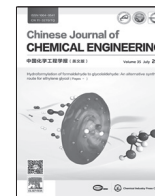




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Full Length Article

Modification of ash flow properties of coal rich in calcium and iron by coal gangue addition

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ABSTRACT

Flow property of coal ash and slag is an important parameter for slag tapping of entrained flow gasifier. The viscosity of slag with high contents of calcium and iron exhibits the behavior of a crystalline slag, of which viscosity sharply increases when temperature is lowered than temperature of critical viscosity (T_{CV}). The fluctuation in temperature near the T_{CV} can cause an accumulation of slag inside the gasifier. In order to prevent slag blockage, it is necessary to adjust the ash composition by additive to modify the flow property of coal rich in calcium and iron. Main components of coal gangue are Al_2O_3 and SiO_2 , which is a potential additive to modify the ash flow properties of these coals. In this work, we investigated the ash flow properties of a typical coal rich in calcium and iron by adding coal gangue with different SiO_2/Al_2O_3 ratio. The results showed that the ash fusion temperatures (AFTs) firstly decreased, and then increased with increasing amount of coal gangue addition. Chemical composition of coal ash rich in calcium and iron moved from gehlenite primary phase to anorthite, quartz and corundum primary phases. The slags with coal gangue addition behaved as a glassy slag, of which the viscosity gradually increased as temperature decreased. Besides, a high SiO_2/Al_2O_3 ratio of coal gangue was beneficial to modify the slag viscosity behavior. Addition of coal gangue with a high SiO_2/Al_2O_3 ratio impeded formation of crystalline phases during cooling. This work demonstrated that coal gangue addition was an effective way to improve the ash flow properties of the coal rich in calcium and iron for the entrained flow gasifier.

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1. Introduction

Coal gasification is a feasible way to realize the clean and efficient utilization of coal resources. Compared with other gasification technologies, the advantages of entrained-flow coal gasification are high carbon conversion, low pollution and good flexibility of feedstock [1,2]. Due to the high operating temperature ($>1300\text{ }^\circ\text{C}$) of the entrained-flow coal gasifiers, the ash is discharged as liquid slag from the bottom of the gasifier. In order to ensure smooth tapping of the slag, the ash flow properties of coal including ash fusion and slag viscosity properties must meet requirement for slag tapping. It is generally accepted that flow temperature (FT) of ash should be less than $1400\text{ }^\circ\text{C}$ and the slag viscosity should be $2.5\text{--}25\text{ Pa}\cdot\text{s}$ within the tapping temperature [3,4]. Therefore, the flow property of coal ash and slag plays an important role in the entrained flow gasification.

The ash flow property is largely influenced by its chemical composition. Relationship between ash flow property and chemical compositions can be explained as follows: the acid oxides (SiO_2 , Al_2O_3 and TiO_2) are prone to increase ash fusion temperatures (AFTs) and slag viscosity at high temperature, while the basic oxides (Fe_2O_3 , CaO , MgO , K_2O and Na_2O) lower the AFTs and slag viscosity [5]. Ash of Yibei coal from Xinjiang province has high contents of calcium and iron ($Fe_2O_3 + CaO > 35.00\%$ (mass)), which results in low FT and slag viscosity at high temperature [6]. In addition, the slag exhibits as the behavior of a crystalline slag, of which viscosity sharply increases when temperature is lowered than temperature of critical viscosity (T_{CV}). The fluctuation in temperature near the temperature of T_{CV} can cause an accumulation of slag inside the gasifier. Therefore, Yibei coal cannot be directly used in the entrained-flow coal gasification.

In order to achieve coal that can be processed in entrained flow gasifiers, it is usually blend the coal with additives to produce a feed with appropriate ash flow property [7]. For the ash rich in calcium and iron, additives with high acid oxide content like SiO_2 ,

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Al_2O_3 should be selected, such as sand, silica or kaolin. Kong *et al.* [8] found that the AFTs of coal with high contents of calcium and iron ($\text{Fe}_2\text{O}_3 + \text{CaO} = 64.63\%$ (mass)) firstly decreased with increasing amount of kaolin, then reached a minimum value before increasing again. Slag viscosity at high temperatures increased with increasing kaolin addition, and the slag behaved as a glassy one when kaolin addition was 12%. Hu *et al.* [9] found that sand addition to coal could improve the slag viscosity property of the Jinjitan coal with high contents of calcium and iron ($\text{Fe}_2\text{O}_3 + \text{CaO} = 38.49\%$ (mass)). When sand addition were 20% and 40%, the slag transformed from a crystalline one to a glassy one. Liu *et al.* [10] found that silica addition could be used to adjust viscosity fluctuation behaviors of the coals YL and YCW1# with high content of calcium and low content of silicon by adding 10.62% and 6.30% of SiO_2 .

Coal gangue is one of the solid waste in the process of coal production, the annual yield is about 10% of the coal output. The accumulated stacking amount of coal gangue has reached more than 5 billion tons, and the amount of annual emission is about 100 million tons in China. Spontaneous combustion and pollution of air, water and soil due to coal gangue stacking have become an urgent issue to be solved. Because main components of coal gangue are Al_2O_3 and SiO_2 [11–13], it can be used as an additive to modify the ash flow properties of Yibei coal, which can not only solve the problems caused by coal gangue stacking but also realize the utilization of coal gangue. However, no studies have been reported on modification of ash flow properties of coal rich in calcium and iron by coal gangue addition.

