



Research paper

Hydrated lime/potassium carbonate as alkaline activating mixture to produce kaolinitic clay based inorganic polymer

Muayad Esaifan^a, Hani Khoury^{b,*}, Islam Aldabsheh^b, Hubert Rahier^c, Mohammed Hourani^a, Jan Wastiels^d^a Department of Chemistry, The University of Jordan, Amman, Jordan^b Department of Geology, The University of Jordan, Amman, Jordan^c Department of Physical Chemistry and Polymer Science, Vrije Universiteit Brussel (VUB), Brussels, Belgium^d Department of Mechanics of Materials and Constructions, Vrije Universiteit Brussel (VUB), Brussels, Belgium

ARTICLE INFO

Article history:

Received 28 December 2015

Received in revised form 20 March 2016

Accepted 21 March 2016

Available online 1 April 2016

Keywords:

Alkali-activation

Hydrated Lime

Potassium carbonate

Inorganic polymer

Kaolinite

Building material

ABSTRACT

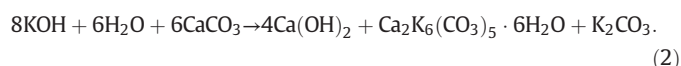
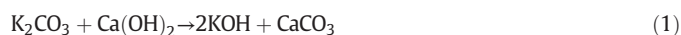
The stability as a function of storage time of an alkaline mixture developed by reacting hydrated lime ($\text{Ca}(\text{OH})_2$) with a solution of potassium carbonate (K_2CO_3) has been studied. The chemical and microstructure of alkali-activated kaolinite using the developed alkaline mixture were studied using XRD, ATR-FTIR, TGA and EDX-SEM. Over storage time of 24 h, the developed alkaline mixture was always composed of solid and liquid fractions. The solid fraction was characterized as a mixture of hydrated lime, butschliite ($\text{Ca}_2\text{K}_6(\text{CO}_3)_5 \cdot 6\text{H}_2\text{O}$) and calcite (CaCO_3), and the liquid fraction as a solution saturated with K^+ ions in a highly alkaline environment (OH^-). The inorganic polymer was dominated by hydrous phase of kaliophilite ($\text{K}_2\text{Si}_2\text{Al}_2\text{O}_8 \cdot 3\text{H}_2\text{O}$), hydrated lime, butschliite and calcite. Different mixing procedures of water, hydrated lime and potassium carbonate with kaolinitic clay were used to produce an inorganic polymer, whose strength and stability were evaluated in terms of unconfined compressive strength under dry and saturated conditions. A maximum dry strength of 39 MPa and a maximum soaked and cycled strength of 11 MPa were obtained. A low cost-environment friendly alkaline mixture was developed to produce kaolinitic clay based inorganic polymer for construction and industrial applications.

© 2016 Elsevier B.V. All rights reserved.

1. Introduction

Inorganic polymers (Duxson et al., 2007; Rahier et al., 1996) and geopolymers in particular (Davidovits, 1991) have been investigated intensively in last decades as alternative and green building materials in terms of low CO_2 emission, mechanical strength and durability (Bernal and Provis, 2014; Davidovits, 1991; Duxson et al., 2007; Esaifan et al., 2015; Pacheco-Torgal et al., 2008; Rahier et al., 1996). The class of inorganic polymers studied here is synthesized by alkali-activation of reactive aluminosilicate sources, starting from naturally abundant materials (e. g. kaolinite, volcanic tuff, etc.) (Esaifan et al., 2015; Lemoungna et al., 2013; Slaty et al., 2013), and even waste or by-products (e.g. fly ash, blast furnace slag, etc.) (Wastiels et al., 1994; Song et al., 2000). The most used alkaline activating solutions are based on chemicals such as NaOH, KOH, and potassium or sodium silicate solutions (Pacheco-Torgal et al., 2008; Slaty et al., 2013). These chemicals are expensive and aggressive when manipulated. This is one of the reasons for

the slow commercial breakthrough of inorganic polymers. The use of $\text{Ca}(\text{OH})_2$ instead of KOH or NaOH solutions could solve some safety issues. However, the use of pure $\text{Ca}(\text{OH})_2$ results in poor mechanical properties if the aluminosilicate source has no pozzolanic properties (Gogo, 1990; Shi et al., 2006). In early work, Gogo (1990) obtained improved mechanical properties with addition of K_2CO_3 , but the results were not published in scientific literature. Only limited information is available about the system $\text{K}_2\text{CO}_3/\text{Ca}(\text{OH})_2/\text{H}_2\text{O}$ (Gogo, 1990). The idea behind mixing K_2CO_3 and $\text{Ca}(\text{OH})_2$ with the addition of water, is the partial dissolution of $\text{Ca}(\text{OH})_2$ and release of OH^- . Gogo (1990) suggested that hydrolysis and precipitation reactions occur in the $\text{K}_2\text{CO}_3/\text{Ca}(\text{OH})_2/\text{H}_2\text{O}$ system. A double salt, named butschliite (Milton and Axelrod, 1947; Mrose et al., 1966; Pabst, 1974), was formed next to CaCO_3 according to the following reactions:



