Conference Paper

Development of Porous Tungsten Mud Waste-based Alkali-activated Foams with Low Thermal Conductivity

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Abstract

Aim of this study was to produce alkali-activated foams with low thermal conductivity. Different precursors' maximum particles sizes of 150μm, 300μm, and 500μm using a blend of 70% tungsten mining waste mud (TWM), 20% grounded waste glass (WG) and 10 % metakaolin (MK) with sodium silicate (SS) and sodium hydroxide (SH) as original material. Aluminium powder (Al) was used as a blowing agent and added first to the dry mix by changing content from 0.1g to 0.5g. Precursors and activators were mixed together to produce a homogeneous mixture, which was placed into a mould (100x200x60 mm^3), and cured in the oven at 60°C for 24 hours. The effect on foaming properties of different precursors maximum particles sizes were studied. The AAFs exhibited 28 day compressive strengths ranging from 2.28 to 16.1 MPa with the different densities from 913 to 1647 kg/m^3 achieved through alteration of the foaming content. The thermal conductivity of AAFs was in the range 0.21– 0.33 W/m*K. Open celled hardened of the AAFs with 0.5g Al shows a high porosity of 58% with the mix made with 500μm. Therefore, tungsten mining waste-based alkali-activated foams shows a promise as thermal insulation material in some situations.

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1. Introduction

Research in the field of alkali-activated materials has led to the development of several new or to the improvement of some types of alkali-activated foams [1]. Several examples of alkali-activated foams which have been obtained from different raw and waste materials, were reported in the literature [2] and they have been proposed as a new idea which involves the production of the lightweight materials, thus combining the performance and the benefits of energy saving (carbon footprint) with the reductions of the cradle-to-gate emission obtained [3–5], in order to reduce the density of the foamed alkali-activated materials [6, 7] holes or lightweight aggregates [8] can be added for such purpose.

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Alkali-activated foams can be made using different methods. The two main methods are pre-foaming and the mixed-foaming that have already been used to produce foamed materials [9]. Several foaming agents, as well as detergents, resin soap, glue resins, saponin, and hydrolysed proteins, such as keratin and similar materials [10] have been produced for this purpose. Among the methods used to produce alkali-activated foamed lightweight materials is the thermal expansion of (Na, K)-poly (silicate-multisiloxo) with ration Si:Al>>6 [11]. Various chemical foaming agents can be used in the foaming process of inorganic polymers [12]. Aluminium powder (Al) has been used to produce foams of inorganic polymers [13, 14] as a chemical foaming agent beside many others such as hydrogen-peroxide (H_2O_2) and sodium-perborate [15, 16].

In the last few years, several examples of alkali-activated foams (AAFs), obtained from different raw and waste materials, were reported in the literature using Al powder as a foaming agent, and the resulting foams had low thermal conductivity [2, 17, 18]. The search for AAFs offering high thermal insulation has increasingly become a target of the modern construction and building industry, as energy-efficiency of buildings in service has become of ever increasing concern [19, 20].

In the recent focus on global warming, insulating materials play an important role on the reduction of energy consumption, the concern on the thermal performance and fire resistance of building materials has become one of the research interests [13]. Research focused the attention on the reuse of waste materials by integrating them into new materials manufacture, protecting the environment and developing sustainable thermal insulation solutions [21].

The main objective of this paper is to investigate the fundamental properties of AAFs from tungsten mining waste mud (TWM), waste glass (WG), and metakaolin (MK) using aluminium powder (Al) as a foaming agent, including compressive strength, dry density and thermal insulation. Understanding of these properties is useful for the widespread uptake of this new material.

2. Materials and Methods

2.1. Materials

In the present study, a combination of tungsten waste mud (TWM), grounded waste glass (WG), and metakaolin (MK) were used as original materials by grouping them into

three different precursors' particle sizes (P1, P2, and P3) sieved under 150µm, 300µm, and 500 µm sieve respectively.

