EFFECT OF MECHANICAL ACTIVATION ON THE POZZOLANIC ACTIVITY OF MUSCOVITE

GENG YAO, HAOYU ZANG, JUNXIANG WANG, PENG WU, JUN QIU, AND XIANJUN LYU[*]

[1]College of Chemical and Environmental Engineering, Shandong University of Science and Technology, Qingdao 266590, China

Abstract—In order to provide a theoretical foundation for the utilization of tailings as supplementary cementitious materials, the pozzolanic activity of muscovite—a typical mineral phase in tailings—before and after mechanical activation was investigated. In this study, significant pozzolanic activity of muscovite was obtained as a result of the structural and morphological changes that were induced by mechanical activation. The activated muscovite that was obtained after mechanical activation for 160 min satisfies the requirements for use as an active supplementary cementitious material, and the main characteristics of the pozzolana were as follows: median particle size (D_{50}) of 11.7 μm, BET specific surface area of 28.82 m^2 g^{-1}, relative crystallinity of 14.99%, and pozzolanic activity index of 94.36%. Continuous grinding led to a gradual reduction in the relative crystallinity and an increase in the pozzolanic activity index due to the dehydroxylation reaction induced by mechanical activation, which occurred despite the fact that the specific surface area showed a decreasing trend when the grinding time was prolonged. Mechanically activated muscovite exhibited the capacity to react with calcium hydroxide to form calcium silicate hydrate, which is a typical characteristic of pozzolana. This experimental study provided a theoretical basis for evaluating the pozzolanic activity of muscovite using mechanical activation.

Keywords—Mechanical activation · Muscovite · Pozzolanic activity · Supplementary Cementitious Material

INTRODUCTION

As a widely used building material, with a life-cycle encompassing all production processes, uses, and scrappage, cement-based materials have a significant effect on the ecological environment (Ludwig & Zhang 2015; Shi et al. 2011). In recent decades, the need to protect the ecological environment and promote sustainable development has motivated researchers to focus on finding new supplementary cementing materials to reduce cement consumption (Lothenbach et al. 2011; Vargas & Lopez 2018). Supplementary materials such as blast-furnace slag, which is a calcium silicate-based by-product obtained using the cold quenching technology of the iron-making industry, or fly ash from coal-fired electricity generation are widely used in blended cements that are more environmentally friendly and resource-efficient (Alujas et al. 2015; Paris et al. 2016; Yüksel 2018). The global demand for Portland cement is expected to increase for the foreseeable future, while the production of coal fly ash, the most commonly used pozzolanic addition, is expected to decrease (Price 2016). Considering the environmental, feasibility, and economic aspects, the use of new and optimized pozzolanic materials as supplementary cementing materials is of great engineering significance and socioeconomic benefit.

In recent years, almost all mineral-producing countries have faced issues with environmental pollution from the accumulation of mine tailings and the need for rational management strategies. Mine tailings, which are among the most abundant industrial solid wastes, are a potential pozzolanic material, as their main chemical composition is similar to that of cement (Bian et al. 2012). Most previous studies on pozzolanic materials have focused on amorphous glassy materials. From a traditional perspective, however, mine tailings cannot react with an alkali activator to form hydration products with gelatinous properties,

because they are a stable crystalline material at room temperature (Argane et al. 2015). Thus, researchers are examining ways to pretreat mine tailings to better utilize them as an active pozzolanic material. Knowledge of reactive activation of crystalline tailings has been improved, and the potential use of this solid waste as a supplementary cementing material has drawn the attention of researchers around the world (Moura et al. 2007; Ozlem et al. 2006). Thermal activation of solid waste is a complex process, involving significant energy consumption and difficult operations. Mechanical activation by grinding, an important pretreatment method for mineral activation, is a very effective approach for improving the pozzolanic activity of mine tailings. In their work regarding the effects of mechanical activation on the pozzolanic activity of gold ore tailings, Yao et al. (2019) found that the pozzolanic activity of the tailings was improved during the grinding process, and the mineral-phase muscovite in gold ore tailings had positive effects on the pozzolanic activity. Mechanical activation uses high-impact-friction materials to produce a more reactive product with a disordered structure in short periods of time. In addition, to separate a variety of useful minerals from the ore in the dissociation of particles, the ore must be ground to a certain fineness to meet the requirements for mineral processing, and thus the mineral processing itself is a mechanical activation process.

