ORIGINAL ARTICLE

Resistance to acid attack, abrasion and leaching behavior of alkali-activated mine waste binders

Pacheco-Torgal Fernando · Jalali Said

Abstract This paper report results of a research project on the development of alkali-activated binders using mine wastes. Abrasion and acid resistance of two ordinary Portland cement (OPC) strength class concrete mixtures (C20/25 and C30/37) and several mine waste (MW) mixtures were compared. This study indicates that MW binders possess higher acid and abrasion resistance than OPC based concrete mixtures. The leaching assessment of the MW binders shows it can be considered an inert material which indicates that it could be used as a building material.

Keywords Alkali-activated binders · Mine wastes · Abrasion · Acid resistance · Leaching performance

1 Introduction

The production of one tonne of Portland cement generates 0.55 tonnes of chemical CO_2 and requires an additional 0.39 tonnes of CO_2 in fuel emissions for baking and grinding, accounting for a total of 0.94 tonnes of CO_2 [1]. Other authors [2] report that the cement industry emitted in 2000, on average, 0.87 kg of CO_2 for every kg of cement produced. Therefore its no surprise that its use tends to become less competitive compared to alternative new binders like the ones obtained by alkali-activation. Alkali-activated binders generates 80% less carbon dioxide than Portland cement [3]. Weil et al. [4] mentioned that in comparison to Portland cement concrete the global warming potential (GWP) of alkali-activated concrete is 70% lower. Although research in this field has been published as "alkali-activated cement, or "alkaline cement" the term "geopolymer" is also for this technology. The alkali-activation involves a chemical reaction between various alumino–silicate oxides with silicates under highly alkaline conditions, yielding polymeric Si–O–Al–O bonds. That indicates that any Si–Al materials could be alkali-activated. Over the last years several authors have reported research in a large number of aspects related to alkali-activated based binders such as: dependence of the nature of source materials (alkali-activated binders synthesised from calcined sources show a higher compressive strength than from raw materials) [5–7], immobilization of toxic metals [8–10], reaction mechanisms and hydration products [11–14], the role of calcium in alkali-activation [15–19], manufacture operations [20, 21], and even the development of lightweight building materials [22]. According to some authors [23] durability is the most important issue on determining the

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success of these new materials (Fig. 1). Although several studies have been published about the alkali-activation of industrial wastes [24–27], very few concern the use of mine wastes. Previous studies concerning the alkali-activation of mine wastes together with a small percentage of calcium hydroxide suggests that a new binder with extremely high early age strength can be produced [28–31]. Therefore, the objective of the present work is to investigate durability and the leaching performance of the new binder.

2 Experimental program

2.1 Materials

2.1.1 MM binders

The chemical composition and specific surface of the mine waste mud before calcination are shown in

Table 1. Mine waste mud used in this study was subjected to a thermal treatment at 950°C during 2 h. Mineralogical composition and thermal conditions were described elsewhere [32].

The XRD patterns indicated that mine waste mud consists mainly of muscovite and quartz which were identified by their characteristic patterns as follows: muscovite (Powder Diffraction File card 46-1409) and quartz (Powder Diffraction File Card 46-1045). For calcination at 950°C during 2 h XRD patterns indicated that dehydroxylation did not result in a complete collapse of muscovite structure. Calcination leads to formation of an amorphous phase, causing an increase of the diffuse scattering of the XRD patterns. This occurs predominantly in the calcination interval from 850 to 950°C, with a thermal behaviour similar to other phyllosilicate clay minerals [33]. The main muscovite peak ($2\theta = 8.8°$) persisted even after the sample had been heated at 950°C although it decreased considerably. Peak area measurements

