

## TRANSFORMATION BEHAVIOR OF ALKALI METALS IN HIGH ALKALI COALS

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**Abstract** – Burning a coal containing a high content of alkali metals (Na, K) in a circulating fluidized bed (CFB) may result in ash-related problems such as bed agglomeration, slagging and fouling on the heat transfer surfaces. This study investigates the transformation behavior of Na in four typical high-alkali coals during combustion. The Na content in the coal is much higher than the K content, and different high-alkali coals have difference in the aspect of their existing forms. Measurement results of the tested coals showed that most of the Na in the coal is H<sub>2</sub>O-soluble Na. During combustion, the released alkali metals increase with the elevated temperature. The H<sub>2</sub>O-soluble Na and CH<sub>3</sub>COONH<sub>4</sub>-soluble Na in the ash decreases after the coal is burnt. This indicates that the released Na is originated from the H<sub>2</sub>O-soluble and CH<sub>3</sub>COONH<sub>4</sub>-soluble Na, with the H<sub>2</sub>O-soluble Na being the dominant constituent. The investigation also reveals that some of the H<sub>2</sub>O-soluble Na in the ash might be converted into HCl-soluble Na and insoluble Na. Furthermore, the low volatility of Na for tested coals is closely associated with the content of Cl, Si and Al in the coal.

**Key words:** high-alkali coals, sodium, transformation behavior;

### 1. INTRODUCTION

Coal, as an important source of primary energy, will continue to play an important role in worldwide energy consumption over the next few decades, particularly in China. However, the high-grade good quality coal has been excessively consumed; thus, low-grade coal has been attracting increasing attention. Unfortunately, some of these coals can give rise to several serious economic and safety problems in coal-fired power plants, including many ash-related problems, such as fouling, slagging, and bed agglomeration (Akiyama et al., 2010). One important factor causing these issues is the high content of alkali metals in coal, especially Na.

During the thermal utilization of high-alkali coals, part of the alkali metals (Na, K) are released into the flue gas. Subsequently, the released alkali metals can condense on the heat transfer surface with a relatively low temperature and form a sticky layer, which results in the growth of an ash deposition layer. Therefore, the transformation behavior of alkali metals is a very important stage for the formation of ash deposition. Many researchers have focused on this subject (Li et al., 2015; van Eyk et al., 2011; Wei et al., 2008; Kosminski et al., 2006).

As is commonly known, not all alkali metals lead to ash deposition problems. Alkali metals exist in two forms in coal: inorganic compounds and organic compounds (Li et al., 2015; van Eyk et al., 2011). Presently, the alkali metals in coal are classified as water-soluble (H<sub>2</sub>O-soluble), ammonium acetate-soluble (CH<sub>3</sub>COONH<sub>4</sub>-soluble), hydrochloric acid-soluble (HCl-soluble) or insoluble by using the chemical extraction method (Li et al., 2015; Zhang et al., 2001). H<sub>2</sub>O-soluble alkali metals are mainly chloride, sulfate and carbonate, all of which can be dissolved in water. CH<sub>3</sub>COONH<sub>4</sub>-soluble alkali metals are those present as exchangeable ions that are organically bound with carboxyl groups. HCl-soluble alkali metals are those present as coordinated oxygen-containing functional groups. The insoluble alkali metals are those highly stable compounds that cannot be dissolved with the above-mentioned solvents, such as silicates (Zhang et al., 2001).

There is a close relationship between the release of alkali metals and their existing forms in coal. The released alkali metals mainly come from H<sub>2</sub>O-soluble and organic alkali metals (Li et al., 2015; Zhang et al.,