The use of mine tailings in construction materials has attracted much attention; however, studying the pozzolanic activity of mine tailings is rather difficult given their relatively complicated mineral phases. Although previous studies have proven that clay minerals, such as the muscovite and kaolinite in mine tailings, positively influence pozzolanic activity (Li et al. 2010a, 2010b), to date no systematic research has been conducted on the different mineral phases in tailings. To offer a theoretical foundation for the utilization of mine tailings as supplementary cementing materials, illustrating clearly the pozzolanic activity of the different mineral phases in tailings is important. For these reasons, the investigation of the effects of mechanical

* E-mail address of corresponding author: lyuxianjun@163.com

activation on the pozzolanic activity of muscovite—a common mineral phase in mine tailings—was necessary.

A previous study (Vizcayno et al. 2010) of supplementary cementitious material used in cement assessed the effects of mechanical and thermal activation on the pozzolanic activity of kaolin containing mica using a Herzog oscillating mill. The researchers found that the mechanical activation process increased not only the surface area of the kaolinite clays, but also their pozzolanic reaction activity; the pozzolanic activity index of activated kaolinite clays was dependent on the mineralogical composition of the raw materials and their grinding time. A similar study (Ilić et al. 2016) reported that mechanical activation of kaolin by dry grinding for 20 h in a conventional horizontal ball mill was as effective for obtaining pozzolana as thermal activation.

Although some studies have investigated the effects of mechanical activation on the pozzolanic activity of clay minerals, such research has not yet been reported for muscovite. Therefore, the main objective of this study was to investigate the changes in structure and pozzolanic activity that occur in muscovite after mechanical activation.

EXPERIMENTAL

Materials

The muscovite used in the present study was obtained from Anhui Henghao Technology Co., Ltd., Anhui Province, China. The chemical composition of the muscovite consisted mainly of Fe_2O_3, SiO_2, and Al_2O_3 (Table 1). The X-ray diffraction (XRD) pattern (Fig. 2) showed that the main mineral phase in the muscovite sample was muscovite with high crystallinity. The muscovite (~150 kg) was dried at 105°C until it reached a constant mass, and was then used for the subsequent experiments.

The Portland cement (PC 42.5) used in the study was obtained from China United Cement Corporation, Shandong Province, China. The major constituents of the Portland cement were SiO_2, Al_2O_3, CaO, and Fe_2O_3 (Table 1).

Analytical reagent-grade calcium oxide (AR, ≥98%), obtained from Shanghai Macklin Biochemical Co., Ltd., Shanghai Province, China, was used as an activator.

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

Chemical composition	Muscovite	Cement
SiO_2	43.88	21.88
Al_2O_3	29.44	4.49
CaO	0.10	64.65
K_2O	13.28	–
Na_2O	0.60	0.51
Fe_2O_3	9.64	3.45
MgO	1.40	2.36
SO_3	0.01	2.44

Mechanical activation

In order to improve the pozzolanic activity of the muscovite, the samples were subjected to mechanical treatment for various durations using a planetary mill (XQM-4, Changsha Tianchuang Powder Technology Co., Ltd., Hunan Province, China) operating at a frequency of 50 Hz, power of 0.75 kW, and rotation speed of 500 rpm. Steel balls of 5, 8, and 15 mm diameter were used as the grinding media, in a ratio of 3:5:2. Each grinding was performed with 60 g of muscovite in a 500-mL steel can containing 600 g of hardened steel balls. In each grinding run, the muscovite samples were ground for periods ranging from 20 to 200 min.

Characterization

The particle-size distribution curves of the muscovite powders were obtained using a BT-9300Z Intelligent Laser Particle Size Analyzer (Dandong Bettersize Instruments, Ltd., Liaoning Province, China) with a particle size range of 0.02–2000 μm.

