



Experimental study and SEM-EDS analysis of agglomerates from gasification of biomass in fluidized beds

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ABSTRACT

Fluidized bed gasification is an attractive conversion technology for biomass. However, processing biomass in fluidized beds suffers from ash related problems. In this work, bed agglomeration of wood pellets and grass pellets was compared, focusing on de-fluidization temperatures and variation in microstructure and composition of agglomerates. The results showed that both biomasses form agglomerates at temperatures between 700 °C and 900 °C. After each test run, residual sand and agglomerates were discharged and the ash/bed material ratio was calculated. Larger amount of accumulated ash and agglomerates were collected from the gasification of grass pellets than that from wood pellets, indicating that different mechanisms are present in the agglomerate formation. The ash/bed material varied from 3 to 10 wt% for the grass pellets and from 0.8 to 1.5 wt% for the wood pellets. A scanning electron microscope with energy dispersive X-ray spectroscopy was used for characterizing morphology and elemental composition of the agglomerates. The findings show that the surface of agglomerates from grass were dominated by Si, Ca and K, while the surface of agglomerates from wood mainly were dominated by Si and K. The cross-sectional analysis concluded that Si, Ca, and K are the ash forming elements that play the most significant role in agglomeration processes.

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1. Introduction

Biomass is a major source of conventional fuel that has a great potential to reduce the dependence on fossil fuels, and at the same time provide energy in a more sustainable and carbon-neutral manner. One of the most suitable approaches for energy production from biomass is gasification in fluidized beds [1]. Gasification of biomass represents a thermochemical process that converts the biomass into useable gaseous products, preferably an energy rich syngas consisting of mainly carbon monoxide (CO) and hydrogen (H₂). The syngas can be directly used in the form of heat and power, or it can be further synthesized into storable energy carriers such as gaseous and liquid transportation fuels and other convenient base chemicals [1,2]. Fluidized beds have been widely used for biomass gasification during the last decades, and the interests in these facilities have grown rapidly in chemical industries [3]. Due to the

important characteristics such as low process temperatures, efficient material mixing and excellent heat transfer, the fluidized beds are considered particularly suitable for handling a wide range of fuels from biomass [4,5]. The major advantage of fluidized bed gasifiers is the uniform temperature profile, which provides a gasification process with improved energy conversion and product gases with high heating values [2–4]. Despite the many benefits with the fluidized bed technology, the industrial applications for processing biomass are still hampered by fuel-specific troubles related to the presence and conversion of biomass ashes [5–8]. In large-scale industrial fluidized bed gasifiers, the main problem with biomass ash is the formation of agglomerates that may lead to partial or complete de-fluidization of the bed. The bed agglomeration occurs when the bed particles are glued together by molten ash, either by formation of a sticky ash layer or by direct adhesion [9]. If not counteracted, the consequence is poor gasification conditions and loss of fluidization, resulting in complete shutdown of the fluidized bed installation [3,5,10–13].

