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Investigation of sulfur forms and transformation during the co-combustion of sewage sludge and coal using X-ray photoelectron spectroscopy

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ABSTRACT

X-ray photoelectron spectroscopy was used to investigate the characteristics and evolution of sulfur (S) in mixtures of bituminous coal and sewage sludge (SS) and their chars during isothermal combustion. Five groups of mixtures with SS content of 0%, 10%, 20%, 30% and 100%, were examined at different burn-off ratios (β) of 0, 30%, 50%, 70% and 100%. The S in the coal mainly exist as the forms of mercaptan (S1), sulfide (S2), thiophene (S3), sulfoxide (S4), sulfone (S5) and sulfate (S6). During the coal combustion process, the content of S1 and S2 decreased, while that of S3 and S5 increased in the early stage and decreased in the late stage. The S4 content increased throughout the entire process of combustion. Small amount of S6 was detected, showing a fluctuated pattern. The trend of S1, S2, S5 and S6 in SS was alike with that in coal, whereas S4 decreased at the end of combustion. The changing process of S3 in SS was opposite to that of coal, while the composition of S in the mixtures resulted from the mixing of coal and SS. The transformation of each functional group during co-combustion were correlated with their transformation characteristics during the mono-combustion of coal and SS, and no obvious interaction was observed, which demonstrated that the coal-origin and SS-origin sulfur in mixtures kept their own characteristics in the combustion. SS may accumulate on the solid surface as α increase, resulting its significant influence on the evolution of each form of S. When α was low, most of the S-contained functional groups presented the characteristics of coal. The percentage of coal-origin functional groups declined as α increased. The transforming trends of most functional groups were similar with that of SS when α reached 30%.

1. Introduction

Co-combustion of sewage sludge (SS) and coal was favored due to several distinct advantages over other disposal methods [1–3]. As a result of growing environmental awareness, pollutant emissions during co-combustion were paid more attentions. SO_x emission mechanism of coal during mono-combustion had been studied by many researchers [4–6], however, the combustion character of SS may be different from coal, resulted from its particular characteristics, such as high contents of volatile matter and ash, and the low content of fixed carbon. Therefore, further studies are needed for better understanding of SO_x emission mechanism during co-combustion of coal and SS. Recently, Folgueras et al. [7] studied sulfur retention induced by Na_2O, K_2O and CaO in ash from co-combustion of three bituminous coals and a sewage sludge, while Elled et al. [8] investigated the interference of phosphorus on sulfur capture during co-combustion of sludge with wood. However, the conversion and evolution mechanisms of microscopic sulfur forms, which are of significance for better sulfur retention and desulfurization methods, had yet been fully explained.

X-ray photoelectron spectroscopy (XPS) can be used to identify elements (except H) on particle surface. It is one of most successful non-destructive techniques for studying the functionalities on coal surface. Schultz and Proctor [9] analyzed a range of organic and inorganic sulfur compounds as well as a number of coals using XPS, which indicated that XPS could distinguish among the forms of sulfur such as organic, pyritic and sulfate sulfur. From then on, Frost et al. [10], Jones et al. [11], Dutta et al. [12], Gorbaty et al. [13,14], Hittle et al. [15], Chen [16], Grzybek et al. [17], Olivella et al. [18] and Kozlowski [19] studied the forms of S in coal, concluding that S functionalities in coal included mercaptan, sulfide, thiophene, sulfoxide, sulfone, sulfone, sulfate and pyrite.

Research on the forms of S in SS received less attention. Dote et al. [20] utilized GC/MS to determine the S in oils derived from liquefaction of dewatered SS, suggesting that its main S species

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Table 1
Proximate and ultimate analysis of coal and sewage sludge.

Coal/SS	Proximate analysis (wt%)				Ultimate analysis (wt%)					Q (MJ kg^{-1})
	M	A	V	FC	C	H	O	N	S	
100/0	1.48	12.56	26.3	59.66	61.89	1.792	6.51	0.978	1.05	28.39
90/10	1.93	12.60	30.08	55.38	58.02	1.787	6.17	0.958	1.03	26.31
80/20	2.38	12.64	33.86	51.11	54.16	1.782	5.84	0.938	1.02	24.23
70/30	2.83	12.68	37.64	46.83	50.29	1.777	5.50	0.918	1.01	22.15
0/100	6.00	12.96	64.12	16.92	23.25	1.743	3.161	0.780	0.946	7.59

were mercaptan, sulfide and thiophene. Meanwhile, Harrison et al. [21], Kienhuis and Geerdink [22] and Merino et al. [23] also pointed out that sulfoxide, sulfone and sulfate existed in SS. Thus, the S-contained functionalities in SS were similar to that in coal.