In this work, we investigated the method of coal gangue addition to modify the ash flow properties of Yibei coal. Two coal gangues were selected. The AFTs and viscosity-temperature curves of slag with coal gangue addition were measured by ash fusion detector and high-temperature rotating viscometer respectively. X-ray diffraction (XRD) was carried out to detect the crystalline minerals. Thermodynamic calculation was used to analyze effect of coal gangue addition on phase transformation of ash and slag.

2. Materials and Methods

2.1. Coal and coal gangue

One coal sample from Yibei, Xinjiang province, was selected and denoted as YB. Two coal gangues with high contents of SiO_2 and Al_2O_3 were selected to blend with YB coal, and denoted as Gangue-A and Gangue-B, respectively. The samples were ground to a particle size of less than 0.200 mm. Proximate and ultimate analyses of the samples are shown in Table 1.

The coal and coal gangues were ashed in the muffle furnace at 815 °C according to the Chinese standard GB/T 212–2008. Chemi-

cal compositions of ashes were analyzed by X-ray fluorescence (XRF) and shown in Table 2. Gangue-A has a high $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio, and Gangue-B has a low $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio.

2.2. Measurement of AFTs

The AFTs of ash with coal gangue addition were measured by a 5E-AF4115 fusion analyzer under CO/CO_2 (6:4, volume fraction) atmosphere according to the Chinese standard GB/T 219–2008. The ash cone was heated to 900 °C at 15 °C·min^{−1}, and then increased at 5 °C·min^{−1} to FT. Four characteristic temperatures were recorded: deformation temperature (DT), softening temperature (ST), hemispherical temperature (HT), and flow temperature (FT). The AFTs of the coal and coal gangue are shown in Table 3. The ash of YB coal has high contents of CaO and Fe_2O_3 ($\text{CaO} + \text{Fe}_2\text{O}_3 = 37.35\%$) and low FT (FT = 1300 °C). However, ash of coal gangue has high contents of SiO_2 and Al_2O_3 ($\text{SiO}_2 + \text{Al}_2\text{O}_3 > 94.00\%$), DT of the coal gangue was higher than 1550 °C.

2.3. Slag viscosity measurement

The viscosity-temperature curves of YB coal ash slag with coal gangue addition were measured by Theta high-temperature rotating viscometer under CO/CO_2 (6:4, volume fraction) atmosphere. For the viscosity measurement, the ash was firstly pre-melted in a high temperature furnace, and the final temperature was usually set to 200 °C higher than FT to ensure that the slag can melt completely and form homogeneous liquid.

The pre-melted slag was crushed to less than 5 mm, and placed into the crucible. The sample was heated at 10 °C·min^{−1} to 1400 °C, then at 5 °C·min^{−1} to the target temperature and held for 20 minutes under CO/CO_2 (6:4, volume fraction) atmosphere. The spindle was slowly immersed into the molten slag. The viscosity and temperature were recorded as the temperature decreased at a cooling rate of 3 °C·min^{−1}, which was proved to be a suitable cooling rate for continuous viscosity measurements [14]. When the viscosity was over 200 Pa·s or the torque was beyond the limits, the spindle was raised out from the slag and the experiment was stopped.

2.4. Mineral characterization

The slag after a viscosity test was dried and crushed to less than 0.075 mm for XRD analysis. To detect the crystalline minerals in the slag, the XRD patterns were recorded by a Bruker D2 X-ray powder diffractometer with Cu K_α radiation.

2.5. Thermodynamic calculation

FactSage (version 6.4) was used to calculate the phase equilibrium, mass fraction of the solid phases, and diagram of mineral phases in a multi-component ash system. In this work, the ash system consisted of SiO_2 , Al_2O_3 , Fe_2O_3 , CaO [15,16]. Databases, FACTPS and FToxid, were selected in the calculation. The calculations were performed from 1100 °C to 1700 °C with an interval of 20 °C under CO/CO_2 (6:4, volume fraction) atmosphere.

3. Results and Discussion

3.1. Properties of YB coal ash

Fig. 1 shows the viscosity-temperature curve of the YB coal ash. It can be seen that the slag viscosity gradually increased till the temperature decreased to T_{CV} . The temperatures corresponding to 25 Pa·s and 2.5 Pa·s were 1356 °C and 1439 °C, respectively. However, the slag viscosity abruptly increased rapidly when the

Table 1

Proximate and ultimate analyses of coal and coal gangue (% (mass))

Sample	YB	Gangue-A	Gangue-B
<i>Proximate analysis, ad</i>			
Moisture	17.58	1.18	1.41
Ash	5.00	91.46	73.38
Volatile	30.05	7.10	16.12
Fixed carbon	47.37	0.26	9.09
<i>Ultimate analysis, ad</i>			
Carbon	68.92	0.96	10.91
Hydrogen	2.64	0.75	1.92
Nitrogen	0.62	0.02	12.15
Sulfur ^①	0.71	0.01	0.15
Oxygen ^②	4.53	5.62	0.08

^① Total of sulphur.

^② By difference, ad: air dried.

