

Synergistic effect in low temperature co-pyrolysis of sugarcane bagasse and lignite

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Abstract—Sugarcane bagasse was co-pyrolyzed with lignite in a fixed bed reactor to investigate the possible interaction during co-pyrolysis. GC-MS revealed that the concentration of phenols and aliphatic compounds in the tar increased with the addition of sugarcane bagasse, while the content of aromatic compounds had the contradictory tendency. The phenol content in co-pyrolyzed tar reached 20.35%, which increased by 142.26% compared with the calculated values. The sugarcane bagasse decomposition peak partly overlapped with lignite pyrolysis peak from TG-DTG curves, which meant more interaction between lignite and sugarcane bagasse during the pyrolysis process. The difference between the experimental and calculated values of pyrolysis products yield, tar components, DTG values and kinetics analysis indicated the synergetic effect between lignite and sugarcane bagasse.

Keywords: Sugarcane Bagasse, Lignite, Co-pyrolysis, Synergetic, Thermogravimetric Analysis

INTRODUCTION

With the exhaustion of fossil fuels and serious pollution to the environment during societal development, it is urgent to seek alternative fuels and high-efficiency method to utilize coal resources. Sugarcane bagasse (BG) is a fibrous residue of the sugarcane stalk after crushing and extraction of the juice, which mainly consists of cellulose, hemicellulose and lignin. It attracts extensive attention as a promising renewable and CO₂ neutral energy source. Compared with other biomass, BG has the advantage of high yields, simple material structure, high H/C and low ash, nitrogen, sulfur content [1-4]. Pyrolysis technology is not only a promising thermochemical conversion technology for producing varieties of fuels and chemical compounds, but also the first and key step of all the other thermochemical technologies [5-8]. Co-pyrolysis technology is extremely important to the utilization of biomass and coal resources, which has the characteristics of mild conditions and utilizes coal in an environmentally friendly way. In addition, interaction between biomass and coal during co-pyrolysis process may have unexpected effect on product distribution and composition.

Many researchers [9-12] have studied the co-pyrolysis process of coal and biomass to prove the existence of synergetic effects, which are likely due to the higher H/C and alkali/alkaline earth metal (AM) content of biomass compared with coal. Nevertheless, some studies [13-15] have reported a lack of synergetic effect during co-pyrolysis of coal and biomass because the devolatilization of coal and biomass particles takes place at different times and the contact time between the pyrolytic products from coal and biomass is short at rapid heating rate. Aboyade et al. [16] have investigated the kinetics and characterization of the devolatilization of

coal blends with corn and sugarcane residues, but the coal sample used was a kind of inertinite rich hard coal and the devolatilization peak of coal had no overlap section with that of biomass. In addition, synergistic interactions between the coal and biomass they proved were only based on the relatively poor quality of fit parameters (reaction order, pre-exponential factor, activation energy) of single fuels with blends.

Therefore, Australia lignite (LC), which has similar devolatilization characteristic temperature with biomass, was co-pyrolyzed



Fig. 1. Picture of coal sample.

Table 1. Size distribution of feedstocks

Size range (mm)	0.178-0.355	0.150-0.178	<0.150
LC	0	63.53	36.47
BG	38.96	54.55	6.49

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Table 2. Ultimate and proximate analysis of samples (w/%)

Samples	Ultimate analysis (ad)					H/C	Proximate analysis (ad)			
	C	H	O*	N	S		M	A	V	FC
LC	56.82	3.20	19.44	0.21	0.38	0.68	18.66	1.29	35.97	44.08
BG	41.67	6.21	41.33	0.37	0.07	1.79	8.64	1.71	73.28	16.37

*By difference

with BG at different mix ratio in a fixed bed reactor to investigate the possible interaction during co-pyrolysis process. Interaction of lignite and biomass was studied by thermogravimetric analysis (TGA) and gas chromatography-mass spectrometer (GC-MS). The kinetic parameters were determined with the Arrhenius equation and Coats-Redfern method [17].

