

EFFECTS OF TEMPERATURE AND ADDITIVES ON ASH TRANSFORMATION AND MELTING OF HIGH-ALKALI-CHLORINE COAL

by

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The high contents of sodium and chlorine in Shaerhu coal aggravate severe slagging ash deposition and corrosion in boilers. Adding proper additives is an effective way to reduce slagging ash deposition and corrosion. Based on the experimental study, this paper investigated the effect of combustion temperature, types of additives, and its amount on the ash transformation and melting of high sodium chloride Shaerhu coal. The ash melting characteristics, elemental compositions and mineral compositions of the ash produced under different conditions were characterized. Results showed that the contents of calcium, magnesium, and sulphur in the coal ash varied slightly with the temperature increasing. Chlorine and sodium released rapidly from coal at 550-815 °C and 550-700 °C, respectively. At 1050 °C, calcium silicate was observed as the main component of the ash. The addition of different additives had no significant effect on chlorine and sodium capture. The addition of silicon species lowered the ash fusion temperatures, while the aluminum had an opposite effect. To prevent the furnace from slagging, a high aluminum additive is proposed to be adopted.

Keywords: *high sodium coal, high chlorine coal, ash melting, ash deposition, mineral transformation, additives*

Introduction

The reserves of Zhundong (ZD) coalfield and Shaerhu (SHE) coal are tremendous in quantity in China [1], especially in Xinjiang, reaching to 390 Gt, 89.2 Gt, respectively [2]. They have similar characteristics like massive reserves, low ash and sulphur (S) contents, good grindability, high volatile contents, and high sodium contents [3]. However, the burning of ZD and SEH coal causes severe slagging and fouling problems in coal-fired boilers [4], due to the high contents of sodium (Na), calcium (Ca), potassium (K), iron (Fe), chlorine (Cl), and S which can be released into the flue gas at high temperature [5]. A high amounts of sulphates of Na and Ca were observed in the ash deposits on the heating surfaces of a 350 MW boiler

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burning ZD coal [6]. Qi *et al.* [7] found that Cl in the flue gas aggravates the deposition/fouling on air-cooled surfaces. In particular, high Cl content in SEH coal can cause corrosion in the boiler heating surfaces [8], which are primarily caused by gaseous corrosive media [9], including HCl(g), NaCl(g), and Cl₂(g) [10]. Wang *et al.* [11] observed a high degree of particle deposition on the boiler surfaces when coal is co-fired with agriculture residues due to their high sodium contents. Tyni *et al.* [12] concluded that the formation of low-melting phases in ash is caused by the high alkali metal contents. For the safe and efficient use of SEH coal, it is crucial to analyze the ash-related problems caused by the SEH coals during combustion.

Several studies have been conducted on the evolution of Na and Cl in coal. Quyn *et al.* [13] found that the sodium in the form of NaCl is more volatile than that in the form of carboxylate in the coal. Hancock [14] performed corrosion experiments in the presence of pure NaCl and fly ash, and found that steel starts oxidation continuously at 500 °C in the presence of NaCl, which is much less than the melting point of NaCl (801 °C). Li *et al.* [15] concluded that Cl plays a key role in the release of alkali metals from the coal. Since Cl is highly volatilized in the range of 600-800 °C, it can combine Na and then released as NaCl. The Na is more vulnerable to reaction with other minerals, silica (SiO₂) and alumina (Al₂O₃), to form aluminosilicates remaining in ash, while Cl in the forms of HCl, Cl₂, NaCl are readily evaporated into the flue gas. Blasing and Muller [16] reported that the coal composition and reaction atmosphere have significant effects on the release of Na, K, Cl, and S species. Oleschko and Muller [17] investigated that Na and K species are released mainly in the form of NaCl and KCl during the combustion products of German brown coals, and their release depends largely on the Cl content of the coals.