Fig. 1 Diagram showing some of the interrelationships between technical and scientific aspects of geopolymer binder technology
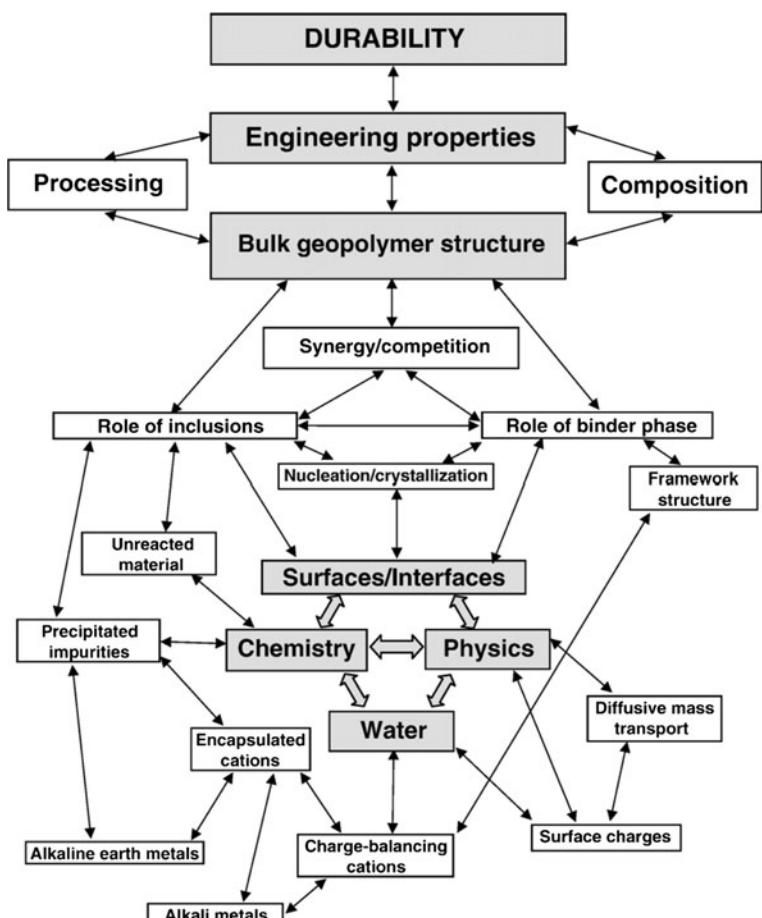
Table 1 Chemical composition and specific surface

Constituents (%)	MW
SiO_2	53.48
Al_2O_3	16.66
Fe_2O_3	12.33
K_2O	7.65
Na_2O	0.62
MgO	1.27
SO_4	3.10
TiO_2	1.39
As	1.28
Other minor oxides	2.22
Blaine fineness (m^2/kg)	357

Table 2 Compressive strength of alkali-activated MW

Curing (days)	Compressive strength (MPa)	
	Raw mine waste	Waste calcined at 950°C during 2 h
7	8.4 (1.2)	28.4 (1.8)
14	9.3 (4.3)	37.0 (1.1)
28	11.2 (1.8)	39.6 (1.8)

Bracketed values are the standard deviation

revealed that about 12% of muscovite survived calcination at 950°C. Molecular changes during dehydroxylation were also examined with infrared emission spectroscopy, confirming decrease in the absorption peaks at 3600–3700 (OH stretch). However the main muscovite peak did not disappear totally indicating only a partial transformation.

Compressive strength tests were used to assess the reactivity of calcined mine waste mud. Compressive strength data was obtained using $50 \times 50 \times 50$ mm^3 cubic specimens. The fresh mortar were cast and allowed to set at room temperature for 24 h before being removed from the moulds and kept at room temperature until tested in compression. Compressive strength for each mortar mixture was obtained from an average of 3 cubic specimens.

The mass ratio of fine aggregate: waste mud: activator was 1:1:1. The fine aggregate used was a crushed schist sand from the same mine with a specific gravity of 2.7, 24 h water absorption of 0.9%, and a fineness modulus of 2.8 mm. Waste and calcium hydroxide were mixed with a mass ratio of 5:1. An activator with sodium hydroxide (12 M) and sodium silicate solution ($Na_2O = 8.6\%$, $SiO_2 = 27.8\%$, $Al_2O_3 = 0.4\%$ and water = 63.2%) was used with a mass ratio of 1:2.5. Distilled water was used to dissolve the sodium hydroxide flakes to avoid the effect of unknown contaminants in the mixing water. To produce a workable mix extra water was been added. The mass ratio of water/dry solid content was 1.8%.

Compressive strength data related to alkali-activated mortars made with, respectively, raw waste

mud and calcined waste mud showed an increase of more than 300% (Table 2) justifying the thermal treatment. Besides, mortars made with raw waste mud weren't very stable from a structural point of view, because they easily showed a lot of efflorescences in the surface of the specimens. That is due to non reacted sodium ions which will react with atmospheric carbon dioxide leading to the formation of sodium carbonate crystals.

