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OPTIMIZATION OF THE ALKALINE ACTIVATION OF PRE-TREATED MUNICIPAL SOLID WASTE INCINERATOR BOTTOM ASHES

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Keywords

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Abstract

Alkaline activation of wastes with a high content of amorphous aluminosilicates generates reaction products somewhat similar to those produced in the hydration of conventional cement and often with equivalent mechanical performance, thereby making it possible to manufacture cement-free construction materials. In the present research, alkali-activated mortars were elaborated, using municipal solid waste incinerator bottom ash (MIBA) as precursor (around 16 million tonnes of MIBA are currently generated per year in the EU). NaOH and Na_2SiO_3 were used as alkaline activators, the amounts of which varied in relation to the Na_2O/binder (4%, 6%, 8%, 10%, 15%) and SiO_2/Na_2O (0, 0.5, 1.0, 1.5 and 2.0) factors, in order to determine the optimal values at which peak mechanical performance is achieved. Since the precursor under study contains 5.46 g of Al per kg of MIBA, which reacts at alkaline pH to produce hydrogen, a pretreatment stage was necessary in order to avoid the production of this gas in the fresh state mixes. This minimized the occlusion of gas pockets in the mortar in the hardened state. Tests carried out on the mortars corresponded to the density and workability in the fresh state, and compressive strength and modulus of elasticity in the hardened state. In parallel, an experimental campaign was carried out with the same criteria in terms of mix designs and properties evaluated, using fly ash, which corresponds to the control material. A 24-hour resting time allowed the stabilization of MIBA in the manufacturing process and thermal curing of AAM but did not inhibit the reaction of the remaining aluminium with Na_2SiO_3, which led to increased internal porosity, while affecting the workability of the mix MIBAs in the fresh state. After achieving the optimum levels of Na_2O/binder and SiO_2/Na_2O for each precursor, it was established that MIBA mortars presented worse mechanical performance when compared with corresponding fly ash mortars.

1. INTRODUCTION

Simulating the chemical process that occurs in the cement´s hydration, to form reaction products with mechanical properties and durability similar to those generated by it, is considered a constant challenge for the scientific community. Understanding in depth new methodologies for the manufacture of binders, with the objective of developing international standards for the future commercialization of new products, values each stage and/or contribution, since they contribute to the detailed and in-depth knowledge of a new material [1][2]. The first reported patent for Portland cement was in 1824, but only until 1870 was it patented in countries like the United States [3], therefore, nearly 50 years passed in which the scientific community was contributing from various areas to finally have the widely disseminated material that is known today, of which, at the present, its production exceeds 1600 billion kg [4]. A contrasting situation occurs with alkali activated materials (AAM), which obtained their first patent in 1908 [5] but nowadays, they are not widely marketed.

AAM are developed through the chemical reaction of an amorphous aluminosilicate- and/or calcium-based precursor with an alkaline solution such as sodium hydroxide (NaOH), obtaining reaction products with mechanical properties and durability similar to those obtained in the hydration of Portland cement [6][7][8]. The precursors of alkaline activation have been categorized by Provis and Van Deventer [1] into two large groups: precursors rich in calcium such as blast furnace slag, type C fly ash (FA), or municipal solid waste incinerator bottom ash (MIBA) and those low in calcium such as type F FA, metakaolin, among others; each group with its own characteristics regarding the products formed, and the properties in fresh and hardened state of the AAM. Type F FA has been one of the most studied precursors since the reported compressive strengths exceed 59 MPa at 28 days [9], showing excellent mechanical and durability characteristics [10], against other precursors such as MIBA, in which authors, such as Carvalho et al. [11] reported compressive strength values of less than 6 MPa at 28 days. The considerable decline in performance was associated to three factors: the release of hydrogen from the reaction of metallic aluminium present in the MIBA with the alkaline solution; the low amorphous content of calcium, silicon and aluminium; and non-incinerated waste content. Owing to some of the shortcomings that this last precursor presents, Huang et al. [12] evaluated two pre-treatment options, which consisted of the controlled elimination of the hydrogen generated in a fresh state, through a retention time of 3 hours of the slurry before its use and the calcination of the MIBA at 1050 °C for 30 minutes. The authors reported that the defoaming step allowed a strength increase from 2.4 MPa to 8.4 MPa at 28 days. In parallel, the calcination pre-treatment stage resulted in a compressive strength of 10.4 MPa at 28 days, thereby showing the necessity of further beneficiation processes to valorise this material. Another challenge that MIBA presents is the phenomenon of efflorescence, for which Huang et al. [13] proposed sealing the specimens during the curing period, in order to avoid the migration of compounds and exchanges of humidity with the environment. This hindered the movement of the alkaline cation and the ion hydroxyl (OH^-) present in the activator, thereby providing them additional time to react with the precursor. In this study, binary mixes were made with two precursors, MIBA and granulated blast furnace slag (GBFS), and sodium silicate (Na_2SiO_3) and NaOH were used as alkaline activators. The compressive strength was greater than 50 MPa at 28 days, reporting an improvement in the mechanical property compared to the 30 MPa of the control material.

