

A Study on the Migration Behavior of K, Na, and Cl during Biomass Gasification

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The migration behavior of the alkali metals and chlorine were studied during rice straw and corn straw gasification in a fixed bed reactor at various temperatures using thermodynamic equilibrium calculations, X-ray diffraction (XRD), and scanning electron microscopy/energy dispersive spectrometry (SEM-EDS). The results showed that K and Na were released mostly in chloride form. The release of potassium, sodium, and chlorine increased upon the increase in temperature from 600 to 1000 °C. The maximum amounts of potassium, sodium, and chlorine that were released from rice straw were 38.9%, 18.7%, and 34.9%, respectively. The maximum amounts of potassium and chlorine that were released from corn straw were 24% and 43.6%, respectively, which occurred at 1000 °C. The maximum amount of sodium released from corn straw was 77.6%, at 700 °C, and the amount of sodium released was greater than that of potassium. Most of the potassium and sodium was converted into insoluble carbonate, sulfate, silicate, and aluminosilicate compounds in the gasification ash.

Key words: Rice straw; Corn straw; Gasification; Alkali metals; Chlorine

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INTRODUCTION

Environmental pollution and energy crises arising because of over-exploitation of fossil fuels have intensified in recent decades (Erlach *et al.* 2012; Johansson 2013). Biomass resources are favored because they are clean, renewable, contain low levels of nitrogen, and can effectively address the pollution and global warming problems presented by traditional energy sources (Ma *et al.* 2008; Khan *et al.* 2009; Qin *et al.* 2010; Kirtay 2011). Biomass gasification technology is an advanced means of biomass energy conversion; however, the high alkali metals content in straw wastes, such as corn straw and rice straw, can accumulate in the ash on gasifier heating surfaces, from slag-bounding in a furnace, and from agglomeration of bed material and similar technologies (Liu *et al.* 2010; Niu *et al.* 2010; Nordgren *et al.* 2013) because of the sticky deposition layer of sodium and potassium (Doshi *et al.* 2009). Therefore, it is essential to study the migration and conversion of alkali metals during biomass gasification.

The mechanism of the release of potassium and sodium from biomass conversion has been studied previously under different conditions. For instance, certain studies have examined the release of alkali and alkali earth metals (AAEMs) from corn straw, rice husk, and cotton stalk using steam gasification and have shown that approximately 53% to 76% of alkali metals (K and Na) and 27% to 40% of alkali earth metals (Ca and Mg) can be released during pyrolysis, while 12% to 34% of alkali metals (K and Na) and 12% to 16% of alkali earth metals (Ca and Mg) can be released during gasification (Jiang *et al.* 2012).

Certain scholars have generated sawdust gasification products using steam gasification to effect multi-stage condensation, showing that the release of AAEMs was considerably affected by the water-soluble tar composition and that the products were mostly released as small-molecule particles at high temperatures (Hirohata *et al.* 2008). Researchers have studied the release of potassium with a high-chlorine biomass by emphasizing the effects of secondary recapture and reactions using a pilot-scale fuel bed and a lab-scale setup (Johansen *et al.* 2013). In addition, chlorine in biomass interacts with alkali metals, and it may react with alkali metal silicates during gasification to promote alkali metal volatilization. However, studies on the conversion features of alkali metals with chlorine in biomass gasification are inadequate, and in-depth studies on the interactive response mechanism of alkali metal with chlorine are necessary.

In this paper, corn straw and rice straw gasification were examined using a fixed bed reactor, and the migration of alkali metals with chlorine was investigated using chemical thermodynamic equilibrium analysis, X-ray diffraction (XRD), and scanning electron microscope/energy dispersive spectrometry (SEM-EDS). The purpose was to provide a theoretical basis for biomass gasification applications.

EXPERIMENTAL

Materials

Rice straw and corn straw were collected from the suburb of Shenyang in the Liaoning province of China, cut using a straw chopper, and then smashed using a grinder. Particles of sizes 0.15 to 0.3 mm were selected for the experiments and the fuel constituents are shown in Table 1.