Calcination of mine waste mud in a static furnace, as it happens in this study may be expensive. Nevertheless, some authors have already developed flash calciner industrial 800 kg/h production units, capable of reducing calcination time to a few seconds and with the additional advantage of no further grinding operations [34, 35]. This will cut down the cost of down mine waste mud thermal treatment.

Figure 2 and Table 3 show the gradation and the physical characteristics of aggregates. Limestone and granitic coarse aggregates have the same Faury maximum dimension (9.52 mm) and similar fineness modulus respectively 5.5 and 5.2 mm. Schist sand has a lower maximum dimension (2.78 mm) and a fineness modulus of 2.38 mm.

2.1.2 OPC concrete

Using the Faury concrete mix design method [36], a C20/25 and a C30/37 strength class OPC concrete mixtures were designed. The concrete mixes and their main properties are described in Table 4. Concrete specimens were cast into cubic moulds with 150 mm high in order to be test in compression after 28 days curing, to confirm the concrete strength class. The concrete specimens for abrasion and acid resistance tests were cured immersed in water during 3 months at 20°C. This curing period provides an almost

Fig. 2 Aggregates gradation
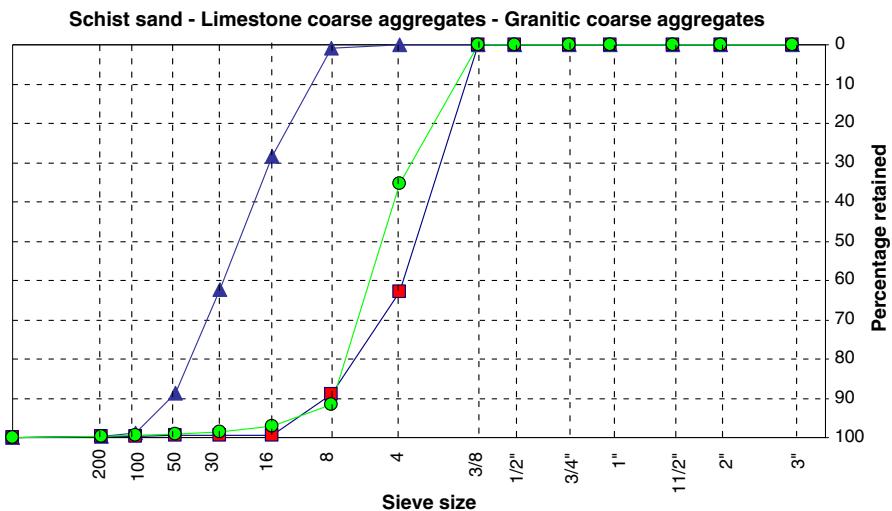
Table 3 Dimensional and physical characteristics of aggregates

Fineness modulus (mm)	2.78	5.5	5.2
Max. dimension (mm)	2.38	9.52	9.52
Water absorption (%)	0.9	0.8	0.4
Density (kg/m^3)	2703	2659	2689

Table 4 Mix proportions and main properties of the OPC concrete binders

Components	C20/25	C30/37
Cement II 32.5 (kg/m^3)	394	504
Fine river sand (kg/m^3)	632	417
Coarse aggregate (kg/m^3)	1032 (limestone)	1154 (granite)
W/C ratio	0.55	0.43
fc$_{28d}$ [a] (MPa)	25.6	37.8

[a] Average value of three specimens ($150 \times 150 \times 150mm^3$)

complete concrete hydration as old concretes in field practice and has been used by other authors [37]. After that time they were cut with an electric masonry saw to obtain $50 \times 50 \times 50 \ mm^3$ cubic specimens.