The specific surface area was measured using a Micromeritics ASAP 2460 (Micromeritics Instruments Corporation, Norcross, Georgia, USA) instrument. The initial and mechanically activated muscovite powders were degassed at 105°C for 10 h under negative pressure, and the specific surface area of the muscovite samples were calculated according to the BET method.

To obtain the powder XRD patterns, a Rigaku Ultima IV (Rigaku Corporation, Tokyo, Japan) diffractometer was used; the XRD patterns of the powder were collected using CuKα radiation ($\lambda = 0.15416$ nm) over a scanning range from 5 to 70°2θ, with a resolution of 0.02°2θ/step and scanning speed of 5°2θ/min.

The infrared (IR) spectra of the muscovite powders in the 4000–400-cm^{-1} range were recorded using a Nicolet iS50 (Thermo Fisher Scientific, Waltham, Massachusetts, USA) Fourier transform infrared spectroscopy (FTIR) spectrometer using the KBr pressed-disk technique. Two milligrams of powder sample was mixed with 200 mg of KBr in an agate mortar, and was then pressed into pellets 13 mm in diameter with a pressure of 8.0 Mpa.

Thermogravimetric (TG) analysis was performed at temperatures from 50 to 1050°C using a Mettler TGA 2 instrument (Mettler Toledo Corporation, Greenville, South Carolina, USA) at a heating rate of 10°C/min in a nitrogen atmosphere.

Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) analyses were conducted using an FEI Nova NanoSEM 430 (FEI, Hillsboro, Oregon, USA) scanning electron microscope at an acceleration voltage of 2.0 kV.

The pozzolanic activity index was evaluated by comparing the compressive strength of two mortar samples (Cheng et al. 2016), one consisting of a standard mix of Portland cement and sand (reference), and the other with 30% of cement replaced with mechanically activated muscovite samples. According to the Chinese National Standard GB/T 12957-2005, the

compressive strength ratio (activity index) of cement-muscovite mortar to cement mortar cured for 28 days should exceed 65%.

RESULTS AND DISCUSSION

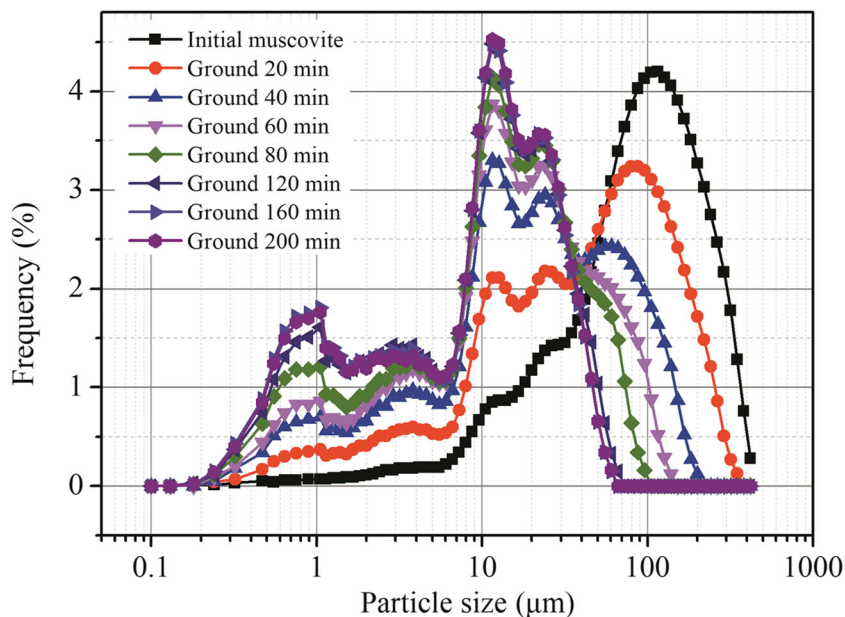### Particle-size distribution and specific surface area