In this paper, first a more in depth study is carried out to better understand which reactions take place in the $\text{K}_2\text{CO}_3/\text{Ca}(\text{OH})_2/\text{H}_2\text{O}$ system. The aim is to study the chemical composition and stability of

* Corresponding author.

E-mail addresses: mu.esaifan@ju.edu.jo (M. Esaifan), khouryhn@ju.edu.jo (H. Khoury), islam.aldabsheh@gmail.com (I. Aldabsheh), hrahier@vub.ac.be (H. Rahier), mhourani@ju.edu.jo (M. Hourani), jan.wastiels@vub.ac.be (J. Wastiels).

the reaction products as a function of time. The use of a matured mixture is compared to the direct addition of one or both of the solid components to kaolinite before adding the other component dissolved in water. The influence of the mixing procedure on the inorganic polymer properties was investigated in terms of unconfined compressive strength under dry and saturated conditions.

2. Materials and experimental methods

2.1. Materials

Red kaolinitic clay (coded as KC) was collected from the BatnEl-Ghoul deposit. This deposit is located in the southern part of the Hashemite kingdom of Jordan. The clay geology and mineralogy of this deposit was studied in details by [Khoury and El-Sakka \(1986\)](#). A two ton gross clay sample was crushed and homogenized using a cone crusher (TRIO model TC36) equipped with micro-filters. About 95% of the particle size is $<20\ \mu\text{m}$ ([ASTM D422-63, 2007](#)). The plastic limit was determined to be 25% according to standard procedure ([ASTM D4318-05, 2005](#)). Pure kaolinite (coded as PK) was supplied by Merck KGaA (Germany), and it was used as a reference material. The chemical composition of the clay samples as determined by X-ray fluorescence (XRF) is listed in [Table 1](#).

The alkaline activating mixture was prepared using $\text{Ca}(\text{OH})_2$ powder (British Drug Houses, UK 96.0%), K_2CO_3 powder (British Drug Houses, UK, 99.0%) and demineralized water. A reference alkaline solution was prepared using KOH pellets (Merck KGaA, Germany, 98.0%). In order to facilitate the mixing and compaction of the specimens to evaluate the mechanical strength and dimensional stability (phase 2 in [Fig. 1](#)), as well as to improve their compactness and dimensional stability, silica sand (M31 supplied by SIBELCO, $D_{50} = 370\ \mu\text{m}$) was used as filler material. This nearly pure quartz sand (99.5% SiO_2) can moreover be considered as inert in the highly alkaline environment of the fresh mixture. It can also improve the fresh mixture workability, reducing drying cracks caused by drying shrinkage, and reducing the material cost without loss of strength by lowering the necessary amount of binding material.

2.2. Methodology

A schematic representation of the experimental program conducted in this investigation is shown in [Fig. 1](#). The work was divided into three phases. Phase (I) consisted of the chemical and physical stability study of the synthesized alkaline mixture, in phase (II) the mixing methodology of $\text{Ca}(\text{OH})_2$ - K_2CO_3 -clay on the strength and dimensional stability of alkali activated kaolinitic clay (KC) was studied, and finally in phase (III) the chemical and structural changes of both raw and produced materials were studied using different mass ratios of alkaline activators.

In phase (I), the alkaline mixture stability study was performed on $\text{Ca}(\text{OH})_2$ (5 M) and K_2CO_3 (5 M) concentrations assumed to produce K^+ in solution with a theoretical maximum concentration of 10 M according to the reaction stoichiometry described in [Eq. \(1\)](#). This concentration of KOH (10 M) is equivalent to the composition used to fabricate inorganic polymer specimens with KOH/Clay mass ratio of 0.14 and $\text{H}_2\text{O}/\text{clay}$ mass ratio of 0.25, which is similar to NaOH activated kaolinitic clay ([Esaifan et al., 2015; Slaty et al., 2013](#)). The alkaline mixture was synthesized by dissolving K_2CO_3 first in water, after which $\text{Ca}(\text{OH})_2$ was added to the solution ([Fig. 1](#)). It was mixed at room temperature for one hour, using a magnetic stirrer with speed

