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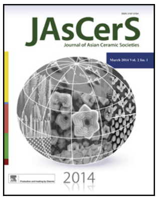

Effect of the rate of calcination of kaolin on the properties of metakaolin-based geopolymers

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ABSTRACT

Kaolin samples of the same mass were treated at 700 °C for the same duration of 30 min by varying the rate of calcination (1, 2.5, 5, 10, 15 and 20 °C/min) in order to obtain metakaolins which were used to produce geopolymers. Depending on the nature of each type of material, kaolin, metakaolins and geopolymers were characterized using thermal analysis, chemical analysis, XRD, FTIR, particle size distribution, specific surface area, bulk density, setting time and compressive strength. FTIR and XRD analyses showed that metakaolins except at 1 °C/min contained residual kaolinite whose quantity increased with the rate of calcination of kaolin and which influenced the characteristics of geopolymers. Thus as the rate of calcination of kaolin increased, the setting time increased (226 min (rate of 1 °C/min)–773 min (rate of 20 °C/min)) while the compressive strength reduced (49.4 MPa (rate of 1 °C/min)–20.8 MPa (rate of 20 °C/min)). From the obtained results the production of geopolymers having high compressive strength along with low setting time requires that the calcination of kaolin be carried out at a low rate.

1. Introduction

The term geopolymer refers to inorganic aluminosilicate polymers. These materials find applications in areas such as construction and civil engineering [1–3], encapsulation of certain toxic waste [4–7], removal of certain heavy metal cations from aqueous solutions [6–8] or the development of biomaterials [9]. These inorganic polymers are generally obtained by polymerization reaction between aluminosilicate and strongly basic alkaline solution [10]. Aluminosilicate raw materials commonly used for the synthesis of geopolymers are fly ash, blast furnace slag, volcanic ash or metakaolin [1,4,11,12]. The variability of chemical composition of fly ash, blast furnace slag or volcanic ash usually leads to geopolymers whose physical and mechanical properties vary from one aluminosilicate raw material to another [7,11]. Regarding metakaolin, the characteristics of resulting inorganic aluminosilicate polymers depend on the conditions of calcination of kaolin [11,13,14]. Elimbi et al. [11] studied the effect of temperature of calcination of kaolin on the properties of inorganic aluminosilicate polymers and found 700 °C as the temperature at which geopolymers with optimal characteristics are obtained. Castelein et al. [15] investigated the effect of heating rate of kaolin to reveal its influence on the formation of mullite. Elsewhere, Castelein et al. [16] studied both influence of temperature and rate of firing of kaolin on the distribution of iron neighboring kaolinite. Ptáček et al. [17] studied the effect of the rate of calcination of kaolin to investigate kinetic decomposition of kaolinite under non-isothermal conditions. The obtained results showed that dehydroxylation of kaolinite is controlled by the rate of third-order reaction under applied condition and the median of overall activation energy and frequency factor correspond respectively to $227 \pm 1\ \mathrm{kJ\,mol^{-1}}$ and $(9.4 \pm 0.3) \times 10^{9}\ \mathrm{s^{-1}}$.

In this study, samples of kaolin of the same mass were treated at 700 °C for the same duration by varying the rate of calcination. The metakaolins obtained were used for the synthesis of geopolymers whose characteristics were determined with a view to evaluate the effect of the rate of calcination of kaolin on their properties. To that end, kaolin was treated at the following calcination speeds: 1, 2.5, 5, 10, 15 and 20 °C/min. Depending on the materials, thermal and chemical analyses, particle size distribution, specific surface area,

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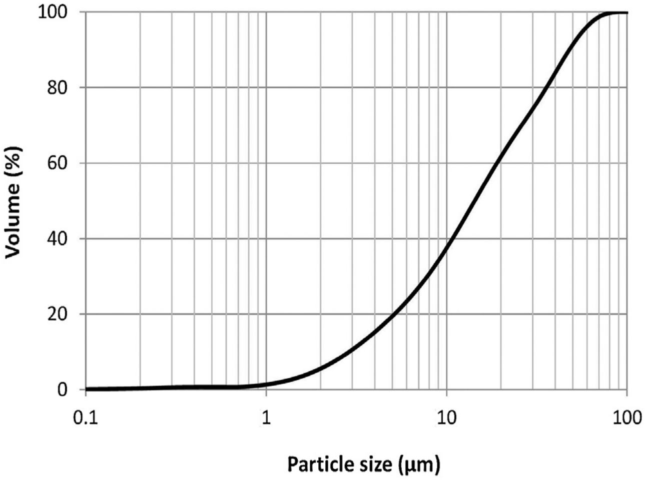

Fig. 1. Particle size distribution of the clay fraction (K).

FTIR, XRD were performed on kaolin, metakaolins and geopolymers. The setting time and compressive strength were determined as well on geopolymers.

