Preparation and Properties of Pulp Black Liquor Briquettes

Baonong Tian, Zhe Ji, and Fushan Chen*

Effects of adding pulping black liquor in the preparation of briquettes were evaluated relative to the properties of the resulting pulp black liquor briquettes. The addition of 2% binder (pregelatinized starch) and a molding pressure of 5 MPa were sufficient to obtain briquettes that complied with DB13/1055 (2009). When 30% of pulping black liquor was added in the preparation of briquettes, the compressive strength, shatter index, moisture content, and the content of volatile matter and ash of the resulting briquettes increased. Moreover, the combustion performance of the briquettes after the addition of black liquor was higher, where the briquettes exhibited an enhanced ignition point, a more stable combustion process, and a 50% higher sulfur-fixation ratio after combustion, compared to the ordinary briquette. However, the thermal stability and calorific value decreased and the ash after combustion of the black liquor briquette contained a higher content of high melting point salts. Therefore, the addition of pulping black liquor in the composition of briquettes should be moderate. The preparation of black liquor briquettes provides an efficient way for utilization of black liquor.

Keywords: Pulping black liquor; Briquette; Molding performance; Sulfur-fixing performance

Contact information: College of Marine Science and Biological Engineering, Qingdao University of Science and Technology, Qingdao, Shandong Province, 266042, China;
** Corresponding author: chenfushan@qust.edu.cn*

INTRODUCTION

Briquette technology can be regarded as a type of clean coal technology. Coal-cleaning technology is one of the methods used both to control the emissions of sulfur dioxide and dust, and to improve fuel quality. Biomass is an alternative renewable energy characterized by zero greenhouse gas emissions because it converts solar energy and CO_2 to chemical energy (Roy and Corscadden 2012). However, low energy density, storage difficulties, and high costs of transportation are disadvantages that hinder the possibilities to extend biomass combustion technology effectively (Dael *et al.* 2013; Stolarski *et al.* 2013). To solve these problems, biomass molded fuel (BMF), which is regarded as an effective method to promote the use of bioenergy, could be a solution. Recently, the utilization of BMF as an alternative fuel has attracted increasing attention in research (Jiang *et al.* 2015). The results of industrial experiments show that briquette burning has a higher efficiency by 10% to 17%, resulting in a coal saving rate of approximately 10% to 26%, and a household stove saving rate of 20% in briquette burning. Simultaneously, the soot emissions of briquette burning can be reduced 71% to 89%, and the emissions of SO_2 and NO can decrease 50% to 60% (Heschel *et al.* 1999; Chen *et al.* 2001).

Alkaline pulping black liquor comprises approximately 65% to 70% of organic matter and 30% to 35% of inorganic matter in the solid matter. Within these percentages, the content of lignin is approximately 20% to 30%, the content of organic acid is approximately 6% to 10%, and the content of total sodium is approximately 20% to 26% (Zhang *et al.* 2012). During pulping, polysaccharides and lignin remain unprocessed and are released into pulping black liquor, which is more difficult to treat (Thielemans *et al.* 2002; Gosselink *et al.* 2004). Pulping black liquor contains large amounts of lignin, hemicellulose, and humic acid, *etc.*, as well as more highly basic substances, such as Na_2CO_3, Na_2SO_4, and NaOH (Xiong *et al.* 2017). These substances, having alkaline character, are used for the pretreatment of sugar production for corn stover (Zhang *et al.* 2011; Xu *et al.* 2012). The viscosity of these substances is in a suitable range so that they can be added directly to briquettes as additives or can be modified prior to addition (Lumadue *et al.* 2012). These additives can increase the volatiles during briquette combustion, thereby increasing its combustion efficiency, while alkaline substances can play a certain role in fixing sulfur, such as reducing the direct discharge contamination of the pulping black liquor, reducing the cost of the briquette preparation, and opening up a new avenue for the highly efficient utilization of pulping black liquor.

EXPERIMENTAL

Materials

Black liquor was prepared through alkali treatment of straw pulp black liquor (SPBL) at the Pulp and Paper Engineering Laboratory of Qingdao University of Science and Technology (Qingdao, China). Pulverized coal was purchased from a mineral powder processing plant (Xinlei Mineral, Hebei Province, Tangxian, China). Pregelatinized starch was purchased from Hebei Yanxing Chemical Co., Ltd. (Qingdao, China). Potassium hydroxide (KOH) was purchased from Sinopharm Chemical Reagent Co., Ltd. (Qingdao, China). All of the chemicals used were of analytical grade.

