SPECIAL ISSUE ARTICLE

Preparation of façade panels based on alkali-activated waste mineral wool, their characterization, and durability aspects

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Abstract

Mineral wool is a widely used insulation material and one of the largest components of construction and demolition waste, yet it mainly ends up in landfills. In this work, we explored the potential recycling of waste stone wool in the pilot production of alkali-activated façade panels. The current work shows mechanical properties, SEM-EDS, and mercury intrusion porosimetry analyses for three different mix designs used for the preparation of façade panels. They are all composed of waste stone wool and differ in the amount of co-binders (local slag, lime, metakaolin, and/or fly ash) selected by the preliminary studies. In this study, co-binders were added to increase early strength and improve the mechanical properties and freeze–thaw resistance. The mechanical properties of each were measured up to 256 days, different durability tests were executed, and, by evaluating the mechanical properties, microstructure, and workability of the mortar, the most suitable mix was selected to be used for the pilot production. In addition, the leaching test of the selected mixture showed no exceeded toxic trace elements and therefore got classified as non-hazardous waste after its use.

KEYWORDS
Alkali activated materials, Façade panels, Mineral wool waste, Recycling

1 | INTRODUCTION

Mineral wool is divided into glass (GW), stone (SW), and slag wool, among which GW and SW wool are commonly used insulation materials. In Europe, about 2.55 mt of mineral wool was produced in 2020 and therefore there is a lot of waste mineral wool that usually ends up in landfills, where, due to its low density, consumes a lot of space.[1] Considering the data for waste mineral wool in Austria (EU country),[2] the calculated amount for Slovenia, taking into account the population number, could be in the range 5–7.5 kt/yr. Therefore, its reuse is crucial, whether being used for further wool production or in the alkali activation process. Mineral wool is almost 100% amorphous and therefore is a promising material for the alkali activation technology. The chemical composition of SW and GW fibers differ, where GW is composed of about $\sim$60−65 wt% SiO_2, $\sim$16 wt% Na_2O, and $\sim$7 wt% CaO, whereas SW consists of about 40−45 wt% SiO_2, 16−18 wt% Al_2O_3, 16−18 wt% CaO, and 9−12 wt% MgO.[3] Amorphous material dissolves in alkali solutions and rearranges into a structure formed of SiO_4 and AlO_4 tetrahedra. Since AlO_4 tetrahedra are negatively charged, positive ions from alkali solutions (such as Na^+ from NaOH or sodium silicate) are used to balance the negative aluminium charge.[4] The precursors most commonly used in the alkali activation

technology are metakaolin (M), fly ash (FA), and blast furnace slag.[5] In recent years, mineral wool[6] and other waste materials containing a higher proportion of amorphous phase (e.g., waste glass) have also been used.[7–9] In the previous study, alkali-activated samples using waste SW and a combination of sodium silicate/NaOH as alkali activators show compressive strengths below 10 MPa after 7 days of curing at the room temperature and below 5 MPa when using GW as a precursor. Therefore, a higher temperature (e.g., 40°C for three days) is recommended to accelerate early strength.[10] However, the study of Yliniemi et al. shows that one day at 40°C is enough for SW and GW when using sodium silicate as an alkali activator, but compressive strength < 3 MPa was measured when GW was alkali-activated by NaOH.[3] To apply a less energy-intensive process in the foreseen pilot production of façade panels, instead of increasing the temperature of curing, the addition of co-binders was preferred. Our previous study indicates acceleration of early strength for alkali-activated SW and GW samples at the room temperature when adding 20 wt % of co-binders such as slags or M.[11]

This paper thus represents the results of prepared mix designs for façade panels from waste SW, with slag, lime, M, and FA added as co-binders that are cured at the room temperature: (1) to increase early strength and thus consume less energy, (2) to improve the mechanical properties and freeze–thaw resistance, and (3) to re-use as much as possible waste SW in the mix design. All the work is part of the WOOL2LOOP project,[12] which is concerned with the development of various building materials (e.g., façade panels, pavement slabs, acoustic panels, and dry mixes) from waste mineral wool.

