



Control of Air Pollutants Emission and Improvement of Incineration Rate During Incineration of Oily Sludge-Based Briquette

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ABSTRACT

The incineration technology of oily sludge-based briquette makes great contributions to oily sludge treatment. In order to resolve these problems, air pollutants (SO₂ and organic gas) emission and low incinerating rate during the briquette incineration, the effects of additives (CaCO₃, CaO; K₂CO₃, Na₂CO₃, NaCl; KMnO₄, KNO₃, NaNO₃; Fe₂O₃, KClO₃) on the generating gas and incinerating rate during incineration were studied. The results show that CaCO₃ is more effective than CaO for fixing sulphur into residue, and K₂CO₃ improved the sulphation efficiency of calcium-based additives, which is more useful than NaCl and Na₂CO₃ did. As for the incinerating rate of briquette, KMnO₄ is more effective than KNO₃ and NaNO₃. The maximum of incinerating rate of briquette is improved from 0.38 to 0.6 g/min and Fe₂O₃ and KClO₃ can apparently reduce the release of organic gas during the incineration of briquette. But KClO₃ can greatly reduce the release of sulphur. When the adding amount of CaCO₃, K₂CO₃, KMnO₄ and KClO₃ is 16‰, 12‰, 18‰, 6‰ respectively in the process of briquette preparation, the sulphation efficiency reached the maximum of 95.3%, and there is still an extremely small amount of organic gas in flue gas. CaCO₃, K₂CO₃, KMnO₄ and KClO₃ used in emission control of air pollutants and full combustion of briquette during incineration of oily sludge based-briquette, demonstrates high efficiency and low emission compared to other additives tested in this study.

INTRODUCTION

Oily sludge, composed of petroleum, minerals and water, generating from oil industry, has been listed in the Chinese national hazardous waste lists (Hu et al. 2013), and there is almost more than three million tons of oily sludge in China (Zhang et al. 2012), which leads to serious impacts on the ecological environment. For example, hydrocarbons, heavy metals and a large amount of petroleum hydrocarbon in oily sludge seriously affect the water, soil and air (Souza et al. 2018).

By now, there have been many effective methods to treat and dispose oily sludge, such as chemical cleanliness (Sahebnazar et al. 2017, Liang et al. 2017), centrifugation (Lin et al. 2017), low-temperature pyrolysis (Zhao et al. 2017), ultrasound treatment (Hu et al. 2017), biological treatment (Casarini et al. 1988), chemical oxidation (Jing et al. 2012, Matsodoum Nguemté et al. 2018) and incineration treatment (Karamalidis et al. 2008). Among these oily sludge treatments, the incineration treatment can not only minimize the volume and reuse thermal energy of oily sludge, but can also transform most of the toxic and harmful

substances into steady and nontoxic substances during the process. And thus, the incineration has been considered as the mainstream technology to treat oily sludge (Hu et al. 2017, Polc et al. 2016).

As the share of non-combustible components in oily sludge is high and the calorific value is not uniform, which cause the cost of direct incineration to be huge (Xu et al. 2014). Thus, the oily sludge-based briquette has been considered to overcome the problems. Oily sludge-based briquette is a mixture of oily sludge, coal and biomass, and can be widely used in industrial boilers (Lopes et al. 2001). What is more, the technology of oily sludge-based briquette may not only resolve the problem of insufficient energy and huge cost during the direct incineration of oily sludge, but can also save the raw materials of industrial briquette. At present, there have been some studies about the preparation of oily sludge-based briquette (Magdziarz et al. 2013, Kijokleczkowska et al. 2016). However, SO₂ and organic gas generated during the incineration of briquette and the low incinerating rate limit the application of briquette in industry. Fortunately, the addition of additives to briquette offers a chance to resolve the problems above. As we know different

kinds of additives play different roles, mainly reducing the release of air pollutants and promote the incineration of coal or other solid fuel (Rulkens et al. 2008, Wu et al. 2002). In addition, the kinds of calcium compounds, such as calcium magnesium acetate and calcium carbonate, can effectively reduce the produce of SO₂ in flue gas (Nimmo et al. 2004). Meanwhile, calcium magnesium acetate can simultaneously reduce NO_x and SO_x when the coal was incinerated (Guan et al. 2003). Alkaline metal compounds have also been used as the additive to coal-firing, which can enhance carbon combustion, reduce air pollutant emissions and promote the sulfation efficiency of calcium compounds (Liu et al. 2004, Li et al. 2007, Zhou et al. 1997). For example, Manquais et al. (2012) systematically performed the influence of alkali metals and alkaline earth metals on combustion performance of coal, and demonstrated that alkali metal carbonate compounds and chloride can effectively promote the burnout of coal, the more coal combustion, and lesser emissions of organic gases during combustion. However, chloride, especially sodium chloride, can distinctly shorten the burnout time of coal compared with other alkali metals and alkaline earth metals.