According to industrial analysis, SPBL contained 12.3% of solid content. Solid ash was 17% and had a calorific value of 12562 kJ/kg and a pH of 14. The pulverized coal contained 3.85% moisture, 3.78% ash, 28.64% volatiles, and 68.28% fixed carbon, with a calorific value of 16782 kJ/kg.

The following equipment was used in this study: a muffle furnace (High-Tech Development Zone, Luoyang Experimental Power Plant, Luoyang, China), a PC thermogravimetric analyzer (STA 409, Netzsch, Selb, Germany), a Philips X'pert diffractometer (Philips, Amsterdam, Netherlands), and a field emission scanning electron microscope (JSM-6700F, Japan Electronics Corporation, Beijing, China) equipped with an X-ray energy dispersive spectrometer (XEDS; Oxford Instruments INCA system).

Methods

Cui *et al.* (2015) determined the shatter index of the briquettes by a shatter test in accordance with MT/T 925 (2004). According to the standard, a number of coal-like products are allowed to fall freely from a height of 2 m on a 15-mm-thick steel plate. For pieces greater than 13 mm, the process was repeated three times. Finally, the weight of pieces greater than 13 mm remaining after the third drop is measured and the percentage of weight loss is calculated. This indicates the shatter index of the briquette.

The thermal stability of briquettes was determined in accordance to MT/T 924 (2004). A certain amount of coal sample was heated in a muffle furnace at 850 °C ± 15 °C for 30 min, then weighed and sieved after cooling. Finally, particles with sizes greater than 13 mm were used to determine the total mass of the residual coke, which was used to calculate the thermal stability index BTS_{+13}.

The compressive strength experiment was conducted using an artificial pressure method. The specific experimental steps began with randomly selected briquettes that were placed in the center of the mechanical test fixture; then a certain load was slowly applied to the briquette until it cracked. Cracked moment readings were recorded. Three samples were selected for the compressive strength test from each group of briquettes, and the arithmetic mean of the measured values for each briquette was taken as the compressive strength (N/ball) of the briquette (Li and Zhang 2011).

The moisture content in the briquettes was determined in accordance with GB/T 211 (2007). A certain amount of a coal sample with a particle size of < 13 mm was dried to a constant mass not exceeding 40 °C. Then the coal sample was crushed to a particle size of < 3 mm and dried in an air stream to a constant mass at 105 °C to 110 °C. The total moisture was calculated according to the mass loss after the two-step drying of the coal sample.

The ash content of the briquettes was analyzed with a rapid ashing method in accordance with GB/T 212 (2008). The briquette sample was weighed to 1 g ± 0.1 g and flattened evenly in a dish and combusted for 40 min in a muffle furnace at 815 °C ± 10 °C.

The dish was removed from the furnace and cooled for approximately 20 min to room temperature before weighing. The sample was repeatedly heated to 815 °C ± 10 °C for 20 min intervals until the weight variation for two consecutive heating cycles did not exceed 0.0010 g. When the ash content was less than 15.00%, it was not necessary to perform this process. The coal ash content was calculated by Eq. 1,

$$A_{ad} = \frac{m_1}{m} \times 100 \tag{1}$$

where A_{ad} is the mass fraction of ash after air drying (%), m is the briquette sample quantity (g), and m_1 is the mass of the residue after ignition (g).

Analysis of volatiles in briquettes

The content of volatiles in the briquettes was determined in accordance with GB/T 212 (2008). The briquette was weighed 1 g ± 0.01 g, evenly spread in a crucible, and combusted for 7 min in a muffle furnace at 920 °C. The crucible was then removed from the furnace and cooled to room temperature before being weighed. The volatiles in the briquette were determined by Eq. 2,

$$V = \frac{m_1}{m} \times 100 - V_1 \tag{2}$$

where V is the mass fraction of volatile components in the air-dried briquette (%), m is the mass of briquette (g), m_1 is the reduced mass of briquette after heating (g), and V_1 is the moisture content of the briquette (%).

Determination of calorific value of briquettes

The calorific value of the briquettes was measured by GB/T 213 (2008) (Polat *et al.* 2015). The instrument used in this experiment was an XRY-1B microcomputer oxygen bomb calorimeter (Nanjing T-BOTA, Nanjing, China). An amount of approximately 0.9 g to 1.1 g of briquette was weighed and placed on a combustion dish. The oxygen bomb was pressurized to approximately 2.8 MPa to 3.0 MPa. Ignition was initiated after installing all of the devices and the inner cylinder temperature was monitored. A sharp rise in the temperature within 30 s indicated that the ignition was successful, and the time from initiation of ignition to the end of combustion in the calorimeter was approximately 8 min to 10 min. After combustion, the test failed if the cartridge and the inside of the combustion vessel had signs of incomplete combustion or black carbon.

