Optimization of alkali-activated mineral wool mixture for panel production

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Abstract. A significant amount of mineral wool waste is generated during the construction and demolition of buildings. At the moment, most of this material ends up in a landfill without further utilization. Alkali activation is one technology already recognized to produce low carbon dioxide binders and other products/materials from several industrial by-products, and could also be employed to recycle mineral wool waste. This study shows the result of milling and homogenization of different types of wool waste and their subsequent use as precursors in the alkali -activation process. Two different types of mineral wool waste were taken from the mining company Termit (Slovenia). Stone and glass wool were milled, pulverized and sieved to a particle size smaller than 63 μm. After homogenization, different alkali-activated pastes were prepared using two different alkali activators (NaOH and/or Na -water glass). The compressive and flexural strength of each alkali-activated material was measured, showing higher values for glass wool in comparison to stone wool. In addition, different curing temperatures were assessed (room temperature and 40 °C). The compressive and flexural strength of glass wool after three days at 40 °C was 34.7 and 8.7 MPa respectively, compared to values of 29.1 and 9.3 MPa for stone wool. After 3 days at room temperature the strengths were not measurable, however, after 28 days the respective compressive and flexural strength were 29.9 and 14.4 MPa in the case of glass wool, and 40.6 and 14.9 MPa for stone wool.

Keywords: mineral wool, construction and demolition waste, recycling, alkali-activated material, mechanical properties, circular economy.

1. Introduction

Mineral wool (stone and glass wool) is the most common insulating building material worldwide. Due to the massive production of mineral wool, a huge amount of construction and demolition waste has been generated. Mineral wool waste has a low density (20-200 kg/m^3), low weight, and contains formaldehyde-based components that could leach into the environment after disposing to landfill. Due to its low weight and

density, it consumes a large amount of space. In addition, sorting and separation, logistics and the lack of economically feasible uses for mineral wool waste impedes its recycling. This study, which is part of the H2020 project Wool2Loop ["WOOL2LOOP," 2019] aims to upgrade construction and demolition wool waste into a valuable raw material which could be used in new products and for applications based on alkali activation technology. By combining smart demolition practices with on-site analysis, the costs of the mineral wool waste can be reduced to a level which would enable reuse. Alkali activation technology is an important method to utilise mineral wool waste as a raw material to convert into new materials. A careful mix design enables the production of various value-added products with different properties (e.g. hollow core slabs, fibre-reinforced panels, acoustic panels, pavement slabs, facade panels, etc.).

Alkali activation technology can be used as an alternative to Portland cement production to prepare a low carbon dioxide binder and other products/materials from several industrial by-products, including mineral wool waste. Moreover, concrete or ceramic-like materials originating from mineral wool generate 50%–80% less CO_2 emissions than regular concrete [Habert & Ouellet-Plamondon, 2016; Provis & Deventer, 2014]. Alkali activated materials are generated through a combination of an alkali source (typically sodium or potassium silicate or hydroxide) and an aluminosilicate component (blast-furnace slag, fly ash, calcined clay, etc.) [Provis & Deventer, 2019]. During alkali activation a structurally disordered, highly cross-linked aluminosilicate gel is formed [Provis, 2014]. Mineral wool waste could be a potential precursor for alkali activation due to its consistent chemical and physical composition, high content of Si and X-ray amorphous mineralogy [Kinnunen, Yliniemi, Talling, & Illikainen, 2016; Yliniemi, Kinnunen, Karinkanta, & Illikainen, 2016].

The goal of the present work is the development of mixtures based on wool waste by applying alkali activation technology. Glass and stone wool, which were used as precursors, were ground, homogenized and activated using sodium hydroxide and/ or sodium silicate. The microstructure before and after grinding, as well as after alkali activation was analysed, and the effect of different curing temperatures on mechanical properties was assessed.