Table 1. Fuel Constituents (wt.%)

	C _{ad}	H _{ad}	O _{ad}	N _{ad}	S _{ad}	Cl	K
Rice straw	39.77	5.53	46.16	0.82	0.24	0.44	0.81
Corn straw	41.44	5.31	48.23	0.84	0.14	0.72	0.77
	Na	Ca	Mg	Al	Si	Fe	Ti
Rice straw	0.32	0.452	0.018	0.352	4.998	0.0349	0.005
Corn straw	0.03	0.24	0.37	0.13	1.63	0.14	0.01

ad: air dried basis

Apparatus and Procedure

A schematic diagram of the experimental setup is shown in Fig. 1. A gas supply system, a fixed bed reactor system, a gas condensate system, and a gas collection system were used. The quartz tube length was 590 mm with an outer diameter of 60 mm; the outlet tube diameter was 10 mm, which was measured from the bottom at 410 mm; the thickness was 2 mm, and a top cover was used for a matte mouth, which was embedded into the outer diameter of the intake pipe at 10 mm with a 10 mm-diameter hole.

As a protecting gas, 0.2 m³/h of nitrogen was continuously bubbled into the reactor before it reached the setting temperature. Next, nitrogen flow was closed and 0.8 L/min of air was bubbled into the reactor, and 2 g of the sample was rapidly placed into the hot zone and left there for 30 min. The water-soluble ions were absorbed by deionized water in a gas-washing bottle. The water-soluble ions on the quartz wall and in the ash were washed with deionized water and collected after the gasification completed.

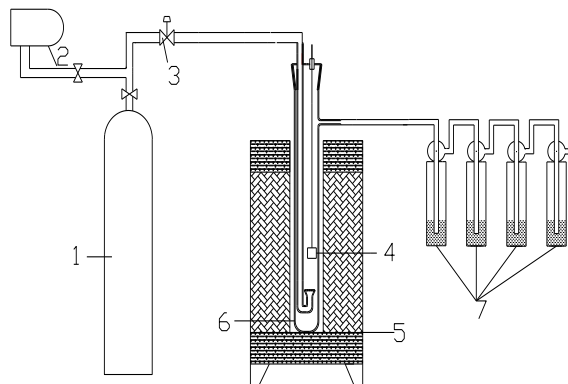


Fig. 1. Sketch of the gasification reactor setup: 1, nitrogen cylinder; 2, air compressor; 3, numerical flow meter; 4, basket; 5, fixed bed; 6, quartz tube reactor; and 7, gas washing bottle

The potassium, sodium, and chlorine in the absorption liquid were measured using a flame photometer (6400A, Cai-Hong, China) and a chloride ion tester (ET6800, Lovibond, Germany). The bottom ash micromorphology was observed using SEM-EDS (S-3400N, Hitachi, Japan), and the crystalline phase was analyzed using XRD (PW3040/60, Philips, Holland).

Chemical Thermodynamic Equilibrium Calculations

The migration and conversion of potassium, sodium, and chlorine in the gas/solid phase at 1.1×10^5 Pa, 500 to 1100 °C, and in an excess air ratio of 0.4 were calculated using HSC chemistry 5.0 (Outotec, Finland). The input parameters (mol) for the simulation are shown in Table 2. The mass fraction of each element in Table 1 was multiplied times 1 kg of material mass, and then the results were divided by the relative atomic mass of each element, giving the data in Table 2.

Table 2. Moles of Each Element in the Fuel

	C	H	O	N	S	Cl	K
Rice straw	33.14	55.3	54.89	98.54	0.08	0.12	0.21
Corn straw	34.53	53.1	56.32	99.06	0.04	0.20	0.20
	Na	Ca	Mg	Al	Si	Fe	Ti
Rice straw	0.16	0.11	0.007	0.13	1.56	0.006	0.009
Corn straw	0.01	0.06	0.15	0.05	0.51	0.03	0.002

RESULTS AND DISCUSSION

Chemical Thermodynamic Equilibrium Analysis of Potassium, Sodium, and Chlorine in the Gas/Solid Phase

Migration analysis for potassium, sodium, and chlorine in the gas phase

The equilibrium concentration distributions for potassium, sodium, and chlorine in the gas phase are shown in Figs. 2 and 3, respectively. The gaseous products from the gasification of the two materials were mostly KCl(g) , $\text{K}_2\text{Cl}_2\text{(g)}$, NaCl(g) , and $\text{Na}_2\text{Cl}_2\text{(g)}$, which indicates that the alkali metals were released in the alkali-chloride form (Froment *et al.* 2013). The sum of the released sodium and potassium, in molar quantities, were close

to the amount of released chorine. The main difference was that small quantities of $K(g)$ and $KCN(g)$ were generated from rice straw in the temperature range of 900 to 1100 °C. Rice straw molar ratio is $(n(K)+n(Na))/n(Cl) \approx 3 > 1$, while corn straw molar ratio is $(n(K)+n(Na))/n(Cl) \approx 1$, which are combined in Table 2. These data predicted that the surplus alkali potassium from rice straw would be released as elemental potassium and carbonitride in the gas phase during the high-temperature gasification region. This might be explained by the following reactions (Chen *et al.* 2007).