Calculation of sulfur-fixing ratio

The sulfur-fixing ratio for briquettes was calculated by Eq. 3 (Ohbuchi *et al.* 2010),

$$R = \frac{\omega \cdot S}{\omega_1 \cdot S_1} \times 100\% \qquad (3)$$

where R is the briquette sulfur-fixing ratio (%), ω is the weight percentage of ash content (%), S is the sulfur content in ash (%), S_1 is the sulfur content of briquette (%), and ω_1 is the weight percentage of briquette (%).

RESULTS AND DISCUSSION

Preparation of Briquettes

A certain amount of pulverized coal and pregelatinized starch were weighed and mixed thoroughly. Then, 25% distilled water was added and mixed well with mechanical stirring. The mixture was placed into a mold and 5 MPa was applied to form the briquettes. They were oven-dried at 90 °C for 2 h, then removed from the oven and allowed to cool to room temperature. Round cakes with a diameter of 45 mm and height of 10 mm were obtained for using them as test samples in experimental research.

Fig. 1. Briquette samples

Effect of Binder Content on Properties of Briquettes

As shown in Fig. 2, the shatter index of the prepared briquettes reached approximately 83% when a pregelatinized starch binder dosage of 2% was added. Moreover, the shatter index exceeded 90% for a binder dosage of 2.5%. It was concluded that the obtained briquettes presented an excellent shatter index, in line with national briquette standard DB13/1055 (2009) that requires a shatter index of 85%.

In contrast, the thermal stability of the briquettes did not exhibit changes with an increased amount of binder. This was explained by the fact that thermal stability was associated with the volatiles of briquettes, and the addition of a small amount of binder had minimal effect on the volatiles. Thus, the thermal stability of the briquettes remained basically unchanged.

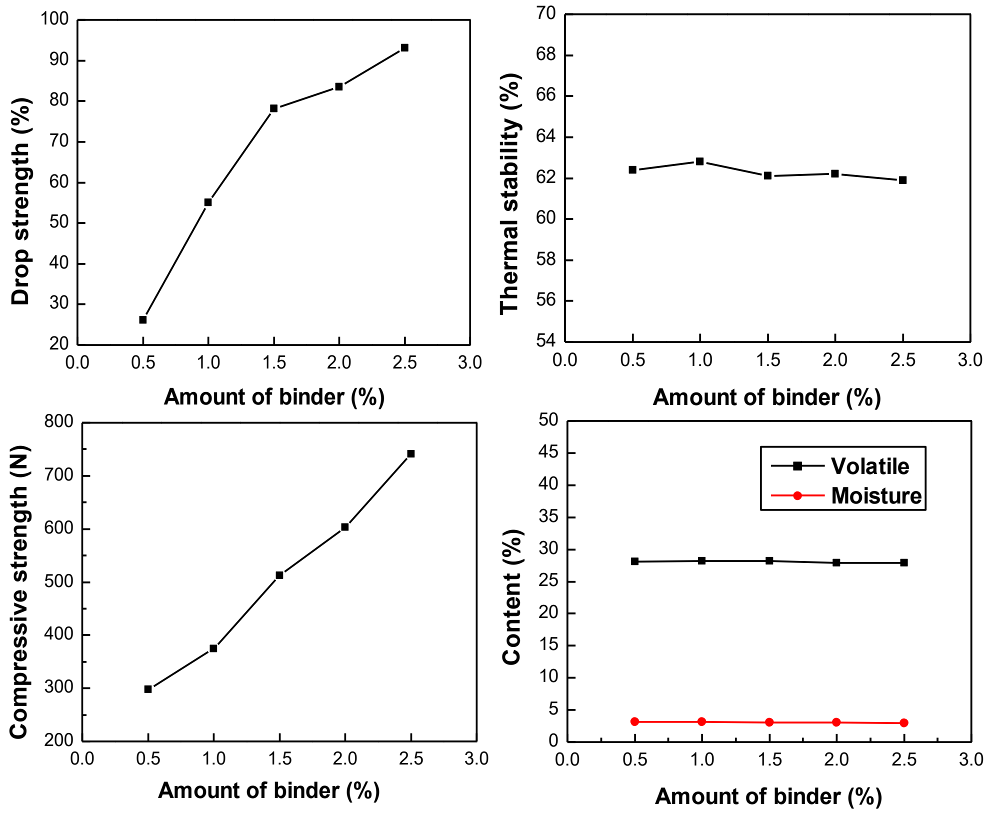

Fig. 2. Relationship between the binder content and the performance of briquettes