2.1.3 MW mix proportioning and specimen preparation

Table 5 shows the composition of MM mixtures. MW paste was a mixture of waste mud, calcium hydroxide, alkaline silicate solution and water. Calcium

hydroxide was used with a percentage substitution of 10%, because it was found that percentage lead to the highest compressive strengths [29]. An activator with sodium hydroxide (24 M) and sodium silicate solution ($Na_2O = 8.6\%$, $SiO_2 = 27.8\%$, $Al_2O_3 = 0.4\%$ and water = 63.2%) was used with a mass ratio of 1:2.5. Previous investigations showed that this ratio lead to the highest compressive strength results in alkali-activated mine waste mud mortars [29]. Distilled water was used to dissolve the sodium hydroxide flakes to avoid the effect of unknown contaminants in the mixing water. The alkaline activator was prepared prior to use. MW mortars were a mixture of aggregates, waste mud, calcium hydroxide, alkaline silicate solution and water. The sand, mine waste mud and calcium hydroxide were dry mixed before added to the activator. To produce a workable mix extra water has been added. Compressive strength data was obtained using $50 \times 50 \times 50 \ mm^3$ cubic specimens according to ASTM C109. The fresh mortar were cast and allowed to set at room temperature for 24 h before being removed from the moulds and kept at room temperature (20°C) until tested in compression. MW binders using schist (SC) fine aggregates with an aggregate/binder ratio of 1.5 was named SC-AG/B 1.5. Similarly when limestone (LS) or granite (GR) aggregates were used was named respectively LS-AG/B 1.5 and GR-AG/B 1.5. MW mixtures made with 2% superplasticiser by mass of binder lime and mine waste mud were named respectively SC/SP and LS/SP.

Table 5 MW mixtures

Constituent	Paste	Mortar				
		AG/B = 0.5	AG/B = 1.5			
		LS	GR	LS	SC-SP	LS-SP
Mine waste mud	90%					
Calcium hydroxide	10%					
Aggregate/binder	–	0.5	1.5			
Solid binder/activator	1					
Sodium hydroxide concentration	24 M					
Waterglass/sodium hydroxide	2.5					
Extra water/dry solid	3.6%		7%			
Superplasticizer/binder	–				2%	

2.2 Test procedures

2.2.1 Abrasion resistance

The abrasion resistance was evaluated using the Los Angeles abrasion apparatus, according to specification EN1097-2:2007. This test consists of a metal cylinder, where eight $50 \times 50 \times 50$ mm^3 cubic specimens with 56 days curing have been placed together with eight steel spheres. The cylinder is then submitted to 1000 full rotations, being that after every 100 full rotations the specimens are weighed to detect the weight change.

2.2.2 Acid resistance

The resistance to acid attack was tested by immersion of the MW and OPC concrete $50 \times 50 \times 50$ mm^3 specimens with 56 days curing in 5% of sulphuric, hydrochloric and nitric acid solutions during 28 days. To keep a constant pH acid solutions were replace after 14 days. After 28 days the specimens were oven-dry to achieve constant weight and detachable particles were removed by hand with a metallic plane spatula. The acid resistance was assessed by the differences in weight of dry specimens before and after acid attack.

2.2.3 Environmental performance

Leaching tests have been carried out according to DIN 38414-S4 [38]. In this test a sample of a MW mixture was placed in a bottle with water. This bottle was then placed in a rotating device during 24 h. After the rotation step the solid part is separated from a filtration process and the chemical composition of the liquid part was evaluated.

2.3 Results and discussion

2.3.1 Chemical composition and specific surface

The figures in Table 1 clearly show that mine waste mud consists essentially of silica and alumina, contaminated with arsenic and sulphur and with a high content of iron and potassium oxide. Although it's known that iron oxide contributes to the strength of Portland cement it is still an open question if iron also contributes to alkali-activation [23]. The SiO$_2$/Al$_2$O$_3$ atomic ratio of mine waste mud is 5.5. This is higher than the one suggested by Davidovits by about 2 for making cement and concrete. However, the final SiO$_2$/Al$_2$O$_3$ atomic ratio in the hardened binder depends mainly on the reactivity of Al–Si because not all the silica and alumina are reactive. So even despite the fact that Al and Si have synchro-dissolution behaviour in alkaline solution. Meaning they dissolve from the mineral in some linked form. One can not expect the same Si/Al ratio in the final hydration product as the one present in the original precursor material. Indeed most of the Al–Si materials cannot even supply sufficient Si in alkaline solution to start the alkali-activation process. This explains why they need extra silica provided in solution by waterglass, which influences the Si/Al ratio of the hardened binder. Since no tests were

performed to evaluate the reactivity of silica and alumina from the mine waste it is not possible to discuss this issue. Mine waste Blaine fineness is low but is in the range of the most used slag based alkaline binders [7]. Despite the fact that increased fineness led to increase reactivity that can be overcome by using highly alkaline-silicate activators.