The tungsten mining waste mud (TWM) was collected from the deposit field (The Panasqueira tungsten mining waste mud in Covilhã, Castelo Branco District, Portugal), and drying in laboratory, as show in Figure 1. the waste mud (TWM) was sieved under three different particles sizes (150 µm, 300 µm and 500 µm) to obtain three different solid fineness of powders.
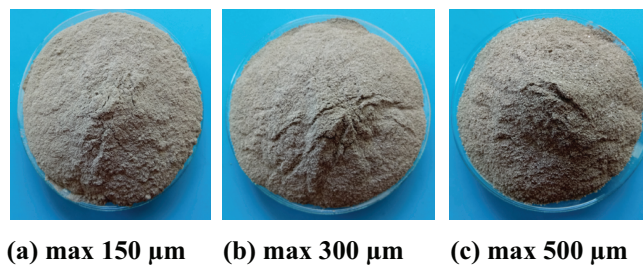

(a) max 150 µm   **(b) max 300 µm**   **(c) max 500 µm**

Figure 1: Images of the tungsten mining waste mud powders (a) max 150 µm (b) max 300 µm (c) max 500 µm.

Grounded waste glass (WG) was obtained from glass bottles by crushing and finer milling them. Waste glass was also sieved through (150 µm, 300 µm, and 500 µm) to obtain three particles sizes ranges that could be used as another source of silicate in the matrix. The images of the WG solid powder are presented in the Figure 2.
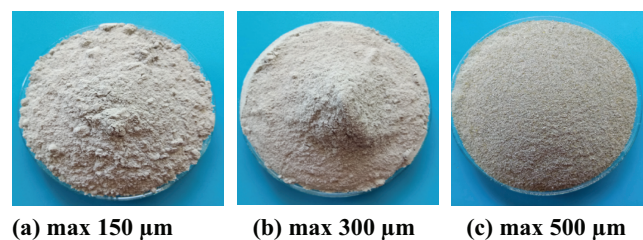

(a) max 150 µm   **(b) max 300 µm**   **(c) max 500 µm**

Figure 2: Images of the waste glass powders (a) max 150 µm (b) max 300 µm (c) max 500 µm.

Metakaolin (MK) provided from BASF with Specific Gravity of 2.50 (g/cm3) and pH = 6 (28% solids). metakaolin particles are shown in the Figure 3.

In addition, the aluminium powder (Al) as presented in Figure 4. was used for the foaming process, provided by ACROS organics with a purity and molar mass of 99% and 26.98 Al, respectively.

The typical chemical compositions of the powders are shown in the Table 1. The composition was determined, in several samples of TWM, WG and MK, through energy dispersive spectrometry (SEM/EDS) at UBI's (University of Beira Interior) optical centre.

Figure 3: Images of the metakaolin particles.

Figure 4: Images of the Al powder particles.

A commercial sodium silicate Na_2SiO_3 (SS) solution with the composition SiO_2/Na_2O = 3.2, and Sodium hydroxide NaOH (SH) (98.6% purity) pellets dissolved in distilled water were mixed to constitute the alkali activators, which were prepared at the laboratory of the University of Beira Interior (UBI). The chemical composition of the activators was given by suppliers (Quimialmel Quimicos e Minerais, Lda. José Manuel Gomes dos Santos, Lda.) are shown in Table 2.

TABLE 1: The chemical composition of TWM, WG, and MK characterized by SEM.

Chemical compound	TWM (%)	WG (%)	MK (%)
SiO_2	46.67	68.13	52.28
Al_2O_3	19.01	2.80	42.99
Fe_2O_3	15.47	2.90	1.49
SO_3	7.90	0.23	-
K_2O	4.90	0.86	0.94
Na_2O	0.85	12.52	0.32
CaO	0.69	10.52	-
MgO	4.83	2.04	0.47

TABLE 2: The chemical composition of the activators.

Oxide/Materials	Chemical composition of the activators (g)	
	Sodium silicate (SS)	Sodium Hydroxide (SH)
Na_2O	19.37	13.02
SiO_2	62.60	0.00
Al_2O_3	0.90	0.00
H_2O	142.32	43.27