The above studies can provide precious experience about the coal-fired additives, but these studies concentrated on the effect of single additive on the combustion of coal. At present, there are few studies about the influence of mixed additives on the air pollutants emission of coal. Therefore, the target of this study is to select the appropriate mixed additives to control the emission of air pollutants (SO₂ and organic gas) and ensure the incinerating rate of briquettes simultaneously. In addition, the effect of adding an amount of mixed additives on the emission of air pollutants and the improvement of incinerating rate was also studied. The additives can be divided into four species: calcium-based additive (being beneficial to reducing sulphur dioxide

emissions), sulphation-supporting additives (being good to improving sulphation efficiency of calcium-based additive), oxidizers (Gong et al. 2009) (being beneficial to improving the incinerating rate) and catalysts (being beneficial to control the emission of organic gas), and the four additives will be added into the briquette. All incineration experiments were carried out under the temperature of 900°C.

MATERIALS AND METHODS

Oily Sludge, Coal and Apricot Shell Samples Collection and Pretreatment

The oily sludge was obtained from ZhangGuanmiao joint station of Wuqi County, Yan'an City, China. The physical and chemical properties are presented in Table 1, and the share of non-combustible components of oily sludge is high according to the Table 1. The coal and apricot shell were obtained from Yulin, China. The elemental analysis and ash constituent results of the experimental material are given in Table 1 and Table 2.

Screening of Added Pharmaceuticals

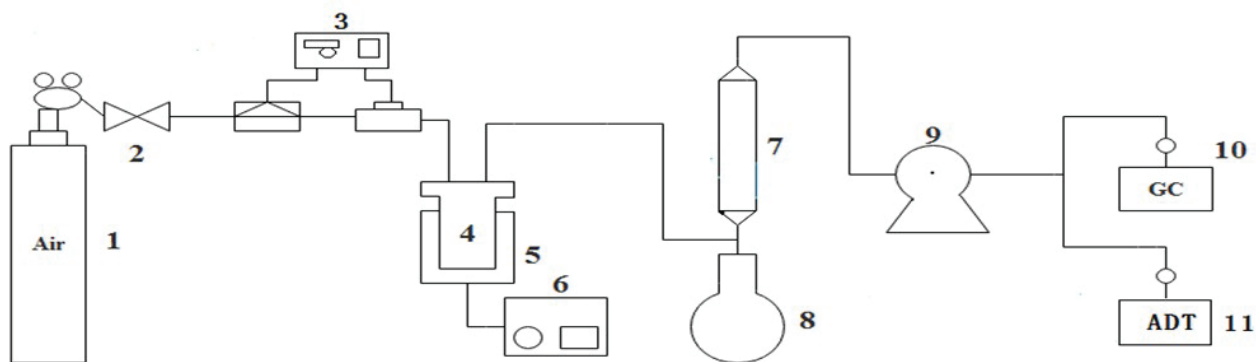
Pharmaceuticals were added into the briquette according to a certain amount. All chemicals and reagents used were of analytical grade. Different species of calcium-based additives (CaO, CaCO₃) were added during the preparation of the oily sludge-based briquette, and the amount of calcium-based additive was changed, and then the oily sludge-based briquette was incinerated directly. The schematic picture of the experiment flow is illustrated in Fig. 1. The sulphur contents in residues were determined by elemental analysis with EDS (Energy Dispersive X-Ray Spectroscopy). The total sulphur in briquette was calculated by equation (1). The sulphation efficiency was calculated by equation (2). According to the equation (2).

Table 1: Ultimate analysis results of experimental materials (wt%).

Sample	Ultimate analysis (wt %)					trace element analysis (mg/kg)								
	C	H	O	S	N	Hg	Cu	Cr	Zn	As	Pb	Ba	Ni	Cd
Coal	74.68	11.13	13.23	0.8	0.16	0.02	5.11	12.42	6.85	0.9	11.8	110	15.31	0.11
Oily sludge	55.45	16.63	21.24	6.46	0.22	0.03	9.62	30.11	100	3.86	13.5	0.02	18.92	0.01
Apricot	52.36	12.92	33.92	0.06	0.81	-	0.01	-	0.21	-	-	-	0.01	-

Table 2: Analysis of ash constituents (wt %).

Sample	SiO ₂	CaO	Al ₂ O ₃	MgO	TiO ₂	SO ₃	K ₂ O	Na ₂ O	P ₂ O ₅	BaO	Cr ₂ O ₃
Coal	67.68	2.41	22.8	2.69	0.81	0.4	1.6	0.5	0.1	0.1	0.01
Oily sludge	68.55	5.47	17.3	2.46	4.73	0.09	0.03	0.02	0.01	0.01	0.01
Apricot	17.16	-	2.43	14.68	-	2.46	31.06	2.48	8.18	0	0.04



1 air cylinder 2 valve 3 volume flow controller 4 reactor 5 heating furnace 6 temperature controller 7 condenser
8 flask with round bottom 9 gas flowmeter 10 gas chromatograph 11 automatic dust (gas) tester

Fig. 1: Schematic picture of experiment flow.

$$S_T = \omega_S \times 6.64\% + \omega_C \times 0.8\% + \omega_A \times 0.06\% \quad \dots(1)$$

(ω_S : the weight of oily sludge in briquette; ω_C : the weight of coal in briquette; ω_A : the weight of apricot shell in briquette; S_T : total sulphur in briquette)

$$\text{sulfation efficiency (\%)} = \frac{S_0}{S_T} \times 100 \quad \dots(2)$$

Where, S_0 : the weight of sulphur in residues.

