

## **Alkali-activation of tungsten mining waste mud blended with waste glass: reactivity, performance and innovative applications**

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### **Abstract**

Mines and quarries waste accounts for about 29% of the total generated waste from industrial processes and households in Europe. The reuse of mud tailings as precursor materials for alkali-activated binders and applications are very promising from a technical and environmental point of view. The aim of the present study was to develop added-value solutions to reutilize tungsten mine mud tailings in alkali activated materials. Some potential uses of these new materials obtained from alkali-activation of tungsten mud waste and other precursor materials, as well incorporating expanded granulated cork, such as a high energy efficient panel for a vegetated surface, and a foamed lightweight brick wall, are presented.

### **Keywords**

Alkali-activation; Recycling; Tungsten mining waste; Waste glass; Waste glass



# Alkali-activation of tungsten mining waste mud blended with waste glass: reactivity, performance and innovative applications

## 1. Introduction

The World is facing serious environment problems caused by global warming and climate changes, mainly because of the increase of carbon dioxide (CO<sub>2</sub>) in the atmosphere [1]. Approximately 5-7% of global anthropogenic CO<sub>2</sub> emissions results of the manufacturing of Ordinary Portland cement (OPC) [2,3]. In 2016, the global anthropogenic CO<sub>2</sub> emissions increased approximately 8% with an estimated total value of about 1.45 ± 0.20 Gt CO<sub>2</sub> [4]. Consequently, the development of alternative low-carbon binders is recognized as a smart option to reduce CO<sub>2</sub> emissions [5]. The alkali-activated materials (AAM) technology brings a new type of cementing materials that don't produce CO<sub>2</sub> emissions like the case of Portland cement (PC) industry [6]. Thus, there is a growing demand to replace PC concrete by alkali-activated concrete. In fact, these innovative alkali-activated construction materials under certain conditions have lower greenhouse gas emissions, causing lower environmental impacts and CO<sub>2</sub> emissions that are associated with their manufacture [7].

Household and industrial waste management remains an issue worldwide particularly in Europe. European economic activity and households produced 2.5 billion tons of wastes in 2012, which 62% comes from construction and mining and quarrying activities, while manufacturing, households, energy and other economic activities produce the other 38%. Mines and quarries waste accounts for about 28% of the total generated waste from industrial processes and households in the EU [8]. Most mining and quarrying and other industrial wastes can be reused in earthworks and construction, namely the coarser fractions into asphalt pavements and concrete [9]. It can also become raw material for industrial applications where the high value of the product does not prejudice its reuse due to transport costs [10]. Other research studies have been focused in developing technical-artistic value added and polymer-based composite materials [11].

The reuse of fine particles from mud tailings as precursor materials for alkali-activated binders and applications have also been conducted and are very promising from a technical, environmental and economic point of view [12,13]. Besides, the alkaline activation of aluminosiliceous industrial by-products is widely known to yield binders whose properties make them comparable to or even stronger and more durable than ordinary Portland cement [14-16]. Besides, several non-conventional waste materials are re-used nowadays as precursors in the alkaline activation, that can be blended/combined with mining waste mud tailings with promising results [17].

This study evaluated the results to produce alkali-activated binders (AAB) by blending tungsten mine waste mud (TMW) and fine waste glass (WG) activated with a composition of sodium silicate (SS) and sodium hydroxide (SH). This research work is part of REMINE research program (H2020 RISE-Marie Curie Action) that aims to the reuse of mining waste into innovative geopolymeric-based structural panels, precast, ready mixes and in-situ applications. The project is coordinated by Beira Interior University (PT) with the following partners: Brunel University (UK), Silesian University (PL) Bologna University (IT), Granada University (SP), Strathclyde University (UK), Kiev National University of Civil Engineering and Architecture (UA), Alsitek Ltd (UK). Sofalca, Lda (PT), Beira Serra (PT)) [18].

Some potential uses of these new materials obtained from alkali-activation of tungsten mud waste and other precursor materials, as well incorporating expanded granulated cork, such as



a safety barrier, a high energy efficient panel for a vegetated surface, and a foamed lightweight brick wall are presented [19,20].