The compressive strength of the briquettes increased with an increased amount of the binder, and it reached 620 N/ball when 2% of the binder was added. A compressive strength of 600 N/ball was sufficient to meet national briquette use standard DB13/1055 (2009). As expected, increasing the binder content in the briquette had little effect on the moisture content and volatiles.

Considering the performance of the briquettes as a function of binder content, an addition of 2% pregelatinized starch was used as the optimum amount of binder in preparing the briquettes.

Molding Performance of Pulp Black Liquor Briquettes

As shown in Fig. 3, it was observed that the rate of moisture loss in the black liquor briquettes and ordinary briquettes were essentially the same with an increase in the number of drying days during the preparation of the briquettes. However, the addition of black liquor led to a reduction in the combustion calorific value, but the overall effect was not notable from approximately 28,500 kJ/kg to 27,000 kJ/kg. Therefore, a high addition of black liquor should be avoided.

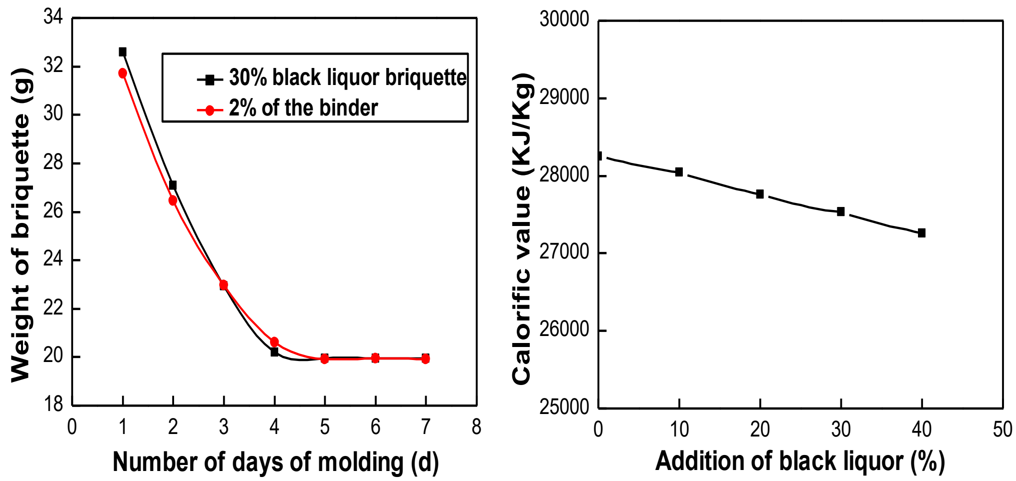

Fig. 3. Loss of moisture during briquette preparation

As shown in Fig. 4, when an amount of 30% of black liquor was added, the shatter index of the briquettes reached 93%. However, the shatter index remained essentially unchanged after further additions of black liquor.

