



## Research article

# Biomass ash induced agglomeration in fluidized bed. Part 2: Effect of potassium salts in different gas composition

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## ABSTRACT

Agglomeration is one of the main challenges for combustion and gasification of biomass in fluidized beds. Bed agglomeration is related to K species present in biomass. Understanding the role of different types of K species on formation of agglomerates at different conditions can reveal the mechanisms of biomass ash induced agglomeration. Extensive experiments are conducted in a laboratory scale fluidized bed reactor, using mixtures of quartz sand and K species, including KCl, K<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>CO<sub>3</sub>, to study the agglomeration mechanisms. The effects of gas composition, including air, H<sub>2</sub>- and H<sub>2</sub>O-containing gas are investigated. The morphology and elemental analyses of the agglomerate samples are examined by SEM/EDS analysis. Thermodynamic equilibrium calculations are performed for verifying the proposed mechanisms. The results show that the role of various forms of potassium salts on agglomeration in fluidized beds is different. Gas composition also has strong impact on the agglomeration tendency. In the air and H<sub>2</sub>-containing gas, defluidization of KCl in a sand bed is caused by the melt of KCl. However, KCl reacts with SiO<sub>2</sub> to form K-silicates in the H<sub>2</sub>O-containing gas, which results in a lower defluidization temperature. No defluidization is observed for K<sub>2</sub>SO<sub>4</sub> in the presence of oxygen or water. However, K<sub>2</sub>SO<sub>4</sub> decomposes and reacts with SiO<sub>2</sub> to form K-silicates, causing defluidization at 850 °C in the H<sub>2</sub>-containing gas. In the air, H<sub>2</sub>- or H<sub>2</sub>O-containing gas, K<sub>2</sub>CO<sub>3</sub> would react with SiO<sub>2</sub> to form potassium silicates and KOH may be additionally formed in the H<sub>2</sub>- and H<sub>2</sub>O-containing gas. The possible mechanisms of agglomeration of various potassium salts at different conditions are discussed.

## 1. Introduction

In our previous work [1], biomass ash induced agglomeration and defluidization phenomena were investigated in different gas composition in order to reveal the mechanisms of agglomeration in fluidized bed combustion and gasification of biomass. It was revealed that gas composition has a significant effect on the agglomeration mechanisms. In high concentration of H<sub>2</sub>O, the agglomeration induced by corn straw and rice straw ash appears to follow the coating-induced mechanism. However, at oxidizing (air) or reducing (H<sub>2</sub>-containing gas) conditions, the melting-induced mechanism seems dominant. Furthermore, defluidization temperature at reducing condition is significantly lower than that at oxidizing condition. It is also observed that the release of

potassium to gas phase is enhanced in the H<sub>2</sub>O-containing gas, while the amount of melted potassium is significant in the H<sub>2</sub>-containing gas. It is speculated that the formation of coating in the H<sub>2</sub>O-containing gas is related to the transformation of KCl and the higher liquidus potassium is caused by the transformation of K<sub>2</sub>SO<sub>4</sub>. However, the speculations need further confirmation.

Gas composition may influence the transformation of different form of K-compounds. The type of K-compounds may play an important role in the formation of agglomerates [2,3]. During fluidized bed combustion or gasification, K-species in biomass ash may react with SiO<sub>2</sub> to form low melting point potassium-silicates, resulting in the formation of agglomerates [4–11]. The gaseous K-species released from biomass may react with bed material, forming coating layers on the particle surfaces

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and increasing the agglomeration tendency [12,13].

KCl,  $K_2CO_3$  and  $K_2SO_4$  are the typical potassium compounds present in biomass ash [14]. Char-bonded K may be oxidized to form  $K_2CO_3$  during combustion [15]. Most chlorine in biomass ash exists likely in the form of KCl [16,17].  $K_2SO_4$  is formed during the combustion of biomass with relatively high content of sulfur [10]. The slag phase of the biomass ash may exist in the form of  $K_2O \cdot nSiO_2$  [7,18], which is formed by the reaction between K-containing compounds and  $SiO_2$  [2,16,19,20]. At high temperatures, potassium in biomass may be released to the gas phase in form of KOH when the chlorine content is low and the presence of  $H_2O$  may enhance the formation of KOH [16,21–24].

Many investigations have been carried out to study the mechanisms of K-induced agglomeration in fluidized bed combustion/gasification using biomass [4,25–27]. The existing form of K-compounds may have large influence on the agglomeration. It has been observed that the agglomeration tendency induced by biomass with similar ash composition have huge differences, which may be related to the existing form of K-compounds [6]. Defluidization experiments of quartz bed with potassium salts were reported [19]. The influence of type of potassium salts, bed temperature, the amount of stepwise added salts and the air with and without steam on defluidization was studied. It is shown that KCl and quartz sand do not react and defluidization was caused by the melting of KCl as glue between sand particles.  $K_2CO_3$  reacts with sand forming potassium silicates, resulting in defluidization. No defluidization occurs for  $K_2SO_4$  at temperatures up to 900 °C. The similar conclusions with respect to the KCl-sand and  $K_2CO_3$ -sand interactions were reported by heating up of a fluidized bed until defluidization occurs [28]. However, systematic study on the contribution of individual K-compound to agglomeration at various gas composition is scarce, though the effect of steam is reported [19].