Table 2

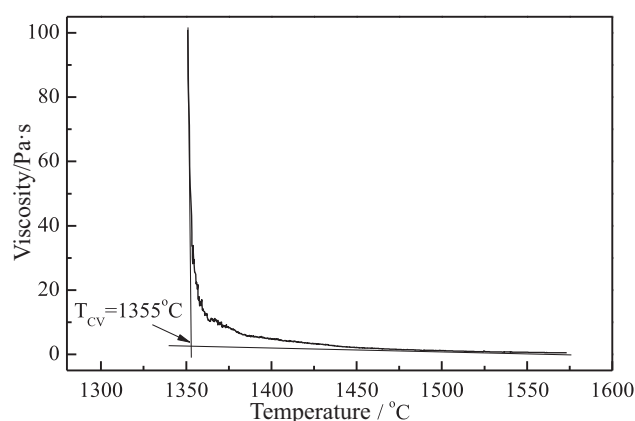
Ash chemical composition of coal and coal gangue (% (mass))

Sample	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	Si/Al
YB	21.06	10.77	12.32	25.03	7.40	18.00	0.27	1.75	1.96
Gangue-A	74.08	20.57	0.88	0.16	0.67	–	2.31	0.17	3.60
Gangue-B	52.11	46.00	0.49	0.11	0.19	–	0.17	0.18	1.13

Si/Al: mass ratio of SiO₂/Al₂O₃.**Table 3**

Ash fusion temperatures of coal and coal gangue (°C)

Sample	DT	ST	HT	FT
YB	1286	1293	1297	1300
Gangue-A	>1550	>1550	>1550	>1550
Gangue-B	>1550	>1550	>1550	>1550

**Fig. 1.** Viscosity-temperature curve of YB coal ash.

temperature lower than T_{CV} , which demonstrated the behavior of crystalline slag. In addition, T_{CV} (1355 °C) was close to the temperature at 25 Pa·s, which was not good for the slag tapping.

In order to explain the slag viscosity of YB coal ash, Fig. 2(a) shows the solid phase formation of YB coal ash. The spinel was firstly separated solid phase when the slag was below 1417 °C. The amount of spinel increased as the temperature decreased. The viscosity of YB increased rapidly below 1355 °C, which indicated the precipitation of spinel led to increase of the viscosity. Kondratiev *et al.* [17] found that formation of spinel lowered Fe, Mg and Al in the liquid phase, and the slag started to show non-

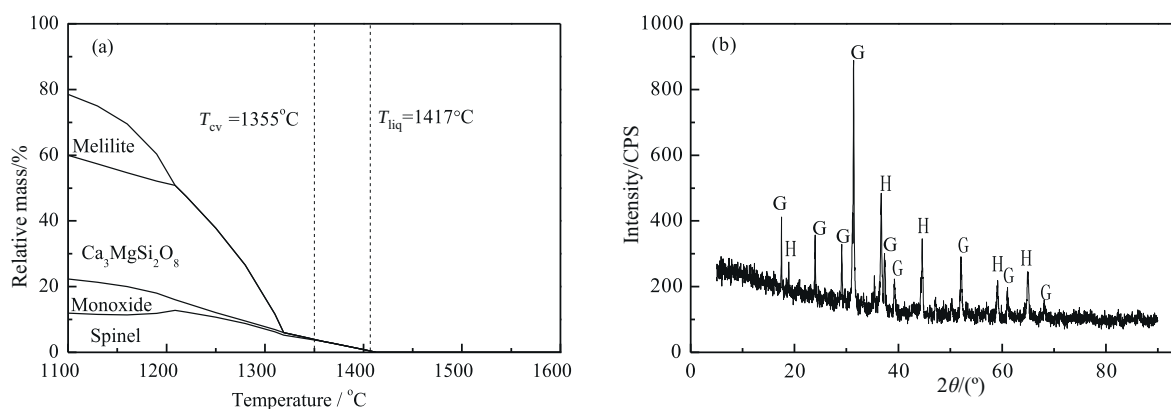
Newtonian behavior with spinel precipitation. Fig. 2(b) shows XRD pattern of YB slag after viscosity test. Oh *et al.* [18] found that formation of crystalline phases during cooling was the main reason for the crystalline slag. It can be seen that gehlenite and hercynite were the crystalline minerals in the slag. That explains why the slag of YB coal ash exhibits the behavior of a crystalline one. Besides, Li *et al.* [19] found that precipitation of gehlenite can cause the slag viscosity fluctuation. It should avoid the formation of gehlenite in the slag to eliminate the viscosity fluctuation problem [20].

In slagging gasifiers with crystalline slags, the fluctuation of operation temperature near T_{CV} can rapidly increase slag viscosity and cause the slag blockage inside the gasifier [21]. Once the slag is blocked, the temperature in gasifier has to be increased to melt the blocked slag. In the worst case, the gasifier has to be shut down so that the blocked slag can be removed by mechanical means. Therefore, it is necessary to add additives to change the slag from a crystalline one to a glassy one.

3.2. Chemical composition of slag vs addition of coal gangue

Silica and alumina are the dominant compositions in coal ash with addition of the coal gangue. Therefore, total of SiO₂ + Al₂O₃ and SiO₂/Al₂O₃ ratio are regarded as the important factors which influence mineral transformation and ash flow properties.