2 | EXPERIMENTAL

Waste mineral wool (stone wool, SW), lime (L), M, electric arc furnace slag (EAFS), and FA were used for the preparation of façade panels. SW and EAFS were pre-treated before use. EAFS was dried and then milled for 1 h. SW was dried at 105°C for 24 h in a drying oven and then put in a homemade grinding machine (with the rate of 10 kg of milled material/hour). The milled waste mineral wool was then sieved through 500 μm and 63 μm sieves. All material passed through the 500 μm mesh, while slightly more than 60% passed through the 63 μm mesh. However, particles passing through 63 μm mesh were also bigger due to the fibrous structure of mineral wool. X-ray fluorescence (XRF) analysis was used to determine the chemical composition of the precursors used (Table 1).

The mix designs for the three façade panels prepared in the present study are shown in Table 2. Alkali-activated mortars were prepared using SW with co-binders (L, M, EAFS, and/or FA), alkali activators NaOH (Donau Chemie

Ätznatron Schuppen, EINECS 215-785-5, Austria) and Na-silicate (with a 12.8% mass percentage of Na_2O, and 29.2% mass percentage of SiO_2 received from mining company Termit d.d., Slovenia). For preliminary testing, the same amount of Na-silicate and NaOH was used in all mixtures, whereas the amount of dry mass for SW, L, M, EAFS, and FA varied as shown in Table 3.

To test the mechanical properties, the mortars were moulded into silicone prisms $80 \times 20 \times 20$ mm³ in size, while the façade panels were prepared in a silicone mould with dimensions of $400 \times 400 \times 50$ mm³. Compressive and bending strengths were measured using a compressive and bending strength testing machine (ToniTechnik Toni-NORM) after 1, 3, 7, 14, 28, 56, 128, and 256 days at the room temperature.

Mix design C was modified and additional façade panels were prepared to test the suitability of different SiO_2/Na_2O modules by using 2.2, 2.3, and 2.5. In addition, polypropylene fibers of 10 mm size in length were used in the experiments.

The pore size of alkali-activated material was determined by mercury intrusion porosimetry (MIP) using Micromeritics®Autopore IV 9500 equipment (Micromeritics, Norcross, GA, USA). All samples were cured 56 days at room temperature and dried at 70°C for 24 h before being analyzed.

Durability was assessed by freeze–thaw (ETAG 004) and leaching tests (EN 12457-2). The workability of the material was estimated based on the slump test according to EN 1015-3.

Rietveld refinement with an external standard (pure Al_2O_3 crystal) was performed to determine the amorphous phase in the façade panels, using X'Pert Highscore plus 4.1 on the X-ray diffraction (XRD) data.

The microstructure of the alkali-activated material (AAM) samples was investigated by low-vacuum scanning electron microscopy (SEM; JSM-5500LV, Jeol, Tokyo, Japan) in BED-C mode and energy dispersive spectroscopy (EDS, Link Pentafet, Oxford Instruments).

3 | RESULTS

3.1 | Mechanical properties

Many different mix designs were prepared, but due to poor frost resistance, only a few were suitable for further testing on a larger scale, and the results are presented in this paper. Based on the preliminary results presented in Table 3, mixes suitable for the production of façade panels were selected. An important parameter for the selection of a suitable mix design for the production of façade panels was the possibility of demoulding the samples after one day and the freeze–thaw resistance of the produced

TABLE 1 Chemical composition of stone wool (SW), lime (L), metakaolin (M), electric arc furnace slag (EAFS), and fly ash (FA) with loss on ignition at 950°C (LOI), determined by the gravimetric method