The selection of sulphation-supporting additives (NaCl, Na_2CO_3 , K_2CO_3) was performed after the screening of calcium-based additives. The three sulphation-supporting additives were mixed with materials (coal, oily sludge and binder) of briquette and calcium-based additive. One of the three sulphation-supporting additives that can enhance the sulfation efficiency of calcium-based additive will be selected to add into the briquette. The screening of oxidizers (KMnO_4 , NaNO_3 , KNO_3), catalysts (KClO_3 , Fe_2O_3) is similar to sulphation-supporting additives. Whereas, the selection of oxidizers is based on average incinerating rate of briquette and the sulphation efficiency of calcium-based additive. The selection of catalysts is based on the share of organic gas in flue gas and the sulphation efficiency of calcium-based additive.

Measuring the Flue Gas of Incineration

The flue gas was measured every 15 minutes with SP-3420A gas chromatograph and Laoying 3022H automatic dust (gas) tester (ADT). The sulphur released into atmosphere can be obtained by difference of the total sulphur in briquette and the sulphur contents in residue.

Measuring the Incinerating Rate of Briquette

The average incinerating rate of different incineration stages

was computed by the ratio of incinerating weight loss to time. The oxidizers were screened according to the average incinerating rate, the higher the incinerating rate, the better the incineration-supporting effect of the oxidizers.

RESULTS AND DISCUSSION

Effect of the Species and Adding Amount of Calcium-Based Additive on the Sulphation Efficiency

From Fig. 2, we can see that when the amount of calcium-based additives was 10%, the sulphation efficiency of CaO and CaCO_3 was 52% and 64% respectively. With the adding amount of CaCO_3 increased from 10% to 16%, the sulphation efficiency of CaCO_3 increased from 64% to 83%. When the amount of CaCO_3 was 16%, the sulphation efficiency of CaCO_3 reached the maximum value as 83%, which indicates that much sulphur transfers to residue and less sulphur transfers to atmosphere during the process of incineration. However, the added CaO slightly affects the sulphur content in incinerating residue. The maximum sulphation efficiency was 60% when CaO was used as the calcium-based additive. According to the analysis above, we can see that the addition of CaO and CaCO_3 can transfer part of sulphur from the flue gas to the residue in the process of incineration. When the amount of calcium-based additives was uniform, the efficiency of sulphation of CaCO_3 was higher than CaO. Therefore, CaCO_3 is more suitable for the calcium-based additives of briquette.

Effect of the Species and Adding Amount of Sulfation-Supporting Additives on the Efficiency of Sulphation

Although the sulphation efficiency of calcium-based

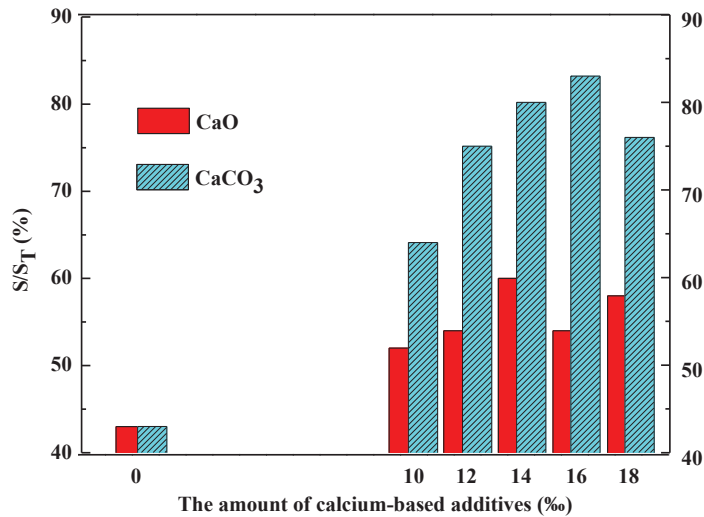


Fig. 2: Impact of the species and amount of calcium-based additives on the sulphation efficiency.

(CaCO₃) can reach to 83%, a certain amount of sulphur was still emitted into the atmosphere in the form of sulphur dioxide. Therefore, it is necessary to add different kinds of sulphation-supporting additives (K₂CO₃, Na₂CO₃, NaCl) during the preparation of briquette to improve the sulphation efficiency of CaCO₃.

Fig. 3 indicates that the addition of sulphation-supporting additives has a certain effect on the efficiency of sulphation. When the amount of Na₂CO₃ was 9‰, the sulphation efficiency of CaCO₃ reached to a maximum value of 88%. When the amount of NaCl was 10‰, the sulphation efficiency of CaCO₃ reached a maximum value

of 85%. In addition, the efficiency of sulphation improved with the increase of K₂CO₃, when the adding amount of K₂CO₃ was 12‰, the sulphation efficiency of CaCO₃ reached the maximum value of 94%. Thus, three kinds of sulphation-supporting additives increased the efficiency of sulphation of CaCO₃ to some extent. In addition, under the same conditions, the effect of K₂CO₃ on the sulphation efficiency of CaCO₃ was extremely obvious. K₂CO₃ can greatly improve the sulphation efficiency of CaCO₃. However, Na₂CO₃ and NaCl only slightly promote the sulphation efficiency of CaCO₃. Thus, K₂CO₃ is more suitable for the sulphation-supporting additives.