2.3.2 Abrasion resistance

MW binder specimens show a low level of weight loss while in OPC specimens a severe weight loss was observed (Fig. 3). This behavior is due to the fact that OPC specimens possess much lower compressive strength (25 and 37 MPa) than MW based specimens.

Fig. 3 Abrasion resistance with the Los Angeles test for OPC and MW binders using 50 mm cubic specimens
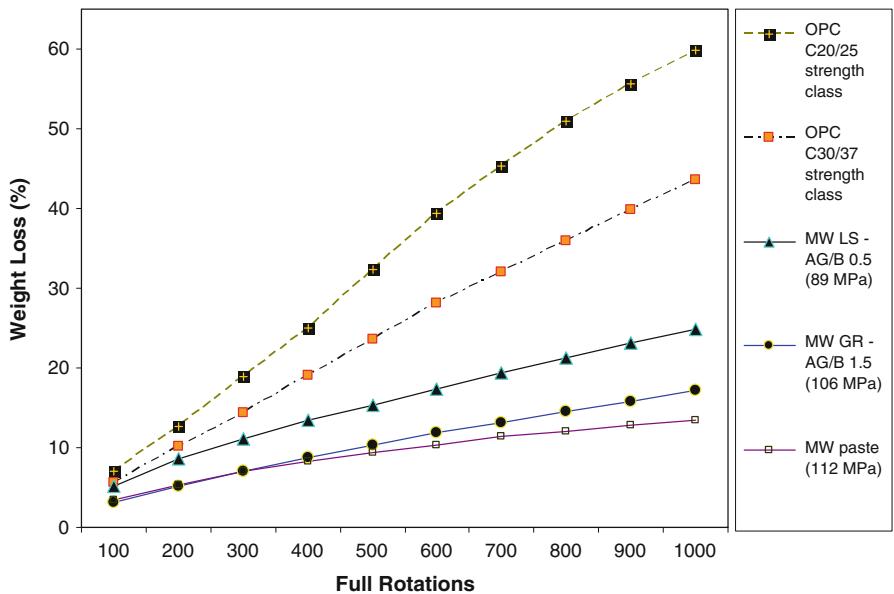

