thermal Processing Technology, Franz-Josef-Straße 18, A-8700, Leoben, Austria*
[3] *Institute of Materials Resource Management, University of Augsburg, Am Technologiezentrum 8, D-86159, Augsburg, Germany*

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ABSTRACT

Mineral wool products are man-made vitreous fibres (MMVFs), such as glass wool and stone wool, mainly used for thermal and acoustic insulation. Demolition of buildings generates mineral wool (MW) waste. With regard to the intended recycling of materials, the European Union's circular economy package currently foresees a landfill ban for certain waste streams by 2030. As a result, Austria will have a landfill ban for MMVFs from 2027. This paper presents an investigated route for the recycling of MW waste into new mineral wool (wool2wool). The recycling route aims to conserve valuable and limited landfill volume and enables the reduction of primary resource consumption and CO_2 emissions. The investigation was based on pH-dependent leaching tests of the fibres according to ÖNORM EN 14429 and hydrogeochemical modelling using LeachXS/Orchestra to identify the solubility-controlling mechanisms. The next step was a thermochemical treatment using correction materials to adjust the chemical composition, followed by rapid cooling through a spinning process. Another focus was on the theoretical determination of the dynamic viscosity to ensure suitable flow behaviour during fibre production. The chemical composition of the mineral wool produced was analysed, and it was shown that the target formulation could not be achieved for all elements within the permitted variation. The variations were due to the dissolution of the kiln lining, which had a more significant influence on the experiment than expected. Overall, the recycling pathway showed a high potential for CO_2 savings, resource savings and other environmental benefits by recycling waste that is currently landfilled.

1. INTRODUCTION

The 2030 Agenda for Sustainable Development of the United Nations (UN) proclaimed 17 Sustainable Development Goals (SDGs) in addition to the Millennium Development Goals (MDGs) (United Nations 2015). The recycling of mineral wool waste can be expected to contribute to the achievement of some of these goals, as waste management is only indirectly addressed in the SDGs. It should also enable the implementation of the Austrian landfill ban for mineral wool waste from 2027 (DepVO 2021).

Mineral wool is the generic term for a man-made vitreous fibre made primarily from glass (glass wool), igneous rock (stone/rock wool) and slag (slag wool). Mineral wool products are used as insulation and in horticulture. Mineral wool waste causes waste management problems due to its high volume and low density. Currently, most of Austria's mineral wool waste is landfilled and not recycled (Sattler et al. 2020a). As it is mandatory to landfill mineral wool waste in big bags in separate landfill compartments, this results in a 400-fold difference in Young's modulus between the general Construction and Demolition (C&D) waste and the mineral wool compartments (Sattler et al. 2020b), causing serious accidents in the form of overturned vehicles due to sudden ground settlement when crossing the landfill and driving on the unstable mineral wool body (Huber 2019). The positive effect of briquetting mineral wool waste on density and strength was shown, where the bulk density of glass wool and stone wool was increased up to 100 times. Therefore, briquetted mineral wool waste is expected to be more stable in landfill than loose mineral wool waste (Sattler et al. 2019).

In line with the waste hierarchy, priority however should be given to recycling rather than landfilling. Studies have

* Corresponding author:
Theresa Sattler
email: theresa.sattler@unileoben.ac.at

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shown that recycling mineral wool waste (glass wool waste and stone wool waste) in the cement industry can decrease production costs, saves primary raw materials and decreases CO_2 emissions in production, and can also save valuable landfill space (Sattler et al. 2020c). Another way in which mineral wool can be recycled is as supplementary cementitious material (SCM) (Doschek- Held et al. 2023), (Sattler et al. 2020d). It can also be used to produce alkali-activated materials (AAMs) (Yliniemi et al. 2016). AAMs are alternative binder systems, activated by alkaline solutions, such as water glass and alkali hydroxides and are used to create concrete-like structures (Tuorila et al. 2020), (Steindl et al. 2023).