Chemical composition of precursors	SiO_2 (wt%)	Al_2O_3 (wt%)	Na_2O (wt%)	CaO (wt%)	MgO (wt%)	Fe_2O_3 (wt%)	LOI (950°C)
Waste stone wool (SW), Slovenia	38.4	17.2	2.00	16.1	11.6	6.45	4.60
Lime (L), Slovenia	1.86	2.05	0.30	68.2	2.07	0.10	24.8
Metakaolin (M), Germany	68.1	25.2	0.09	0.45	0.16	2.21	2.25
Local slag (EAFS), Slovenia	13.7	5.20	0.28	27.9	23.3	4.64	20.5
Fly ash (FA), Slovenia	44.8	23.0	1.19	12.4	2.80	10.7	0.51

TABLE 2 Mix designs for façade panels presented in terms of the precursors, activators, and aggregates (0–2 mm) added

Laboratory mixture label	Ratio SW: Na-silicate: NaOH: L: M: EAFS: FA: aggregate
A	72: 90: 1: 0: 0: 9: 9: 30
B	72: 90: 1: 4.5: 13.5: 0: 0: 30
C	63: 90: 1: 1.8: 16.2: 9: 0: 30

mix. Since the façade panels will be placed outdoors, each prepared mixture was subjected to 150 freeze–thaw cycles and only those that were resistant were then selected for further production of larger $40 \times 40 \times 3$ cm^3 panels. The waste SW could only be demoulded after three days and the mechanical properties were measured after 7 days. The addition of a co-binder (L, M, EAFS, and FA) showed frost resistance only in the case of the addition of L (10 wt%) (the values for these results are from the previous study[11]). Most alkali-activated samples could be demoulded after one or two days, and mechanical properties were measured after three days for all mixtures except in the case of FA. Due to the fast hardening, the amount of added L was reduced (1–5 wt% L was used). The amount of co-binder(s) added was in the range of 10–30 wt% and varying the mass fraction of SW, L, M, FA, and EAFS resulted in different mechanical properties (Table 3). The addition of M as a source of aluminium improved the workability of the mixtures, but mixture containing 5 wt% L hardened during moulding ("flesh setting"). Although this sample was frost resistant, it was not useful for further production. The mixture containing 80 wt% SW, 4 wt% L, and 16 wt% M had better workability due to slower hardening, and was frost resistant. A higher percentage of L improved the compressive

and bending strengths after three days of curing as well as the frost resistance of the prepared mixtures as can be seen in Table 3, but decreased the workability of the mortar. In order to prolong the setting time and improve the workability, the percentage of L was decreased and the addition of EAFS and FA was tested (mixtures with 70 or 75 wt% SW, 2 wt% L, 18 wt% M and 5 or 10 wt% FA and EAFS). The mixtures with FA were not frost resistant, while the alkali-activated mixture with 10 wt% EAFS was suitable for further use. Mixtures with the addition of 1 wt% L and various wt% M were not suitable due to low mechanical properties after three days and lack of frost resistance. Another mix design, also selected for further production of larger panels, contained 10 wt% FA and 10 wt% EAFS. The sample exhibited some delamination of the top layer immediately after immersion in water, but the bulk of the sample was frost resistant for up to 150 cycles.

Based on preliminary results shown in Table 3, three different mixes (the composition shown in Table 2) were selected for the production of the façade panels (selected mix designs are bolded in Table 3), for which targeted values of 30 MPa and 10 MPa were set for compressive and bending strength, respectively. Figure 1 shows the mechanical properties of the selected mixes, labeled A, B, and C. After 28 days, values of compressive strength were above 30 MPa in mixtures B and C and close to 30 MPa in mixture A. The bending strength was above 10 MPa in all façade panels after 28 days. After 256 days, the compressive strength slightly decreased in sample A; while the plateau had not yet been reached in samples B and C, the strength exceeded 50 MPa in all specimens. After 256 days, the bending strength increased in samples A and C (above 20 MPa), while in mix B the bending strengths after 28 and 256 days were similar (around 16 MPa).