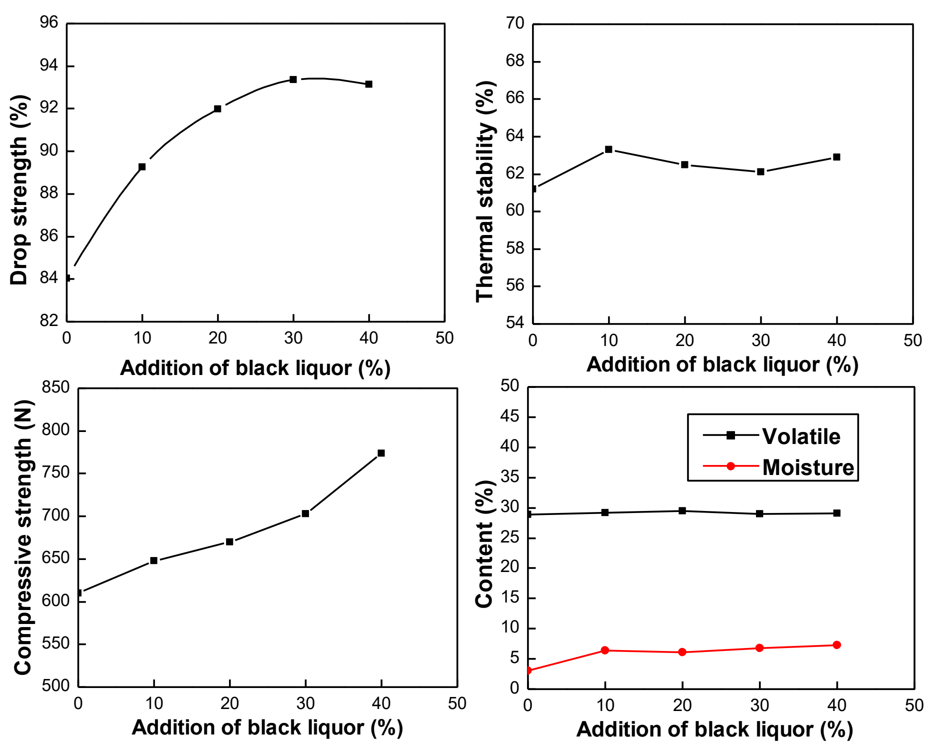

Fig. 4. Relationship between the content of pulp black liquor and briquette performance

With the increase in the black liquor content, the thermal stability of the briquettes increased at first and then decreased. This can be explained by the fact that the organic matter in the briquette is volatile and it decomposed at higher temperatures. Therefore, the stability of the briquette was diminished, but the overall effect was not considerable.

In addition, with the increase in the amount of black liquor, the compressive strength of the briquettes also increased, which was due to the lignin present in the black liquor itself, which also played a certain role in bonding. The moisture content in the black liquor briquette also increased with an increase in the amount of black liquor. This may have been due to the sodium lignosulfonate in the black liquor, which has a certain hydrophilicity. Hence, the sodium lignosulfonate contributed to maintaining a certain amount of water within the briquette. The content of volatile in the black liquor briquette remained essentially unchanged, and the difference in the volatilization weight loss of the briquette at high temperatures was mainly due to the loss of moisture.

Thermogravimetric Analysis of Black Liquor Briquettes

For thermogravimetric analysis (TGA), ordinary and black liquor briquettes were subjected to experimental conditions beginning with a heating range between 20 °C to 700 °C, at a heating rate of 10 °C/min under an air atmosphere. The combustion process of briquettes is generally believed to be divided into four stages, where the first stage is the evaporation of water in the briquette, the second stage is the release of volatile matter and combustion, the third stage is the combustion of the coke, and the fourth stage is the briquette charcoal burnout stage. However, there are quite a few overlaps between the third stage and the fourth stage and these tasks can be considered to be completed in the same stage.

As shown in Fig. 5, the first stage of the combustion process of the briquettes was due to the loss of moisture, when the internal moisture vaporized from the pulverized coal, *etc*., as indicated by the sharp and broad endothermic peak in the corresponding derivative thermogravimetric analysis (DTG) profile. Moreover, the endothermic peak in the DTG curve of the black liquor briquette was larger than that of the ordinary briquette. This indicated that the black liquor briquette contained more water than the ordinary briquette at this stage, and for this reason, the rate of weight loss was faster. The TG curves of the briquettes sloped gently downward with an increase in temperature, which corresponded mainly to the release of the volatile matter in the briquettes.

