



Original Research Paper

Properties of metakaolin based geopolymer incorporating calcium carbonate

A. Aboulayt^{a,*}, M. Riahi^b, M. Ouazzani Touhami^b, H. Hannache^{c,d}, M. Gomina^e, R. Moussa^a^a University Hassan II Casablanca, Faculty of Sciences Ain Chock, Laboratory of Physical Chemistry of Inorganic Materials, Casablanca, Morocco^b University Hassan II Casablanca, Faculty of Sciences Ain Chock, Laboratory of Mechanics, Casablanca, Morocco^c University Hassan II Casablanca, Faculty of Sciences Ben M'sick, Laboratory of Materials Engineering, Casablanca, Morocco^d Materials Science and Nanoengineering Department, Mohamed VI Polytechnic University, Lot 660 – Hay Moulay Rachid, 43150 Benguerir, Morocco^e CRISMAT, UMR 6508 ENSICAEN/CNRS, 6 Boulevard Maréchal Juin, 14050 Caen cedex 4, France

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ABSTRACT

An alkaline solution, thermally activated kaolinite clay and a mineral additive (calcium carbonate) were mixed with the aim to elaborate a geopolymer material with physical and mechanical properties comparable to those of classical construction materials.

The starting reagents were characterized by quantitative chemical analyses (XRF), mineralogical analyses (XRD), thermal gravimetric analyses (TGA), and grain size distribution measurements. The setting of the mixture (polymerization) was implemented by measuring the evolution of the viscosity as a function of time at different temperatures.

The geopolymers were synthesized at a temperature of 40 °C. The investigation of the mechanical behavior reveals that these materials display acceptable characteristics: the flexural and compression strength are around 4.6 and 26 MPa respectively, for an added calcium carbonate over dry matter ration up to 12% by weight.

The promising results exposed in this paper show that the geopolymer formulations can be adapted for applications in construction and civil engineering structures as an alternative to conventional materials.

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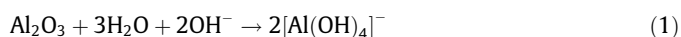
1. Introduction

The manufacturing process of construction materials is one of the most energy-consuming industries. In recent decades, manufacturers have sought to minimize the energy costs and to reduce the environmental impact by designing and developing new materials. In this scope, geopolymers have emerged as a promising alternative to conventional materials such as cementitious materials, ceramic coatings, etc. [1–3]. These materials are manufactured at low temperatures (<100 °C), without emitting greenhouse gases. The hydrothermal stability and resistance to acidic environment and/or reducing atmosphere are arguments favoring the use of these materials [4,5].

The synthesis of geopolymers was studied first by Davidovits [6]. In general, these materials are a result of a reaction between a solid aluminosilicate compound and an alkaline solution. Aluminosilicate compounds may be of natural origin such as metakaolin,

wollastonite and pozzolan, or come from industrial waste containing aluminum and silicon compounds such as fly ash from thermal power plants or slag from blast furnaces [2,7].

The alkaline attack results in the dissolution and hydrolysis of the silicon and aluminum through the alkaline reagent MOH where M is an alkali or alkaline earth cation (Na⁺, K⁺, etc.) according to the following sequences:



A condensation process follows, where a gel is formed among aluminates and silicates species to form aluminosilicate oligomers with amorphous three-dimensional structure. The final hardening stage comes next. Geopolymer materials can be described by the empirical formula $M_p [-(\text{SiO}_2)_z - \text{AlO}_2]_p$, w H₂O, in which the index p is the polymerization degree and z is the Si/Al molar ratio. These materials are then either of polysialate type (z = 1), of polysialate-siloxo (z = 2) or of polysialate-disiloxo (z = 3) type [6].

* Corresponding author.

E-mail address: abdelilah.aboulayt@etu.univh2c.ma (A. Aboulayt).

Although the elaboration of metakaolin-based geopolymers does not require high temperature conditions, some components, especially kaolinite-based clays, require a thermal activation to increase their reactivity and allow their transformation into metakaolin. That raises the overall elaboration cost of these materials. Indeed, recent studies are increasingly focused on the reduction of geopolymers' dependence on aluminosilicate clay sources by replacing the activated clay with some widely available and inexpensive natural or industrial powders. These sources can be used either to enrich the reactional medium with silicates and aluminates species or by acting as an inactive filler to improve the properties of geopolymer materials.

Although the use of carbonates in alkaline binders dates from ancient Egypt, only few research deals with their introduction in geopolymers. Based on chemical and mineralogical studies, Davidovits stated that the pyramid blocks were made of a mixture of limestone sand, calcium hydroxide, sodium carbonate and water. According to his investigations, pyramid blocks were not made of calcium fossilized layers as would happen in natural rock, but are rather oriented in a random manner similar to that of an artificial binder. XRD diffraction patterns of pyramid specimens indicate that calcite is the major crystalline phase [8].