2001; Naruse et al., 1998; He et al., 2015), but not all volatilized alkali metals will be completely released during combustion. Some of them will be retained in the ash. The element Cl is an important factor for Na release. Rassk (1985) thought that H<sub>2</sub>O-soluble Na is released mainly in the form of NaCl. Oleschko et al. (2007) analysed the combustion products of German brown coals on line. The results showed that the release content of NaCl and KCl strongly dependent on the Cl-content. Nevertheless, it is difficult to elucidate the relation between the Cl-content and Na release completely during combustion. The release of Na and Cl from coal containing NaCl was disproportionate (Kosminski et al., 2006). Moreover, according to the research results of Gottwald et al. (2001), although the content of Cl in the coal was decisive for the level of alkali released in PFB combustion, the presence of clay minerals in the coal or the fluidized bed strongly suppressed alkali release. Thus, the Si and Al elements in coal have significant influence on the release of Na during combustion as well (Naruse et al., 1998), especially for the coal with high Na content, but low Cl-content. The transformation behavior of Na for these coals is seldom reported, and the distribution mechanism of volatilized Na in the gas phase and solid phase should be further investigated.

The aim of this article is to investigate the transformation behavior of Na in high-alkali coals with low-Cl content during combustion. The combustion experiments were conducted in an electrically heated tube reactor at a temperature of 500~1000 °C. The existing forms of alkali metals (Na and K) in coal/ash were measured by the chemical extraction method. Meanwhile, to gain a better understanding of the transformation behavior of Na species, the equilibrium calculation was used by FactSage 5.2.

## 2. EXPERIMENTAL METHOD

### 2.1 Sample preparation

Four kinds of coals with high alkali metals (Na, K) were investigated in this article. They are referred to as coal 1, coal 2, coal 3 and coal 4, respectively. Among these, coal 1 comes from the Zhundong coalfield, which was newly discovered in China. This coalfield has caused great concern because of its huge reserve, low-ash content and excellent combustibility etc. (Li et al., 2012; Liu et al., 2016). The other coals come from the largest coalfield in Pakistan, known as the Taer coalfield. These four coals all have serious ash deposition propensity due to the high Na content.

The received coal was put in an electric thermostatic drying oven with the temperature set as 50 °C, and the air drying process lasted for 2 h. Then, the air-dried coal was crushed and sieved to obtain particles with a size of ≤ 200 μm. These coal samples were used for further analysis. Additionally, the proximate, ultimate and ash composition analysis are given in Table 1, Table 2.

Table 1 Proximate and ultimate analysis of coals

Coal sample	Proximate analysis $w_{ad}/\%$				Ultimate analysis $w_{ad}/\%$					
	<i>M</i>	<i>A</i>	<i>V</i>	FC	C	H	O	N	S <sub>t</sub>	Cl
Coal 1	11.17	6.43	27.91	54.49	62.89	3.04	15.41	0.55	0.51	0.019
Coal 2	9.24	12.77	44.17	33.82	54.43	3.90	16.89	1.04	1.73	0.014
Coal 3	7.97	21.15	40.62	30.26	46.82	3.70	16.85	0.84	2.67	0.011
Coal 4	8.86	13.36	44.90	32.88	54.72	4.02	16.07	1.11	1.86	0.020

Table 2 Ash composition analysis of coals

Coal sample	Content $w/\%$							
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>
Coal 1	41.57	11.16	4.79	16.21	6.48	0.51	3.50	8.05
Coal 2	30.42	17.46	12.76	12.11	6.96	0.29	3.13	14.02
Coal 3	45.55	18.55	14.13	4.75	6.06	0.22	1.76	6.51
Coal 4	36.91	16.75	13.27	11.47	6.23	0.14	2.91	11.06

## 2.2 Measurement of alkali metals (Na, K)

The chemical extraction method was widely used to measure the content of alkali metals (Na, K) (Li et al., 2015; Zhang et al., 2001; He et al., 2015; Wang et al., 2015). First, H<sub>2</sub>O-soluble, CH<sub>3</sub>COONH<sub>4</sub>-soluble and HCl-soluble alkali metals were extracted sequentially from coal or ash by H<sub>2</sub>O, CH<sub>3</sub>COONH<sub>4</sub> (1 mol/L) and HCl (1 mol/L). Then, the residue was digested with a microwave digestion system. Lastly, the extraction and digestion solutions were analysed with inductively coupled plasma optical emission spectrometry (ICP-OES), and the content of the alkali metals in different existing forms was obtained.