This paper focuses on the characteristics of agglomeration induced by different potassium compounds in different gas composition. The same procedure as out previous work [1] is applied, e.g. the mixture of potassium compounds and quartz sand was heated in fluidized bed until defluidization occurring or the temperature reaching 1020 °C. Defluidization temperature is used to indicate the tendency of agglomeration. The surface morphology and elemental distribution of agglomerates are further analyzed to reveal agglomeration mechanisms. The transformation of potassium salts in the presence of  $SiO_2$  in the different gas composition is indicated by thermodynamic equilibrium calculations.

## 2. Experimental

### 2.1. Apparatus

The experiments were carried out in a bench scale bubbling fluidized bed (BFB). The details of the setup are described elsewhere [1]. Here, only a brief summary of the setup is given. The setup consists of a fluidized bed reactor, a gas dosing system and a control/data acquisition system. The reactor is made of quartz with a height of 900 mm. A sintered quartz distributor is located in the middle of the reactor, below which is a preheating section, and above is the dense bed section. A freeboard section is present at the top of the reactor. The dense bed section is 32 mm in the inner diameter and 240 mm in height. The freeboard section is 120 mm in the inner diameter and 150 mm in height. A filter is installed at the exit of the reactor to collect fine particles. The reactor is electrically heated with a maximum temperature of 1020 °C.

The gas flow rate is controlled by mass flow controllers. The flow rate of  $H_2O$  is controlled by a precise water pump. The water stream is introduced to the bottom of the reactor and evaporated to steam in the preheating zone.

The pressure drop over the bed is measured by a differential pressure transducer. Two thermocouples are used to measure and control

**Table 1**  
Melting and evaporation points of the potassium compounds /°C [29].

K species	Melting point	Evaporation point
KCl	771	1437
$K_2SO_4$	1069	1670
$K_2CO_3$	901	N.A.
KOH	360	1323

the reactor temperature, respectively. A thermocouple is located at 20 mm above the gas distributor. A controlling thermocouple is situated between the furnace and the reactor close to the distributor. Signals of temperatures and pressure drop are continuously logged by a computer through a data acquisition system.

### 2.2. Materials

Quartz sand, with a solid density of 2600 kg/m<sup>3</sup>, is used as bed material. The particle size of the quartz sand ranges between 0.15 mm and 0.35 mm with a mean diameter is 0.28 mm. The composition of quartz sand is analyzed by an X-ray Fluorescent (XRF, AXIOS-MAX), showing a high content of  $SiO_2$  (98.33%), with a minor amount of  $Al_2O_3$ .

The potassium salts, KCl,  $K_2SO_4$  and  $K_2CO_3$ , are used as model potassium compounds. Potassium hydroxide is also tested in a few tests. The properties of the potassium compounds, such as the melting and evaporation temperatures are shown in Table 1 [29].  $K_2CO_3$  decomposes before evaporation occurs, thus, the evaporation point of  $K_2CO_3$  is not available as indicated in Table 1. All these potassium compounds are analytically pure (> 99.0%). The particles size distributions of the quartz sand and potassium salts are shown in Table 2.

### 2.3. Experimental conditions and procedure

Potassium salts and quartz sand particles are well mixed. The molar ratio of K to Si in the mixtures is kept constant of 0.01 in all experiments. The amounts of different potassium salts and quartz sand used in each experiment are summarized in Table 3.

Air, mixture of  $H_2/N_2$  (5 vol%  $H_2$ ) and  $H_2O/N_2$  (25 vol%  $H_2O$ ) are used as the fluidization gas to simulate different gas composition, respectively. The mixtures of  $H_2/N_2$  and  $H_2O/N_2$  are applied for simulation of the reducing condition and  $H_2O$ -containing gas condition in gasifiers, respectively. Air is used for simulation of oxidizing condition in combustors. The gas flow rate is kept constant of 1.5 NL/min. in all experiments. Similar to the procedure used in literature [10,30], the fluidized bed reactor is first heated to 550 °C at a heating rate of 20 °C/min to reduce the experiment time. After that, the heating rate is decreased to 3 °C/min and heating up is continued until defluidization occurs or until 1020 °C in case of no defluidization occurs. In this way, the defluidization temperature can be determined accurately. The temperature of 550 °C, at which the heating rate is changed, is the ashing temperature of the previous work [1], Here the same procedure is used for comparable results.

**Table 2**  
Accumulative particle size distribution of potassium salts and quartz sand /wt %.

Size/μm	KCl	$K_2CO_3$	$K_2SO_4$	Quartz sand
950	100	100		
750	94.6	99.8	100	
550	77.6	97.2	95.0	
350	25.2	84.1	64.3	100
250	8.0	76.0	45.8	58.4
150		63.4	18.6	6.2
60		48.8	6.4	

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