EXPERIMENT

1. Materials

Raw materials used in the study were lignite from Australia and agriculture residues sugarcane bagasse (BG) collected from Guangdong province. The coal sample had lower metamorphic grade with some visible wooden structure to the naked eye. A photograph of the coal sample is shown in Fig. 1. Lignite was ground below 0.178 mm particle size and conserved in a hermetic bag according to GB 474-2008. BG was dried for 24 hours at 40 °C in drum wind drying oven to moisture content less than 10 wt%, then milled below 0.355 mm and stored in a desiccator. The particle size distribution of feedstocks is presented in Table 1. The ultimate and proximate analysis are shown in Table 2.

2. Experimental Method

Pyrolysis of lignite, biomass and their blends was performed in a self-designed fixed bed reactor to investigate the influence of coal-biomass mix ratio on product distribution. Biomass fraction (mass fraction of BG accounted for blends) used was 0%, 10%, 20%, 25%, 30%, 40% and 100%, respectively. 10 g (± 0.001 g) raw mate-

rials/blends were fed into the pyrolysis reactor in a single batch. The reactor was heated to 510 °C from 10 °C at rate of 10 °C/min and then held for 30 min at final temperature at 1atm. The co-pyrolysis experiments were performed four times for each run to guarantee experimental stability and accuracy. The relative error between each run was less than 5%. The optimum coal-biomass mix ratio was determined according to the maximum deviation between theoretical and experimental data. A schematic diagram of the homemade pyrolysis apparatus is shown in Fig. 2.

NETZSCH STA 449F3 thermogravimetric analyzer (Germany) was employed for the thermogravimetric analysis of LC, BG and the blends. The operating conditions were under argon flow of 60 ml/min and temperature increase to 1,000 °C from 30 °C at a heating rate of 10 °C /min. GC-MS experimental apparatus and methods are used as reported by He et al. [12].

The instrument of EDX-7000 was used to measure the metal contents in the pyrolysis char of BG and LC. Results are shown in Table 3.

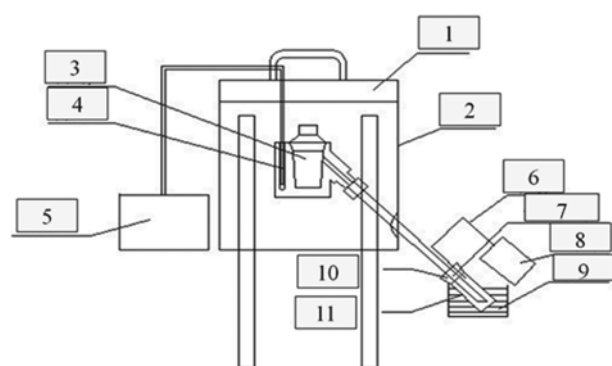
RESULTS AND DISCUSSION

1. The Distribution of Pyrolysis Products

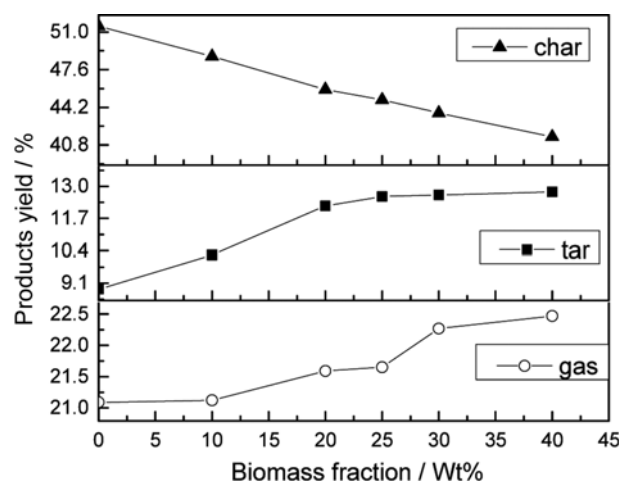
According to GB/T480-2010 and related research papers [12,18],

