

New Porous Material Made from Industrial and Municipal Waste for Building Application

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The aim of this study was to find a new method for usage of the hazardous waste coming from recycling industry. Two hazardous wastes – aluminium recycling final dross or non-metallic product (NMP) and lead – silica glass (LSG) were investigated. It is generally considered that NMP is a process waste and subject to disposal after residual metal has been recovered from primary dross. NMP is impurities which are removed from the molten metal in dross recycling process and it could be defined as a hazardous waste product in aluminium recycling industry. LSG comes from fluorescence lamp recycling plant and could be classified as hazardous waste due to high amount of lead in the composition and re-melting problems.

The new alkali activated material, which can be defined as porous building material, was created. Composition of this material consisted of aluminium recycling waste, recycled fluorescent lamp LSG, sintered kaolin clay as well as commercially available alkali flakes (NaOH) and liquid glass ($\text{Na}_2\text{SiO}_3 + n\text{H}_2\text{O}$). Physical and mechanical properties of the obtained material were tested.

Density of the obtained material was from $(460–550) \text{ kg/m}^3$ and the total porosity was from 82 %–83 %. The compressive strength of the material was in range from 1.1 MPa to 2.3 MPa. The thermal conductivity was determined. The pore microstructure was investigated and the mineralogical composition of porous material was determined.

Keywords: aluminium recycling waste, lead silica glass waste, porous building material, alkali activation.

1. INTRODUCTION

Due to the several advantages, building materials made from non-limestone based binders start to play an important role in the society of scientists and engineers. Scientists and “green thinking” engineers have focused their attention on developing of alternative binders which do not require the use of fossil fuel to heat large kilns and virgin raw materials. One alternative can be “geopolymer” technology, which requires mixing of post-industrial and municipal waste as well as naturally occurred aluminosilicate materials with an activation agent at the ambient temperature or slightly increased temperature in order to produce a long lasting, eco-friendly cementitious material. The production of different types of alkali activated binder (AAB) and concrete (AAC) results in a near zero-carbon dioxide emission, which is significantly less compared to the production of ordinary portland cement or lime binder [1]. The AAB and AAC, which are basically inorganic polymer composites, exhibit some superior engineering properties and have the potential to form a substantial element of an environmentally sustainable construction by replacing/supplementing the conventional concretes.

The microstructure and properties of AAB and AAC depend on the initial raw (waste) materials; they can have high compressive strength, low shrinkage, extra fast or fast setting, good acid and fire resistance and low thermal conductivity [2–4]. Wallah and Rangan [5] proved that the alkali activated concrete resistance to sulphuric acid and sulphate attack of heat cured AAC was significantly better

than that of the portland cement concrete. Temujin et. al. [6] reported that the aluminosilicate composites have good adhesion to metal substrates and excellent thermal resistance. Shi et. al. [7] have demonstrated the useful engineering properties of AAB using fly ash and an alkaline liquid containing sodium silicate and sodium hydroxide solution for structural applications. The alkaline liquids used for this type of binder are still commercially manufactured, therefore, additional industrial pollution is appearing. Deevasan and Ranganath [8] had idea that the geopolymer technology could be more environmentally friendly if the alkaline solution could be replaced with the industrial waste. Therefore they made experiments with the alkaline solution from poly-fibre industries and concluded that it is possible to produce AAC of strength up to 55 MPa using industrial by-products (waste) such as low calcium fly ash and effluent alkaline solution.

Also, it could be possible to work with reactive aggregates in the building industry by using alkali activated binder. When reactive aggregates are present, the alkali-aggregate reaction induces disruption in ordinary portland cement (OPC) concrete causing serious durability problems to reinforced cement concrete structures. The presence of calcium in OPC induces expansion of the alkali-silica reaction products, whereas in the case of alkali-activated, fly ash-based binders, the alkali-aggregate reaction results in the formation of zeolite crystal deposits in the pre-existing pores which are not expansive in nature. Although alkali used in the production of AAB could react with the aggregate, in the long term and in the absence of calcium in the system, the product is not expansive [9].

AAB and AAC are commonly formed by alkali activation of industrial aluminosilicate waste materials,

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such as coal fly ash [10], rice husk ash [11], fly ash [12–14], ground granulated blast furnace slag [15], lignite bottom ash [16] wastes from metallurgy [17] etc., and they have a very small carbon footprint when compared to the traditional concretes.

