



Effect of the calcinations temperatures of phosphate washing waste on the structural and mechanical properties of geopolymeric mortar

R. Dabbebi^{a,b,*}, J.L. Barroso de Aguiar^b, A. Camões^b, B. Samet^a, S. Baklouti^a

^a University of Sfax, Laboratory of Industrial Chemistry, Sfax 3038, Tunisia

^b University of Minho, C-TAC Research Centre, Guimarães 4800-058, Portugal

HIGHLIGHTS

- Phosphate washing waste is one of phosphate sub product.
- The calcined phosphate washing waste was used as geopolymer precursors.
- The performance and the formed phases after alkali activation depend with calcination temperature.

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ABSTRACT

The phosphate industry is facing serious environmental problems. This issue is caused by the waste after the extraction of the phosphate ore. The waste termed as phosphate washing waste (PWW) was filtered and dried at 105 °C for 24 h to remove the water. The dried PWW was milled, sieved in a 100 μm sieve and characterized by X-ray fluorescence (XRF). The resulting waste was calcined at 600, 700 and 800 °C, the calcined and uncalcined waste were investigated by X-ray powder diffraction (DRX), Fourier transform infrared (FTIR), simultaneous differential thermal and thermogravimetric analyses (DSC-TG) and scanning electron microscope (SEM). The PWW was activated with sodium hydroxide (NaOH) and sodium silicate to produce geopolymeric materials. It was found that the calcination temperature and the extra water added to the mixture to assure the good workability play an important role on the development of the system's workability and compressive strength.

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1. Introduction

The solid waste generation have considerably accelerated because of the fact that the global population increase, rapid urbanization and consumption [1]. The global quantities of solid waste generation such as factories mills and mines waste were about 11 billion tons per years. This waste is mainly created by the use of natural resources, every year about 120–130 billion tons of natural sources are consumed [2]. This large amount of waste is one of the major problems which leads to severe environmental pollution and significant land occupation. There are numerous type of industrial waste such as mining waste, processing waste and metallurgical waste [3–5]. The recycling and reuse of these waste is important ways to conserve some of the natural resources for the new generation, save energy, reduction of costs, practical application, financial returns and encourage innovation [4]. These

industrial wastes were differently reused by numerous researchers to reduce its load by exploitation as alternative in different fields like the Table 1 shows.

Phosphate ore is one of important minerals in the world. According to industry analyst, its production was about 147 million tons in 2017, which can increase to reach 168 million tons in 2021 [6]. This rock mine is one of essential product extracted by Gafsa Phosphate Company (CPG). It extracts an average of about 8 million of rocks in recent years [7]. Tunisian phosphate ore is composed by fluorapatite, silica, carbonate (calcite), gypsum, organic and aluminosilicates materials [7–9]. This ore is used in the production of phosphoric acid and as fertilizer to be used in agriculture and other fields [10]. To increase the percentage of P₂O₅ and produce nearly 80% of phosphate ore for commercial use, the ore was enriched by the extraction of the gangue such as aluminosilicates, silica and carbonate. The CPG use the washing and flotation as techniques to eliminate these components. These operations create a large volume of waste which generates real damage to the environment.

* Corresponding author at: University of Sfax, Laboratory of Industrial Chemistry, Sfax 3038, Tunisia.

E-mail address: dabbebirawia@gmail.com (R. Dabbebi).

Table 1
Data collection related to the use of waste as alternative in different fields.

Wastes	Alternative	References
Fly ash	Alkali activated materials	[11–13]
	Geopolymer materials	[14–16]
	Removal of heavy metals	[5]
	Synthesis of zeolites	[17]
Ceramic waste	Thermal protective coating TPC	[18]
	Geopolymer materials	[19]
	Aggregate	[20]
	Building materials	[3,21]
	Synthesis of zeolites	[22]
Blast furnace slag	Alkali activated materials	[23]
	Geopolymer materials	[15]
	Removal of heavy metal	[5]
	Glass-ceramic	[24]
Waste glass	Aggregates	[25–27]
	Geopolymer materials	[25]
	Building materials	[28]
	Alkali activated materials	[29]
	Ceramic	[30]
Red mud	Geopolymer materials	[25]
	Synthesis of zeolite ceramic	[31]
	ceramic	[32]
Steel slag	Geopolymer materials	[33]
	Cement and concrete ceramic	[34,4]
Phosphate sub-product	Recovery of waste water	[35]
	Building materials	[36]
	Membrane	[37–40]
	Ceramic	[41,42]
	lightweight	[43]

This waste has different names used in the literature [38] which are phosphate sub-group [41,42], phosphate slimes [36,45,46] and phosphate tailing [38,43,47]. This waste can be divided in two categories. The first is the waste with particle size more than 2 mm and the second is the waste with particle size less than 70 μm [8]. The last was stored in a pond with around dozen hectares [44] as shown in the Fig. 1 and named phosphate slimes or phosphate washing waste (PWW).

