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## TALC AS RAW MATERIAL FOR CEMENTITIOUS PRODUCTS FORMULATION

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**Abstract:** This study reports the characterisation of a talc from Cameroon as a possible source material for cement formulation. To that end, the talc sample was characterized and mixed with a solution of sodium polyphosphate to formulate the cementitious products. Addition of magnesia (MgO) was done to analyse the effect of available MgO on the products. Fourier transform infra red, X-rays diffraction, linear shrinkage, compressive strength and Scanning electron microscopy were used to analysed the products. The compressive strength increased with MgO addition and the linear shrinkage decreased. All the analyses indicate that talc is a raw material of interest in cementitious products formulation however, the availability of the MgO is a key factor for a better performance of the products.

**Keywords:** Talc; Magnesia; Cement; compressive strength

## 1- Introduction

Cement formulation is presently a growing research area due to the variety and development of construction and building material. The ever growing concerns over global warming and other detrimental ecological changes has spurred the research and development of new kinds of cementitious materials produced from non conventional resources. The reason is that the production of 1ton of hydraulic cement will generate about 900 kg of carbon dioxide and consume about 5 billion joules of electrical power and fuel energy [1, 2]. The geopolymers for instance are a family of cementitious materials developed in late 70's and appear as an alternative of interest [3]. Kaolinite is nowadays a material largely used in geopolymers making as shown by the increasing number of publications that deal with metakaolin based geopolymers [4-8]. Some new classes of raw materials are now being introduced in this domain. For instance, the use of volcanic ashes as raw material in geopolymer making is now established [9-10].

Talc is a hydrated layered magnesium silicate with chemical formula  $Mg_3Si_4O_{10}(OH)_2$ . It is a hydrophobic material that easily blends and disperses within organic media including

polymers. Talc is widely used as reinforcing filler in several industrial products such as paper, paints, rubbers, polymers, ceramics, refractory materials [11,12]. The composition of talc make it a potential raw material in cement formulation since magnesium can beneficially interact with phosphate to give cementitious material, as in phosphate-magnesia cements [13-16]. Talc-based cementitious material are already used as prosthesis in dental surgery [17]. Recently, Pusch et al., (2013) [18], have used talc as a super plasticizer for the formulation of cement poor concrete to be used for boreholes sealing in rocks.

Given that some talc deposits are found along the pan-African Yaoundé fold belt at the border of the northwestern edge of the Congo craton [19,20], it is obvious that research seeking for talc valorization is to be undertaken. In the present work, talc from Cameroon is used as raw material for the formulation of cementitious products. Because the mobilization of magnesia is expected, a study of the effect brought by the addition of magnesia is also carried out. Analysis of some properties of cement paste made from talc is presented here as a first step towards valorization of talc as raw material in cement formulation. Powder X-ray diffraction (XRD), Fourier Infra red spectroscopy (FTIR), scanning electron microscopy (SEM), mechanical testing (compressive strength) and linear shrinkage measurements are used to characterize the formulations.

## ***2- Materials and methods***

A talc from Lamal Pougue in the Boumyébèl area at about 93 Km from Yaoundé (Center region , Cameroon) was used. The area description is presented in Nkoumbou et al., (2008) [19]. The raw material was manually ground and sieved at 100  $\mu\text{m}$  prior to characterization.

Sodium polyphosphate  $((\text{NaPO}_3)_n)$  analytical grade containing 60%  $\text{P}_2\text{O}_5$  purchase at Riedel de Haen AG, was used as phosphate source.

The magnesia used is a analytical grade product from Rhône-Poulenc. The IR spectrum (Figure 1) shows traces of sulphur compounds through the stretching band of

sulphate at  $1423\text{ cm}^{-1}$  and S-O stretching at  $847\text{ cm}^{-1}$ . The bands at  $1459\text{ cm}^{-1}$  indicates the presence of carbonate. Some silicates impurities are detected from the band at  $1077\text{ cm}^{-1}$  due to forsterite ( $\text{Mg}_2\text{SiO}_4$ ) as also observed from the XRD pattern (Figure 2). The X ray pattern also indicates that the magnesite is almost completely converted into magnesia.

