

# The thermal activation process of coal gangue selected from Zhungeer in China

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**Abstract** The thermal behavior of coal gangue selected from Zhungeer, Inner Mongolia Autonomous Region of China, was investigated by X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy, thermogravimetry (TG), derivative thermogravimetry (DTG), and scanning electron microscope (SEM). The XRD data indicated that the mineral compositions of the coal gangue were kaolinite, boehmite, and quartz. The coal-gangue sample was considered as belonging to a typical mixture of kaolinite and boehmite. The XRD and FT-IR spectra clearly showed that the structural changes and dehydroxylation of coal gangue occurred with increased temperature from 100 to 900 °C. The reaction activity of coal gangue could be effectively improved by calcination. The calcined coal gangue contained considerable active amorphous Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> and had significant loss on ignition. The optimum activation temperature range of coal gangue was from 600 to 700 °C. The dissolution contents of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> were 92.31 and 64.44 %, respectively, when the calcination temperature at 700 °C.

**Keywords** Coal gangue · Mineral composition · Kaolinite · Thermal activation · Activity

## Introduction

Coal gangue is a solid waste of coal produced in excavation and washing processes. Currently, coal gangue has been one of the largest industrial solid wastes in China. Coal gangue has caused serious damage to the environment, including taking up large land and farmland, and polluting the atmosphere and water quality [1–5]. In China, the total of accumulative stockpile of coal gangue reaches 4.5 billion metric tons. Kaolinite, a layered silicate mineral consisting of siloxane- and gibbsite-like layers, has a wide range of applications [6, 7]. Coal gangue with the high content of kaolinite can be modified by calcination and surface modification to be used as active polymer composite materials. The activation method of coal gangue includes thermal activation, mechanical activation, chemical activation, microwave radiation activation, and composite activation [8–11]. The thermal activation is an effective method to stimulate the activity of coal gangue due to that it could take advantage of the severe thermal motion produced by the microstructure of coal gangue particles at a high temperature, removed bound water in minerals, and reselected interstitial cations positions, such as calcium ion, magnesium ion, and iron ion. Coal gangue contains a large number of active alumina and silica after thermal activation [12, 13]. Studies have shown that the generated amorphous reactive SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> after thermal activation improve the reactivity of coal gangue [14, 15]. Extensive studies have used specific types of coal gangue to find the optimal activation process to achieve high chemical reactivity of coal gangue. The mineral composition of Fujian coal gangue is studied, which mainly consists of illite, quartz, and calcite, and the optimal activation temperature is 750 °C. The dissolution of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> of coal gangue differs in different calcination

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temperatures [16]. The mineral composition of Zibo coal gangue in Shandong includes kaolinite, alpha quartz, gypsum, and calcite, and the optimum activation temperature is 700 °C. Thermal activation is necessary for activating coal gangue [17]. The physico-chemical transformations and dissolution of the active ingredients of Jiahe coal gangue calcined at different temperatures were investigated. The calcination of coal gangue could remove water and organic contents, form amorphous material and glass phase, and make the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> contents much more soluble [18]. In this study, the structure changes of coal gangue in Zhungeer in the thermal activation process were systematically analyzed by X-ray diffraction (XRD) and Fourier transform infrared (FT-IR) spectroscopy. Calcination is an effective means to stimulate the activity of coal gangue. The thermal activation process of coal gangue contains considerable active amorphous silicon dioxide and aluminum oxide [19–23].

## Experimental

### Material and method

The coal-gangue samples collected from Zhungeer, Inner Mongolia, China, were used in this study. All coal-gangue samples were ground and sieved to a particle size below 100 μm. The treated coal-gangue samples were heated at 100, 200, 300, 400, 500, 600, 700, 800, and 900 °C for 2 h at a rate of 10 °C min<sup>-1</sup> in a muffle furnace under an air atmosphere. Activated coal gangue (10 g) and hydrochloric acid solution (60 g, 25 % mass) were mixed finely and stirred at 95 °C for 3 h in a three-neck flask, with a stirring speed of 200 r min<sup>-1</sup>. The products were filtered after the reaction. Filtrate A was obtained, and the filter residue was dried at 100 °C until constant mass. The filter residue and sodium hydroxide solution were then mixed and stirred at 95 °C for 3 h in a three-neck flask, with a stirring speed of 200 r min<sup>-1</sup>. Filtrate B was obtained, and the filter residue was dried.