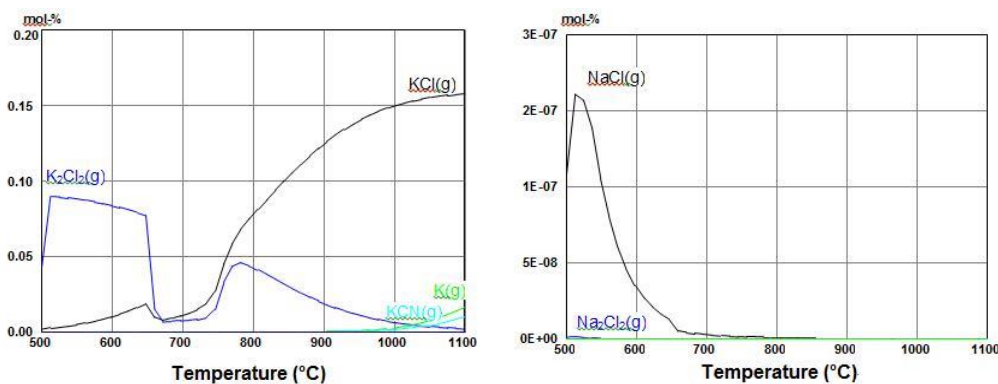
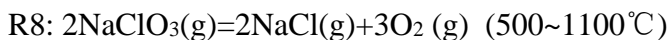
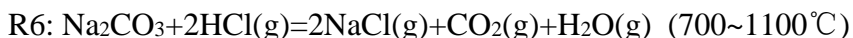
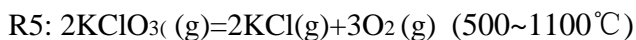
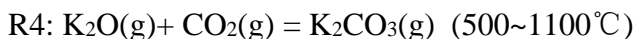
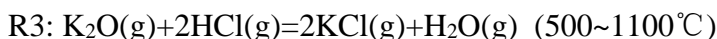
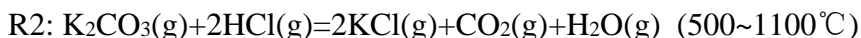
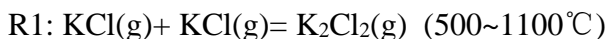


Fig. 2. K, Na, and Cl distributions for the rice straw gas phase equilibrium concentrations

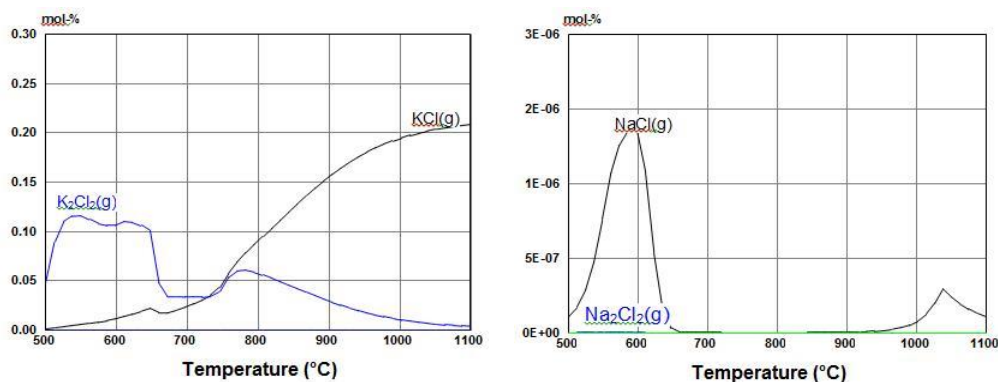


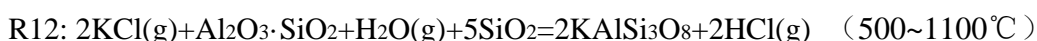
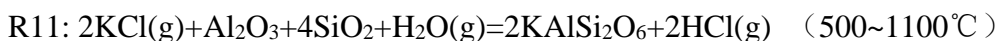
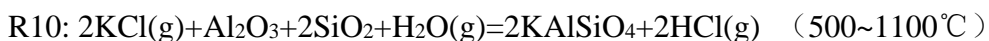
Fig. 3. K, Na, and Cl distributions for the corn straw gas phase equilibrium concentrations