Fig. 3 illustrates effect of coal gangue addition on the total content of SiO₂ + Al₂O₃ and SiO₂/Al₂O₃ ratio of YB ash. When the coal gangue-A (or B) additions were 2%, 4%, 8% and 10%, the total content of SiO₂ + Al₂O₃ of the ash with gangue-A was close to that with the same proportion of that with the coal gangue-B, while the SiO₂/

**Fig. 2.** (a) Phase transformation of YB coal ash and (b) XRD pattern of YB slag after viscosity test. G: Gehlenite (Ca₂Al₂SiO₇); H: Hercynite (FeAl₂O₄).

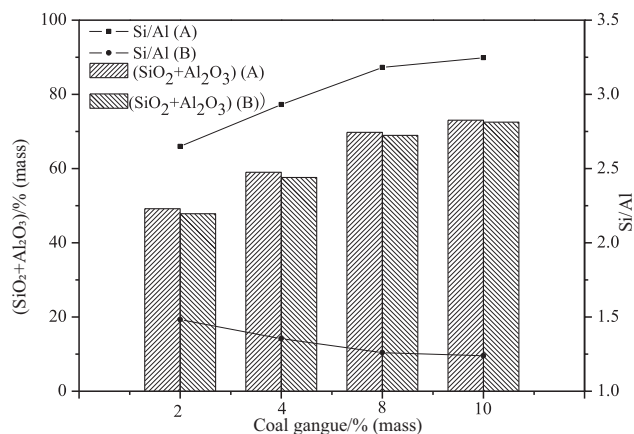


Fig. 3. Total content of $\text{SiO}_2 + \text{Al}_2\text{O}_3$ and $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of YB ash with coal gangue-A (or B) addition.

Al_2O_3 ratio obviously changed, which became the main factor that affect the composition of the ashes with the coal gangue-A(or B) addition. $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio was also one important factor affecting slag tapping in the gasifier [22]. For the ash slag with same content of $\text{SiO}_2 + \text{Al}_2\text{O}_3$, an increase of $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio usually prevents the crystallization of slag and is beneficial to increase the viscosity.

3.3. Effect of coal gangue on AFTs of YB

As shown in Fig. 4, the AFTs firstly decreased with increasing coal gangue addition, and then reached to the minimum value when the coal gangue-A (or B) addition increased up to 2% before increasing once more. When the coal gangue addition was 2%, the FT of ash with Gangue-A (or -B) addition was 1170 °C and 1211 °C, respectively. In addition, when the coal gangue addition more than 2%, the AFTs increased slowly with increasing of gangue-A addition ratio, while they increased rapidly with increasing of gangue-B addition ratio. The FT of ash with 10% gangue-A addition was 1242 °C, while it was 1390 °C with the same proportion of gangue-B. Because coal gangue-A with a high $\text{SiO}_2/\text{Al}_2\text{O}_3$ had the higher content of SiO_2 , it was beneficial for low eutectic with silicate. This demonstrated that addition of gangue B showed a more significant effect on the AFTs of YB coal.

Fig. 5 shows primary phase of YB coal ash with addition of coal gangue by Factsage calculation. The chemical composition of YB coal ash was located in the gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$) primary phase. When the coal gangue-A (or B) addition was 2%, chemical composition of the ash moved to the anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) primary phase. As for the coal gangue-A addition increased up to 14%, it moved into the quartz (SiO_2) primary phase. However, as for the coal gangue-B addition was 10%, it moved into the corundum (Al_2O_3) primary phase. The melting point of gehlenite was 1593 °C, which was higher than that of anorthite (melting point:

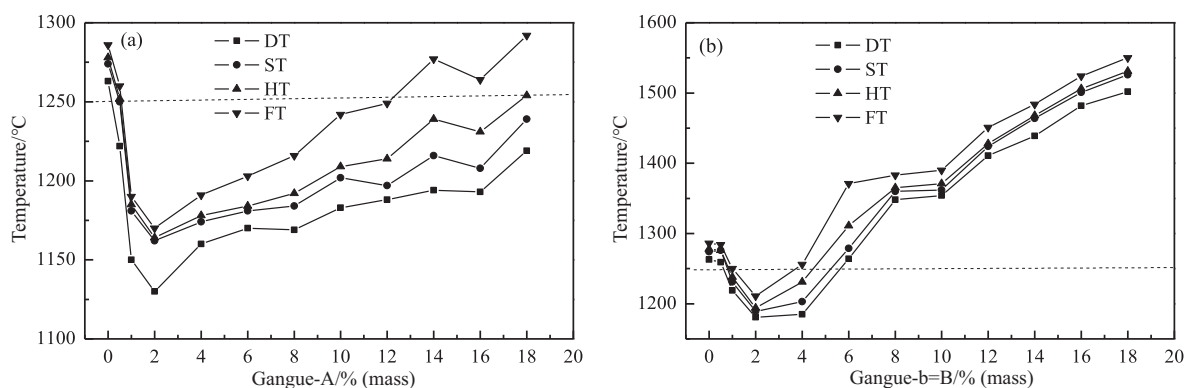


Fig. 4. AFTs of YB with different additions of Gangue-A (or -B).