Filtrate A was for determining the content of Al<sub>2</sub>O<sub>3</sub> in activated coal gangue. The Al<sub>2</sub>O<sub>3</sub> content was determined by the national standard GB15892-2009 aluminum poly-chloride [24]. The SiO<sub>2</sub> content in the coal gangue was measured through the method of alkali-soluble carbon. The specific steps were as follows: Filtrate B was added to the three-neck flask equipped with a stirring system heated at 60 °C with a stirring speed of 200 r min<sup>-1</sup>, and then, CO<sub>2</sub> gas was slowly passed into the flask with a uniform motion. The product was filtered after 0.5 h of reaction, and hydrochloric acid solution was continuously sprayed to the filter cake. The impurities in the filter cake were removed until the filter cake had no bubble. The filter cake was then

rinsed with distilled water, weighed, and dried in a vacuum oven. The SiO<sub>2</sub> content of activated coal gangue was extracted.

### Characterization

The X-ray diffraction (XRD) patterns of the raw and the thermally treated samples were generated using a Rigaku D/MAX 2500PC powder X-ray diffractometer with Cu Kα radiation ( $\lambda = 1.54059 \text{ \AA}$ ), with a scanning rate of 4° min<sup>-1</sup> in the range of 2.6° to 60°, and operated at 40 kV and 40 mA.

Fourier transform infrared (FT-IR) spectra were analyzed by Nicolet 6700, Thermo Fisher. The samples were mixed with KBr and ground in an agate mortar for 5 min. The mixture was pressed into a pellet for transmittance infrared spectroscopic measurements. The FT-IR spectra of prepared samples between 400 and 4000 cm<sup>-1</sup> were recorded.

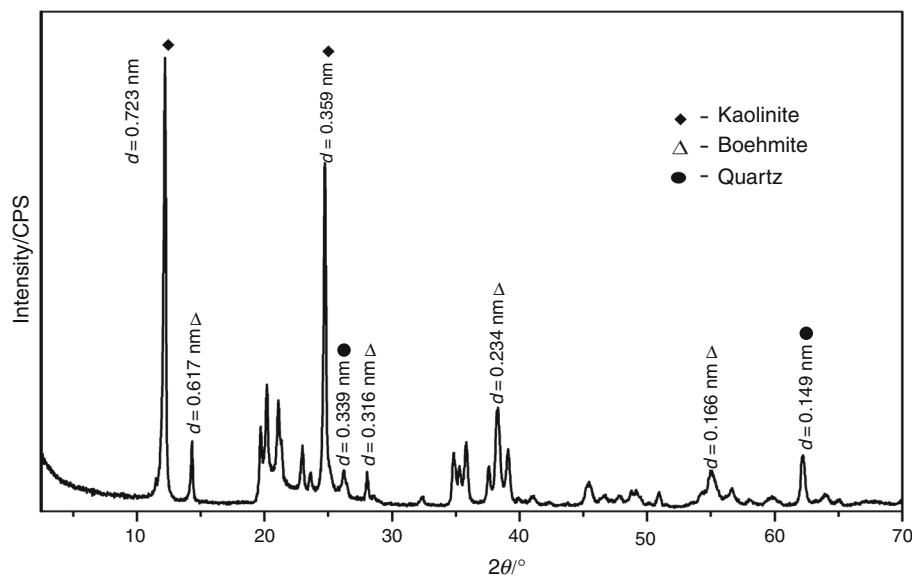
A Switzerland Mettler Toledo TGA/DSC1/1600HT was used for thermogravimetric combustion experiments. The sample was heated from room temperature up to a maximum temperature of 1100 °C at a heating rate of 10 °C min<sup>-1</sup>.