In the view of the above, the study of S in mixtures of SS and coal during co-combustion was mainly focused on S retention or capture, whereas the conversion and transformation mechanisms of microscopic sulfur forms were rarely discussed [24–26]. Meanwhile, the study of S transformation and evolution of pure SS and pure coal were concentrated in pyrolysis, and was seldom presented for combustion. This study using XPS would improve the knowledge base on this subject. The first aim was to discuss the transformation mechanism of S in coal and SS during their mono-combustion. The second aim was to discuss evolution mechanism of S during co-combustion, as well as the relationship between mono- and co-combustion based on the findings from mono-combustion.

2. Experimental

2.1. Sample preparation

Experiment was performed on a bituminous coal sample and a SS sample. The SS was collected from a municipal sewage plant, Wuhan, China. Both samples were dried at 105 °C and ground into fine powder by pestle and mortar, and then passed through a 120-mesh screen. Five groups of mixtures, in which the mass percent α of SS were 0%, 10%, 20%, 30% and 100%, respectively, were obtained by finely stirring and mixing until uniform in appearance. Analysis data of samples were given in Table 1. Chars were prepared at the temperature of 850 °C under the environment of N_2 and O_2 (N_2:O_2 = 80:20) in a tuber reactor furnace. The excessive air coefficient value was 1.2. Five groups of chars (25 samples in all) were obtained, corresponding to coal and SS and their mixtures, and the burn-off ratio β was 0%, 30%, 50%, 70% and 100%, respectively. The experimental furnace was illustrated in Fig. 1. Chars were stored in vacuum prior to XPS analysis.

2.2. Apparatus

XPS experiment was performed using XSAM800 ESCA spectrometer (made by KRATOS Company) equipped with Mg Kα (1253.6 eV), excitation source being operated at 12.5 kV and 16 mA (200 W) at a base pressure of 5×10^{-7} Pa. The spectrometer was run in FRR mode at analyzer pass energy of 160 eV and 20 eV for whole and narrow scans, respectively. The spectra were recorded with middle resolution of 0.9 eV and sensitivity of 0.1%. The calibration was carried out to the main C 1s peak at 284.6 eV. Samples were mounted on double-sided adhesive tape. The vertical axis represented electronic number in the spectra while horizontal axis was electronic binding energies. The area of peak reflected the relative content, and the height contained information strength in S2p spectral line.

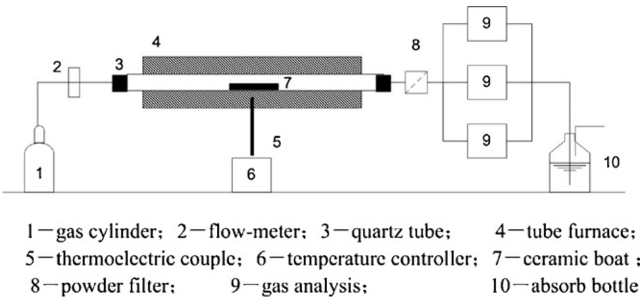


Fig. 1. The scheme of experiment for char preparation.

3. Result and discussion

Six to seven kinds of S functionalities were contained in coal and SS according to the literature, shown in Table 2. The formation process, as well as the formation condition, of coal and SS was quite different from each other, resulting in different combinational environment and various properties of the same functional group in these two substances.

3.1. S Functionalities in coal

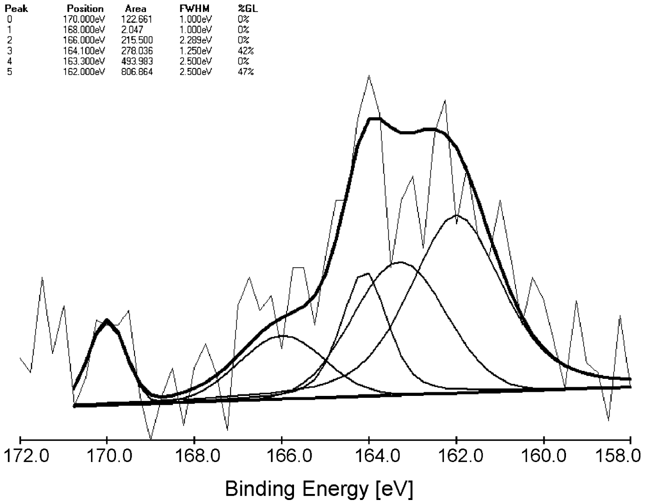
S2p spectra line of raw coal and peak-splitting result were presented in Fig. 2. Six components were required to achieve an acceptable fit in the XPS spectra, corresponding to S1, S2, S3, S4, S5 and S6, respectively. The concentrations of these S-contained functionalities derived from the fitted S2p components were 42.0%, 25.7%, 14.5%, 11.2%, 0.1% and 6.4% apart, which was in agreement with the findings in the literatures [13,24,26,27]. Since most of S7 was picked out manually, as well as oxidized to S6, no obvious S7 signal was found in the spectra.