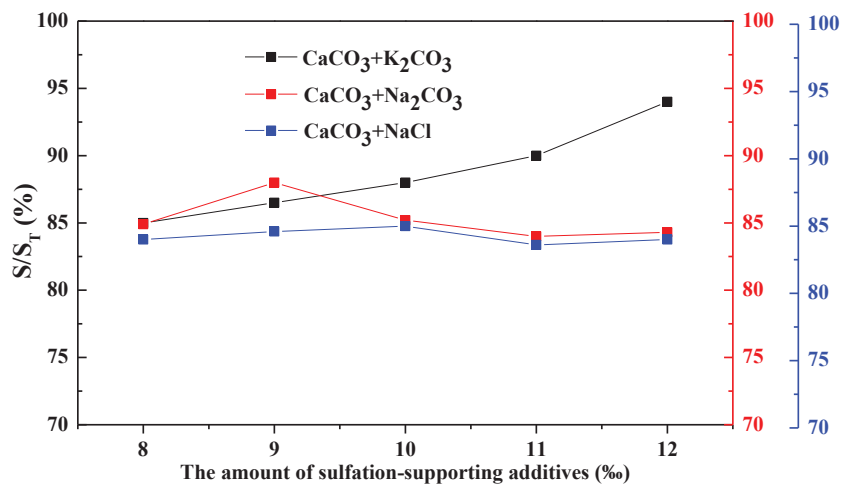


Fig. 3: Impact of species and adding amount of sulphation-supporting additives on the sulfation efficiency of CaCO₃.

There are two reasons why K_2CO_3 can improve sulphation efficiency of $CaCO_3$. Firstly, the added K_2CO_3 can react with SO_2 , the possible reaction can be as below:



Secondly, K_2CO_3 presents a melting state at high temperature as $891^\circ C$, which can change the surface area and microstructure of CaO that is produced through decomposition of $CaCO_3$. The results of X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) can also prove the above conclusions.

The (XRD) results given in Fig. 4 illustrate that the fixed sulphur by calcium-based additives turned into $CaSO_4$ when no sulphation-supporting additive was added. At the same time, barium and magnesium compounds in briquette can also act as sulphur fixation during the incineration process of briquette. Sulphur is converted to $Mg_3S_2O_8(OH)_2$ and $BaSO_4$ in the residue. In addition, $CaCO_3$ is still contained in the residue, which indicates that $CaCO_3$ is not fully utilized. When the sulphation-supporting additive K_2CO_3 was added, the fixed sulphur remained in residue in the form of $CaSO_4$, K_2SO_4 and $BaSO_4$. Calcium in the residue exists in the form of $CaSO_4$, which indicates that the added $CaCO_3$ is fully utilized. Therefore, the sulphation-supporting additive K_2CO_3 not only can react with sulphur to fix sulphur, but can also improve the utilization of $CaCO_3$.

The picture located in the upper of Fig. 5 is a sample calcined solely from $CaCO_3$ at $900^\circ C$ for an hour, the bottom is the sample calcined from the mixture of $CaCO_3$ and

K_2CO_3 at the same condition, and the surface characteristic of the samples above is distinctive. The surface of the upper of Fig. 5 is smooth and the bottom is coarse, which indicates that the added K_2CO_3 can promote surface area and enrich surface pore of CaO that was produced through decomposition of $CaCO_3$. The sulphation of $CaCO_3$ at high temperature contains three stages: the decomposition reaction of $CaCO_3$, the diffusion and transfer process of gaseous phase in solid phase, and the combined between CaO and SO_x . And thus, the pore and large surface area are beneficial to the release of CO_2 and the fast combination of SO_2 with CaO (Li 2012).

Effect of Oxidizers on the Incinerating Rate of Briquette

In order to increase the incinerating rate of briquette, different species of oxidizers were also added during the preparation of the briquette and average incinerating rate of the briquette was measured at $900^\circ C$. With the same amount of addition (12%), different oxidizers were added into the briquette, the average incinerating rate of the briquette is shown in Fig. 6.

It can be seen from Fig. 6 that the oxidizers promote the incineration of the briquette and increase the incinerating rate of the briquette. Especially in the early stage, the incinerating rate has been greatly improved. Because the three kinds of oxidizers can produce oxygen under the condition of certain temperature, oxygen can promote the incineration of the briquette. However, the decomposition