The idea of this research is usage of aluminium scrap recycling waste as a raw material for production of alkali activated binder. It is well known that the consumption of aluminium scrap recycling waste is rising continuously worldwide, which is great stimulus for developing a non-waste technology [18–20]. The recovery of metal from black or white dross (primary dross) in aluminium recycling industry is done through the conventional metallurgical process all over the world. Residual aluminium recycling waste (final dross) containing alumina, salts, impurities and a small amount (3%–5%) of metallic aluminium was obtained during the second recovery process. Processing of the NMP created in the aluminium scrap recycling companies is one of the most challenging tasks due to its toxic nature – in accordance with the Basel Convention, Annex III, marking of this waste is H 4.3 (reaction with water results in highly inflammable substances) and H 10 (reaction with water results in increased concentration of toxic gases, for instance, ammonia). The given type of waste may have different names in various scientific publications, for example: NMP, the aluminium recycling waste, the residual oxide mix, the residues and even recycled or residual dross. It is generally considered that the NMP is a process waste and subject to disposal after metal has been recovered from dross. The term NMP is used interchangeably in this study.

Waste from aluminium scrap recycling industry (NMP) and LSG waste as well as sintered kaolin clay were used as basic raw materials for production of porous alkali activated binder in this research.

2. EXPERIMENTAL DETAILS

2.1. Test methods

Chemical composition of the raw materials was determined according to LVS EN 196-2 with precision $\pm 0.5\%$. Analysis of the elements was carried out with help of inductive coupled plasma optical emission spectrometry (ICP-OES), atomic absorption spectroscopy (AAS) and potentiometer titration analyses. The mineralogical composition of NMP was determined by using the XRD analysis. Specific surface area was detected by BET method ("Nova 1200 E-Series, Quantachrome Instruments"). Effective diameter was detected by Zeta potential ("90 Plus" un "MAS ZetaPALS Brookhaven Instr"). Microphotographs were obtained by "Tescan" "Mira/LMU Schottky".

The flexural and compressive strength of AAB were tested according to LVS EN 1015-11 using 3 specimens with dimensions (40×40×160) mm. The density of AAB was measured in accordance with EN 1097-7. The water absorption was determined according to EN 1097-6. The thermal conductivity was performed with heat flow meter instrument LaserComp FOX 660 using air-dry A10 sample with dimensions of (300×300×50) mm. The upper and lower plate temperatures were 0 °C and 20 °C with average temperature of 10 °C.

The mineralogical composition of specimens was determined by X-Ray diffractometer "RIGAKU ULTIMA+". The samples were observed by using scanning electron microscope (TESCAN Mira\LMU Field-Emission-Gun) and the elemental composition of the observed phases was assessed by energy dispersive X-ray spectrometry (EDS, Oxford Instruments 7378) performed on the scanning electron microscope. The accelerating voltage was 15 kV.

2.2. Raw materials

The raw materials used in the current study of alkali activated binder were metakaolinite (MK), NMP received from aluminium scrap recycling industry and LSG waste received from glass waste recycling factory. Alkali-activation of raw material composition was done by modifying sodium silicate solution with an addition of sodium hydroxide.

Metakaolinite (MK)

In the current study, MK was obtained in the laboratory furnace by the sintering of commercially available kaolinite clay in the temperatures from 500 °C to 800 °C. The results showed that the sintering rate and time make significant influence to the pozzolanic reactivity of MK (Table 1). The sintering process of MK used in this study lasted approximately 3 h with temperature increase of 15 °C/min and holding of MK in the maximum temperature (800 °C) for 1 hour. This sintering method was chosen to obtain precise sintering temperature and time. It was also possible to use a rotary furnace (RF) with sintering time no longer than 15 min and maximum temperature 700 °C, because the pozzolanic reactivity of MK remained the same (Table 1), but process can be controlled. Both of these sintering methods require grinding of the agglomerated products, therefore MK was ground in the laboratory planetary ball mill Retsch PM 400 for 30 min with speed 300 rpm. Specific surface area of ground MK was 15.86 m²/g. Effective diameter of MK particles was 743.1 nm.

According to the chemical analyses, the amount of amorphous aluminium and silica oxides increased to 32%–33% in the composition of MK sintered in the temperature range from 700 °C to 800 °C. Amount of amorphous SiO₂ did not exceed 1.85% during the heat treatment procedure (Table 1).