In addition to the evolution of Na and Cl, the melting characteristics of coal ash are also the key parameters reflecting the fouling and slagging problems, which are mainly influenced by the mineral composition [18]. Ash fusion temperatures (AFT) of coal ash with high SiO₂ and Al₂O₃ contents decreases significantly with the increase of sodium oxide (Na₂O) contents due to the formation of fusible minerals, such as albite, nepheline, and the eutectics [19]. Albite and nepheline are formed when ash contains about 83% SiO₂+Al₂O₃ and 5-20% Na₂O, which leads to a decrease of AFT [20]. Vassilev *et al.* [21] observed that the high contents of Ca, Na, Mg in coal ash lowered AFT, while the increase of Si and aluminum (Al) contents have the opposite effect, *i. e.* increased the AFT. Jing *et al.* [22] identified the low melting point material due to the presence of Fe, Na, and Ca contents which leads to the decrease of AFT. Hence, there have been many studies to improve AFT through additives, Niu *et al.* [23] concluded that lime and kaolin additives effectively increased AFT, but sodium carbonate (Na₂CO₃) and SiO₂ additives demonstrated V shaped functions between the AFT and additive amount for the ZD coal ash. It was observed that when the ratio of Si-Al additives increases up to 9%, the resulted AFT can higher than the ones of raw coal ashes [24]. Wei *et al.* [25] found that the additives (SiO₂, kaolin, and fly ash) are capable of capturing Na, and among these additives, SiO₂ has the best Na capture ability. Wang *et al.* [26] studied the effect of aluminum additives (Al₂O₃, SiO₂, kaolin, and diatomite) on the chemical form transformation of Na, Ca, and Fe, and found that the Si-Al additives promoted the conversion of a large amount of water-soluble Na to an insoluble form. The aforementioned studies have already concluded that it is an efficient method for improving AFT and curing Na by changing the composition of ash with additives.

In summary, the evolution of Na and the ash fusion characteristics have been widely conducted for ZD coal. However, the properties of SEH coal are different, with extremely

high Na content in coal ash (10%), and extremely high Cl content in coal as-received basis (>1%). The slagging and fouling problems from burning SEH coal will be much more aggravated compared with ZD coal. In comparison with the huge amount of studies on ZD coal, there are still not enough specific studies on the ash melting and mineral evolution for SEH coal. In terms of reducing the slagging and fouling in boilers burning SEH coal by Si-Al additives with an economic cost, it is also suggested to use the additives nearby with cheap transportation expenses.

In this paper, the effect of Si-Al additives on the AFT of SEH coal is studied for a circulating fluidized bed (CFB) boiler burning SEH coal, in Xinjiang province, China. Five types of additives (coal cinder, aluminum ash, kaolin, desert sand, and washed sand) nearby the SEH coal mine are collected and used in this study to control the blending cost. The ash melting temperature, elemental composition, and mineral compositions are characterized by ash melting-point equipment, X-ray fluorescence (XRF), and X-ray diffraction (XRD), respectively. A CaO-SiO₂-Al₂O₃ pseudo-ternary phase diagram based on thermal equilibrium calculation is adopted to analyze the fusion characteristics of major compositions. The effects of additive types and addition ratios on the fusion characteristics and mineral transformation are mainly studied. The results from this study are expected to suggest the additive type and ratio to reduce the slagging and fouling problems of CFB boilers burning SEH coal.

Experimental section

In this section, properties of coal, *i. e.* proximate and ultimate analysis, the composition of coal ash and additives, ash preparation and characterization method are illustrated.

Properties of coal and additive samples

The SEH coal and five kinds of additives, Dihua (DH) coal cinder, aluminum ash, kaolin, desert sand, and washed sand, are used. The DH coal cinder, desert sand, and washed sand are purchased from the area near the factory. The location where these additives are available is shown in fig. 1.