Table 3. The metal content in the pyrolysis chars of LC and BG (%)

	K	Ca	Na	Mg	Al	Fe	Si
LC	0.04	0.21	0.77	0.52	0.13	0.64	0.12
BG	1.14	0.48	0.78	0.71	0.11	0.99	0.07

**Fig. 2. Schematic diagram of homemade pyrolysis apparatus.**

1. Furnace cover
2. Furnace body
3. Retort body
4. Thermocouple
5. Temperature control
6. Airway
7. Tar exported tube
8. Air pocket
9. Ice bath
10. Rubber plug
11. Test tube

**Fig. 3. Yields of co-pyrolysis products.**

co-pyrolysis products were obtained at final temperature of 510 °C and 1 atm. Fig. 3 shows the product yields obtained from pyrolysis of each sample. As shown in Fig. 3, char was the major pyrolysis product, and yield decreased with the addition of BG. However, the yields of pyrolysis tar and gas had a contradictory tendency with the addition of BG. The pyrolysis tar yield reached to 12.78% with BG ratio of 40%, which increased by 44.24% compared with single coal pyrolysis. The yield data (gas, tar and char) for BG were 19.87%, 20.28% and 26.58%, respectively. The char yield decreased because carbon content of BG was far less than the coal sample, just the same as high volatile matter and oxygen content of BG to the increase of pyrolysis gas and tar. From the perspective of structural and chemical characteristics, BG consisted of hemicelluloses, cellulose and lignin macro-molecules linked together by relatively weak R-O-R bonds, which were easy to decompose to smaller molecules. By comparison, coal released comparatively less volatile as it is composed of highly cross-linked aromatics, held together by significantly stronger C-C bonds. The nonlinear change of the pyrolysis products yield in Fig. 3 proved the interaction of biomass and coal.

Comparing the experimental values of co-pyrolysis products yield with the theoretical values in Fig. 4, there existed an obvious deviation. The deviation enlarged with increasing proportion of BG in the feed mixture and the maximum positive deviation existed at BG ratio of 20%. The relative deviation of tar yield was 9.61% compared with calculated values. Researchers have shown that the pyrolysis process was affected by the pyrolysis atmosphere [19]. Biomass pyrolysis process took precedence over coal samples, so the pyrolysis gas (CO , CO_2 , CH_4 , H_2) of biomass could react with coal sample [20], thus changing the reaction mechanism, pyrolysis conversion and distribution of pyrolysis products. In addition, H/C in BG (1.79) was higher than that of lignite (0.68). High hydrogen content stopped the free radicals that generated during coal pyrolysis reacting or recombining with chars [9]. Instead, hydrogen radical combined with free radicals fragments generated during pyrolysis to form small molecule compounds, thus promoting the pyrolysis conversion. Besides, alkali/alkaline earth metal elements such as K, Ca, Na in BG were higher than that in lignite as shown in Table 3. Alkali/alkaline earth metal that combined with functional group and side chain in coal basic structural unit weakened

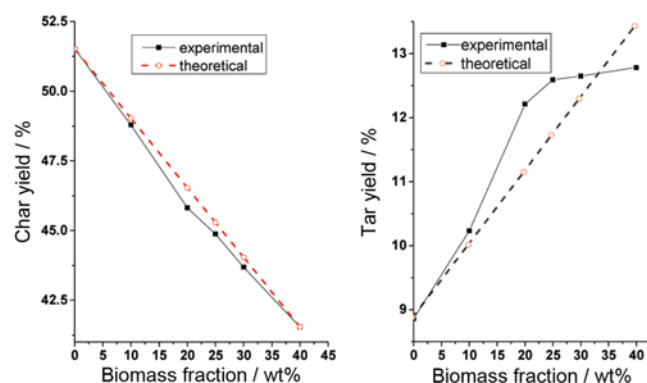


Fig. 4. Experimental values and theoretical values of co-pyrolysis product yields.