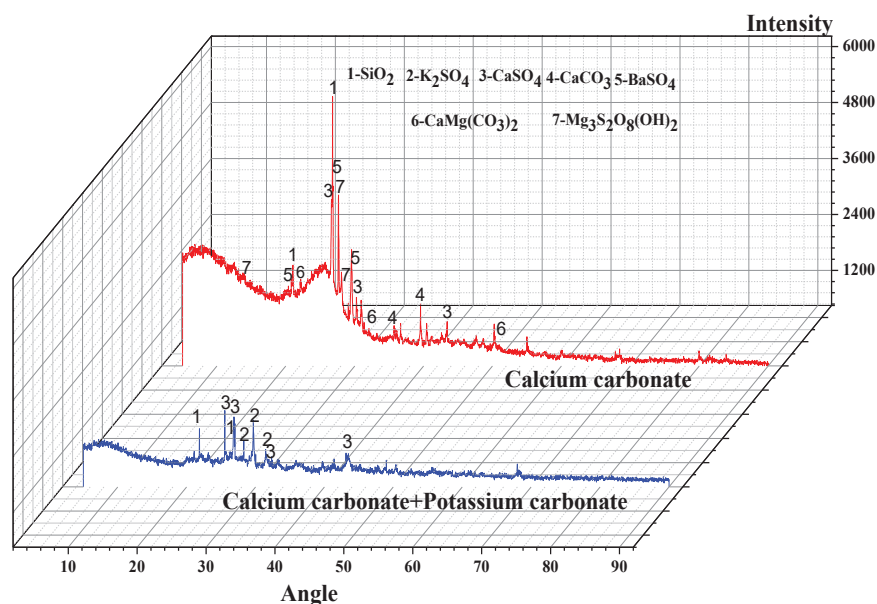


Fig. 4: The XRD spectrum of the incinerating residue.

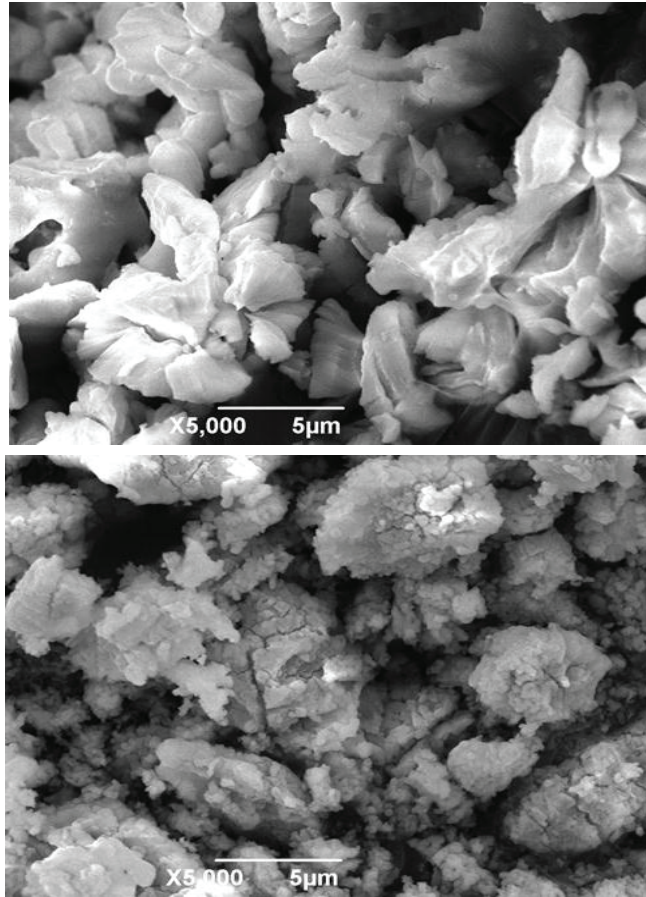


Fig. 5: SEM images of CaO produced through decomposition of CaCO₃.

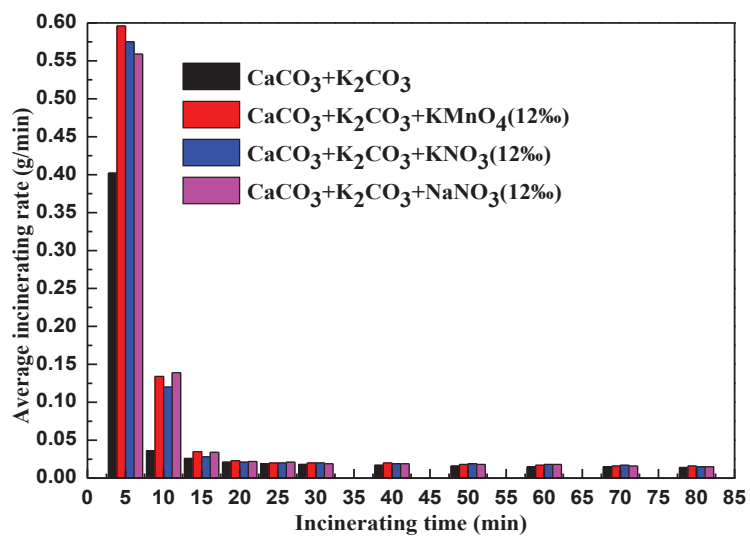
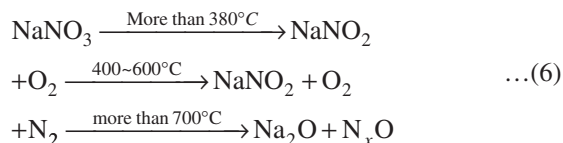
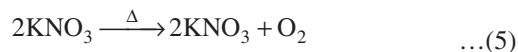
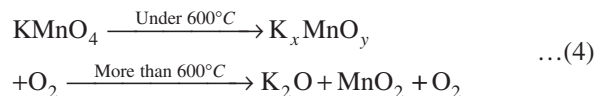


