

SYSTEMATIC STUDIES OF ASH COMPOSITION DURING THE CO-COMBUSTION OF RAPESEED CAKE AND BARK

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Abstract: Agglomeration occurring during the firing of phosphorus-rich biomass is not fully understood. It has become clear that the ash-forming matter in agricultural biomass (e.g., rapeseed cake) is phosphorus dominated and that phosphate species are involved in agglomeration. Understanding the agglomeration mechanisms would provide valuable insight into possible strategies for preventing defluidization. The objective of this work was to study the ash composition resulting from the combustion of a phosphorus-rich fuel, in particular, the phosphorus compounds formed; the focus was the effect of ash composition on the formation of sticky ash particles, which cause defluidization. Eleven mixtures of rapeseed cake and bark with varying Ca/P molar ratio were studied. Using a 5 kW quartz BFB reactor operated at approximately 800 °C, eight-hour combustion tests were performed prior to each agglomeration experiment. Fly ash and bottom ash samples were extensively analyzed with SEM/EDX, and the fuel properties were also determined. Rapeseed cake ash consists of particles containing different K/Na-Ca/Mg-P compounds. The ash composition responsible for low defluidization temperatures was found to lie in the region of the ternary diagram containing 30-40 mol% P₂O₅, 20-40 mol% (CaO+MgO), and 30-50 mol% (K₂O+Na₂O). Sodium probably formed CaNaPO₄, but it was also found in mixtures with other phosphates. The addition of bark favoured the formation of orthophosphates containing 40-50 mol% (CaO+MgO), which increased the first melting temperature of the ash.

Keywords: rapeseed cake, bark, co-combustion, phosphorus, agglomeration

INTRODUCTION

The tendency of bed particles to agglomerate can cause defluidization and limit the operation of fluidized beds at high temperatures. The FBC agglomeration mechanisms during the firing of woody fuels are well-described; they mainly involve the reaction of high concentrations of alkali metals with bed material grains. The resulting inner reaction layer on the bed grains leads to viscous flow sintering (*Skrifvars et al., 1997; Öhman et al., 2000; Brus et al., 2005; Zevenhoven et al., 2006*). The same mechanisms are observed during bark combustion. However, bark contains a high concentration of calcium, which dissolves into the potassium silicate in the inner reaction layer, decreasing the amount of melt. With the introduction of agricultural residues into the fuel mixture, phosphorus has acquired a new importance as an element influencing the ash chemistry during combustion.

Phosphorus may play a significant role in the formation of agglomerates during biomass combustion (*Barišić et al., 2008; Piotrowska et al., 2010*). Phosphorus dominates silicon in the competition for the basic cations K⁺, Mg²⁺, Ca²⁺, and thus, with P-rich fuels, phosphates will form prior to silicates (*Boström et al., 2009*). The effect of phosphorus in minimizing the formation of the inner reaction layer on silica bed grains was observed in previous studies (*Grimm et al., 2011*). Phosphate-rich ash with a low melting temperature formed, resulting in defluidization. The introduction of calcium-based additives was found to be beneficial during the combustion of phosphorus-rich fuels (*Lindström et al., 2007; Steenari et al., 2009*).

This work is a continuation of a previous study on the agglomeration characteristics of rapeseed

cake-bark fuel mixtures, in which defluidization temperature curves were determined for the fuel mixtures and will be published elsewhere. The combustion of rapeseed cake, whose ash chemistry is dominated by phosphorus and alkali metals, leads to defluidization at approximately 800 °C. Increased defluidization temperatures were observed for mixtures with a minimum of 60 wt% bark which was correlated with an increasing Ca/P molar ratio. In this study, a detailed investigation of ash composition was performed. The results of SEM/EDX point analyses of particles in bottom and fly ash were presented in ternary diagrams with the aim of determining the phosphorus compounds formed during the combustion of rapeseed cake-bark fuel mixtures. The results can be used to enhance the understanding of how the ash chemistry influences the bed agglomeration process.