Some works were interested in the role that can play calcium-based additives, in the elaboration of metakaolin-based geopolymers, such as calcium hydroxide [9], blast furnace slag (GBFS) [10,11] as well as certain sources of calcium silicates, such as cement, wollastonite, tremolite, anorthite etc. [11]. These studies agree on the fact that the reactivity of these additives is highly dependent on several essential factors: the thermal history of the calcium silicate sources, their crystallinity as well as the alkalinity of the alkaline activator. In a strongly alkaline medium, the main product is the geopolymer matrix which coexists with an amorphous C–S–H gel and unreacted particles [9–12]. A specific study of the impact of the addition of calcite (CaCO₃) and dolomite (CaMgCO₃) on the mechanical properties of metakaolin-based geopolymers was conducted by Yip et al. [13]. According to this investigation, the addition of 20 wt% of calcite is found to ameliorate the mechanical strength. Above that content, the mechanical strength falls and the shrinkage is increased. The reported improvement for calcite content below 20 wt% is imputed to the contributions of the calcium and magnesium ions to the increase of the contacts between the particles and the polymeric gel (despite their weak concentration in the mixture). Furthermore, the authors focused attention to the very low contribution of these ions in the polymerization mechanisms. Calcite is present in several natural rocks such as limestone, this later is a sedimentary carbonate rock composed mostly of calcium carbonate associated to other crystalline phases such as dolomite (calcium-magnesium carbonate, Ca Mg[CO₃]₂) and other impurities. A recent study conducted by Qian and Song [14] focused on the impact of the addition of limestone (up to 30 wt%) on a geopolymer prepared by alkali activating metakaolin (with potassium hydroxide). They report that 10 wt% addition of limestone powder increased the mechanical properties and the workability of fresh geopolymer (the fluidity was determined by the Mini-slump spread test). They suggest that it could be attributed to the formation of a more compact structure and a better particle size distribution ("filler effect").

In this work, we examine the influence of the partial replacement of metakaolin by different amounts of calcium carbonate

on the characteristics and performance of metakaolin-based geopolymers in both fresh and hardened states. These materials were elaborated in a strongly basic medium and the maximum of substitution rate was 12 wt%.

1.1. Materials and experimental methods

1.1.1. Starting materials

A number of starting materials were used for the elaboration of the geopolymers:

- Sodium and potassium hydroxides from Cadilhac Company, of purity 98.5% and 90%, respectively.
- alkaline solution of sodium silicate from Cadilhac Company with 45 wt% of dry matter, SiO₂/Al₂O₃ molar ratio of 2 ± 0.1 and density of 1.54.
- commercial kaolin powder from Sibelco Company, UK.
- commercial calcium carbonate from Janssen Chimica Company, of purity 98.5%.

1.1.2. Characterization methods

The investigation of the physical and structural characteristics of the materials requires the implementation of several techniques:

- X-ray fluorescence (Magixpro, Panalytical) (see the results in Table 1) was used to determine the quantitative chemical composition of the kaolin, and X-ray diffraction (Panalytical X'Pert Pro) (Fig. 2) was applied to the mineralogical compositions analyses of the kaolin and the calcium carbonate.
- Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) methods were carried out using a SHIMADZU instrument (DTG-60 H) at heating rate of 5 °C/min.
- Haak Rheostress type I rheometer in the plane-plane configuration (PP60Ti) was used to determine the evolution of the viscosity of the mixtures from the first moments of the polycondensation in order to study the setting as a function of temperature (between 30 °C and 60 °C) and the geopolymers composition. Mixtures were stirred for two minutes before being injected into the rheometer.
- Itron type 5566 universal testing machine equipped with a 10 kN load cell was used for the mechanical characterization of samples aged 28 days at a cross-head displacement rate of 1mm/min. Three-point bending tests were performed on 2 × 2 × 8 cm³ specimens) while for the compression tests the samples dimensions were of 2 × 2 × 3 cm³.
- Clemex type apparatus with 20 kN capacity tester was used for Vickers hardness assessment.
- Carl Zeiss Supra 55 type scanning electron microscope (SEM) served for microstructure observation of gold-sputtered specimen surfaces.
- Vertex 70 FTIR spectrometer operating in the spectral range 400 cm⁻¹–4000 cm⁻¹ in the Attenuated Total Reflection (ATR) mode was used for Fourier transform infrared spectroscopy (FTIR) analyses. This is considered to be an appropriate method to study the structural evolution of amorphous aluminosilicates exhibiting high heterogeneity [15]. Indeed, analyses of infrared absorption bands enable identifying specific molecular components and structures.

Table 1
Quantitative chemical composition of the kaolin oxides in wt%.

Oxyde	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	P ₂ O ₅	MnO	SO ₃	TiO ₂	P.A.F.
wt%	47.5	35.49	0.89	0.43	0.33	2.15	0.09	0.05	0.01	0.02	0.18	12.6

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