Design of alkali-activated materials for a modular green wall and green roof system

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Abstract. This study presents the work developed with alkali activated mixtures to be used as component of a new modular green wall and green roof system (GEOGREEN). The aim is to find the most appropriate composition of alkali-activated mixture to maximize water absorption and porosity and also find a good mechanical strength with reduced density. Alkali-activated mixtures were produced using two precursors, mine waste mud from Panasqueira mine (W) and ground waste glass (G) and two alkaline activators, sodium silicate (SS) and sodium hydroxide (SH). A ventilated oven was used to speed up the curing process. Variables as percentage substitution of W per G, molar concentration of SH, cure length and temperature, were tested to identify the reference mixture. After these tests different percentages of aggregates as sand (S), expanded cork granules (C) and expanded clay (A) were added to reference mixture (REF). Results indicate that S25 obtained the maximum compressive strength of 35 MPa after 7 curing days. However, about 30% compressive strength loss is observed after immersion of this mixture in water for 24h. Capillary absorption coefficient can reach to 4.77 kg/m².h⁰.⁵ with C25 and to 4.11 kg/m².h⁰.⁵ with S25. Also C50 enables a 20% density reduction compared to REF.

1 Introduction

1.1 Background

The construction sector is one of the main waste producer in European Union (EU) representing 35% of total produced waste. Mine and quarrying comes just after representing around 28% of total waste production [1]. Therefore it is important to find alternative applications for construction and mine waste, promoting their recycling and reuse.

Panasqueira mine in Portugal generates several hundred tons of coarse aggregate and waste mud every year [2]. Recent research indicates that mine waste mud from Panasqueira mine is rich in silica and alumina and show good reactivity with alkaline activators like sodium silicate and sodium hydroxide allowing to produce alkali-activated binders [3].

Studies indicate that alkali-activation can be improved by mixing Panasqueira mine waste mud with other sources of amorphous silica [4, 5, 6].

Besides, lightweight aggregates as expanded clay, expanded polystyrene (EPS) or granulated cork are also being tested in several types of cementitious concrete mixtures [7, 8].

1.2 Modular system design

Environmental conditions of urban areas are becoming deteriorated, due to crescent pollution, densification and lack of green areas. In this context, new solutions of green walls and green roofs are being developed to create vegetated surfaces in buildings [9] without any land occupation [10]. In fact they can be used as a potential strategy of urban rehabilitation [11, 12], having several environmental, social and economic benefits [13]. Recent studies focus on green walls and green roofs ability to improve the urban environment by reducing the heat island effect [14], decrease flood risk and air pollution [15, 16], encouraging the fruition of urban areas [17], increase biodiversity [18, 19, 20], and improve quality of life [17]. In a building scale green walls and green roofs can also reduce energy demand for heating and cooling [21, 22, 23] and improve buildings thermal [24] and acoustic envelope [25, 26, 27].

A new modular system (GEOGREEN) for vegetated surfaces (Fig. 1-3) was designed with the aim of creating more sustainable green roofs and green walls [28, 29]. Based on the reuse of industrial waste, alkali-activated base plate is part of this modular system with the purpose of absorbing water and slowly release it to the plants, minimizing its irrigation needs.
1. Goals

This study aims to identify the composition of alkali-activated mixtures which have the best characteristics to be used as a component of GEOGREEN. The goal is to develop an alkali activated binder that has good mechanical strength and water absorption, porosity, and lower density than conventional alkali-activated binders.

2. Materials and Methods

Several materials make part of the alkali-activated mixtures. As precursors were used waste mud (W) from Panasqueira tungsten mines and milled waste glass (G).

As activators were used sodium silicate (SS) and sodium hydroxide (SH).

Aggregates like, sand (S), expanded cork granules (C) and expanded clay (A) were also added to the mixture.

Waste mud was obtained from Panasqueira tungsten mine at Portugal. It comes in the form of a powder. Glass bottles were obtained at the local municipality. Glass bottles are cleaned and labels removed. Glass is broken in small particles, dried and sieved with a 125 µm sieve. Sand is sieved to obtain the desired particles size.

2.1. Materials characterization

Mine waste mud from Panasqueira mines is rich in alumina-silicates and has very good reactivity with alkaline activators like sodium silicate and sodium hydroxide. From the chemical analysis it is evident that mine waste mud is rich in silicon dioxide (52%) and aluminium oxide (11%). The amorphous part of the silica and alumina present in the mud are fundamental for the alkaline activation.

Waste glass has mainly amorphous silica (74%) in its composition and smaller amounts of oxides of calcium and sodium. Waste glass is milled and sieved under 125µm to be incorporated into the mixture. It is used in the mixture to increase the precursor’s percentage of amorphous materials.

