



Talc-based cementitious products: Effect of talc calcination



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ABSTRACT

This study reports the use of calcined talc for cementitious products making. The calcination is used to enhance the availability of magnesium from talc to react with phosphate for cement phase formation. It is shown that previous calcination of talc leads to products having enhanced mechanical performance due to the formation of more cement phase than in products based on raw talc. Talc fired at 900 °C was found to be the one in which magnesium release was maximal. Firing at temperature higher than 900 °C leads to the stabilization of enstatite, which decreased the magnesium availability. The cement phase is struvite, which was better detected on the X-ray patterns of the products involving fired talc. All the products have very rapid setting time and low shrinkage.

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1. Introduction

Cements are materials of common use for human welfare. Although these are ancient materials with well-known chemistry, it is necessary to enhance the cement quality and to find new cementitious raw material to overcome the global warming challenge due to the production of the largely used Portland cement. This research area arouses interests, which focuses on the reduction of the CO₂ emission and for alternative cement formulation using non-classical raw materials [1,2]. Indeed, the Portland cements are considered to be a cause of the global warming increase due to the large amount of CO₂ that is produced by the related industries [3]. Another reason to seek for alternative cement formulation is the related energy cost for the production of hydraulic cements [4,5]. In addition, the need of cement products to be used in humid or acidic environments has aroused interest in studies that focused on the development of cements with low setting time. In this category, magnesium phosphate cements (MPCs) are among promising products [6–9]. MPCs are chemically bonded phosphate ceramics, which were discovered and developed as dental cement in the late 19th century [10]. In these materials, the chemical bonding

is formed by a through-solution acid-base reaction between dead burned magnesia and phosphate. In early 1970s, magnesium phosphate cements were investigated as structural materials for usage as fast setting repair material. Recently, a wide range of applications of MPC were developed in civil engineering. These include uses of light magnesium cement foamed material [11], and wastes stabilized and solidified [12,13]. MPCs also exhibit improved characteristics such as low setting time, ability to set and harden at low temperature as –20 °C, high bonding strength, and very good durability including resistance to chemical attack and permeation [14].

In the prospect of developing alternative material source of magnesium, we recently reported the possibility to use talc, which is a hydrated layered magnesium silicate [15]. In this study, evidences that magnesium from the talc structure can be involved in a reaction with phosphate to form cementitious products were demonstrated. However, the products' performance, namely compressive strength, was low and when talc was partially replaced by magnesia (MgO) up to 10%, the performance was increased. This was an indication that the mechanical response of the products could be increased if the magnesium contained in talc is made more available for the reaction process. Thermal treatment of the talc was proposed as a potential way to increase magnesium availability.

In the present study, talc was thermally treated at various temperatures and used for cementitious products formulation with potassium dihydrogenophosphate. Fourier transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRPD), and scanning electron microscopy coupled with energy dispersive X-ray

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spectroscopy (SEM/EDS) were used to analyze the products. Setting time, linear shrinkage, and compressive strength are the mechanical parameters that are evaluated.

2. Materials and methods

A talc from Lamal Pougue in the Boumnyébèl area at about 93 km from Yaoundé (Center region, Cameroon) was used. The area description is presented in Nkoumbou et al. [16]. The raw material, indexed T0804, was manually ground and sieved at 100 μm . The chemical composition of this talc is given in Table 1. The infrared and X-ray diffraction characterizations of the raw talc can be found in Ngally Sabouang et al. [15].

The raw talc is thermally treated using a muffle furnace Carbolite, type CWF 11/5. The temperature rate was set at 5 $^{\circ}\text{C}/\text{min}$ and for each firing temperature, the sample is left to soak at the final temperature for 2 h. The heating temperature range was from 400 $^{\circ}\text{C}$ to 1000 $^{\circ}\text{C}$ with an increment of 100 $^{\circ}\text{C}$ between two consecutive heating temperatures. The corresponding fired products are indexed consecutively as T400 up to T1000 prior to infrared spectroscopy and X-ray diffraction analyses.

Analytical burned magnesia from Rhône-Poulenc, sieved at 80 μm , was used at a dosage of 10% in replacement of part of talc as previously established [15]. As described in Ngally Sabouang et al. [15], using X-ray diffraction, the product is mainly made of magnesia with some traces of forsterite and dolomite.

Potassium dihydrogenophosphate was purchased from Alfa Aesar and used as phosphate ingredient.