The particle-size distribution curves (Fig. 1) of the initial and mechanically activated muscovite milled for various durations in a planetary mill revealed that the curve corresponding to the initial muscovite was unimodal. The curve later became bimodal or multimodal, depending on the grinding. After 60 min of grinding, the particle-size distribution curve transformed to a broad bimodal curve with two distinct particle size ranges of 0.5–6.0 μm and 8–30 μm. After 120 min of grinding, particles larger than 80 μm disappeared, and the number of fine particles <2 μm in size increased significantly as the grinding time increased. The characteristic diameters D_{10}, D_{50}, and D_{90} were used to characterize the effects of the mechanical activation on the particle-size distribution. These indices represent the particle size at which 10%, 50%, or 90% of the weight is finer. The variation in the characteristic diameter and specific surface area with grinding time (Table 2) indicated that the characteristic diameters D_{10}, D_{50}, and D_{90} decreased significantly over 20 min of grinding, from 17.22 μm, 95.24 μm, and 253.90 μm to 5.69 μm, 47.76 μm, and 169.23 μm, respectively. Thereafter, a gradual change was observed. The size reduction appeared to reach a limit at 160 min of grinding, with particle size increasing with further grinding. Similar change trends have been reported by other researchers using the mechanical activation method (Alex et al. 2014; Andrić et al. 2014; Ilić et al. 2016). The specific surface area increased significantly, from 0.81 m^2/g to 28.14 m^2/g, with grinding time of 120 min, after which the changes in the specific surface area were insignificant. With further grinding, the specific surface area of the 200-min mechanically ground muscovite was less than that of the 160-min ground muscovite. Combined with the particle size distribution analysis, this result was inconsistent with previously reported findings, where the reduction in the particle-size and the increase in the specific surface area occurred in the initial stages, while a prolonged grinding time resulted in decreased specific surface area (Mitrović and Zdujić 2014; Perrin-Sarazin et al. 2010).

Based on the analysis above, the following changes occurred during mechanical activation: the particle size reduction appeared to reach a limit at 160 min of grinding. However, the tendency was toward increased particle size with a further increase in grinding time, probably due to aggregation of the ground fine particles. This tendency was also somewhat more apparent from the significant decrease in surface area at 200 min of grinding (28.82 m^2/g) as compared to 160 min of grinding (23.44 m^2/g).

XRD analysis

The XRD patterns of the initial and mechanically activated (for various grinding times) muscovite with (Fig. 2) revealed that the muscovite reflections decreased significantly with grinding time. The decrease in reflection intensity is a manifestation of the amorphization (Kitamura & Senna 2001), and the loss of reflection intensity illustrates the breaking of the bonds between the muscovite layers. Such an observation implies that significant amorphization was mechanically induced in the muscovite structures during grinding (Mitrović & Zdujić 2014). The reduction in the reflection intensity was insignificant in the initial stages—especially within the first 40 min—but significant changes in the particle size and specific surface area were observed during this period. With prolonged grinding, further changes in particle size and specific surface area were

Fig. 1 Particle size distribution curves of the muscovite samples ground for different durations

Table 2 Variation in the characteristic diameters (D_{10}, D_{50}, and D_{90}) and specific surface area with grinding time

| Grinding time, min	Characteristic diameters, μm			Specific surface area, m²/g
	D_{10}	D_{50}	D_{90}	
0	17.22	95.24	253.90	0.81
20	5.69	47.76	169.23	8.40
40	2.81	21.54	95.42	15.88
60	2.29	16.36	65.79	18.77
80	1.63	14.13	48.51	22.84
120	1.30	12.07	34.27	28.14
160	1.17	11.70	32.07	28.82
200	1.20	11.87	32.35	23.44

insignificant compared with the significantly decreased intensity of the muscovite reflections. Regarding the energy entering the muscovite particle that was obtained from mechanical activation, some of it was released rapidly and some of it may have been stored (Dandurand et al. 1982). In the initial stages, energy storage existed mainly in the form of new surfaces, but with prolonged grinding, energy storage may have caused the formation of internal defects in the muscovite which was the main reason for the amorphization. Crystalline muscovite continued to be observed, indicating that the mechanically induced amorphization was incomplete.

