



# The use of different by-products in the production of lightweight alkali activated building materials



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## HIGHLIGHTS

- Industrial by-products as precursor of lightweight alkali activated materials.
- Highly porous lightweight building materials were obtained with a density from 380 to 470 kg/m<sup>3</sup>.
- Steel plant waste retards pore formation providing homogeneous pore distribution;
- Alkali activation of raw materials shift the crystalline phase to an amorphous;
- 3D porous structure with X-ray micro-tomography was investigated;

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## ABSTRACT

This paper investigates the value of different industrial by-products and residues in the production of lightweight alkali activated materials (AAM). Waste metakaolin, recycled waste glass, aluminium scrap recycling waste, and steel-plant waste (SPW) were considered as secondary raw materials. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Energy-dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM), and X-ray micro-tomography examinations were performed in order to analyse the reaction products, to identify their phase composition, and to observe the micro-morphology.

The results showed that highly porous lightweight building materials could be obtained, with densities ranging from 380 to 470 kg/m<sup>3</sup>, heat conductivities ranging between 0.14 and 0.15 W/m-K, and compressive strengths ranging between 1.1 and 2.0 MPa. It was also shown that the main hydration products were amorphous aluminosilicate gels, whereas the crystalline compounds from the SPW remained in the structure of the AAM. The results of X-ray micro-tomography showed that lightweight AAM contained 72.0–89.0 vol% of pores, with sizes ranging between 1 and 5 mm. In the paper a brief description is given of the waste management method which can be applied to obtain materials that can be used in the building industry as lightweight load-bearing insulation materials.

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## 1. Introduction

Alkali activated materials (AAM) have become a remarkable research area, since, by means of the alkali activation processing of different industrial by-products, it is possible to obtain financially beneficial and environmentally friendly materials for use in the construction field as load-bearing and insulating materials. According to the Kyoto protocol, which commits countries to important reductions in CO<sub>2</sub> emissions, the replacement of ordi-

nary Portland cement (OPC) by ‘greener’ material has become increasingly important; so that the valorisation of industrial by-products is a further goal which represents a promising option for the achievement of sustainable development [1].

Inorganic binders used in the building industry can be divided into fundamentally different groups according to their chemical composition. OPC is characterized by its high Ca and low Na content [2], whereas a high amount of Al is characteristic for fire-resistant alumo-silicates [3]. Similarly, AAM could be divided into high calcium (Ca) systems and low Ca systems. The primary binding phase of high Ca AAM systems consists of calcium silicate hydrates (C-S-H) and calcium aluminate silicate hydrates (C-A-S-H), whereas in the case of low Ca AAM systems sodium aluminate

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silicate hydrates (N-A-S-H) make up the predominant binding phase [4]. It is clear that the choice of the raw material plays a very important role in the formation of AAM, and has a strong effect on the binding strength [1]. Due to its reactive chemistry and very suitable oxide composition, mixtures of metakaolin and fly ash have provided a 'model system' for the studying of activation process [5–7]. However, many scientists have shown that industrial by-products such as blast furnace slag [8–10], steel slag [11], waste calcined kaolin clay (MK), aluminium scrap recycling waste (ASRW), and glass waste (G) together with sodium silicate solutions, could be used to create low Ca AAM [12,13].

One of the main advantages of using porous, lightweight construction materials is that they can significantly reduce the loads acting on structures, as well as improving their thermal and acoustic properties. Material porosity is the main parameter in lightweight material characterization. It is described by means of overall porosity, open and closed porosity, and pore size, as well as pore distribution in the material. Considerable efforts have been made to create porous lightweight AAM using highly porous siliceous materials, by incorporating them in the AAM matrix, so that materials with densities ranging between 0.93 and 1.50 g/cm<sup>3</sup> and compressive strengths of up to 2.4 MPa were obtained [14]. The incorporation of aggregate from recycled lightweight blocks has also been attempted. This could enhance the waste management of demolished buildings and result in lightweight AAM concretes with densities from 860 to 1400 kg/m<sup>3</sup>, water absorption of 10–31%, and compressive strengths from 1.0 to 16.0 MPa [15]. Lightweight AAM can also be obtained by adding a foaming agent, which creates gas by mean of a chemical reaction. In this case the development of porosity within the structure of a material depends on the following factors: the supply of the gas, which is the result of the chemical reaction and the heat flux; the breaking down of walls and the coalescence of the pores, with diffusion of the small bubbles into larger ones due to the differences in pressure in the fresh stage of the material [16]. Henon et al. used free silicon for hydrogen gas production in order to create a porous AAM based on metakaolin. As a result a material with foamed pores ranging in size between 0.6 and 1.5 mm was created, with a material bulk density between 0.29 and 0.36 g/cm<sup>3</sup> [17]. By using peroxide as a pore forming agent, an AAM with a total porosity of up to 75% was obtained [18]. Zhao et al. has reported that a porous AAM, which was obtained from coal fly ash, sodium dodecyl benzene sulfonate and glutin, with a density of 0.414 g/cm<sup>3</sup>, a water absorption of 126.5%, and a compressive strength of 6.76 MPa, could be used as waste water filter [19].