The behavior of S functionalities in coal surface during combustion was shown in Fig. 3. In the process of combustion, the contents of S1 and S2 declined to 0 gradually. There are two reasons: (a) these S-contained functionalities would be released into environment as volatilize; (b) the alky sulfur, having poor thermal stability, was easily transformed into the more stable modes, such as S3–S5, at the presence of oxygen [13,14,25,26].

The contents of S3–S5 rose up in early stage of combustion, which was supported by the previous studies [13,14], may include two aspects: first, the oxide sulfur (S4, S5) and aromatic sulfur (S3) was s at low temperature and easily to retain in the char; second, the decreasing of S1 and S2, being released as volatilize or trans-

Table 2
Binding energies of sulfur functionality in coal.

S-contained functionality	Symbol	Binding energy/eV	References
Mercaptan	S1	162.2 ± 0.3	[16,34]
Sulfide	S2	163.3 ± 0.4	[17,19,24,27,34]
Thiophene	S3	164.1 ± 0.2	[17–19,27,34]
Sulfoxide/sulfide	S4/S4′	166.0 ± 0.5	[17,19,24,27,29,34]
Sulfone	S5	168.0 ± 0.5	[16,17,19,24,27,34,35]
Sulfate	S6	170.0 ± 1.0	[10,17,19,24,27,34,36]
Pyrite	S7	162.5 ± 0.5	[19,25,34,37]



Peak	Position	Area	FWHM	%GL
0	170.000eV	122.661	1.000eV	0%
1	168.000eV	2.047	1.000eV	0%
2	166.000eV	215.500	2.289eV	0%
3	164.100eV	278.036	1.250eV	42%
4	163.300eV	493.983	2.500eV	0%
5	162.000eV	806.864	2.500eV	47%

Fig. 2. S2p spectral line of raw coal.

formed into S3–S5, induced by the increasing relative contents of these functionalities.

S3 and S5 contents decreased in terminal stage of combustion. S3 may be oxidized into the oxide-S in the oxygen-enriched environment, resulting in the decrease of its content. However, it was reported that the S3 content increased and reached the maximum of 80% under the severe conditions of pyrolysis [26,28]. Actually, oxygen was absent in these pyrolysis experiments, which was opposite to this work. This discrepancy may result from the distinct react conditions. The temperature was so high on particle surface that S5 began to be decomposed at the end of combustion, which was in accordance with the study by Liu et al. [29].

S4 content increased throughout the entire combustion process. Previous studies [24] showed that S4 can be decomposed at 673 K, indicating that its tendency of it, like that of S5, may decline from middle stage of combustion, which was different from this experiment. In fact, the chemical valence of sulfite was identical to that of S4, suggesting that the binding energies of the two functionalities were close to each other. Part of S may react with mineral substance on coal surface and transformed into sulfite during combustion, leading to the increasing of the component ascribed to S4 in XPS spectra. Thus the increasing of S4 in terminal stage may be related to the formation of sulfite.

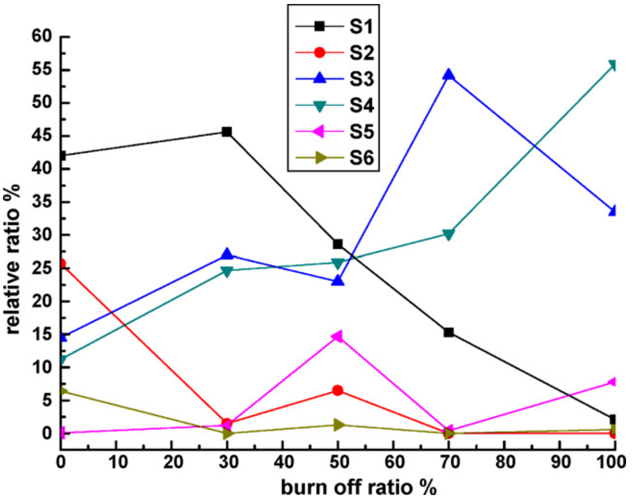
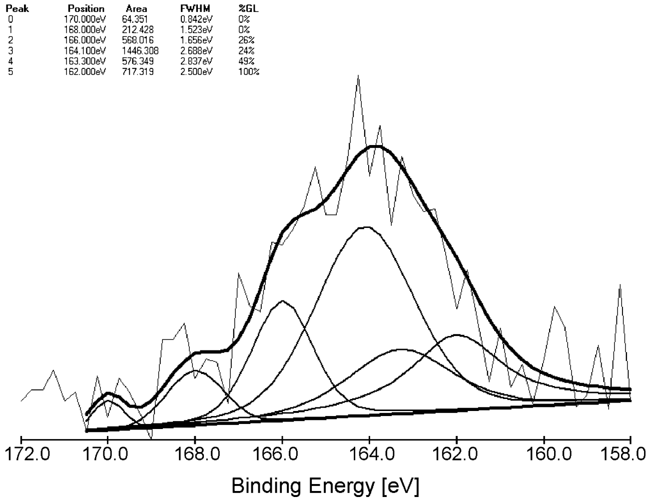


