



Invited lecture/Review

Green Transition in Slovenian Building and Civil Engineering Industry: 10 Years of Research on Alkali-Activated Materials and Alkali-Activated Foams

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Abstract:

The building and civil engineering industry yearly causes more than 40% of man-made CO₂ and consumes raw materials for two-thirds of Mont Everest. To decrease the carbon footprint and consumption of raw materials, alkali-activated materials (AAMs) are researched as an alternative to conventional building and civil engineering products like cements, mortar, and ceramics. Ideally, locally available waste materials are used as ingredients: (i) as precursors that react with alkali and form an aluminosilicate network, and (ii) as fillers that get permanently encapsulated and safely stored in AAMs. The addition of gas bubbles and lightweight fillers transforms AAMs into alkali-activated foams and alkali-activated lightweight materials that have the potential to be used as thermal and acoustic insulation materials. Although AAMs are researched worldwide, this review focuses on the state-of-the-art localised solely to Slovenia, particularly on the materials and curing procedures used, as well as on the scientific contribution of the basic research. Besides, the year 2024 marks 10 years of research on alkali activation of raw and waste inorganic materials in Slovenia.

Keywords: Secondary raw materials; Alkali-activated materials; Alkali-activated foams; Low-temperature curing; Microwave curing; Circular economy



1. Introduction

The amount of waste is increasing worldwide with a steep slope (by 2050 increase of solid waste is predicted to be 70%, i.e. $3.4 \cdot 10^9$ t) because of the increased consumption and small recycling rate (less than 20%) (Alves, 2023). At the same time, the non-renewable natural resources are being depleted due to constant (large-scale) excavation and outtake from nature: with the current consumption rate, coal will be depleted in 175 years, and oil and natural gas in 45 years (“Natural Resources Depletion,” 2022).

Globally, mankind excavates more than $500 \cdot 10^9$ t of raw materials every year. This amount can be estimated as two-thirds of the mass of Mount Everest. Half of all excavated natural resources are consumed by the building and civil engineering industry, which causes 40% of global CO₂ emissions (Miller, 2021). However, the need for building and civil engineering will be always present, therefore, the challenges have to be recognised and solved.

Among building and civil engineering products available on the market, conventional cement represents a challenge that has to be addressed among the first. Namely, besides that for the production of cement available on the market raw materials are needed, these raw materials are treated at temperatures up to 1450 °C (Shepherd and Rankin, 1911). Such high energy has an additional negative environmental impact connected to cement. Its use in civil and building engineering is wide because it is used also as an ingredient in other products, like mortar and concrete, making the “cement” issue even more prominent.

Although raw materials are already partially replaced in cement by (fly) ash, municipal wastes, and (ground granulated blast furnace) slag (Nwankwo et al., 2020; Rashad, 2018), the replacement is just partial. Therefore, material, that can be made solely from waste material, and at temperatures below 100 °C, and still have at least similar properties as cement, will sooner or later replace nowadays cement. One of the qualifying options is alkali-activated materials (AAMs) (Roy, 1999) which are being researched worldwide regarding the availability of local secondary materials (waste materials) and are showing promising results regarding mechanical properties (Ding et al., 2016) and durability (Wang et al., 2020).

AAMs can be made either from natural resources or waste materials that contain a sufficient amount of silicon (Si) and aluminium (Al) in amorphous content. Si and Al that are present in precursor dissolve in liquid alkali (usually hydroxides and alkali silicates) and rearrange into an aluminosilicate network (ASN). The rate of dissolution depends on many factors, however, the most important is the size of the particles in the precursor, as is presented in **Figure 1**.