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The biomass materials derived from various sources contain different concentration of ash and certain ash forming elements. The different ash-forming elements are present in varying concentrations, and the ash characteristics are closely associated with the type of biomass that are used [8,9,11,14,15]. Woody biomass often has high content of Ca. Whereas the herbaceous biomass that die at the end of the growing season will tend to have higher ash content than forestry biomass that are build up over years [13–15]. In addition, ash from fast growing herbaceous biomass contains large amounts of alkali metals such as K and Na, while ash from biomass originating from woody materials has relatively large content of alkali earth metals such as Ca and Mg [16–18]. At increased temperatures, the alkali metal compounds will primarily react with other ash-forming elements such as Si, Cl and S. The release of the alkali metals takes place through a set of complex reactions affected by the physical characteristics and chemical compositions of the biomass, as well as the fluidized bed conditions including the reactor temperature and the air-to-fuel ratio [5,6,11]. Several research studies concerning the high temperature ash behaviour agree that the critical components that lead to operational problems in biomass conversion processes are Ca, Mg, K, Na, Si and P [8,17–23]. Once the organic materials are oxidized, the remaining substances form oxides that correspond to the main ash-forming elements in the biomass. Due to their low ionization number (+1) in oxidized forms, the alkali metals are highly reactive and will therefore readily react as gases. During biomass gasification, K and Na will initially react with water vapour producing the stable hydroxides KOH (g) and NaOH (g) [18]. Depending on the local temperature variations in the gasifier, part of the hydroxides may condense on the surface of bed material or in the free board zone [9,11,18,24]. The alkali earth metals, on the other hand, have high ionization numbers in oxidized forms and stay solid in all zones in the gasifier. The oxides CaO (s) and MgO (s) are less reactive and will mainly be released as fine particles during the gasification process. The same applies to Si, where the corresponding oxide SiO₂ in most cases will stay solid throughout the heating process [3,18]. Vassilev et al. [17] studied the ash-forming elements and their impact on bed agglomeration during biomass gasification in fluidized beds. They found that the alkali earth elements Ca and Mg typically increase the ash melting temperature, while the Si, P and the alkali metals K and Na decrease the ash melting temperature. The combination of high Si and high K and/or Na (K–Na) content is especially problematic for fluidized bed systems due to the formation of complex silicates (K₂O·nSiO₂ and Na₂O·nSiO₂) that melt at low temperatures [17]. Other research studies focusing on the critical elements in biomass ash came to the same conclusions, i.e. that systems rich in Si–P and K–Na and lean in Ca–Mg typically give high risk for agglomeration of bed material [3,10,19,24–26]. Furthermore, the same studies showed that systems with low content of Si–P in combination with high content of K–Na and Ca–Mg typically cause formation of chemical compounds that either remain in the bed as bottom ash or pass through the reactor as a non-sticky dust (fly-ash). However, the amount of portion of ash remaining in the bed decreases with less Ca and Mg present in the biomass. The ratios of Si/K and K/Ca are therefore important in these systems [19,24,25]. Large amounts of Ca can outcompete K in the interaction with Si from the bed material, and thereby cause more K to evaporate preventing it from binding to the bed particles providing a lower risk for bed agglomeration [10,14,19]. Many studies have been carried out and reported on agglomeration and de-fluidization during fluidized bed combustion of biomass. However, detailed studies on agglomeration during gasification of biomass in fluidized bed gasifier are still relatively less. This is especially true for herbaceous biomass such grass, as the previous studies mainly focused on conversion of woody

biomass materials. Monitoring and mechanisms of agglomeration of during fluidized bed gasification of grass is seldom.

De-fluidization due to bed agglomeration can be avoided by increasing the fluid flow or changing the fuel feed conditions in the fluidized bed system. The success of these actions, however, requires that any changes in the fluidized conditions need to be detected at an early stage in the process. The objective of this work was to study the formation of agglomerates during gasification of two types of biomasses. Grass was selected as herbaceous biomass that represents a biomass feedstock with high ash content and high alkali content. Wood was selected as a representative for biomass with low ash content and high fraction of alkali earth metals. The gasification experiments were carried out in a 20 kW bubbling fluidized bed gasifier. The biomasses were gasified in the temperature range from 700 °C to 900 °C using quartz sand with particle diameter ranging from 200 to 600 µm as bed material. The experiments were run continuously until the fluidized bed collapsed as a consequence of bed agglomeration and subsequently de-fluidization. The mass ratio of ash to bed material was introduced as a new, comparable unit of measurement, which could be calculated from the amount of accumulated ash in the bed at the time when agglomerates were formed in the bed. Agglomerates and residual bed particles were collected after ended experiments for each of the controlled agglomeration runs. The morphology and the present of elements in the different samples were characterized using a scanning electron microscope with energy dispersive X-ray spectroscopy (SEM-EDS). The SEM-EDS analysis on the agglomerates were conducted with two purposes: 1) to analyse and compare the morphology of agglomerates generated under different operational conditions, and 2) to examine micro ash chemistry and to reveal possible agglomeration mechanisms. The SEM-EDS analysis on typical agglomerates have been widely used to study both the surface and the cross section of agglomerates. Although the EDS analyses are semi-quantitative, clear trends and correlations can still be identified through sufficient and representative EDS spot analyses and mapping on the different elements. The SEM-EDS analysis has been reported in previous studies as an efficient way to study detailed agglomeration severity, chemistry, and mechanism during thermal conversion of biomass in a fluidization system [8,27–29].

2. Materials and methods

Coniferous wood from Norwegian forests and grass from timothy, lucerne and clover were used for the study. The biomass samples used were cylindrical pellets sized with a length of 5–20 mm and a diameter of approximately 0.6 cm. The purposed pellets are packed and sealed in plastic bags. Before one gasification experiment, the pellets in the sealed bags were taken out and fed into the gasifier directly without further storage and being exposed to ambient atmosphere. The as-received biomass pellets were characterized, and results are shown in Table 1. The analyses are determined according to standardized methods carried out by the certified laboratory Eurofins AS Norway. The proximate and ultimate analyses show that grass pellets have high fraction of ash, Cl and S compared to wood pellets that have low ash content and contains only traces of Cl and S. Fig. 1 compares the elemental composition of ash from grass pellets and wood pellets. Extra focus was given to the ash-forming elements that contribute to the critical amount of ash in fluidized bed gasifiers. As shown in Fig. 1, Si, K and Ca are the predominant elements in both biomass ashes, and count for more than 80% of the major ash forming elements. Higher relative amount of Si, P and the alkali metals K and Na are found in grass pellets, while wood pellets have higher level of Mn and high relative amount of the alkaline earth metal Ca. The

Table 1
Fuel characteristics and standardized methods for analyses of the biomass (as received).