The cement pastes were prepared as follows:

In an acidic solution of potassium phosphate, a known amount of thermally treated talc and, if needed, MgO are added and thoroughly mixed during 5 min using a M&O mixer. The viscous paste obtained is used for samples molding and for setting time determination. The samples were molded using a cylindrical mold having a diameter of 30 mm and a height of 60 mm. The pastes in the mold are vibrated for 10 min on an electrical vibrating table (M&O, type 202, No. 106) to remove entrapped air bubbles. The samples are covered with a polyethylene bag and left for consolidation. The consolidated samples are kept at the ambient temperature (24–27 $^{\circ}\text{C}$). During samples consolidation, linear shrinkage is evaluated every 7 days up to 28 days. The 28-day samples are used for compressive strength measurement and for SEM/EDX, XRD and FTIR analyses.

Two series of samples are prepared for each thermally treated talc sample: a sample without addition of MgO and a sample containing 10% MgO. For each formulation, the ratio water/dry matter is 0.2 (in absence of MgO) and 0.4 (in presence of MgO) and the ratio phosphate/talc (or phosphate/talc + MgO) is 0.4. The products' indexations are given in Table 2.

Powder X-ray diffraction patterns were recorded using a Philip Panalytical PW3710 diffractometer equipped with an iron anticathode operating with a Fe K α radiation ($\lambda = 1.9373 \text{ \AA}$) at 40 kV and

30 mA. The diffraction patterns were obtained from 5 $^{\circ}$ to 75 $^{\circ}$ with a step of 0.025 $^{\circ}$.

Infrared spectra were recorded in transmission mode using a Nicolet Nexus spectrometer. The spectra, recorded from 4000 cm^{-1} to 600 cm^{-1} with a resolution of 1 cm^{-1} , are accumulation of 32 scans.

SEM images are recorded using JEOL JSM-840A and for coupled SEM–EDS analysis, an XL 30 microscope by FEI Company is used. The samples were carbon-coated prior to analysis for coupled SEM–EDS.

Setting time was accessed using a Vicat apparatus according to the EN 196-3 standard.

Measurements of linear shrinkage were done using a Calliper on the hardened mortars aged of 1, 7, 14, or 28 days respectively. Linear shrinkage was calculated using the following equation:

$$R_L = \frac{(L_0 - L) \times 100}{L_0}$$

where L_0 is the initial length of specimens at first days and L is the length of specimens after a given number of days [15].

Compressive strength was determined on 28-day cylindrical samples with 60 mm height using an electro-hydraulic press (M&O, type 11.50, No. 21) with capacity of 60 kN. The samples are submitted to a compressive force at average rate of 3 mm/min until the sample failed according to ASTM C 109 standard test method. The compressive strength was calculated using the following equation:

$$\delta = \frac{F}{A},$$

where F is the failure force and A is the cross-section of the sample.

3. Results and discussion

3.1. Characterization of the fired talc

The XRPD patterns of the various calcined talc samples are presented in Fig. 1. It can be noted that for 400–800 $^{\circ}\text{C}$, no significant transformation is observed. At 800 $^{\circ}\text{C}$, a decrease of the d_{002} peak of talc at 9.3 \AA is observed and was associated with the dehydroxylation of the talc in accordance with the work by Villieras [17] in which dehydroxylation was found to be situated between 800 $^{\circ}\text{C}$ and 850 $^{\circ}\text{C}$. At 900 $^{\circ}\text{C}$, the decrease of this d_{002} peak is more obvious, confirming the dehydroxylation of talc. At this temperature, the appearance of peaks at 2.85 \AA , 3.11 \AA , and 2.48 \AA associated to enstatite formation is observed. At 1000 $^{\circ}\text{C}$, the enstatite peaks are better defined. As also reported by Villieras [17], it is observed that during talc firing between 900 $^{\circ}\text{C}$ and 1000 $^{\circ}\text{C}$, talc is converted to enstatite. Silica, as a product of dehydroxylation, is not detected given that silica is in amorphous form and will crystallize into cristobalite after 1200 $^{\circ}\text{C}$.

Table 1

Chemical analysis of major oxide in the used talc.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	LOI ^a	Total
T0804	51.71	0.61	6.30	0.09	32.08	0.18	<L.D.	<L.D. ^b	0.01	<L.D.	8.01	98.99

^a Loss on ignition.

^b Detection limit.

Table 2

Designation of the various formulations with raw or fired talc.

Firing temperature	Raw	500	700	800	900	1000
Formulation without MgO	TB000	TB500	TB700	TB800	TB900	TB1000
Formulation with 10% MgO	TB001	TB501	TB701	TB801	TB901	TB1001

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