Even with the reported drawbacks, MIBA is a noticeably valuable precursor for AAM production, and thus should not be disposed of in landfills. Discovering new outputs is especially important considering that the world generation of this waste increases with the growing population; in Europe alone, about 74.7 million tonnes of municipal solid waste are generated [14], therefore, investigating its reuse to enter the supply chain represents a high advantage from an environmental point of view [15]. This study evaluated the density and workability of mixes in the fresh state and the compressive strength and dynamic modulus of elasticity in the hardened state of alkali-activated pre-treated MIBA, using NaOH and sodium silicate (Na_2SiO_3) as alkaline activators. Type F FA-based mortars were produced as control AAM under the same conditions as those made with MIBA. Both precursors were characterized using X-ray fluorescence (XRF) and laser diffraction particle size distribution, and their density was tested using the helium pycnometer technique.

2. MATERIALS AND METHODS

2.1. PRECURSORS

In this study, MIBA was provided by the Valorsul company, in São João da Talha, Portugal. The sample was dried at 105 °C, cleaned to remove non-incinerated garbage and finally ground in 3 stages, achieving a particle size distribution between 0.5 µm and 120 µm determined by laser diffraction analysis. MIBA pulverized density was determined by the helium pycnometer technique, obtaining a value of 2.704 g/cm^3. FA was provided by the company EDP - Gestão da Produção de Energia, S.A. at the Sines Production Center factory with a particle size distribution between 0 µm and 120 µm and a density of 2.425 g/cm^3. The chemical composition by XRF for MIBA and FA is shown in Table 1.

Table 1. Chemical compositions of the precursors.

Oxides	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SO_3	Cr_2O_3	TiO_2	ZnO	P_2O_5	V_2O_5	CuO	MnO_2
FA (%)	25.6	2.28	6.92	2.75	1.83	1.30	56.4	0.80	0.49	1.14	0.02	0.44	0.05	0.00	-
MIBA (%)	8.85	18.3	6.69	1.59	4.02	6.55	48.9	1.36	0.06	0.48	0.35	2.52	-	0.16	0.12

2.2. ACTIVATORS

The alkaline solution is composed of liquid Na_2SiO_3, with a relative density of 1.36 g/ml, which contains 26.4% silicon oxide (SiO_2), 8% sodium oxide (Na_2O) and 65.6% water, and NaOH with 98% purity and relative density of 2.13 g/ml. Tap water according to Directive 98/83/CE (CEU, 1998) was used for the preparation.

2.3. AGGREGATES AND SUPERPLASTICIZER

Fine sand 0/1 and coarse sand 0/4 were used as aggregates, according to the EN 12620 standard [16]. Dry density was determined as 2620 kg/m^3 for fine sand$_{0/1}$ and 2610 kg/m^3 for coarse sand$_{0/4}$, water absorption for fine sand$_{0/1}$ was 0.2% and for coarse sand$_{0/4}$ was 0.3%, the particle size of fine sand$_{0/1}$ varies from 2 mm to 0.0625 mm and, for coarse sand$_{0/4}$ varies from 8 mm to 0.0625 mm. SikaPlast 717 was used as water reducing admixture, which contains a combination of water-based synthetic organic dispersants with a pH of 10 ± 1.