Based on the XRD pattern data for muscovite, analysis of the relative crystallinity was conducted using the intensity of the strongest characteristic peaks (Makó et al. 2001), namely, the intensity of the muscovite (009) reflection. The calculated results (Fig. 3) according to Eq. (1) revealed that the main

decrease in the relative crystallinity occurred after 40–160 min of grinding, and it was reduced to 14.99% at 160 min.

$$\alpha = \frac{I_{ground}}{I_{initial}} \times 100 \qquad (1)$$

In Eq. (1), α is the relative crystallinity of the muscovite, I_{ground} is the intensity of the muscovite (009) reflection of the mechanically ground muscovite, and $I_{initial}$ is the intensity of the initial muscovite (009) reflection (assuming an integral intensity of 100%). Thereafter, the variations were insignificant. However, a continuous decrease in relative crystallinity occurred despite the fact that the specific surface area decreased when the grinding time was prolonged, which was consistent with a previous study (Mitrović & Zdujić 2014).
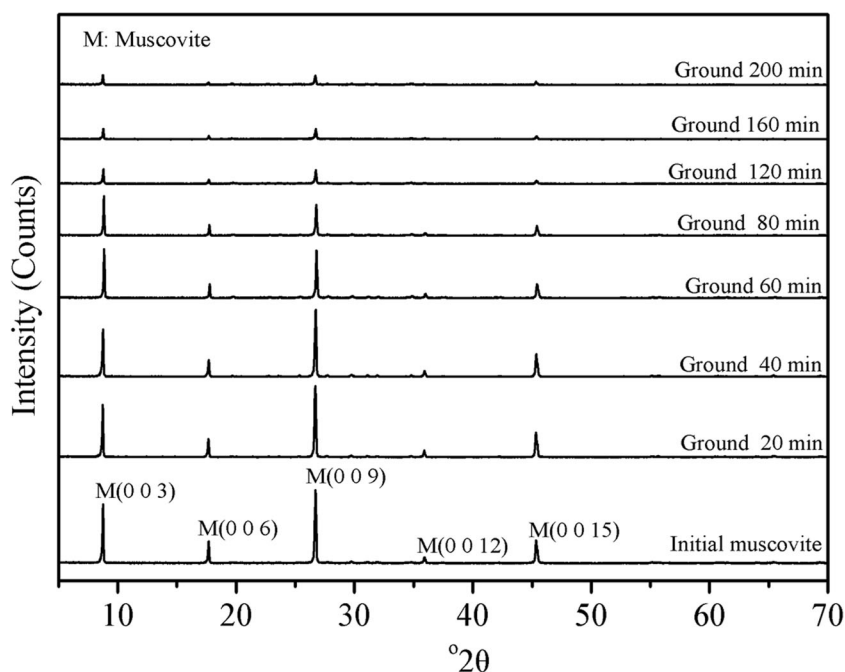
Fig. 2 XRD patterns of the initial and ground (for various amounts of time) muscovite
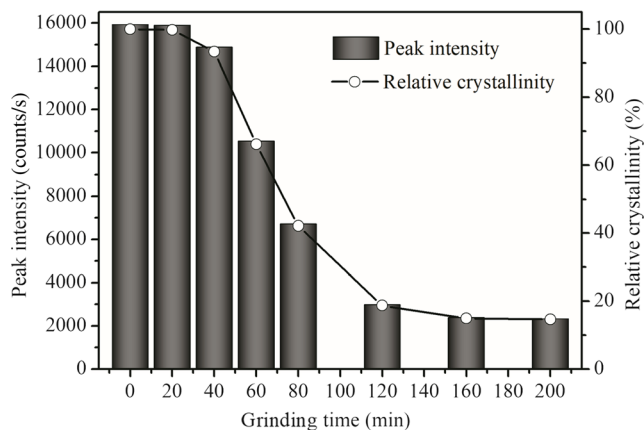
Fig. 3 Variation in the intensity of the muscovite (009) reflection and the relative crystallinity with grinding time