From the point of view of the waste hierarchy, a recycling at the mineral wool plant is preferable. Currently, mineral wool manufacturers only take back their own new, non-hazardous mineral wool waste for recycling. This is because mineral wool fibres may have adverse health effects in case of low solubility in the human lung (Muhle et al. 1998), (Costa & Orriols 2012) which is generally not known for old mineral wool fibres in C&D waste. The carcinogenity of mineral wool fibres depend on their dimension, dosis and durability (Barly et al. 2019). A dimension with a length of more than 5 µm, a diameter of less than 3 µm and a length-to-diameter-ratio of 3 or more is considered as "critical" for lung dieseases according to the World Health Organization (WHO), so that such fibres are called WHO fibres (IARC 1988). However, even WHO fibres can be destroyed in the human lung by so-called clearing mechanisms. The dwell time of a fibre in the human lung is described by the biopersistance which is affected by mechanical clearance mechanisms and biochemical dissolution (IARC 1988). Therefore, since the 1990s many countries introduced compulsory biosolubility tests for newly produced mineral wool (97/69/EC 1997), (GefStoffV 2010) and the industry developed corresponding labels such as RAL (RAL 2023) and EUCEB (EUCEB 2023). The EUCEB (EUropean CErtification Board for mineral wool products) was established in 2000 to increase the confidence of authorities, customers, and consumers in the safety of mineral wool. The general purpose of these labels is to provide voluntary certification that the manufactured fibres have a chemical composition within the ranges of the approved reference fibres. In these in-vivo tests, mineral fibres are implanted into animals and after 40 days the animals are killed and the number of still existing fibres is compared with the initial amount. An alternative are in-vitro-tests in which the lung environment is modelled by experimental solutions. In this case the pH values of the extracelular environment (7.4) and within the macrophages (4.5) must be investigated (Oberdörster 1991). For stone wool, a mineral wool subgroup, the biosolubility increase was reached by a higher alumina content (Guldberg et al. 2000) but it has to be considered that the dissolution rates of the fibres are also influenced by the binder of the stone wool (Wohlleben et al. 2017). The relation between the Al content and the biosolubility is highly complex. For Al/(Al+Si) ratios below 0.1, which is typical for glass wool, the dissolution rate at pH 7.4 decreases with increasing Al content, whereas it increases with increasing Al content above ratios of 0.2 which are typical for stone wool (Guldberg et al. 2000). It has to be mentioned that the observed trend is only valid for pH 7.4 which is typical for the pleural fluid. In the macrophages at pH 4.5 also low-Al stone wool dissolves well which has to be considered in an overall health assessment. In consequence, for stone wool, "new" products with Al_2O_3 concentrations of 19 to 22 wt% and SiO_2-concentrations of 37 to 43 wt% compared to "old" products with Al_2O_3 concentrations of around 14 wt% and SiO_2 concentrations of about 46 wt% were produced. However, due to the landfill ban, demolition and waste management face a severe challenge and request manufacturers to recycle all mineral wool waste. For this purpose, it is necessary to investigate how old, hazardous mineral wool waste can be spun into new mineral wool. The adverse health effects of hazardous mineral wool are thought to be determined by certain fibre characteristics such as fibre length, fibre diameter and also in vivo durability and persistence (IARC 1988). In vivo tests (Eastes et al. 2000) and in vitro tests (Guldberg et al. 2001) found that an increasing aluminium content and decreasing silicon content increased the dissolution/degradation rate at an acidic pH in stone wool, which is preferable from a health point of view. However, beside the aspect of health, the chemical composition effects the properties of the melt. The implementation of the aforementioned EUCEB certificate led to an increase in the aluminium oxide content of stone wool products. This circumstance is essential for the chemical modification in a wool-to-wool recycling concept and influences the thermal behaviour of the melt, e.g. the dynamic viscosity, during the fibre production process.

This study aimed to investigate the recycling of low aluminium mineral wool in order to obtain new mineral wool with a higher aluminium content. The results of this work show that it is possible to recycle mineral wool waste by conditioning it with other correction materials.

2. MATERIALS AND METHODS

2.1 Sampling

In order to obtain a representative sample material of mineral wool waste, a staggered sampling was conducted at an Austrian landfill. The waste material was collected from each truck over the course of a month, and then placed in hermetically sealed bags. 13 stone wool waste samples were taken.

Seven mineral wool product samples were purchased from Austrian construction markets.

2.2 XRF analyses

The chemical composition of the mineral wool waste and mineral wool product samples, as well as the new mineral wool spun in this study, was determined by XRF. The fiber samples were milled and analysed for loss on ignition at 1025°C before producing melt-fused tablets. Subsequently, the chemical composition was determined using X-ray fluorescence spectroscopy (XRF) by a Panalytical XRF instrument (Axios PW2400) from CRB GmbH at a voltage of 60 kV.