Fig. 3. Evolution of S functionalities in coal during combustion.



Peak	Position	Area	FWHM	%GL
0	170.000eV	64.351	0.842eV	0%
1	168.000eV	212.428	1.523eV	0%
2	166.000eV	568.016	1.656eV	26%
3	164.100eV	1446.308	2.688eV	24%
4	163.300eV	576.349	2.837eV	49%
5	162.000eV	717.319	2.500eV	100%

Fig. 4. S2p spectral line of SS.

Small amount of S6 was detected and the fluctuant characteristic of its content was observed, reflecting the enrichment and liberation of organic sulfur on granule surface during combustion. S6 decreased when the organic sulfur were concentrated on coal surface and increased when organic sulfur were decomposed and released.

3.2. S Functionalities in SS

The S functionalities in SS were similar to those in coal, however, the different tendency of transformation was observed. S2p spectral line of SS was given in Fig. 4. S1–S6 were used to obtain an acceptable fit, and their contents were 20%, 16.1%, 40.4%, 15.8%, 5.9% and 1.8%, respectively. The S1 and S2 contents in SS were lower than that in coal while S3 was higher, being relative with their distinct formation conditions.

The evolution of S functionalities in SS surface during combustion was presented in Fig. 5. S1 and S2 decreased gradually, which was identical to that in coal. The explanation could be similar to coal, being released into environment as volatilize or transformed into other forms.

S5 was relatively low and presented the tendency of increasing at the beginning and decreasing at the end, being consistent with that in coal except that tendency was milder. The reasons may be

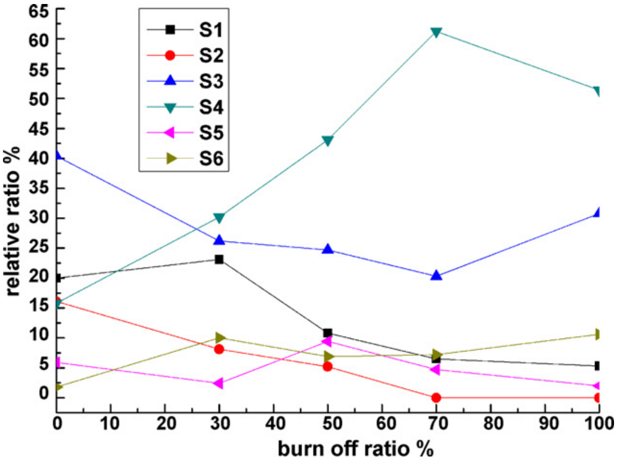


Fig. 5. Evolution of S functionalities in SS during combustion.

referred to the coal character. Besides, S5 was mainly influenced by the variation of other forms of S because of its low concentration.

The fluctuant characteristic of S6 was also obtained in SS. However, the concentration of S6 in SS was higher than coal, fluctuating around 10%. The large CaO and MgO contents present in SS ash could retain part of SO_2 produced during combustion, forming S6. This was in agreement with the previous studies [7,30].

S4 increased at the beginning of combustion, showing no difference with that in coal. However, the decrease of this functionality in the end of combustion was observed. S4 may be formed by the conversion of S1 and S2 under low temperature at the presence of oxygen in early stage and decomposed to other forms under high temperature in terminal stage [13,14,28]. It also demonstrated that the increasing mount of S4 in the end of combustion in coal could be caused by the formation of other S-contained functionality, such as sulfite.

The decreasing trend of S3 in SS was observed, being opposite to that of coal. Reference data [31,32] showed S3 in coal usually contained multi-ring and even more complex structure may be expected with the coalification degree. The formation condition of SS was different from that of coal, without the process under high temperature and pressure for long-period. Single-ring contained S3 may be expected as the main component of S3 in SS. Single-ring S3 may be released as volatilize at the temperature of 673 K [24]. Thus the single-ring contained S3 may be decomposed when combustion started while multi-ring contained S3 were more stable and easily retained in chars, inducing the different trend of S3 in coal and SS. Besides, the discrepancy may also be ascribed to the inherent nature of coal and SS, which still need to be discussed.