Table 1. Amount of amorphous SiO₂ and R₂O₃ in MK sintered at different temperatures (w %)

Sintering temperature	SiO ₂ total	R ₂ O ₃ total	SiO ₂ amorphous	R ₂ O ₃ amorphous
500 °C	53.50	42.24	1.00	6.04
600 °C	53.54	42.86	1.27	21.06
700 °C	54.98	43.34	1.10	32.76
700 °C RF	54.70	43.77	1.85	32.19
800 °C	55.86	43.70	1.28	32.67
Sensibility ± absol.%	0.5	0.5	0.10	0.3

MK has clay minerals in its composition. The crystalline layer structure, which is a common

characteristic of clay minerals, leads to a fine particle size (1 μm –3 μm) and plate like morphology in MK (Fig. 1).

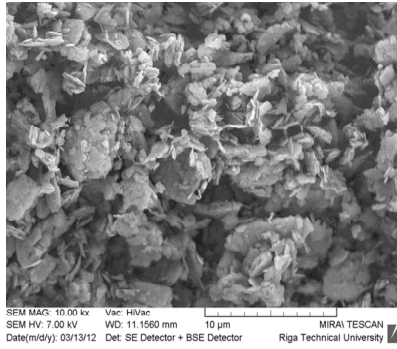


Fig. 1. Microphotograph of MK particles sintered in the laboratory furnace at temperature 800 °C

Non-metallic product (NMP)

The NMP is the final waste product at the aluminium scrap recycling factories. Useful utilisation of the industrial waste is a much more attractive alternative than to deposit it on the waste sites as it is practiced in Latvia and other small countries. Recycling of industrial waste into building materials reduces possible pollution of the environment and decreases the costs of waste deposits, as well as saves resources of natural raw materials and gives economic benefits for manufacturing of new materials and building units.

In order to increase homogeneity and fineness of NMP it was ground for 30 minutes by using planetary ball mill Retsch PM 400. Chemical composition of NMP was determined according to LVS EN-196-2 with sensibility ± 0.5 w% (Table 2).

Table 2. Chemical composition of NMP and LSG (w%)

Chemical component	NMP	LSG
Al ₂ O ₃	63.19	1.03
SiO ₂	7.92	69.07
CaO	2.57	1.39
SO ₃	0.36	–
TiO ₂	0.53	–
MgO	4.43	–
Fe ₂ O ₃	4.54	0.19
PbO	–	20.02
Na ₂ O	3.84	8.02
K ₂ O	3.81	1.17
LOI, 1000 °C	6.21	–

Analysis of the NMP elements was carried out with the help of ICP-OES, AAS and potentiometer titration analyses. Results showed the following content of NMP: aluminium (Al) – 34.40 %, silicon (Si) – 4.40 %, magnesium (Mg) – 2.44 %, calcium (Ca) – 1.32 %, sodium (Na) – 1.69 %, potassium (K) – 2.31 %, sulphur (S) – 0.07 %, chlorine (Cl) – 4.23 %, iron (Fe) – 3.60 %, copper (Cu) – 0.99 %, lead (Pb) – 0.14 % and zinc (Zn) – 0.6 %. These data correspond to the chemical composition of NMP, which is given in Fig. 2. In terms of chemical composition the analysed NMP contained also aluminium nitride (AlN) – on average 5 w%, aluminium chloride (AlCl₃) – on average 3 w%, potassium and sodium

chloride (NaCl+KCl) – total 5 w% and iron sulphite (FeSO₃) – on average 1 w%.

The mineralogical composition of NMP was determined by using the XRD analysis (Fig. 2). According to the XRD analysis data, the NMP contained metallic aluminium (Al), iron sulfite (FeSO₃), aluminium nitride (AlN), corundum (Al₂O₃), aluminium iron oxide (FeAlO₃), magnesium dialuminium (MgAl₂O₄), quartz (SiO₂), aluminium chloride (AlCl₃) and aluminium hydroxide (Al(OH)₃).