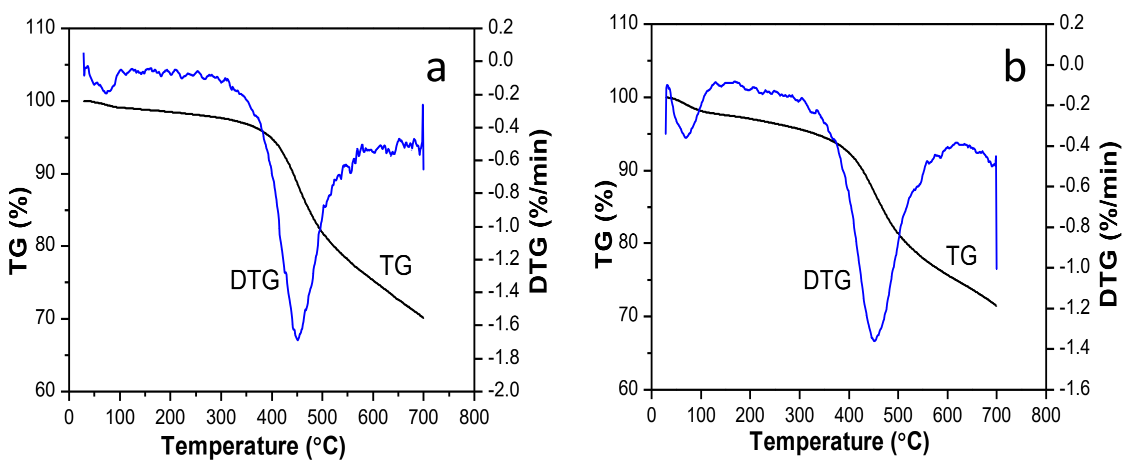

Fig. 5. Thermogravimetric curves of briquettes: a- Ordinary briquette; b- 30% black liquor briquette

The TG curve of the black liquor briquettes exhibited a sharper slope, as the black liquor briquettes contained more volatiles. The ignition temperature of ordinary briquettes is approximately 400 °C (Li *et al.* 2007), whereas when comparing the two sets of curves, it was observed that the ignition temperature of the black liquor briquettes was approximately 350 °C. A small sharp peak was observed in the DTG curve due to the heat of combustion of the organic matter (cellulose, lignin, and hemicellulose, *etc*.) in the black liquor.

These species have a high calorific value and further improve the ignition heating value of the black liquor briquette when the volatiles are released. The ignition point of the black liquor briquette was lower than that of the ordinary one, and the third stage corresponded to the fixed carbon combustion stage of the black liquor briquette. Furthermore, the TG curve of the black liquor briquette was steeper, which indicated a more rapid weight loss and a faster rate of combustion.

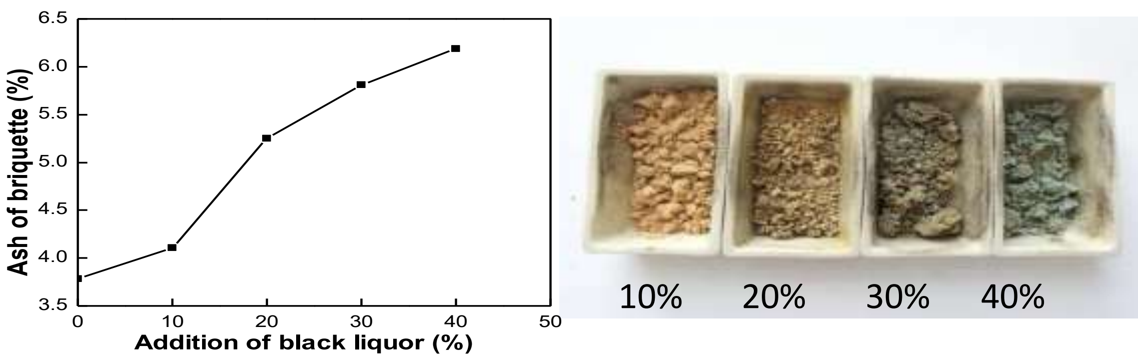

Fig. 6. Ash content of black liquor briquettes

As shown in Fig. 6, with the increase in the amount of black liquor, the ash content of the black liquor briquette also increased, but it remained under the maximum of 12% allowed by DB13/1055 (2009). Due to the increase in the content of alkaline metal salts in the black liquor component, the amount of crystalline compounds that formed in the ash of the black liquor briquette also increased, which determined a crystalline texture of the ash. Thus, the addition of too much black liquor to briquettes can lead to clogging of the furnace, which can require frequent cleaning. Considering the discussion above, it may be concluded that the addition of black liquor in the preparation of briquettes should not exceed 35%.

Analysis of Sulfur-fixing Ratio of Black Liquor Briquettes

Pulping black liquor contains highly alkaline substances (KOH and K_2CO_3, *etc*.,), and some surfactants such as lignin sulfonate and humic acid. The presence of these substances during high-temperature combustion of briquettes offers the possibility of generating solid sulfur. Samples were analysed by scanning electron microscope equipped with an X-ray energy dispersive spectrometer. A 200× magnification was adopted that allowed the detection of particles with diameters in the ranges 25 μm to 125 μm. An XEDS analysis was conducted on the black liquor briquette before combustion, and the results are summarized in Table 1.

The content of the elements is expressed as a relative percentage; when the element content was relatively low, the element had little effect on sulfur fixation.