Thermal behavior

In order to study the thermal characteristics of the initial and mechanically activated muscovite during thermal treatment from 50 to 1100°C, TG and derivative thermogravimetric (DTG) analyses of typical samples (Fig. 4) were carried out. Chemically, the crystal structure of the muscovite dehydroxylation at high temperatures can be summarized as $2(OH)^{-1} \rightarrow H_2O + O^{2-}$. The weight loss of initial muscovite at temperatures up to 400°C was only 0.39 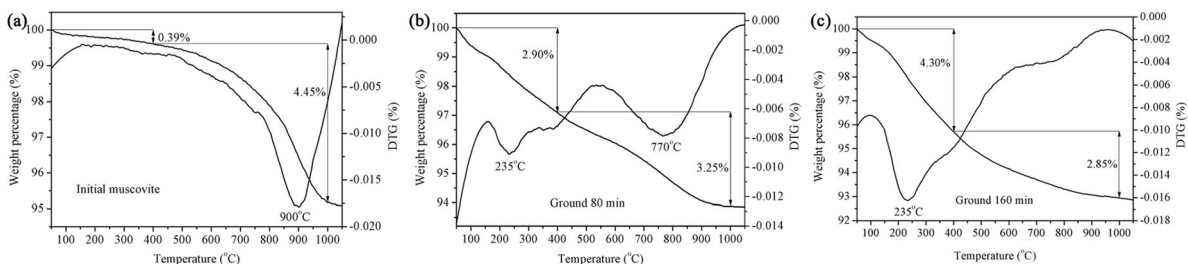wt.% (Fig. 4a), which was attributed to the physically adsorbed water. The total weight loss at 1000°C was 4.45%, and dehydroxylation began to form a dehydroxylated phase at ~600°C, which means that most of the hydroxyls were removed at temperatures between 600 and 1000°C. This result is inconsistent with previously reported findings, where most of the hydroxyls were removed at temperatures below 850°C (Gridi 2008). In their work on the thermal behavior of muscovite, Kodama & Brydon (1968) reported a lower dehydroxylation temperature for mechanically induced muscovite with smaller particle sizes. With prolonged grinding, the dehydroxylation temperature decreased from ~900 to 770°C at 80 min of grinding time (Fig. 4b). Between 600 and 900°C, a stepwise weight-loss with a large and asymmetric peak occurred, which may be due to the formation of a disordered or partially amorphous structure as a result of the mechanical activation. In the sample mechanically activated for 160 min of grinding, the dehydroxylation process

was no longer detected, indicating that mechanical activation transformed the crystal structure of the muscovite into a metastable dehydroxylated phase. Several studies have discussed the dehydroxylation reaction for muscovite (Gridi 2008; Mazzucato et al. 1999; Tokiwai & Nakashima 2010). Guggenheim et al. (1987) reported that dehydroxylation occurred through the loss of hydroxyl groups, where the residual oxygen moved to the *z*-coordinate position of the Al cation, and the Al-O polyhedra was in fivefold coordination in the dehydroxylated phase of the muscovite. The weight-loss peak at 235°C was attributed to the adsorbed water of the mechanically ground muscovite. On the basis of the TG data, the amount of adsorbed water (under 400°C) showed an increasing trend (0.39%, 2.90%, and 4.30% for 0, 80, and 160 min, respectively) with grinding, attributed mainly to the increase in the specific surface area. In contrast, the weight loss of the samples corresponding to dehydroxylation exhibited a decreasing trend with grinding, reflecting the dehydroxylation reaction that occurred during the grinding process.

FTIR spectra

The structural changes in the initial and mechanically activated muscovite (ground for 80 min and 160 min) were determined using FTIR spectroscopy (Fig. 5). The spectra for the initial muscovite showed the following characteristic bands: 3623, 687, and 412 cm^{-1} (O–H vibrations); 1026, 748, 527,

Fig. 4 (**a**) TG and DTG curves of the initial (KAl$_2$(Si$_3$Al)O$_{10}$(OH,F)$_2$) and mechanically activated muscovite after grinding for 80 min (**b**) and 160 min (**c**)

Fig. 5 FTIR spectra of the initial and mechanically ground (for 80 and 160 min) muscovite

and 472 cm^{-1} (Si (Al)–O and Si–O–Si (Al) vibrations); 826 cm^{-1} (Al–OH); and 3447 and 1646 cm^{-1} (OH and HOH vibrations of the adsorbed water) (Ilić et al. 2016; Tang et al. 2016; Vizcayno et al. 2010; Zhang et al. 2010).