The cement pastes were prepared as follows:

In an acidic solution of sodium polyphosphate, known amounts of talc and calcined MgO were added. The mixture is thoroughly mixed during 5 minutes using a M&O mixer.

This mixture is used to prepare cylindrical samples using cylindrical mould having a diameter of 30 mm and a height of 60 mm. The paste in the mould are vibrated for 10 minutes on an electrical vibrating table (M & O, type 202, No 106) to remove entrapped air bubbles. The samples are cover with a polyethylene bag and left for consolidation. The consolidated samples are kept at the ambient temperature ( $24\text{-}27^\circ\text{C}$ ) and the mechanical analysis are carried out on the 28 days aged samples. The sample indexed CITA<sub>0</sub> is prepared without any added magnesia. For this sample, 50 g of talc with 50 g of 40% (in weight) solution of sodium polyphosphate are used. For samples in which magnesia is added, the amounts of magnesia, expressed in percentage mass with respect to the initial mass of talc (50 g), were: 5%, 10%, 15% and 20%. The obtained products are indexed CITA<sub>1</sub> to CITA<sub>4</sub> in the increasing order of magnesia content.

The cement sample are analyzed using powder X-ray diffraction (XRD), Fourier transform infra red (FTIR) spectroscopy, Scanning electron microscopy (SEM) coupled to energy dispersive X-ray spectroscopy (EDS), linear shrinkage and compressive strength measurements.

Chemical analysis of major elements in the raw talc was obtained by inductive coupled plasma by atomic emission spectrometry (ICP-AES) at the *Service d'Analyse des Roches et des Minéraux* (SARM, Nancy).

Powder X-ray diffraction patterns were recorded using a D<sub>8</sub> Advance Bruker diffractometer equipped with a Co K $\alpha$  radiation ( $\lambda = 1.7890 \text{ \AA}$ ) operating at 35 kV and 45 mA. The diffraction patterns were obtained from 1.5° to 32° at a scanning rate of 1° min<sup>-1</sup>.

Infrared spectra were recorded in diffuse reflection mode using a Bruker IFS 55 spectrometer. The spectra, recorded from 4000 cm<sup>-1</sup> to 600 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup>, are accumulation of 200 scans.

The SEM micrographs and EDS data were obtained on a Hitachi S-4800 using a YAG (Yttrium Aluminium Garnet) backscatter secondary electron detector. The samples were carbon coated prior to analysis.

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

$$R_L = (L_0 - L) \times 100 / L_0$$

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 [10].

Compressive strength was determined on cylinder samples with 60 mm height and aged 28 days 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 calculate using the following equation:

$$\delta = F/A, \text{ where } F \text{ is the failure force and } A \text{ is the initial cross section of the sample.}$$