Table 1. XEDS Analysis of Black Liquor Briquette Before Combustion (%)

Species	C	O	Na	Mg	Al	Si	S	K	Ca	Fe
0%	79.78	18.6	0.02	0.02	0.23	0.54	0.19	0.05	0.12	0.38
10%	80.47	17.45	0.03	0.01	0.22	0.61	0.16	0.47	0.18	0.39
20%	78.98	17.93	0.01	0.03	0.18	0.51	0.17	1.61	0.14	0.46
30%	77.35	19.06	0.03	0.04	0.17	0.48	0.16	2.10	0.10	0.48
40%	77.41	18.09	0.02	0.03	0.18	0.43	0.14	3.17	0.16	0.37
50%	74.91	18.98	0.02	0.02	0.18	0.47	0.14	4.49	0.26	0.49

The XEDS analysis was also performed on the ash obtained after combustion of the black liquor briquette, as summarized in Table 2.

Table 2. XEDS Analysis of the Ash from Black Liquor Briquette After Combustion (%)

Species	C	O	Na	Mg	Al	Si	S	K	Ca	Fe
0%	17.11	49.87	0.60	0.82	5.74	14.65	1.48	0.96	4.82	3.23
10%	42.23	36.10	0.25	0.38	2.70	6.31	1.58	5.63	2.43	2.13
20%	14.82	44.00	0.38	0.34	2.15	5.67	1.56	23.90	4.73	2.34
30%	26.16	45.24	0.21	0.10	0.44	1.71	1.79	22.81	0.89	0.63
40%	38.34	39.47	0.45	0.05	0.31	1.45	1.89	15.71	1.61	0.65
50%	43.33	35.66	0.26	0.04	0.07	0.49	1.63	18.08	0.26	0.36

From Tables 1 and 2, it may be noted that as the amount of black liquor in the briquettes increased, the content of the K element remarkably rose, which can be explained by the use of KOH as a treatment agent in the alkali treatment of ginger cellulose. This trend is less obvious for other multi-alkaline metal ions, providing a basis for solid sulfur in the coal. From the analyses completed before and after combustion, it was established that the addition of black liquor had a greater impact on the amount of S and K elements.

Using the solid sulfur rate calculation as shown in Eq. 3, the graph in Fig. 7 was able to be completed. It may be noted from the graph that the rate of sulfur fixation was best when adding an amount of 40% of black liquor to the briquettes. Compared with the ordinary briquette, which had no black liquor in its composition, the black liquor briquette had a higher rate of sulfur fixation, which increased by 50%. However, the rate of sulfur fixation presented a downward trend when the amount of black liquor was excessively high.

This could have been due to the excessive content alkaline metals in the black liquor, which produced an overly high amount of crystals, and was not conducive to the reaction of sulfur dioxide and alkali metals, thus affecting the rate of sulfur fixation of the briquettes to a certain extent.

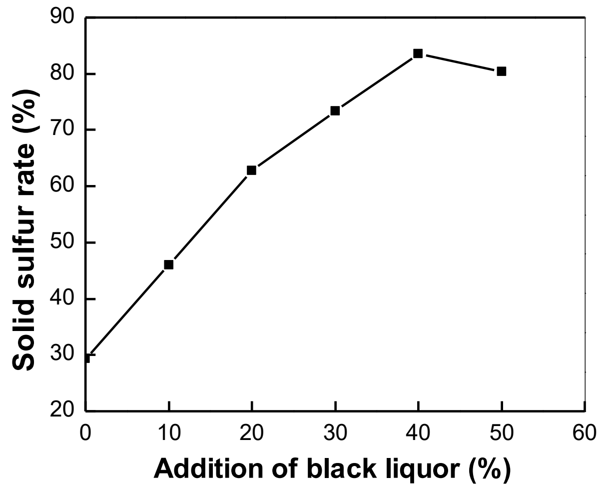

Fig. 7. Solid sulfur rate of black liquor briquettes

X-ray Diffraction (XRD) Analysis of Black Liquor Briquettes

Ash samples from the two kinds of briquettes were analyzed by XRD, at 2θ angles of approximately 20° to 80°, to compare the main forms of the oxides in the ash (Li *et al.* 2007; Sola and Atis 2012).

When the black liquor briquette was combusted in the furnace, ash was easily generated. The ash was mainly comprised of K, Fe, and Ca salts, and complex salts of these elements.