To illustrate the characteristics of SEH coal, a typical ZD coal is considered for comparison. The proximate analysis, ultimate analysis, and heating values, respectively, are shown in tab. 1, while the chemical compositions of SEH coal ash and five additives are depicted in tab. 2. The chlorine content of SEH coal as shown in tab. 2, is significantly higher than the ZD coal as shown in tab. 1. Calcium oxide (CaO), Na₂O, and Cl contents in SEH coal are as high as 38.1%, 9.5%, and 9.2%, while the contents of SiO₂ and Al₂O₃ is as low as 11.2% and 5.3%. While the additives chemical compositions have the opposite trend, with a high Si content and a low Ca content. The chemical compositions of desert sand and washed sand are almost similar, which can be considered as the same category materials.

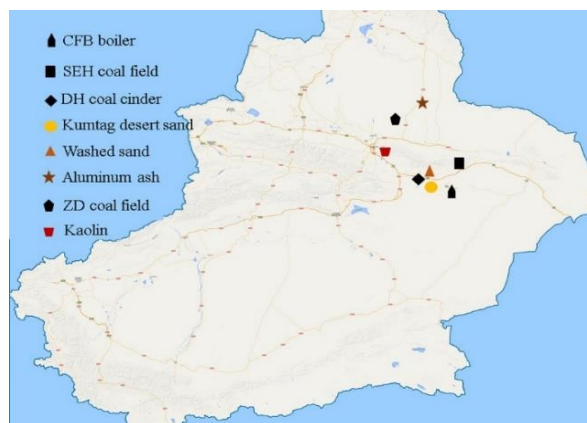


Figure 1. Distribution of coal field, CFB boiler, and additives used in this study (Map of Xinjiang province, China)

Table 1. Proximate and ultimate analyses of coal samples

Samples	Proximate analysis [wt.%]				Ultimate analysis [wt.%]					$Q_{gr,d}$ [MJkg ⁻¹]
	M_{ad}	V_{ad}	FC_{ad}	A_{ad}	C_{ad}	H_{ad}	N_{ad}	S_{ad}	Cl_{ad}	
SEH coal	14.7	28.54	53.96	5.27	61.63	4.09	0.67	0.39	0.468	22.31
ZD coal	8.55	29.41	57.67	4.37	53.76	2.79	0.37	0.42	0.056	27.72

Note: ad – air-dried basis, d – dry basis, gr – gross calorific value

Table 2. Compositions of coal ash and additives

Component	K ₂ O	Fe ₂ O ₃	MgO	SO ₃	SiO ₂	Al ₂ O ₃	Cl	Na ₂ O	CaO	CO ₂
SEH coal ash	0.4	5.3	5.0	2.1	11.2	5.4	9.2	9.5	38.1	12.7
DH coal cinder	1.6	6.6	2.8	6.7	40.6	24.8	0.5	2.2	11.8	0.155
Aluminium ash	5.56	0.32	8.83	0.34	1.5	71.3	5.17	2.59	0.79	-
Kaolin	1.91	1.66	0.63	2.31	46.20	20.90	-	0.41	0.9	24
Desert sand	3.23	4.5	2.01	0.17	65.7	13.8	0.04	3.45	5.4	0.48
Washed sand	3.1	6.5	2.54	0.096	59.4	15.6	0.03	3.8	6.46	0.96

Ash preparation and characterization methods

The raw coal is dried and milled < 200 μ m [4]. Ash is prepared at low temperatures to inhibit the release of Na and Cl from the coal. Coal and additives are initially heated up to 300 °C by using muffle stove at a rate of 10 °C per minute and kept it for 30 minute, then heated to 550 °C at a rate of 10 °C per minute and held it for 2 hours. Additives are mixed with the coal ash at 25%, 50%, and 75% by mass basis. Then the well-mixed sample is initially heated to 550 °C at a rate of 10 °C per minute and keep it for 30 minute before rising to the target temperature, then the sample is heated to the target temperature (700 °C, 815 °C, 950 °C, and 1050 °C) at a rate of 10 °C per minute and again keep it for 2 hours.