The morphology of the raw coal gangue and the calcined coal gangue was observed by a cold field emission scanning electron microscope (SEM, S-4800, Hitachi). During the observation, an accelerating voltage of 15 kV was selected, and the resolution was of ±2 nm.

## Results and discussion

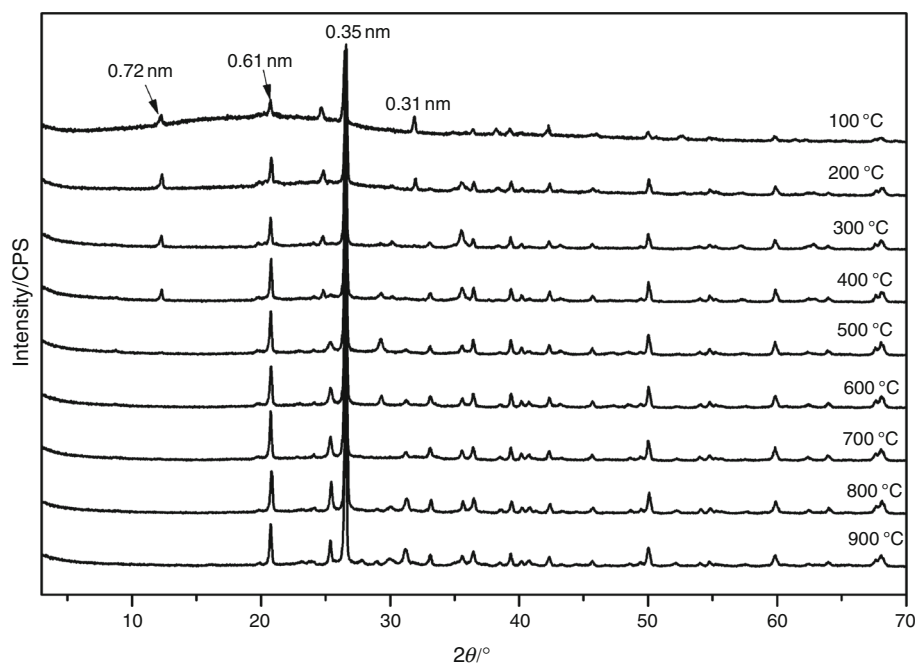
### XRD

The XRD patterns of raw coal gangues are presented in Fig. 1. As illustrated in Fig. 1, the major mineral compositions of the original coal gangue are kaolinite (PDF card No. 14-0164), quartz (PDF card No. 65-0466), and boehmite (PDF card No. 21-1307). The strong diffraction peaks with the values of 0.72 and 0.359 nm at  $2\theta = 12^\circ, 24^\circ$  are attributed to the diffraction of (001) and (002) crystal planes, respectively. Three diffraction peaks with the values of 0.45, 0.439, and 0.420 nm at  $2\theta = 18^\circ\text{--}24^\circ$  are attributed to the diffraction of (020), (110), and (111) crystal surface reflections, respectively [25]. Six diffraction peaks at  $2\theta = 35^\circ\text{--}40^\circ$  have favorable separation condition and peak shape. Two diffraction peaks with  $d_{(001)}$  value of 0.72 nm and  $d_{(002)}$  value of 0.35 nm present six distinct peaks in the highly ordered kaolinite XRD patterns, and the relative intensity of the diffraction peaks can also be changed with the decreased number of the characteristic diffraction peaks [26]. The diffraction peaks by quartz at

**Fig. 1** XRD patterns of raw coal gangue

$2\theta = 26.7^\circ$  and  $62.1^\circ$  are weak, which display that the content of quartz is insignificant [27]. The characteristic diffractions with values of 0.617 and 0.317 nm at  $2\theta = 14.1^\circ$ ,  $28^\circ$  are attributed to boehmite [28, 29]. The characteristic peaks with the  $d_{(031)}$  value of 0.234 nm and  $d_{(151)}$  value of 0.166 nm are attributed to boehmite. A certain content of boehmite exists in the coal-gangue sample, which illustrates that the aluminum content in coal gangue is high, and the  $\text{SiO}_2/\text{Al}_2\text{O}_3$  molar ratio of the coal-gangue sample is less than the theoretical value. This result is consistent with the result of chemical analysis.