2.3 Chemical composition and solubility of stone wool

Three stone wool samples (SW_1, SW_2 and SW_3) were selected from the 20 available (13 waste samples, 7 products) for further experiments based on their chemical composition. pH-dependent leaching tests based on ÖNORM EN 14429 were conducted to study the solubility of the samples (Bauer 2021). The shredded samples weighing 25 g were suspended in 1 L of ultrapure water and shaken for 48 hours using an overhead shaker at 6 rpm. To achieve the desired pH level, a predetermined amount of acid or base, determined in separate pre-tests, was added before the experiments. After completion of the experiments, the solutions were separated from the solids through filtration at 0.45 µm. In contrast to the study of Guldberg et al. (2000) who determined dissolution rates, i.e. kinetic constants, in this approach a thermodynamic equilibrium between solid and aqueous solutions should be achieved. Prior to the leaching tests, samples were comminuted using an Eirich mixer to maximum fibre lengths of 1 to 1.5 cm. In deviation to the standard, a liquid/solid (L/S) ratio of 15 instead of 10 was chosen to prevent complete absorption of the liquid by the solid, which had been observed for L/S = 10. The leachate was analysed after the experiment for pH, filtrated at 0.45 µm and analysed for cations using inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7500 CE) and for anions using ion chromatography (IC, Thermo Fisher Scientific ICS 2000). The leachable concentrations at all pH values were used as input for hydrogeochemical modelling which was done using LeachXS/Orchestra and the Minteqv4 database. The program uses the maximum leachable concentrations as "available" concentrations and predicts the precipitation of solid phases from this hypothetical solution as well as the sorption of dissolved species to sorbents such as hydrous ferric oxides (HFO). The modelling strategy was based on the inclusion of all phases possibly precipitating from the solution. If the model yielded lower concentrations than in equilbrium with these phases, the corresponding phases were excluded from the model. This procedure was applied iteratively until modelled and analysed leached concentrations matched each other. Since carbonate was not determined in the leachates due to the rapid interaction with the atmosphere, the available carbonate concentrations were fitted to the measured Ca concentrations since calcite saturation is suggested to be a leaching controlling mechanism. The corresponding values were 20,000 mg/kg dry matter (DM) for the Al-rich samples and 500 mg/kg DM for the Al-poor sample.

2.4 Wool2wool – Mineral wool recycling

The stone wool sample with the lowest aluminium content, i.e. 1.65 wt%, was selected as the conditioned sample due to its presumed low solubility. This value is far below the "old" stone wool composition given in the literature and rather suggests a high solubility (Guldberg et al. 2000). The target formulation in the experiment was the chemical composition of a new, non-hazardous mineral wool. The Urbain model was used to ensure comparability with the dynamic viscosity published by Peternelj et al. (2017). The dynamic viscosity was calculated for the chemical composition of the melt of the newly spun mineral wool and the target composition range using the EUCEB deviation criteria.

TABLE 1: Amount of stone wool and residual correction materials.

component	weight [g]
stone wool (SW_3)	17,500.0
dolomite slurry	7,001.6
corundum dust	7,001.1
SiO_2 (pure)	2,455.0
MgO (pure)	1,049.0

2.4.1 Chemical composition and dynamic viscosity

In order to achieve the target composition (Table 3), the appropriate amount of addition of waste and correction materials to be added to the stone wool was calculated, using a Microsoft Excel Solver Add-In; these correction materials were dolomite slurry and corundum dust, while SiO_2 and MgO were added as pure materials (Table 1). The chemical composition of the stone wool, dolomite slurry and corundum dust are shown in Figure 1.

To calculate dynamic viscosity, an aluminium oxide content of stone wool ranging from 14 to 18 wt% (Yliniemi et al. 2021) was anticipated. Peternelj et al. (2017) published a dynamic viscosity 1.88 Pa*s at 1400°C for his spinning wheel tests using a stone wool sample with an aluminium oxide content of about 17 wt%. The dynamic viscosity was calculated using the Urbain model which considers cations such as glass formers, modifiers and amphoteric cations (Urbain 1987).

2.4.2 Mechanical processing

The first step was to prepare the mineral wool using a forced action mixer to reduce particle size and homogenise the material with the other residual and pure materials (correction materials), followed by wet pelletising. Prior to the thermochemical treatment, the pellets were dried, preheated at 120°C and separated in 2 batches of 18 kg each.

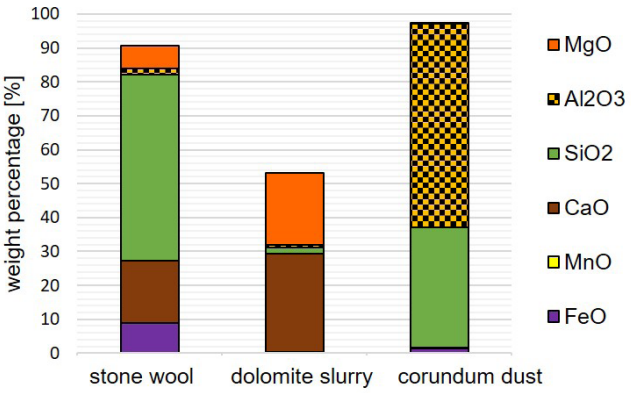

FIGURE 1: Chemical composition of stone wool and waste correction waste materials.

2.4.3 Thermochemical conditioning

A top blown rotary converter (TBRC) furnace was loaded with the preheated pellets. The material was then heated to a maximum temperature of 1500°C in the furnace. As the melting point of the pellets was exceeded, a melt was formed. When the target temperature was reached, the furnace was tilted and the melt stream was poured over a steel nose onto a 2-wheel spinner. At the spinner, the melt was defibred by compressed air and spun into conditioned, new mineral wool (Figure 2). The melting process of both batches was monitored and documented using a thermal imaging camera, a high-speed camera and several pyrometers.