The AFT is measured under a reducing atmosphere according to the Chinese standard procedures (GB/T 219-2008) [4]. A mixture of CO (60 $\pm$ 5)% and CO₂ (40 $\pm$ 5)% is introduced into the furnace to control the weak reducing atmosphere. Ash samples are shaped into triangular pyramids and heated at 20 °C per minute up to 900 °C and then heated to 1500 °C at a rate of 5 °C per minute. The four characteristics of the melting temperature: deformation temperature (DT), softening temperature (ST), hemisphere temperature (HT), and fluid temperature (FT) are analyzed by observing the ash cone morphological changes in the heating process.

The qualitative and semi-quantitative elemental analysis is performed using XRF (S4-Pioneer, Bruker Co., Germany). The main crystalline compounds in ash samples and additives samples are identified by XRD using a D/max 2400X power diffractometer (Netherlands) by providing 40kV and 40mA electricity. Peak identification is performed by comparison with the reference standards of the JADE6 software package.

Results and discussion

In this section, based on the experimental study the ash fusion, mineral transformation of high NaCl coal, effect of combustion temperature, types of additives and its amount on the ash transformation and melting of high NaCl are discussed. The effect of five additives on ash slagging and deposition in a full-scale CFB boiler is described.

Ash slagging and deposition in a full-scale CFB boiler

Figure 2 shows that coal ash and bed material formed large slags inside the furnace. The slag sample is in a molten state with no apparent stratification, and the area of the slag block is 0.8 m². The slagging area locates in the furnace dense phase area, where the flue gas temperature is in the range of 850-920 °C. It can be inferred that when the boiler operates in this temperature range, some ash particles in the flue gas are either melted or semi-melted. These particles are highly adhesive and formed adhesive layers to which other particles bind and grow, causing slagging, which can lead to poor fluidization and serious safety hazards in the furnace.

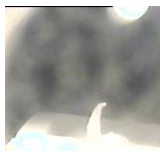
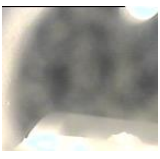


Figure 2 Slags in CFB boiler wall

Ash fusion and mineral transformation of high sodium and chlorine coal

Table 3 shows that the AFT of SEH coal are not low. The DT is 1279 °C and the ST of coal ash has a higher value as 1365 °C. The FT is only 40 °C higher than the ST.

Table 3. The SEH coal AFT

AFT	DT [°C]	ST [°C]	HT [°C]	FT [°C]
Image				
Temperature	1279	1365	1368	1405

The elemental release for 1 kg fuel under different conditions is calculated:

$$R_{e,T} = 1000 \frac{A_{550\text{ °C}} C_{e,550\text{ °C}} - A_T C_{e,T}}{1 - M} \quad (1)$$

Assumption: Na, Cl, Ca, Mg, and Al are not released when burning at 550 °C. The part $A_{550\text{ °C}} C_{e,550\text{ °C}}$ means the initial mass of an element in 1 g of a mixed sample, and $A_T C_{e,T}$ means the mass of element at T , so $A_{550\text{ °C}} C_{e,550\text{ °C}} - A_T C_{e,T}$ means the elemental release for 1 g mixed sample. It is assumed that the additives are stable and cannot release the element under 1050 °C. The part $(A_{550\text{ °C}} C_{e,550\text{ °C}} - A_T C_{e,T}) / (1 - M)$ means the elemental release for 1 g fuel. Symbol $R_{e,T}$ [g/kg] is the mass of the release element from 1 kg of raw coal under different conditions, e is any element such as Na, Cl, Ca, and etc., $A_{550\text{ °C}}$ is the ash yield at 550 °C, $C_{e,550\text{ °C}}$ is the mass content of e element in residual ash at 550, A_T is the ash yield at T , $C_{e,T}$ is the mass content of e element in residual ash at T , M is the percentage addition ratio, and T is the burned temperature °C.