## 2.3 Combustion experiment

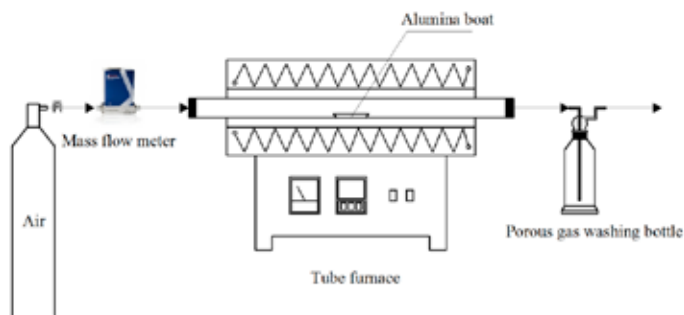


Fig. 1 The setup of the combustion experiment

The combustion experiments were conducted in an electrically heated tube reactor (as shown in Fig. 1). To investigate the transform behavior of alkali metals during the coal combustion, 2 g of coal was loaded in an alumina boat that was positioned in the centre of the tube reactor. During each experiment, the air was continually supplied by a gas cylinder, and the flowrate was set as 1.0 mol/L via the mass flow metre. Temperature is an important parameter for coal combustion, and it was increased from room temperature to the setting temperature at a rate of 10 °C/min, then maintained for 1 h in order to ensure that the coal sample was completely combusted. After combustion, the ash was collected and analysed. It is well known that the content of alkali metals complies with the conservation law. They are partly released into the gas phase, the other remainder is retained in the ash. Thus, the content of alkali metals in the gas phase can be obtained when those retained in ash are analysed.

## 2.4 Equilibrium calculation on the transformation of Na species

Thermodynamic equilibrium calculations were carried out using the FactSage 5.2 software program to simulate the transformation behavior of Na species during combustion. The calculated temperature range was set as 500–1200 °C, which corresponded with the combustion temperature of circulating fluidized beds, and the temperature step was 50 °C at normal pressure. The required data for the calculations included: C, H, O, N, S, Cl, Si, Al, Fe, Ca, Mg, K and Na. The theoretical air volume was calculated using the equation:  $V^0 = 0.0889([C] + 0.375[S]) + 0.265[H] - 0.0333[O]$ , and the excess air coefficient was 1.2. The calculation method was based on the principle of minimization of Gibbs free energy.

# 3. RESULTS AND DISCUSSION

## 3.1 Content of alkali metals (Na, K)

Ash deposition problems (slagging, fouling) are related to the total amount of alkali metals and their existing forms in coal. As shown in Fig. 2(a), the largest amount of Na exists in the form of H<sub>2</sub>O-soluble Na, and HCl-soluble Na is found in the lowest proportions for the different coals. For instance, H<sub>2</sub>O-soluble Na accounts for 84% of the composition of Na in coal 2, and insoluble Na is only 4%. Compared with Na, the existing forms of K show some variance (shown in Fig. 2). The largest component in coal 1 is insoluble K, but there is no significant difference between different K for coal 2, 3 and 4. This is because coal 1 and coals 2–4 come from different coalfields, and there are obvious differences between the primitive plants, coal-forming processes and geological environments in different coalfields (Zhang et al., 1999). Additionally, the total amount of Na is higher than that of K in the coals. According to the analysis results,

the contents of sodium in all of the coals are more than 2200  $\mu\text{g/g}$ , while that of potassium was only 296~459  $\mu\text{g/g}$ .