2.   Experimental part

Different types of mineral wool waste (glass and stone wool) were obtained from an open waste dump belonging to the company Termit d.d.. Samples were first separated, shaken by hand to remove particles such as pebbles and wood, and cut into small pieces. About 1 kilogram of wool waste was placed in a classic concrete mixer and mixed for 2 hours. The sample was homogenized and dried at 105 °C for 24 hours in a drying oven then sieved to a particle size below 63 μm. The residue was ground again in the homogenizer, sieved and a very small amount left for disposal.

Alkali-activated materials (AAM) were prepared using glass (labelled as GW) or stone (labelled as SW) wool sieved below 63 μm, and alkali activators NaOH (Donau

Chemie Ätznatron Schuppen, EINECS 215-785-5) and/or Na-water glass (received from mining company Termit, with mass percentage of Na_2O 12.8 %, and mass percentage of SiO_2 29.2 %). NaOH and Na-water glass were prepared in different proportions, stirred until the liquid became clear (when NaOH was added into Na-water glass), and poured into the sample whilst constantly mixing.

Table 1: AAMs mix design and liquid to solid ratio (calculated according to precursor) for glass and stone wool waste.

	GW1	GW2	GW3	GW4	SW1	SW2	SW3	SW4
Mineral wool waste (g)	150	150	150	150	150	150	150	150
NaOH (g)	12	3	/	6	12	3	/	6
Na_2SiO_3 (g)	/	60	96	91	/	60	129	117
H_2O (g)	65	22	/	/	98	42	/	/
L/S	0.43	0.38	0.37	0.35	0.65	0.51	0.5	0.45

The paste mixtures (precursors and activators) were moulded into prisms of 80x20x20 mm^3. Compressive and flexural strength were measured using a compressive and flexural strength testing machine (ToniTechnik ToniNORM) after three days cured at 40 °C and after 1, 3, 7, 14 and 28 days at room temperature.

XRD analysis of glass and stone wool samples was performed using an Empyrean PANalytical X-ray Diffractometer (Cu X-Ray source) between 4 and 70 degrees at intervals of 0.0263 degrees , under cleanroom conditions in powder sample holders.

For XRF analysis (Thermo Scientific ARL Perform'X Sequential XRF) powder samples were first treated for two hours at 950 °C in a furnace (Nabertherm B 150) to remove organic compounds and CO_2, and were then mixed with Fluxana (FX-X50-2, Lithium tetraborate 50 % / Lithium metaborate 50 %) in a ratio of 1:10 in order to lower the melting temperature. XRF analysis was performed with program OXAS on melted disks, while data was characterized using the program UniQuant 5.

The specific surface area (BET) of the milled glass and stone wools was determined by nitrogen adsorption at 77K over a relative pressure range of 0.05-0.3 (Micrometrics ASAP 2020, Micrometrics, Norcross, GA, USA). Before BET analysis samples were heated at 105 °C for 24 hours and degassed to 10^{-3} Torr (Micrometrics Flowprep equipment, Micrometrics, Norcross, GA, USA).

Scanning electron microscopy (SEM; Jeol JSM-IT500) with a tungsten filament cathode as an electron source was investigated under high vacuum conditions. Samples were dried at 40 °C in a vacuum chamber before observation.

3.   Results and discussion

3.1.   *Chemical and mineralogical analysis*

The data showing loss of ignition at 550 °C and the specific surface area of the different types of mineral wool is presented in Table 2. Ignition loss is higher in glass wool suggesting that glass wool contains more organic species [Heiri, Lotter, & Lemcke, 2001]. BET analysis demonstrates similar results for glass and stone wool; however, glass wool has a bigger specific surface area. XRF analysis shows the presence of different oxides in glass and stone wool (Table 2). Mineral wools are mainly composed of silicon, aluminum, calcium, magnesium and iron oxides. The glass wool contains 54.0 wt % of SiO_2 and 12.1 wt % of Na_2O, which is slightly lower than in typical glass wool, but 8.7 wt % of Al_2O_3 and 7.9 wt % of MgO which is a little higher than in typical glass wool collected from Termit d.d. [Horvat, Češnovar, Pavlin, & Ducman, 2018]. In contrast, the stone wool has a composition as expected. In this study, it is assumed that the glass wool may not be pure glass wool but contains some proportion of stone wool. During the collection of waste glass wool, it is hard to separate glass and stone wool completely, and the final material could be a mixture of both.