The XRD patterns of all thermally treated coal-gangue samples calcined at  $100\text{--}900^\circ\text{C}$  are shown in Fig. 2. The diffraction peak shape of the calcined one at  $100\text{--}400^\circ\text{C}$  is similar to that of the raw coal-gangue sample. The diffraction peak a value of 0.72 nm at  $2\theta = 12.6^\circ$  gradually weakens as the temperature increases. At the calcination temperature of  $600^\circ\text{C}$ , the diffraction peaks of kaolinite completely disappear through removing the inner hydroxyl structure, which may be due to the destruction of the crystal face (001) and the formation of an amorphous substance. The  $\alpha$ -quartz diffraction peak with the value of

**Fig. 2** XRD patterns of the coal gangue samples calcined at different temperatures

0.35 nm is intense. The result may be attributed to the further loss of water in kaolinite, boehmite, and free carbon, thereby generating amorphous  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ , which makes the content of quartz increase [30].

### FT-IR analysis

The FT-IR spectra of raw coal gangue in Zhungeer are shown in Fig. 3. The infrared spectra of kaolinite mainly include the characteristic absorption bands of Si–O, –OH, and H–O–H. As illustrated in Fig. 2, in the high-frequency region ( $4000\text{--}3000\text{ cm}^{-1}$ ), two –OH absorption bands exist at  $3694$  and  $3619\text{ cm}^{-1}$ , which are attributed to the surface and inner hydroxyl stretching vibration bands. The intensity of the absorption band at  $3619\text{ cm}^{-1}$  is stronger than that of the absorption band at  $3694\text{ cm}^{-1}$ , and the band at  $3411\text{ cm}^{-1}$  is associated with the OH stretching vibration mode of absorption water. However, the other two feature bands of kaolinite at  $3668$  and  $3652\text{ cm}^{-1}$  in the high-frequency region do not appear, thus showing that the bottom surface structure of coal kaolinite is imperfect. In the intermediate-frequency and low-frequency regions, the characteristic vibration bands at  $2919$  and  $2850\text{ cm}^{-1}$  are assigned to aliphatic or naphthenic C–H. The band at  $1598\text{ cm}^{-1}$  may be attributed to the vibration band of aromatic hydrocarbons or carboxylate salts. The band at  $1384\text{ cm}^{-1}$  is attributed to the methyl or methylene vibration band. These bands indicate that coal-gangue samples contain carbonaceous components. The bands at  $1094$  and  $1036\text{ cm}^{-1}$  are attributed to the symmetric