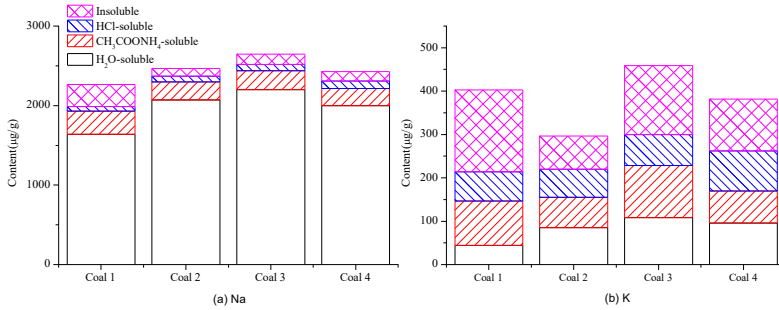


Fig. 2 Content of alkali metals (Na, K) in the coals

### 3.2 Release behavior of alkali metals during combustion

After coal combustion, ash was retained in the alumina boat. The ash rate can represent the amount of ash which was produced during coal combustion, and it can be computed by:

$$\text{Ash rate (\%)} = \frac{\text{The weight of ash retained in the alumina boat}}{\text{The weight of coal before combustion}} \times 100\% \quad (1)$$

As shown in Fig. 3, the ash rate decreases when the combustion temperature increased, which indicates that high temperature is beneficial for the complete combustion of coal. In addition, the ash rate of coal 3 is larger than those of the other coals. Meanwhile, the ash rates of coal 2 and coal 3 are almost the same, and it is lowest for coal 1. These results all correspond with the proximate analysis shown in Table 1.

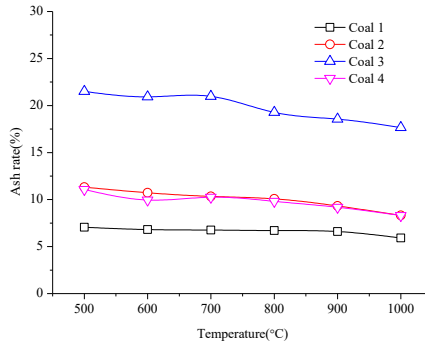


Fig. 3 Ash rate of the different coals

With an increase in temperature, the content of alkali metals (Na and K) retained in ash decreased constantly at temperatures of 500~1000  $^{\circ}\text{C}$  (as shown in Fig. 4). As a whole, the volatility of Na is not high, and the content of Na retained in the ash is 88%~65%. Only a small amount of Na volatilizes into the flue gas. When the analysis result of coal 1 is compared with those of Zhundong coal obtained by Li et al. (2015) and Liu et al. (2014), the released content of Na in coal 1 is lowest. Li et al. (2015) analysed the crystalline phases of ash at different temperatures. The results indicated that NaCl was clearly present in low-temperature ash, and NaCl(g) was the main form of Na volatilized from coal at temperatures of 600~800  $^{\circ}\text{C}$ . Liu et al. (2014) measured the  $\text{Cl}^-$  content extracted by the  $\text{H}_2\text{O}$ , which was much higher than the total content of Cl in coal 1. Thus, the low Cl content might be responsible for the low volatility of Na.

Additionally, the order of the volatility of Na can be listed as: coal 1 > coal 4/coal 2 > coal 3. Although the volatility of Na in coal 1 is highest among these four coals, the content of  $\text{H}_2\text{O}$ -soluble Na is lowest (as shown in Fig. 2). There is no simply positive relationship between the volatility of Na and the content of

H<sub>2</sub>O-soluble Na. Furthermore, the Cl contents in coals 1~4 are all low (as shown in Table 1), and the mole ratio of Cl/Na is 0.03~0.05. Therefore, the promotion effect of Cl on the Na release could be ignored for these coals. Li et al. (2014) investigated the release and transformation of alkali metals during co-combustion of coal and sulfur wheat straw. The results indicated that the amounts of K and Na released during co-combustion could be reduced by the effects of Fe, Ti, S, Si and Al from blended fuels. The mole ratios of Si/Na and Al/Na in coal are given in Fig. 5, according to the analysis results of ash composition. The Si and Al content have a positive relationship. Their order is: coal 3>coal 4> coal 2 >coal 1. The high Si and Al content in coal means much higher levels of relative alumina-silicates are likely to exist in coal. These minerals in coal/ash might have chemical absorption functions on alkali metals (Wei et al., 2016; Kyi and Chadwick, 1999). Moreover, the order in the Si and Al content almost matches that of the volatility of Na, Therefore, the Si and Al have a large influence on the release of Na as well.