The simulation results indicate that the R1 of potassium generated primarily $K_2Cl_2(g)$ at 500 to 650 °C, while R2 and R5 occurred at 650 to 750 °C, and while $K_2Cl_2(g)$ rapidly decreased. Additionally, R3, R4, and R6 occurred as the temperature increased, but

the equilibrium concentration of KCl(g) increased. The equilibrium concentration of the sodium compounds was clearly lower than that of the potassium during the gasification process, and only small quantities of NaCl(g) were produced in the range of 500 to 700 °C. One reason for this phenomenon was that the potassium content of the biomass was greater than the sodium content; a second reason was that the activity of potassium is stronger than that of sodium. The equilibrium concentration of gaseous alkali metal chloride was lowest at 700 °C, which, in theory, can be effectively reduced if the temperature is maintained at approximately 700 °C. Furthermore, the probability of fouling and slagging decreases in the gasification process.

Migration analysis for potassium, sodium, and chlorine in the solid phase

The equilibrium concentration distributions for the alkali metals in the solid phase are shown in Figs. 4 and 5. The solid product was the same for the two materials, while the equilibrium concentration differed slightly from that for gasification process. The solid potassium products were mainly generated at 650 to 750 °C and were the greatest at 670 °C; the solid product trend presented as an inverted V. This might be explained by the reactions (Wei *et al.* 2005; Fan *et al.* 2011).



Figures 4 and 5 show that R9 through R113 might react in the range of 650 to 750 °C. The $\text{K}_2\text{O} \cdot 2\text{SiO}_2$ and KAlSiO_4 were the main rice straw products, while corn straw also generated $\text{K}_2\text{O} \cdot \text{Fe}_2\text{O}_3$ but did not generate $\text{K}_2\text{O} \cdot 2\text{SiO}_2$ because of its relatively high iron concentration. The sodium solid product occurred at all temperatures and mainly generated NaCN (Han *et al.* 2014). Sodium content of biomass is lower than potassium in an order of magnitude. HSC chemistry has limitations and the simulation results might be have deviation.

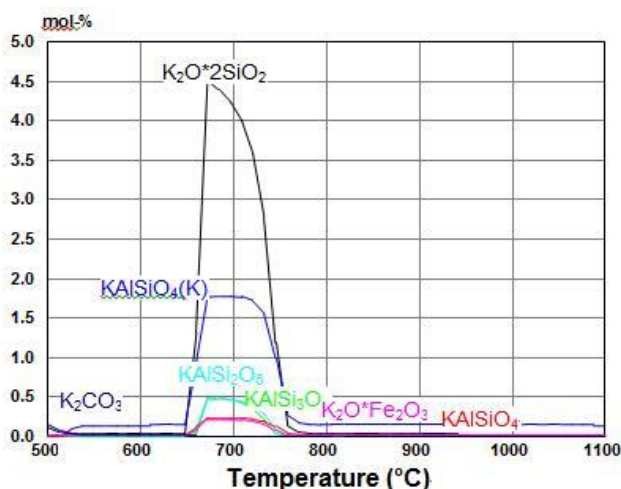


Fig. 4. Potassium distributions for the rice straw solid-phase equilibrium concentrations

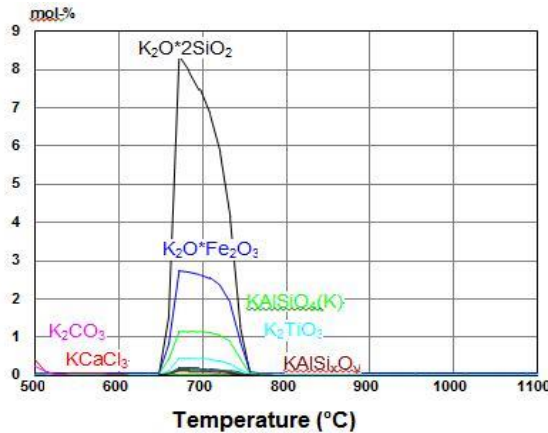


Fig. 5. Potassium distributions for the corn straw solid-phase equilibrium concentrations

The Migration of Alkali Metals during Gasification

Figure 6 shows that the rice straw release rates of K^+ , Na^+ , and Cl^- increased with increasing temperatures, and that the maximum release rates were 38.9%, 18.7%, and 34.9% at 1000 °C, respectively. The reason for these data was that increasing temperatures increased the vaporization of alkali metals and chlorine, which made the rate of water solubility decrease for the solid phases of K^+ , Na^+ , and Cl^- (Yang *et al.* 2014). The variable trends for K^+ and Cl^- were similar to those of rice straw, and the maximum release rates were 24% and 43.6%, respectively, at 1000 °C. The release rate of Na^+ for corn straw was clearly higher than that for rice straw, and the maximum was 77.6%, at 700 °C. The trend remained linear as temperature increased, and Na^+ was released easily, perhaps because of the porous structure of corn straw (Du *et al.* 2013). Thus, the influence of the porous structure of corn straw on the release rate of Na^+ was greater than the influence of temperature.