This research deals with alkali activated industrial by-products such as waste metakaolin, recycled glass, ASRW, and steel-plant waste (SPW). It has been shown that components of ASRW, such as aluminium nitride (AlN) and iron sulphite (FeSO<sub>3</sub>), react in alkali media and create gases – ammonia and sulphur dioxide, thus providing a porous structure, so they could be used as a pore-forming agent [20].

The aim of the work described in this paper was (i) to create porous lightweight alkali activated building materials based on waste materials and residues as low Ca precursors, (ii) to describe the AAM properties which are affected by the choice of raw material.

## 2. Experimental

### 2.1. Testing methods

The particle size distribution of the powdered raw materials was determined by means of a Analysette 22 Nano Tec laser granulometer. Their specific surface area was detected by the BET

method ('Nova 1200 E-Series, Quantachrome Instruments'). Effective diameter was detected by Zeta potential ('90 Plus' and 'MAS Zeta PALS Brookhaven Instr.').

The mineralogical composition of the raw materials and the AAM was determined by X-ray diffraction (PANalytical X'Pert PRO), the beam energy and intensity being set to 40 kV and 30  $\mu$ A, and Cu K Alpha 1 radiation being used. X'Pert Highscore software with an ICDD PDF-2 data base, was used for the data processing. The chemical composition of the raw materials was determined according to LVS EN-196-2 with a sensibility of  $\pm 0.5$  wt%.

Structural characterizations of the different functional groups were performed for the raw materials and the AAM by means of a Fourier transform infrared (FTIR) spectrometer (VARIAN 800 FT-IR), between 400 and 4000 cm<sup>-1</sup>.

A Tescan Mira/LMU Scanning electron microscope (SEM) was used to investigate the microstructure of the raw materials, using 28 days old AAM samples. Chemical characterization was performed by means of EDX (energy dispersive X-ray spectrometry – EDS, Oxford instruments 7378).

Flexural and compressive strengths were determined according to LVS EN 1015-11, using test specimens with typical dimensions of 40  $\times$  40  $\times$  160 mm. The density of the AAM was measured in accordance with EN 1097-7, and water absorption according to EN 1097-6. Open porosity was determined by water absorption taking into account the volume of the prepared samples. The samples were immersed in water for 72 h. Total porosity was obtained from specific gravity obtained by using the Le Chatelier flask method (ASTM C188).

The temperatures which occurred due to the exothermic effects during the binding and hardening of the AAM were recorded using the methodology proposed by the company Alcoa [21].

The thermal conductivity was measured with a LaserComp FOX 660 heat flow meter instrument, using air-dried samples with typical dimensions of 300  $\times$  300  $\times$  50 mm. The upper and lower plate temperatures were 0  $^{\circ}$ C and 20  $^{\circ}$ C.

X-ray micro-computed tomography using an 'Xradia  $\mu$ CT-400' tomograph (XRadia, Concord, California, USA) was used to investigate the structural characteristics of the AAM specimens. The beam energy and the intensity were set to 80 kV and 125  $\mu$ A, respectively. 1600 projection images and an exposure time of 5 s per projection were acquired on the CCD camera, which was equipped with a 0.39X magnification optical objective. Due to the presence of large macro pores, the

X-ray micro-XCT scans were performed on a large sample size (40  $\times$  40  $\times$  40 mm), which consequently resulted in a lower resolution of 48  $\mu$ m. Avizo Fire 3D-image analysis software was used to reconstruct the three-dimensional internal pore structure of the samples, as well as for estimation of overall porosity and pore size

**Table 1**  
Chemical composition of the investigated raw materials (wt%).

Chemical component	Raw material			
	ASRW	SPW	MK	G
Al <sub>2</sub> O <sub>3</sub>	63.2	0.6	34.2	1.0
SiO <sub>2</sub>	7.9	3.0	57.2	68.1
CaO	2.6	7.0	0.3	1.4
SO <sub>3</sub>	0.4	3.3	–	–
TiO <sub>2</sub>	0.5	0.1	2.9	–
MgO	4.4	2.7	0.4	–
Fe <sub>2</sub> O <sub>3</sub>	4.5	42.0	1.5	0.2
MnO	–	2.7	–	–
PbO	–	1.7	–	20.0
Na <sub>2</sub> O	3.8	–	0.3	8.0
K <sub>2</sub> O	3.8	3.9	2.7	1.2
ZnO	–	18.0	–	–
Others	2.6	5.0	0.3	0.1
LOI, 1000 $^{\circ}$ C	6.2	10.0	0.2	–

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