Based on previous research of the phosphate ore, these two types of waste contain probably quartz, carbonates, aluminosilicates and fluorapatite with consideration of the mineralogical composition of phosphate rocks [9,48–50]. These wastes were used and valorised in different fields [51,52]: ceramic industry [26,53], brick manufacture and aggregate production for road and concrete [44,54]. In accordance to the literature, no studies have dealt with the reuse of Tunisian PWW. The composition of the PWW can be suitable for the synthesis of many products like ceramics and alkali activated materials (geopolymeric materials).

Geopolymer is a subclass of alkali activated materials. This term was applied for the first time in 1970 by Davidovits [55]. This material is inorganic polymer with lower CO_2 footprint. It is produced from a large number of natural minerals [56,57] or industrial waste [12–15,18,25,33,56,58] activated by an alkaline solution at

ambient or slightly high temperature. It presents sometimes problems of cost and durability [59].

The geopolymeric mortars are composed of basic components such as aluminosilicate precursor, sand and alkali activator solution which can be basic or acid [60–62], and by supplementary materials like extra water, plasticizer and fiber.

This study focuses on the synthesis and the characterization of geopolymeric mortars based on the Tunisian PWW calcined at different temperatures.

2. Experimental work

2.1. Materials

The phosphate washing waste used in this study was taken from Metlawi's storage ponds (LaveriesIV), a region in the south of Tunisia. The waste was in the form of slurry containing a flocculent. The slurry is filtered and dried at 105 °C for 24 h to remove the water. The dried PWW was crushed and sieved to the grain size less than 100 μm , then the powder was calcined at 3 different temperatures 600 °C, 700 °C and 800 °C for 2 h with heating rate 10°/min in a muffle furnace LAB TechDaihan LAB TECH CO. LTD.

2.2. Experimental techniques

The obtained powder was characterized with X-ray fluorescence (Philips X' UNIQUE II), X ray diffraction (XRD) model Bruker D8 Discover with $\text{Cu K}\alpha$ radiation $\lambda = 1.54060 \text{ \AA}$ at 40 kV and 40 mA, the sample was scanned from 5° to 60° at a speed of 0.02° s^{-1} , and by the Fourier transform infrared (FTIR) model Perkin Elmer spectrum BX spectrophotometer apparatus in transmittance mode in the wave number range between 4000 and 400 cm^{-1} . The grain size was measured by laser diffraction (FRITSCH Analysette 22 MicroTec plus). The microstructure observations of the samples were performed using a scanning electron microscope SEM 200 operating at 15 kV. The samples were sputter coated with 40 nm film of Au-Pd (80–20 wt%) prior of the analysis. DSC-TGA of the samples were done by TA Instruments – SDT 2960 Simultaneous DSC-TGA under argon atmosphere with heating rate 10 °C/min from room temperature to 1000 °C.

2.3. Geopolymeric mortars preparation

The composition of the mixtures is presented in the Table 2. The powder calcined at 3 different temperatures 600 °C, 700 °C and 800 °C were activated using alkaline solutions based on: sodium hydroxide NaOH solution 10 M (SH), and sodium silicate solution Na_2SiO_3 (SS) with chemical composition $\text{Na}_2\text{O} (\text{SiO}_2)_x \cdot y (\text{H}_2\text{O})$ with $3.19 < x < 3.53$ and $50\% < y < 60\%$ from the Portuguese Industry MERKANDA. The $(\text{Na}_2\text{SiO}_3/\text{NaOH})$ ratio is of the order of 1.

First the solution of NaOH 10 M was prepared with water and pellets of NaOH and after 24 h it was mixed with the sodium silicate solution. The mortar compositions were determined for a unitary volume of 1 m^3 [63]. This method takes into account the densities of the materials present in the Table 3. First the liquid/solid ratio was fixed after preliminary essays, next the needed quantities of the extra water and the sand were determined to obtain the adequate workability for the geopolymeric mortars. The calcined PWW powder and the sand were mixed first for 30 s, then the alkali solutions and extra water were added and all the mixture was mixed for 90 s with low speed and other 90 s with high speed. Right after mixing the mortar, the flow table test of fresh geopolymeric mortar was determined in conformity with the European Standard EN1015-3 [64]. The diameter obtained with each measurement was only considered when equal to $150 \pm 1 \text{ mm}$. The mortar was placed in the mold with $50 \times 50 \times 50 \text{ mm}^3$. Then, the molds were vibrated for 30 s with a vibration table to eliminate the air bubbles. The samples were covered with a



Fig. 1. Tunisian phosphate washing waste pond.

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