up to 1500 rpm. After about 30–40 min of mixing, the mixture had formed a white viscous gel. After finishing the mixing process, the solution was left to rest. After a few hours, the viscous gel started to separate into a precipitate and a liquid fraction. After different gel storage times (6, 12 and 24 h), the solution was filtered to separate the liquid (LF) and solid (SF) fractions using a cellulose acetate filter via a Buchner funnel attached to a vacuum pump. From the liquid fraction, a dilution of 200 times was applied to reduce the solution concentration to within the readability ranges of pH meter and atomic absorption spectrometer (AAS). The solid fraction was washed several times with distilled water after the separation, then dried at $80\ ^\circ\text{C}$ using a ventilated oven (Heraeus model TU100/150) for one day, crushed and finally characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and thermo-gravimetric analysis (TGA).

The best mixing methodology of alkali-activated inorganic polymer components (phase II) was selected based on mechanical strength and dimensional stability tests. Seven inorganic polymer mixes were prepared as described in [Table 2](#) (mixes 1 to 7). $\text{Ca}(\text{OH})_2$ (mix 1), K_2CO_3 (mix 2) and KOH (mix 3) were mixed separately in water and then applied as single alkaline additive for comparison. The fourth mix was prepared by mixing $\text{Ca}(\text{OH})_2$ and K_2CO_3 as solid additives with clay and silica sand, then water was added to the solid additives and mixed together. In the fifth mix, K_2CO_3 was premixed as solid additive with dry sand and clay, then $\text{Ca}(\text{OH})_2$ was mixed with water and added to the premix. In the sixth mix, $\text{Ca}(\text{OH})_2$ was premixed as solid additive with dry sand and clay. K_2CO_3 was dissolved completely in water, and added to the premix. For the last mix (mix 7), K_2CO_3 was dissolved completely in water, after which $\text{Ca}(\text{OH})_2$ was added to the solution and mixed for one hour, then added immediately to the sand and clay. All seven batches were mixed using a Hobart planetary mixer (model A200) at 198 rpm for a total period of 15 min. After the mixing process, each batch was divided into nine specimens (about 50 g each) and molded immediately in a stainless steel cylinder (internal diameter 25 mm, height 150 mm) with a plunger at a pressure of 15 MPa. The demolded specimens were cured for 24 h at $80\ ^\circ\text{C}$ in a ventilated oven. This mixing and curing procedure led to optimal results in previous studies for the activation of kaolinite with NaOH ([Esaifan et al., 2015; Slaty et al., 2013](#)). After curing, the specimens were divided into three groups of three specimens each. Group 1 specimens were dried in a ventilated oven at $40\ ^\circ\text{C}$ for seven days in order to avoid uncontrolled absorption of air humidity, after which the unconfined compressive strength test was conducted to evaluate the dry strength (S_{dry}). Group 2 specimens were immersed in demineralized water at room temperature for seven days, after which the stability of the specimens was evaluated by measuring the soaked strength (S_{soaked}). Group 3 specimens were subjected to three immersions/drying cycles, being one day immersed in demineralized water at room temperature, followed by drying for one day at $40\ ^\circ\text{C}$. Finally these specimens were kept immersed for one more day before evaluating their durability by measuring the residual soaked strength (S_{cycle}).

In phase (III), four groups of batches were prepared for the mineralogical and microstructural studies of the alkali-activated kaolinitic clay, as listed in [Table 2](#) (mixes 8 to 23). The first group of batches (mixes 8 to 11) used pure kaolinite (PK) activated with different mass ratios of KOH/clay. The second group of batches (mixes 12 to 15) used pure kaolinite activated using $\text{Ca}(\text{OH})_2/\text{clay}$ and $\text{K}_2\text{CO}_3/\text{clay}$ mass ratios assumed to produce mass ratios of KOH/clay equivalent to 0.14, 0.30, 0.40 and 0.50, according to the stoichiometry described in [Eq. \(1\)](#). These batches were used as model system. Except for the first mix,

Table 1
Chemical composition (wt.%) of the raw materials.

Material	Al_2O_3	SiO_2	CaO	Fe_2O_3	K_2O	MgO	MnO	TiO_2	Na_2O	L.O.I	Total
KC	28.1	50.4	0.3	5.9	1.2	0.7	0.2	0.8	0.2	11.9	99.7
PK	36.1	47.8	0.1	0.8	1.4	0.2	0.3	1.2	0.1	11.6	99.5

Download English Version:

<https://daneshyari.com/en/article/1694067>

Download Persian Version:

<https://daneshyari.com/article/1694067>

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