Effects of Additives Blended in Corn Straw to Control Agglomeration and Slagging in Combustion

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Four additives including two specimens of kaolin clay, limestone, and a byproduct of a sugar mill (BSM) (mainly CaCO₃) were utilized to increase ash fusion temperature (AFT) of corn straw. The results showed that the ash softening temperature (ST) was increased by 250 to 380 °C and agglomeration or slagging could be avoided during combustion with each additive. Meanwhile, the slagging/fouling tendency of all ash samples fell within the "low" range according to alkali index. Lime was shown to have the best effect, which indicated that calcium oxide was the best compound to increase the AFT of corn straw densification (CSDF). Both kaolin specimens made the fusion range very narrow. BSM had the least effect on ST among the four. All the additives diluted the concentration of chlorine by more than 50%. No agglomeration or slagging phenomenon appeared in real boilers burning CSDF with lime blended as additive.

Keywords: Additives; Corn straw; Agglomeration; Ash fusion temperature

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INTRODUCTION

As a renewable and CO_2 -neutral energy source, biomass has a high potential to mitigate greenhouse gas emission for heat and electricity generation in the future. Biomass densification fuel is made of the straw compressed by specialized equipment at a certain temperature and pressure into rod, granular, or other forms. It solves the main problems of biomass including transportation, storage, and fire control. Thus, it will have an extensive development prospect and present tremendous economic benefits (Berlanga *et al.* 2012; Chen *et al.* 2015).

Grate furnaces and circulating fluidized beds (CFBs) are widely used in the combustion of biomass. However, the maximum temperature in the grate furnace is between 1100 °C and 1300 °C, so slagging is easily formed on the grate due to the low ash fusion temperature (AFT) of biomass fuel. When the temperature in CFB is higher than 900 °C, the formation of bed agglomeration is also probable, which may lead to unscheduled plant shutdown. The fouling on heat exchangers decreases the thermal efficiency of the power plant and hinders their cleaning considerably. It is generally believed that the agglomeration, slagging, and fouling can be attributed to the concentrations of K, Cl, S, Al, and Si in ash (Fryda *et al.* 2010; Xiao *et al.* 2011; Michel *et al.* 2012; Teixeira *et al.* 2012; Zhu *et al.* 2017; Yao *et al.* 2017; Liu *et al.* 2018; Batir *et al.* 2019).

Until now, two possible bed agglomeration formation mechanisms have been proposed, namely melt-induced agglomeration and coating-induced agglomeration (Brus

et al. 2005; Öhman et al. 2005; Chirone et al. 2006). Co-combustion and the addition of chemical materials (additives) (Davidsson et al. 2007, 2008) or alternative bed materials (Llorente et al. 2008) for bubbling fluidized bed or CFB combustion is a cheap and practical means to reduce the sintering caused by alkaline compounds. The purpose is to increase the AFT of the biomass ash. According to reactive components, additives can be divided into four groups, including aluminium silicates-based additives (such as kaolin, halloysite, cat litter, emathlite, clay, illite, detergent zeolites, sewage sludge, paper sludge, peat ash, and coal fly ash), sulphur-based additives (such as ammonia sulfate, aluminum sulfate, and iron sulfate,), calcium-based additives (such as limestone, lime, marble sludge), and phosphorus rich additives (such as ammonia phosphate and phosphoric acid) (Wang et al. 2011; Wu et al. 2011; Grimm et al. 2011; Hupa 2012; Wang et al. 2012b). Kaolin clay, which is an aluminium silicate, is a typical additive. It can react with KCl (g) and generate kalsilite (KAlSiO₄) and leucite (KAlSi₂O₆), which have high melting temperatures of about 1600 °C and 1500 °C, respectively (Steenari et al. 1998). Lime is more efficient in abating ash sintering and slagging because the reactions are more likely to occur at high temperatures and with prolonged time (Wang et al. 2012a).

The aim of this work was to solve ash-related problems by blending additives (kaolin, lime, and BSM) in corn straw (CS) to increase the AFT. The CS was first cut by a lab-scale biomass chopper, and the four additives were blended evenly in the CS powder by manual work respectively. After the samples were burnt in a muffle furnace, part of the ashes were collected for further measurement of ash melting points with an ash melting test apparatus. Ashes from fuel combustion were analyzed by XRF and XRD to obtain chemical composition and main mineralogical compounds in each of them. Slagging tendency can be replaced with slagging tendency evaluation.

