



Research paper

Investigation of the relationship between the condensed structure and the chemically bonded water content in the poly(sialate-siloxo) network



Sorelle J.K. Melele^a, Hervé K. Tchakouté^{a,b,*}, Charles Banenzoué^c, Elie Kamseu^{d,e},
Claus H. Rüscher^b, Fernanda Andreola^e, Cristina Leonelli^e

^a Laboratory of Applied Inorganic Chemistry, University of Yaounde I, Faculty of Science, Department of Inorganic Chemistry, P.O. Box 812, Yaounde, Cameroon

^b Institut für Mineralogie, Leibniz Universität Hannover, Callinstrasse 3, D-30167 Hannover, Germany

^c The University of Douala, Faculty of Science, P.O. Box. 24157, Douala, Cameroon

^d Local Materials Promotion Authority, P.O. Box 2396, Nkolbikok, Yaoundé, Cameroon

^e Department of Engineering “Enzo Ferrari”, University of Modena and Reggio Emilia Via Vivarelli 10, 41125 Modena, Italy

ARTICLE INFO

Keywords:

Metakaolin
Hardeners
Geopolymer cement
Compressive strength
Chemically bonded water
Condensed structure

ABSTRACT

The main objective of this work was to investigate the relationship between the condensed structure and the chemically bonded water content in the metakaolin-based geopolymer network. The kaolin used in this work as an aluminosilicate source was transformed to metakaolin by calcination at 700 °C. The powder of the waste glass and the silica fume were used as silica sources for the synthesis of the hardeners, dissolving with caustic soda solution. The obtained hardeners were characterized by infrared spectroscopy and MAS-NMR ²⁹Si. The metakaolin and the hardeners were used for producing geopolymer cements. The synthesized products were characterized by X-ray diffractometry, infrared spectroscopy, mercury intrusion porosimetry, scanning electron microscopy, MAS-NMR ²⁹Si and ²⁷Al, thermal analyses (TG and DSC) and compressive strength. The results show that the compressive strength of geopolymer cements using hardener from silica fume and the one from waste glass are 61.93 and 25.51 MPa, respectively. The microstructure (SEM observations) of geopolymer cements obtained using hardener from silica fume is homogeneous, compact and dense with an average pore diameter around 13 nm. Whereas, the one obtained using hardener from waste glass are heterogeneous and contains larger pores (with average pore diameter around 168 nm). MAS-NMR ²⁹Si and ²⁷Al results show that the specimen obtained using hardener from the silica fume contains more aluminum in four-fold coordination in its network than the ones using hardener from waste glass. This indicates a higher degree of crosslinking of poly(sialate-siloxo) chains which could lead to a smaller pore sizes and a higher water uptake in the structure of the sample. The amount of chemically bonded water contained in the network of geopolymer cements using hardeners from waste glass and silica fume were 6.82 and 11.23%, respectively, as determined from weight loss in the range 100–300 °C. All these results indicate that the higher content of chemically bonded water in the network of geopolymer cement obtained using hardener from silica fume is related to the much smaller average pore size diameter and the hydrophilic character of aluminum, which reveals obviously better mechanical and microstructural properties of the specimen. This could indicate here a higher degree of condensation using silica fume based hardener for geopolymerization.

1. Introduction

The term geopolymer was introduced in the early 1970s by Joseph Davidovits. This inorganic polymeric material is synthesized using different aluminosilicate sources (such as metakaolin, volcanic ash, fly ash, slag, etc.) with a hardener so called monomer solution. Geopolymers consist of repeating siloxonate $-(\text{Na}, \text{K}, \text{Ca})-(\text{Si}-\text{O}-\text{Si}-\text{O}-)$ or sialate $-(\text{Na}, \text{K}, \text{Ca})-(\text{Si}-\text{O}-\text{Al}-\text{O}-)$ units