3. RESULTS AND DISCUSSION

3.1 Chemical Composition of stone wool products and stone wool waste

The chemical composition of the stone wool products is shown in Figure 3 and the composition of the analysed stone wool waste is presented in Figure 4. Stone wool products tend to be richer in SiO_2, Al_2O_3, Na_2O and MgO, whereas values for CaO, K_2O and Fe_2O_3 tend to be slightly higher in stone wool waste. The variation in stone wool waste is explained by two factors. Firstly, the chemistry of old mineral wool waste has changed compared to new mineral wool waste after the EUCEB label for new mineral wool products

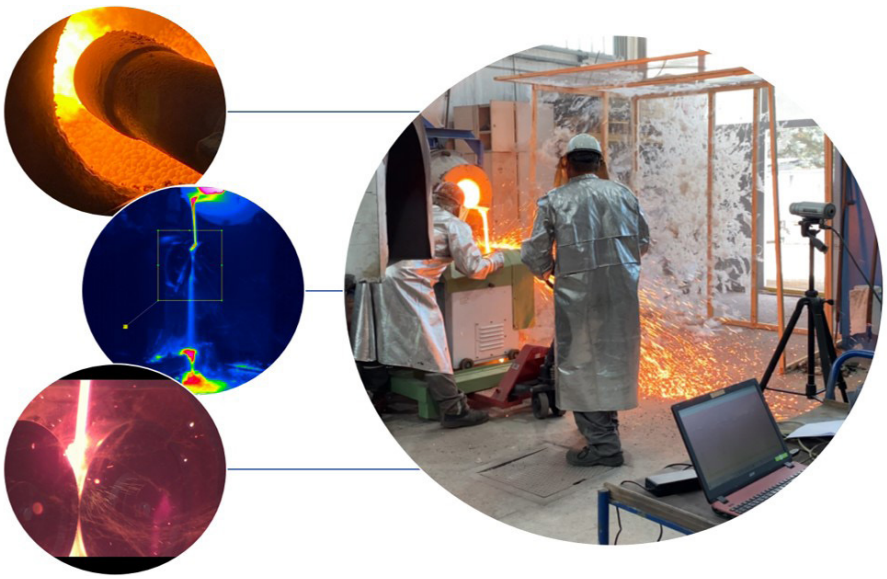

FIGURE 2: Overview of thermochemical conditioning and spinning process.

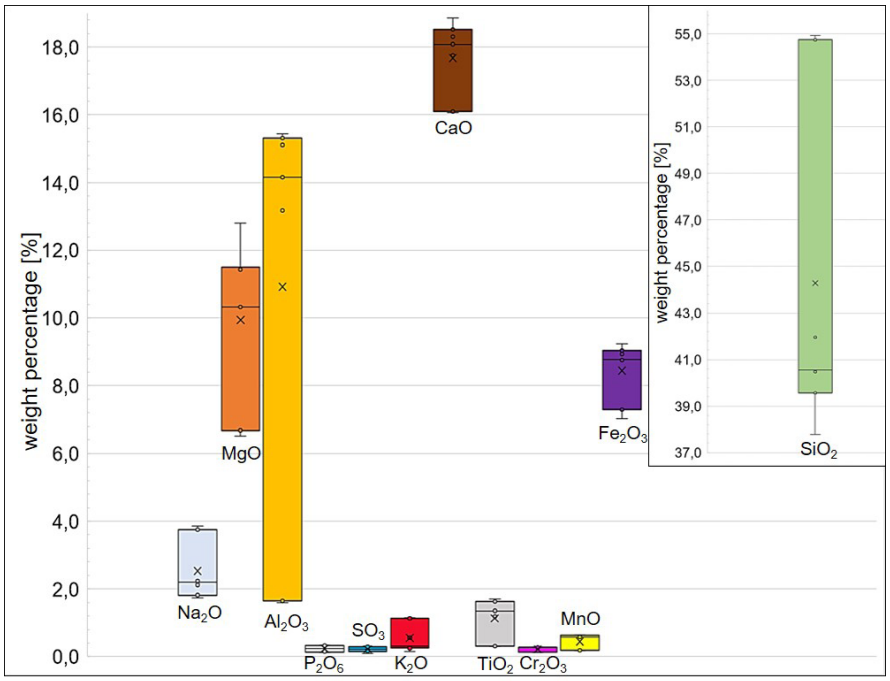

FIGURE 3: Chemical composition of the stone wool products (n=7).