EXPERIMENTAL

Biomass and Additives

Corn stalk (CS), as the most abundant agro-residue and main fuel in many bioplants in the Northeast of China, was selected for this work. The CS used in the experiment was obtained from the countryside of Heilongjiang province. It was milled to the size of range 0 to 1 mm by a biomass chopper and dried at 105 °C in a drying oven for 2 h to a final total moisture of less than 5%.

Four additives were chosen in this paper, namely two specimens of kaolin clay, lime, and a byproduct of production sugar mill (BSM). The former three were bought from chemical product shops and directly utilized in this work, while BSM was obtained from a sugar mill. The main composition of BSM is calcium carbonate.

Additives were ground to pass a 250 mesh sieve and then mixed with the CS evenly. The proportions of additives added in the CS were 1, 2, 3, and 4% by weight. When only one additive was added in one sample, this will be referred to as "used singly", whereas when any two kinds of additives were both used in combination, this will be called "used together".

Both raw biomass samples and those with additives were combusted at 550 $^{\circ}$ C in a conventional muffle furnace for 2 h with its door half opened to realize complete combustion. The obtained ashes were ground to the size of less than 0.25 mm and tested in an ash melting point test apparatus to get the AFT.

Ultimate Analysis										
Element	С	Н	0	Ν	S	CI	K	Na	Si	other
Weight (wt%)	33.76	4.15	36.37	0.38	0.95	1.57	1.92	17.64	1.78	1.48
			Pr	oximate A	nalysis					
Component		Ash		Volatile			Fixed carbon			
Weight (wt%)		24.39		50.00				25.6	61	

Table 1. Ultimate and Proximate Analyses (Dry Basis) of RBL

Experimental Instruments

The muffle furnace (Youxin Technology Development Co., Ltd., Changsha), in which the biomass samples were combusted, operated at a temperature range from room temperature to 1200 $^{\circ}$ C.

The ash melting point test apparatus (Youxin Technology Development Co., Ltd., Changsha) contained a computer, data acquisition card, video capture unit, high temperature furnace, thermocouple, and control system, *etc.* Its heating rate was 20 to 30 °C /min before 900 °C, and 5 °C \pm 1 °C /min after 900 °C. The temperature ranged from room temperature to 1500 °C with a power of 8 KW.

Analytical Methods

Analyses of calorific value, volatile matter, ash, moisture, bulk density, distribution of particle size, chlorine, and ultimate analysis (C, H, N, S) were carried out for biomass samples according to internal procedures, which are mainly based on ASTM norms for wood, refuse derived fuels, and coal.

X-ray diffraction (XRD) was utilized to determine the inorganic compounds in the ashes using a D/Max 2500 diffractometer (Rigaku, Osaka, Japan). Identification of compounds was made using the diffractometer software with the JCPDS database as a source of reference data. Peak identification was performed through a comparison with standards coming from Jade[®] 5.0 software. An X-ray fluorescence spectrometer (XRF, AXIOS-PW4400, Netherlands), which is widely used to measure the elemental composition of material *via* a spectroscopic method, was utilized to carry out the analyses for the different samples.

The ash fusibility was based on the changes in shape detected during the heating process of a cylindrical pellet of ash from room temperature to 1500 °C in a weak reducing atmosphere. The characteristic temperatures measured were deformation temperature (DT), softening temperature (ST), hemisphere temperature (HT), and fluid temperature (FT), following the DIN forms 51730-1998 and DIN 51730-1994, the norm DIN 51730-1994 was only followed to determine the FT, which was established when the height of the ash pellet is at 1/3 of the initial height.

Slagging/fouling Index

Several empirical indices have been proposed to investigate the behaviors and deposition tendencies of ash for biomass. We selected the alkali index (AI) to predict the ash deposition tendencies, which take the form as follows:

$$AI = \frac{kg(K_2O + Na_2O)}{GJ}$$
(1)

The alkali index represents the quantity of alkali oxides in fuel ash per unit fuel energy. When the value of the alkali index falls within the range of less than 0.17 kg/GJ,

slagging or fouling in the fluidized bed is low, and if the value falls within the range of 0.17 to 0.34 kg/GJ, slagging or fouling is probable, whereas slagging or fouling is virtually certain to occur when these values are greater than 0.34 (Miles *et al.* 1996).

RESULTS AND DISCUSSION

Biomass Characterization

The physical and chemical characterizations of the CS are summarized in Table 1. The CS sample had a low content of ash (4.2 wt%) and sulphur (0.12 wt%), while the volatile content was very high (70.3 wt%), which strongly influenced the characteristics of the biomass combustion. When burned in the boiler, the CS fuel ignited easily with less ash remaining, as well as very little sulfur dioxide emissions. The air-dried CS had a relatively high low-heating-value (LHV) (15080 kJ/kg), which was comparable to that of middle rank coals.