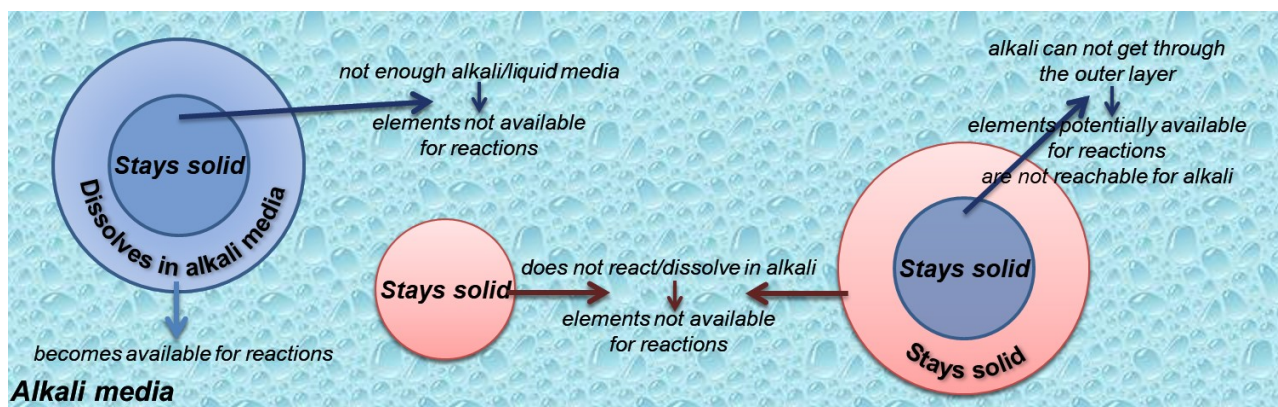


Figure 1. The dissolution potential of powder material containing amorphous Si and Al in alkali liquid. Red does not dissolve in alkali, lighter blue can get dissolved in alkali, darker blue cannot (blue circle on the left: not enough alkali to reach the core blue circle in red on the right: alkali cannot reach blue through the red).

If the alkali cannot reach the part that is prone to dissolve or if there is not enough alkali available, not everything will have the possibility to dissolve. If the addition of alkali is



limited by avoiding the efflorescence, the molar ratio between alkali metal and amorphous Al can be equal to or lower than 1 (Horvat & Ducman, 2019). The reason for aiming the molar ratio Al and alkali metals to be equal to 1 is that alkali metals compensate the extra bond of Al with O in the tetrahedron that is connected through oxygen bridges with other tetrahedra that have in the centre either Si or Al (Škvára, 2007). These tetrahedra are building blocks in a chain, so they represent an inorganic polymer system. If the precursor is metakaolin (calcined clay), this inorganic polymer can be called geopolymer (Davidovits, 1991, 1989), otherwise it should be addressed as AAM.

AAMs can encapsulate many different materials: hazardous and radioactive waste (Shi and Fernández-Jiménez, 2006), organic and inorganic, fibrous and “spherical” (Elijah, 2015), materials that enhance the desired properties regarding the intended purpose of the product and materials that just have to be safely stored. However, due to European legislation, AAMs are not utilised, especially as large-scale building and civil engineering products. On the other hand, in Australia, which has different legislation than Europe, there are already several big-scale projects finished and in operation: (i) aircraft pavements at Brisbane West Wellcamp Airport, Toowoomba (in operation since November 2014; main ingredient was slag) (Glasby et al., 2015), (ii) a 4-story public building, University of Queensland’s Global Change Institute, Queensland (“World’s first public building with structural Geopolymer Concrete – Geopolymer Institute,” 2013), where fly ash and slag were used.

Slovenian researcher, dr. V. Ducman, brought AAMs to Slovenia and designed a mixture from fly ash that was used as a sculpture material by J. Kočica. This sculpture has been permanently placed at the Slovenian National Chemical Institute since 2012/2013 and marks the beginning of art from AAMs in Slovenia. However, the official beginning of scientific research on AAMs was the year 2014, with the Slovenian national project “Mechanisms for the strengthening of different types of ash by means of a geopolymerization process” led by Dr V. Ducman. With this review, we are celebrating 10 years of research on AAMs in Slovenia. The selection of the bibliography was performed using the database SICRIS, however, only studies (or their parts) fully performed in Slovenia, relevant original research and online-available conference papers were taken into account. The works are presented time-wise from older to the newest published to show the progress in the field.