Fig. 6: Effect of oxidizers on average incinerating rate of briquette.

temperature of KMnO_4 , KNO_3 and NaNO_3 to produce oxygen is respectively 240°C , 334°C and 380°C . In the incineration process, the order of the oxygen released by the three kinds of oxidizers is KMnO_4 , KNO_3 and NaNO_3 . Therefore, in the early stage of incineration, the incinerating rate of the briquette containing KMnO_4 is the highest, and the incinerating rate of the briquette containing NaNO_3 is the minimum. Meanwhile, NaNO_3 can produce oxygen continuously under the condition of heating. In other words, a certain amount of oxygen is produced in different temperature ranges. However, KNO_2 formed by thermal decomposition of KNO_3 is relatively stable (Ravanbod et al. 2016). Maybe the reason is that the incinerating rate of the briquette containing NaNO_3 is greater than containing KNO_3 at 10 minutes. In addition, all the three kinds of oxidizers increased the incinerating rate at the early stage of incineration. The increase of incinerating rate is mostly obvious when KMnO_4 was added into the briquette. Moreover, from Fig. 7, we can see that the content of NO_x in flue gas will increase when the three kinds of oxidizers were added into the briquette in the early stage of incineration. When KMnO_4 was used as oxidizer, the content of NO_x in flue gas is not only less than that of KNO_3 and NaNO_3 , but the content of NO_x in flue gas is close to that without oxidizer. Therefore, we can think that the increase of NO_x content in flue gas is due to the addition of potassium nitrate and sodium nitrate in the briquette. As the content of nitrogen in briquette is less, the addition of

nitrate causes the increase of NO_x concentration in the flue gas. Therefore, KMnO_4 was selected as the oxidizer. The possible reaction can be as below:



It can be seen from Fig. 8 that with the increase of amount of KMnO_4 , the incinerating rate of briquette is gradually increasing. When the content of KMnO_4 in briquette increased from 10% to 12%, the incinerating rate of the briquette increased from 0.47 g/min to 0.58 g/min. When the content of KMnO_4 in briquette exceeded 12%, the incinerating rate of briquette increased slowly, from 0.58 g/min to 0.63 g/min. The effect of KMnO_4 content in the briquette on the sulphation efficiency of CaCO_3 is shown in Fig. 9. With the content of KMnO_4 in the briquette increased from 10% to 18%, the sulphation efficiency of CaCO_3 increased from 52.5% to 80.7%. However, the efficiency of sulphation decreased to 72.3% as the KMnO_4

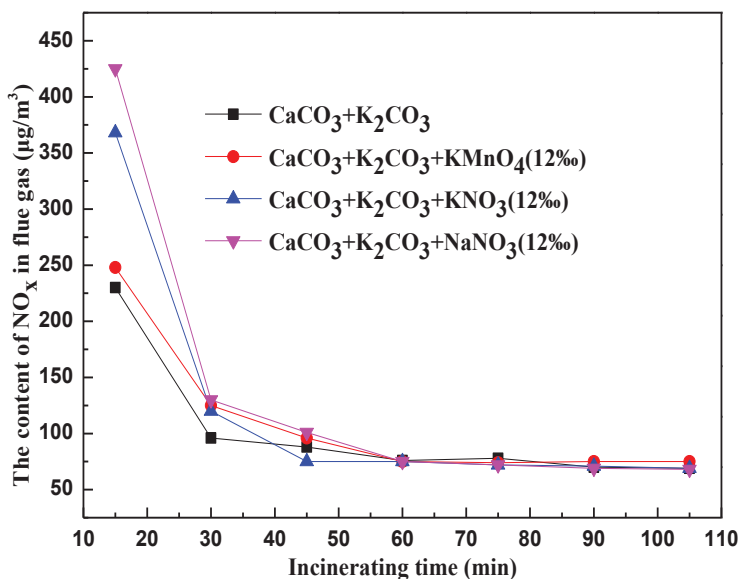


Fig. 7: Effects of oxidizer on NO_x concentration in flue gas.

content of briquette was 16%. Taking the incinerating rate and the sulphation efficiency into account, the amount of KMnO_4 in the briquette was determined as 18%.

Effect of the Type and Amount of Catalysts in Briquette on the Share of Organic Gas in Flue Gas and the Sulphation Efficiency

The results in Table 4 show that Fe_2O_3 and KClO_3 can apparently reduce the release of organic gas during the incineration of briquette. The addition of Fe_2O_3 effectively reduces the share of CO , C_3H_8 and C_4H_{10} in flue gas. However, KClO_3 strongly affects the proportion of CH_4 , C_2H_4 , C_3H_8 and C_4H_{10} in flue gas. In addition, the addition of Fe_2O_3 and KClO_3 can also reduce the content of C_2H_6 and C_3H_6 . It is difficult to define the type of catalysts from

the share of organic gas in flue gas.