(oligomers), polycondensed into typically ceramic, covalently bounded, non-crystalline (amorphous) 3D networks. Further research widened their definition by adding ferro-sialate and aluminophosphate oligomers, as well as acidic (using phosphoric or humic acids as chemical reagent) geopolymerization routes. The empirical formula used to describe the inorganic polymer is $\text{M}_n\{-(\text{SiO}_2)_z-\text{AlO}_2\}_n \cdot w\text{H}_2\text{O}$, wherein z is 1, 2, or 3; M is a monovalent cation (such as Na^+ or K^+); n is the degree of polycondensation and w is the water content (Davidovits,

* Corresponding author at: Laboratory of Applied Inorganic Chemistry, University of Yaounde I, Faculty of Science, Department of Inorganic Chemistry, P.O. Box 812, Yaounde, Cameroon.

E-mail addresses: htchak@yahoo.fr, hervetchakoute@gmail.com (H.K. Tchakouté).

<https://doi.org/10.1016/j.clay.2018.01.029>

Received 9 October 2017; Received in revised form 8 January 2018; Accepted 24 January 2018
0169-1317/ © 2018 Elsevier B.V. All rights reserved.

2011). During the geopolymerization reaction, water is gradually separated and the tetrahedral units are alternately bonded together by oxygen atoms thus forming a semi-crystalline geopolymer network. Although the effects of water have already been mentioned by some authors (Van Jaarsveld et al., 2002; Hongling et al., 2005; Perera et al., 2007), the investigation was not sufficient. Water is involved in the hydrolysis and polycondensation reactions during the synthesis of geopolymers (Zuhua et al., 2009) and is present after hardening as free or interstitial water, chemically and physically bound water and a hydroxyl group, OH (Davidovits, 2011). Zuhua et al. (2009) studied the role of water in the synthesis of calcined kaolin-based geopolymer. They concluded that water plays an important role in the whole process of geopolymer synthesis. Especially chemically bonded water is necessary to keep the strength stable. Fang and Kayali (2013) investigated the fate of water in fly ash-based geopolymers and they reported that the fact that minerals and non-evaporable (chemically bonded water) still exist in a geopolymer even at the high temperature suggests that the fly ash-based geopolymer could be a good fire resistant material. Other authors such as Temuujin et al. (2012) and Tchakouté et al. (2016a, 2016b) reported that a more condensed geopolymers structure contains structural water (called chemically bonded water) in their cavities, while in a less condensed structure, water is present mostly as free water and released during drying and provokes more pores and voids in the matrix. According to Davidovits (2011), water plays a critical role during geopolymerization because a part of water generated during polycondensation remains within the tridimensional polymeric frameworks. However, the investigation of the relationship between the amount of chemically bound water and the condensed geopolymers structure has not been done. Considering the above, the investigation in deep of the relationship between the more condensed geopolymer structure and the chemically bonded water content in the network of geopolymer cement could be interesting. The obtained geopolymer cements were characterized by mechanical testing, scanning electron microscope (SEM), X-ray diffractometry (XRD), infrared spectroscopy (IR), thermogravimetric and differential scanning calorimetry analysis (TG and DSC), Mercury Intrusion Porosimetry (MIP) and MAS-NMR ^{29}Si and ^{27}Al .

2. Materials and experimental methods

2.1. Materials

Kaolin (K) from Dibamba in the Littoral Region of Cameroon was used as an aluminosilicate source in this work. Waste glass bottles as a low-value silica-rich waste were collected from garbage cans and broken it into pieces. Kaolin and the resulting pieces of waste glass were separately crushed in a ball mill (MGS Sarl) with a porcelain jar and microspheres of high-grade alumina as grinding medium. The resulting powders were denoted as Dib1 and WG, respectively. The powder of kaolin was calcined in a programmable electric furnace (Nabertherm, Mod_LH 60/14) for 4 h at a heating/cooling rate of 5 °C/min at 700 °C to obtain metakaolin (MK). Commercial silica fume, $\text{SiO}_2 \cdot x\text{H}_2\text{O}$ (Merck, No 10 279-57-9) and the powder of waste glass were used as silica sources to prepare the alkaline hardeners. NaOH pellets were supplied by Merck (KGaA, 64271 Darmstadt, Germany). The physico-chemical characterization of kaolin was already studied by Elimbi et al. (2011). Metakaolin, waste glass and silica fume were already studied by Tchakouté et al. (2016b), Tchakouté and Rüscher (2017). The chemical composition of kaolin and waste glass were given in Table 1. Figs. 1 and 2 show the X-ray pattern and thermal analysis (TG/DSC) of kaolin (K), respectively.