As can be seen from fig. 3, the contents of Ca, Mg, and S in the coal ash hardly changes with the increasing of temperature. The Ca and Mg may not be released at a temperature less than 1050 °C, while the release of S is comparatively low. So, the release of Na and Cl is the focus of researchers. The Cl element is rapidly evaporated at 550-815 °C and stops after 815 °C which means that Cl element released completely up to 815 °C. The Na element is released quickly in the range of 550-700 °C and slowly released at 700-1050 °C.

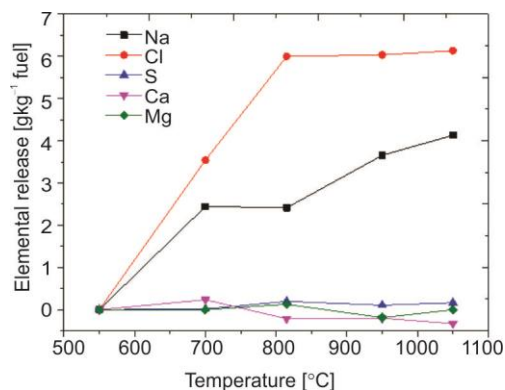


Figure 3. Elemental release per kilogram of SEH coal

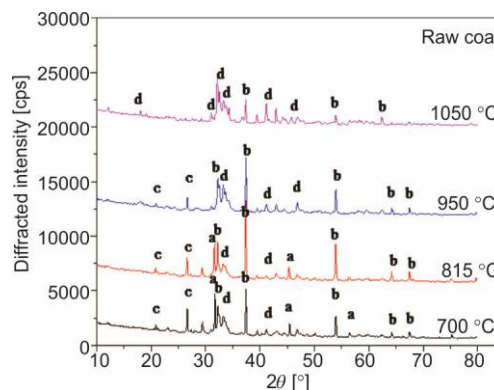


Figure 4. The XRD patterns of raw SEH coal ash at different temperatures (a – NaCl, b – CaO, c – SiO₂, d – Ca₂SiO₄)

Figure 4 shows the XRD trends of SEH coal ash at 700 °C, 815 °C, 950 °C, and 1050 °C. Different peaks in the XRD trends indicate different kinds of minerals, while the intensity represents the mineral's relative content qualitatively [4]. It can be seen that NaCl pattern appears at 700 °C and disappears at 950 °C. The SiO₂ and CaO were also found in the ash at less than 950 °C. When the temperature increases to 1050 °C, the Ca₂SiO₄ peak increases while CaO and SiO₂ peak reduces. The primary transformation path may follow:



Effect of additives on ash fusion

By the ternary phase diagram method, the locations of different mixed coal ash are shown in fig. 5. The addition of aluminum ash caused the mixed coal ash to fall in the mullite

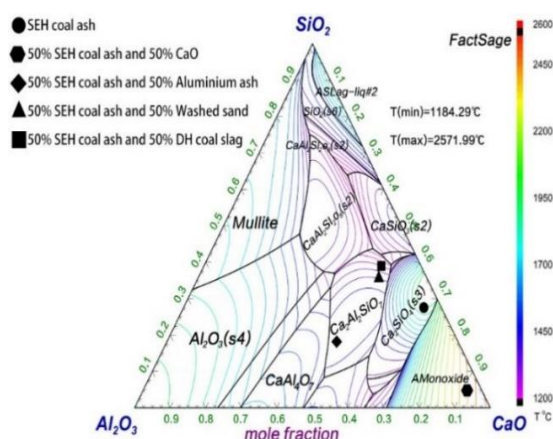


Figure 5. The CaO-SiO₂-Al₂O₃ pseudo-ternary phase diagram

area and is advantageous for increasing the ash melting point. The additions of DH coal cinder and washed sand caused the mixed coal ash to fall in the area of gehlenite (Ca₂Al₂SiO₇) and reduces its ash melting point. The bottom lake coal slag and the washed sand are much closed to each other in the phase diagram, and the effect on the ash melting point is similar.