TABLE 3 The results of compressive and bending strengths for mixtures prepared from SW and co-binders cured 3 days at room temperature. In the last column are the results of frost resistance (150 freeze–thaw cycles) for each mixture performed after 28 days of curing at room temperature

Tested mix design	Compressive strength (MPa)	Bending strength (MPa)	Freeze–thaw resistance 150 cycles
SW	/*	/*	No
80 wt% SW + 20 wt% EAFS	4.0±0.3	2.1±0.4	No
80 wt% SW + 20 wt% M	2.6±0.7 (0)	1.3±0.2 (0)	No
90 wt% SW + 10 wt% L	16.5±1.5	3.8±0.6	Yes
80 wt% SW + 20 wt% FA	/*	/*	No
90 wt% SW + 8 wt% M + 2 wt% L	4.7±0.3	1.8±0.1	No
85 wt% SW + 13 wt% M + 2 wt% L	7.5±0.4	4.3±0.4	No
85 wt% SW + 14 wt% M + 1 wt% L	2.5±0.1	1.6±0.1	No
80 wt% SW + 19 wt% M + 1 wt% L	3.1±0.2	1.6±0.1	No
80 wt% SW + 15 wt% M + 5 wt% L	22.2±1.8	9.2±0.2	Yes
80 wt% SW + 18 wt% M + 2 % L	12.7±0.9	5.4±0.1	No
80 wt% SW + 16 wt% M + 4 wt% L	**16.2±1.9**	**6.6±0.8**	**Yes**
80 wt% SW + 10 wt% FA + 2 wt% L + 8 wt% M	6.2±0.4	2.8±0.2	No
75 wt% SW + 19 wt% M + 1 wt% L + 5 wt% FA	2.3±0.1	/	No
70 wt% SW + 18 wt% M + 2 wt% L + 10 wt% FA	6.2±0.9	1.9±0.2	No
80 wt% SW + 10 wt% FA + 10 wt% EAFS	**3.9±0.2**	**1.4±0.1**	**Yes**[**]
75 wt% SW + 19 wt% M + 1 wt% L + 5 wt% EAFS	3.8±0.1	1.3±0.1	No
70 wt% SW + 18 wt% M + 2 wt% L + 10 wt% EAFS	**7.2±0.1**	**2.2±0.1**	**Yes**

Notes:

*Mixtures are too soft to be measured.

**Upper layer delaminated when immersing in water, otherwise frost resistant.

3.2 | Microstructure, porosity, and the amount of the amorphous phase

The SEM micrographs in Figure 2 show the microstructures of the three different mix designs, A, B, and C. Mixture design B is the most porous, having visible microcracks. Compared to it, mixtures A and C show few or no microcracks. In all cases, many unreacted particles are seen, where larger particles represent unreacted slag and quartz sand, while the small grey elongated particles are unreacted waste SW fibers. It follows that also the dissolution of the SW is only partial. However, waste SW alone could be alkali-activated as confirmed in our previous study,[10] but it takes one week to be able to demould and measure bending and compressive strengths and therefore co-binders such as M, slag, and L were added. In order to see the differences in dissolution and formation of the matrix, we performed SEM-EDS point analyses and calculated the average Si/Al, Si/Na, Ca/Al, and Na/Al ratios.

The Si/Na ratios are similar, while the Si/Al ratio is highest in mixture A, followed by B, with sample C having the lowest ratio. In samples B and C, Al enters the system due to the addition of M, and hence the Si/Al ratio is lower than in the A samples, as FA contains less Al than M. The EAFS added in the mixture contains a much lower amount of Al compared to FA. The Ca/Si ratio is similar in all cases, while the Na/Al ratio is less than 1 in all cases, implying that there should be no efflorescence. This, however, is not the case, as all samples show at least a minor amount of efflorescence after some time. The amount of amorphous phase calculated by Rietveld refinement shows that the amount of the amorphous phase was similar in all three samples, at about 80%.