Fig. 4 Weight loss after acid attack
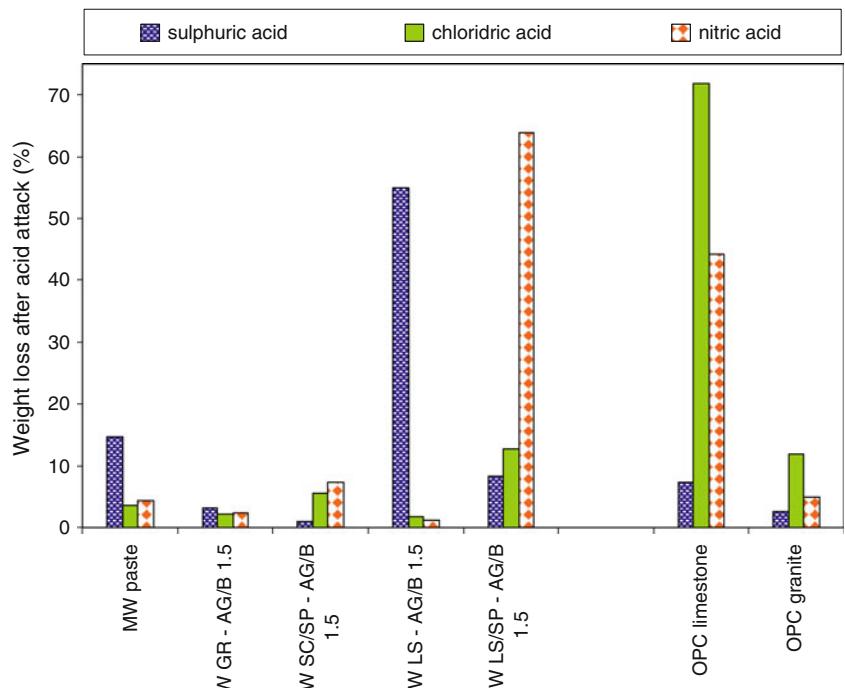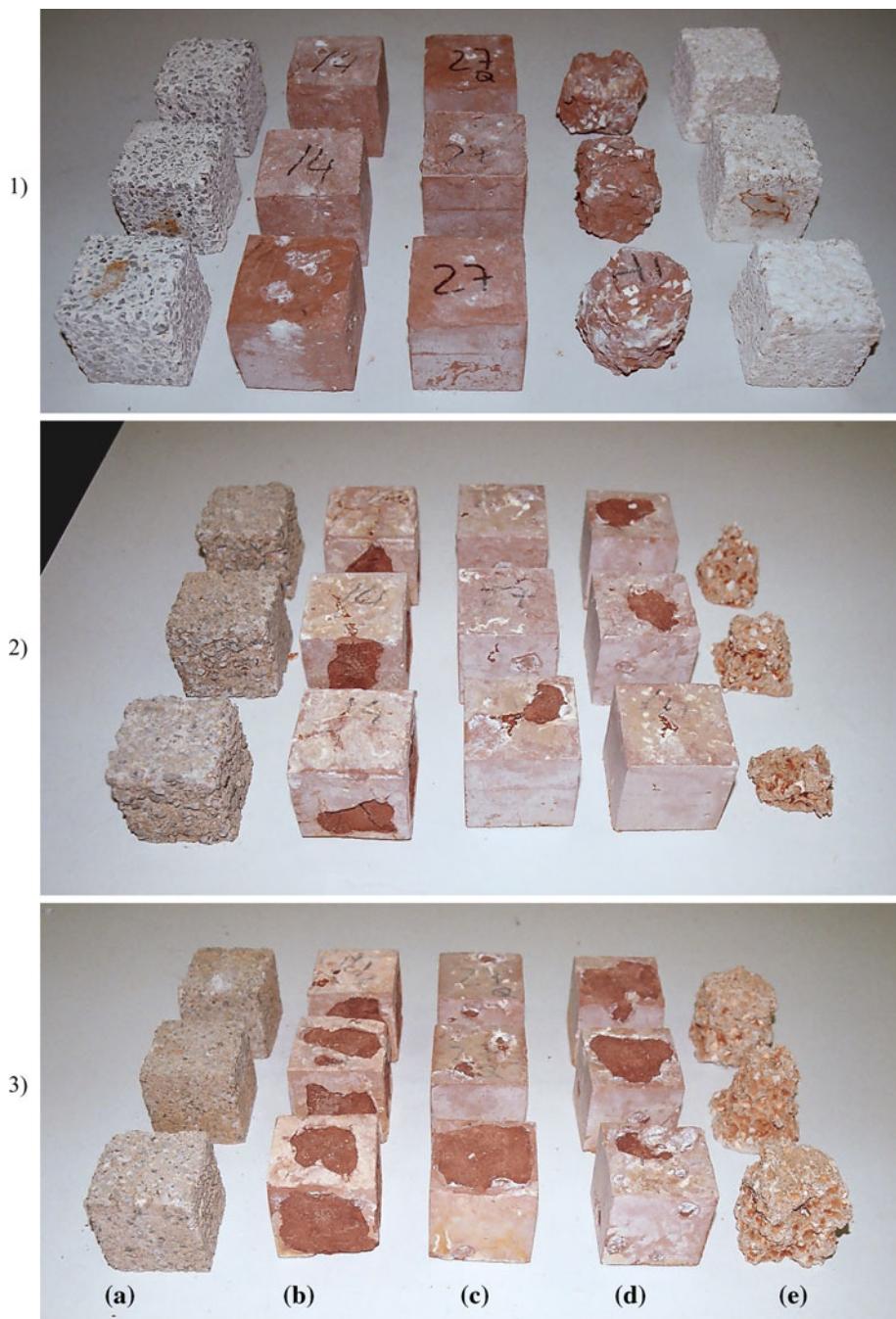
Fig. 5 Specimens exposed to acid attack: (**1**) sulphuric acid; (**2**) hydrochloríc acid; (**3**) nitric acid (a) OPC concrete with granitic aggregates; (b) MW paste; (c) GR–MW with granitic aggregates; (d) CL–MW with limestone aggregates; (e) OPC concrete with limestone aggregates

Hu et al. [39] also confirm the high abrasion resistance of alkali-activated binders. For MW binders the higher abrasion resistance was achieved in paste specimens. This result is partially related to the fact that MW paste had the highest compressive strength (112 MPa). Another reason as to do with the fact that contrary to OPC binders, MW mortars possess very high flexural strength [28]. Since OPC pastes traditionally possess

Fig. 6 Sulphuric acid attack. Specimens before detachable particles have been removed

lower strength than OPC concrete, this means that abrasion resistance seems to be more influenced by the compressive strength than for the aggregates used in the mix. Other authors reported that for OPC binders, abrasion resistance is influenced by aggregate resistance [40].