Mechanical activation can destroy the crystal structure of muscovite by breaking or weakening the O–H, Al–OH, Al–O–Si, and Si–O bonds (Kodama & Brydon 1968; Papirer et al. 1990; Vizcayno et al. 2010). Prolonged grinding gradually decreased the absorption intensity of the hydroxyl vibration bands at ~3623, 687, and 412 cm^{-1}, and the bands at 687 and 412 cm^{-1} disappeared after 160 min of grinding, indicating that structural modifications occurred due to the partial dehydroxylation. The existence of bands arising from hydroxyl

vibrations at 3622 cm^{-1} after 160 min of grinding suggested that the dehydroxylation of muscovite was not completed. The decrease in absorption intensity of the bands at approximately 826, 748, 527, and 472 cm^{-1} was attributed to the increase in the structural disorder of muscovite with prolonged grinding. In addition, the HOH bending vibration of the adsorbed water at ~3434–3447 cm^{-1} became wider due to an increase in the amount of adsorbed water connected to the particle surface following grinding, and this finding was consistent with the results of the TG analysis.

Pozzolanic activity

According to the analysis above, the mechanical activation process decreased particle size, increased specific surface area, and caused the dehydroxylation reaction of muscovite powders, which increased the amorphous structure. Changes in the pozzolanic activity index were observed as a function of the grinding time, increasing from 36.98% to 95.02% with 200 min of grinding (Fig. 6). During the initial stages—within the first 40 min—the corresponding relative crystallinity of the mechanically activated muscovite was 93.39% according to XRD analysis, and the pozzolanic activity index increased to 61.24%, which still did not qualify it as an active pozzolan. This finding was mainly due to the high relative crystallinity of muscovite. At 60 min of grinding, the relative crystallinity decreased sharply decreased to 66.14%, and the pozzolanic activity index increased to 75.57%, which qualified the material for use as an active pozzolan (≥65% at 28 days according to the Chinese National Standard GB/T 2847-2005). After 160 min of grinding, the changes in the pozzolanic activity index were insignificant, and the result was consistent with the change in the relative crystallinity. Therefore, it could be inferred that the increase in the pozzolanic activity was the consequence of the increased specific surface area, reduced particle size, decreased relative crystallinity, and dehydroxylation caused by mechanical activation.

The present study indicated that the pozzolanic activity index of muscovite could be improved by using mechanical activation because of the amorphous structure produced by the

Fig. 6 Changes in the pozzolanic activity index for the initial and mechanically ground muscovite after 28 days of curing

Fig. 7 Ca(OH)$_2$ contents in the paste samples vs the hydration time as determined by TG

dehydroxylation reaction after grinding. The pozzolanic activity is the capacity of pozzolan to react with calcium hydroxide to form calcium silicate hydrates (Cheng et al. 2016). To further verify the pozzolanic activity of activated muscovite, the amount of calcium hydroxide consumed by the initial and mechanically activated muscovite (typical samples ground for 80 min and 160 min) at various hydration times was measured using the TG technique. Three different types of paste samples were prepared by mechanical mixing of lime, muscovite (ground for 0 min, 80 min, and 160 min), and water at a mass ratio of 3:7:7, and the paste samples were then cured in a curing chamber with relative humidity ≥95% and temperature of 20 ± 1°C for different lengths of time. The calcium hydroxide content in the pastes cured for 1, 3, 7, 14, and 28 days was detected using TG analysis. The results (Fig. 7) demonstrated that the Ca(OH)$_2$ content in mechanically activated muscovite–lime systems decreased significantly with curing time, especially at the early hydration age; the consumption of Ca(OH)$_2$ revealed that the mechanical activation caused a pozzolanic reaction between Ca(OH)$_2$ and activated muscovite to yield hydration products. However, the change in the Ca(OH)$_2$ content in the initial muscovite–lime system was insignificant, indicating that the initial muscovite was a relatively stable material with no pozzolanic activity. In addition, the consumption of Ca(OH)$_2$ presented an increasing trend

with grinding and curing time, indicating that the pozzolanic activity of muscovite was improved via mechanical activation.