2. Materials and methods

2.1. Materials processing

Metakaolins used in this study were obtained from kaolin provided by the NUBRU HOLDING Group which is involved in the valorization of certain local raw materials in Cameroon. Before being used, the kaolin was enriched with kaolinite by the sedimentometry process based on Stokes's law [18]. The clay fraction obtained labeled as K was first maintained at ambient laboratory temperature for a week, cured at 105 °C until its mass became constant and then it was ground and sifted through a sieve of mesh 90 µm. The resulting material was calcinated at 700 °C, the choice of this temperature being based on the work of Elimbi et al. [11]. Soaking of a mass of 1.5 kg of K at 700 °C in an electric muffle furnace on ceramic plates (Nabertherm Mod LH 60/14) lasted 30 min at the following rates of calcination: 1 °C/min, 2.5 °C/min, 5 °C/min, 10 °C/min, 15 °C/min and 20 °C/min. The obtained metakaolins were respectively labeled as MK_1, $MK_{2.5}$, MK_5, MK_{10}, MK_{15} and MK_{20}.

The alkaline solution was prepared by mixing sodium silicate and sodium hydroxide solution (12 M) so as to obtain Na_2O/SiO_2 molar ratio of 0.7. The sodium hydroxide solution was obtained by dissolving sodium hydroxide pellets with a purity of 99% in distilled water and sodium silicate was made up of (mass%) SiO_2 (26.5), Na_2O (8.0) and H_2O (65.5).

2.2. Paste and concrete samples preparation and analytical techniques

The preparation of geopolymer paste samples consisted of mixing alkaline solution with metakaolin powder according to liquid/solid mass ratio of 1:1 in an automatic Hobart mixer (*Controlab*) according to EN 196-1 standard [19]. Setting time of geopolymer pastes was determined using Vicat apparatus according to EN 196-1 standard [19]. For the determination of compressive strength, cubic samples of ($2 \times 2 \times 2\,cm^3$) were made by mixing standardized sand [19] and metakaolin powder and alkaline solution in mass ratio of 3:1:1. The molded mortar specimens were cured at 20 °C in a controlled room at 20 ± 2 °C and 98% RH condition. Demolding was carried out 24 h later and the cubic samples were covered with polyethylene film and stored at ambient temperature (20 ± 2 °C) under 98% RH condition for 28 days. According to the rate of calcination of kaolin, the cubic geopolymer specimens were respectively labeled as GPC_1, $GPC_{2.5}$, GPC_5, GPC_{10}, GPC_{15} and GPC_{20}. The cubic specimens aged of 28 days were subjected to compressive strength using an automatic electro-hydraulic press (*IGM*) operating at an average rate of 0.2 kN/s. Hardened geopolymer pastes aged of 28 days were crushed and sifted through a sieve of mesh 80 µm and powder was subjected to XRD (*Siemens D 5000 diffractometer using CoKα radiation with a rear monochomator*) and FTIR analysis using a Bruker Alpha-p IR spectrophotometer operating in absorbance mode (interval of wave number ranging was 4000–400 cm^{-1}). The particle size distribution was determined using a granulometer (*CILAS – LIQUID 1090*) whose interval of investigation ranged between 0.04 and 500 µm. The thermal analyses (TG and DSC) of K were performed using a *NETZSCH STA-449F3* operating at the rate of 20 °C/min in air. The chemical analyses of K and metakaolins were performed according to the ICP-OES process. Specific surface areas of metakaolins powders were

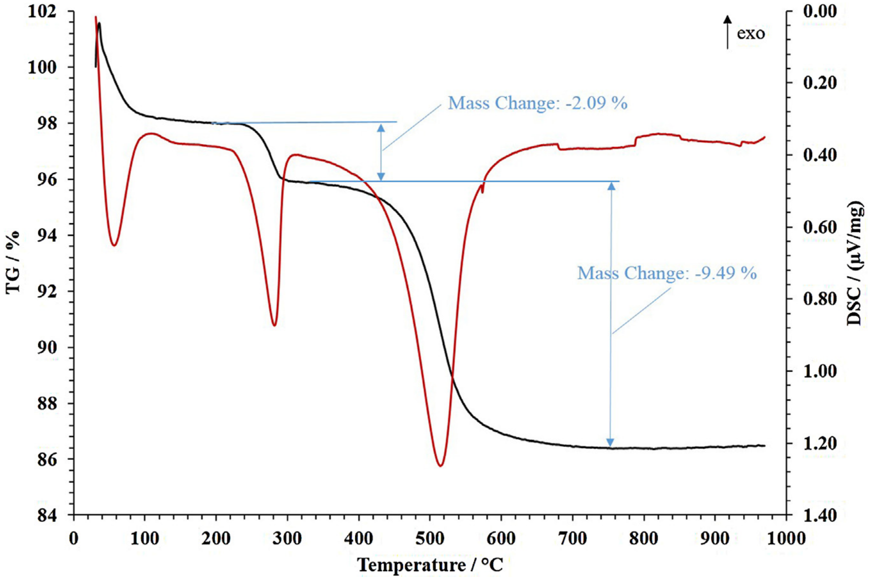

Fig. 2. Thermal analysis of the clay fraction (K).