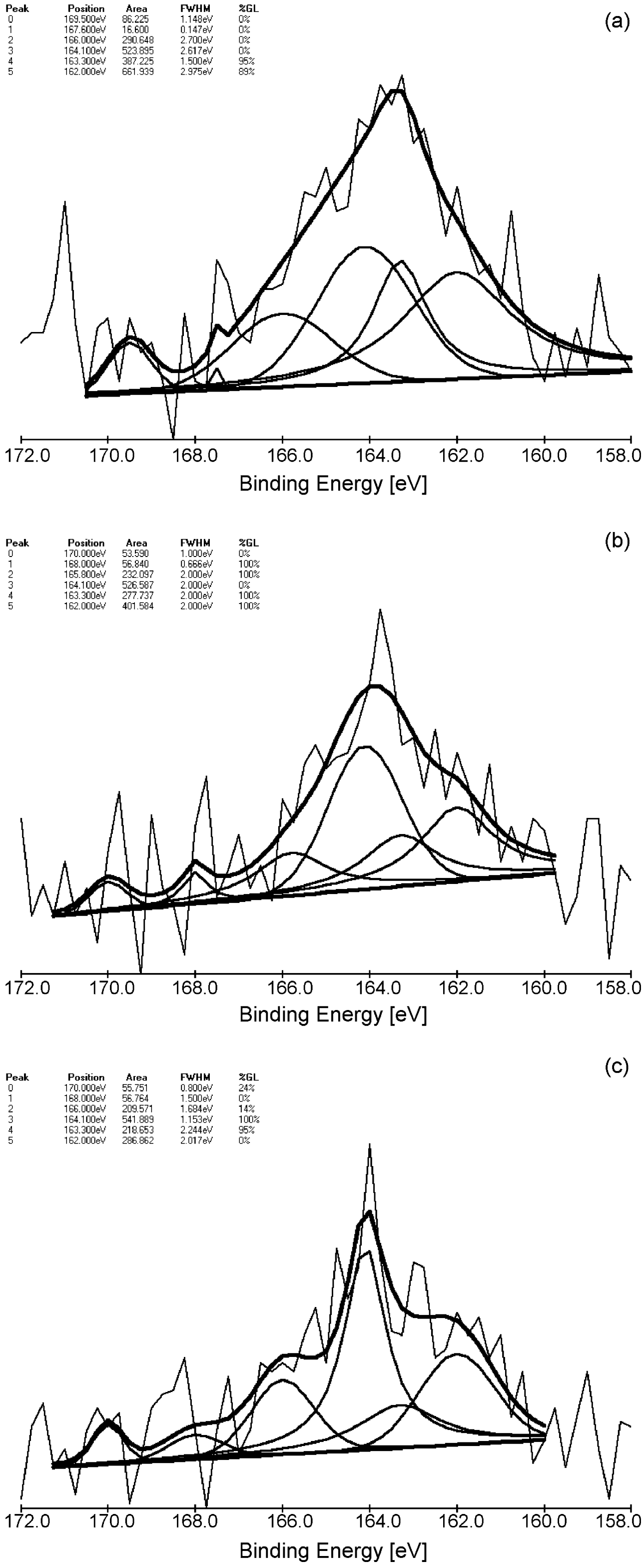
3.3. S functionality in the mixtures of SS and coal

3.3.1. S functionality in original mixtures of SS and coal ($\beta = 0$)

S2p spectra line of original mixtures of coal and SS ($\beta = 0$), as well as peak-splitting result, was given in Fig. 6. The mass percent α of SS were 10%, 20% and 30%, respectively. There were also six components in the mixtures, corresponding to S1–S6 separately. However, their contents were different from that in coal or SS.

The evolution of S functionalities in original mixtures with mass percent α of SS, was presented in Fig. 7. Fig. 7a was obtained in experiment while Fig. 7b was calculated by the contents of each functional group in coal and SS, as well as the atomic proportion of S from coal and SS in mixtures. The tendency of each component in Fig. 7a was consistent with that in Fig. 7b, illustrating that S in the mixtures resulted from the accumulation of that in coal and SS and no obvious reaction was observed.