Table 4 shows the AFT of 50% SEH coal ash and 50% additives mixture. Only aluminum ash is more advantageous in terms of the coal ash AFT, while all the other additives have an adverse effect on AFT. The ST and FT for all the four additives are less than

1230 °C and 1250 °C, respectively. The DH coal cinder has a better effect than desert sand and washed sand, but worse than the aluminum ash.

Table 4. The AFT of 50% SEH coal ash and 50% additives

Additives	DT [°C]	ST [°C]	HT [°C]	FT [°C]
The DH coal cinder	1182	1229	1230	1241
Desert sand	1172	1204	1206	1211
Washed sand	1145	1197	1200	1214
Kaolin	1159	1192	1200	1203
Aluminium ash	1253	1412	1426	1443

The effects of washed sand addition on ash fusibility are shown in fig. 6 indicating that with the increase of washed sand blending ratio, AFT first decrease and then increase, the lowest AFT is observed with 75% blending ratio, *e.g.*, the ST which is 1181 °C. The difference between ST and DT is about 70 °C, which indicates a sticky melt, which may lead to significant slagging problems.

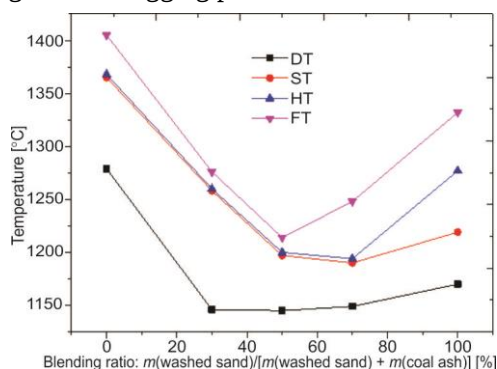


Figure 6. The AFT of SEH coal ash with washed sand

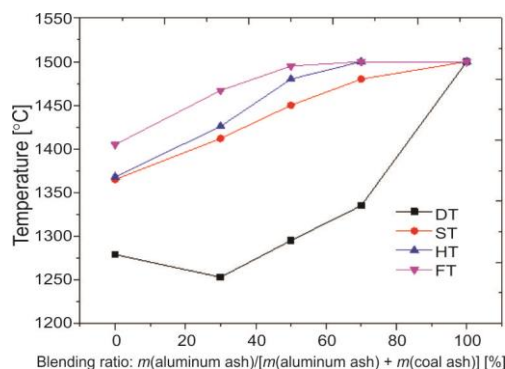


Figure 7. The AFT of SEH coal ash with aluminium ash

The effect of aluminum ash addition on fusion temperatures of SEH coal ash is shown in fig. 7. With the increase in the blending ratio, the AFT increase continuously. It can be seen that there is a significant difference between the four characteristic temperatures. The ST of aluminum blended sample is higher than that of the washed sand. Therefore, adding aluminum ash will be more helpful to prevent serious slagging problems for the higher AFT. The four characteristic temperatures are 1500 °C in pure aluminum ash, although the AFT are higher than 1500 °C.

The effect of kaolin and desert sand addition on SEH coal ash fusion is shown in figs. 8 and 9, respectively. With the gradual increase in kaolin or desert sand, AFT first decrease and then increase. Among these three additives (kaolin, desert sand and washed sand), kaolin addition is the worst one with the lowest AFT.

From the results shown in figs. 6-9, it is concluded that the addition of silicon species might further reduce AFT due to the high CaO contents in SEH coal ash, while the addition of aluminum species may increase AFT. To avoid slagging of a furnace, it is recommended to use high aluminum additives.