Contrary with Na, the volatility of K is much higher, as shown in Fig. 4(b). Except for coal 1, more than 50% of potassium volatilized into the gas phase at a temperature range of 500~1000 °C. Although the proportion of soluble K is not as high as that of soluble Na in coal, the high volatility of K might result from the low absorption reactivity between soluble potassium and silicates/alumina-silicates. Moreover, there may be another reason responsible for this phenomenon. K, which exists inside silicates, will diffuse to the outer surface of silicate particles and then volatilize in the form of K atoms within the temperature range of the research (Liu et al., 2016).

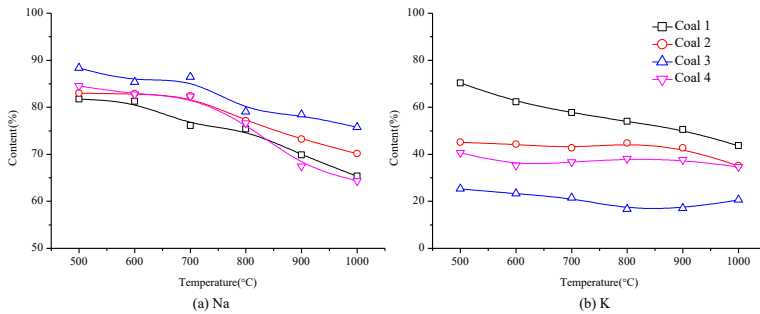


Fig. 4 The content of alkali metals retained in the ash after combustion

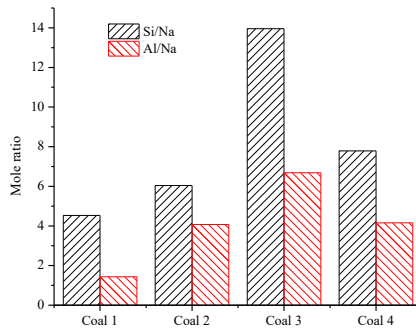


Fig. 5 The mole ratio of Si/Na and Al/Na in coals

### 3.3 Transformation behavior of Na

Although the volatility of K is higher than that of Na, Na deserves much attention due to its high content. Volatilized Na is very important during the ash deposition process, and different forms of Na have different volatilities. It is imperative to learn about the source of volatilized Na, especially when the pre-treatment method is applied before coal combustion, as it can provide theoretical guidance for choosing an appropriate solvent to eliminate the volatile Na in coal, which results in the mitigation of ash deposition. Fig. 6 shows the forms of Na retained in the solid phase after combustion, and their contents are acquired based on the total content of Na in the coal. The content at 20 °C indicates the Na content in coal, which is the same as the Na content shown in Fig. 3.

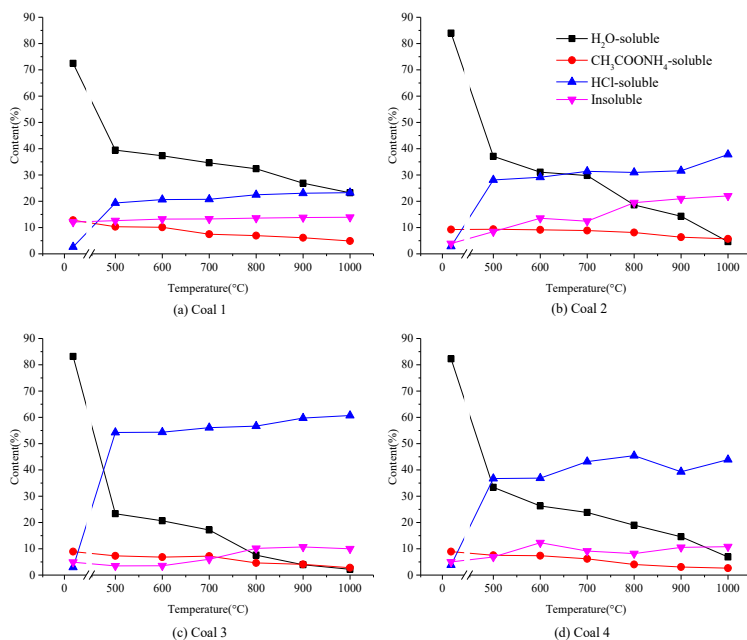


Fig. 6 The existing forms of Na retained in the solid phase (coal/ash)