TABLE 4 Ratios of Si/Na, Si/Al, Ca/Si, and Na/Al in each of the three mix designs (A, B, and C), calculated based on SEM-EDS point analyses. In addition, the amount of the amorphous phase and porosities are shown for each of the mixtures

	Si/Na	Si/Al	Ca/Si	Na/Al	Amorphous phase (%)	Porosity (%)
Mixture A	1.92	8.03	0.14	0.24	83.7	21.2
Mixture B	2.02	4.87	0.17	0.42	79.6	30.0
Mixture C	2.04	3.78	0.16	0.88	77.1	25.4
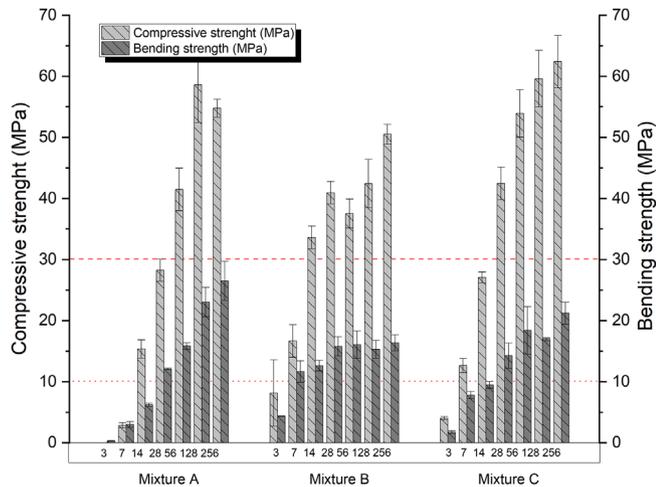
FIGURE 1 Mechanical properties of mixtures A, B, and C after 1, 3, 7, 14, 28, 56, 128, and 256 days. All mixes were cured at room temperature

To identify differences between the façade panels, microporosity was determined using MIP. The percentage of pores in the mixes A, B, and C are shown in Table 4. Sample B has the highest porosity (hence the lowest mechanical strengths), followed by mixture C and then mixture A with the lowest porosity (21.2%). Pore size distributions are shown in Figure 3, where it can be seen that all three mixes contain similar pore size distributions, with different proportions in the different ranges. All three mixtures have a similar proportion of pores sized between 0.1 and 1 μm, but the percentage of pores between 10 and 100, 2 and 10, and 1 and 2 μm slightly vary. Compared to mixtures A and B, for example, mixture C has a similar proportion of pores between 2 and 10 μm to mixture A and smaller proportion of pores than B, a larger proportion of pores sized between

1 and 2 μm, and a smaller proportion of larger pores ranging from 10 to 100 μm.

3.3 | Durability tests

Since façade elements from this study were meant to be used in a real case study on the façade, all three samples were exposed to different conditions that could simulate the behavior of the panels when they are placed outdoors. Freeze–thaw tests were therefore conducted.

3.3.1 | Freeze–thaw test

For this test, we used a modified method according to ETAG 004. After curing for 28 days at room temperature, all specimens were immersed in water (at room temperature) for 24 h. The samples were frozen at −15°C and thawed at 15°C. The results presented in Figure 4 show the freeze resistance of the façade panels after 150 cycles. Mixtures B and C were resistant to freezing (Figure 4E,F), while mixture A (Figure 4D) was not suitable for further testing due to surface damage, especially at the corners.