2. Review of 10 years of basic research on alkali-activated materials in Slovenia

After testing the viability of AAMs in larger sculptures, the first research was performed using local low-calcium fly ash as a precursor, and sodium hydroxide (NaOH) and liquid sodium silicate (Na-silicate) as alkalis, cured at room conditions (Kramar & Ducman, 2015). The amounts of alkali and water were varied in the study. Compressive strength showed dependence on water content, i.e., the lower the amount of water, the higher the compressive strength and the fuller the ASN. The highest compressive strength reached in this study was ~50 MPa (Kramar & Ducman, 2015).

With the addition of foaming agents, i.e., solid pulverized Al and (liquid) hydrogen peroxide (H₂O₂), fly ash that was activated with NaOH and Na-silicate, and cured for 24 h at 70 °C before being kept at room conditions, foamed (Ducman & Korat, 2016). The highest addition of Al was 0.2 mass percentage (m%), and H₂O₂ was 2 m%. Total porosity was up to ~60%, which resulted in low mechanical strengths (less than 5 MPa for samples with a density lower than 1 kg/l). However, using H₂O₂ resulted in smaller pores (Ducman & Korat, 2016).

Foaming of fly ash with H₂O₂ (up to 1.5 m%) during alkali activation (using NaOH and Na-silicate) was controlled by stabilizing agent sodium dodecyl sulphate (SDS; up to 4 m%). The mixture was cured for 24 at 70 °C, after that they were kept at room conditions. The addition of a stabilising agent decreased the compressive strength and density because it was successful at stabilizing-separating bubbles in the ASN. At lower additions of H₂O₂, the limit value of density and compressive strength was reached at 4 times higher addition of SDS (Korat & Ducman, 2017).

Upcycling with alkali activation technology was studied on waste mineral wools, i.e., rock and glass wool, both as construction and demolition wastes, delivered to the landfill. Both



types of wool should have been completely amorphous and differ only in chemical compositions, but as they were waste materials, they both had 5 to 10 m% of minerals. Alkali used in the study was 10 M NaOH mixed with Na-silicate solution in a mass ratio of 1 to 1. Dry precursor, milled and sieved below 90 μm , was added to the alkali mixture until viscosity was not measurable anymore. The mass ratio precursor to 10 M NaOH to Na-silicate solution was for glass wool 1.5:1:1 and rock wool 2:1:1, respectively. However, the amount of used alkali exceeded the amount of needed alkali, i.e. efflorescence took place (thermonatrite formed). Alkali-activated glass wool had ~ 3 times lower shrinkage, ~ 4 times higher bending strength and ~ 2 times higher compressive strength (Horvat et al., 2018).

Although waste foundry sand is not a promising material for alkali activation, its usefulness for the reaction has been evaluated by the addition of KOH/NaOH and K/Na-silicate solution in different ratios, as well as the addition of extra water and molarities of KOH/NaOH. The baseline ratio of amorphous Si to Na normalised to amorphous Al was set to be 1.9 to 1 to gain the highest compressive strength and avoid efflorescence according to the literature (Duxson et al., 2005). Curing of alkali-activated local waste foundry sand was performed at room conditions and 70 $^{\circ}\text{C}$ for 24 h, then left to further dry at room conditions, in a drier at 110 $^{\circ}\text{C}$ for 24 h or with microwave irradiation (in a microwave oven with magnetron source working in cycles, 2.45 GHz, 700 W, 2.33 min). If curing was performed solely at room conditions, samples did not solidify. A smaller amount of liquid phase resulted in higher mechanical strengths, both for NaOH and KOH. Maximal strength was reached without the addition of hydroxides, i.e., compressive strength reached ~ 30 MPa, which is an impressive result for material with less than 40 m% of amorphous content, than 10 m% of amorphous Si, and 4 m% of amorphous Al. Activating precursor with Na-silicate solution instead of K-silicate solution had 2 times higher compressive strength. Volumetric drying (microwave irradiation) was, regarding the shrinkage, faster than drying at 110 $^{\circ}\text{C}$, which was faster than drying at room conditions. However, faster drying causes bigger cracks and a decrease in bending strength but increases compressive strength (Horvat et al., 2019a).