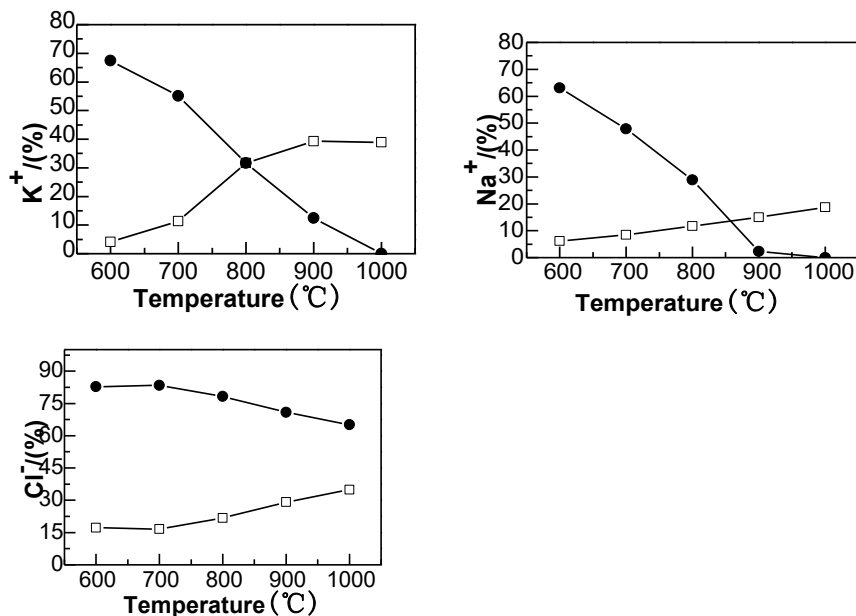


Fig. 6. The K^+ , Na^+ , and Cl^- distributions from rice straw for each phase

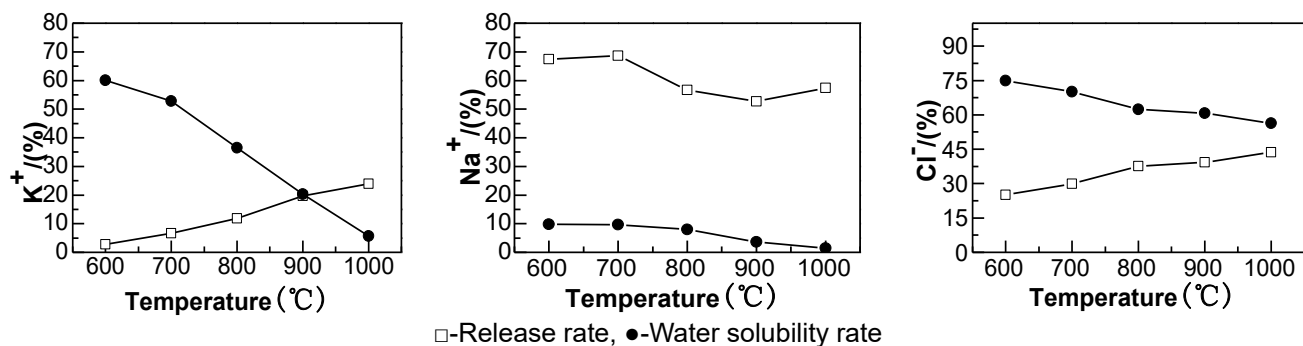


Fig. 7. The K⁺, Na⁺, and Cl⁻ distributions from corn straw for each phase

The water-solubility rate for the solid-phase K⁺, Na⁺, and Cl⁻ from the two materials decreased with the increase in temperature; the trends for K⁺ and Na⁺ were similar, and K⁺ and Na⁺ nearly approach zero at 1000 °C. These results were attributed to the tendency of the steam-gas phase toward generating steam with the increase in temperature and because alkali metal with chlorine becomes water-insoluble in the solid phase. As supported by the results from the chemical thermodynamic equilibrium analysis, at 650 to 750 °C, the alkali metals in the solid phase were found primarily in the form of K₂O·2SiO₂ and KAlSiO₄, which are water-insoluble substances. During the real gasification process, when the chemical thermodynamic equilibrium analysis conditions are in their ideal state, the water-soluble alkali metals with chlorine are converted into water-insoluble substances at higher temperatures.