2.4. MIX DESIGN

22 combinations for the preparation of the alkaline activator from the ratios Na_2O/binder (4%, 6%, 8%, 10%, 15%) and SiO_2/Na_2O (0, 0.5, 1.0, 1.5 and 2.0) were tested. The amount of Na_2O in both ratios corresponds to the sum of the present contributions of this compound in the Na_2SiO_3 and NaOH. The water/binder ratio was 0.5, in which the amount of water provided by the sodium silicate is included. The amount of tap water to be added to each combination was used to prepare the NaOH solution in the MIBA pre-treatment stage. Heat generated by the dissolution of the NaOH in water was used to accelerate the corrosion reaction of the aluminium. The mix was left to rest of 24 hours. After the treatment, the Na_2SiO_3 was added (when applicable) to the rest of materials to produce the AAM mixes. The superplasticizer/binder ratio was 2%, wherein 1% was used in the pre-treatment stage and then another 1% was used 24 hours later in the mixing process. For mixes with FA precursor, the superplasticizer was not required. The ratio of the sands corresponded to 30% fine sand and 70% coarse sand, and the volume binder/volume aggregate ratio was 0.33. Each mix was named with the initial letter of binder (M) next to the percentage of Na_2O/binder and the denominator was the SiO_2/Na_2O ratio. When the mortars were made, they were left for 24 hours at environmental conditions, covered by a plastic film and, after that, were cured at a temperature of 80 °C for 24 hours more. Finally, the mortars were demoulded and placed in a dry chamber at 20 ± 3 °C and a relative humidity of ~60% until the day of the test.

2.5. TEST METHODS IN MIBA AND AAM

The aluminium content present in MIBA was determined through an experimental test, in which the hydrogen generated was quantified from the reaction of 10 g of MIBA with a 2.5 M sodium hydroxide solution. The mixes in the fresh state were tested according to EN 1015-6 [17] and EN 1015-3 [18] for density and workability, respectively. The AAM in the hardened state were evaluated at 28 days of curing according to the standard EN 1015-11 [19] and ASTM-E1876 [20] for compressive strength and dynamic modulus of elasticity, respectively.

3. RESULTS AND DISCUSSION

3.1. QUANTIFICATION OF METALLIC ALUMINIUM IN MIBA

Figure 1 shows the laboratory setup to quantify metallic aluminium in MIBA. The chemical reaction to produce gaseous hydrogen from the reaction between metallic aluminium and NaOH solution corresponds to equation 1.

$$2Al^0 + 2NaOH + 6H_2O \rightarrow 2NaAl(OH)_4 + 3\,H_2\uparrow \quad (1)$$

The aluminium in contact with an excess NaOH solution produces 7.91 L of $H_2\uparrow$/kg of MIBA, concluding that there is ~5.5 g of Al/kg of MIBA.

Figure 1. Experimental setup for quantification of metallic aluminium in MIBA.

3.2. FRESH STATE PERFORMANCE

Table 2 shows the density values in the fresh state for all mixes made. The fresh density of MIBA mixes showed that all values vary between 1.459 g/cm^3 and 2.144 g/cm^3 for families M15/1.0 and M4/0.5, respectively. For FA mixes, the density values vary between 1.79 g/cm^3 and 2.49 g/cm^3 for families F4/2.0 and F6/0.5. The results show that, for mixes with MIBA, the effect of the Na_2O/binder does not have a relevant impact on the density, while when increasing the SiO_2/Na_2O ratio there is a slight decrease in density. This behaviour is due to the foaming effect presented by the sodium silicate when reacting with the metallic aluminium remaining in the precursor, which was also reported by Eliche Quesada et al. [21], therefore, weight decreases slightly. Figure 2 shows the values obtained for the workability of mortars made with MIBA. When increasing the SiO_2/Na_2O ratio, the mixes with Na_2O/binder of 4% and 6% presented a dry consistence. Those with a Na_2O/binder of 8% and with an increasing SiO_2/Na_2O ratio presented a high initial workability that degraded in a short period of time; therefore, it was not possible to determine this property for all mixes. These findings are in accordance with that reported by Puertas [22].