The potential of different local foundry wastes was evaluated regarding their usefulness in alkali activation at room conditions. Different waste foundry sands, foundry flue gas, and waste casting cores were activated with a mixture of 10 M NaOH and Na-silicate solution (mixed in the mass ratio of 1 to 1). The addition of precursor to alkali mixture was determined experimentally, i.e. the chosen ratio was when viscosity was not measurable anymore. One of the foundry sands was mixed with "green waste ceramics", and with "green waste ceramics and (bottom) ash", to shorten the time of solidification from 1.5 years to less than a week (all solid ingredients were mixed in an equal mass ratio), i.e., time of solidification can be controlled by selection of a mixture of precursors when curing ovens must be avoided. Waste casting cores were once ground and sieved below 90 μm , and once gently ground and sieved below 600 μm . The first did not solidify in more than 2 years (remained rubber-like), and the latter fell apart when demoulded. Compressive strengths for all other samples were from 5 to ~ 10 MPa. Therefore, these materials can be used as reactive fillers (Horvat et al., 2019b).

Another potential precursor, which has not been widely researched up to date (Soriano et al., 2023), was local green ceramics waste that was activated using NaOH and Na-silicate, but in calculated ratios following molar ratios of Al, Si and the sum of the chemical elements of the first group of the periodic system, as also the mass of whole water (from all sources) content present in the mixture. Besides, mathematical formulas running behind calculations were published and software for the calculations and mixture design with the ability to choose the alkalis was developed. AAMs were also chemically foamed using powder Al, powder Na-perborate (monohydrate), liquid H_2O_2 , and a stabilizing agent SDS. All were varied from 0 to 10 m%. Curing conditions were room temperature, 70 $^{\circ}\text{C}$ and 90 $^{\circ}\text{C}$, for different amount of time. From the time development of mechanical strengths, it was concluded that the chemistry of the mixture is the most important parameter, i.e., molar ratios of amorphous Si to Na (normalised to amorphous Al) should be 1.9 to 1, respectively. Mixtures with this ratio gained a limit value of mechanical strengths fastest no matter the curing temperature. If the ratio was not optimal, the mixture might have reached high early compressive strength, which decreased by more than 50% of the



early value. In the study, the influence of the viscosity on the mechanical properties was reported: lower viscosity and higher temperature led to lower mechanical strength (which was reached faster), and higher viscosity and lower temperature led to higher mechanical strengths because curing was gentler, but to reach the limit values took more time. The density of foamed samples, where compressive strength was between 1 and 5 MPa, was lowest at 0.7 kg/l (Horvat & Ducman, 2019).

Different fractions of local electric arc furnace slag were alkali activated with K-silicate solution with the addition of water in mass ratios equal for all samples which were cured for 3 days at 70 °C. The highest compressive strength reached samples made from the smallest fraction (~60 MPa samples from slag with particle size below 63 µm, compared to ~20 MPa for samples from slag with fraction between 90 and 125 µm), which showed the highest reactivity and had the biggest surface area. Using this fraction resulted also in the smallest total porosity of alkali-activated slags (Traven et al., 2019).