### **3 Results and discussion**

#### ***Characterization of the used talc***

The chemical analysis of the used talc is given in table I. For the ideal talc formulation ( $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ ), the theoretical content of equivalent magnesium oxide is 31.9% and silica is 63.4%. Hence, the content in Magnesia (32.08%, MgO) and silica (51.71%,  $\text{SiO}_2$ ) is consistent with the magnesium silicate nature of the Talc. However, the material shows a deficiency in silicon, probably substituted by part of iron (6.30%,  $\text{Fe}_2\text{O}_3$ ). The IR spectra and the XRD pattern of the raw material are given in figures 1 and 2. On the FTIR spectrum (Figure 1), the O-H stretching vibration band of OH surrounded by 3 Mg ( $\text{Mg}_3\text{-OH}$ ) appears at  $3676\text{ cm}^{-1}$ . The band at  $3660\text{ cm}^{-1}$  is consistent with the stretching of OH surrounded by 2Mg and 1 Fe ( $\text{Mg}_2\text{Fe-OH}$ ) while the  $3649\text{ cm}^{-1}$  band is associated to OH surrounded by 1 Mg and 2 Fe ( $\text{MgFe}_2\text{-OH}$ ). The OH libration band is observed at  $667\text{ cm}^{-1}$ ; the stretching band for Si-O-Si is observed at  $1050\text{ cm}^{-1}$  and the combination bands of Si-O are observed at  $779\text{ cm}^{-1}$  [21-24]. Trace of calcite and/or dolomite is evidenced by the vibration bands of carbonate group at  $2535\text{ cm}^{-1}$ ,  $2922\text{ cm}^{-1}$ ,  $1818\text{ cm}^{-1}$  and  $1459\text{ cm}^{-1}$  [25]. The OH bands for chlorite (in the range  $3590\text{-}3415\text{ cm}^{-1}$ ) [24, 26] was not clearly observed and this indicates that the content in chlorite is rather low.

The XRD pattern (Figure 2) shows the typical talc reflections corresponding to (001), (002) and (003) basal planes at  $9.37\text{ \AA}$ ,  $4.68\text{ \AA}$  and  $3.12\text{ \AA}$  respectively. The presence of chlorite as associated mineral to talc is shown by the reflections at  $14.38\text{ \AA}$ ,  $7.10\text{ \AA}$  and  $4.76\text{ \AA}$ . The intensities of these peaks are relatively low and confirm the conclusion from FTIR of low content in chlorite. Trace amount of amphibole is suggested by a low intensity diffraction peak at  $8.25\text{ \AA}$ . The carbonate trace content observed from IR is also indicated by the reflections at  $3.05\text{ \AA}$  on the XRD pattern of the talc.

### ***X-ray diffraction and FTIR analysis of the cementitious products***

In figures 1 and 2, the infra red spectra and XRD patterns of the cements are presented together with the spectra of the raw products (talc and magnesia). The XRD pattern of CITA<sub>0</sub>

(formulation based on raw talc only) is given as reference together with CITA<sub>3</sub> and CITA<sub>4</sub>. Because the addition of MgO is done in order to improve the compressive strength, through an increase of cement phase formation, hence, given that CITA<sub>3</sub> and CITA<sub>4</sub> exhibited the highest values of compressive strength (see compressive strength section), which indicate more cement phase formation, they were chosen for both FTIR and XRD analyses.

It is easily noticeable from both IR spectra and XRD patterns that there is not formation of new crystalline phase. This observation is in accordance with what is reported for phosphomagnesia cement prepared with sodium phosphate [27]. The remaining Talc is clearly evidenced from X ray through the reflections at 9.31 Å and 3.11 Å. The disappearance of characteristic diffraction peak of magnesia in CITA<sub>3</sub> and CITA<sub>4</sub> clearly shows that almost all the magnesia has reacted. A part from water characteristic bands (3280 cm<sup>-1</sup> ( water OH stretching) and 1649 cm<sup>-1</sup> (in plane deformation)), the OH bands between 3700 and 3400 cm<sup>-1</sup> are characteristic of OH linked to magnesium (Mg-OH). The OH stretching of OH in a 3 magnesium environment at 3676 cm<sup>-1</sup> are characteristic of talc. The changes in the intensity of these bands are indicative of reaction of the magnesium from the talc. The bands at 667 cm<sup>-1</sup> show a more marked change. Given that this band was assigned to talc OH libration, then it is suggested to be an indicative of the magnesium from the Mg-OH interactions with the added phosphate. The band at 3699 cm<sup>-1</sup> is rather characteristic of magnesium from the added magnesia and the change in intensity of this band compared to the bands in the pure magnesia spectrum is indicative of the reaction of this magnesia. Two new bands at 1129 cm<sup>-1</sup> at 918 cm<sup>-1</sup> are observed in the FTIR spectra of CITA<sub>3</sub> and CITA<sub>4</sub>. The band at 1129 cm<sup>-1</sup> is assigned to asymmetric bending of P-O in PO<sub>3</sub> or PO<sub>4</sub> groups and the bands at 918 cm<sup>-1</sup> is assigned to asymmetric bending of P-O-P groups [28]. The presence of these phosphate characteristic bands **is an evidence** for the presence of phosphate in the matrix of the obtained products. It is then obvious that cement formation occurs and magnesium interaction with the