Table 2. Density in the fresh state of MIBA and FA mortars

Alkaline activator	**4/0**	**4/0.5**	**4/1.0**	**4/1.5**	**4/2.0**	**6/0**	**6/0.5**	**6/1.0**	**6/1.5**	**6/2.0**	**8/0**
FA (g/cm^3)	2.16	2.19	2.23	2.23	1.79	2.20	2.49	2.21	2.29	2.27	2.24
MIBA (g/cm^3)	2.00	2.14	1.93	1.94	1.85	1.99	2.11	1.94	1.85	1.74	1.93
Alkaline activator	**8/0.5**	**8/1.0**	**8/1.5**	**8/2.0**	**10/0**	**10/0.5**	**10/1.0**	**10/1.5**	**15/0**	**15/0.5**	**15/1.0**
FA (g/cm^3)	2.21	2.22	2.24	2.21	2.27	2.27	2.26	2.23	2.22	2.24	2.24
MIBA (g/cm^3)	1.99	1.73	1.46	1.69	1.96	1.62	1.61	1.70	1.92	1.47	1.46

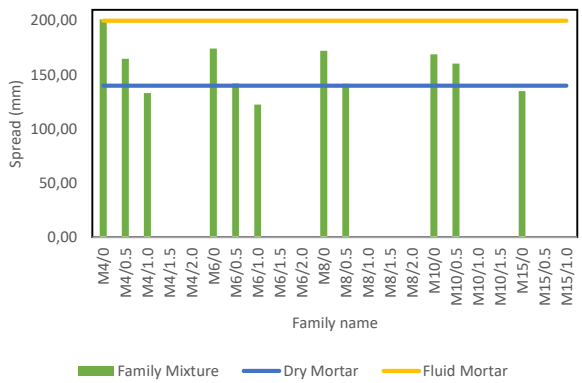


Figure 2. Workability of MIBA mortars.

The mixes with the highest workability are those made with the lowest percentages of Na_2O/binder and SiO_2/Na_2O (i.e. M4/0). Mixes M6/0, M8/0 and M10/0 were classified as plastic mortars according to the standard (140 mm < spread < 200 mm), M15/0 was classified as a dry mortar. The results show that increasing the concentration of Na_2O decreases the workability. It has been established that the viscosity of the alkaline solution increases with concentration of the solute [23][24]. Therefore, increasing the viscosity is likely to decrease the workability of mortars in the fresh state. FA mortars' workability presented a more stable behaviour since all mixes were classified as fluid.

3.3. HARDENED AAM STATE PERFORMANCE

Figure 3 shows the influence of Na_2O/binder and SiO_2/Na_2O on the mechanical properties evaluated at 28 days of curing for AAM with pre-treated MIBA. The contour graph presented in Figure 3a reveals that the compressive strength improves with the increase of the Na_2O/binder concentration. It reaches its maximum performance at 8% (compressive strength of 5.47MPa), after which significant deterioration was observed. A decline in performance was also observed with the addition of sodium silicate. The worst performance was presented by the M4/0 mix, with a 28-day compressive strength of 0.87 MPa. This result is consistent with what is reported in the literature. Although the dissolution of aluminosilicates and amorphous calcium occurs in high alkaline environments [25], it has been observed elsewhere [26] that an increase in the concentration of NaOH for AAM with MIBA as precursor, may lead to the deterioration of the material's mechanical performance. In this case, the excess of the sodium cation competes with aluminium and amorphous calcium, which results in a reduction in the formation of the reaction products. On the other hand, the negative effect when sodium silicate was added was due to the fact that a considerable part of the aluminium was not oxidized in the pre-treatment stage thereby reacting with the sodium silicate, which increased the foaming effect [21]. For this reason, the compressive strength of pre-treated MIBA decreased at both low and high SiO_2/Na_2O ratios.