Table 2: Chemical composition of glass and stone wool, loss of ignition at 550 °C determined by the gravimetric method and BET analysis for glass and stone wool.

Chemical component (%)	Glass wool [GW]	Stone wool [SW]
Na_2O	12.1	3.9
SiO_2	54.0	43.7
Al_2O_3	8.7	16.0
MgO	7.9	11.6
Fe_2O_3	3.0	5.3
CaO	12.5	16.8
Dry matter content (%)	99.9	99.9
LOI 550 °C (%)	5.38	4.62
BET (m^2/g)	0.4948	0.3722

XRD analysis of precursors is shown in Figure 1. As expected, the curves show that both types of wool are almost completely vitrified and amorphous. Traces of minerals are present in the mineral wools: quartz, calcite and dolomite in the glass wool (Figure 1a), and quartz and dolomite in the stone wool (Figure 1b).
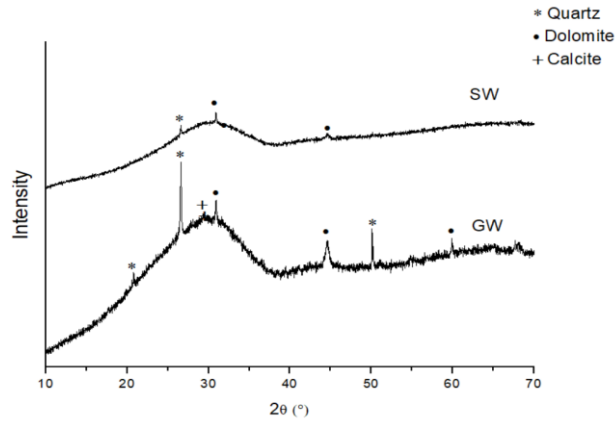

Figure 1: XRD of precursors glass wool and stone wool.
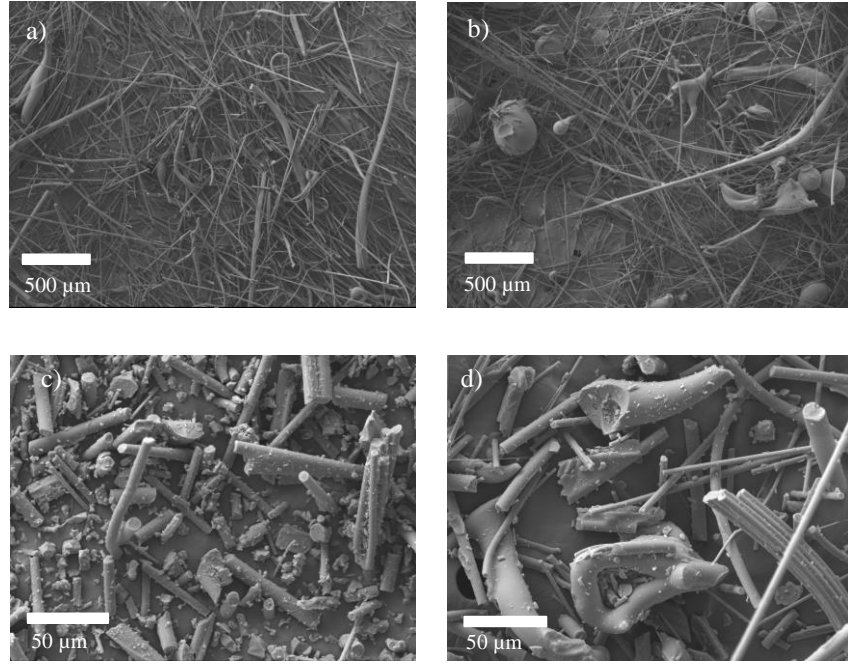
3.2. *Microstructural analysis*

Figure 2: SEM pictures before pulverizing the glass (a) and rock wool (b). After grinding, samples were sieved to below 63 μm. Figure c) represents glass wool with different elongated particles, while in figure d) many random-shape particles are also observed.