Table 1. Oxide chemical composition of mine waste mud and waste glass.

| Constituents (%) | Mine waste mud | Waste glass |
|-----------------|----------------|-------------|
| SiO₂            | 51.72          | 73.93       |
| Al₂O₃           | 10.53          | -           |
| FeO₂            | 12.93          | 0.40        |
| SO₃             | 10.40          | -           |
| K₂O             | 2.68           | 0.69        |
| CaO             | 0.87           | 12.83       |

As activators were used sodium silicate (SS) D40 and sodium hydroxide with 10-12M molar concentration. In these mixtures was established a ratio of sodium silicate to sodium hydroxide of 4 and a ratio of precursor to sodium silicate of 4.5.

Different types of aggregates like 1-2mm river sand (S), 4-8mm expanded cork granules (C) and 1-2mm expanded clay (A) were used in the mixtures.
2.2 Synthesis of Samples

Precursors were mixed together in a dry state until obtaining a uniform mixture. Activators like sodium silicate and sodium hydroxide solution were mixed together in a mixer for 10 minutes at low speed. After mixing the precursor mixture is slowly added to the alkali-activator solution. After obtaining a homogeneous mixture, water was slowly introduced. Finally, aggregates were added to the mixture.

Alternatively, aggregates surface can be sprinkled first before being inserted into the mixture. The resulting paste was mixed at a lower rate until uniformity was obtained and then placed into the mould. The paste was manually compressed to ensure full mould filling. The mould was covered with plastic to avoid a rapid moisture loss in the beginning of the curing process. Samples were demoulded and left uncovered after 48 hours.

2.3 Compressive Strength

Cubic specimens with 40x40x40mm size were prepared for compressive strength testing. Compressive strength was tested using an ELE 3000 kN compression equipment in accordance with EN 196-1 [30], with some adaptations. The recorded compressive strength value was the average of values obtained from six specimens.

2.4 Water immersion

Specimens with 40x40x40mm size of each mixture were also produced to identify if mixtures mechanical strength is affected by water. Six specimens of each mixture were tested after a curing period of 7 days. All samples were allowed to cool after curing and then inserted in a tin with approximately 1 litre of water at room temperature for 24h. This was considered the time necessary to get specimens saturated. After each immersion period, specimens were removed from water, dried until constant mass acquired and submitted to a compressive strength test in accordance with EN196-1 [30], with some adaptations.

2.5 Capillarity Water Absorption

Capillarity is defined as the penetration of a liquid into the material by effect of surface tension generated at the water-air-wall pores interface. Water penetration depends normally of the quantity, size, shape and pores connectivity in the material structure. Test samples were prepared according to the Portuguese Specification E393-1993 – Concretes, with some adaptations. Five samples with 4x4x14cm of each mixture were prepared to perform this test. Samples were dried at a constant temperature until constant mass is acquired, then allowed to cool at room temperature. Test samples were inserted into a tray with 5 to 10 mm of water above the underside of the samples. Measurements were made at the end of 3h, 6h, 24h and 72h from the moment samples were placed in contact with water.

3 Results and discussion

Prior to adding aggregates to the mixture, some characterization tests were performed to identify the reference mixture. In this context different molar concentrations, waste glass percentages and curing times were tested.

3.1 Influence of glass substitution on mixtures compressive strength

This analysis is based on a replacement of a percentage of 20% to 30% of mine waste mud (W) by milled waste glass (G). Each result reflects the average value from six specimens per each G replacement value and age. These mixtures were tested after 7, 14 and 28 days curing at a constant curing temperature of 60ºC.

In Fig. 4 it can be observed that for 7 curing days the compressive strength increases with the addition of G content in the mixture. However, the increase of strength is not so marked in longer curing periods. W70G30 specimen shows even a loss of strength after a 14 curing days. In fact mixture W80G20, with a lower G content, was the mixture that presented a greater increase of strength over time. Therefore this mixture was used forward as reference mixture.

![Compressive strength of alkali-activated mixtures with 20% to 30% of W replacement by G.](image)

3.2 Influence of sodium hydroxide molar concentration in compressive strength

Considering that mixture W80G20 demonstrated a better behaviour it was used as reference for the following studies. In this case different molar concentrations of sodium hydroxide were tested to identify its compressive strength with different curing periods (Fig.5). Results indicate that mixtures with 12M present slightly higher
compressive strength in shorter curing periods of 7 days. However the strength results are very similar for 10M and 12M concentrations.

Fig 5. Compressive strength of alkali-activated mixtures with different SH molar concentrations.