2.2. Methods

2.2.1. Mixing alkaline solutions

The synthesis of the AAF includes three steps: (a) preparation of the activation solutions, after the magnetic stirring at a rate of 500 r.p.m for about 5 minutes of (10M) sodium hydroxide NaOH (SH) with liquid sodium silicate Na_2SiO_3 (SS), sodium hydroxide is used only 24 hours after preparation; (b) mixing of the raw materials (dry powders and Al powder) the precursors were prepared by dry mixing the powders; tungsten waste mud (TWM), grounded waste glass (WG) and, metakaolin (MK). Different measured amounts of Al powder from 0.1g to 0.5g were added to the precursors then all dry components were mixed well by hand for 1 minute to obtain a homogeneous pore distribution; and (c) moulding and curing the specimens after mixing solid components with the alkaline activating solution, which is alkali metal hydroxide and sodium silicate, for about 2 minutes at a fast speed. AAF mixes were manufactured with P/A = 2.5;1 of

precursor/activator ratio when using precursor P1 (under 150 μm), besides P/A = 3;1 when using precursors P2 and P3 (under 300 μm and 500 μm, respectively), and the same activators ratio of SS/SH = 3;1 (Sodium silicate/Sodium Hydroxide) to study the effects of different precursors' particles sizes on the expansion parameters by adding different amount of Al powder from 0.1g, to 0.5g. The reaction between the Al powder and the alkaline activator occur very quickly, as shown in the equation (1);

$$2Al + 2NaOH + 2H_2O \rightarrow 2NaAlO_2 + 3H_2. \tag{1}$$
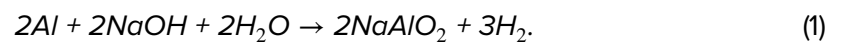
2.2.2. Mix design

Fifteen AAF mixes with all varieties of precursors (P1, P2 and P3) were produced with different amounts of Al powder; ranging from 0.1 to 0.5 g, as presented in Table 3. The mixes were poured into (100*100*60mm^3) covered acrylic moulds and placed firstly in the oven at 60 °C for 24 hours for curing to speed up the alkali activation.

TABLE 3: Mix proportions of the AAFs.

Precursors Particles sizes	Precursors (%)			Activators (%)		Al powder (g)	P/A
	TWM	WG	MK	SS	SH		
< 150 μm	70	20	10	74.99	25.01	0.1	2.5;1
	70	20	10	74.99	25.01	0.2	
	70	20	10	74.99	25.01	0.3	
	70	20	10	74.99	25.01	0.4	
	70	20	10	74.99	25.01	0.5	
< 300 μm	70	20	10	74.99	25.01	0.1	3;1
	70	20	10	74.99	25.01	0.2	
	70	20	10	74.99	25.01	0.3	
	70	20	10	74.99	25.01	0.4	
	70	20	10	74.99	25.01	0.5	
< 500 μm	70	20	10	74.99	25.01	0.1	3;1
	70	20	10	74.99	25.01	0.2	
	70	20	10	74.99	25.01	0.3	
	70	20	10	74.99	25.01	0.4	
	70	20	10	74.99	25.01	0.5	

After curing in the oven, the samples were removed from the mould and set aside to continue the curing in laboratory conditions, the samples were cut and prepared in the following days as shown in Table 4.

TABLE 4: AAFs tests.

Compressive strength (MPa)	Density (Kg/m^3)	Porosity	Thermal conductivity
3 Cubes (40*40*40)mm^3	D = m/V	P (%) = ($Ws - Wd$) / Wd × 100	2 slabs (100*100*30)mm^3

3. Results and Discussion

Table 5. shows the compressive strength, density, expansion volume, thermal conductivity and thermal resistance results of the AAF.

TABLE 5: Compressive strength, density, expansion volume, thermal conductivity, and thermal resistance results.

Compressive Strength (MPa)	Dry Density (Kg/m^3)	Expansion volume (%)	Thermal conductivity (W/m.K)	Thermal resistance (m^2*K/W)
16.1	1647.59	11.50	0.3340	0.0898
9.5	1331.89	29.63	0.3142	0.1185
5.09	1178.47	39.81	0.3003	0.1247
3.65	1126.41	41.21	0.2734	0.1382
4.25	1081.62	42.90	0.2466	0.1469
14.2	1518.18	19.05	0.3070	0.0985
4.25	1255.54	36.20	0.2893	0.1031
4.28	1090.80	41.46	0.2626	0.1122
3.23	1054.14	45.31	0.2448	0.1265
3.03	1038.09	48.57	0.2340	0.1315
10.97	1517.10	23.86	0.3408	0.0878
4.02	1266.07	40.84	0.2647	0.0947
3.58	1119.04	44.26	0.2335	0.0985
2.45	981.55	50.37	0.2209	0.1022
2.28	913.26	57.56	0.2116	0.1204

3.1. Compressive strength

The compressive strength test for the alkali-activated foams was carried on AUTO-GRAPH AGS-X, 10 KN SHIMADZU testing equipment. For each mixture, three 40 x 40 x 40 mm^3 cubes were tested. The cubes were obtained by cutting for the casted 100 x 100 x 60 mm^3 with a diamond cutting blade.