EXPERIMENTAL SET-UP

Reactor

The experiments were carried out in a bench-scale 5 kW bubbling fluidized bed reactor (BFB), described in detail by *Öhman and Nordin (1998)*. The stainless steel reactor is 2 m high. The minimum fluidization velocity is 8 Nl/min, corresponding to 0.1 m/s. After leaving the freeboard section, the flue gases pass through a cyclone separator with a cut size of approximately 10 µm. The maximum temperature that can be reached in the bed is 1045 °C. A constant temperature in the reactor is achieved by using preheated primary air, in conjunction with electrical heaters in the freeboard section. A perforated stainless steel plate at the bottom of the fluidized bed with 1% open area functions as an air distributor. The temperature and pressure drop in the bed were continuously monitored using two thermocouples and two pressure probes. The defluidization temperatures were determined based on the measured pressure and temperature profiles as will be reported elsewhere.

Combustion and Agglomeration Tests

Eleven mixtures of rapeseed cake and bark were studied. The proportion of bark in the fuel mixture ranged from 0-100 wt%. The test procedure comprised two stages: combustion and agglomeration. During the eight-hour combustion stage, 4-5 kg of each mixture was continuously fed into the reactor, and the temperature in the bed and that in the reactor raiser were maintained at approximately 800 °C. For each experiment, 540 g of fresh quartz sand (200-250 µm) was used as the bed material. After the combustion stage a sample of bottom ash was collected, and a controlled fluidized bed agglomeration test was carried out. During the agglomeration test, the bed temperature was increased at 3 °C/min until defluidization occurred or the maximum reactor temperature (1045 °C) was reached. During the agglomeration tests, propane was burned below the air distribution plate to allow proper mixing of gases and to maintain a combustion atmosphere in the reactor. After defluidization was achieved and the reactor was cooled down, also an ash sample from the cyclone was collected. The procedure for determining the defluidization will be published elsewhere.

Table 1. Fuel Properties

Fuel	Rapeseed cake	Bark
LHV [MJ/kg db]	20.6	19.8
Volatile [% db]	77.8	72.3
Fixed carbon [% db]	14.7	22.8
Ash [% db]	7.5	4.9
C [% db]	50.0	52.3
H [% db]	6.6	5.7
N [% db]	4.9	0.4
O (calculated) [% db]	29.8	36.6
S [% db]	0.47	0.02
Cl [% db]	0.67	0.03
P [mg/kg db]	12918	506
Si [mg/kg db]	462	4642
Al [mg/kg db]	53	1006
Mg [mg/kg db]	4161	790
Ca [mg/kg db]	7933	9648
Na [mg/kg db]	4518	356
K [mg/kg db]	12618	2192
Mn [mg/kg db]	61	479
Fe [mg/kg db]	207	497
Zn [mg/kg db]	64	96

Table 2. Experiments, Molar Ratios and Defluidization Temperatures

	in dry fuel*	B100	B90	B80	B70	B60	B50	B40	B30	B20	B10	B0
Ash content	wt %	4.9	5.2	5.4	5.7	5.9	6.2	6.5	6.7	7.0	7.2	7.5
Mass bark/total fuel mass	%	100	90	80	70	60	50	40	30	20	10	0
Ash bark/total ash inflow	%	100	85	72	60	49	40	30	22	14	7	0
Si _{fuel} /P	molar	9.17	2.67	1.40	0.88	0.60	0.42	0.30	0.21	0.14	0.08	0.04
Ca/P	molar	19.06	4.19	2.41	1.67	1.27	1.01	0.84	0.71	0.61	0.54	0.47
(Na+K)/P	molar	5.03	2.06	1.67	1.51	1.42	1.36	1.32	1.30	1.28	1.26	1.25
Na/K	molar	0.16	0.41	0.47	0.51	0.54	0.56	0.57	0.59	0.60	0.60	0.61
(Na+K)/(Mg+Ca)	molar	0.24	0.41	0.55	0.68	0.80	0.92	1.03	1.13	1.23	1.32	1.41
IDT***	°C	>1045	919	910	896	860	DC**	DC	DC	DC	DC	DC
TDT****	°C	>1045	>1045	>1045	1043	1041	1013	982	974	954	873	~800

*based on bark and rapeseed cake ash content; **DC stands for defluidization during combustion; **initial defluidization temperature (IDT), ***total defluidization temperature (TDT)