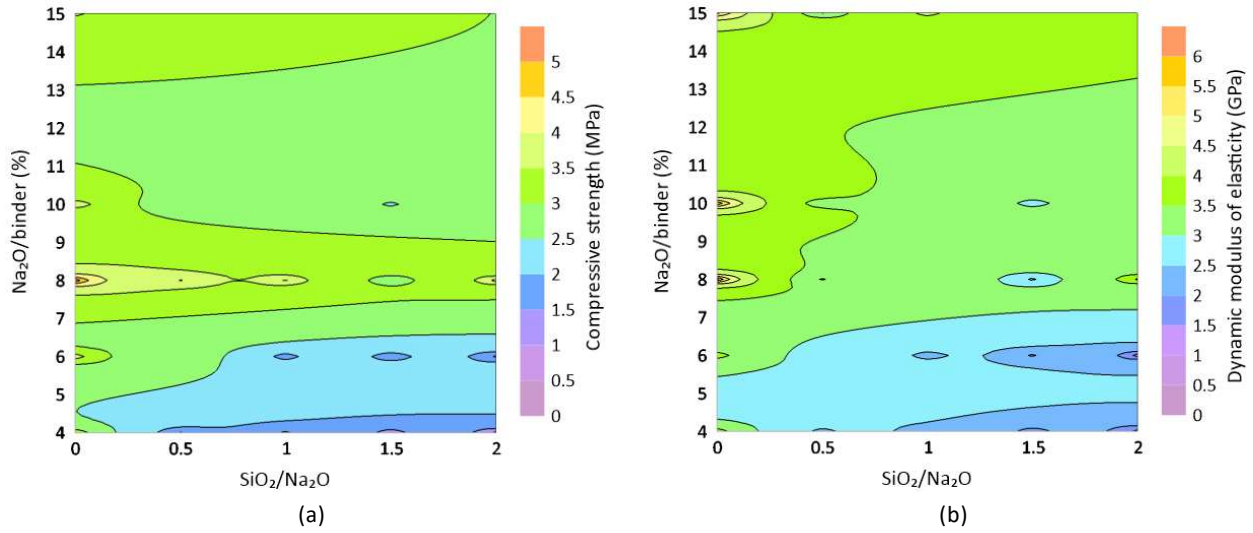


Figure 3. Hardened properties AAM with MIBA (a) compressive strength and (b) dynamic modulus of elasticity.

The contour graph presented in Figure 3b shows that the modulus of elasticity is influenced in a similar way as the compressive strength by the Na_2O/binder and SiO_2/Na_2O ratios. The M8/0 mix showed the best performance at 6.25 GPa, whereas the lowest values came from M4/0 at 1.25 GPa. The foaming effect triggered by the addition of sodium silicate caused high porosity of the hardened specimens. Not only does this contribute to a lower modulus of elasticity, but microcracks would likely be generated as a result of the higher porosity of the already unstable specimens thereby decreasing the property even further [27].

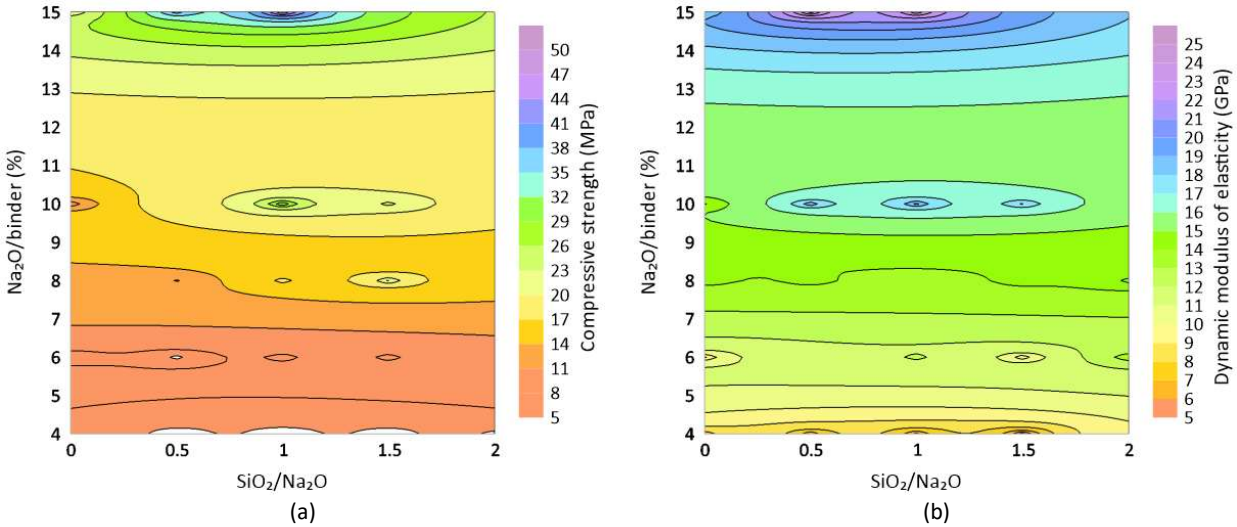
Regarding the pre-treated MIBA, the heat released in the reaction of dissolving NaOH in water was used as catalyst for the release of hydrogen (46.2 kJ/mol at a NaOH concentration of 1 mol/L), since the temperature stimulates the oxidation of metallic aluminium in alkaline OH^- environments [15]. Because of the treatment process, all MIBA mortars were dimensionally stable, showing only slight expansions in the specimens of mixes with higher Na_2O/binder and SiO_2/Na_2O ratios. This was due to the very high concentration of the activator (17.17 M for the M15/1.0 mix), resulting in a lower amount of water used in the pre-treatment. This was the equivalent of having a water/MIBA volumetric ratio of 0.20, thereby leaving some of the MIBA particles unwetted by the alkaline solution. Consequently, 24 hours later, a large quantity of metallic aluminium was still available to react with the sodium silicate when making the mixes. Figure 4 shows the aluminium oxidization process for the M8/0 and M15/1 mixes, where the appearance of the two processes confirms the lower efficacy of the treatment for the latter mix.

