



## Research paper

# Effects of mechanical and thermal activation on pozzolanic activity of kaolin containing mica



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## ABSTRACT

Kaolin from a Serbian deposit, characterized by high content of impurities, such as mica and quartz, disordered kaolinite and high specific surface area, was subjected to thermal and mechanical activation on a large scale. Activations were carried out with a goal to investigate the effects of applied methods on the chemical, structural and morphological changes that occurred in the kaolin, and their influence on the pozzolanic activity. The changes were monitored using XRD, FTIR, TG/DTA, PSD, BET and SEM analyses. Pozzolanic activity was evaluated by determination of the 7-day compressive strength conducted on the lime mortars.

The results showed that mechanical activation by milling the kaolin for 20 h in the conventional horizontal ball mill was competitive for obtaining pozzolana as thermal activation. Pozzolanic activity of thermally activated kaolin was mainly influenced by formation of metakaolin. Combined effects (such as significant specific surface area increase, as well as partial kaolinite and complete mica phase amorphization) were dominant in pozzolanic activity of mechanically activated kaolin.

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## 1. Introduction

In recent decades, environmental considerations and energy efficiency requirements have motivated researchers to focus on finding new and optimized pozzolanic material, specially intended for use in cement-based systems (Sabir et al., 2001; Schneider et al., 2011). Among the new generation of such materials, both thermally and mechanically activated kaolin, have been a subject of many studies (Vizcayno et al., 2010; Rashad, 2013; Fabbri et al., 2013; Alujas et al., 2015; Fitos et al., 2015; Hamzaoui et al., 2015; Soury et al., 2015) due to a significant variation in the composition of kaolin, leading to the increased diversity of obtained products and their technical benefits (Siddique and Klaus, 2009).

Metakaolin is produced by thermal activation (calcination) of kaolin (Sabir et al., 2001; Siddique and Klaus, 2009). Thermal activation at appropriate temperature transforms a crystalline kaolinite phase into a disordered phase (metakaolinite) through a crystal lattice failure. During the thermal activation of kaolin, two hydroxyl groups from the surface of kaolinite, join to form a water molecule leaving the remaining oxygen as a superoxide anion (Maiti and Freund, 1981; Brindley and Lemaitre, 1987; Frost and Vassallo, 1996). The instabilities caused by the anion imbalance result in crystal structure collapse (rearrangement of Si and Al atoms leading to a decrease in octahedral Al and the

appearance of penta- and tetra-coordinated Al (Rocha and Klinowski, 1990)), thus forming metastable, anhydrous aluminum silicates. Dehydroxylation and disordering of the kaolinite lead to a formation of reactive material with pozzolanic activity, i.e. the ability of materials to react with calcium hydroxide (CH) in presence of water and produce calcium silicate hydrate, stratlingite, tetracalcium aluminate hydrate and other compounds having binding properties (Oriol and Pera, 1995; Wild and Khatib, 1997; Cabrera and Rojas, 2001). Pozzolanic activity of the metakaolin depends on several factors, most important being the nature and abundance of clay minerals in the raw material, thermal activation conditions (He et al., 1994; Kostuch et al., 1996; Badogiannis et al., 2005) crystallinity of the original kaolinite (Kakali et al., 2001), and the average grain size of the metakaolin (Oliveira et al., 2005; Fabbri et al., 2013).

During the last years, a significant number of studies (Badogiannis et al., 2005; Potgieter-Vermaak and Potgieter, 2006; Gutierrez et al., 2008; Ilić et al., 2010; Fernandez et al., 2011; Said-Mansour et al., 2011; Tironi et al., 2012; Fabbri et al., 2013; Alujas et al., 2015) have been focused on optimization of thermal activation process and the influence of the process parameters (temperature and heating time) on the pozzolanic activity of obtained metakaolin. The optimum temperature and heating time necessary to obtain material with a high pozzolanic activity varies in literature (Rashad, 2013) as a result of huge differences in the kaolin composition and structure.

Although the capability of metakaolin as pozzolanic material to improve mechanical properties and durability of concrete when used as

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a partial replacement of Portland cement is well documented in literature (Sabir et al., 2001; Siddique and Klaus, 2009; Siddique and Iqbal Khan, 2011; Aiswarya et al., 2013), its utilization in building industry is quite limited, mainly due to its high price. Continuous efforts are being made to develop processes that enable the production of materials with better technical characteristics at a lower price and with a lower impact on the environment. Among them, mechanical activation attracted special attention because of the advantages it has over the traditional technological procedures (Boldyrev, 2006; Balaž, 2008).

Mechanical activation of kaolin has been often studied in recent years (Horvath et al., 2003; Vizcayno et al., 2005; Vizcayno et al., 2010; Valášková et al., 2011; Dellisanti and Valdre, 2012; Mitrović and Zdujić, 2014; Hamzaoui et al., 2015). It has been found that mechanical activation of kaolin leads to structural changes (breaking the kaolinite bonds, losing of the kaolinite surface hydroxyls and replacing them with water both coordinated and adsorbed on the surface of the octahedral sheet). As a result, a new material with significantly altered surface of kaolinite, different cation-exchange capacity, porosity and water absorption capacity, reduced crystal size and a higher specific surface area is formed (Kristof et al., 1993; Frost et al., 2001; Horvath et al., 2003).