Analyses of Fuels and Ash Samples

The fuel samples were analyzed by an external laboratory according to Swedish standards. Elemental analyses were performed with ICP-AES and ICP-SFMS. For this purpose, a dry fuel sample was digested with LiBO₂ and dissolved in HNO₃. The fuel properties are shown in Table 1. Two types of ash samples were analyzed using SEM/EDX: fuel-derived ash found in the bottom ash, and fly ash. Fuel-derived ash particles ($d_p < 200 \mu\text{m}$) from the bottom ash sample were sieved, and their composition was studied. For simplicity, this sample is called bottom ash. Fly ash samples were collected from the cyclone ($d_p < 10 \mu\text{m}$) after each experiment when temperature in the reactor dropped to room temperature. For the SEM/EDX analyses, ash samples were mounted on carbon tape and covered with a thin layer of carbon. The results are expressed as mol% oxides on a carbon-free basis. The experiments with 0 wt%, 10 wt%, 20, wt%, 30 wt%, 40 wt%, 50 wt%, 60 wt%, 70 wt%, 80 wt%, 90 wt%, 100 wt% bark are denoted by B0, B10, B20, B30, B40, B50, B60, B70, B80, B90, and B100, respectively. For the bottom ash sample from each of the experiments B0, B10, B50, B60, B70, and B90, between 50 and 90 point SEM/EDX analyses were performed. For the pure bark combustion test (B100), only a few bottom ash particles could be obtained, so only 12 data points are available for this test. For the fly ash sample from each of the experiments B0, B10, B50, B70, B90, and B100, between 60 and 100 SEM/EDX point analyses were performed. For the purpose of this work, approximately 470 data points were obtained from the bottom ash and 480 data points from fly ash samples. The results were grouped and are presented in ternary diagrams. The composition of each data point was normalized to 100% of the major oxides corresponding to the corners of the diagrams. To ensure a reliable correspondence between the ternary diagrams and the compositions represented by the data points, the oxides were chosen so that, together, they account for at least 90 mol% before normalization; in fact their sum is often close to 100 mol%.

RESULTS AND DISCUSSION

Fuels and defluidization temperatures

The fuel properties are shown in Table 1. The elemental composition of rapeseed cake is dominated by high concentrations of phosphorus, alkali, and alkaline earth metals. An unusual feature of the rapeseed cake is its relatively high sodium content (Na/K molar 0.6) compared to that of other biomass fuels. The sulphur and chlorine contents are also high. Bark is dominated by calcium, silicon, and potassium. Based on the laboratory studies of woody fuels reported in *Werkelin et al. (2010)*, the silicon in bark is usually considered to be a fuel contaminant resulting, for example, from contact with soil.

Eleven tests were carried out with mixtures having different proportions of bark. The aimed proportion of bark is indicated by the name of the test (Table 2). The properties of the mixtures in Table 2 are derived from those of the original fuels (Table 1) using the proportion of bark in the fuel mixture, i.e., the ratio of bark mass to total fuel mass. The ash content of the bark is roughly half that of the rapeseed cake, so the theoretical ash inflow (based on ash content) is dominated by rapeseed cake ash for mixtures up to 50 wt% bark. With these mixtures, there were problems maintaining stable operation during the combustion phase at 800 °C. Pressure drop in the bed was constantly decreasing, so instead of listing the initial defluidization temperatures (IDT), Table 2 indicates that defluidization occurs during combustion, despite the observed increase in the total defluidization temperatures (TDT). In the mixture with 60 wt% bark, the content of bark ash and that of rapeseed cake ash are approximately equal. For mixtures with 70 wt% bark or more, the theoretical ash inflow is dominated by bark ash. Mixtures with 60 wt% bark or more had significantly higher defluidization temperatures than mixtures dominated by rapeseed cake ash. This difference could be correlated with the higher Ca/P molar ratio. The first melting temperatures of phosphates increase with higher contents of alkaline earth metals (*Lindström et al., 2007; Barišić et al., 2008; Steenari et al., 2009*). In the first part of this study it was shown that bark can be used as a calcium source during co-combustion with phosphorus-rich biomass.

Overall composition of the bottom ash and the fly ash

Morphological studies of the bottom samples and the fly ash samples performed with SEM/EDX are summarized in Fig. 1. The major elements identified in the bottom ash are silicon, phosphorus, sulphur, calcium, magnesium, potassium, and sodium. In the fly ash sample, the same elements were identified, but chlorine was also detected. This finding is consistent with the elemental analyses of the fuel samples (Table 1). However, there is another important source of silicon in the ash: some of the bed material may find its way into both types of ash samples due to mechanical stress. In Fig. 1, it can be observed that most of the samples contain less than 10 mol% silicon (expressed on an oxide basis). The samples containing a significant amount of silicon are mainly ash particles from mixtures with a high proportion of bark (B90 and B100); for these mixtures, approximately 40% of the fly ash data points lie on the line between the corners corresponding to SiO₂ and earth metals. In these tests, there were no significant operational problems due to fluidization, indicating that the troublesome ash chemistry is dominated by anions associated with the other corner (SO₃ and P₂O₅). With this in mind, only samples containing less than 10 mol% silicon are considered in the discussion below.