C-C bonds inside the hydrocarbon molecule [21], so coal/char was more easily decomposed into gases and lighter tar. In addition, the existence of alkali/alkaline earth metal decreased the activation energy of the reaction during pyrolysis process, as well as inhibited the progress of char graphitizing and increased char reactivity [22,23]; thus the yield of tar and gas was increased effectively. AM played a catalytic role in the co-pyrolysis process, so there was a char-coal reaction in addition to gas-coal reaction during the co-pyrolysis process [24]. Nevertheless, the experimental values of pyrolysis tar yield were lower than the theoretical values after 30%. The large amount of biomass and its pyrolysis carbon black produced in advance would cover the surface of coal or clog the coal pores because of the low density of biomass, thus affecting the heat transfer and precipitation of volatile in coal sample. There was an optimal mix ratio to improve the yield of volatile matter because of the competition of promotion and inhibition interaction. However, optimal mass mix ratio of BG and lignite (20%) was lower than the results reported by other researchers [12,18]. It was due to the low bulk density of the air dried BG sample (80-90 kg/m³) when compared with algae (300-400 kg/m³). In addition, Si content in BG was 0.07%, which was much lower than that of in algae and lignite, and it has been reported that Si had a negative effect on pyrolysis process [25]. Consequently, the optimal mass mix ratio obtained in the study was 20% and less than others' results.

2. Tar Composition Analysis

Pyrolysis tars are complex mixtures of organic compounds consisting of a wide range of organic chemical groups, which mainly include aliphatic compounds, aromatic compounds, phenols, acids, aldehydes, esters and heteroatom-containing compounds [26]. Fig. 5 shows the chemical composition of tar products from sugarcane bagasse, lignite and their mixture (20%). Wherein aliphatic compounds mainly consisted of octadecane, nonadecane, tetracosane and pentacosane; 2,3-dihydro-Benzofuran was the representative of aromatic compounds; phenols were mostly comprised of phenol, 4-methyl-phenol and 3,4-dimethyl-phenol; acid mainly included vaccenic acid and aldehydes mainly included furfural.

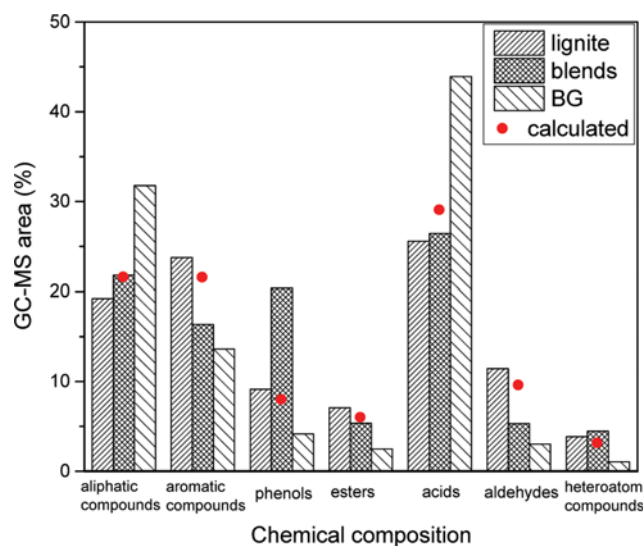


Fig. 5. Chemical composition of tar.