3.3.2 | Selection of a façade panel for pilot production

For the pilot production, only mixtures of suitable workability can be used, so the slump test was performed. This test indicates the water to cement ratio required for the slump desired, which could be affected by many factors, including the material properties, mixing methods,
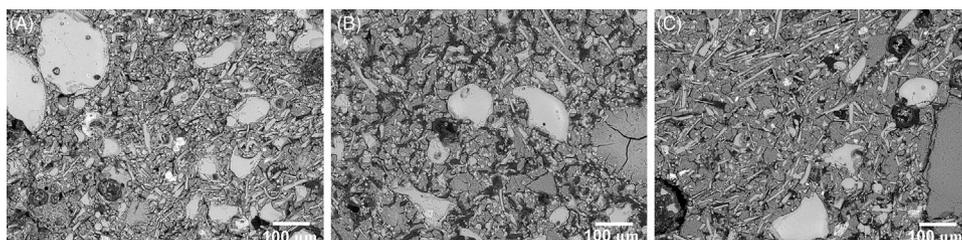

FIGURE 2 Scanning electron microscopy (SEM) microstructures of the three mixtures, A, B, and C 150× magnified
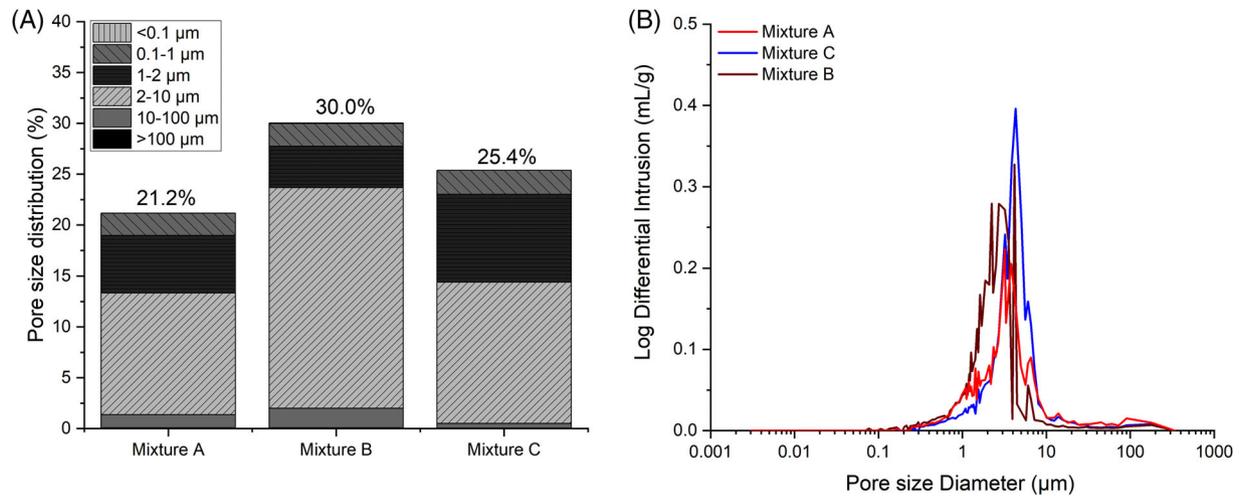
FIGURE 3 Pore size distribution of the façade panels A, B and C. Graph (A) represents column stacked graph of pore size distribution for mixtures A, B, and C, and graph (B) shows differential mercury intrusion porosimetry vs. pore size diameter curves
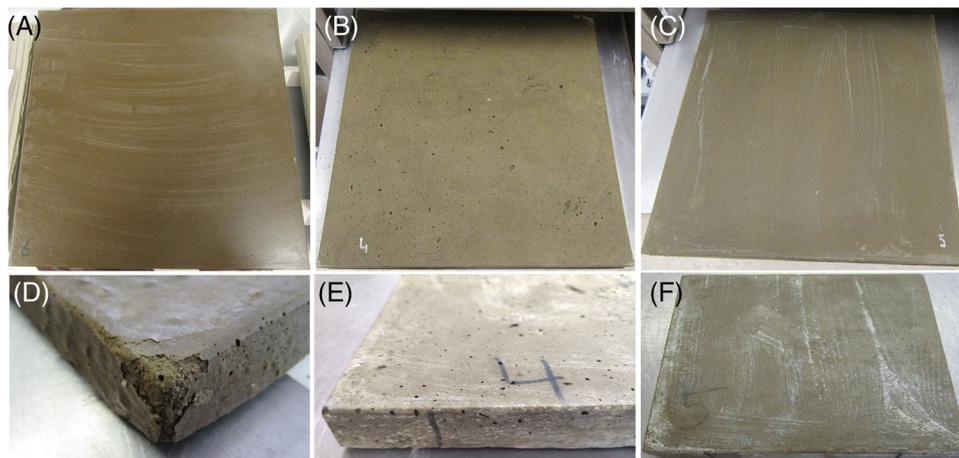