Bark and Rapeseed cake

The compositions of ash particles found in the fly ash samples and bottom ash samples from the combustion of bark (B100) and rapeseed cake (B0) are shown in Fig. 2. The compositions of the fly ash samples and the bottom ash samples from the combustion of mixtures dominated by bark (B90) and rapeseed cake (B10) are shown in Fig. 3. After bark combustion (B100), the bottom ash samples

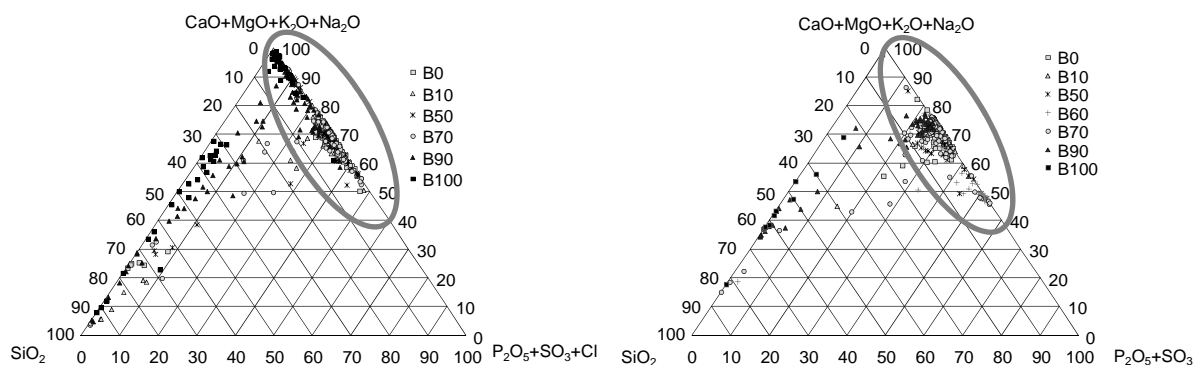


Figure 1. Composition of fly ash (left) and bottom ash (right) samples based on SEM/EDX. The composition is expressed in mol% of major elements in the form of oxides. The composition is normalized to 100%. The ellipses indicate point analyses with less than 10 mol% silicon.

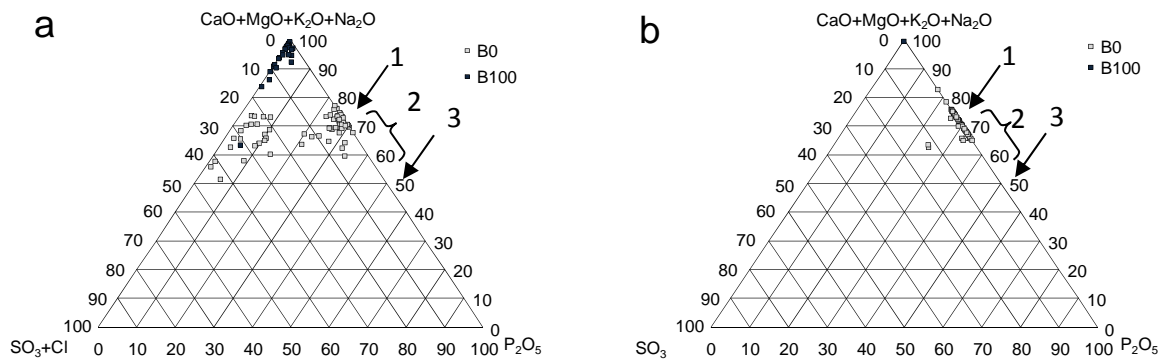


Figure 2. Composition of fly ash (a) and bottom ash (b) samples from mixtures containing 0 wt% bark (pure rapeseed cake) and 100 wt% (pure bark) based on SEM/EDX. Here, only points corresponding to $\text{SiO}_2 < 10 \text{ mol}\%$ are shown.