More information could be obtained by the comparison of Fig. 7a and b. The experimental contents of the functionalities, which were in low concentration in SS such as S1 and S2, were significantly lower than the calculated values in the mixtures, whereas that in high concentration in SS such as S3 and S4, were obviously higher than the calculated values. This may be caused by the enrichment of SS on mixtures surface. More notable deviations were observed as the mass percent α of SS grew. When α reached 30%, the contents of S1–S4 in mixtures had been close to that in SS, but far away from the calculated values, showing serious enrichment of SS on mixtures surface. The behaviors of S5 and S6 were less evident for their low concentrations.



Peak	Position	Area	FWHM	%GL
0	169.500eV	86.225	1.148eV	0%
1	167.600eV	16.600	0.147eV	0%
2	166.000eV	290.648	2.700eV	0%
3	164.100eV	523.895	2.617eV	0%
4	163.300eV	387.225	1.500eV	95%
5	162.000eV	661.939	2.975eV	89%

Peak	Position	Area	FWHM	%GL
0	170.000eV	53.590	1.000eV	0%
1	168.000eV	56.840	0.666eV	100%
2	165.800eV	232.097	2.000eV	100%
3	164.100eV	526.587	2.000eV	0%
4	163.300eV	277.737	2.000eV	100%
5	162.000eV	401.584	2.000eV	100%

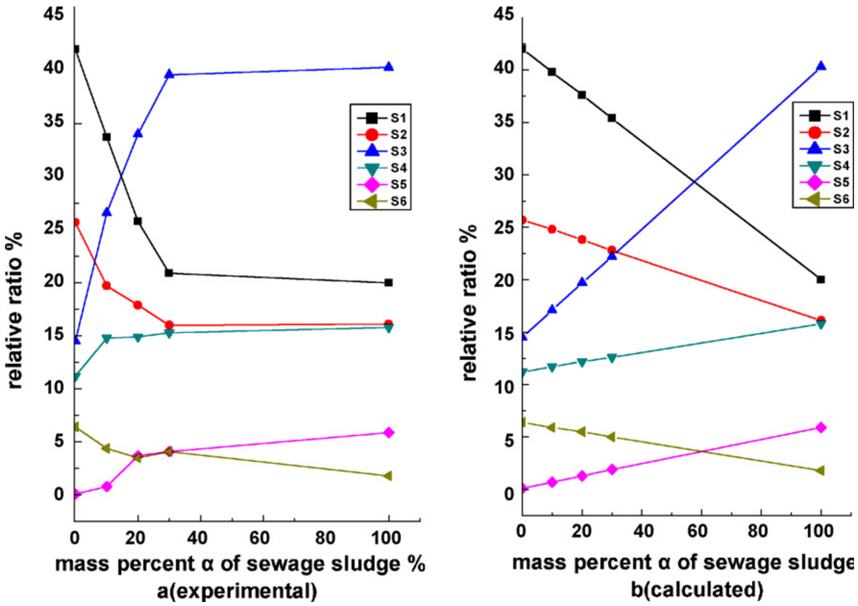
Peak	Position	Area	FWHM	%GL
0	170.000eV	55.751	0.800eV	24%
1	168.000eV	56.764	1.500eV	0%
2	166.000eV	209.571	1.684eV	14%
3	164.100eV	541.889	1.153eV	100%
4	163.300eV	218.653	2.244eV	95%
5	162.000eV	286.862	2.017eV	0%

Fig. 6. S2p spectral line of blends ((a) $\alpha = 10\%$; (b) $\alpha = 20\%$; (c) $\alpha = 30\%$).

3.3.2. S functionalities in the chars of mixtures

The evolutions of S-contained functionalities in mixtures during co-combustion were shown in Figs. 8–10. Mass percent α of SS were 10%, 20%and 30%, respectively. Figs. 8a–Fig. 10a were gained in this experiment while Fig. 8b–Fig. 10b were calculated by the atomic ratio of S from coal and SS in mixtures and the contents of each functional group during their mono-combustion (shown in Figs. 3 and 5).

The experimental results of all the functionalities agreed well with that of calculated values at different mass percent α of SS in Figs. 8–10, indicating that combustion of S in mixtures both kept their own characteristics in the process of reaction. If it was not the



a(experimental)

b(calculated)