The influence of curing/drying methods was further researched on alkali-activated waste casting cores that did not solidify in more than 2 years at room conditions no matter their particle size (Horvat et al., 2019b). Both granulations (below 600 µm and 90 µm) were activated with 10 M NaOH and Na-silicate solution, all mixed in a mass ratio (2.5/3.3/3.75):1:1, respectively, and cured for 24 h at 70 °C. This curing regime was not enough for any mixture or tested granulation. However, for larger granulation of precursor, additional curing/drying at 110 °C for 24 h or microwave treatment for less than 1 minute (with a microwave oven working at 700 W, with 2.45 GHz, and a magnetron source working in cycles) resulted in the compressive strength ~20 MPa. In the smaller fraction used, the highest compressive strength was achieved when the largest proportion of precursor was additionally treated at 110 °C for 24 h (~25 MPa), while microwave treatment for less than 1 min resulted in a compressive strength of 5 MPa. If the addition of precursor was lowered, so was compressive strength, and if microwave treatment was prolonged, the mixture foamed (density of 1.6 kg/l lowered to 0.6 kg/l, compressive strength to 2 MPa). Self-foaming was observed as a consequence of degradation of the organic compound while being irradiated with microwaves (Horvat and Ducman, 2020a). Besides testing waste materials regarding their potential to be used as precursors and fillers, waste bottle glass and waste cathode-ray tube glass were studied focusing on their usefulness for the synthesis of alkali activators. Activators were produced using hydrothermal synthesis changing the synthesis time and temperature, as well as the particle size of the waste glass. The highest concentration of dissolved Si (19 g/l) and Al (0.9 g/l) was prepared at 120 °C treated for 24 h. However, using the best hydrothermally synthesised alkali for alkali activation of local fly ash resulted in a compressive strength of ~30 MPa, while when commercial Na-silicate solution was used, the compressive strength was ~70 MPa (König et al., 2020).

Self-foaming was further researched with the reaction between waste refractory materials and Na-silicate solution in the mass ratio of 5 to 3 for precursor, at 70 °C for 24 h. The precursor was only sieved below different fractions, where the smallest fraction was 1 mm. All fractions had more Al than Si in the amorphous content, which led to foamed material, but only in the smallest fraction. All bigger fractions had big enough voids among particles for the released gasses to accumulate, while in the smallest fraction, released gasses had accumulated in the ASN. The highest compressive strength had a fraction between 1 and 2 mm, 20 MPa. This fraction was tested further to evaluate the dependence of the temperature and time of curing: at room conditions, it did not cure, i.e., after 1 month the compressive strength was 2 MPa, which was reached in 1 week if the sample was cured at 40 °C for 1 week, but if the sample was cured at 70 °C for 1 week, compressive strength was 80% of the compressive strength when the sample was cured at the same temperature for only 1 day. However, after 1 month the compressive strength of the sample treated at 70 °C for a longer time reached the same value as it was treated for a shorter time (Horvat & Ducman, 2020b).

Local fly ashes, alkali activated with Na-silicate solution and NaOH, were foamed with Na-perborate and stabilised using SDS, cured at 70 °C for 24 h and exposed to 1000 °C. The lowest density obtained was ~0.3 kg/l, highest compressive strength was ~6 MPa. The best performance had fly ash that contained the highest amount of Si and Al, as well as the highest amount of amorphous content. Also when exposed to elevated temperature,



i.e., its density did not change, while compressive strength increased by ~50%. Fly ashes with a lower amount of Si and Al, and a smaller amount of amorphous content, showed higher shrinkage after exposure to 1000 °C, therefore the density increased, as the compressive strength (for ~800%) (Korat & Ducman, 2020).

A “cradle-to-cradle” life-cycle assessment was performed for the prefabricated alkali-activated façade cladding panels made from large fractions of recycled construction and demolition waste (wood, and as alternative waste inorganic materials like fired clay, mortar, and concrete) used as fillers. Precursors used were metakaolin, ground granulated blast furnace slag, and class F fly ash, while alkali activators were Na-silicate and K-silicate solutions; the final mixture was obtained by project partners (Panizza et al., 2018). The biggest environmental burden comes from the manufacturing process, which can be lowered by optimising the need for electricity requirements. However, during the use of the product, the energy needed for heating decreases by ~20 to ~40%. Comparison to façade cladding panels made from different virgin materials was calculated on base “cradle-to-gate”. Results suggest that products made via alkali activation and incorporating waste fillers are environmentally friendlier solutions, particularly when the heating during the use stage is considered. Therefore, not only that alkali-activated materials are environmentally friendlier when compared to conventional materials like cement, mortar, concrete and ceramics, but so are the final construction products assessed in the study (Kvočka et al., 2020).

