



Research paper

An investigation on pozzolanic activity of Iranian kaolins obtained by thermal treatment

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ARTICLE INFO

Article history:

Received 14 May 2014

Received in revised form 29 October 2014

Accepted 3 November 2014

Available online 20 November 2014

Keywords:

Kaolin

Metakaolin

Thermal treatment

Pozzolanic activity

ABSTRACT

Use of metakaolin as a cementitious supplementary material has found interesting applications during recent years. This interest is in part due to the technical advantages and related to environmental and energy issues, particularly in CO₂ gas emission challenges in another part. However, kaolins that have effective pozzolanic activity and meet the desired requirements are still being studied. Current research aims to investigate calcination of two types (from Zonouz and Khorasan districts, Iran) of kaolin at temperatures of 600–850 °C for production of metakaolin with pozzolanic activity. The samples were analyzed using Particle Size Distribution (PSD), BET, DTA/TG, XRD, SEM, and FTIR techniques to specify the mineralogical composition and extent of de-hydroxylation. Pozzolanic activity of the resulting calcined samples were evaluated by determination of the residual quantity of Ca(OH)₂ via STA method on hydrated mixtures of 50% metakaolin and 50% calcium hydroxide. Then, optimal parameters in calcination of kaolin to metakaolin were calculated for each of the two samples. The calcination temperature and time for conversion of kaolin to metakaolin in the Zonouz and Khorasan samples were found to be 700 °C and 800 °C, respectively, with a holding time of 3 h. Content of the pozzolanic activity for Zonouz metakaolin was obtained as 77.5%, while that for the Khorasan metakaolin could even hardly reach 55.0%.

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1. Introduction

Development of construction materials with marvelous technical and environmental advantages is one the main challenges of the new millennium. Cement as an important development industry was unfortunately known to be responsible for 5–8% of the emissions of carbon dioxide gas in the world [Scrivener and Kirkpatrick, 2007; Rashad, 2013]. The cement industry not only released carbon dioxide gas, but also generated SO₃ and NO_x gases which caused the greenhouse effect and incurred serious environmental impacts [Sang-Sook and Hwa-Young, 2008; Rashad and Sayieda, 2011]. Metakaolin and other supplementary cementitious materials were found to be useful as a partial substitute for cement or a new source of cement-free materials [Rashad, 2013]. Beside its advantageous environmental contributions, metakaolin has improved mechanical properties and durability compared to mortar and concrete [Siddique and Klaus, 2009]. Therefore study of metakaolin has raised a lot of interests in recent years.

Kaolinite is the mineralogical term for hydrated aluminum disilicate (Al₂Si₂O₅(OH)₄) and the main component of kaolin. Kaolinite is reported

to possess a two-layered structure in which a sheet of octahedrally coordinated aluminum is connected to a tetrahedrally coordinated silicon sheet [Wypych and Satyanarayana, 2004; Vizcayno et al., 2010]. Thermal treatment or calcination break down the structure of kaolin such that the alumina and silica layers become puckered and lost their long-range order. Resulting from this dehydroxylation and disorder is metakaolin, a highly reactive transition-phase, amorphous material with pozzolanic and latent hydraulic reactivity, suitable for use in cementing applications [Bensted and Barnes, 2002]. At ambient temperature, metakaolin reacts with Ca(OH)₂ in the presence of water and produces a cementing compound like C-S-H and alumina-containing phases including C₄AH₁₃, C₃AH₆ and C₂ASH₈ [Tironi et al., 2012a,b]. The crystalline products are mainly dependent on the ratio of metakaolin to calcium hydroxide and reaction temperature [Siddique and Klaus, 2009].

Optimal calcination temperature, which leads to pozzolanic characteristics and reactivity of the metakaolin, is reported differently by various researchers. However, they all address the range of 600–850 °C considering the type of kaolin [Bich et al., 2009; Tironi et al., 2012a,b; Morsy et al., 2012; Ramezani-pour and Bahrami, 2012]. Heating at higher temperatures causes recrystallization and formation of mullite or spinel and hence loss of pozzolanicity [Badogiannis et al., 2005;

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Tironi et al., 2012a,b]. The key point in the production of metakaolin for use as a supplementary cementing material is to achieve as much dehydroxylation as possible without over-heating [Sedigh, 2008].

The purpose of the current study was to determine the optimal calcination parameters of two types of Iranian kaolins for producing metakaolin as a supplementary cementitious material and also for showing their pozzolanic activity.

2. Experimental

2.1. Raw materials and calcination

Two types of Iranian kaolins, namely Zonouz (North west, over 32 million tons deposit of clayey ore beds) kaolin (KZ) and Khorasan (North east, over 3 million tons deposit of clayey ore beds) kaolin (KK) were used as raw materials in this study. The materials' chemical and mineralogical compositions, particle size distribution and the specific surface area (SSA) are listed in Table 1.

For calcination of the kaolins, 50 g from each material were put in an alumina crucible and were then thermally treated by a laboratory furnace at temperatures of 600, 650, 700, 750, 800, and 850 °C for 3 h. After thermal treatment, the samples were cooled down to room temperature at ambient conditions.