In order to determine the morphology and chemical composition of the hydration products formed by pozzolanic reaction between mechanically activated muscovite (ground for 160 min) and lime, the activated muscovite–lime paste cured for 28 days was selected for SEM-EDS analyses. Based on the EDS analysis (Fig. 8, region A), the amorphous reticulate structural morphology (Fig. 8) in the activated muscovite–lime paste was identified as a C–S–H gel that solidified Al^{3+} and K$^+$ during the hydration process. The SEM-EDS analysis results again demonstrated that mechanical activation could improve the pozzolanic activity of muscovite, and the major hydration product in the activated muscovite–lime system was an amorphous C–S–H gel.

CONCLUSIONS

In this study, the effects of mechanical activation on the pozzolanic activity of muscovite were investigated, and the following conclusions were drawn.

(1) The reduction in particle size was evident in the initial stages of the mechanical activation process, and appeared to reach a limit at 160 min of grinding. However, the specific surface area exhibited a decreasing trend when the grinding time was prolonged.

(2) FTIR and TG analysis showed that mechanical activation induced the dehydroxylation reaction of muscovite. The relative crystallinity of the muscovite mechanically activated for 160 min decreased to 14.99%, and the pozzolanic activity index increased to 94.36%. Continuous grinding led to a gradual reduction in the relative crystallinity and an increase in the pozzolanic activity index.

(3) The calcium hydroxide was consumed due to the pozzolanic reaction in the activated muscovite–lime paste system, and the calcium hydroxide content decreased significantly with curing time. The SEM-EDS analysis revealed that mechanically activated muscovite reacted with calcium hydroxide to form a C–S–H gel that solidified Al^{3+} and K$^+$ during the hydration process.
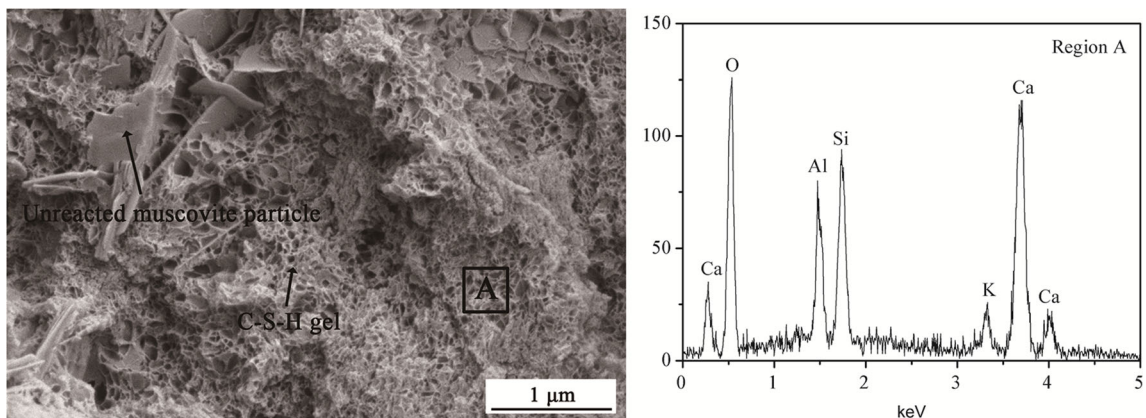
Fig. 8 SEM images and EDS analysis of the activated muscovite–lime paste sample cured for 28 days

(4) The pozzolanic activity of the typical mineral-phase muscovite in the tailings was improved by mechanical activation, and the pozzolanic activity index satisfied the requirements for usage as an active addition in cementitious materials.

Compliance with ethical standards

Conflict of Interest
The authors declare that they have no conflict of interest.

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