## 2. Experimental

### 2.1. Materials

The main materials used in this investigation consisted of tungsten mine waste mud (TMW) from Panasqueira mine in Covilhã, Portugal, milled waste glass (WG), sodium hydroxide (NaOH) (SH), and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) (SS). Some specimens tested in this research also included tungsten mine coarse wastes (TCW), metakaolin (MK), granulated expanded cork (GC) and aluminum powder (AL). Metakaolin was provided by the German chemical company BASF, while GC was supplied by Sofalca Lda, located in Abrantes, Portugal). The study was carried on using natural (as obtained in deposit) (N-TMW) and heat-treated ( $950^\circ\text{C}$  for two hours) (C-TMW) tungsten mine waste mud. WG was produced after milling for 6 hours using a ball mill. Both TMW and WG were used with particle size lower than  $300\mu\text{m}$ , obtained by sieving analysis. The chemical composition of the TMW and WG was obtained by SEM-EDS. Table 1 shows the average values of TMW (natural and thermal treated) and WG chemical composition. The density of precursor materials was obtained by helium pycnometer (Micromeritics AccuPyc 1330). TMW density was  $3,031\text{ g/cm}^3$  for N-TMW and  $2,950\text{ g/cm}^3$  for C-TMW. Specific surface was obtained by the Blaine method (ACMEL LABO BSA1). C-TMW specific surface is slightly lower ( $2117\text{ cm}^2/\text{g}$ ) than N-TMW ( $2417\text{ cm}^2/\text{g}$ ). Grain size distribution analysis was made for TMW and WG by laser diffraction analysis. The TMW has a mean particle size of  $12,1\mu\text{m}$  (N-TMW) and  $16,3\mu\text{m}$  (C-TMW) while the WG has a mean particle size of  $39,7\mu\text{m}$ . Sodium hydroxide solution was prepared by dissolving sodium hydroxide pellets (98% purity obtained from Fisher Scientific, Schwerte, Germany) in deionized water and allowed to cool before use. Sodium silicate (obtained from Solvay SA, Póvoa de Santa Iria, Portugal) had a  $\text{SiO}_2/\text{Na}_2\text{O} = 3.23$  (8.60% by weight  $\text{Na}_2\text{O}$ , 27.79% by weight  $\text{SiO}_2$ , 63.19% by weight  $\text{H}_2\text{O}$ , and 0.4% by weight  $\text{Al}_2\text{O}_3$ ).

Table 1- Chemical composition (% by weight) of TMW and WG determined by SEM-EDS (Supra 35VP/EDAX, Oberkochen, Germany)

Chemical compound	N-TMW	C-TMW	WG	MK
$\text{SiO}_2$	49.33	49.66	73.93	52.28
$\text{Al}_2\text{O}_3$	16.28	14.42	0.00	42.99
$\text{Fe}_2\text{O}_3$	13.67	22.44	0.40	1.49
$\text{SO}_3$	8.93	4.70	0.00	0.00
$\text{K}_2\text{O}$	4.38	4.44	0.69	0.94
$\text{Na}_2\text{O}$	0.64	0.44	9.72	0.32
CaO	0.83	0.97	12.83	0.00
MgO	4.93	1.80	0.00	0.47
$\text{TiO}_2$	1.00	1.13	0.00	0.00

### 2.2. Mixing and curing conditions

All sample preparation was carried on at room temperature (around  $20^\circ\text{C}$ ). Alkali activated binders (AAB) were produced by blending TMW with WG and MK, with different percentages. For the constituents of the AAB the following parameters were selected: Molarity of SH= 10M; Weight ratio of SS:SH=4 and Weight ratio of precursor/activator = between 2.8 to 3 (except for samples containing higher percentage of MK were lower ratio, between 1 to 2, was used). To produce AAB samples TMW, WG and MK were first mixed in dry state for about 3 minutes. The SS and SH were also mixed together for a period about 5 minutes. Then alkali-activator solution was stirred together with the dry mix for about 5 minutes. To produce Alkali activated concrete (AAC) tungsten coarse wastes (TCW) were added to the composition and mixed in the dry state with TMW and WG, before adding the alkali-activator solution. To produce lightweight alkali-