	Grass	Wood	Analytical method
Proximate analyses (d.b., wt %)			
Moisture	8.4	7.9	EN-ISO 18134 - 1, 2, 3 (2015)
Ash content	9.5	0.6	EN-ISO 18122 (2015)
Volatiles	75.9	83.9	SS-EN ISO 18123 (2015)
Fixed Carbon	6.2	7.7	Calculated by difference
Ultimate analyses (d.b., wt %)			
C, H, N			EN-ISO 16948 (2015)
C	46.9	51.3	
H	5.7	6.1	
N	3.2	<0.1 ^a	
S, Cl			EN-ISO 16994 (2016)
S	0.3	<0.1 ^a	
Cl	0.075	<0.0011 ^a	
O	33.7	42.0	Calculated by difference
Heating value (MJ/kg)	19.396	20.382	SS-EN ISO 18125 (2017)

^a The sensitivity of the methods is limited to 0.1 for N, S and Cl.

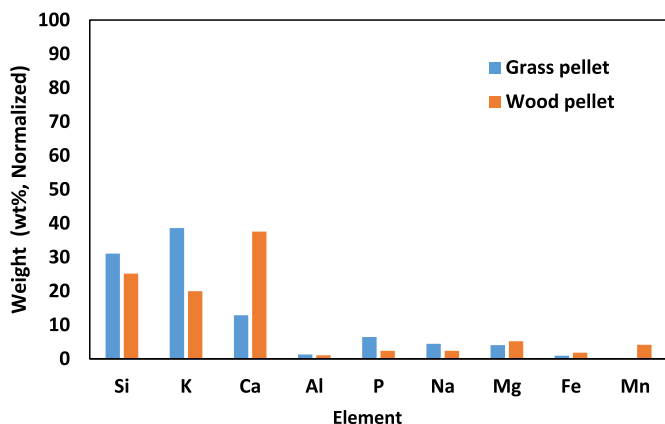


Fig. 1. Comparison of elemental composition of ash from grass pellets and wood pellets.

differences between the Mg, Al, Fe and Ti in content the two biomasses are rather small.

2.1. Experimental setup

A 20 kW laboratory scale bubbling fluidized bed gasifier (BFBG) was used for the agglomeration experiments. The system consists of a fluidized bed reactor, a fuel feeding system, a set of pressure transducers and thermocouples, a data acquisition unit and a gas analysis system. A more detailed description of the experimental set-up and procedure is described by Jaiswal et al. [30] and Jakobsen et al. [31]. A schematic of the BFBG system is shown in Fig. 2. The reactor is made of a high temperature resistant steel column with an inner diameter of 10 cm and a height of 100 cm. To achieve necessary operating temperatures and to obtain isothermal bed conditions, the reactor is equipped with three electrical wall-heating elements. The gasification agent is preheated air that flows through two pipes from the bottom of the reactor. The airflow is controlled by a BROOK precision mass flowmeter. The biomass is fed to the reactor by a screw feeder positioned 21.2 cm above the air inlet. The bed pressures and temperatures are measured at five different locations along the height of the reactor. The temperature and pressure operating conditions are continuously recorded during the test runs and are logged to a computer by the LabVIEW data acquisition unit. For the present experiments, the sensors T2/P2 were used, which are located in the bed 14.3 cm above the air inlet [31]. The syngas leaves the reactor from the top.

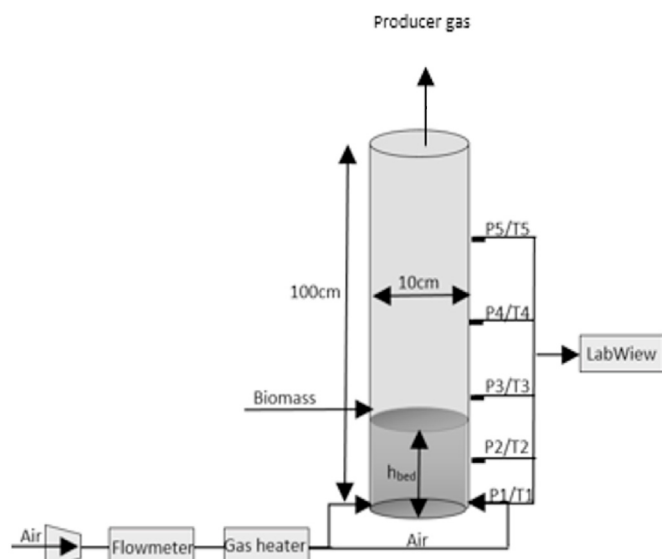


Fig. 2. Schematic of the 20 kW BFBG system used in the experiments.

