

Structural Properties of Phosphate-Washing Waste Based Geopolymeric Mortars

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Abstract

The phosphate ore extraction activities often generate serious environmental problems, particularly those engendered by the phosphate washing waste. The waste was characterized by X ray fluorescence. The powder was calcined at 700 °C. The calcined and uncalcined samples were then investigated by means of X-ray powder diffraction (XRD). The calcined PWW was activated with sodium hydroxide and sodium silicate to produce geopolymeric materials. The reached results prove to reveal that the present phases in the powder appeared to have a remarkable effect on the result of compressive strength.

Keywords

Phosphate-washing waste • Calcination • Sodium hydroxide • Sodium silicate • Geopolymeric mortar

1 Introduction

Phosphate ore stands as the principal mineral product in Tunisia. This ore is extracted by the Gafsa Phosphate Company (CPG), based in the Tunisian southwest. This ore

is composed of fluorapatite, silica, carbonate, gypsum, organic and aluminosilicates materials [1, 2]. The CPG uses the washing and flotation techniques as means whereby the aluminosilicate, silica and carbonate could be eliminated, and a high percentage (~80%) of phosphate ore recovered for commercial use. This process creates a large volume of waste, engendering a real environmental problem.

This waste named phosphate washing waste (PWW), contains quartz, carbonate, aluminosilicate and fluorapatite in respect of the phosphate ore relating mineralogical composition of [3, 4]. Based on the relevant literature, such waste can be explored in different fields as lightweight [5], Ceramic [6], Membrane [7, 8]. The composition of the PWW can be also adequate for the synthesis of other products, particularly, alkali activated materials (geopolymeric mortars).

2 Materials and Methods

The phosphate washing waste, as used in this study, is collected from the Metlawi storage pond (Lavrie IV). It was filtered and dried at 105 °C, for 24 h, to remove the water, then crushed and sieved to the grain size of less than 100 µm. The obtained powder was calcinated at 700 °C for 2 h in a muffle furnace at a heating rate of 10 °C/min.

The powder was, then, characterized via X-ray fluorescence, and the samples via X ray diffraction (XRD), with Cu K α radiation $\lambda = 1.54060 \text{ \AA}$ at 40 kV and 40 Ma. The sample was then scanned from 5° to 60°, at a speed of 0.02 s⁻¹.

The powder was activated using alkaline solutions based on a sodium hydroxide NaOH solution 10 M, and a sodium silicate solution Na₂SiO₃. The (Na₂SiO₃/NaOH) ratio is of the order of 1.

The obtained samples of geopolymers mortar were covered and left in the laboratory at ambient temperature, to be characterized at 7 and 28 days.

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3 Results

3.1 Chemical Composition

The data figuring on Table 1 display the chemical composition of the PWW, revealing that the silica, calcium, aluminum and phosphorus persist in major quantities, while the other elements are present in trace amount.

3.2 XRD Characterization

Figure 1 depicts the X-ray diffractograms of the PWW at 25 and 700 °C. The pattern of PWW at 25 °C shows the persistence of carbonates, essentially as a calcite, at 3 Å. It displayed peaks at 8.76 Å, indicating the presence of natural zeolite heulandites as a main aluminosilicate mineral. The PWW also contains a fibrous clay palygorskite, displayed at a peak of 10.09 Å, a small amount of quartz with a peak of 3.41 Å, and gypsum with a peak of 2.8 Å.

The patterns reveal crystalline and amorphous phases, which are important for the powder calcined at 700 °C. It displays that the fluorapatite mineral still appears after calcinations. The curves also indicate that the calcite, palygorskite, and heulandite phases were progressively decreased. This decrease in the calcite phase is related to the decarbonation. For the heulandites, the decrease was related to the temperature effect on the decomposition of the heulandites structure [9]. In addition, reflections of new phase emerge, mainly, C₂S.

4 Discussion

Figure 2 reports the geopolymer XRD as based on the powder calcined at 700 °C (G⁷⁰⁰) at 7 and 28 days. The patterns represent a crystalline and amorphous phases. The comparison established between the XRD calcined PWW results at 700 °C with those of the hardened samples, reveals that the crystalline phases, originally existing at 700 °C as the Heulandites, palygorskite and anhydrite proved to disappear. Traces of calcite can be detected in the

geopolymeric mortars, which could well be the result of either the samples' carbonation, or the quantities persistent in the calcined sample. The patterns appear to reveal that the fluorapatite was unaltered by the alkali solution. In the patterns, the peaks of quartz appear more intensely than those relevant to the calcined materials. This is mainly due to some sand particles used as aggregate, which could not be totally eliminated during the XRD samples' preparation. Furthermore, the crystalline sodium compounds did not appear in the XRD patterns; it could be assumed that the sodium was included in the amorphous geopolymers phase. This G⁷⁰⁰ associated performance can be approved by the SEM image, as appearing in Fig. 3, highlighting that the mortar was noticeably heterogeneous with a compact structure.

The compressive strength, depicted in Fig. 4, indicates that the geopolymer phases persistent in G⁷⁰⁰, as resulting from the aluminosilicates phases related destruction, stem from the heulandites and the palygorskite. The evolution of the compressive strength has its interpretation in the amorphous phases and the C₂S predominant in the powder calcined at 700 °C. According to Sanchez et al. [10], the C₂S prevalent in the alkali medium proves to have a positive effect on the mechanical strength. The reached compressive strength value is of the range of ~11 MPa, satisfying the BS 6073 relating requirements, which entails a value ≥ 7 MPa of compressive strength for the masonry brick application [11].

5 Conclusions

The most obvious finding emanating from the conduction of such a study is that the calcination, as associated with the phosphate washing waste, proved to have a noticeable effect on the phases persistent in the powders.