The compressive strength results are presented in Figure 5. It is well observed that the highest compressive strength values are achieved with samples the less amount of Al powder that is with less porosity. Compressive strength results for all samples with diverse amounts of Al powder ranged between 2.28 MPa and 16.10 MPa for all samples.

Therefore, the increase of Al powder amounts decreases of the density and high porosity which results in a gradual decrease of the compressive strength. As it was expected, the compressive strength is strongly affected by the porosity of the AAFs.
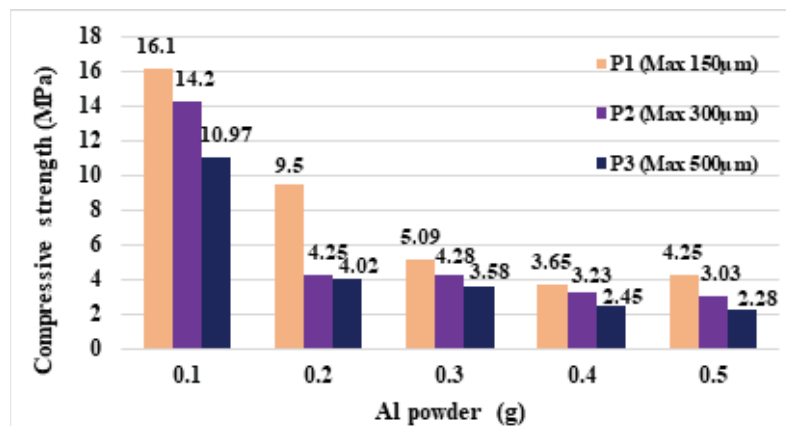

Figure 5: The compressive strength of the AAF at 28 days.

3.2. Dry density and porosity

The density of the foams was determined by measuring the weight of specimens with a precision balance scale and their dimensions with a precision electronic calliper. The density (d) of the samples was computed by dividing the dry mass (m) by the volume (v) (i.e. so-called geometrical density). Cubic specimens (40*40*40mm^3) were used for the dry density measurements.

The dry density of the AAF is reported in Figure 6. It is clearly observed that the fineness particle size of the precursor (mainly mining waste mud) does not lead to a low density but the increase of the porosity does. The lowest density for each precursor P1, P2, and P3 was achieved at about 1081 Kg/m^3, 1038 Kg/m^3, 913 Kg/m^3 respectively,

when adding 0.5g of the Al powder amount. Furthermore, the increase of the Al powder amount enlarges the pore size of the samples, and a numbers of voids inside the material filled with air could be generated, thus resulting in the reduction of the density.
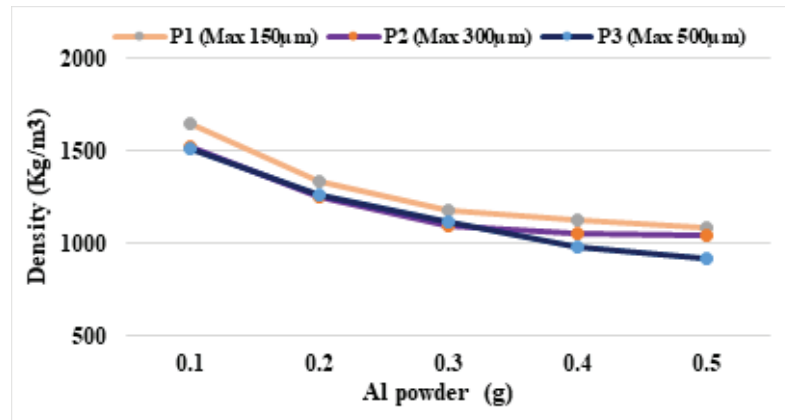

Figure 6: The relation between the dry density and the Al powder amounts of the AAFs.