It can be seen from Fig. 10 that the catalysts Fe_2O_3 and KClO_3 have a certain influence on the sulphation efficiency of CaCO_3 . With the improvement of the amount of catalyst, the sulphation efficiency of CaCO_3 increases gradually, and then the sulphation efficiency decreases when Fe_2O_3 was added. However, when KClO_3 was used to be catalyst, the trend of sulphation efficiency of CaCO_3 is adverse. When the amount of catalyst is identical, the effect of KClO_3 on sulphation of CaCO_3 is more obvious than that of Fe_2O_3 . When the amount of KClO_3 is 6%, the sulphation efficiency of CaCO_3 can reach 95.8%. In addition, according to the results of Table 5, organic component in flue gas distinctly decreases with the improvement of KClO_3 . Although there is still a small amount of CO in the

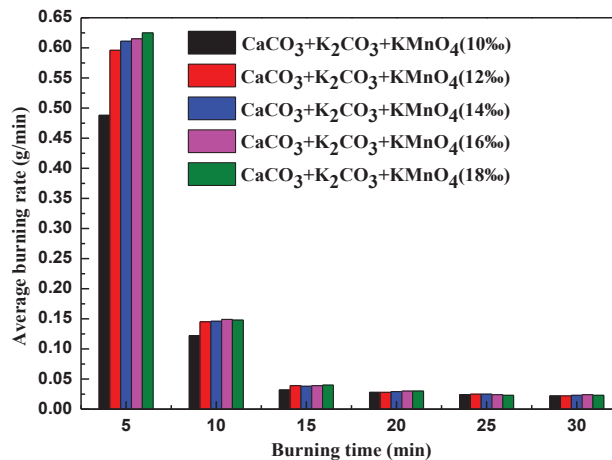


Fig. 8: Effect of the adding amount of KMnO_4 on average incinerating rate of briquette.

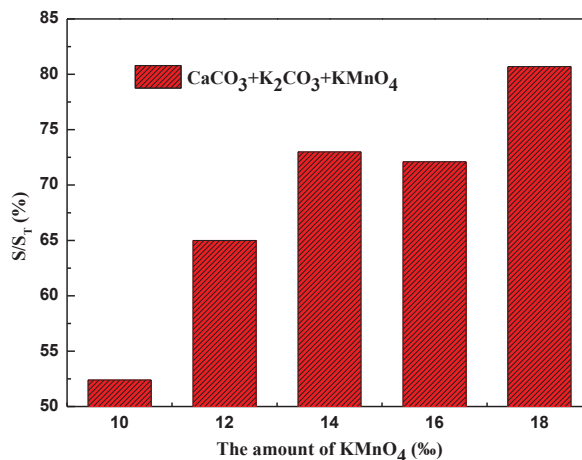


Fig. 9: Effect of the adding amount of KMnO_4 on the sulphation efficiency of CaCO_3 .

Table 3: The composition of incineration gases (%).

	The composition of combustible component of incineration gases (%)						
	CO	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ H ₁₀
No catalyst	1.2827	0.8125	0.1151	0.0986	0.1724	0.0889	0.1026
Fe ₂ O ₃ (6%)	0.0899	0.0020	0.0021	0.0027	0.0030	-	-
KClO ₃ (6%)	0.1012	0.0008	0.0014	0.0026	0.0032	0.0006	-

Table 4: Effect of catalysts in briquette on the share of combustible gas in flue gas.

	The composition of combustible component of incineration gases (%)						
	CO	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ H ₁₀
No catalyst	1.2827	0.8125	0.1151	0.0986	0.1724	0.0889	0.1026
KClO ₃ (6%)	0.1012	0.0008	0.0014	0.0026	0.0032	0.0006	-
KClO ₃ (8%)	0.0413	0.0002	0.0012	0.0011	0.0004	-	-
KClO ₃ (10%)	0.0120	-	0.0006	-	-	-	-
KClO ₃ (12%)	0.0182	-	-	-	-	-	-
KClO ₃ (14%)	0.0174	-	-	-	-	-	-

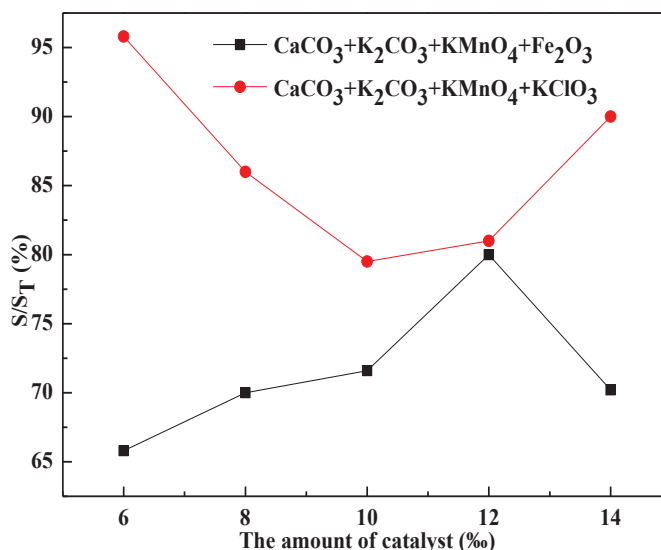


Fig. 10: Effect of varieties of catalysts and adding amount on the sulphation efficiency.

flue gas, the share of organic component in flue gas is little when the adding amount of KClO₃ is 6%. Taking the share of organic component and sulphation efficiency of CaCO₃ into account, KClO₃ is more suitable for the catalyst of the briquette.