Alkali-activated thermally insulating façade panels with inorganic construction and demolition waste used as filler, which were evaluated regarding their life cycle performance, were analysed regarding their mechanical strength and durability tests: capillary water uptake, water vapour permeability, impact resistance, bond strength, freeze-thaw behaviour, freezing in the presence of de-icing salt, resistance to carbonation, alkali-silica reactivity, and sulphate resistance. Samples performed comparably to cement-based panels, except at the test evaluating resistance to freezing in the presence of de-icing salt, where alkali-activated panels ended up severely damaged (Frankovič et al., 2020).

Further development of alternative alkali activators from pulverised waste materials (rock and glass wool, bottle glass and cathode-ray tube glass) was performed at 120 °C at normal pressure (no hydrothermal synthesis) for 4 and 24 h. In 200 ml of 10 M NaOH, 5 and 20 g of pulverised glassy waste was constantly stirred. Synthesised alkali was tested with local thermopower plant fly ash, which was used as received, and electric arc furnace slag from the metallurgical industry, which was milled and sieved below 90 µm; the mass ratio precursor to alkali was kept constant. As a reference 10 M NaOH was used. In the case of fly ash, mechanical strengths did not show noticeable changes, while for slag ~70% increase of already low compressive strength happened when activators based on waste bottle glass and waste glass wool were used. Activators might be better than 10 M NaOH, but are not yet comparable to the commercial Na-silicate solutions (König et al., 2021).

Waste glass and rock wool, and their mixtures, were studied as sole precursors in alkali activation with 5 M NaOH, and with different ratios of NaOH dissolved in Na-silicate solution, cured at room conditions and for 3 days at 40 °C. Higher temperature allowed faster reaching higher mechanical strength values, rock wool solidified faster, but did not reach as high final compressive strength as glass wool. However, a mixture of 25 m% of rock wool and 75 m% of glass wool resulted as an optimal choice, i.e., samples gained faster the early mechanical strengths but also reached higher final values (Pavlin et al., 2021).

The development of deformation during the early stages of curing was studied on ladle slag and electric arc furnace slag, both activated with Na-silicate solution, in dependence on the temperature (room temperature, 40 and 60 °C) and humidity (30 and 90%). The focus was placed on autogenous and drying shrinkage in the first 72 h. The autogenous shrinkage had the highest rate at the highest tested temperature. The highest tested temperature and lowest humidity also gave the highest rate of drying shrinkage, i.e., the conditions were most favourable for water evaporation. The smallest deformations happened when the curing temperature was 40 °C for the first 24 h (Češnovar et al., 2021).

Alkali-activated foams, foamed with H₂O₂ and stabilized with SDS, based on fly ash were thermally improved by the replacement of fly ash with metakaolin and the replacement of Na-based with K-based alkalis (hydroxides and silicate solutions). The replacement of



fly ash with metakaolin delayed the start of the shrinkage from ~600 to ~700 °C, the replacement of Na-based with K-based activator from ~600 to ~800 °C, and the replacement of fly ash with metakaolin along with the replacement of Na-based with K-based activator from ~600 to ~900 °C (Traven et al., 2021).

Alkali-activated local fly ash and slag mixture (ladle slag and electric arc furnace slag, in mass ratios of 1 to 1), activated with Na-silicate solution and NaOH, foamed with Na-perborate and H₂O₂, and stabilised with triton, with/without added polypropylene fibres, embedded lightweight aggregates (expanded clay, perlite, polystyrene, expanded glass). While bending strength was below 1 MPa, and compressive strength was below 5 MPa, thermal conductivity was between ~100 and ~200 mW/(m·K). If lightweight aggregates had large open pores, alkali-activated foam expanded into them and mechanically fixated aggregates into the ASN. Otherwise, under the scanning electron microscope, a chemical reaction between alkali-activated slurry and aggregates was not observed (Traven et al., 2022b).

Alkalis based on glass wool, rock wool, bottle glass and cathode-ray tube glass were synthesized in KOH (5 and 20 g of glassy waste was treated in 200 ml of 10 M KOH for 4 and 24 h at 120 °C). Their usefulness in alkali activation was tested on electric arc furnace slag and compared to activation with commercial K-silicate solution and KOH, and compared to the study where alkalis were prepared in NaOH. Compressive strength results of alkali-activated slag were higher when alternative activators were made in KOH than in NaOH, and higher or comparable to reference activated with KOH and NaOH. However, compressive strength results were lower compared to slag activated with commercial Na-silicate and K-silicate solution 3 to 4-fold (Pavlin et al., 2022c).