contained very few fuel ash particles; Fig. 2b B100 shows the composition of a single bottom ash particle. Bark formed mainly fly ash, indicated by the data points near the top corner of the triangle (Fig. 2a). The corresponding samples consist mostly of calcium (expressed in terms of oxide). The calcium compounds occurring in biomass ash are CaO , CaCO_3 , and Ca(OH)_2 . However, in this study, these compounds could not be distinguished and so are described as CaO . Some calcium sulphate and chloride also formed. Higher amounts of sulphates and chlorides formed in test B90 (Fig. 3) than in the pure bark test (B100). This could be explained in terms of the inflow of rapeseed cake ash (Table 2). Sulphates and chlorides, shown in Figs. 2 and 3 (samples containing $< 10 \text{ mol}\%$ P_2O_5), occurred almost entirely in areas high in calcium. Sulphates and chlorides also formed during the combustion of rapeseed cake (B0) (Fig. 2a) and of the rapeseed cake dominated mixture (B10) (Fig. 3a); in this case as well, they were generally found together with calcium. Only a few particles with both alkali and sulphur could be observed.

For the rapeseed cake ash samples, alkali metals were generally observed together with phosphorus. This is in agreement with *Boström et al., 2009*, who concluded that alkali metals will primarily form phosphates. In the bottom ash from B0 (Fig. 2b) and B10 (Fig. 3b), mainly phosphates were found. The numbers 1, 2, and 3 in all the ternary diagrams represent different forms of phosphates. Number 1 indicates orthophosphates (PO_4^{3-}), corresponding to approximately 25 mol% P_2O_5 . Phosphorus can also occur in other structures like polymeric ions such as metaphosphates ($\text{P}_3\text{O}_9^{3-}$) and pyrophosphates ($\text{P}_2\text{O}_7^{4-}$) (*Lindberg et al., 2011*). The formation of tripolyphosphates ($\text{P}_3\text{O}_{10}^{5-}$) in biomass ash samples was also reported by *Boström et al., 2009*. Pyrophosphates and tripolyphosphates are visible in the diagrams at the levels of approximately 33 mol% and 38 mol% P_2O_5 ; this range of phosphorus content is indicated by number 2. On the other hand, this composition could also correspond to mixtures of the different types of phosphates mentioned. Therefore, it is difficult to determine if number 2 indicates pure pyro- and tripoly-phosphates or if it denotes the formation of phosphate mixtures. Number 3 corresponds to the formation of methaphosphates.

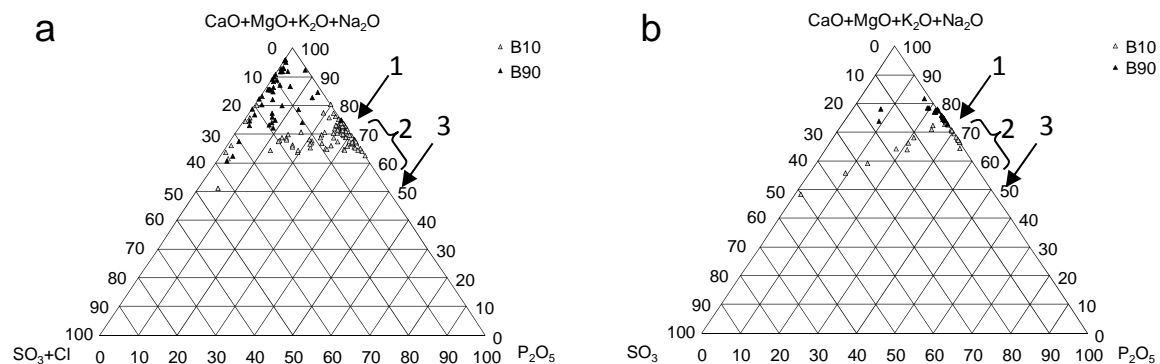


Figure 3. Composition of fly ash (a) and bottom ash (b) samples for B10 and B90 based on SEM/EDX. Here, only points corresponding to samples with $\text{SiO}_2 < 10 \text{ mol}\%$ are shown.