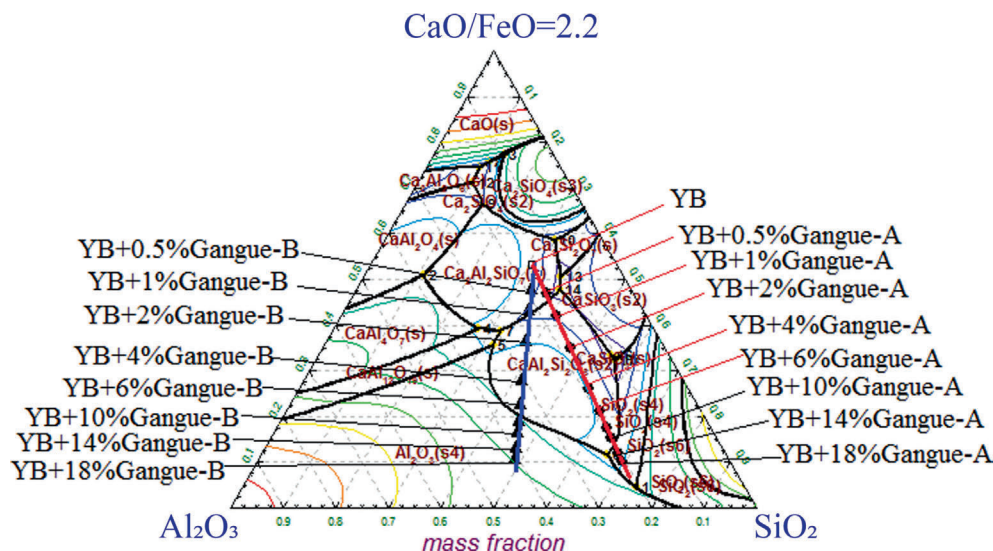


Fig. 5. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-(CaO/FeO)}$ phase diagram calculated by FactSage.

1550 °C) but lower than that of quartz (melting point: 1750 °C) and corundum (melting point: >2000 °C). Therefore, the AFTs firstly decreased, and then increased with increasing coal gangue addition.

3.4. Effect of coal gangue addition on slag viscosity

The viscosity-temperature curves of samples with different gangue addition are shown in Fig. 6. It can be seen that the slag viscosity increased with coal gangue of adding proportion. Coal gangue addition that is beneficial for the slag exhibited the behavior of a glassy slag, particularly coal gangue-A. The slag viscosity increased gradually with temperature decreasing, which was beneficial for smooth slag tapping in the gasifier.

Fig. 6 also shows that the lowest operating temperature when the viscosity was 25 Pa·s increased with increasing coal gangue addition, which was lower when the same addition of gangue-B. When the coal gangue-A addition was 2%, the temperature was 1248 °C, while it was 1204 °C for coal gangue-B. The results indicated that the operating temperature of slag tapping with coal gangue B was lower than that with the same proportion of coal gangue-A.

3.5. Effect of coal gangue on phase transformation

To account for the effect of coal gangue addition on slag viscosity of YB coal, Factsage was used to calculate the solid phase and liquid phase of slag. Fig. 7 gives phase transformation of YB coal ash with coal gangue-A addition. The solid started to deposit when

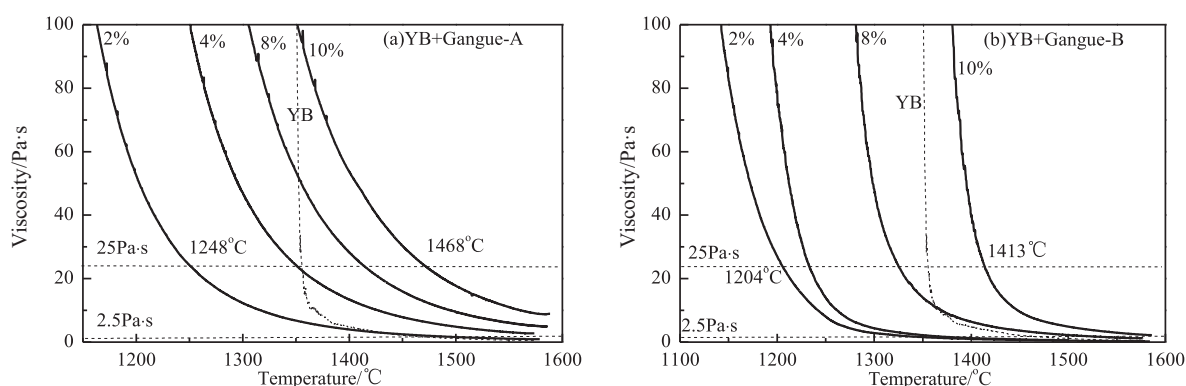


Fig. 6. Viscosity-temperature curves of YB with coal gangue-A (or -B) addition.