The influence of (three-roll mill) homogenization of the freshly mixed alkali-activated slurry on mechanical strength was studied on different precursors: fly ash, fly ash with metakaolin (mixed in mass ratio of 3 to 1 for fly ash), glass wool, waste green ceramics, and slag mixture (ladle slag and electric arc furnace slag in mass ratios of 1 to 1). Precursors were activated by NaOH, Na-silicate solution, and laboratory-produced alkali based on waste cathode-ray tube glass, in recipes that were not comparable, so that the only parameter left would be homogenization and no homogenization. Therefore, curing regimes (60 and 70 °C) and drying methods (room temperature and 105 °C) also differed. Moreover, the slag mixture was foamed using Na-perborate and stabilized by triton, and the mixture of fly ash and metakaolin with Na-perborate and stabilized by SDS. Homogenization lowered the viscosity, therefore workability and moulding were easier, the distribution of particulates and pores was more uniform, and mechanical strengths were higher. In the case of fly ash, the non-combusted cellulose got combusted during homogenization (smoke was visible during the procedure and under the scanning electron microscope no cellulose was found after homogenization). If the onset of the foaming was delayed, homogenization could be performed for foamed materials without loss of gasses and increase of density, ending with smaller pores (Horvat et al., 2022).

To improve mechanical properties, different fibres (polypropylene, polyvinyl-alcohol, basalt, and waste glass wool) in several quantities (0.5, 1.0, 1.25, 1.5, and 2.0 volumetric percentage) were added into alkali-activated foam based on a mixture of ladle slag and electric arc furnace slag (their mass ratio is 1 to 1). As an activator Na-silicate solution was used, as a foaming agent H₂O₂, and as a stabilising agent the surfactant triton. Compressive strength showed improvement in all samples while bending strength showed improvement only when polypropylene and polyvinyl-alcohol were added (Traven et al., 2022a).

Besides the addition of fibres into alkali-activated slag foams, 1 m% of different fibres (basalt, cellulose of 2 types, glass, polypropylene, polyvinyl alcohol and steel fibres) were added to alkali-activated milled waste rock wool (activator used was Na-silicate solution), that is fibrous on its own. Bending strength after 1 month was highest when glass fibres were used, while compressive strength was high for all fibres except basalt and cellulose packed in cubes (Pavlin et al., 2022b).

Waste rock wool was further investigated regarding its potential to be used as a precursor in alkali-activated foams. The activator used in the study was a Na-silicate solution, the foaming agent was H₂O₂ (added from 1 to 3 m%), and the surfactant used was triton (1.5 m%). The samples were cured at 70 °C for 3 days. The sample with the lowest density (~0.5



kg/l) had a compressive strength ~1.5 MPa and total porosity below 80%. The lowest thermal conductivity was ~90 mW/(m·K) (Pavlin et al., 2022a).

Alkali-activated fly ash and ground-granulated blast furnace slag were analysed as adhesives to different substrates (concrete, ceramic tiles, wood-based geopolymer and high-density geopolymer). They were mixed in different ratios and activated with K-silicate solution and KOH. Good bonding (high pull-off strength) was achieved in connection with concrete and ceramic tiles, while not with either of the geopolymeric samples. On the interfaces it was confirmed, that also chemical adhesion took place, but the mechanism is not yet understood (Wisniewski & Ducman, 2022).