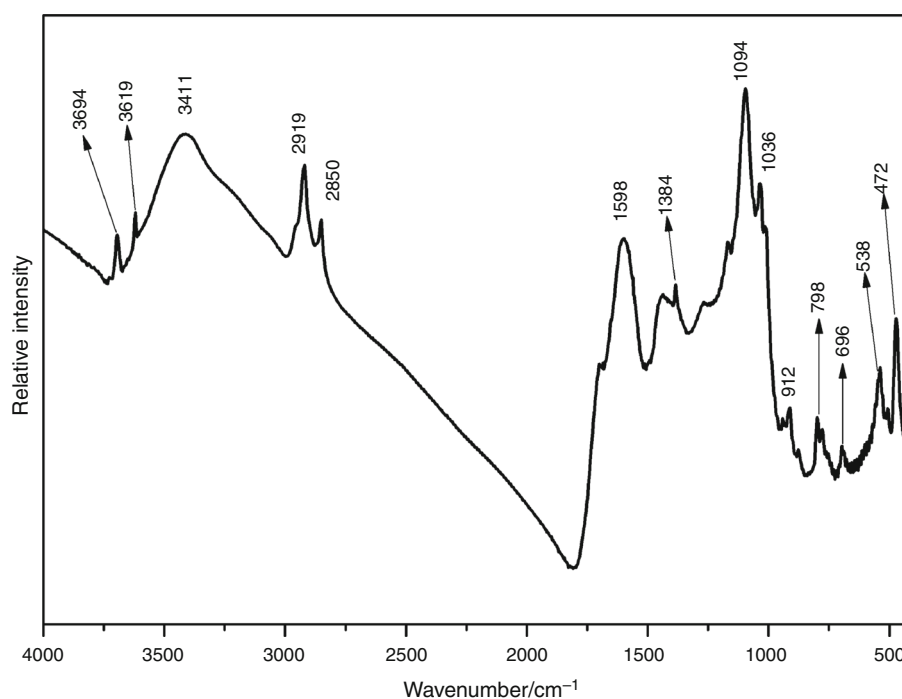
stretching vibration of Si–O–Si. The bending vibration band differentiation of the inner surface hydroxyl groups is not obvious. The band approximately at  $696\text{ cm}^{-1}$  is the stretching vibration mode of Si–O–Al, and the bands at  $538, 472\text{ cm}^{-1}$  are the bending vibration mode of Si–O–Al.

The results of FT-IR under different calcined temperatures are presented in Figs. 4 and 5. The band at  $3695$  and  $3650\text{ cm}^{-1}$  is formed by the vibration of the internal and external hydroxyl groups of kaolinite in coal gangue [31]. According to a study, the band at  $3695\text{ cm}^{-1}$  is related to the stretching vibration of the hydroxyl groups of kaolinite in coal gangue [32]. The intensity of the two bands at  $3694$  and  $3619\text{ cm}^{-1}$  gradually decreases and disappears at  $600\text{ }^\circ\text{C}$ , which indicates that the hydroxyl groups are removed in comparison with that of raw coal gangue. The intensity of the bands at  $915$  and  $472\text{ cm}^{-1}$  gradually decreases and disappears at  $900\text{ }^\circ\text{C}$ . The result is attributed to the breakages of Al–OH and Si–O–Al as temperature increases. The intensity of the band at  $1094$  and  $1036\text{ cm}^{-1}$  also gradually weakens as the temperature increases, and a wide band occurs when the temperature is  $900\text{ }^\circ\text{C}$ . The results are associated with the depolymerization and collapse of silica tetrahedrons structure [33, 34].