Table 1. Physical and Chemical Characterizations of CS	Samples
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Proximate Analysis (air dry,	Moisture	Volatiles	Fixed carbon	Ash	LHV (kJ/kg)
wt%)	4.68	70.29	20.83	4.20	15080.64
Ultimate Analysis (daf, wt%)	Carbon	Hydrogen	Nitrogen	Sulphur	Oxygen
Olimate Analysis (dal, wt%)	42.17	5.45	0.74	0.12	33.20

Table 2 presents the characteristic of the CS ash. It can be seen that the CS ash had a low fusion temperature (DT, ST, HT, and FT were 929 °C, 959 °C, 1005 °C, and 1160 °C, respectively), which was much lower than the maximum temperature in the grate furnace (about 1100 to 1300 °C) and could not meet the need for CFB combustion as well. Therefore the bed agglomeration was easily formed in CFB, and slagging or fouling on the heating surface in grate furnace were unavoidable when burning raw CS (Brus *et al.* 2005). The ash analysis results from XRF showed that the CS ash had large amounts of K and Cl (20.24 and 6.83, respectively), which chiefly caused the low fusion point of the ash as stated previously. The high concentration of Cl contributed to the corrosion on grates and heating surfaces as well. The AI of the CS ash sample was 0.26, falling within the range of "probable slagging/fouling".

Table 2. Characterizations	s of the Raw CS A	۱sh
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Ash Analysis ^a	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃	CI	AI
(dry ash, wt %)	0.90	15.96	1.73	26.79	2.75	20.24	21.66	0.18	1.31	6.83	0.26
Fusibility	DT		ST		HT			FT			
Temperatures (°C)	92	29	959		1005			1160			
Slagging/Fouling Tendency					Pr	obable					

^aThe ultimate analysis of ash is given in the normalized form from XRF

The raw CS was burned at 1000 °C in the muffle furnace. The results showed that sintering had formed to some extent.

Additives Characterization

The results of the inorganic chemical components of additives detected by XRF analysis are listed in Table 3. The ingredients under 0.1% by weight in all the four additives were omitted. The crystalline mineral species of additives had been identified by XRD analysis.

Ingredients	CaO	AI_2O_3	SiO ₂	MgO	Fe ₂ O ₃	P_2O_5	SO ₃	K ₂ O	TiO ₂
Kaolin-1	0.21	38.14	44.83	5.27	0.34	0.02	-	-	0.50
Kaolin-2	0.28	21.74	61.26	0.41	2.68	0.06	0.04	1.71	0.97
Lime	90.00	0.34	0.81	0.42	0.20	0.04	0.16	-	-
BSM	66.77	0.42	0.77	0.80	0.50	0.95	0.41	-	0.06

Table 3. Ultimate	Analysis	of Additives	(wt%))
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Note: The ultimate analysis of additives is given in the normalized form from XRF.

It can be seen from Table 3 that 90% of the lime was calcium, which was present as calcium oxide (CaO) with a melting point of 2670 °C. Other compounds were relatively low.

The main chemical components of kaolin-1 were Al_2O_3 (38.16 wt%) and SiO_2 (44.83 wt%), which existed in the form of mullite ($Al_6Si_2O_{13}$), simimanite (Al_2SiO_5) and kalsilite (KAlSiO_4) by XRD analysis. The main chemical components of kaolin-2 were Al_2O_3 (21.74 wt%) and SiO_2 (61.26 wt%). The compounds in the kaolin-2 were the most complex while the silicon existed in the form of quartz (SiO_2) with a melting point of 2230 °C. When comparing two kaolin specimens, the most difference was the ratio of silicon to aluminum.

The absolute proportion of calcium by XRF analysis of BSM was 66.8%, while its relative value was more than 94%, as the elements detected by XRF were converted into the form of oxides and the element carbon had been excluded in the analysis. The existing form of calcium was mainly CaCO₃, as detected by the XRD analysis.

Ash Fusion Temperature Test

The ash samples of the CS with different additives were detected in the ash melting point test apparatus, and the results are shown in Table 4. The comparison of the AFT of the different samples is demonstrated in Fig. 1. It can be seen that all the ash melting temperatures of CS with additives increased remarkably compared to the raw CS ash.