3.3. Thermal conductivity

The tests for the determination of the thermal conductivity were carried out on a **NETZSCH HFM 436/3 Lambda** flow meter at **Sofalca Lda** (Estrada Nacional 2, Km 412,2 Bemposta, 2205-213 Abrantes, Portugal). The thermal conductivity tests were carried out in accordance with the European Standard **EN 12667: 2001-en** - "Thermal performance of building materials and products - Determination of thermal resistance by means of guarded hot plate and heat flow meter methods - Products of high and medium thermal resistance". The specimens were preconditioned for a period of 120 h, in an environment characterized by an ambient temperature of 23 ± 2 °C and a relative humidity of 50 ± 5 % and the thermal conductivity tests were carried out at the same environment of 23 ± 2 °C temperature and 50 ± 5 % relative humidity. The specimens were tested in the dry state at a mean temperature of 10 ± 0.3 °C and dried in an oven at about 70 ± 2 °C. The accuracy of the determination of this thermal parameter is estimated at about ± 2.8 %.

The average thermal conductivity values obtained for the foams are presented in Figure 7. The alkali-activated foams mix P3-F5 have the lowest thermal conductivity value, equal to 0.21 W/m*K, while P1-F1 foams display the highest one 0.33 W/m*K. Thermal conductivity depends, for a large part, on the total pore volume achieved after the foaming process; indeed, thermal conductivity decreases by increasing the total porosity, as shown in Figure 7 the higher the porosity, the lower is the thermal

conductivity. This is because a higher porosity means more voids and the thermal conductivity of air within the voids is much lower than that of solid substance, as consequence, an overall lowering of the thermal conductivity of the material, since the thermal conductivity of air within the voids is much lower than that of solid substance [22, 23].
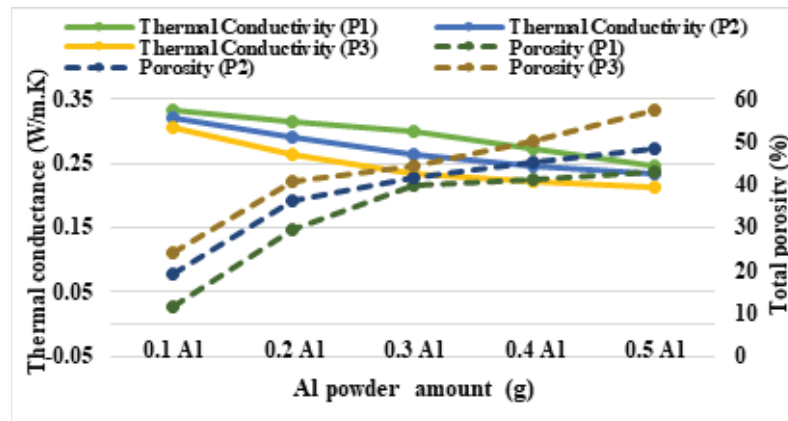

Figure 7: Relationship between total porosity and thermal conductivity of the AAFs as a function of Al powder content.

4. Conclusion

The study was a development of AAFs using three different precursors' (P1, P2, and P3) maximum particles sizes of (150 μm, 300 μm, and 500 μm) respectively, from a combination of 70 % tungsten mining waste mud (TWM), 20 % waste glass (WG), and 10 % metakaolin (MK), by the chemical foaming with aluminium powder (AL) as a blowing agent. The following findings were made:

- Highly porous foams were obtained through the increase of the Al powder amount resulting in the decrease of the density of the AAFs.

- The lowest density for each precursor P1, P2, and P3 was achieved at about 1081 Kg/m^3, 1038 Kg/m^3, 913 Kg/m^3 respectively, when adding 0.5g of the Al powder amount.

- The compressive strength results of the produced AAF are relatively good (4.25 MPa, 3.03 MPa, and 2.28 MPa were obtained for the higher porosity of P1, P2 and P3 respectively).

- The thermal conductivity of AAFs was in the range 0.21– 0.33 W/m*K.

- The results proved that the Al powder has a potential impact on the reduction of thermal conductivity of AAFs by creation of a porous system.

- Open celled hardened of the AAFs with high porosity 58%. Hence, AAFs tungsten mining waste-based shows promise as thermal insulation material in some situations.

Acknowledgment

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