FIGURE 4 Façade panels before (A–C) and after 150 cycles of the freeze–thaw test (D–F). Samples (A) and (D) represent mixture A, (B,E) mixture B; and (C,F) mixture C

batching, and admixtures. The slump test values (flow table spread) are given in Table 5, showing that mixes A and C have a suitable slump, while mix B does not. According to the results from Table 5, we excluded mixture B (the

flow table spread showing good workability of prepared mortars is normally in the range 165–185 mm, while the spread showed for mixture B is much smaller and thus it is not suitable) and mixture A (not resistant to freezing, and not demouldable after 1 day) from further processing, and selected mixture C for the production of façade panels.

TABLE 5 Comparison of different parameters important for the selection of an appropriate mix design for further pilot production

Façade panels	Mixture A	Mixture B	Mixture C
Freeze–thaw resistance	–	+	+
Slump test (workability) (mm/mm)	169/171	135/140	172/183
Demolding time (days)	3	1	1

3.4 | Leaching tests

After selecting mixture C as a suitable mixture for the preparation of façade panels, we tested the leachability of this panel to evaluate its environmental aspect. The standard procedure EN 12547-2 was used in this experiment. The toxic trace elements leached in relation to legislation are presented in Table 6. All values were compared to landfill legislation (for inert and non-hazardous waste) as well

TABLE 6 The results of leached toxic elements according to standard procedure EN 12457-2, alongside values given in legislation

mg/kg	As	Ba	Cd	Co	Cr	Cu	Hg	Mo	Ni	Pb	Se	Sb	Zn
Sample C	0.54	0.25	0.003	0.04	0.65	2.60	0.005	1.69	0.83	0.09	0.16	0.04	0.15
Inert waste*	0.5	20	0.04	/	0.5	2.0	0.01	0.5	0.4	0.5	0.1	0.06	4
Non-hazardous waste*	2	100	1	/	10	50	0.2	10	10	10	0.5	0.7	50
New product from recycled material**	0.1	5	0.025	0.03	0.5	0.5	0.005	0.5	0.4	0.5	0.6	0.3	2

Notes:

*Decree on waste landfill (Uredba o odlagališčih odpadkov: Uradni list RS, št. 10/14, 54/15, 36/16, 37/18 in 13/21).

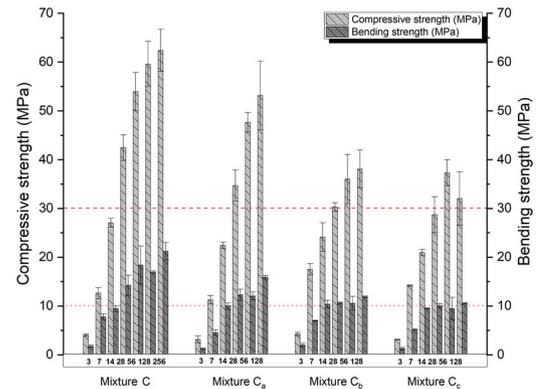**Decree on waste (Uredba o odpadkih: Uradni list RS, št. 37/15, 69/15 in 129/20).