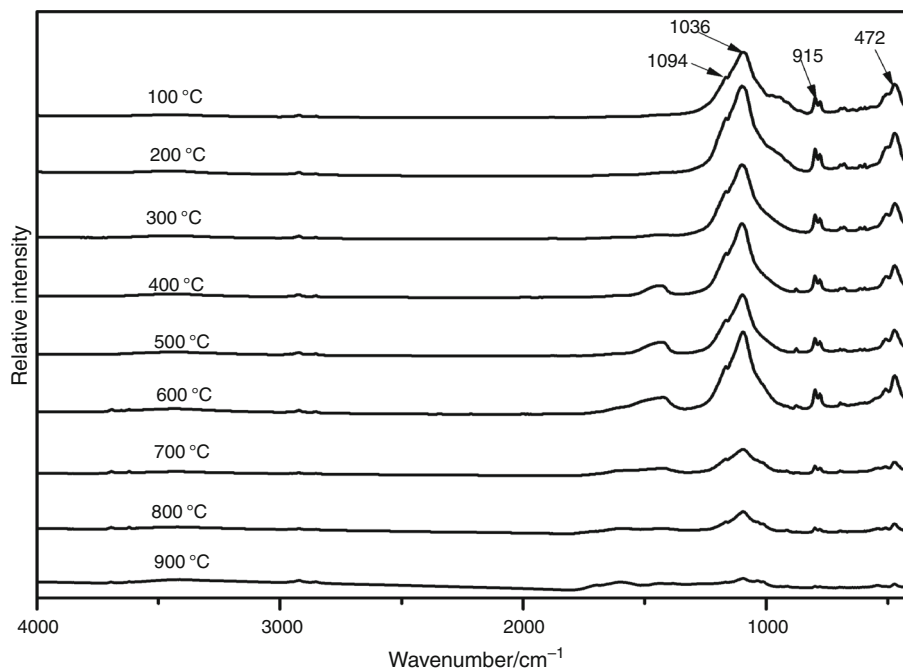
### Thermal analysis

The TG-DTG curves of coal gangue samples in Zhungeer are presented in Fig. 6. An endothermic peak of raw coal gangue exists at approximately  $100\text{ }^\circ\text{C}$ , with mass loss of  $10.23\%$ , which is attributed to the loss of the adsorbed

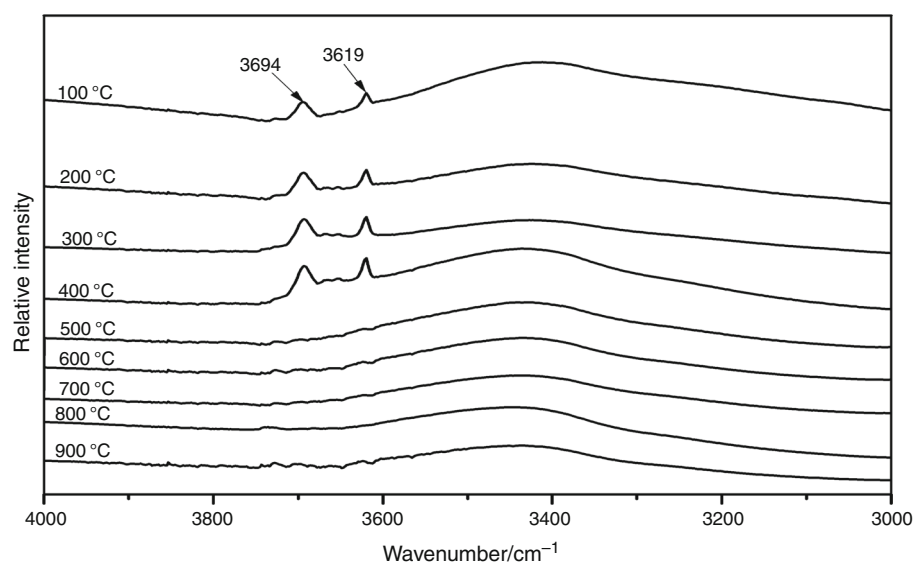
**Fig. 3** FT-IR patterns of raw coal gangue



**Fig. 4** FT-IR patterns of the coal gangue samples calcined at different temperatures



**Fig. 5** FT-IR patterns of the coal gangue samples in the 3000–4000  $\text{cm}^{-1}$  region at different calcined temperatures