Alkali-activated materials made from waste precursors have a lower carbon footprint (Kvočka et al., 2020) compared to conventional building and civil engineering alternatives. However, the environmental imprint can be further lowered by decreasing the need for energy and time for production. The potential candidate is a replacement of the surface with volumetric heating, therefore microwave irradiation of alkali-activated materials in their early stage was studied. Alkali-activated metakaolin slurry was irradiated with microwaves at 100 W and 1000 W for 1 min (microwave of inverter type working at 2.45 GHz) and compared to the sample cured solely at room conditions. Microwave irradiation with low powers resulted in a higher early compressive strength, while irradiation with high powers foamed and solidified alkali-activated slurry. If the dehydration during irradiation was hindered, compressive strength was higher. However, alkali activation particularly enhanced the leaching of As, while irradiation with 1000 W increased the leaching of Cr and As, all compared to the precursor. With the decreased potential of immobilisation of Cr and As, microwave irradiation has the potential to be used for the removal of heavy elements and as a cheap and efficient method for recovery or remediation (Horvat et al., 2023).

Rock wool mixed with lime, metakaolin, and electric arc furnace slag, was alkali activated with Na-silicate solution with added NaOH, and treated at room conditions, 40 and 60 °C, and at 30, 60 and 90% humidity. At upscaling, curvature and highly varying mechanical properties arose, which was because of unevenly milled batches of rock wool. This was the reason for the constant need to change the recipe, however, when the milling was of constant quality, so was the recipe. The final choice for curing to avoid curvature of the façade panels was 3 days at room temperature in the PVC bag (to hinder dehydration), and an additional 3 days at 60 °C and 60% humidity demoulded on a level metal mesh to allow uniform drying (Pavlin et al., 2023).

Calcined river sediments, rich in clay, were evaluated regarding their potential to be used as precursors in alkali activation with Na- and K-silicate solution, and with NaOH and KOH. Clay was calcined at 900 °C, and alkali-activated samples were cured at 60 °C for 3 days. Replacement of the calcined sediment was done up to 30 m% with fly ash and ladle slag. Calcined clay can be used as a precursor, however, the addition of more reactive ingredients is beneficial for mechanical strengths (Žibret et al., 2023).

The influence of microwave irradiation in the early stage of alkali-activated local ladle slag, local thermopower plant fly ash and metakaolin was evaluated regarding the time and power of irradiation (2.45 GHz, magnetron source). Mixtures with Na-silicate and NaOH synthesised had the needed addition of alkali calculated by fixing the Si at an Al to Si molar ratio of 1 to 1.9. Then the first group of the periodic system was set to avoid efflorescence by not exceeding the molar amount of amorphous Al, if possible. Besides, the amount of liquid to solid was varied to study the dependence of water content in the alkali-activated slurry during microwave irradiation. Even if the material absorbed the same amount of dose, it is clear that it is important if the dose was received gently and slowly, or fast under harsh conditions. This was different for every mixture, not just every precursor. Additional water decreased the compressive strength, which was slightly higher after irradiation. However, there were no mineralogical changes observed because of the irradiation.



3. Conclusions

10 years of research on alkali-activated materials in Slovenia brought the research to the edge, i.e., with just chemical and mineralogical evaluation of any material containing silicon and aluminium, the optimal algorithm for calculating the recipe was proposed. This recipe is the starting point in experimental analysis on the TRL4 level, while 10 years ago everything started on the TRL1 level. With a theoretical approach, several pilot products and testing fields in cooperation with the local industry and from local waste were designed.

Besides levelling up the start of the research from the TRL1 to TRL4 with a theoretical approach, foaming through chemical reactions and heat was researched. Chemical foaming was performed through the introduction of different foaming and stabilizing agents into the raw ingredients (not into the fresh mixture) as induced foaming, while a correct choice of precursors in an alkali environment led to a delayed self-foaming. As self-foaming did not need an introduction of additional chemicals, so did not physical foaming with volumetric heating. However, the last curing-foaming technique is still ongoing research in the first national project on the influence of microwave irradiation on alkali-activated materials.

Nonetheless, if the waste is collected and stored as raw materials are, in big enough quantities, the future of the building and civil engineering products made with alkali activation of the secondary materials is viable but has to be tested first on a bigger scale. However, bigger countries with different industries that produce different wastes, have a much wider pallet of materials to choose from and select the most suitable regarding the desired properties of the product and environmental impact.

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