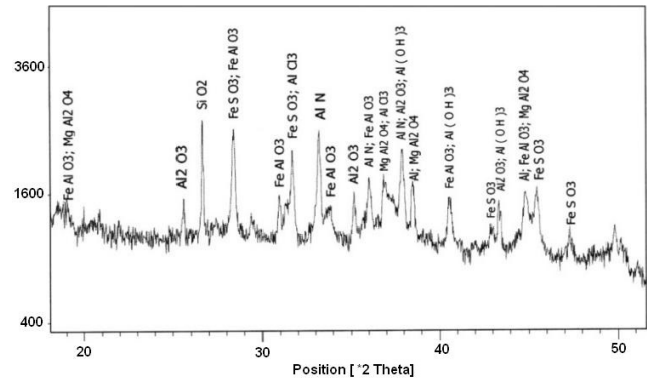


Fig. 2. XRD analysis of NMP

Lead-silicate glass waste (LSG)

The LSG was obtained from fluorescent lamp recycling process, which includes lamp classification, glass separation, cleaning from harmful components and glass grinding. The chemical composition of lead glass is given in Table 2.

In order to increase chemical activity and fineness of waste glass, it was additionally ground in the laboratory planetary ball mill Retsch PM 400. The applied grinding period was 30 minutes to ensure homogeneity and fineness of the waste glass particles.

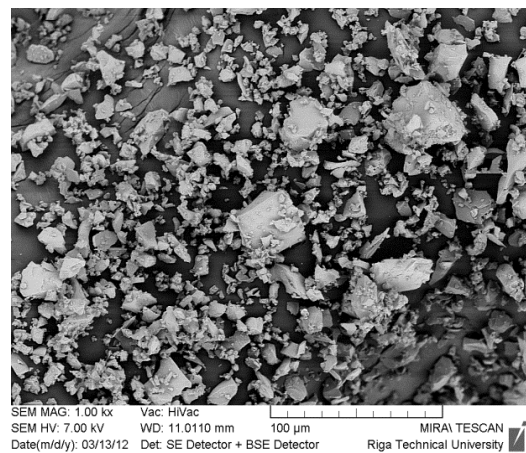


Fig. 3. Microphotograph of ground LSG particles

The particle size distribution in powder material was determined by laser diffraction method. The grading analysis results showed that the additionally ground material contains a wide range of particle sizes: from 8 μm to 30 μm with the average grain size of 26.3 μm . It is comparable with the data obtained from the investigation made by scanning electron microscope SEM (Fig. 3). The ground glass particles have smooth surface. The following geometrical results were obtained: circularity coefficient 0.868, convexity coefficient 0.978 and elongation

coefficient 0.335. The specific surface area of waste glass ground for 30 min. was 1.126 m²/g.

Alkali activator solution

The alkali activated binder specimens were prepared by using alkali activator solutions with silica modulus M_s 1.93 (mixture A7.5) and 1.67 (mixture A10). Alkali activators were prepared by using sodium silicate solution characterized by the silica modulus $M_s = 3.22$ (Table 3). To obtain alkali activation solution with the required chemical composition, the modification with an addition of sodium hydroxide was done. Commercially available sodium hydroxide flakes with 97 % purity were used.

Table 3. Properties of sodium silicate solution

Density	40.4–42.0 °Bé
Dry Matter	37.50–39.50%
Molar Ratio	3.30–3.50
Weight ratio	3.20–3.40
SiO ₂	29.50 %
Na ₂ O	9.00 %

2.3. Sample preparation and curing conditions

Alkali activated binder mixtures were cast in (40×40×160) mm prismatic moulds and the curing conditions were kept constant at the different temperature regimes from 40 °C to 100 °C for 24 h. The optimal curing temperature for A7.5 and A10 was 80 °C for 24 h. The components of NMP – aluminum nitride (AlN) and iron sulphite (FeSO₃) – react in the alkali environment and create gases – ammonia and sulfur dioxide, which provide the porous structure in the obtained material.

After 24 h, samples were demoulded and kept in ambient environment. Physical and mechanical properties of hardened alkali activated mortar bars were tested.

Several series of alkali activated binder mixtures were prepared. The variable between all mixtures was composition of raw materials and silica modulus of activator solution. Two most promising mixtures were selected. The alkali activator of the first mixture A7.5 has a silica modulus of $M_s = 1.93$, but the mixture A10 a silica modulus of $M_s = 1.67$. The composition of the presented mixtures by mass was prepared with the same ratio: 1 part NMP, 1 part LSG and 1 part sintered clay (T – 800 °C).