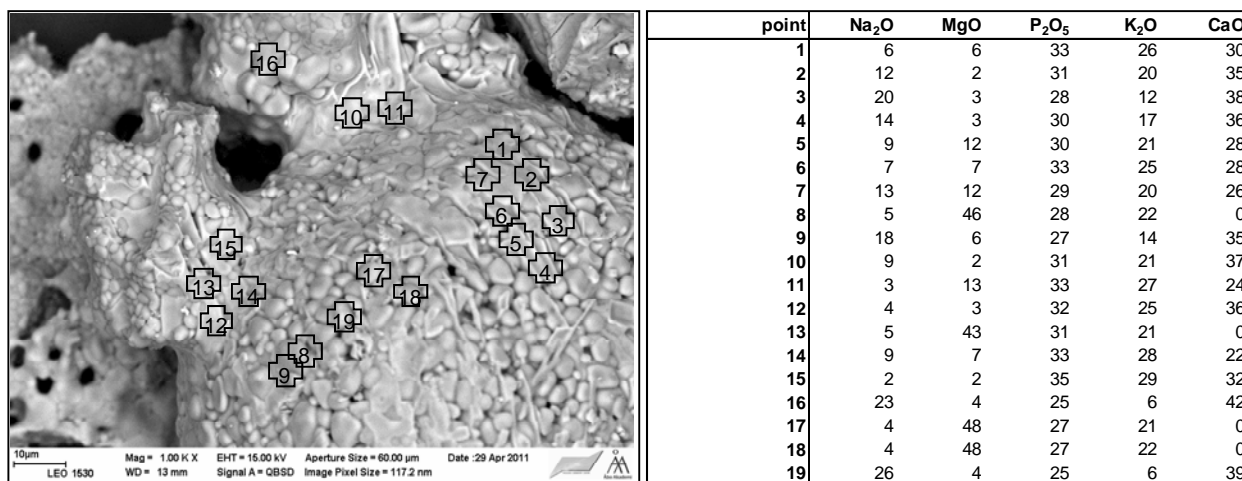


Figure 4. Example of an RC ash particle found in the bottom ash sample after 8 h of fluidized bed combustion at 800 °C and its composition expressed in mol%, on an oxide basis, obtained from SEM/EDX studies. Composition is normalized to 100 mol%. Before normalization, the composition ranged between 94 mol% and 100 mol% for the elements shown.

The compositions of rapeseed cake ash particles lie in the ranges designated by 1 and 2 (Figs. 2 and 3). Particles in these ranges caused defluidization at fairly low temperatures (~800 °C) (Table 2). The low defluidization temperature and the difficulty in maintaining stable FBC operation can be attributed to the formation of partly molten ash particles dominated by phosphates. However, alkali and calcium orthophosphates (number 1 in all the ternary diagrams) melt at very high temperatures (*Lindberg et al., 2011*), so, during the combustion of phosphorus-rich biomass, the formation of orthophosphates is desirable. It is the composition indicated by number 2 that is responsible for the formation of low melting temperature ash during rapeseed cake combustion. The SEM/EDX point analyses make it possible to distinguish the compounds involved, however, due to the normalization of composition results and limitations of SEM/EDX for quantitative studies, interpreting of the data should be done carefully.

Fig. 4 shows an example of a rapeseed cake ash particle from B0; it has a heterogeneous composition with a high phosphorus content. It is plausible that different types of phosphates could be present in a single ash particle. There seem to be correlations between forms of phosphates: K and Mg are found together in higher concentrations at several points, as are Na and Ca. Based on SEM/EDX analysis and stoichiometry the formation of some compounds may be inferred. Figure 4 suggests that orthophosphates such as CaNaPO_4 (point 16) and KMgPO_4 (points 17 and 18) are present together with other phosphates having a composition, for example, close to that of $\text{CaK}_2\text{P}_2\text{O}_7$ (point 1). Two of them (KMgPO_4 and $\text{CaK}_2\text{P}_2\text{O}_7$) have previously been identified in biomass ash samples (*Lindström et al., 2007*). With rapeseed cake used in this study, sodium-rich compounds have also been identified (e.g., CaNaPO_4). In addition, the point analyses indicate the presence of complex phosphate mixtures. Both these complicate the melting behaviour: the substantial amount of sodium found in the rapeseed mixtures could have a considerable influence on the melting behaviour of the phosphates. The problem is also complicated by the fact that the melting behaviour of mixtures can differ dramatically from the melting behaviour of the pure compounds, affecting the amount of melt and the first melting point of the ash.