(a) (b)

Figure 4. Pre-treatment stage (a) M8/0 and (b) M15/1.0.

Figure 5 shows the influence of Na_2O/binder and SiO_2/Na_2O on the mechanical properties of activated FA after 28 days of curing. The contour graph presented in Figure 5a shows that the compressive strength improves with increasing Na_2O/binder, reaching its peak for a SiO_2/Na_2O ratio of 1; the F15/1.0 presented a 28-day compressive strength of 50.7 MPa, whereas the lowest value was that of F4/1.5, corresponding to 1.03 MPa. The alkali activation of FA generally leads to a maximum value for an optimum content [28], after which it begins to exhibit a decline in performance. However, this was not witnessed in the present research. The aforementioned results are consistent with those reported in the literature; a study [29] indicated that an increase in the concentration of NaOH leads to an improvement in the mechanical property, since high concentrations of the OH^- ion favour the dissolution of the amorphous aluminosilicates present in FA, resulting in the formation of reactions products. Additionally, the addition of sodium silicate influenced positively the compressive strength of all mixes, confirming that the addition of external sources of amorphous silicate improves the compressive strength [30].

The contour graph presented in Figure 5b shows that the modulus of elasticity is affected in a similar way to the compressive strength with varying Na_2O/binder and SiO_2/Na_2O ratios. The maximum value was gotten by F15/1.0 mix, corresponding to 24.8 GPa, and the lowest values came from F4/1.5 at 4.1 GPa. Nath & Sarker [31], in their research, evaluated the elasticity modulus of alkali-activated concrete mixes, using type F FA as precursor and a solution of NaOH and Na_2SiO_3 as alkaline activator. They reported 28-day values in the range of 21.6-23.2 GPa, which is comparable to those in this research for the same alkaline activator concentrations.



(a) (b)

Figure 5. Hardened properties AAM with FA (a) compressive strength and (b) dynamic modulus of elasticity

4. CONCLUSIONS

AAM made with pre-treated MIBA showed dimensional stability in the hardened state. This means that it is possible to release the greatest amount of hydrogen in the plastic state without requiring additional energy during the AAM production, by taking advantage of the temperature and time variables available in the process. Mortars with FA as precursor have more stable densities and workability than those made with MIBA. In the latter, when adding Na_2SiO_3, unexpected foaming was observed as it oxidized the remaining aluminium particles that had remained unreacted during the pre-treatment process.

M8/0 and F15/1.0 mixes presented the best mechanical results at 28 days with values of 5.57 MPa and 50.7 MPa respectively. This discrepancy was due to the low content of amorphous aluminosilicates and calcium present in the MIBA when compared to those available in the FA, which led to a compressive strength almost 10 times greater than that of MIBA. Therefore, it is recommended that further research is carried out on the combination of the two precursors in new mix designs, seeking the highest possible MIBA content within a minimum strength loss scenario.

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