2.3.3 Acid resistance

Acid resistance of OPC and MW binders as weight loss are shown in Fig. 4. Results are dependent not only from the type of acid but also from the type of aggregate. For MW binders the mixtures GR e SC/VS have low weight loss results for the several acids. The mixtures LS e LS/VS their weight loss behavior is influenced by the type of acid. Specimens immersed in hydrochloric acid have low weight loss performance independent of aggregate type. As for specimens submitted to nitric and sulphuric acid attack they are influenced by the presence of limestone aggregates.

Figures 5(1) and 6 show the behavior of mixture LS after sulphuric acid attack, where it is possible to see that a reaction with increase volume had take place. This kind of reaction is typical of sulphuric acid attack [41, 42] because sulfate ions reacts generating voluminous reaction products.

With the exception of the mixture LS, all the other MW specimens immersed in sulphuric acid had low weight losses. As for the OPC specimens their weight loss is due to the reaction between calcium hydroxide present at the surface of the specimens and the acid, which is clear in Fig. 5 from exposed aggregates.

In MW binders weight loss is due to the detachment of little particles from the leaching of unreacted sodium in the matrix, leading to an increase in porosity and allowing the acid solution to enter inside the specimen and removing calcium compounds. For the mixture SC/VS, the weight loss is even lower which is due to the fact that the acid solution is unable to go inside the specimen because of their low porosity (<4%) [30].

The weight loss results of MW under hydrochloric acid attack are lower than the ones reported by others [20]. That is probably due to the lower surface area of the specimens used by that author ($40 \times 40 \times 40$ mm^3).

Hydrochloric acid reacts with calcium compounds leading to the formation of calcium chloride which has a extremely high solubility (46.1%) [42]. This helps to explain the behavior of the exposed limestone aggregates in the sawn OPC concrete specimens, and also the highest weight loss in OPC concrete specimens.

For the remaining MW specimens it can be said that the low degree of acid attack is related to the unreacted sodium taking into account that the highest weight loss occurs to the mixture LS/VS. A similar behavior takes place for the attack with nitric acid, since that acid reacts with calcium compounds forming calcium nitrate which has a solubility (56%) even higher than the one of calcium chloride, explaining the destruction of limestone aggregates in OPC specimens. In LS/VS MW specimens, a voluminous reaction had taken place (Figures 7(3) and 8).

Being the mixture with the higher unreacted sodium in the matrix, probably the leaching of free