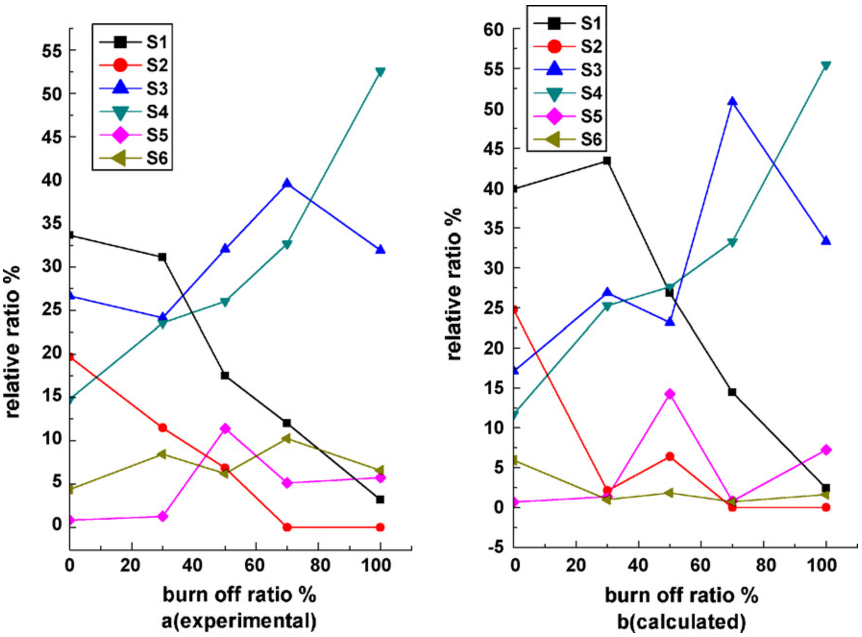
Fig. 7. Evolution of S functionalities in original blends as α grows.

case, more deviation may be expected between experimental data and calculated value.

The experimental evolution of each functional group was completely consistent with that of calculated value at different burn-off ratio β in Fig. 7, except that experimental values of S1 and S2 were a little less than the calculated values, whereas that of S3 and S4 were a little higher when $\beta = 0$ (stated by Section 3.3.1). From comparison of Figs. 3 and 5 with Fig. 8a, it was obtained that the evolution of S1–S5 during co-combustion showed no difference with that in coal during mono-combustion, but showed great difference with that in SS. S6 also presented fluctuant characteristic during co-combustion and no obvious inclination to coal or SS was observed. Thus, it was deduced that S functionalities in coal and SS both kept their own characteristics during co-combustion, which resulted that the characteristic of the component in high proportion would play a dominant role. The mass percent α of SS was 10% while that of coal was 90% in Fig. 8, causing that the trend of S functionalities in mixtures was mainly in agreement with that of coal.

The well agreement between experimental and calculated data was also gained in Fig. 9, except the further deviation of experimental data of S1–S4 from the calculated value caused by the enrichment of SS on coal surface when $\beta = 0$. The transformation of S1, S3 and S4 in mixtures was still consistent with that of coal by the comparison of Fig. 3, Fig. 5 with Fig. 9a. However, the tendency of S6 in mixtures was mainly in agreement with that of SS. This was ascribed to the higher content of S6 in SS and its chars than coal during combustion. S2 and S5 in mixtures presented the both characteristics of coal and SS.

The situation in Fig. 10 was similar to that in Fig. 9. Part of the S functionalities began to represent the characteristic of SS. The ten-



a(experimental)

b(calculated)

Fig. 8. Evolution of S functionalities in blends as β grows ($\alpha = 10\%$).

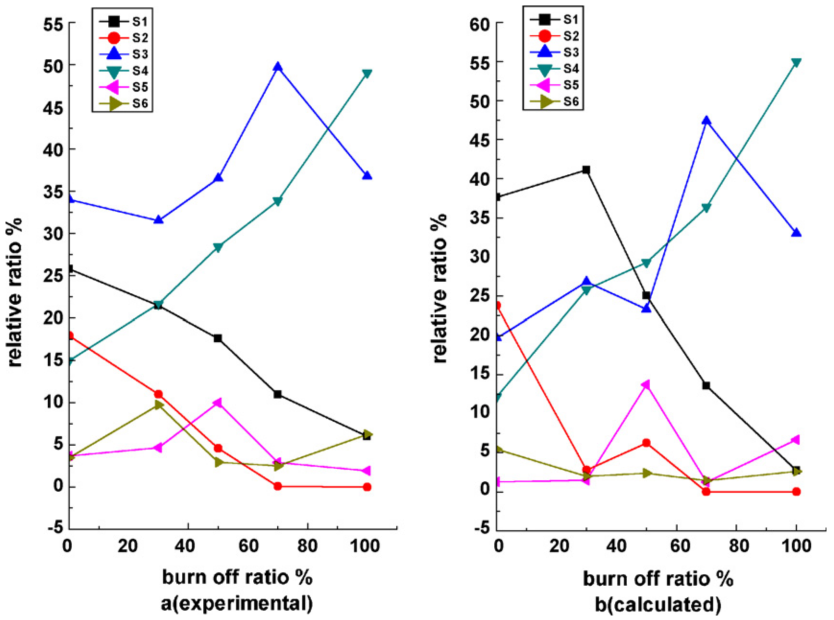


Fig. 9. Evolution of S functionalities in blends as β grows ($\alpha = 20\%$).