The molar ratios of Cl⁻ to K⁺, and Na⁺ released during the gasification of rice straw and corn straw are shown in Figs. 8 and 9. The data showed that $n(\text{Cl}^-)/(n(\text{K}^+)+n(\text{Na}^+)) > 1$ for rice straw only at 600 °C, while $n(\text{Cl}^-)/(n(\text{K}^+)+n(\text{Na}^+)) > 1$ for corn straw at all gasification temperatures. As supported by the chemical thermodynamic equilibrium analysis, the data showed that KCl (g) and NaCl (g) were generated from the two materials. However, HCl(g) was generated from rice straw only at low temperatures, while it was generated from corn straw at all gasification temperatures (Porbatzki *et al.* 2011) because chlorine levels are lower in rice straw than in corn straw.

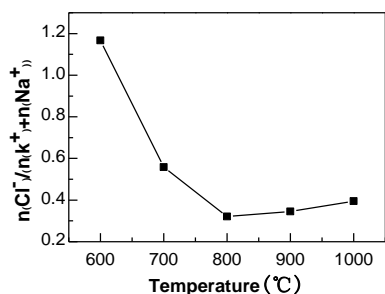


Fig. 8. Molar ratio of Cl⁻, K⁺, and Na⁺ released from rice straw

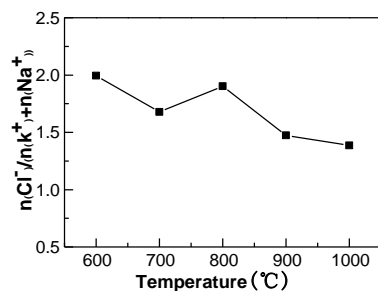
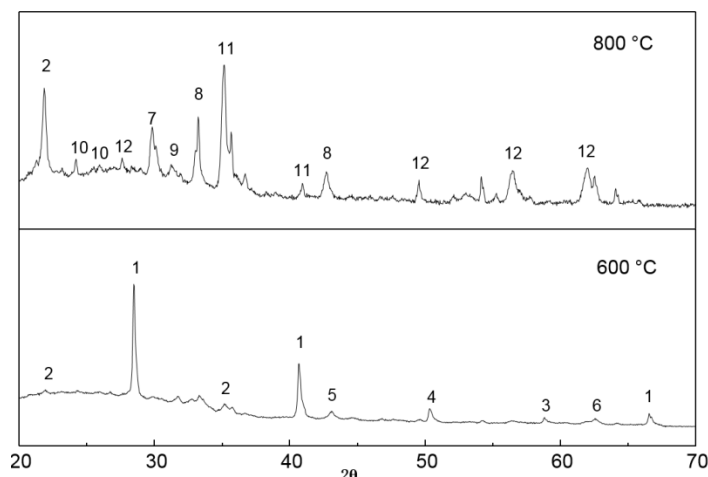


Fig. 9. Molar ratio of Cl⁻, K⁺, and Na⁺ released from corn straw

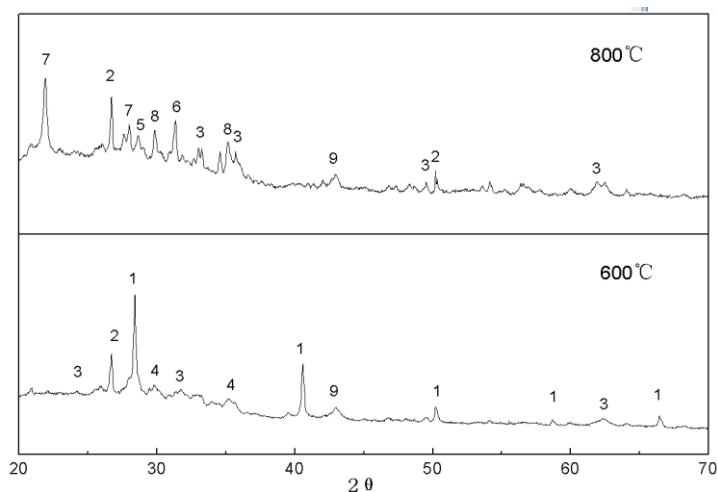
The Microstructure of the Solid Residue from and Distribution Characteristics of Alkali Metals