Mechanically activated kaolin is different from thermally activated, due to the presence of coordinated water in amorphized kaolinite, formed as a result of the mechanical dehydroxylation of kaolinite. Using a high energy ball mill Hamzaoui et al. (2015) established that mechanically activated kaolinite has a similar X-ray diffraction pattern as thermally activated one.

Most studies of mechanical activation of kaolin focused on structural and morphological changes occurring during the milling. The pozzolanic activity of mechanically activated kaolinite, particularly important for its use in cement-based systems, has been investigated only in few papers (Vizcayno et al., 2010; Mitrović and Zdujić, 2014; Fitos et al., 2015; Souri et al., 2015). Reported data show that pozzolanic activity of the mechanically activated kaolinite is a function of the mineralogical composition of the raw kaolin and the milling time (Vizcayno et al., 2010; Mitrović and Zdujić, 2014) as well as the particle size (Mitrović and Zdujić, 2014) and specific surface area (Souri et al., 2015).

Comparison of thermal and mechanical activation on the large-scale milling of kaolin and its influence on the pozzolanic activity has not been reported so far. Hence, the main goal of this research is to compare structural and morphological changes that occurred in kaolin after thermal and mechanical activation and the effect they have on the pozzolanic activity of materials. In this work, kaolin from a Serbian deposit, having a significant content of impurities, such as mica and quartz, disordered kaolinite and high specific surface area, was subjected to thermal and mechanical activation on a large scale, in order to obtain materials with similar pozzolanic activity. The changes were monitored using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), thermogravimetric/differential thermal analysis (TG/DTA), particle size distribution (PSD), specific surface area (BET) and scanning electron microscopy (SEM) analyses. Pozzolanic activity of the activated samples was evaluated by determination of the 7-day compressive strength on the lime mortars.

## 2. Materials and methods

### 2.1. Properties of starting kaolin

Kaolin used in this work was collected from deposit Vrbica, Arandjelovac basin, Serbia. The kaolin (~150 kg) was dried at 105 °C until constant mass, milled 10 min in a ball mill, (capacity of 50 kg) and homogenized.

Chemical composition was determined using X-ray fluorescence spectroscopy on an Oxford ED 2000 instrument. Reactive SiO<sub>2</sub> content of the starting and activated kaolins was determined in accordance with SRPS EN 197-1 and SRPS EN 196-2 standards. Chemical composition

of starting kaolin is given in Table 1. The major oxides of starting kaolin were silica (SiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>) in total content of 80.09% (mass percent) and the kaolin also contained a small amount of impurities (K<sub>2</sub>O, Fe<sub>2</sub>O<sub>3</sub>, and TiO<sub>2</sub>).

The semi-quantitative mineralogical estimation of starting kaolin, based on the characteristic XRD peaks of each mineral, in combination with the bulk chemical analysis, showed following phase composition (in mass percent): 61% kaolinite, 16% mica, 14% quartz and 9% other phases. According to the literature (Aras et al., 2007) starting kaolin was a medium-quality raw material.

### 2.2. Kaolin activation procedures

#### 2.2.1. Thermal activation

Thermal activation of kaolin samples weighting 500 g was carried out in a laboratory furnace using a heating rate of 8 °C/min. Temperatures and times were chosen based on the results obtained in previous experiments with the kaolin from the same basin (Ilić, 2010; Ilić et al., 2010). Thermal activation was carried out at 700 °C and 750 °C with the activation time ranging from 30 to 180 min. After thermal activation, the calcined material was left in the furnace to slowly cool down to the room temperature.

#### 2.2.2. Mechanical activation

Mechanical activation was carried out in a conventional horizontal ball mill, under the same milling conditions as previously applied for another Serbian kaolin (Mitrović and Zdujić, 2014). A cylindrical steel vial of inner diameter 360 mm and height 340 mm (volume 0.0346 m<sup>3</sup>) filled with hardened steel balls of 20–60 mm diameter was used as the milling media. The total mass of the balls was 50.9 kg and the balls-to-powder mass ratio was about 10. The angular velocity of a vial was 4.8 s<sup>-1</sup> (46 rpm). In each milling run, 5 kg of kaolin was milled from 30 min to 20 h.

### 2.3. Characterization

#### 2.3.1. XRD

The XRD data were collected on a Philips PW 1710 diffractometer using Cu-Kα graphite-monochromatized radiation ( $\lambda = 1.5418 \text{ \AA}$ ) in the  $2\theta$  range 4–90° (step size: 0.02°  $2\theta$ , time per step: 0.8 s). The working conditions were 40 kV and 30 mA.

#### 2.3.2. FTIR spectroscopy

FTIR measurements were performed on a Nicolet 6700 Thermo Scientific spectrometer on the solid sample prepared using KBr pressed-disk technique over the wave number range of 4000–400 cm<sup>-1</sup>. Spectral manipulation such as baseline adjustment, smoothing, and normalization was performed using the software package OMNIC.

**Table 1**  
Chemical composition of starting kaolin.

Starting kaolin, mass %	
Loss of ignition	11.90
SiO <sub>2</sub>	49.66
Al <sub>2</sub> O <sub>3</sub>	30.43
Fe <sub>2</sub> O <sub>3</sub>	3.78
CaO	0.65
MgO	0.48
Na <sub>2</sub> O	0.10
K <sub>2</sub> O	1.90
SO <sub>3</sub>	<0.01
P <sub>2</sub> O <sub>5</sub>	0.18
TiO <sub>2</sub>	0.89
SUM	99.97
Reactive SiO <sub>2</sub>	19.52

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