The formation of mixed sulphate and phosphate salts was observed for B10 (Fig. 3a) and in fly ash from 100% rapeseed cake combustion (B0) (Fig. 2a). Alkali phosphates are completely miscible with many alkali salts, e.g., sulphates (*Lindberg et al., 2011*), which could lower their solidus temperature and increase the amount of melt occurring during combustion, leading to the formation of sticky ash particles and the agglomeration of bed material grains. Only a few points indicate the presence of a mixed salt in mixtures dominated by rapeseed cake ash. Accordingly, it is thought that the formation of mixed sulphates and phosphate salts and the subsequent effects do not to have a significant influence on the defluidization temperatures.

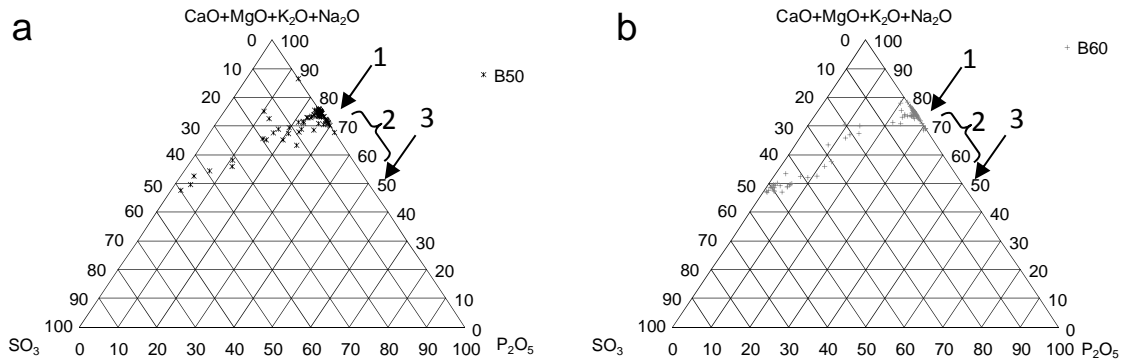


Figure 5. Composition of bottom ash for tests B50 (a) and B60 (b) based on SEM/EDX. Points with $\text{SiO}_2 > 10$ mol% were excluded. Composition is normalized to 100%.

Mixtures

Fig. 5 shows the compositions of ash particles from the bottom ash samples collected after the combustion of 50 wt% bark (B50) and 60 wt% bark (B60). It can be observed that sulphates formed in the ash, but almost no calcium was present. Increasing the amount of bark in the fuel mixture, clearly, leads to a change in the ash chemistry for both B50 and B60, phosphates are found mainly in the region corresponding to orthophosphates, whereas sulphates most probably occur as $(\text{K},\text{Na})_2\text{SO}_4$.

Alkali and alkaline earth metals

The composition of phosphates found in the bottom ash (points corresponding less than 10 mol % SO_3 in Figs. 2, 3, and 5) are also presented in Figs. 6 and 7. The ternary diagrams for P_2O_5 were plotted separately to determine the approximate amount of alkali and alkaline earth metals in the phosphate compounds, which is of crucial importance in discussions of phosphate chemistry since the amount of alkali and alkaline metals could affect the melting behaviour of phosphates (Lindström et al., 2007). In Fig. 5, it can be seen that the phosphates formed during rapeseed cake combustion contain no more than 20 mol% $\text{CaO}+\text{MgO}$. The ash particles, whose composition lies in range 2, contain 20-40 mol% of $\text{CaO}+\text{MgO}$. The phosphates found in the bottom ash from B90 seem to be orthophosphates (indicated by 1) with an alkaline earth metals content of approximately 50 mol% or higher. This is consistent with, for example, the CaKPO_4 reported to form during biomass combustion (Lindström et al., 2007), a compound with a high melting temperature. Almost all the point analyses for B60 and B70 (Fig. 6) indicate the formation of orthophosphates. As shown in Table 2, the $(\text{K}+\text{Na})/(\text{Ca}+\text{Mg})$ fuel molar ratio decreases with increasing proportion of bark in the fuel mixture, favouring the formation of alkaline dominated phosphates (Figs. 6 and 7).