2.2. Characterization

2.2.1. Chemical composition

The chemical composition of the kaolins was determined by the XRF-fused cast-bead method.

2.2.2. Particle size distributions and specific surface area (BET)

The particle size distributions (PSD) were measured in water suspension using a laser particle sizer (Fritsch Analysette 22). The Brunauer-Emmet-Teller (BET) specific surface area (SSA) of the samples was determined with an analyzer (Micromeritics Gemini 2375 V4) using nitrogen gas at liquid nitrogen temperature. Note that in order to make sure the powders were well dried and outgassed, they were kept in the BET tubes under nitrogen gas for 16 h at 80 °C temperature before the measurement.

2.2.3. Thermal analysis

Thermal behavior of the starting kaolins including thermal gravimetry and differential thermal analysis (TG-DTA) were examined using an analyzer (Netzsch STA 409). The samples were heated at a constant rate of 10 °C/min from ambient temperature to 1050 °C.

2.2.4. Phase analysis and SEM analysis

Mineral compositions of the starting and thermally treated kaolins were analyzed using X-ray diffractometry (Philips PW 1800). Microstructure of the samples was analyzed with a scanning electron microscope (SEM, Philips XLF 30 FEG). The accelerating voltage was set to 15 kV and the samples were coated with carbon.

2.2.5. FTIR spectrophotometry

A Fourier transform infrared spectroscopy (FTIR) apparatus (Spectrum 400) was used to confirm the presence of kaolinite specific bands in the raw samples or absence of these bands in the thermally treated kaolin samples.

2.3. Pozzolanic activity of metakaolins

The pozzolanic activity of the metakaolin samples was determined according to un-reacted calcium hydroxide using thermal analysis (Netzsch STA-449 C Jupiter). A paste with an appropriate concentration was prepared from a mixture of 50% metakaolin and 50% Ca(OH)₂, poured in a cylindrical container, and made completely sealed and air-tight. The samples were treated in a dryer at 50 °C temperature for seven days. Afterwards, the samples were pulverized and dried in a nitrogen atmosphere. The powders were exposed to thermal treatment up to 1000 °C at a heating rate of 10 °C/min and their DTG and TG curves were recorded. After measuring the content of ignition loss of the endothermic reaction around 450–550 °C, referable to the dehydroxylation of Ca(OH)₂, this value was multiplied in the molecular ratio between Ca(OH)₂ and H₂O in order to obtain the amount of un-reacted calcium hydroxide.

3. Results and discussion

3.1. Material characterization

The chemical composition of the samples from XRF analysis summarized in Table 1 allows easy evaluation of the grade of the kaolins. A maximum kaolinite content was estimated by comparison to the theoretical composition of the kaolinite mineral (Al₂Si₂O₅(OH)₄). Maximum kaolinite contents for KZ and KK were 57 and 46%, respectively. KZ and KK can therefore be considered as low-grade products, containing mainly SiO₂ as a secondary component.

Table 1 shows that by thermal treatment the BET of both kaolins decreased compared to their untreated state; in KZ the BET changed from 8.1 m²/g for the untreated sample to 6.5 m²/g after thermal treatment and in the case of KK from 7.3 m²/g to 5.6 m²/g. The reason for this decrease is likely the sintering and particle agglomeration occurring at the temperature of thermal activation.

3.2. Thermal behavior of kaolins

The thermal behavior of KZ and KK samples is presented in Fig. 1. The most significant changes according to TG and DTA analysis for starting samples are summarized below:

The DTA pattern of KZ (Fig. 1-a) showed an endothermic peak in the temperature range 450–600 °C, which was associated with de-hydroxylation and formation of the metakaolin phase.

The center of the endothermic peak was located at 538 °C for the KZ sample, while for the KK sample the endothermic peak of metakaolin formation was located in the temperature range 500–650 °C and its center was located at approximately 545 °C. Position and content of the hydroxyl groups differ considerably from one structure to another. For kaolinite, most of the hydroxide groups were located on the inter-layer surface of the octahedral sheet, while for illite most of these hydroxyl groups were located in the core of a layer between two tetrahedral sheets. Therefore, kaolinite used a greater number of OH groups in comparison with the other clay minerals [Fernandez et al., 2011]. In

Table 1

Chemical and mineralogical composition, average particle size and specific surface area of materials.

Oxides, (mass%)	KZ	KK	Phase (mass%)	KZ	KK
SiO ₂	63.39	64.81	Kaolinite	57	46
Al ₂ O ₃	24.10	24.11	Illite	7	13
Fe ₂ O ₃	0.45	0.23	Quartz	33	39
CaO	0.93	0.54	Other	3	2
MgO	0.25	0.07			
Na ₂ O	0.31	0.18			
K ₂ O	0.34	1.48			
TiO ₂	0.03	0.29			
SO ₃	/	0.19			
L.O.I.	10.20	8.10			
d ₅₀ (μm)	3.82	5.40			
BET (m ² /g)					
Starting	8.1	7.3			
Thermally treated	6.5	5.6			

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