3. RESULTS AND DISCUSSION

Physical and mechanical properties

Pore structure developed in the new porous material during hardening process. Due to the chemical reactions and gas evaporation, highly porous material was obtained (Fig. 4). The pore volume and density could be controlled by alkali concentration in the activator. Mechanical and physical properties of the porous alkali activated specimens are given in Table 4. The specific gravity of hardened samples was tested. The material with higher alkali concentration provided lower density and higher porosity. Samples with the mixture composition A10 provided density in a range from (460–510) kg/m³, while

samples with mixture composition A7.5 provide density from 580 kg/m³ to 600 kg/m³. The material density was in the range of (2822–2838) kg/m³.

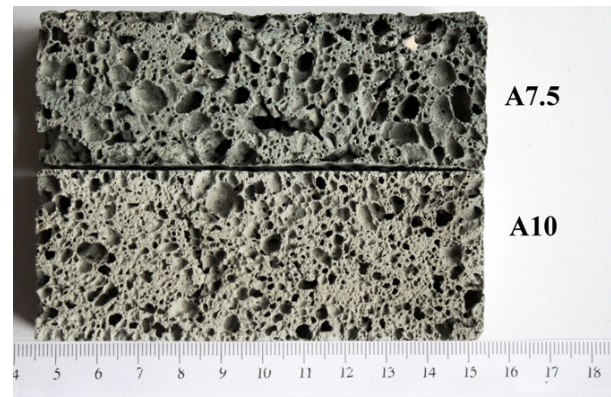


Fig. 4. Macrostructure of porous alkali activated specimens made by using activator solution with different silica modulus

Samples with the mixture composition A10 provided open porosity 29 %–32 % and total porosity 82 %–83 %, but samples with the mixture composition A7.5 provided lower open and total porosity – 25 %–27 % and 80 %, respectively.

Due to the lower density than water, samples with the mixture composition A10 provided very high water absorption – 67 %–70 % by mass of dry sample. The water absorption of samples A7.5 with the mixture composition was little lower from 47 % to 48 %. All specimens floated in the water due to their high proportion of closed pores.

Table 4. Mechanical and physical properties of alkali activated specimens

Composition	A10	A7.5
Density kg/m ³	460–510	580–620
Open porosity, %	29–32	25–27
Total porosity, %	82–83	80
Water absorption, %	67–70	47–48
Compressive strength, MPa	1.1–1.4	1.8–2.3
Flexural strength, MPa	0.1	0.1
Thermal conductivity, W/(m·K)	0.14	0.15

Compressive strength of hardened samples was 1.1 MPa–1.4 MPa for A10 samples and 1.8 MPa–2.3 MPa for A7.5 samples. The flexural strength was 0.1 MPa for all samples with no dependence on composition. The thermal conductivity of porous alkali activated specimens was between 0.14 W/(m·K)–0.15 W/(m·K) (Table 4).

XRD and EDX analyses

Mineralogical composition of alkali activated binder specimens was determined using the XRD analysis. The results are given in Fig. 5. The results indicate amorphous structure of the obtained material with no crystalline phase which is possible to identify for both compositions.

SEM analysis

The obtained alkali activated binder has porous structure with a wide range of pores (see Figs. 4, 6). Micropores

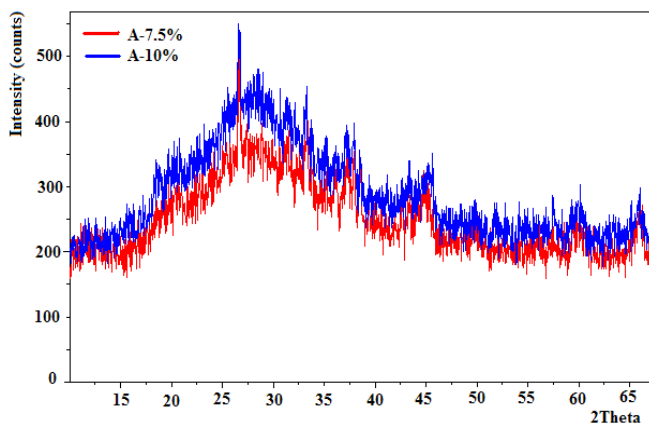


Fig. 5. XRD analysis of hardened alkali activated binders: A10 upper line, A7.5 lower line