Tars from lignite pyrolysis contained more aromatic compounds, phenols, aldehydes, esters, and heteroatom-containing compounds compared to sugarcane bagasse tar. Aromatic compounds accounted for 23.74 wt% in lignite tar, which was nearly twice of the sugarcane bagasse. This was attributed to high polycyclic aromatic hydrocarbon structure in coal. In contrast, aliphatic compounds and acids content in sugarcane bagasse tar were 65.16% and 71.89% higher than that of lignite, respectively, due to the high H/C and O/C molar ratio of sugarcane bagasse. Analysis of tars from co-pyrolysis of sugarcane bagasse and lignite demonstrated that the concentration of phenols and aliphatic compounds in the tar increased with the addition of biomass, while the content of aromatic compounds had a contradictory tendency. It indicated that the sugarcane bagasse promoted the formation of the micromolecule organics just as phenols during co-pyrolyzed with lignite. The results were consistent with the analysis of pyrolysis products. As shown in Fig. 5, the experimental phenol content in co-pyrolysis tar reached 20.35%, which increased by 142.26% compared with the calculated values. Thus, co-pyrolyzed tar had the potential of being raw materials for the extraction of phenolic compounds. The positive deviation values between the experimental and calculated values of tar components indicated the synergistic effect between sugarcane bagasse and lignite.

3. Thermogravimetric Analysis of Individual Fuels

Fig. 6 shows the TG-DTG curves for BG and coal individual fuels. The initial reduction in DTG curves around 100 °C was attributed to the dehydration of the samples. Dehydration peak of coal sample was more obvious than that of BG due to the high moisture content in lignite. However, the fast pyrolysis peak of BG was distinct with lignite as BG had two evident pyrolysis weight loss peaks. The first one approximately at 214.5 °C was ascribed to the decomposition of hemicellulose and second peak nearby 336.6 °C in a narrow temperature range were the decomposition of cellulose, as cellulose consisted of linear chains with strong units, whereas hemicellulose had an amorphous structure with little strength. It was in accordance with the results of other researchers [6,11]. By contrast, lignite had only one big degradation peak between 250 and 600 °C. The ending segment was the carbonization stage of both coal and biomass under an inert atmosphere.

Table 4. Pyrolysis characteristic parameters of individual samples

Sample	$T_1/$ °C	$(dm/dt)_{max}/$ (%·min ⁻¹)	$T_{max}/$ °C	$T_2/$ °C	$(dm)_{max}/$ %
LC	253.5	1.59	411.9	573.2	50.37
BG (peak 1)	184.8	4.26	214.5	358.1	75.07
(peak 2)	254.2	6.58	336.6	369.3	

Tangent method [27] was applied to obtain the pyrolysis characteristic parameters of the sample in this study. T_1 was the initial degradation temperature and T_2 was the termination precipitation temperature; $(dw/dt)_{max}$ and T_{max} were maximum pyrolysis rate and its corresponding temperature respectively; $(dm)_{max}$ was maximum weight loss value. Pyrolysis characteristics parameters of LC and BG are illustrated in Table 4.

As shown in Table 4, the main pyrolysis temperature ranges of BG and LC were 184.8–369.3 °C and 253.5–573.2 °C respectively. It was totally different from the results obtained by Aboyade et al. [8], which demonstrated synergistic behavior between coal and biomass as biomass pyrolysis process had been basically finished before the volatile devolatilization of the coal sample. In this study, the hemicellulose decomposition of BG was before lignite pyrolysis, but cellulose decomposition peak was overlapped with lignite pyrolysis peak, which was more likely to exist as interaction between lignite and BG. Moreover, the maximum pyrolysis rate of BG was four-times faster than that of LC; thus the addition of BG could increase the pyrolysis rate of blends. The initial degradation temperature of BG was 68.7 °C ahead of lignite's due to the weak chemical bonds in biomass as mentioned before. Part of the oxygen containing functional groups in biomass were highly active and low activation energy, which made BG pyrolyzed easily under low temperature, producing a large amount of the volatile gas such as CO and H₂. The volatiles that released during BG pyrolysis affected the follow-up coal pyrolysis process. Consequently, hemicellulose pyrolyzed in advance and cellulose co-pyrolyzed with lignite were beneficial to interaction and synergistic behavior between BG and LC.