2.2. Experimental methods

2.2.1. Methods of characterization of silica fume and waste glass

The specific surface area of the powders of waste glass and silica

Table 1
Chemical composition (mass%) of kaolin (Dib1) and waste glass (WG).

Oxide	Dib1	WG
SiO_2	54.50	68.70
Al_2O_3	27.40	1.90
Fe_2O_3	2.55	0.56
K_2O	0.28	0.75
TiO_2	2.21	< 0.1
MgO	0.09	2.33
Na_2O	< 0.1	12.6
CaO	0.10	14.30
SO_3	< 0.02	/
P_2O_5	0.079	/
ZnO	/	/
MnO	/	< 0.1
Rb_2O	/	/
SrO	/	/
ZrO_2	/	/
LOI	11.19	0.55

LOI: Loss on ignition.

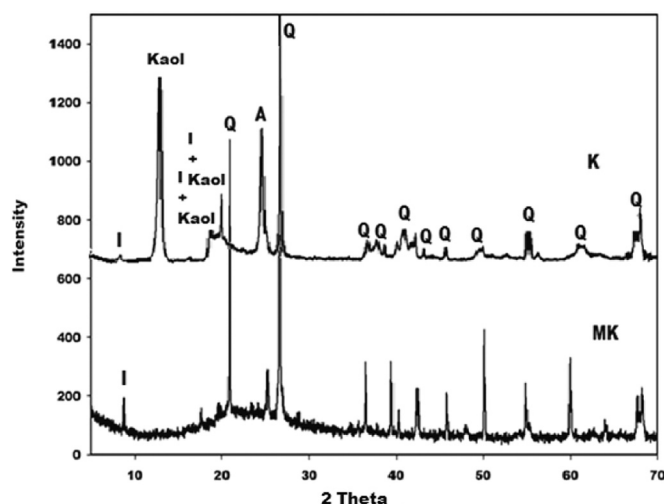


Fig. 1. X-ray pattern of kaolin (K) and metakaolin, MK. I, Q, Kaol and A denote reflections of illite, quartz, kaolinite and anatase, respectively.

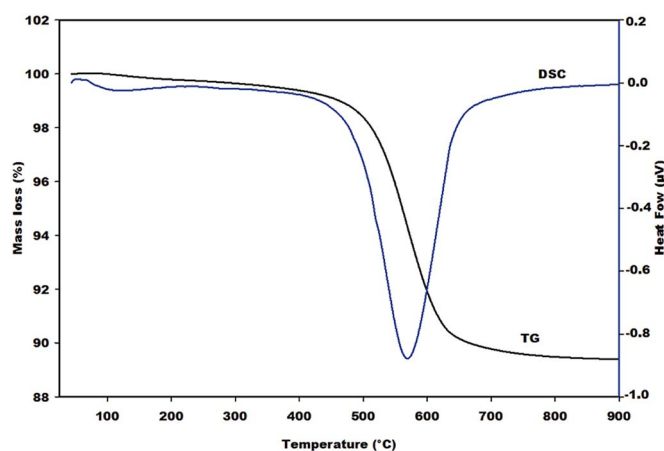


Fig. 2. TG and DSC of kaolin (K).

fume were determined using the Brunauer–Emmett–Teller (BET) method. It was performed via nitrogen adsorption using an automatic homemade apparatus.

Download English Version:

<https://daneshyari.com/en/article/8046081>

Download Persian Version:

<https://daneshyari.com/article/8046081>

[Daneshyari.com](https://daneshyari.com)