The X-ray diffractograph for rice straw, given in Fig. 10(a), shows that the alkali metal and chlorine compounds found in the solid products at 600 °C were mostly KCl, KClO₃, KAlSi₃O₈, NaCN, and NaSiO₃·5H₂O, while the compounds found at 800 °C were

mostly NaAlSiO_4 , $\text{Na}(\text{AlSi}_3\text{O}_8)$, K_2CO_3 , KHCO_3 , and K_2SO_4 . The quantity of chlorine compounds decreased, which indicated that the alkali metal chloride was being released gradually as the temperature increased, and the alkali metals were mostly converted into insoluble carbonate, sulfate, silicate, and aluminosilicate compounds. The solid water-soluble substances, such as $\text{NaSiO}_3 \cdot 5\text{H}_2\text{O}$, generated at 600°C were mostly converted into solid water-insoluble substances, such as NaAlSiO_4 and $\text{Na}(\text{AlSi}_3\text{O}_8)$, at higher temperatures (Jordan and Akay 2012). An X-ray diffractograph for corn straw is shown in Fig. 10(b), the compounds identified in the gasification ash were primarily KCl , KAlSiO_4 , and $\text{KNaCO}_3 \cdot 6\text{H}_2\text{O}$ at 600°C , while the alkali metal compounds were found to consist of K_2SO_4 , KSCN , $\text{Na}(\text{AlSi}_3\text{O}_8)$, and $\text{KNaCO}_3 \cdot 6\text{H}_2\text{O}$ at 800°C . Notably, the water-insoluble $\text{NaSiO}_3 \cdot 5\text{H}_2\text{O}$ and $\text{KNaCO}_3 \cdot 6\text{H}_2\text{O}$ from the two materials were not fully converted into solid water-insoluble substances as the temperature increased. This phenomenon was due to the presence of alkali metals, derived from carbon and sulfur, remaining in the two materials; the effect of carbon on corn straw was weaker than that on rice straw.



(a) X-ray diffractograph of rice straw gasification ash: 1-KCl, 2-SiO₂, 3-NaCN, 4-NaSiO₃·5H₂O, 5-KClO₃, 6-KAlSi₃O₈, 7-NaAlSiO₄, 8-K₂CO₃, 9-KHCO₃ 10-K₂SO₄, 11-Na(AlSi₃O₈), and 12-Fe₂O₃



(b) X-ray diffractograph of corn straw gasification ash: 1-KCl, 2-SiO₂, 3-Fe₂O₃, 4-KAlSiO₄, 5-K₂SO, 6-KSCN, 7-Na(AlSi₃O₈), 8-Fe₃O₄, and 9-KNaCO₃·6H₂O

Fig. 10. X-ray diffractographs of rice straw and corn straw gasification ash at 600 and 800 °C

The SEM-EDS results shown in Figs. 11 and 12 demonstrated that potassium was present at all scanning points for the alkali metals, which indicated that potassium was widely distributed in the gasification ash. The EDS data showed that the K, Na, and Cl in the ash decreased as the temperature increased from 600 °C to 800 °C. For the most part, the potassium remained in the gasification ash, with the exception of the potassium released in the form of KCl(g) ; meanwhile, sodium was mostly released in the form of NaCl(g) (Du *et al.* 2013), which agrees with the simulation results from thermodynamic equilibrium analyses. The data from the XRD analysis indicated that the potassium reacted with the Al, Si, C, and S in the form of oxide to generate carbonate, sulfate, and aluminosilicate, which produced more extreme levels of agglomerate potassium compound deposition.