Figure 1a shows the AFT comparison of samples with 4% additives singly. The lime had the best effect on increasing the AFT. No obvious deformation phenomenon was observed when the temperature reached 1340 °C. The ST was increased by more than 440 °C compared to the raw CS ash, and the FT reached 1455 °C. For kaolin-1, kaolin-2, and BSM, the ST was enhanced by 285 °C, 262 °C, and 257 °C, respectively (as seen in Table 4). As the ST is a very important and indicative temperature of the ash relating to ash fusibility and the agglomeration or sintering in furnace, these increases demonstrate the remarkable effect of the additives. The first specimen of kaolin had a better effect than kaolin-2 because all the four characteristic temperatures (DT, ST, HT, and FT) of the sample with kaolin-1 were higher than that with kaolin-2. The BSM did not have a strong effect on ST as others but had a greater effect on HT and FT compared to the kaolin specimen, which resulted in the widest fusion range (107 °C between ST and FT).

Table 4. The AFT	of Fuel with Additives
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Samples	DT/°C	ST/°C	HT/°C	FT/°C	Fusion Range Between ST and FT
Raw CS	929	959	1005	1160	201
CS + 4% kaolin-1	1230	1244	1248	1260	16
CS + 4% lime	1340	1400	1421	1455	55
CS + 4% kaolin-2	1211	1221	1228	1245	24
CS + 4% BSM	1212	1216	1276	1323	107
CS + 2% lime + 2% kaolin-2	1202	1212	1248	1257	45
CS + 3% lime + 1% kaolin-2	1263	1267	1273	1279	12
CS + 2% lime +2% kaolin-1	1260	1265	1275	1290	25
CS + 3% lime + 1% kaolin-1	1300	1310	1324	1329	19
CS + 2% BSM +2% kaolin-2	1200	1211	1218	1225	14
CS + 2% BSM + 2% kaolin-1	1186	1209	1235	1245	36
CS + 2% lime	1216	1222	1292	1295	73
CS + 2% kaolin-2	1176	1218	1234	1238	20
CS + 2% kaolin-1	1217	1259	1262	1267	8
CS + 2% BSM	1119	1162	1179	1190	28

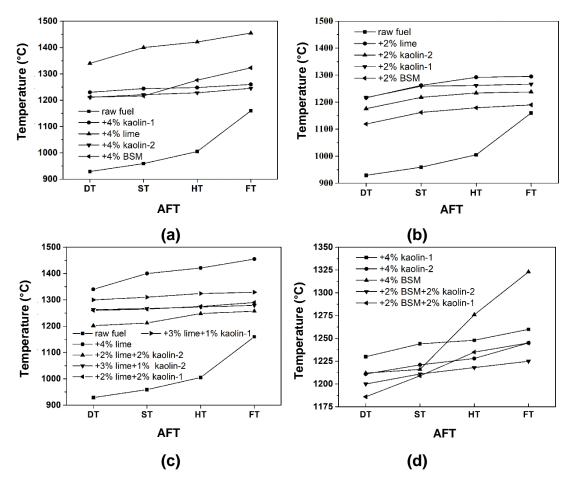


Fig. 1. The comparison of the AFT of different samples

As can be seen from Table 4 and Fig. 1b, when additives were added singly in the raw CS by 2%, the lime was still the best among them. The kaolin-1 was still better than kaolin-2, as all the four characteristic temperatures of the sample with kaolin-1 were still

higher than that with kaolin-2. But the AFT of the sample with 2% lime was much lower than the one with 4% lime. Thus, it can be concluded that it is more effective to blend the lime in the fuel by 4% to achieve a higher AFT. The effect of the sample with 2% kaolin-1 was a little better than that with 4% kaolin-1, though the reason needs further study. The effect of the sample with 2% kaolin-2 was a little worse than that with 4% kaolin-2, and the effect of the sample with 2% BSM was much worse than that with 4% BSM.

It can be seen from Fig. 1c and 1d that the CS with 3% lime plus 1% kaolin-1 had a higher AFT than that with 2% lime plus 2% kaolin-1. The CS with 3% lime plus 1% kaolin-2 also had a higher AFT than that with 2% lime plus 2% kaolin-2, but all were inferior to the CS with 4% lime. This indicated that the AFT increased with the increasing proportion of the lime in the samples. All the AFT of CS with 2% BSM plus 2% kaolin-1 were lower than that with 4% kaolin-1 or that with 4% BSM. Therefore, it can be said that the effect with 2% BSM plus 2% kaolin-1 is worse than that with 4% kaolin-1 or that with 4% BSM. Also, the same conclusion can be obtained that the CS with 2% BSM plus 2% kaolin-2 was not as good as that with 4% BSM nor as that with 4% kaolin-2.