Effect of additives on ash mineral transformation

Figure 10 shows the mass contents of sodium release from 1 kg of raw coal prepared under different conditions. Washed sand, in which sodium is present intrinsically, released a hi-

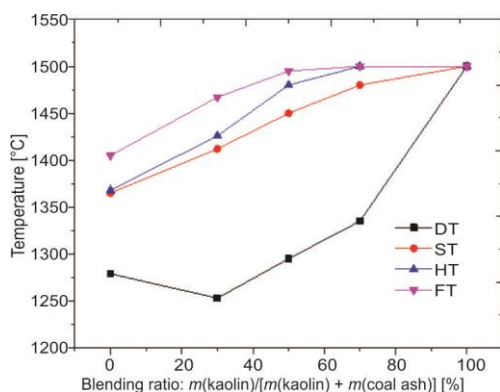


Figure 8. The AFT of SEH coal ash with kaolin

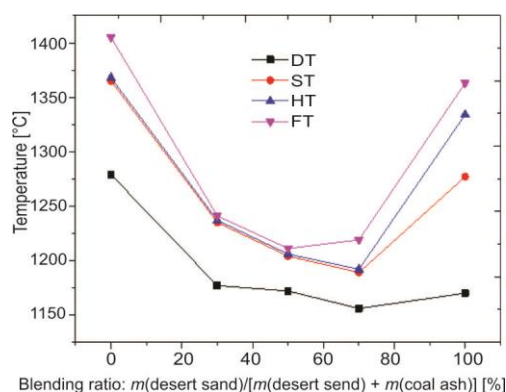


Figure 9. The AFT of SEH coal ash with desert sand

gher quantity of sodium per kg of coal when mixed with the SEH coal. Besides the washed sand, DH coal cinder enhanced the release of sodium at a lower temperature. It can be observed that at 1050 °C, all of these additives have no significant effect on the ability to capture sodium. Figure 11 demonstrates the mass content of chlorine release. All of these additives have no significant effect on chlorine capturing ability, the chlorine is always released at a normal rate with temperature and is not affected by these additives.

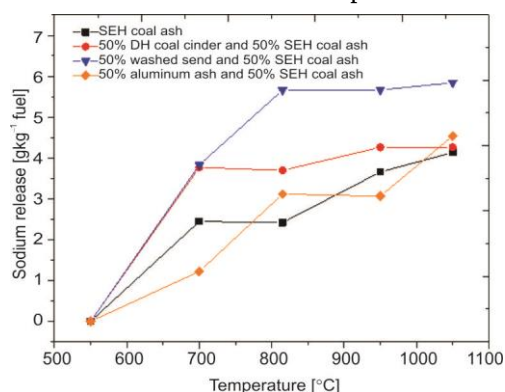


Figure 10. Sodium release per kilogram of SEH coal under different additives

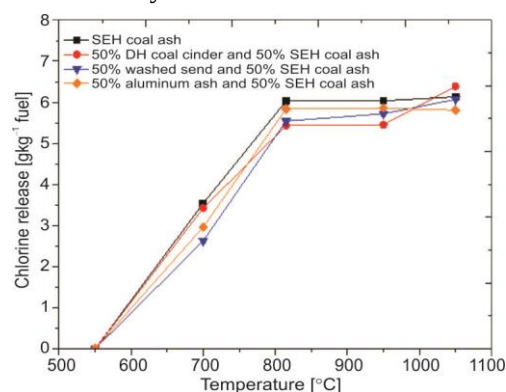


Figure 11. Chlorine release per kilogram of SEH coal under different additives

The XRD patterns of SEH coal ash with washed sand at different temperatures are shown in fig. 12. It can be seen that Ca_2SiO_4 and SiO_2 patterns appear to be the dominant phase at less than 815 °C. Apart from them, $\text{Ca}_2\text{Al}_2\text{SiO}_7$ and $(\text{Ca},\text{Na})(\text{Al},\text{Si})_4\text{O}_8$ were also found in the ash at less than 815 °C. When the temperature increases to 1050 °C, $\text{Ca}_2\text{Al}_2\text{SiO}_7$, and $(\text{Ca},\text{Na})(\text{Al},\text{Si})_4\text{O}_8$ become the dominant phase.