TABLE 7 Mixtures C, C_a, C_b, and C_c with different sodium silicate ratios and the diameters measured in the slump test

Mix design	Na-silicate (SiO_2/Na_2O ratio)	Slump test (mm)	Addition of microfibers (PP)
C (original mix design)	2.3	172/183	–
C_a	2.3	180/178	+
C_b	2.2	175/180	+
C_c	2.5	180/184	–

FIGURE 5 Mechanical properties of mixtures C, C_a, C_b, and C_c after 1, 3, 7, 14, 28, 56, and 128 days. All mixtures were cured at room temperature

as to the decree on waste. As panels from the final selected mixture are made from waste mineral wool and local slag, which are classified as waste materials or by-products, the comparison of the data with the legislation for decree on waste should be carried out, as the waste material is used in the production of the new building materials. The results in Table 6 show that As, Co, Cr, Cu, Mo, and Ni (slightly) exceeded the limits for the production of new recycled materials, as well as the limits for the inert waste, but they comply with the values for non-hazardous waste. Further testing is required to confirm and to understand how the mix design could be modified to reduce the leaching of these elements.

3.5 | Modification of the mix design for the façade panels

Due to the slight curvature of the façade panels made using the selected mix design, three additional modifications were prepared to minimize the curvature (mixes C_a, C_b, and C_c, Table 7), but at the same time to not affect desired properties. Mixture C_a was the same as C but with the addition of polypropylene (PP) microfibers, which do not react with alkali. In mixture C_b lower SiO_2/Na_2O modulus was used and PP microfibers were added to the mortar, while in C_c no PP microfibers were added and the modulus was higher compared to the other mixtures. The workability of all prepared mortars was good as confirmed by

the slump tests table 7. The results show a similar spread in all cases, indicates no influence—due to the addition of microfibers or due to the slight changes in SiO_2/Na_2O modulus.

The mechanical properties of the prepared mixtures are shown in Figure 5. Compressive and bending strengths decrease for all modified mixtures compared to the original C mix design, and after 128 days the highest values of bending and compressive strength were seen in the mix design without added microfibers (C_c). An increase in compressive and bending strengths could be seen in sample C_a, while in C_c compressive strength had not increased after 128 days and the values of bending strength were similar to those at 14 days of curing at the room temperature. It follows that none of the modified mix designs improved the mechanical properties.

All three panels were subjected to freeze–thaw exposure and none of them was completely resistant, as after 150 cycles of freeze–thaw exposure the top layer showed surface damage, but the bottom layer did not. This shows that the original mix design had the optimal properties in terms of freeze–thaw resistance, workability, and mechanical properties. Additional testing considering curing conditions is, however, required to avoid curvature.

4 | CONCLUSION

This study represents the preparation and selection of the most appropriate mix design for the pilot production of façade panels based on waste mineral wool. As part of the WOOL2LOOP project, the amount of waste mineral wool in the samples is as high as possible (70% based on dry mass, studied in previous research), considering the requirement for demoulding after one day. We selected among three different mix designs, mixture A composed of 80 wt% of SW, 10 wt% of FA, and 10 wt% of EAFS, mixture B contains 80 wt% of SW, 16 wt% of M, and 4 wt% of L, and mixture C consisted 70 wt% of SW, 18 wt% of M, 2 wt% of L, and 10 wt% of EAFS. According to the results above, we excluded mixture A because it was not freeze–thaw resistant (and did not demould after one day), and mixture B because of its poor workability. For pilot production, mix design C was chosen.

Façade panels (from mixture C) cured at room temperature for 28 days reached maximal compressive and bending strengths of 42 and 14 MPa, respectively. The mechanical strengths continued to increase until 256 days. Specimens produced using the optimal mix design were frost resistant up to 150 freeze–thaw cycles. Since the façade panels are designed to be installed outdoors, an environmental assessment was executed by conducting leaching tests to determine toxic trace elements, which showed that the permissible values for non-hazardous waste were not exceeded. Additional studies are needed to avoid the curvature of the façade panels.

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