SEM pictures before (a and b) and after (c and d) grinding the glass and stone wool are presented in Figure 2. Samples are composed of different fibers which vary in size and diameter, cylindrical rods and irregular random-shape particles. Although all material was sieved below 63 μm, some particles are bigger. Some particles with a diameter smaller than 63 μm may enter perpendicularly during the sieving process (Figure 2, c and d).

The SEM of mineral wool waste after alkali activation is presented in Figures 3 (glass wool) and 4 (stone wool). The AAMs differ according to which activator was used. When NaOH was used almost no matrix is formed (GW1 and SW1). Na-water glass as an activator gave a porous structure with both mineral wools (GW2-GW4 and SW2-SW4). However, fewer pores are present in the samples GW4 and SW4. It can be seen that initial fibres of glass wool (Figure 3) are better incorporated into the matrix compared to stone wool fibres (Figure 4), no matter which activator was used. μm
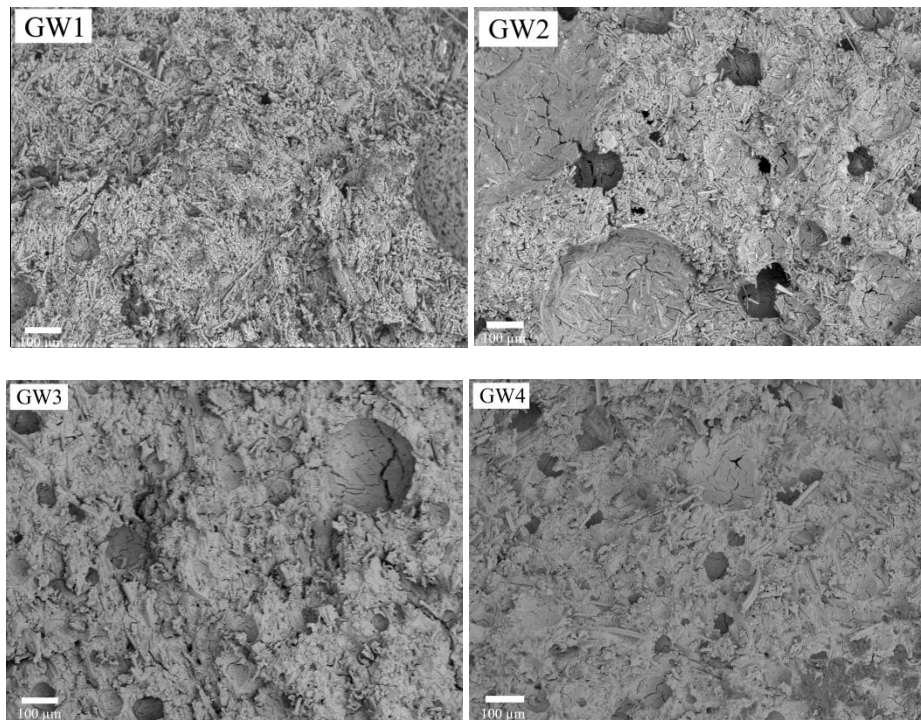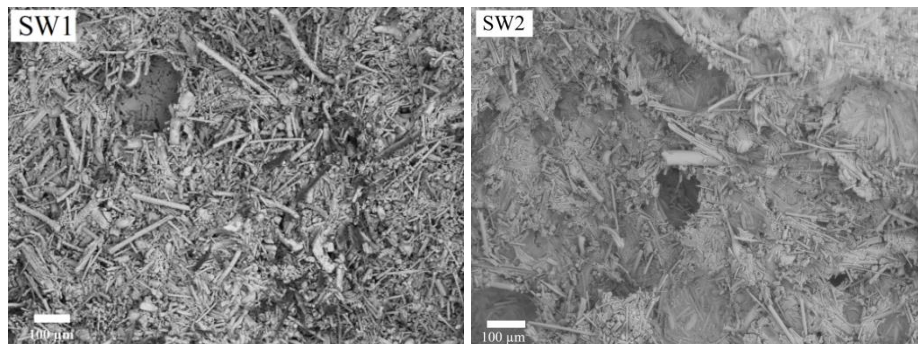
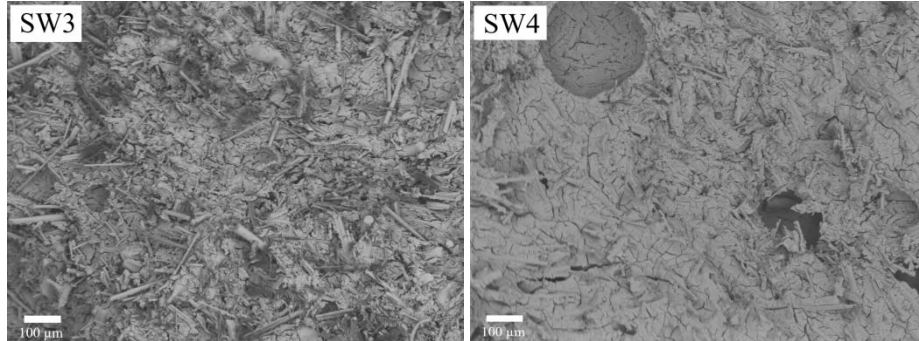**Figure 3:** SEM micrographs of the matrix from alkali activated glass wool.