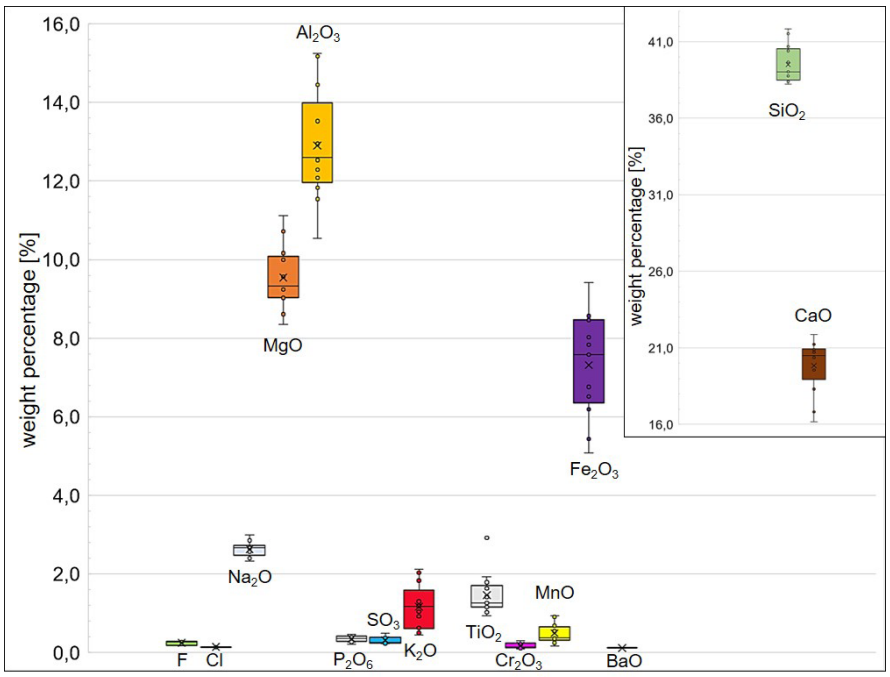

FIGURE 4: Chemical composition of the stone wool waste (n=13).

was achieved. This explains in particular the variation in Al_2O_3 content. Secondly, some impurities are introduced into the waste as a result of contamination during the demolition of buildings. Typical impurities in stone wool waste are gypsum adhesions, iron filings and aluminium facings. The presence of these materials results in increased levels of CaO and Fe_2O_3.

The full list of chemical analyses can be found as supplementary content alongside the electronic version of this article.

3.2 Solubility of Stone Wool

The chemical composition of the investigated samples within the solubility tests (Table 2) shows that one sample (SW_3) is significantly depleted in Al compared to the other two samples. The Al/(Al+Si) ratio of this sample is 0.03 compared to 0.23 and 0.26, respectively. According to the data of Guldberg et al. (2000), the Al-poor sample should have an increased biosolubility compared to the Al-rich samples, whereas even higher Al/(Al+Si) ratios of around 0.4 would also yield a high biosolubility.

The pH-dependent leachability of Si and the corresponding hydrogeochemical models of the investigated samples is displayed in Figure 5, modified after Bauer (2021).

Si can be considered as a proxy for stone wool dissolution as it is the network former of the glass structure. At first, the solubility at pH 4.5, the environment inside the macrophages, shall be compared. In this range, 150 mg/L Si are leached from sample SW_1, 18 mg/L from sample SW_2 and less than 1 mg/L from sample SW_3. In contrast to the results for the dissolution rates of Guldberg et al (2000), these equilibrium tests show the lowest leaching of Si for the lowest Al concentration and lowest Al/Si ratio. The sample with the highest Al concentration and highest Al/Si ratio plots, however, does not show the highest, but intermediate leaching. At pH 7.5, the environment outside the macrophages in the lung fluid, 30 mg/L Si are leached from sample SW_1, 6 mg/L from sample SW_2 and only 0.5 mg/L from sample SW_3. This means, that the order between the samples remains the same, but the overall leaching of Si is lower at neutral pH than in the acidic range. The overall pH-dependent trend of Si leaching shows an amphoteric behavior with lowest leaching in the neutral pH range and increasing leachability towards both acidic and alkaline pH. However, the low-leaching Al-rich sample SW_3 has the leaching maximum in the alkaline pH range - with similar values as the two other samples, whereas the higher-leaching samples have a leaching maximum in the acidic pH range. The main difference between the Al-rich

TABLE 2: Chemical composition of the stone wool samples chosen for solubility tests.