4. Synergetic Effects During Co-pyrolysis in TGA

DTG curves of blends (BG ratio 20%) and LC are shown in

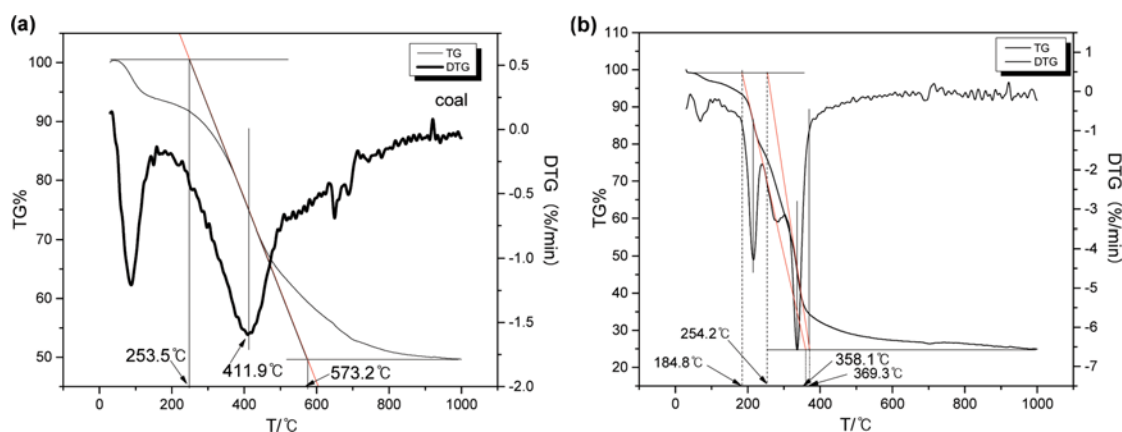


Fig. 6. TG-DTG curves of coal (a) and BG (b).

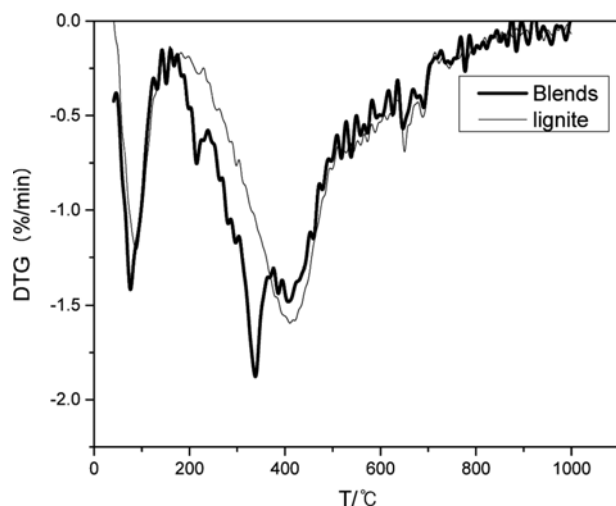


Fig. 7. DTG curves of the samples.

Table 5. Pyrolysis characteristic parameters of mixture samples

Sample	$T_1/^\circ\text{C}$	$(\text{dm}/\text{dt})_{\text{max}}/(\%\cdot\text{min}^{-1})$	$T_{\text{max}}/^\circ\text{C}$	$T_2/^\circ\text{C}$	$(\text{dm})_{\text{max}}/\%$
Peak 1	168.9	0.74	214.8	434.1	
Peak 2	255.1	2.07	337.0	465.8	57.49
Peak 3	267.5	1.52	405.1	512.7	