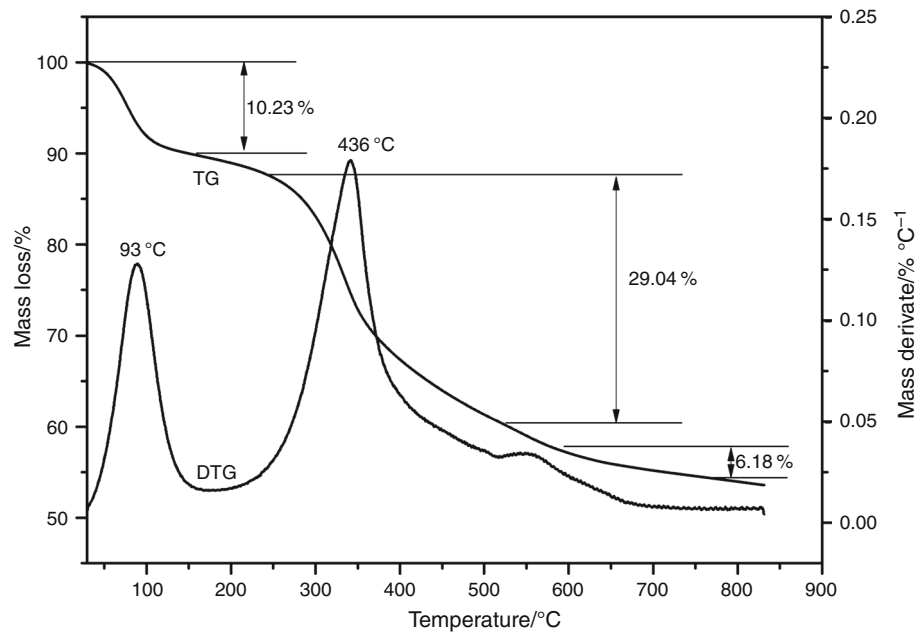
water. The endothermic peak presented at 436 °C with mass loss of 29.4 % is associated with the dehydroxylation of kaolinite in coal gangue because of the dehydroxylation of the crystal lattice [35]. The theoretical loss of the structural water of kaolinite is 14.4 %. The mass loss of coal-gangue samples is higher than the theoretical value of the kaolinite structure water, which indicates that dehydroxylation reaction and carbon loss of kaolinite and boehmite occur [36].

The peak presented at 436 °C in the derivative TG curve is contributed by the dehydroxylation of coal gangue, which results in the metakaolin transformation of the

thermodynamic metastable state [31], and indicates that the lattice is severely damaged and the structural water is driven off. This temperature is lower than the dehydroxylation reaction temperature for ordinary coal gangue. This phenomenon may be due to the poor crystallization degree of coal gangue in Zhungeer. Low crystallinity generally leads to the low temperature of dehydroxylation, and vice versa [37], which is consistent with the crystal-order analysis in the XRD analysis section.

The ignition loss of 32.34 % is associated with the considerable free carbon of coal gangue. The coal gangue in Zhungeer can be effectively utilized.

**Fig. 6** TG-DTG patterns of raw coal gangue

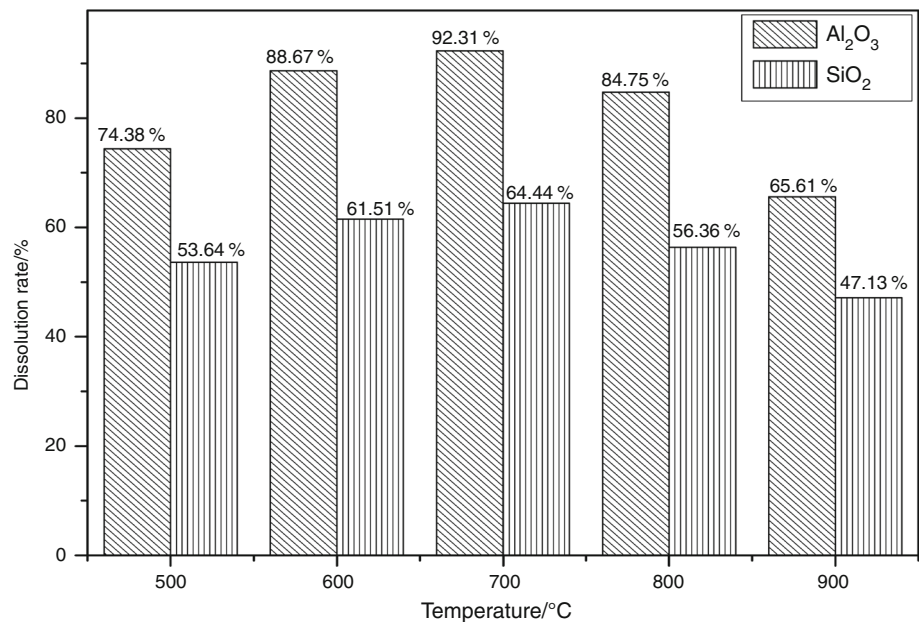


### Thermal activation analysis

This section presents the thermal activation analysis of coal-gangue in Zhungeer. The dissolution contents of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in different calcination temperatures are studied. The dissolution content of coal-gangue samples in Zhungeer is shown in Fig. 7. The dissolution content of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> is reviewed with increasing temperatures from 500 to 900 °C. The dissolution contents of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> increase