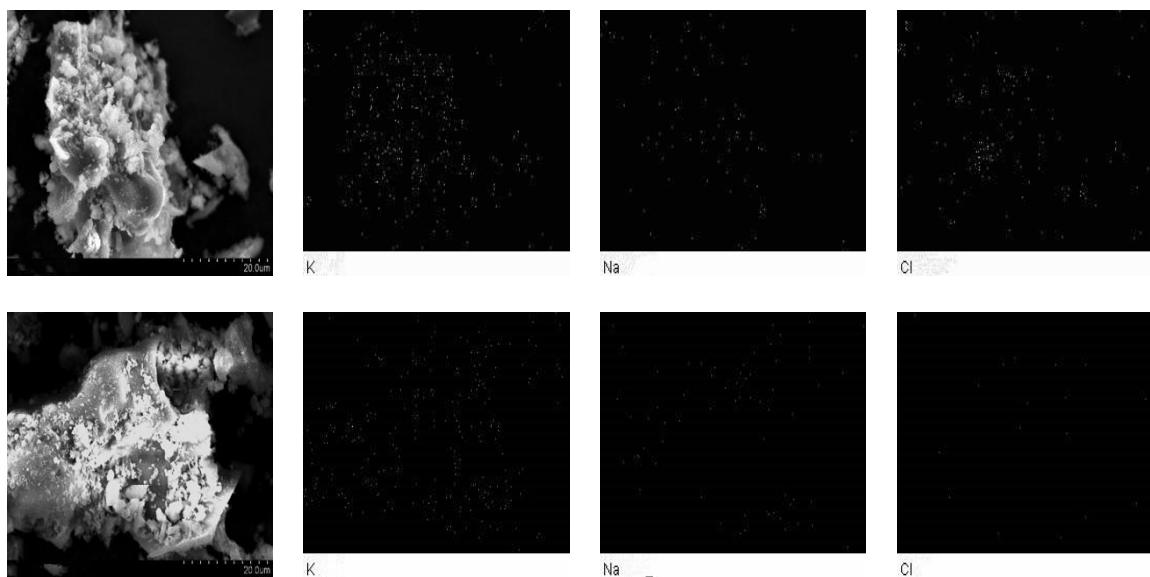


Fig. 11. SEM-EDS graph of rice straw gasification ash at 600 (top) and 800 °C (bottom)

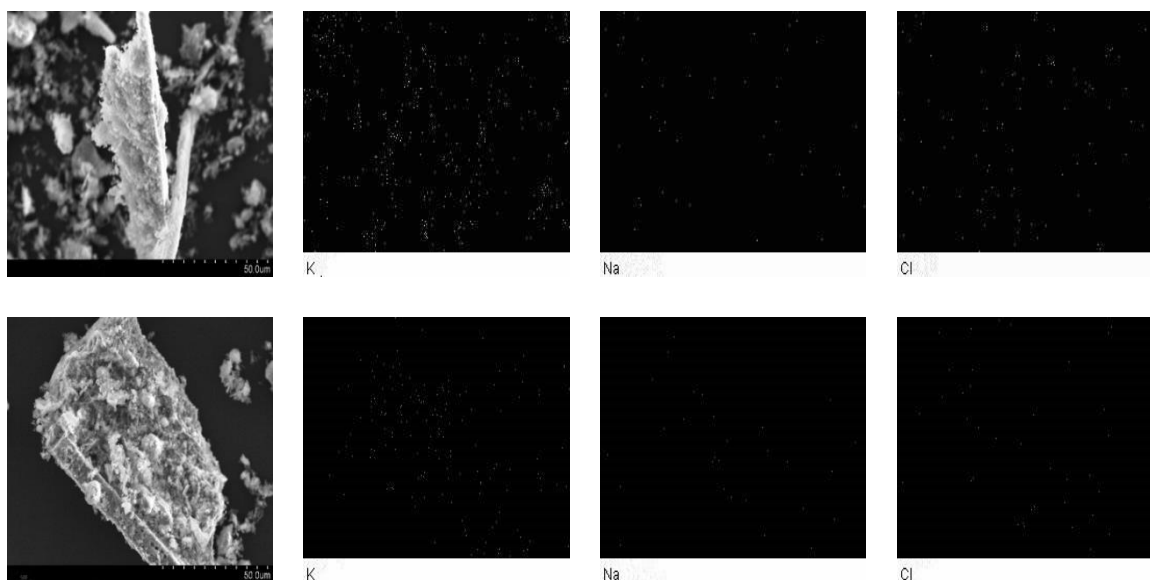


Fig. 12. SEM-EDS graph of corn straw gasification ash at 600 (top) and 800 °C (bottom)

CONCLUSIONS

1. The results from the thermodynamic equilibrium analysis showed that potassium was released mostly in the form of KCl (g), K₂Cl₂ (g), K (g), and KCN (g), and that sodium was released mostly in the form of NaCl (g) and Na₂Cl₂ (g).
2. The potassium, sodium, and chlorine released from the two materials increased with the increase in temperature from 600 to 1000 °C. The maximum amounts of K, Na, and Cl released from rice straw were 38.9%, 18.7%, and 34.9%, respectively, and the maximum amounts of potassium and chlorine released from corn straw were 24% and 43.6%, respectively, which occurred at 1000 °C. The maximum Na released from corn straw was 77.6%, at 700 °C, and the Na released was greater than K.
3. The X-ray diffraction results show that as the temperature increased from 600 to 1000 °C, the potassium and sodium in the gasification ash were mostly converted into carbonate, sulfate, silicate, and aluminosilicate.

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