activated materials (L-AAM) granulated expanded cork (GC) was added to the mixture of TMW and WG and stirred in the dry state. To produce foamed alkali-activated materials (F-AAM) aluminum powder (AL) was added with different percentage to the dry mix (TMW, WG, MK) and mixed with all together for additional one minute. The resulting AAB, AAC, L-AAM F-AAM mixes were then placed in prismatic molds of different sizes (accordingly to different tests) and cured in oven at 60°C for 24 hours, prior to testing. Most details of mix preparation and curing procedures were developed in previous studies [21].

### 3. Results and discussion

#### 3.1. Binders compressive strength

The mechanical properties of alkali-activated binders (AAB) were evaluated by compressive strength testing. The formation of organic compounds was analyzed by Fourier Transforms Infrared Spectroscopy (FTIR). The highest compressive strength (65 MPa at 28 days) was achieved for samples containing 20% waste glass, as presented in figure 1. Overall, the best mechanical behavior was found out on binder specimens with natural waste mud (N-TMW) instead of C-TMW. AAB produced with less than 25% waste glass exhibit a very good mechanical behavior, at 28 days, increasing strength along time. On the other hand, AAB mixes containing more than 25% WG (higher CaO content) showed a decrease in compressive strength over time. While, AAB containing less than 5% waste glass showed a slow alkaline activation reaction. The FTIR analysis revealed a reduction in the absorption intensity, of the main bands, for lower TMW content and along curing time. It indicates that higher WG content and time of curing led to the further dissolution of Si-O from the TMW.

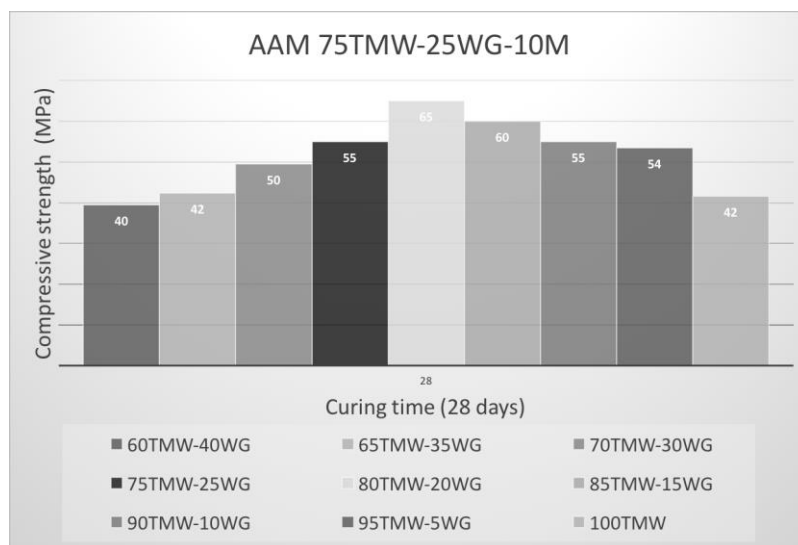


Figure 1 - Compressive strength at 28 days of AAM containing between 5 to 40% WG

#### 3.2. Reactivity based on chemical leaching

Reactivity of AAB's with different composition was evaluated by chemical leaching based on electrical conductivity measurements. For this procedure 2.5 x 2.5 x 2.5 cm cubic specimens were used, after being cured for 7 days. First, specimens to be tested were placed in a goblet containing distilled water at a constant level of 150 ml. Then the electrical conductivity was measured by means of a probe that recorded values from hour to hour until reaching a constant electrical conductivity value. Since the electrical conductivity changes with the number of free ions that migrated from the samples to the distilled water, the higher the sample conductivity, the lower the alkaline activation reactivity extent. Thus, from table 2 results we can conclude



that the best reaction can be obtained on AAB mixes where TMW is replaced by 10% weight of both WG and MK (i.e. AAB mix = 80%N-TMW + 10%WG + 10%MK).