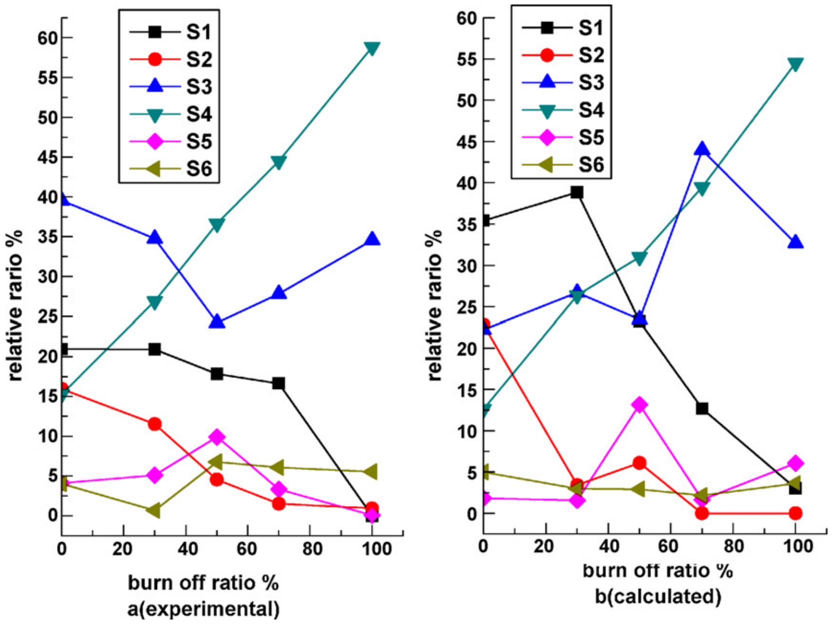


Fig. 10. Evolution of S functionalities in blends as β grows ($\alpha = 30\%$).

dency of S1–S6 in mixtures became to accord with that in SS while S4 performed the effect of both coal and SS. On one hand, the surface content of SS outclassed its bulk content in mixtures as the growing of mass percent α and the enrichment of SS on mixtures surface, leading the majority of functionality to exhibit the characteristic of SS. On the other hand, the each content of S4 in coal and SS was in high level, showing the both characteristic of the two components.

In summary, most of the S-contained functionality in mixtures presented the characteristic of coal under low mass percent α of SS. However, SS gathered on mixtures surface gradually and some S-contained functionality began to exhibit the characteristic of SS as the mass percent α of SS increased. When α reached 30%, the tendency of most functionality was in agreement with that in SS. Meanwhile, the enrichment of SS on mixtures surface had great influence on the evolution of the functionality. When the bulk mass percent of SS was only 30%, the majority of the functionality on mixtures surface performed the characteristic of SS. This demonstrated that even though the mixing amount of SS was low in mixtures, it maybe have obvious effect on the co-combustion, because the fuel surface properties were very important for its combustion. Besides, the S6 contents obtained in this experiment were always higher than the calculated values from Figs. 8–10, contributed to the reaction between SO_2 and alkaline oxides contained in SS ash.

4. Conclusions

XPS was utilized to investigate the evolution of S functionalities presented in coal, SS and their chars during mono- and co-combustion. The following conclusions were obtained:

1. The S in the bituminous coal mainly existed in the forms of mercaptan (S1), sulfide (S2), thiophene (S3),sulfoxide (S4), sulfone (S5) and sulfate (S6). The contents of S1 and S2 decreased during combustion while that of S3 and S5 increased in early stage and decreased in terminal stage of combustion. S4 ascended in whole combustion period. Small amount of S6 was detected and the fluctuant characteristic of it was reached.
2. S functionalities in SS were similar to that in coal, and also exist as S1–S6. The tendencies of S1, S2, S5 and S6 in SS were consistent with those of coal. However, S4 decreased in the end of combustion. The evolution of S3 in SS was opposite to that of coal.
3. S in the mixtures resulted from the accumulation of that in coal and SS respectively. SS may gather onto the mixtures surface as the mass percent α of SS grew up.
4. The combustion of S in mixtures both kept their own characteristics in the process of reaction, and no obvious interactions were observed. When α was low, most of the S-contained functional groups presented the characteristics of coal while part of the functionalities were inclined to SS as α grows. Even more, the tendencies of most functional groups were in agreement with that of SS when α reached 30%. The enrichment of SS on mixture surface had great influence on the evolution of the functionality.

Acknowledgements

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