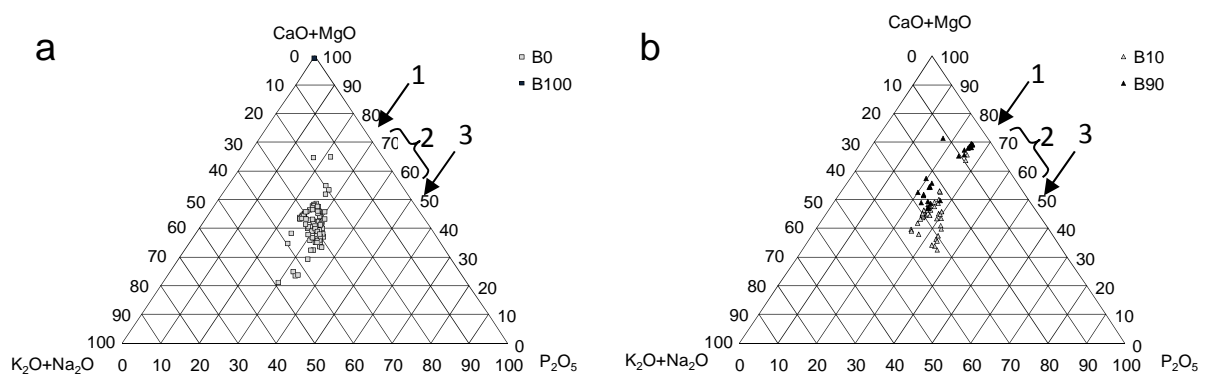


Figure 6. Composition of ash particles in bottom ash samples from tests with 100% fuel (a), B0 and B100, and mixtures (b) with a high proportion of rapeseed cake (B10) and with a high proportion of bark (B90) based on SEM/EDX. Composition is normalized to 100%. Before normalization, the sum of the oxides corresponding to the corners was above 90 mol%.

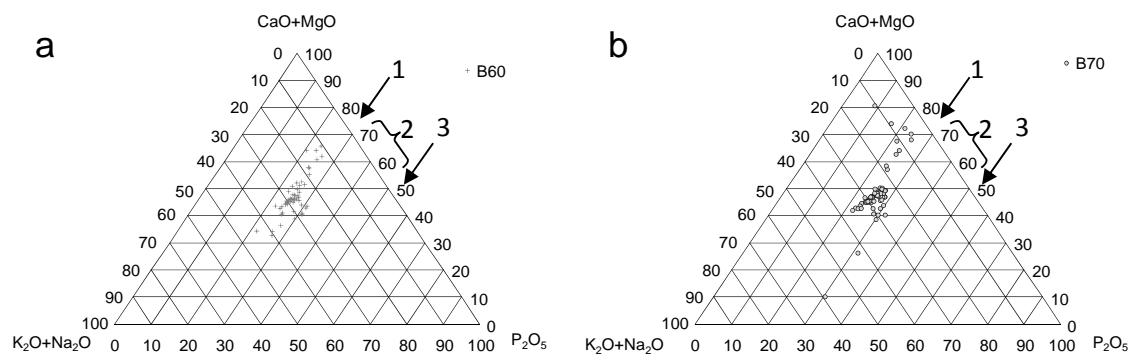


Figure 7. Composition of ash particles in bottom ash samples for B60 (left) and B70 (right) based on SEM/EDX. Composition is normalized to 100%. Before normalization, the sum of the oxides corresponding to the corners was above 90 mol%.

CONCLUSIONS

Systematic studies were performed to determine the ash compositions resulting from the combustion of rapeseed cake and bark mixtures. As the proportion of bark increased, the Ca/P molar ratio increased, while the (Na+K)/(Ca+Mg) molar ratio decreased. This corresponds to higher defluidization temperatures for fuel mixtures compared to those for 100% rapeseed cake. Extensive SEM/EDX point analyses of bottom and fly ash samples indicate that phosphate dominated ash is responsible for the low defluidization temperatures recorded. Rapeseed cake ash, most probably partly molten, consists of particles that are heterogeneous in composition. Ash particles with a composition in the range 30-40 mol% P_2O_5 and 20-40 mol% CaO+MgO, with the remainder alkali metals, may be responsible for the formation of low melting temperature ash during rapeseed cake combustion. This ash contributes to the low defluidization temperature for rapeseed cake and rapeseed cake dominated mixtures. Addition of bark to the fuel mixture favours the formation of orthophosphates (approximately 25 mol% of P_2O_5) with an alkaline earth metals content 40-50 mol%. The influence of sodium on the melting behaviour of phosphates needs to be investigated further.

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