Figure 4: SEM micrographs of the matrix from alkali activated stone wool.

3.3. Mechanical analysis

The glass and stone wool were alkali-activated using NaOH, Na-water glass or a combination of the two. The results for compressive and flexural strength of the different AAMs are presented in Figures 5 and 6. All samples were cured for three days at 40 °C. The results show better compressive strengths for the mixtures prepared with glass wool, compared to those using stone wool. However, the strength is dependent on the type of activator. If using only NaOH (GW1 and SW1), the results for compressive strength are below 11 MPa for glass wool and below 9 MPa for stone wool. Using Na-water glass as an activator or a combination of both activators (NaOH and Na-water glass) (significantly) improves the compressive strength, giving values above 30 MPa for glass wool (GW2-GW4, Figure 5) and 20 MPa for stone wool (SW2-SW4, Figure 6). The addition of water slightly decreases the compressive strength in the case of glass wool (26.7 MPa, GW2 in Figure 5), while compressive strength reduces significantly the stone wool (below 15 MPa , SW2 in Figure 5). The highest compressive strengths were achieved in mixtures GW4 and SW4, eliciting values of 34.7 and 29.1 MPa, respectively.

The flexural strength of all tested mixtures was below 10 MPa when cured at 40 °C for three days. In glass wool, the flexural strength was 3.3 MPa when using NaOH as an activator compared to 7.8 MPa when Na-water glass was used; for stone wool these values were 3.9 and 5.9 MPa respectively, indicating a higher flexural strength when Na-water glass is used. Flexural strength increases if both activators are used simultaneously (around 9 MPa) but decreased with the addition of water (6.9 MPa for glass and 4.3 for stone wool), similar to compressive strength.
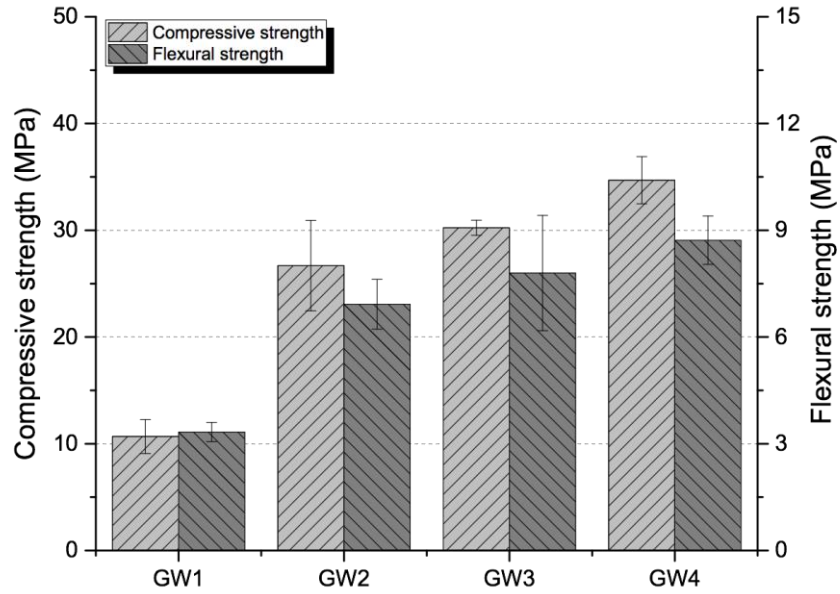
Figure 5: The results of compressive and flexural strength for different alkali-activated glass wool materials after 3 days of drying at 40 °C.
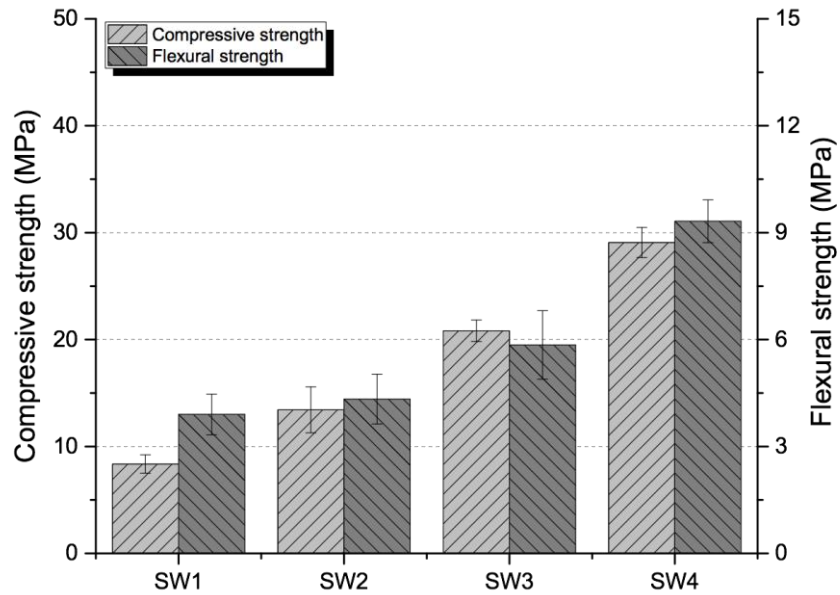

Figure 6: The results of compressive and flexural strength for different alkali-activated stone wool materials after 3 days of drying at 40 °C.

Data regarding the density of different mixtures is available in Table 3. The density of AAMs depends on the type of alkali activator. If NaOH is used (GW1 and SW1), the density is between 1.2-1.6 kg m^{-3} compared to a density of more than 1.8 kg m^{-3} in the AAMs prepared using Na-water glass (GW3 and SW3). Using both activators (a combination of NaOH and Na-water glass) for alkali activation results in a slightly lower density compared to the Na-water glass alone (GW4 and SW4). However, with the addition of water, density decreased (GW2 and SW2).

Table 3: The density of each prepared sample calculated after three days at 40 °C.