Overall, all additives had good effect on increasing AFT, whether they were used singly or together. Of the four additives, the lime had the best effect on increasing the AFT, which indicated that calcium oxide was the best compound of additive for the CSDF. Kaolin-1 was superior to kaolin-2 while both of them made the fusion range remarkably narrow and a lower ratio of silicon to aluminum in the additive resulted in a higher AFT. 4% BSM had the worst effect on the ST among the four additives but led to the widest fusion temperatures range.

Ash Analyses

Results of analysis by XRF for the ash samples of the fuel with additives and the slagging/fouling indices are listed in Table 5.

It is obvious that after adding the lime or BSM in the fuel, the content of CaO increased remarkably, with the decrease of MgO, SiO₂, and K₂O. This is probably due to the fact that the calcium contents in these two additives are quite high, causing dilution of other elements in the ash. The AI decreased to 0.1 and 0.14, respectively. By adding the kaolin-1 or kaolin-2 in the fuel, the concentrations of Al₂O₃ and SiO₂ rose sharply while MgO, K₂O, and CaO dropped, which was associated with the presence of aluminum and silicon in these two additives. The AI was 0.15 and 0.17, respectively. Regarding the four additives, AIs were no more than 0.17 therefore the ashes fell within the range of low slagging/fouling.

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Ash samples	Na₂O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO2	Fe ₂ O ₃	AI	Slagging/fouling tendency
CS + 4% kaolin-1 (%)	1.59	10.87	19.50	39.85	1.28	10.56	10.60	0.31	0.82	0.15	Low
CS + 4% lime (%)	0.23	8.23	0.85	15.45	1.23	8.11	61.74	0.15	0.66	0.10	Low
CS + 4% kaolin-2 (%)	1.48	9.21	7.74	46.36	1.64	12.44	13.93	0.50	2.06	0.17	Low
CS + 4% BSM (%) 0.50 10.90 1.29 17.71 1.57 11.43 51.43 0.14 0.83 0.14 Low											
Note: The ultimate analysis of ash is given in the normalized form from XRF.											

Table 5. Ash Analys	es of Fuel with Additives
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In addition, Cl concentrations in the ash samples are shown in Table 6. A comparison with the raw CS revealed that the content of Cl in the samples with 4% kaolin-1, lime, kaolin-2, and BSM decreased by 62%, 65%, 58%, and 54%, respectively. The decrease was mainly due to the dilution by the additive and partly to the reaction between

the additive and the raw fuel ash. As Cl plays an important role as a facilitator in alkali deposition and a contributor to corrosion in combustion (Miles *et al.* 1996), this significant dilution of Cl concentration will mitigate these problems greatly.

Ash samples	CI (%)	Diluted proportion (%)
Raw CS	6.83	0.00
CS + 4% kaolin-1	2.62	62
CS + 4% lime	2.36	65
CS + 4% kaolin-2	2.85	58
CS + 4% BSM	3.15	54

Table 6. CI Concentrations of the Ash Samples

Discussions Toward Industrial Processes

The samples of the CS with each additive by 4% singly were also burnt at 1000 °C in the muffle furnace. As a result, all the ashes were very loose and no agglomeration or sintering appeared. In practical application, the CSDF with 4% lime had burned in a 25 MW water cooled vibrating grate boiler imported from Danmark, no slagging appeared on the grate surface.

The above findings are valuable for the future application of CSDF with additives for power generation. The CSDF with 4% additives such as kaolin-1, lime, kaolin-2 and BSM has remarkably improved the fuel characterization and the ash fusibility. Thus it is suitable for the application in grate furnaces, CFB and other biomass boilers.

CONCLUSIONS

- The four additives had a remarkable effect on increasing the ash fusion temperature (AFT) of corn straw (CS). Lime had the best enhanced effect which meant that calcium oxide was the best compound to increase the AFT of CS. After blended with lime, ST increased by more than 440 °C compared to the raw CS ash and the FT reached 1455 °C. And the effect ranking of four additives was lime>kaolin-1> kaolin-2>BSM. Although the effect of 4% BSM for increasing the ST is not as good as with 4% kaolin-1 and with 4% kaolin-2, it had a better effect for increasing the HT and FT, which resulted in the widest fusion range between ST and FT.
- 2. Based on the ash analyses, it can be seen that after blending the four additives, AIs were no more than 0.17 which implied that the ashes had low a possibility of slagging or fouling. In addition, Cl concentrations in the ashes decreased by more than 50%, which could mitigate corrosion problem.

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