The XRD results given in Fig. 11 illustrate that the fixed sulphur was turned into CaSO₄ and K₂SO₄. Calcium in the residue exists in the form of CaSO₄, which indicates that the added CaCO₃ is fully utilized. The Fig. 12 is the SEM of sample calcined from the mixture of CaCO₃ and KClO₃ at

900°C. The surface of sample is coarse compared with the surface of the upper of Fig. 5, which indicates that the added KClO₃ can promote surface area and enrich surface pore of CaO that was produced through decomposition of CaCO₃. During the process of calcining, KClO₃ was decomposed into KCl and oxygen by thermal decomposition. The alkali metal compound KCl not only presents the molten state at high temperature, but can form the liquid phase eutectic with the low melting point with CaO produced through decomposition of CaCO₃, which increases the migration and

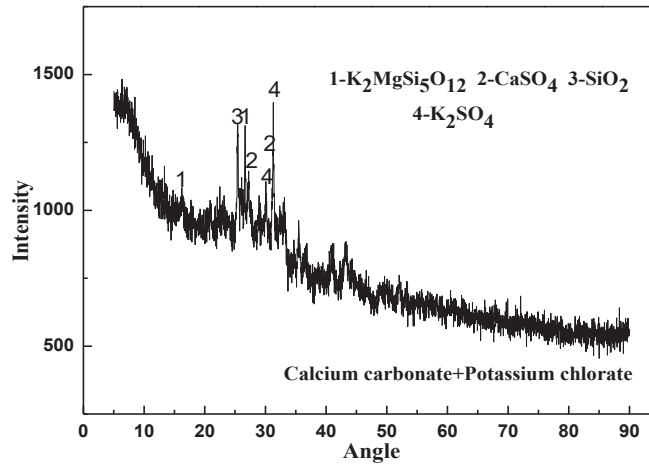


Fig. 11: The XRD spectrum of the incinerating residue (The additive of coal sample are CaCO_3 and KClO_3).

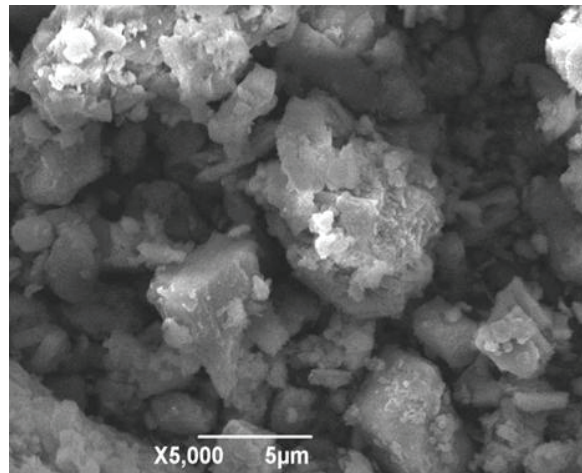


Fig. 12: SEM images of CaO produced through decomposition of CaCO_3 and KClO_3 .

diffusion capacity of the alkali metal ions. The migration and diffusion of the alkali metal ions make the pore size of CaO larger, and the larger pore size is beneficial to the reaction of SO_2 and CaO (Zhou et al. 1997).

CONCLUSIONS

During the incineration of briquette, the addition of CaCO_3 is more effective than CaO for fixing sulphur into residue, K_2CO_3 is more useful than NaCl and Na_2CO_3 to improve the sulphation efficiency of CaCO_3 , the order of improving sulphation efficiency of CaCO_3 by sulphation-supporting additives is K_2CO_3 , Na_2CO_3 , NaCl . The sulphur in briquette is fixed in the form of CaSO_4 , K_2SO_4 and BaSO_4 . The SEM

and XRD results indicate that the sulphation-supporting additive K_2CO_3 not only can promote surface area and enrich surface pore of CaO produced through decomposition of CaCO_3 , but can improve the utilization of CaCO_3 . The addition of KMnO_4 is more effective than KNO_3 and NaNO_3 for improving the incinerating rate of briquette. However, KMnO_4 slightly promotes the release of sulphur.

Fe_2O_3 and KClO_3 can apparently promote the burning of briquette and reduce the release of organic gas during the incineration of briquette, but the effect of KClO_3 on sulphation of CaCO_3 is more obvious than that of Fe_2O_3 .

The adding amount of CaCO_3 , K_2CO_3 , KMnO_4 and KClO_3 is 16%, 12%, 18%, 6% respectively in the process of briquette preparation. The sulphation efficiency reaches

the maximum of 95.3%, the maximum of incinerating rate of briquette is improved from 0.38 to 0.6 g/min, the rate of improvement is more than 50%. And there is still an extremely small amount of organic gas in flue gas.

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