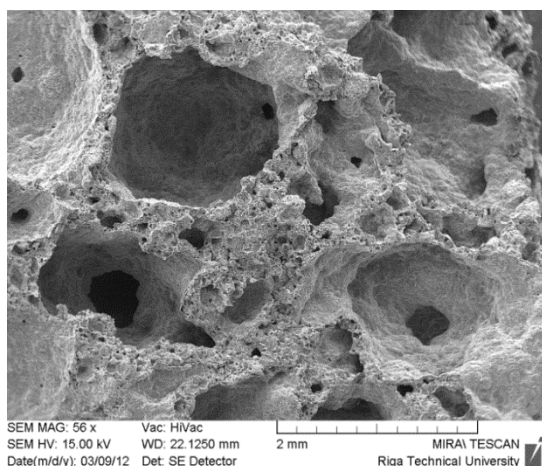


Fig. 6. Microphotograph of porous microstructure of hardened alkali activated binder mixture A10

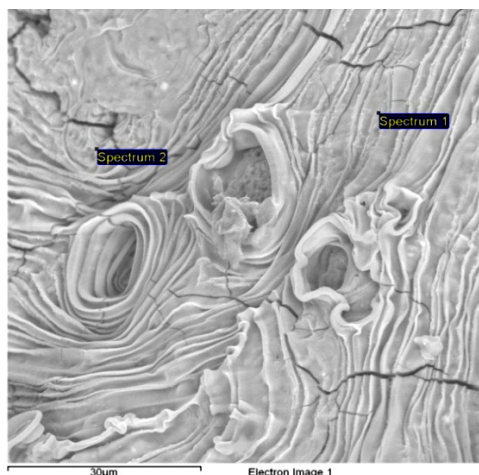


Fig. 7. Microphotograph of A10 sample indicates places where EDX analysis was done. Texture of surface indicates development of pore structure due to gas evaporation

were observed also in the large scale walls. Pores in the range of 87.5 µm to 258 µm were noticed (Fig. 6).

The microstructure of hardened alkali activated binder looks amorphous and homogenous (Fig. 7). An additional research was done regarding investigation of the correlation between curing conditions and microstructure of alkali activated binder. The optimal curing temperature

80 °C was selected for 24 h to obtain specimens without shrinkage cracks. However, some microcracks have been observed in the material structure (Fig. 7). This could be caused by the temperature regime during hardening process of specimens or could have appeared during sample preparation procedure. The microcracks do not affect strongly the functional properties of alkali activated binder due to its high porosity and low compressive strength.

Table 5. EDX results illustrated by Fig. 7, atomic weight %

Spectrum	Compound, %							
	O	Na	Mg	Al	Si	K	Ca	Fe
Spectrum 1	55.13	5.16	–	9.15	25.40	0.80	2.44	1.93
Spectrum 2	64.88	7.31	0.31	8.41	17.78	0.34	0.56	0.39

The EDX results indicate that the compounding elements such as Al, Si, Na and O were present in material structure for both mixtures (Table 5). The metals Pb and Cu were not identified. Most possibly they are encapsulated or immobilized in the structure of alkali activated binder.

4. CONCLUSIONS

The new type of porous alkali activated binder was obtained by using industrial by-products such as NMP from the aluminium scrap recycling industry and lead-silicate waste glass from recycling of fluorescent lamps as secondary raw materials together with the natural raw materials (sintered kaolinite clay) and activator solution. Alkali activated binder could be used as a new progressive porous material in building industry. The presented material was characterized by density in the range 460 kg/m³ – 620 kg/m³. The density can be controlled by moulding fresh mixture and alkali content in activator solution. However, due to the low density and specific pore structure development, mechanical properties of the material were limited. Due to high porosity (82 % – 83 %), the flexural strength of material was 0.1 MPa and the compressive strength of material was in the range of 1.1 MPa – 2.3 MPa. The thermal conductivity of porous alkali activated specimens was between 0.14 (W/(m·K)) and 0.15 (W/(m·K)).

Further research on introduction of sand in the mixture of the alkali activated binder composition and curing conditions optimization would be necessary in order to improve mechanical properties of the material. The pore structure development and pressure build up inside the material during hardening process by gas evaporation must be investigated in order to control the material density and mechanical and physical properties, which could lead to a wide range of material application in the building industry.

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