Figure 13 shows the XRD patterns of SEH coal ash with aluminum ash addition. The mineral composition of adding aluminum ash is more complex than that of adding washed sand. From 700 °C to 1050 °C, it seems that the mineral composition has not changed. The Al_2O_3 , CaSiO_4 , MgAl_2O_4 , and $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ become the dominant phase in ash. MgAl_2O_4 and CuAl_2O_4 belong to aluminum ash and have no chemical reaction with the coal ash.

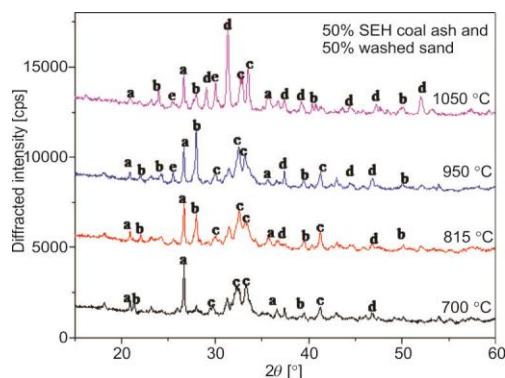


Figure 12. The XRD patterns of 50%SEH coal ash and 50% washed sand at different temperatures (a – SiO_2 , b – $(\text{Ca,Na})(\text{Al,Si})_4\text{O}_8$, c – Ca_2SiO_4 , d – $\text{Ca}_2\text{Al}_2\text{SiO}_7$, e – CaSiO_3)

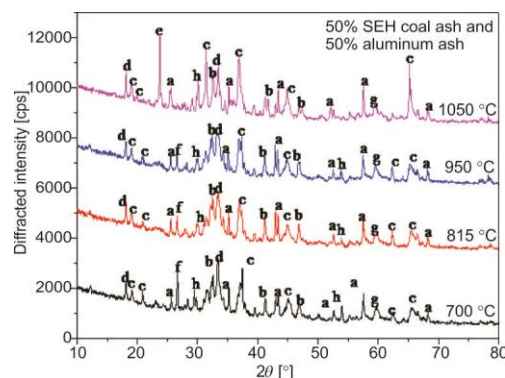


Figure 13. The XRD patterns of 50%SEH coal ash and 50% Aluminium ash at different temperatures (a – Al_2O_3 , b – CaSiO_4 , c – MgAl_2O_4 , d – $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$, e – $\text{Ca}_2\text{Al}_2\text{SiO}_7$, f – SiO_2 , g – CuAl_2O_4 , h – CaO)

Conclusions

In this paper, the influence of additives (DH coal cinder, aluminum ash, kaolin, desert sand, and washed sand), ash fusion characteristics, mineral transformation, and elemental release of SEH coal ash were investigated. The main conclusions are as follows.

- In pure SEH coal ash, the AFT were higher *i. e.*, DT was 1279 °C, and ST was 1365 °C.
- When the additives were mixed with SEH coal, the AFT of the mixture were reduced very sharply.
- In a wide range of blending ratio, the addition of silicon species lowered the AFT, while the aluminum had an opposite effect.
- The addition of a high quantity of aluminum significantly reduced the slagging effect in the boilers.
- To prevent the furnace from slagging, a high aluminum additive is proposed to be adopted.
- The contents of Ca, Mg, and S in the coal ash varied slightly with the temperature increasing.
- The Cl and Na released rapidly from coal at 550 to 815 °C and 550 to 700 °C, respectively.
- Although, the released rate of Na from 700 °C to 1050 °C was not significant.
- At 1050 °C, Ca_2SiO_4 was observed the main component of the ash.
- All additives (DH coal cinder, aluminum ash, and washed sand) had no significant effect on capturing ability of chlorine and sodium in the temperature range of CFB boiler.

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