	SW_1 [wt%]	SW_2 [wt%]	SW_3 [wt%]
Na_2O	2.85	2.24	3.87
MgO	9.24	12.80	6.68
Al_2O_3	11.83	15.11	1.65
SiO_2	39.68	41.96	54.94
P_2O_5	0.28	0.09	0.05
SO_3	0.49	0.07	0.29
K_2O	1.83	0.14	0.32
CaO	20.71	18.86	18.31
TiO_2	1.42	1.70	0.31
Cr_2O_3	0.11	0.04	0.03
MnO	0.62	0.19	0.05
Fe_2O_3	6.19	7.30	8.93

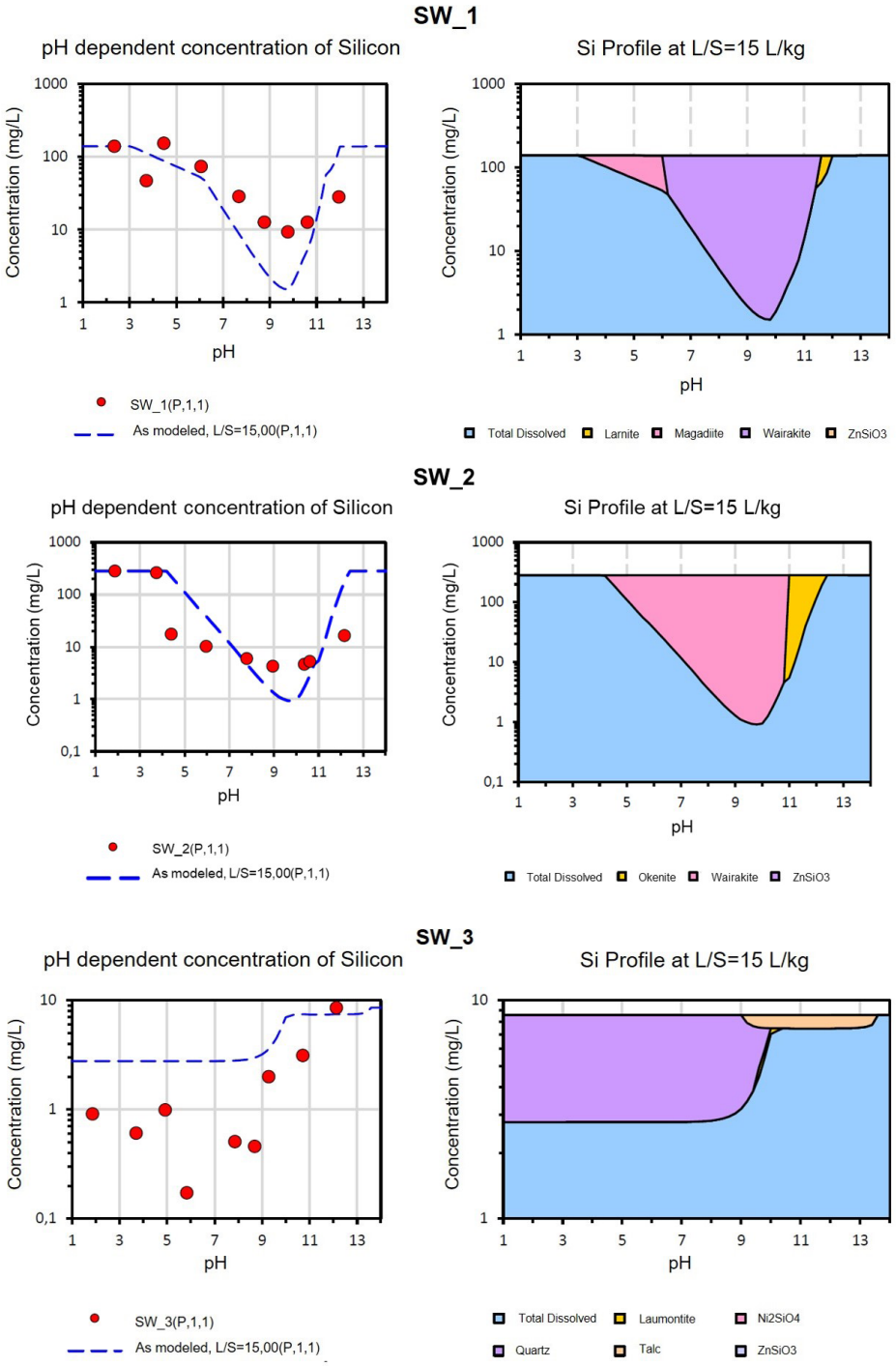

FIGURE 5: Hydrogeochemical models regarding the pH-dependent leachability of silicon for SW_1, SW_2 and SW_3 (modified after Bauer (2021)).

sample and the other two samples is the suppression of Si leaching in the acidic to neutral pH range. It has to be considered that the experimental approach in this study is a simplification of the actual conditions in the human lung, not considering organic species present and not accounting for kinetic effects or phagocytosis. However, this simplification allows to identify the effect of inorganic processes such as dissolution and precipitation of primary and secondary phases, respectively. In contrast, in the Al-rich sample, the entire available Si concentration, i.e. more than 100 mg/L, is leached at acidic pH and any leaching control mechanism is absent. It can be suggested that the presence of Al in the leachate yields to the formation of dissolved Al-Si species increasing the solubility of Al-rich stone wool and preventing precipitation of secondary mineral phases. In contrast, in the alkaline pH range, leaching control mechanisms are present. For the Al-rich samples, the precipitation of wairakite, $Ca(H_2O)_2[Al_2Si_4O_{12}]$, is suggested to control the leaching. For the Al-poor sample, the precipitation of talc, $Mg_3[(OH)_2|Si_4O_{10}]$, yields similar leach-

able concentrations of <10 mg/L Si at pH 10. Anyway, this pH range is not relevant to assess the health risks associate with stone wool waste.