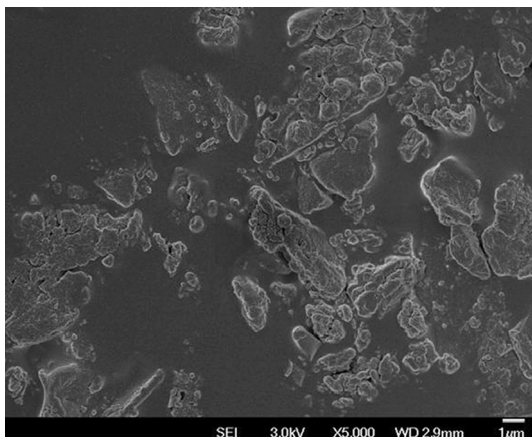
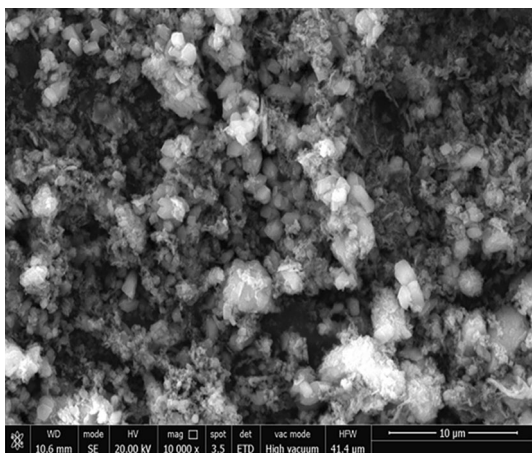
as the temperature rises. At the calcination temperature of 700 °C, the dissolution contents of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> reach maximum at 92.3 and 64.44 %, respectively. When the calcination temperature continues to increase, the dissolution contents of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> gradually decrease. When the calcination temperature is 600 °C, the dissolution contents of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> are also high. Therefore, the optimal active region is 600–700 °C. The results are correlated with the results of TG–DTA and XRD analysis (Table 1).

**Fig. 7** Dissolution contents of the coal gangue samples calcined at different temperatures



**Table 1** Chemical composition of raw coal gangue sample

Coal gangue	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	K <sub>2</sub> O	Na <sub>2</sub> O
Mass fraction	34.58 %	31.95 %	0.76 %	1.00 %	0.3 %	0.02 %	0.42 %

**Fig. 8** SEM image of raw coal gangue**Fig. 9** SEM image of coal gangue calcined at 700 °C

### The microstructure analysis

According to the results of the above analysis, the coal gangue calcined at 700 °C has the best activation effect. The SEM images of raw coal gangue and the coal gangue calcined at 700 °C are presented in Figs. 8 and 9. The microstructure of raw coal gangue presented clumps, schistose, and nonuniform size distribution. The chunks disappear and the fragments increase after coal gangue calcined at 700 °C. The overall particle distribution of calcined coal gangue is uniform and loose compared with that of raw coal gangue. Calcined coal gangue is basically

loose because of the component volatilization and frame expansion [38]. Thermal activation could destroy the stable structure of coal gangue and improve the reaction activity.

### Conclusions

The following conclusions can be drawn from this study:

1. The mineral composition of coal gangue selected from Zhungeer was kaolinite, boehmite, and quartz.
2. The free carbon and organic volatile matter in coal gangue could be removed by thermal activation and calcination. The long-chain polymeric structure that consisted of Si–O bond and Al–O bond was damaged. The reaction activity of coal gangue was improved, and activated amorphous Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> were increased.
3. Thermally activated analysis at different temperatures showed that the optimum activation temperature region of coal gangue in Zhungeer was 600–700 °C. The dissolution contents of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> reached 92.31 and 64.44 %, respectively.

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