Table 2. Electrical conductivity of AAB mixes, containing N-TMW, WG and MK

AAB mix composition	Conductivity ( $\mu\text{s}/\text{cm}$ )	Number of hours to reach a constant conductivity value
20%N-TMW + 80%WG	14014	99
50%N-TMW + 50%WG	17781	330
80%N-TMW + 20%WG	15894	50
80%N-TMW + 10%WG + 10%MK	12908	64
100% N-TMW	14610	428
100% WG	10000	66
100% MK	4122	40

### 3.3. Thermal resistance after 800°C temperature exposure

Thermal resistance of AAB's was evaluated after being exposed to 800°C temperature, for 2 hours' period. For this study, 15 different AAB mixes were produced (see table 3) with a precursor/activator ratio of 2.85, except for mixtures 100%MK and 50%WG+50%MK where this ratio was 1.1 and 2.17, respectively. Compressive strength tests were performed on cubic specimens with 4 cm size and were carried on in two phases. Initially, AAB specimens were tested for compressive strength. Later, specimens with the same chemical composition were tested for compressive strength after being exposed to 800°C temperature, for 2 hours' period. A static furnace (muffle) was used for the temperature exposure. Before the exposure period (800°C for 2 hours), the temperature was gradually increased. Based on results obtained, most of AAB's produced with N-TMW, WG and MK present an increase of compressive strength when exposed to 800°C. Besides the presence of GC, as a lightweight aggregate, is also beneficial regarding the increase of compressive strength of different AAB' mixes after being exposed to 800°C.

Table 3. Thermal resistance of AAB mixes at 800°C, containing N-TMW, WG, MK and GC

ABB mix composition	Weight loss (%)	Initial compressive strength [MPa]	Compressive strength, after 800°C [MPa]
100%N-TMW	7,99	0,00	21,40
100%WG	7,45	16,36	0,00
100%MK	13,67	20,42	17,84
80%N-TMW+10%WG-10%MK	11,08	10,58	29,06
80%N-TMW+20%WG	8,71	3,70	30,48
80%N-TMW+20%MK	8,82	18,44	24,72
60%N-TMW+20%WG+20%MK	9,93	17,90	23,80
55%N-TMW+27%WG+18%MK	10,92	21,04	30,26
50%N-TMW+25%WG+25%MK	8,67	30,90	25,98
50%WG+50%MK	10,03	21,40	34,34
80%N-TMW+10%WG+10%MK+10%GC	8,69	10,44	26,52
80%N-TMW+10%WG+10%MK+20%GC	8,86	7,63	28,98
60%N-TMW+20%WG+20%MK+10%GC	8,59	17,38	23,56
55%N-TMW+27%WG+18%MK+10%GC	10,68	19,80	26,12
55%N-TMW+27%WG+18%MK+20%GC	11,07	10,94	41,54

### 3.4. Concrete compressive strength and elastic modulus

The elastic modulus of alkali activated concrete (AAC) produced with tungsten coarse wastes



(TCW) was also determined in this study. Seven prisms and one cube were subjected to the tests at the 28<sup>th</sup> day. The elastic modulus (EM) results are presented in table 4. It shows a variation between 1.20 to 7.96 GPa. The EM was obtained by three different Standard calculation methods: The ASTM method with a result of 3.13 GPa, per European Standard with values of 5.28 GPa and 4.75 GPa and per LNEC method a value of 4.30 GPa. The overall average of the results was 4.36 GPa. The presented results show a five times smaller elastic modulus when compared with the elastic modulus of an ordinary Portland cement concrete through the standard ACI 318-89 which is 22.30 GPa [22]. Therefore, results indicate that AAC is more elastic than ordinary Portland cement concrete.