3.3 Influence of curing temperature in compressive strength

The influence of curing temperature was also identified (Fig. 6). Curing at 60ºC presents a continue increase of compressive strength along the time. The same doesn’t happen if the cure is increased from 60ºC to 80ºC. Specimens cured at 80ºC show the highest compressive strength, 44 MPa, when cured for 7 days. However there is a 26% loss of strength when curing is increased from 7 to 14 curing days. If the cure is prolonged from 14 to 28 days the tendency of both mixtures is to increase their strength.

Ideally curing time and temperature should be minimized when possible to reduce the embodied energy of alkali-activated mixture and consequently the CO₂ emissions regarding its production. The results indicate that a good compressive strength is reached at 7 days with a curing temperature of 60ºC.

Fig 6. Compressive strength of alkali-activated mixtures with different curing temperatures.

Considering these variables the mixture W80G20 with 20% of G content, a molar concentration of 12M, and a curing period of 7 days in a ventilated oven at 60ºC was the one that presented a greater increase of compressive strength over time. Therefore this mixture was used forward as reference mixture (REF) to add different percentages of aggregates.

3.4 Aggregates influence on compressive strength

Several alkali-activated samples were prepared adding to REF 25% to 50% of precursor’s mass of the following aggregates: 4 to 8mm expanded cork granules (C), 1 to 2mm river sand (S), 1 to 2mm expanded clay (A). These mixtures were tested to compressive strength with 7 days of curing at 60ºC (Fig.7).

Mixture S25 was the only one that revealed better compressive strength than REF. This may results from the fact that sand has silica in its composition helping to increase the amount of amorphous material to react in the mixture. However sand was the aggregate that revealed greater loss of resistance when the percentage is increased to 50%. Mixtures with 25C and 50C were the ones with lower compressive strength, 17 and 13 MPa, respectively. These results represent a 45-58% loss of strength compared to REF. However the results are satisfactory for all mixtures given that it is desired to develop a cladding material.

Fig 7. Compressive strength of mixtures with aggregates.

3.5 Effect of water immersion on mixtures compressive strength

Six specimens of the same mixtures prepared to test the influence of aggregates on compressive strength were used for this test. A comparative analysis is shown in Fig. 7. The results indicate that all mixtures tend to lose compressive strength after water immersion for 24h. Only S50 presents a 12% increase of compressive strength.
compared to REF (W80G20). This increase may result from the presence of amorphous silica in the sand which also reacts. It must be pointed out that cork mixtures were the only ones in which the process of water immersion for 24h didn’t affect their compressive strength.

3.6 Influence of aggregates in water absorption by capillarity

Water absorption of these specimens was compared with the reference mixture REF with no aggregates (Fig. 8). Mixture C25 is the one that shows higher water absorption along the time. However S25 shows higher results in the first hours. Regarding the increase of aggregates percentage from 25% to 50% it proves to be detrimental, leading to a significant reduction in capillary absorption capacity in all mixtures. Although expanded clay is a porous material, the blends with this material have been found to have the lowest capillary absorption capacity of all mixtures with aggregates.

3.7 Relation between mixtures density and water absorption

By establishing the relation between mixtures bulk density and water absorption coefficient (Fig. 9), we can identify which are the most advantageous for the design of GEOGREEN modules.

All aggregates contribute to increase the mixture water absorption coefficient (Aw). However, the ones with better results are C25 with 4,77 kg/m².h⁰.⁵ and S25 with 4,11 kg/m².h⁰.⁵.

The results indicate that all mixtures containing sand, S25 and S50, have higher bulk density than REF. The mixture with expanded clay, A25 and A50, are the ones with lower water absorption.

C50 is the mixture with lowest bulk density, 1,55 g/cm³ and C25 is the mixture with highest water absorption coefficient.

3 Conclusions

Mixture S25 can reach a maximum compressive strength of almost 35 MPa, after 7 curing days at a constant temperature of 60°C in a ventilated oven. However a 30% compressive strength loss of is observed after immersion in water for 24h.

Capillarity absorption coefficient can reach between 1,89 kg/m². h⁰ for A50 to 4,77 kg/m². h⁰ for C25. Density can be reduced with the introduction of expanded cork granules as aggregates in the mixture. C50 enables a final density of 1,55 g/cm³, which represents a 20% reduction from REF.

Therefore expanded cork granules are considered the most suitable aggregate to add to these alkali-activated mixtures.

Results indicate that alkali-activated mixture C25 that has the most appropriate combination of properties to be used in GEOGREEN modules. It obtained one of the lowest compressive strength with 17MPa. However, it obtained a water absorption coefficient of 4,77 kg/m².h⁰.⁵ and a density of 1,85 g/cm³.

Further tests could be prepared to identify if the percentage of cork granules could be slightly increased.

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