During the G⁷⁰⁰ curing time, for 7 and 28 days at ambient temperature, the compressive strength appeared to evolve, due mainly to the presence of the aluminosilicate phases.

The microscopic observation revealed well that the G⁷⁰⁰ turns out to display a noticeable heterogeneous and dense structure.

Table 1 The PWW chemical composition

Oxides	SiO ₂	CaO	P ₂ O ₅	Al ₂ O ₃	Na ₂ O	K ₂ O	MgO	SO ₃	Fe ₂ O ₃	F ₂ O	LOI
%	42	26.5	10	9.77	1.12	0.673	3.09	3.39	2.31	0.96	17

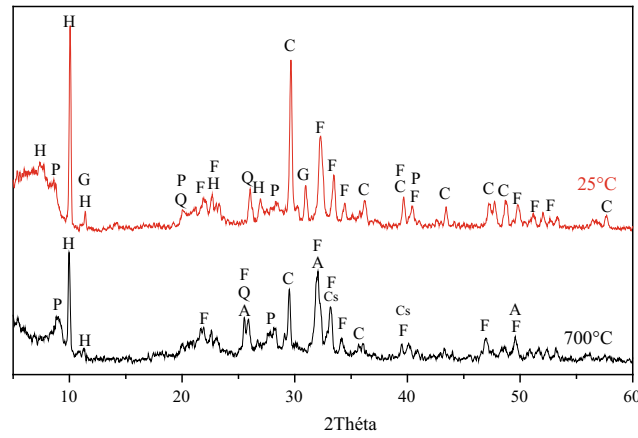


Fig. 1 X ray diffractograms of the PWW uncalcined and calcined 700 °C. C: Calcite, H: Heulandites, G: Gypsum, Q: Quartz, F: Fluorapatite, P: Palygorskite A: Anhydrite, Cs: C₂S

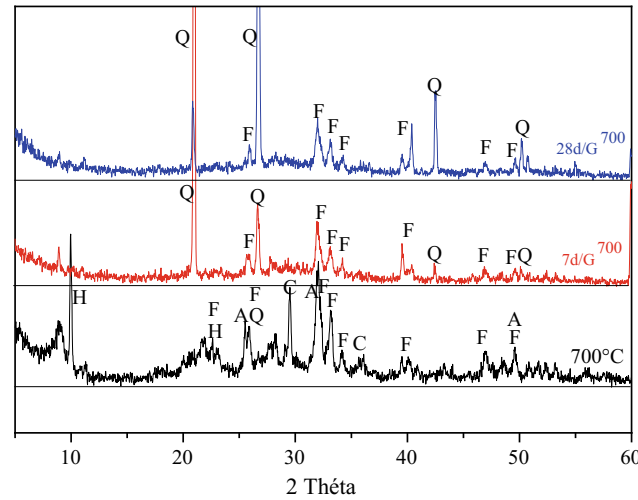
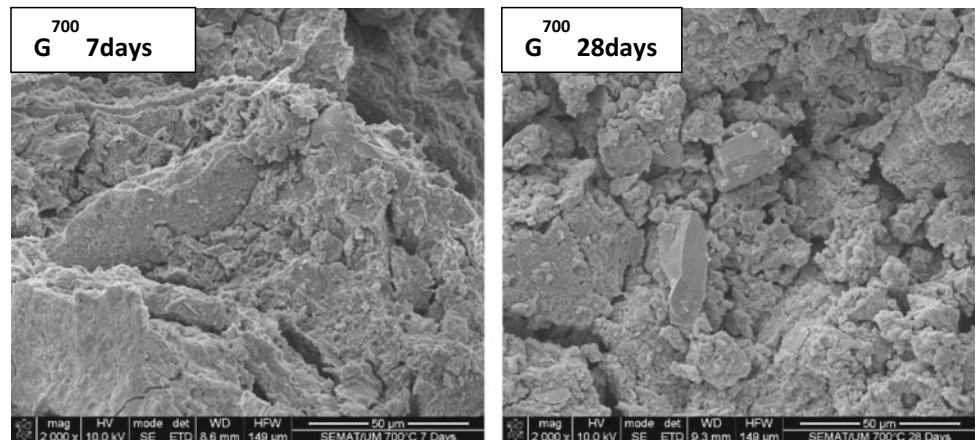


Fig. 2 X ray diffractograms of the PWW calcined 700 °C and G⁷⁰⁰

Fig. 3 SEM of G⁷⁰⁰ at 7 and 28 days



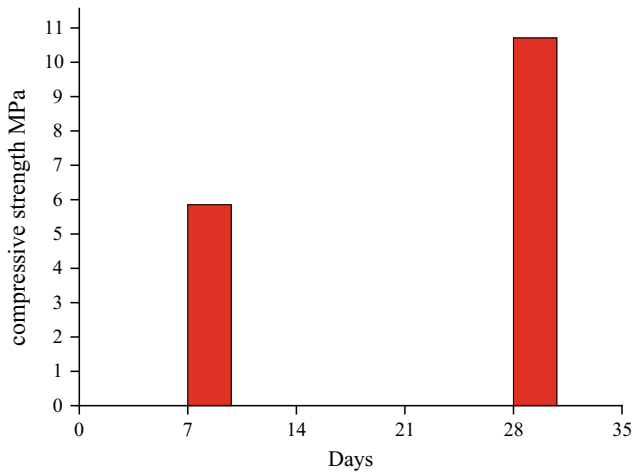


Fig. 4 Compressive strength of the G700

Based on the BS 6073 requirements, the compressive strength reached results make the product fit for masonry applications.

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