Samples	Density (kg m^{-3})
GW1	1.52
GW2	1.57
GW3	1.86
GW4	1.83
SW1	1.23
SW2	1.52
SW3	1.87
SW4	1.83

Since manufacturing AAMs at room temperature would be favourable for the project Wool2Loop (to avoid the energy required for curing at elevated temperatures), in the next step, the most promising mixtures of glass (GW4) and stone wool (SW4) were chosen. Compressive and flexural strength was measured after 1, 3, 7, 14 and 28 days of drying at room temperature. The results are shown in Figure 7. After three days of curing at room temperature, samples were still too soft to be demoulded. After one week, the compressive strength for stone wool was below 10 MPa. In the case of glass wool, it was not possible to measure the compressive strength after one week due to the fragility of the material. Curing at room temperature for 28 days (Figure 7) results in better compressive strength for stone wool (40.5 MPa) compared to glass wool (30.0 MPa), whereas when samples were cured at 40 °C for three days (Figures 5 and 6) better compressive strengths were observed for glass wool (34.7 MPa) compared to stone wool (29.1 MPa). The flexural strength after one week at room temperature ranged between 2 and 3 MPa for both types of AAM. After 28 days of curing, the flexural strength ranged between 14 and 15 MPa, which is better than the flexural strength of around 9 MPa which was achieved after drying the AAMs for three days at 40 °C.
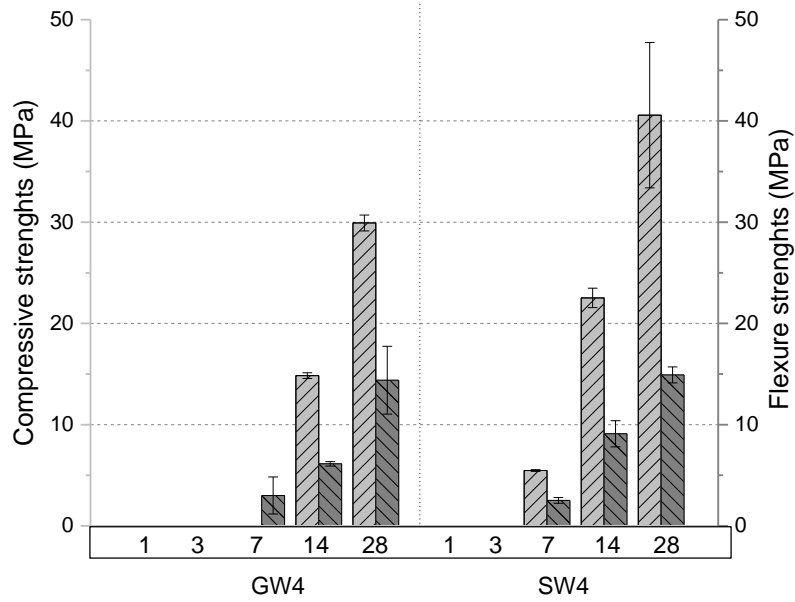
Figure 7: The compressive and flexural strengths of alkali activated GW4 and SW4 measured after 1, 3, 7, 14 and 28 days, dried at room temperature.

4.   Conclusions

Glass and stone wool waste were used for the preparation of AAMs. Both types of wool were milled, ground and sieved under the same conditions, and the effects of different activators on the final compressive and flexural strengths has been studied. NaOH as an alkali activator results in low compressive and flexural strength, whilst using Na-water glass, or a combination of both activators, significantly improves the strength properties. Compressive and flexural strength also depend on the curing temperature, whereby a higher temperature (40 °C) results in faster drying and better compressive and flexural strengths after three days compared to curing at room temperature. However, the compressive strengths of glass wool after three days at the higher temperature are comparable to those measured after drying for 28 days at room temperature. After 28 days, stone wool shows better compressive strength versus glass wool with respect to the three days curing at 40 °C. The microstructure indicates differences between the matrices of glass and stone wool, whereby the glass wool is more reactive. The matrix depends on the type of activator used. Where a mixture of both NaOH and Na-water glass is used, more mineral wool reacts with the activator and forms an amorphous gel.

This study indicates the potential secondary use of mineral wool waste for different building/ construction materials. Due to the huge amount of waste, using mineral wool waste as a raw material for different applications is an important step for the circular economy.

Acknowledgements

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