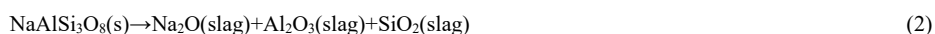
As shown in Fig. 6, the H<sub>2</sub>O-soluble and CH<sub>3</sub>COONH<sub>4</sub>-soluble Na retained in ash decrease continuously with increasing temperature for the four coals, while the other Na forms remained almost the same or increase slightly. This reveals that the Na released during combustion might originate from H<sub>2</sub>O-soluble and CH<sub>3</sub>COONH<sub>4</sub>-soluble Na. As shown in Fig. 6(a), the content of H<sub>2</sub>O-soluble Na decreases from 72% to 39% after the combustion at 500 °C, while that of CH<sub>3</sub>COONH<sub>4</sub>-soluble Na just decreases from 13% to 10%. Thus, it can be concluded that H<sub>2</sub>O-soluble Na takes a dominant role in the release of Na compared with CH<sub>3</sub>COONH<sub>4</sub>-soluble Na. The same conclusion can be reached from the other three coals.

The H<sub>2</sub>O-soluble Na content retained in the solid phase decreases by 33% at 500 °C for coal 1, but the total released Na during combustion is only 18%, while the HCl-soluble Na increases from 3% to 19%. Some of the H<sub>2</sub>O-soluble Na is transformed into HCl-soluble Na during combustion. Insoluble Na remains almost the same as the temperature increased for coal 1, but increases slightly for both coal 2, coal 3 and coal 4 according to Fig. 6(b)-(c). Thus, a small component of H<sub>2</sub>O-soluble Na is also possibly transformed into insoluble Na by reacting with silica, silicates or alumina-silicates.

Finally, there is a difference between the transformation reactions between H<sub>2</sub>O-soluble Na and HCl-soluble Na in terms of the reactivity, and its order is coal 3 > coal 4/coal 2 > coal 1. At a temperature of 1000 °C, HCl-soluble Na increases by 58%, 39%, 35% and 21% for coal 3, coal 4, coal 2 and coal 1, respectively. The reactivity order corresponds with the order of released Na content as shown in Fig. 5 (a). This also proves that the released Na content is related with the transformation reactions.

### 3.4 Equilibrium calculation on transformation behavior of Na species

Fig. 7 shows the equilibrium calculation results on the transformation behavior of Na species. In the figure, “s” represents the solid state, “g” represents the gaseous state and “slag” represents the molten state. At a temperature of 500~1200 °C, the total content of gaseous Na species including NaCl is low. As shown in Fig. 7(a), Na exists mainly in the form of albite (NaAlSi<sub>3</sub>O<sub>8</sub>) at temperatures below 850 °C. Albite and other Na-compounds will lead to eutectic reactions taking place easily; thus, the ash fusion temperature decreases and the ash deposition problem is aggravated. When the temperature reaches 900 °C, Na<sub>2</sub>O (slag) appears and albite disappears. This is ascribed to the following reaction:



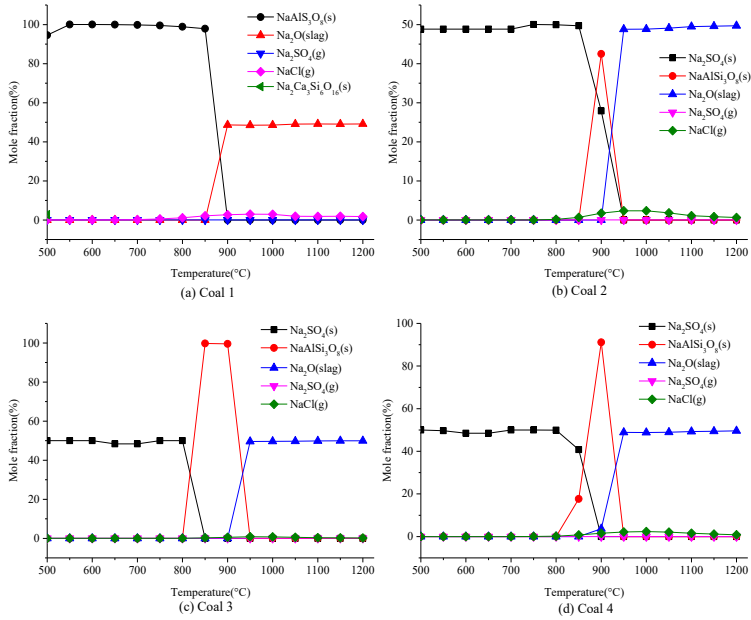
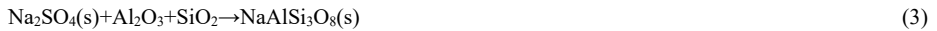


Fig. 7 Transformation behavior of sodium species

However, the main Na species is  $\text{Na}_2\text{SO}_4(\text{s})$  at relatively low temperatures according to Fig. 7(b), Fig. 7(c) and Fig. 7(d). As shown in Table 1, the content of sulfur in coal 1 is only 0.51%, and in coal 2, coal 3 and coal 4, it is 1.73%, 2.67% and 1.87%, respectively. It can be inferred that Na species are prone to reacting with  $\text{SO}_2/\text{SO}_3$  to form  $\text{Na}_2\text{SO}_4$  rather than alumina-silicates at relatively low temperature ( $<850^\circ\text{C}$ ), when the content of sulfur is comparatively high. Additionally,  $\text{NaAlSi}_3\text{O}_8(\text{s})$  appears approximately 850–900 °C accompanied with  $\text{Na}_2\text{SO}_4$  disappearing. The relative reaction is:



$\text{NaAlSi}_3\text{O}_8(\text{s})$  then disappears when the temperature increased above 900 °C, according to the reaction (2). It should be noted that the mole fractions of generated albite for coal 2, coal 3 and coal 4 at 900 °C are different. The mole fraction is almost 100% for coal 3, while it is 42% and 91% for coal 2 and coal 4, respectively. Therefore, combined with the content of Si and Al shown in Fig. 5, it can be concluded that significant Si and Al result in increasing amounts of generated Na-containing alumina-silicates.

#### 4. CONCLUSIONS

To investigate the transformation behavior of Na for high-alkali coals, the combustion experiments were performed in an electrically heated tube reactor, while the chemical sequential method was applied to analyse the existing forms of alkali metals in coal/ash. Additionally, the FactSage 5.2 software program was used to simulate the transformation behavior of Na species. The four tested coals include one Zhudong coal and three Taer coals. The results show that  $\text{H}_2\text{O}$ -soluble Na is the main existing form in the coal and insoluble Na is the least form. The existing form of K is different with that of Na. Insoluble K is the majority part for Zhudong coal, but the contents of the different K forms are close for Taer coals.

During the coal combustion, the temperature has significant influence on the release of alkali metals. The higher temperature will result in more alkali metals being released. Furthermore, the released Na comes from  $\text{H}_2\text{O}$ -soluble Na and  $\text{CH}_3\text{COONH}_4$ -soluble Na, while  $\text{H}_2\text{O}$ -soluble Na is the majority constituent. Part of the  $\text{H}_2\text{O}$ -soluble Na might be transformed into HCl-soluble Na and insoluble-Na by reacting with silicates/alumina-silicates.

Despite the high Na content, its volatility is relatively low in the four tested coals. As a whole, the Na released during combustion was 12%–35% at a temperature of 500–1000 °C. There are two reasons for the

low volatility of the Na. On the one hand, the coals have low Cl content, and thus the promotion effect of Cl on the Na release is limited and can be ignored. On the other hand, Si and Al in coal will react with volatilized Na, resulting in a decrease of released Na.

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