Fig. 7 and co-pyrolysis characteristics of the blends are presented in Table 5. Dehydration peak, hemicellulose decomposition peak, cellulose decomposition peak and lignite pyrolysis peak existed in the DTG curves of blends in Fig. 7. It is obvious that the BG decomposition peak partly overlapped with LC pyrolysis peak, which means more interaction between lignite and BG during the pyrolysis process. Comparing the pyrolysis characteristics parameters of blends with data in Table 4, T_{max} and T_2 of LC were 6.8°C and 60.5°C lower than that of coal pyrolysis alone, respectively. By contrast, pyrolysis characteristic parameters (T_1 and T_{max}) of BG in the blends remained relatively unaltered compared to parameters of single fuels. It indicated that the addition of BG improved the lignite reactivity to a certain extent by changing the coal pyrolysis process to lower temperature region. However, $(\text{dw}/\text{dt})_{\text{max}}$ of BG in mixtures was 2.07%/min, which reduced 68.54% compared with single BG, but $(\text{dw}/\text{dt})_{\text{max}}$ of LC changed inconspicuously. It was the coal particles that had dilution effects on BG and covered the surface of BG particles, thus affecting the heat and mass transfer and stopping the precipitation of volatiles [28] in BG. But $(\text{dm})_{\text{max}}$ of LC in mixtures (57.49%) was higher than that of single lignite (50.37%) and also higher than the theoretical value, which agreed with the result of product yield and proved the existence of synergy. Nevertheless, Song et al. [7] proved the synergetic effect between pine sawdust and lignite during the co-pyrolysis just based on the DTG peaks shifting towards lower temperatures. Biomass decomposition was easier than coal's and the DTG peaks shifting towards lower temperatures could be attributed to the addition of biomass.

To further illustrate the interaction between BG and LC during

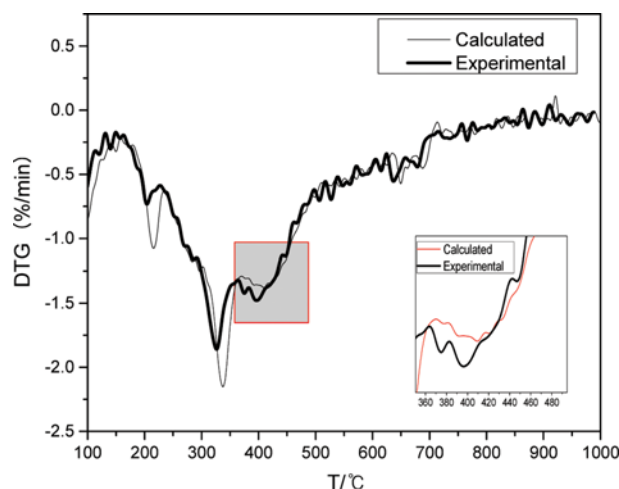


Fig. 8. Experimental and calculated DTG curves for mixture sample.

co-pyrolysis, the experimental and calculated values of the DTG curves were compared. Experimental values refer to the DTG curve of the mixture sample, and calculated values were the sum of the DTG curves of each individual sample, which were obtained as follows:

$$T_{\text{calculated}} = T_{\text{coal}}x_{\text{coal}} + T_{\text{BG}}x_{\text{BG}}$$

$T_{\text{calculated}}$ stands for the weight loss of the blend at each temperature point; T_{coal} and T_{BG} are the weight loss of LC and BG pyrolysis alone, respectively; x_{coal} and x_{BG} are ratios of LC and BG in the blend, respectively. The experimental and calculated DTG curves for mixture sample are shown in Fig. 8.

As revealed in Fig. 8, the experimental values of the hemicellulose decomposition peak and cellulose decomposition peak were smaller than the calculated values. It illustrated that LC inhibited precipitation of volatiles in BG during co-pyrolysis before 360°C. However, the experimental values of blends were greater than the calculated values between 360°C and 440°C, where the obvious synergy existed during co-pyrolysis process. The experimental values roughly agreed with the calculated values after 500°C at BG ratio of 20%, which suggested a weak interaction between BG and LC at high temperature region. The figure not only shows the interaction between BG and LC during co-pyrolysis, but also provides information to control the distribution of co-pyrolysis products by changing the temperature. More gas and liquid products would be obtained at low temperature (<500°C), and high temperature was more beneficial to the formation of solid products. The reasons for this phenomenon were the distinct chemical reactions between BG and LC at different temperature range. Chemical decomposition and depolymerization reaction played a dominant role in the co-pyrolysis process at low temperature range, and high content hydrogen in BG stopped the free radicals recombining with chars, thus generating a large number of pyrolysis tar and gas. In high temperature period, the thermal polycondensation reaction of chars to generate coke was the primary reaction.