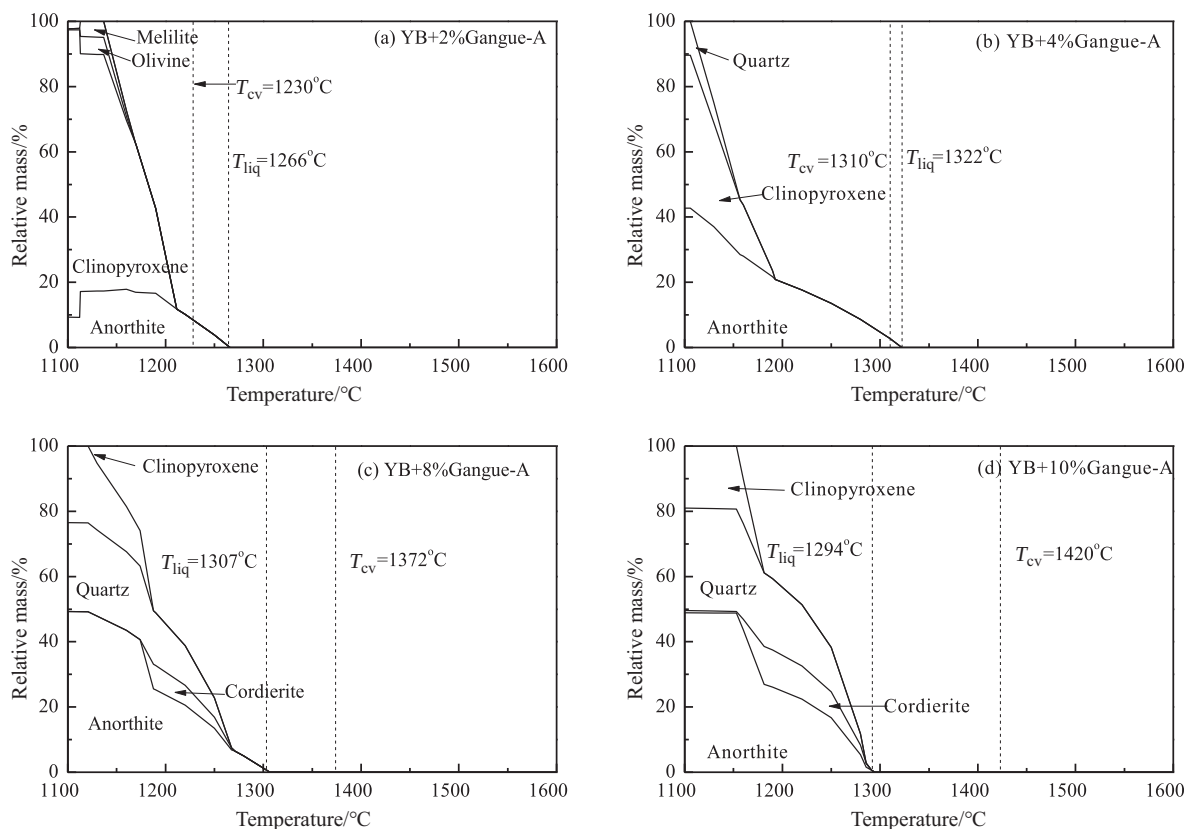


Fig. 7. Phase transformation of YB with different additions of Gangue-A.

the temperature lowered than the liquidus temperature (T_{liq}), the T_{liq} at which the slag was a fully liquid without solid phase. As for the coal gangue-A addition, the anorthite was firstly the separated solid phase instead of spinel. In Fig. 7(a) and (b), the anorthite started to form at 1266 °C and 1322 °C, the viscosity increased rapidly below 1230 °C and 1310 °C respectively, which indicated the separation of anorthite was the main reason for increase of the viscosity. In Fig. 7(c) and (d), the viscosity increased rapidly without solid phase, which indicated change of the slag bulk composition led to increase of the viscosity [23].

Fig. 8 presents phase transformation of YB coal ash with coal gangue B addition. When coal gangue B addition was 2% (Fig. 8 (a)), the spinel was firstly separated solid phase when the temper-

ature was below 1415 °C, but the T_{CV} was lower than before with the coal gangue-B addition. The anorthite started to form at 1250 °C, which was coincided with the T_{CV} . This implied that the precipitation of anorthite led to increase of the viscosity. In Fig. 8 (b), the viscosity increased due to separate of anorthite largely. In Fig. 8(c) and (d), the first solid phase became mullite when the coal gangue-B addition 8% and 10%. The T_{CV} increased due to increase of the mullite with increasing of the coal gangue-B addition. This demonstrated that formation of mullite was the reason for increasing of the viscosity when the coal gangue-B addition more than 8%.

Fig. 9 shows XRD patterns of the slags with addition of coal gangue after viscosity test. In Fig. 9(a), there was no crystalline