Fig. 7 MW binder specimens made with superplasticiser exposed to acid attack (a) specimens with schist aggregates, (b) specimens with limestone aggregates): (**1**) sulphuric acid; (**2**) hydrochloríc acid; (**3**) nitric acid
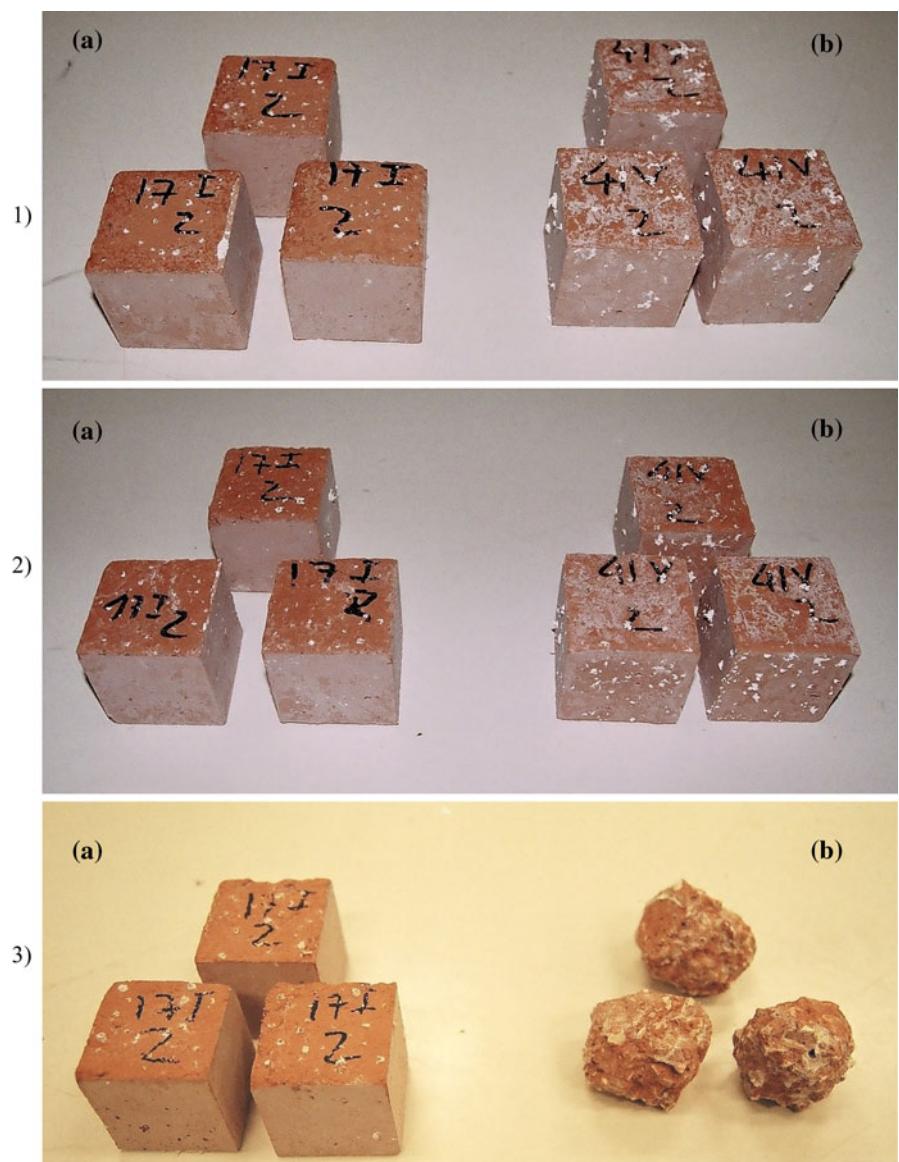

Fig. 8 MW binder specimens made with a superplasticiser submitted to nitric acid before detachable particles have been removed

Table 6 Contaminant concentration in the wastewater by leaching process standard DIN 38414-S4

Contaminant	Test results (mg/l)	Limits (DIN 38414-S4)	
		Max.	Min.
Zinc	0.011	2	5
Arsenic	<0.002	0.1	0.5
Lead	0.197	0.5	1.0
Copper	0.062	2	5
Manganese	0.019	–	–
Iron	0.203	–	–
Potassium	123.75	–	–
Sodium	3792.5	–	–
Magnesium	0.163	–	–
Sulphates	<0.003	500	1500

sodium had certainly contribute to increase the porosity allowing the ingress of acid solution inside the specimen where it reacted with limestone aggregates. Weight loss results for mine waste binders are not too dependent on the type of acid. Other authors report different results for alkali-activated binders based on fly ash and blast furnace slag [43–45].

MW based binders have low weight loss under acid attack, this behavior may be due to the low water absorption of these binders and also to their low content in calcium hydroxide.

2.3.4 Leaching performance

Leaching results show that all chemical parameters are below the limits established by the standard and can be considered an inert material (Table 6). The results for the sulphates indicate that MW mixtures have potential to immobilize this contaminant. The same seem to happen about arsenic. As to the limits for water contamination set by the Portuguese Decree 236/98, it can be stated that although some chemical parameters are above the limits for drinkable water, all limits are met concerning irrigation purposes.

2.3.5 Conclusions

The following conclusions can be drawn from this study:

Abrasion resistance for MW binders is higher than the one presented by current OPC binders although that behaviour is due to a higher strength. MW

binders without limestone aggregates show good acid resistance higher than the one presented by OPC concrete. One believes that performance is due to the fact that MW binders have very low water absorption (<4%) and also low content in calcium which generates less soluble compounds. Acid resistance of MW binders is influenced by the amount of the unreacted sodium in the matrix. Leaching tests show that the new binder is considered an inert material which indicates that it could be used as a building material.

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