5. Reaction Kinetics Analysis

According to the law of conservation of mass, Arrhenius equation and the derivative method, activation energy (E) and pre-expo-

Table 6. Pyrolysis kinetic parameters of samples

Sample	T/°C	Fitting equation	Ea/kJ.mol ⁻¹	A/min ⁻¹
LC	253.5-523.2	$y = -2356.0x - 10.022$ ($R^2 = 0.9906$)	19.59	1.046
BG	214.8-319.3	$y = -2723.4x - 8.2915$ ($R^2 = 0.9920$)	22.64	6.825
Blends	255.1-512.7	$y = -1696.4x - 10.692$ ($R^2 = 0.9933$)	14.10	0.385

nential factor (A) of samples were obtained through the Coats-Redfern method. The fundamental rate equations used in kinetic studies and specific methods to derive fitting equations, values of Ea and A were referred to the research of Zheng et al. [17]. Data points in TG were used to derive fitting equations for each experimental run. As thermogravimetric analyzer recorded a point every 0.1 °C, so fitting equations was obtained by thousands of data points in TG. Pyrolysis kinetic parameters of lignite, bagasse and mixture at ratio of 20% are shown in Table 6.

$$Y = -2356x - 10.022 \quad R^2 = 0.9906 \quad 19.59 \quad 1.046$$

As shown in Table 6, all co-pyrolysis reactions were in accordance with first-order kinetic equation model. In the main pyrolysis region, activation energy (E) of BG was slightly higher than that of LC and pre-exponential factor (A) was significantly greater than the lignite's in Table 6. The lower activation energy (E) reflected higher reactivity, and the greater pre-exponential factor (A) meant the faster the reaction rate. Thus, it showed that the reaction rate of BG was faster than LC, but the reactivity was lower than LC. In general, the reactivity of coal was lower than biomass, especially the high rank coal. But the coal sample used in this study was a kind of lignite with high reactivity and low pyrolysis characteristic temperature (T_1 , T_{max} , T_2) as proved by thermogravimetric analysis. So E of LC was slightly lower than sugarcane bagasse. The high similitude between lignite sample and biomass made the high correlation coefficient (R^2) of fitting equation during blends co-pyrolyzed process. In addition, the activation energy (E) of the blends was less than that of lignite and biomass evidently during pyrolysis, which indicated the synergy between LC and BG. However, the decrease of E and A demonstrated the compensation effect between pyrolysis reactivity and reaction rate, but the synergetic effects between LC and BG were mainly manifested in the increased reactivity.

CONCLUSION

Lignite was co-pyrolyzed with sugarcane bagasse in a fixed bed reactor to investigate the possible interaction during co-pyrolysis process. The main conclusions are summarized below.

(1) The tar yield increased gradually with the rise of biomass ratio. Comparing the calculated values with the experimental values, the deviation enlarged with increasing proportion of BG in the feed mixture and the maximum positive deviation existed at BG ratio of 20%.

(2) The concentration of phenols and aliphatic compounds in the tar increased with the addition of biomass, while the content of aromatic compounds had the contradictory tendency. The experimental phenol content in co-pyrolysis tar reached 20.35%, which

increased by 142.26% compared with the calculated values.

(3) From TGA experiments, the BG decomposition peak partly overlapped with LC pyrolysis peak, which meant more interaction between lignite and BG during pyrolysis process. Moreover, the addition of BG improved the lignite reactivity to a certain extent by changing the coal pyrolysis process to a lower temperature region.

(4) The difference between the experimental and calculated values of pyrolysis products yield, tar components, DTG values and kinetics analysis indicated the synergetic effect between lignite and sugarcane bagasse.

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