After complete combustion of the black liquor briquette at high temperatures, the salt underwent melting and sintering at high temperatures, and the molten slag was easily deposited and accumulated on the furnace wall, which could affect the combustion efficiency of the boiler in the long term, and could even clog the furnace. Therefore, it was necessary to determine the composition and content of the molten ash from the black liquor briquette, which is helpful in determining the combustion temperature of the black liquor briquette. In this study, the main salts of K, Fe, Ca, and the complex salts of these elements in the ash were studied, and the crystal products in the ash were determined by XRD analyses.

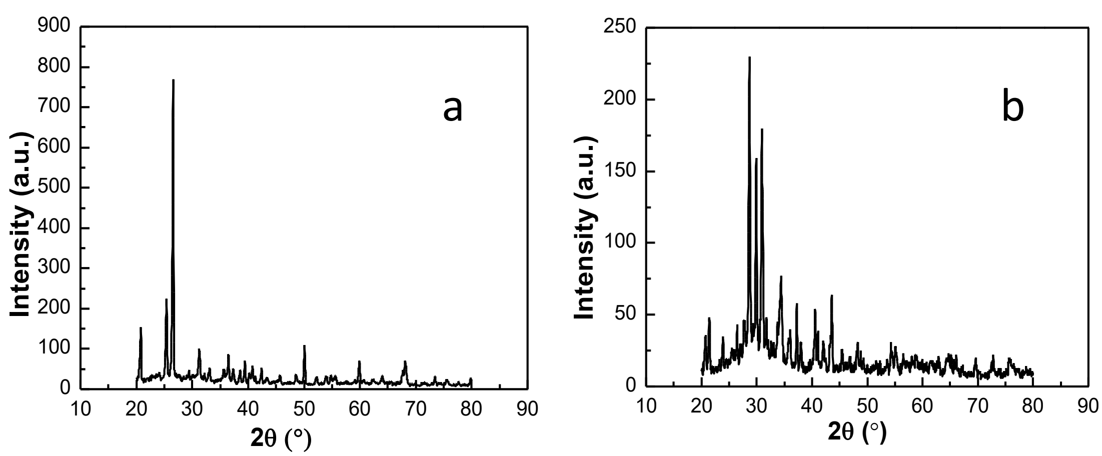

Fig. 8. XRD pattern of the ash from briquettes: (a) ordinary briquette and (b) 30% black liquor briquette

A comparison of the XRD spectra of the ordinary briquette and the black liquor briquette, combined with the XEDS data, showed that the content of silicates and high-melting-point K salts and Fe salts in the black liquor briquette were higher. The melting point of these inorganic salts in the ash was higher, which means that the temperature of the boiler furnace for combustion of the black liquor briquette should be below 1100 °C (Li *et al.* 2007), given that a higher temperature would lead to adhesion of the molten inorganic salts on the furnace wall. Moreover, decomposition of sulfuric acid would lead to a re-release of sulfur and affect the sulfur-fixing efficiency.

The results showed that the alkaline substances (KOH and K_2CO_3) were the main sulfur fixation agents in the pulping black liquor. When the black liquor briquette was combusted at 800 °C to 1000 °C, the SO_2 in the flue gas reacted with KOH to produce sulfate:

$$S + O_2 \rightarrow SO_2 \quad (4)$$

$$2KOH + SO_2 \rightarrow K_2SO_3 + H_2O \quad (5)$$

$$K_2SO_3 + 1/2O_2 \rightarrow K_2SO_4 \quad (6)$$

The thermal stability of sulfate was high and the compounds were not easily decomposed at a high temperature. Thus, most of the sulfur in the briquette was fixed in the slag, with good sulfur-fixation efficiency.

CONCLUSIONS

1. Briquettes were prepared integrating pulping black liquor and were subjected to combustion, characterized, and analyzed. The combustion performance of the black liquor briquette was higher than that of ordinary briquettes, and the ignition point was lower. Moreover, the combustion process was more stable, and the sulfur-fixation ratio after combustion increased 40% to 50% in the case of the black liquor briquette compared to ordinary briquettes.
2. The results demonstrated that increasing the proportion of pulping black liquor caused the ash content to rise after combustion of the black liquor briquette. The ratio of black liquor added to the briquette should be moderate to ensure a moderate thermal value of the black liquor briquette, looser ash, higher whiteness, and less induration slag generated after combustion of the briquette.
3. Considering the preferred amount of black liquor to be added, 30% was established as the optimum amount. Therefore, the addition of pulping black liquor to briquettes leads to an enhanced sulfur-fixation effect. In this way the black liquor treatment can play a good role in protecting the environment.

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