Table 4. Elastic modulus of AAC produced with TCW

ACC specimen	Compressive strength (MPa)	Elastic Modulus (GPa)			
		ASTM	ASTM		ASTM
			$E_{c,0} = E_{c,s} (A)$	$E_{c,s} (B)$	
Prism 1	25.09	3.40	5.35	4.25	3.85
Prism 2	23.90	3.95	4.70	4.23	3.81
Prism 3	21.40	2.82	3.86	3.51	3.23
Prism 4	17.27	3.24	3.87	3.27	3.38
Prism 5	26.52	1.20	7.96	7.07	6.52
Prism 6	19.09	1.65	5.15	5.14	4.60
Prism 7	22.71	5.65	6.07	5.77	4.69
Average	22.28	3.13	5.28	4.75	4.30

## 4. Innovative applications

### 4.1 Modular system for vegetated surfaces

GEOGREEN is a modular system of prefabricated elements with vegetation, suitable for new buildings and retrofitting. Its concept is based in a new design for a modular living wall system (LWS) which incorporates industrial waste materials and industrial sub-products. Each module includes a base in an alkali activated material (AAM) and a top layer of expanded cork board (ICB). These modules were designed to be adaptable to different supports, and to obtain a continuous and uniform layer of vegetation. The system can be applied manually and each module can be easily removed for maintenance purposes. The materials and plants used in the system aim to minimize the irrigation needs, to improve buildings thermal behaviour [23] and their acoustic conditions [24].



Figure 2. GEOGREEN module: with lower layer in alkali-activated binder (a) and upper layer in expanded cork with circular openings (b).



The alkali activated precast base used in this system is obtained from the mixture of two industrial wastes used as precursors, mine waste mud from Panasqueira mine and milled glass. Milled glass is used to increase the amount of amorphous material in the mixture. The binary blend is activated using a composite activator based on a mixture of sodium silicate and sodium hydroxide. Expanded cork granules are also added to the mixture. They are a lightweight aggregate which result from the crushing of waste expanded cork agglomerate. In the alkali activated mixture they are used to reduce the binder density and increase its water absorption. The mixture is casted into a plastic mold and subject to a curing process in a ventilated oven at 60°C during 7 days.

Several alkali-activated mixtures were prepared using different variables and aggregates [25]. Variables as percentage substitution of mine waste per glass waste, molar concentration of sodium hydroxide, cure length and temperature, were tested to identify the reference mixture. After these tests, different percentages of aggregates as sand, expanded cork granules and expanded clay were added to reference mixture. Results indicate that the mixture with 25% of sand obtained the maximum compressive strength of 35MPa after 7 curing days. Capillary absorption coefficient can reach to 4,77 kg/m<sup>2</sup>.h<sup>0,5</sup> when adding 25% of cork granules and to 4,11 kg/m<sup>2</sup>.h<sup>0,5</sup> with when adding 25% of sand. Also, if the addition of cork granules is increased to 50% it enables a 20% density reduction when compared to the reference mixture.

#### 4.2. Alkali-activated foamed materials

Recently, novel ideas have been proposed for the manufacture of alkali-activated foamed materials to produce lightweight materials combining the performance and the benefits of energy saving with the emission reductions obtained [20,26] . The two main ways of producing foamed materials are by introducing mechanically pre-formed foam into the binder or by using a chemical foaming technique.



Fig.... Photographs of alkali-activated foam sample.

Alkali-activated materials have been foamed for suitable thermal conductivity and low density for the application of new types of thermal insulation materials [27]. The processes used to manufacture alkali-activated foam materials have been the same as for conventional aerated concrete, i.e. pre-foaming or mixed foaming [28,29]. Nevertheless, the porosity and thermal insulation properties of alkali-activated foamed materials prepared by the mixed foaming process have not exceeded those of traditional porous materials such as glass foam or autoclaved aerated concrete. Currently, these are the problems limiting the use of alkali-



activated foam as alternative thermal insulation materials to compete with traditional porous inorganic materials.

Besides foaming, density reduction of alkali-activated materials can be done by using lightweight aggregate as alternative to the normal weight granules, where they show better thermal insulating features [30]. Expanded granulated cork has been studied in various industries as lightweight aggregate and the fact that cork is a natural product their usage has eco-efficient advantages [31,32].

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