Based on the chemical analyses and the solubility studies, the sample SW_3 was selected as a potentially hazardous material to demonstrate that such kind of wastes can be recycled into non-hazardous, highly soluble new stone wool. The composition of sample SW_2 was defined as a target composition which would be non-hazardous and could be sold under current legislation. This was done, because the even more soluble sample SW_1 was a waste sample, whereas the chosen SW_2 was indeed a "new" stone wool product with EUCEB certificate. The aim of the subsequent thermochemical treatment test was therefore to adjust the composition of SW_3 to the composition of SW_2 by adding Al-rich residues.

3.3 Recycling test

The XRF analysis of the newly spun mineral wool (Figure 6) showed that the target formulation within the permitted range of variation of the EUCEB quality label was not achieved for all elements (Table 3). The Al_2O_3 content after spinning was 2.73 wt% higher in batch 1 and 4.23 wt% higher in batch 2 than calculated using the composition and mixing ratio of the input materials. There were also deviations in the magnesium oxide and iron oxide contents. This can be explained by the fact that the refractory lining has dissolved and mixed with the melt. It was observed that all components detachable from refractory lining were raised in both batches of newly spun mineral wool.

3.4 Dynamic Viscosity

The determination of dynamic viscosity over a temperature range of 1000°C to 1700°C was effectively determined using the Urbain model (1987), with the chemical compositions serving as the controlling factor. Importantly, both batch 1 and batch 2 met the specified EUCEB deviation criteria for the desired target composition. Consequently, both batches are well within the specified dynamic viscosity thresholds. It should be emphasized that there is a high degree of consistency between the results of the two test batches, indicating a good reproducibility and methodological reliability. This consistency is evident in Figure 7, which shows not only the internal agreement but also the agreement with the dynamic viscosity results published by Peternelj et al. 2017. These agreements support the validity of the Urbain model and enhance its applicability in predicting the dynamic viscosity trends for similar compositions and conditions.

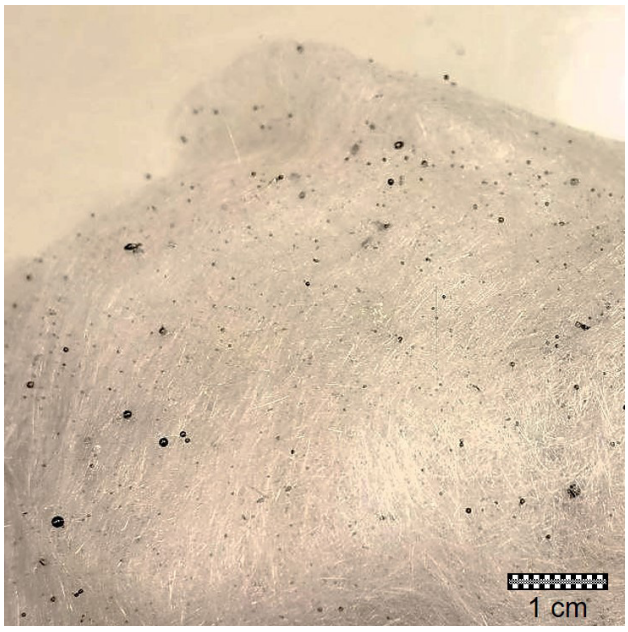

FIGURE 6: Newly spun mineral wool.

TABLE 3: Composition of the newly spun mineral wool (batch 1 and batch 2) compared to target composition.

	initial composition [wt%]	batch 1 [wt%]	batch 2 [wt%]	target composition [wt%]
Na_2O	3.87	2.1	2.2	1.72
MgO	6.68	12.5	12.5	9.77
Al_2O_3	1.65	18.6	20.1	15.87
SiO_2	54.94	43.4	42.8	41.09
P_2O_5	0.05	0.1	0.0	0.36
SO_3	0.29	<0.025	<0.025	0.33
K_2O	0.31	0.2	0.2	1.29
CaO	18.31	16.9	16.3	15.65
TiO_2	0.31	0.2	0.2	1.34
Cr	0.0	0.07	0.07	0.0
MnO	0.05	0.1	0.0	0.45
FeO	8.04	5.22	5.04	3.69