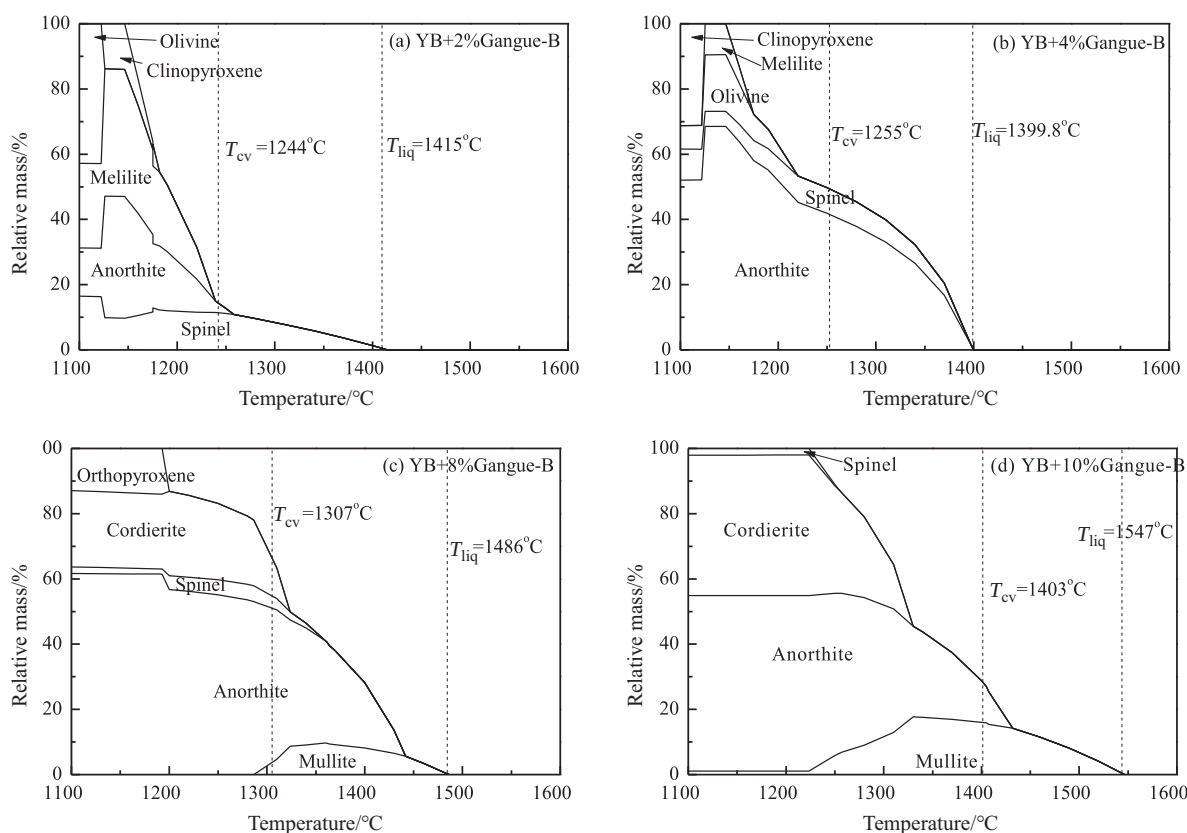


Fig. 8. Phase transformation of YB with different additions of Gangue- B.

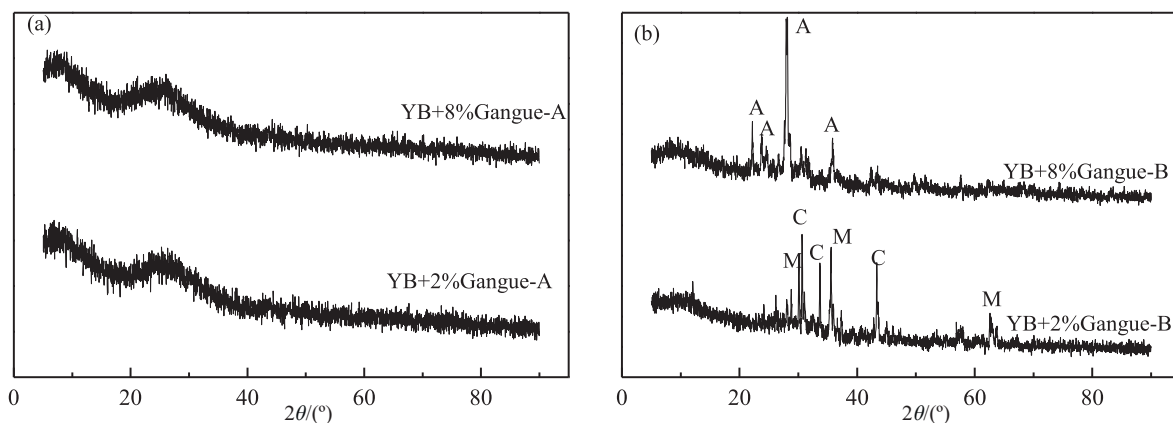


Fig. 9. XRD patterns of the slags with coal gangue-A (or -B) addition after viscosity test. A: Anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$); M: Magnesioferrite (Fe_2MgO_4); C: Calcium Aluminum Iron Oxide ($\text{Ca}_2\text{Fe}_{1.4}\text{Al}_{0.6}\text{O}_5$).

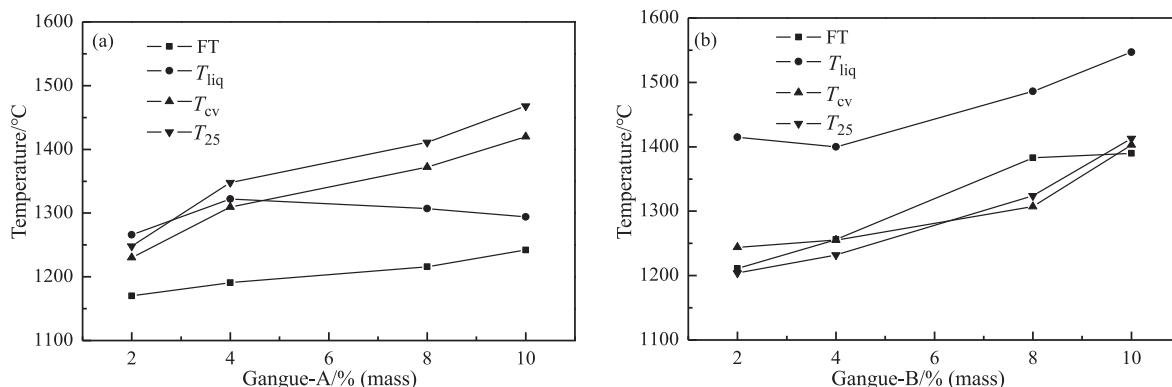


Fig. 10. Slag tapping characteristic temperatures of YB coal with coal gangue addition.