phosphate is responsible of cement phase formation but the added magnesia is more reactive than the magnesia from the talc structure.

### **Linear shrinkage**

The linear shrinkage as a function of the age of the hardened mortars, determined at the ambient temperature of the lab ( $24 \pm 3$ ), are presented on figure 3. The overall trend indicates that linear shrinkage decrease with increasing MgO content in the mixture. Linear shrinkage originates from capillary tension within the paste framework during synthesis [29]. Thus, high shrinkage is an indication of insufficient cement phase formation. Hence, the comparison of linear shrinkage, amongst the studied samples, indicates that the mobilization of MgO for cement phase formation is rather low in raw talc. From figure 5, it is obvious that increase cement formation is observed for 20% added MgO sample (CITA<sub>4</sub>). The availability of the MgO from the talc should then be improved in order to ameliorate the binder phase formation and hence the performance of the products.

### **Compressive strength**

The compressive strength of 28 day aged samples are presented on figure 4. The value of the strength varied from 3.35 MPa (Talc cement with 0% added MgO) to 4.50 MPa (Talc cement with 20% added MgO). The addition of the calcined magnesia (MgO) lead to an increase of the compressive strength. This increase is associated to increased cement phase formation and corroborates with more cement phase formation consecutive to MgO addition as previously concluded from the linear shrinkage. The mobilization of the MgO from Talc appears then as a key to better mechanical performances of the samples. It is evidenced that talc is a potential raw material for the formulation of cementitious materials.

### **Microstructure**

From SEM images for CITA<sub>3</sub> and CITA<sub>4</sub> are presented of figure 5. It can be observed that the materials have a homogeneous feature in which platy disc particles characteristic of talc could

be observed. These remaining platelets are held together by a binder phase due to the reaction of the magnesium with phosphate. The presence of many pores is coherent with the low mechanical performance and relatively high shrinkage. These pores are favorable to fluids capillary diffusion that induces shrinkage of the products.

The EDS analyses on figure 6 evidence unreacted mixed oxide of iron, chrome and Nickel. These elements are characteristic of identified minerals associated to talc (amphibole, chlorite with iron substitution and probably iron oxide). The sulphur is rather associated to the added calcined magnesia as observed from FTIR.

The cementitious materials could be ameliorated by increasing binder phase formation so that the remaining talc could be better held in the matrix and reduction of pores will be probably observed.

#### **4: Conclusion**

The present work was aimed at evaluating the potentiality of talc as a raw material for cementitious products preparation. For this prospect, formulated products were studied by infra-red spectroscopy, X-ray diffraction, linear shrinkage, compressive strength and SEM/EDS.

The infra-red and X-ray diffraction indicate that magnesia is the actual reactive phase for the cement phase formation that served as binder for the remaining talc particles to form cement. The linear shrinkage indicates low binder phase formation which is corroborated by the compressive strength and the SEM micrograph analysis. From compressive strength and linear shrinkage, it is shown that the addition of magnesia improve the performances of the products. It is then concluded that a better mobilization of the MgO from talc could be beneficially used to produce better cementitious products. All the results show that talc is a potentially interesting raw material in cementitious materials formulation. For the purpose of ameliorating the MgO mobilization from talc, a thermal treatment of the talc prior to

cementitious product formulation is presently being carried out and will be presented in an upcoming paper.

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