4. CONCLUSIONS

The feasibility of recycling post-consumer mineral wool waste of unknown origin and potential hazardous properties into safe, new mineral wool products is demonstrated by the recycling route (wool2wool) investigated. Post-consumer mineral wool waste from C&D activities is often lacking labels indicating health safety and is therefore considered hazardous. In-vitro and especially in-vivo tests for (bio)solubility are not feasible for mineral wool waste due to temporal and financial efforts. The relationship between chemistry and solubility generally allows to predict the health impact of mineral wool. However, mineral wool manufacturers and quality associations do often not disclose the chemical composition of their products and the outcome of (bio)solubility tests, excluding a scientific assessment of the complex relations between chemistry and solubility. pH-dependent leaching tests revealed that low-Al fibres (1.65 wt% Al_2O_3) are characterized by a low solubility in the biologically relevant acidic to neutral pH range (<1 mg/L Si), whereas high-Al fibres (11.83 wt% and 15.11 wt% Al_2O_3) exhibit more than one order of magnitude higher solubility (>10 mg/L Si). Hydrogeochemical modelling suggested reasons for the different leaching of Si from the three stone wool samples. Leaching is the property of a chemical element to be released from a sol-

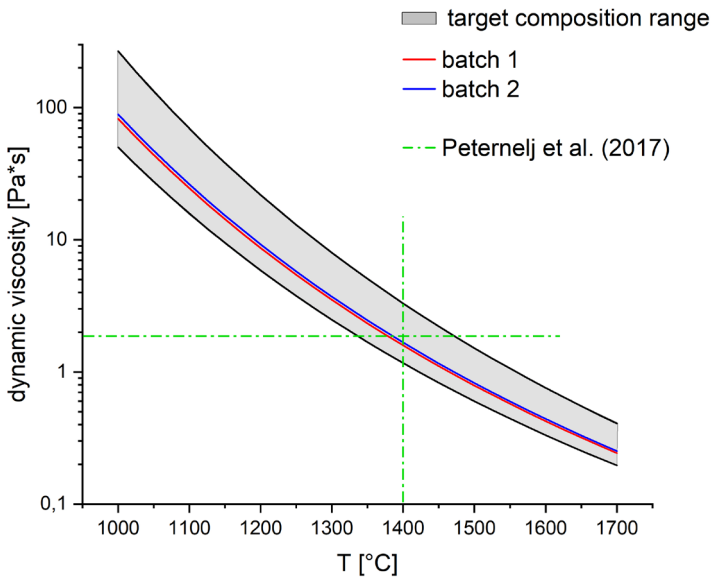

FIGURE 7: Hydrogeochemical models regarding the pH-dependent leachability of silicon for SW_1, SW_2 and SW_3 (modified after Bauer (2021)).

id to the liquid. Leaching is associated with solubility in thermodynamic equilibrium, if precipitation/dissolution reactions control the leaching. Solubility is the property of a solid phase to dissolve in an aqueous solution. Both the dissolution of a solid phase in an undersaturated solution and the precipitation of this phase from a supersatured solution yield the same equilibrium concentration of the dissolved species in the leachate. In both cases, the aqueous solution is saturated with respect to the coexisting solid phase. According to our model, in the Al-rich sample SW_3 the low leaching of Si in the acidic to neutral pH can be explained by the low solubility of quartz. Considering that quartz is not present in the mineral wool, which is amorphous, the model assumption is that the glassy phase dissolves, releasing Si species into solution. Then this solution gets supersaturated with respect to secondary mineral phases which may precipitate at the surface or in the free solution volume. Alternatively, and additionally, surface adsorption processes may take place which are also considered in the model. However, it seems unlikely that quartz precipitates from aqueous solutions at room temperature, but also the slightly higher solubility of amorphous silica would be a reasonable leaching control mechanism. In this context, leaching control mechanism means that the detected Si concentration in solution is the equilibrium concentration coexisting with amorphous silica. Assuming such a situation, it may still be the case, that the overall fibre dissolution continues, if the solubility of the glassy phase is above that of amorphous silica, but the excess Si released into the solution, would be precipitated as amorphous silica.

Hydrogeochemical modelling suggests that low solubility is due to the presence of amorphous pure silica, whereas the formation of dissolved alumino-silicate species fosters the dissolution of high-Al mineral wool fibres. Therefore, mixing Al-poor and therefore potentially hazardous mineral wool waste with Al-rich corundum dust and additional correction materials, and remelting the mixture to produce Al-richer mineral wool products is a feasible strategy to cope with the challenge of mineral wool waste management. In order to calculate the required mixing ratio, the dissolution of refractory material in the melt must also be considered. Further research is required to develop appropriate refractory concepts and to predict the dissolution of refractories in silicate melts obtained from multi-waste mixtures. In summary, thermochemical treatment of mineral wastes allows to destroy hazardous properties such as biopersistance or fibre geometry.

Since, unlike landfilling, thermo-chemical treatment can destroy the hazardous character, it should be further investigated for the recycling of other hazardous wastes such as asbestos.

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