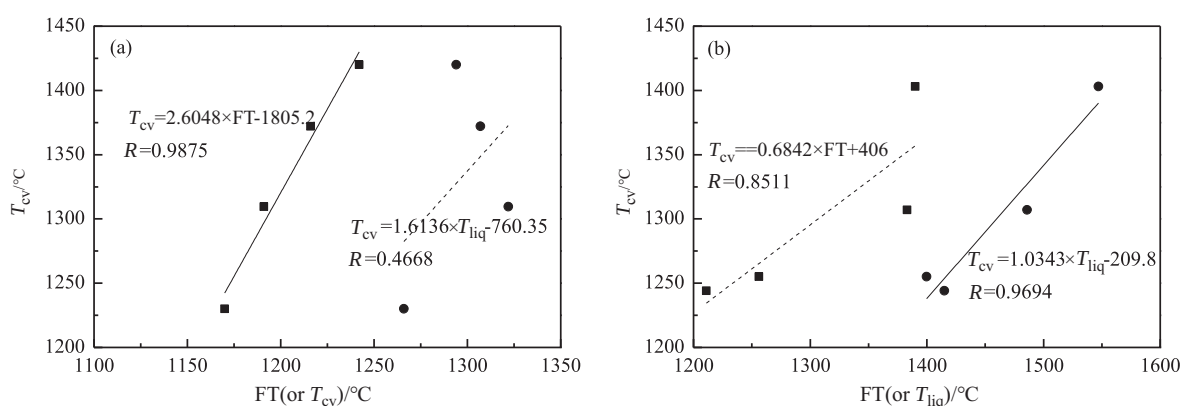


Fig. 11. Fitting of T_{cv} with FT or T_{liq} .

mineral in the slags with coal gangue-A addition, which accounted for formation of the glassy slag. However, in Fig. 9(b), the crystalline minerals, calcium aluminum iron oxide and magnesioferite, were detected in the slag with 2% coal gangue-B addition. The crystalline mineral was anorthite when coal gangue-B addition was 8%. All of these crystalline minerals contributed to the formation of the crystalline slags with coal gangue B addition.

3.6. Effect of coal gangue on characteristic temperatures of slag tapping

T_{cv} and T_{25} (the temperature at which the viscosity was 25 Pa·s) are two important temperatures to determine operating temperature of slag tapping and gasification temperature. However, the measurement of slag viscosity is high cost and not available in most laboratory. In hence, a lot of researches have correlated T_{cv} with ash fusion temperatures and the T_{liq} [24].

Fig. 10 shows the FT, T_{liq} , T_{cv} and T_{25} with different additions of Gangue-A (or -B). The trend of FT, T_{cv} and T_{25} with increasing gangue-A addition was similar. The T_{cv} was higher than T_{liq} , which indicated the viscosity of liquid phase may have a stronger effect on the viscosity. However, the T_{cv} and T_{25} with increasing gangue-B addition were close to that of T_{liq} . The T_{cv} with gangue-B addition was far below T_{liq} , which indicated increase of viscosity was mainly due to solid precipitation.

Fig. 11 shows the fitting result of T_{cv} with FT (or T_{liq}), and the R values which were used to analyze the correlations between the T_{cv} and FT (or T_{liq}). Yan et al. [22] found that T_{cv} of the glassy slags can be predicted with FT, while T_{cv} of the crystalline one can be predicted with T_{liq} . Generally, an R value of 0.9 or higher is excellent for predicting the T_{cv} values of slag. It can be seen in Fig. 11

that the T_{cv} can be well correlated with the FT for the slag with gangue-A addition, while the T_{cv} of slag with gangue-B addition showed a good relationship with T_{liq} .

4. Conclusions

Yibei coal ash was rich in calcium and iron, which exhibited the behavior of a crystalline slag. As a result, the coal was not suitable directly for the entrained-flow gasifier. In this work, we used coal gangue with different $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio to modify the ash flow properties. The results demonstrated that coal gangue addition was an effective way to improve the ash flow properties of YB coal. The main conclusions are as follows:

- (1) The AFTs decreased firstly, and then increased with increasing coal gangue addition. The coal gangue with the low $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio showed a more significant effect on the AFTs of YB coal due to existence of high melting point Al_2O_3 . Chemical composition of YB coal ash moved from gehlenite primary phase to anorthite, quartz and corundum primary phases.
- (2) The slag viscosity of Yibei coal ash increased with increasing coal gangue addition. The slag which behaved as a crystalline slag changed to that which exhibited as a glassy one with addition of coal gangue A.
- (3) Addition of coal gangue with a high $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio impeded the crystallization of anorthite during cooling. A low $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of coal gangue was favorable for the crystallization of calcium aluminum iron oxide, magnesioferite and anorthite.

- (4) T_{CV} of slag can be correlated with FT and T_{liq} , which was largely dependent on the viscosity behavior of slag with coal gangue addition. The T_{CV} of the glassy slag should be correlated with the FT, while T_{CV} prediction of the crystalline one should be used T_{liq} .

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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