2.2. Experimental procedure

The BFBG was operated under controlled bed conditions. The bed material used was quartz sand with particle density 2650 kg/m³. The particle diameter varied from 200 μm to 600 μm. The sand contained more than 80% SiO₂ by weight, and only smaller amounts of other mineral impurities. The chemical composition of the sand particles is given in Table 2. More details about the bed material can be found from previous work [32].

The initial mass of bed materials was 2.4 kg for all experiment. To ensure good fluidization conditions in the bed during the gasification process, the fluidization ratio (superficial air velocity/minimum fluidization velocity) was higher than 2.5 for all experiments. The mass flow rate of air supplied to the reactor is measured with a BROOK air flowmeter. The biomass pellets were conveyed to the reactor via a cold screw attached to the silo and a hot screw connected to the reactor. Prior to the experiments, the feed rates of the different biomasses were calibrated by measuring the average flow rates at specified motor speeds. The fuel feed rates were adjusted to 2.7 kg/h and 2.4 kg/h for the grass pellets and the wood pellets, respectively. The operating bed temperature was maintained between 700 °C and 900 °C. During all the experiments, the formation of agglomerates was provoked. The experiments were run continuously until the fluidized bed collapsed as a consequence of de-fluidized conditions.

2.3. SEM-EDS analyses

Representative agglomerates discharged from the gasifier were selected and examined by scanning electron microscopy (SEM, Zeiss SUPRA 55-VP), equipped with energy dispersive X-ray spectroscopy. The agglomerates were mounted on a carbon tape and directly scanned using the SEM. After scanning, the agglomerates were embedded in epoxy resin before being cut and polished for

Table 2
Chemical composition of the bed material [32].

Oxide	SiO ₂	Al ₂ O ₃	K ₂ O	Na ₂ O	Fe ₂ O ₃	CaO	MgO	Ti ₂ O
Fraction (wt %)	83.6	7.8	2.5	2.3	1.5	1.5	0.5	0.2

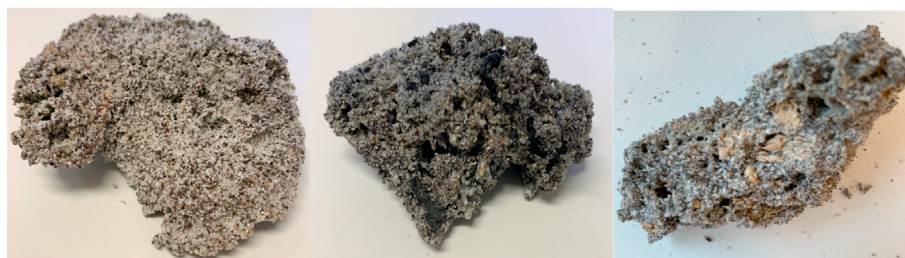


Fig. 3. Agglomerates formed during bubbling fluidized bed gasification of grass pellets at (a) $< 750\text{ }^\circ\text{C}</math>, (b) $700\text{--}800\text{ }^\circ\text{C}</math> and (c) $800\text{--}900\text{ }^\circ\text{C}</math>.$$$

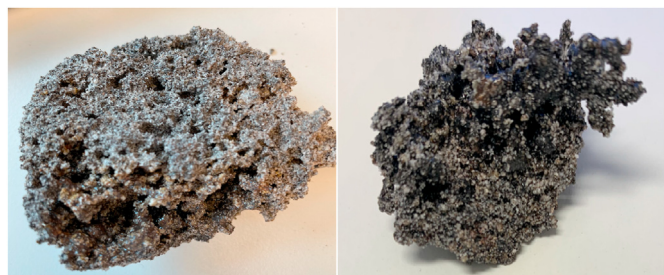


Fig. 4. Agglomerates formed during bubbling fluidized bed gasification wood pellets at (a) $700\text{--}800\text{ }^\circ\text{C}</math> and (b) $800\text{--}900\text{ }^\circ\text{C}</math>.$$

the SEM-EDS analyses of the cross-sectioned agglomerates. Back-scattered images were taken from one sample to give a better view of the distribution of the chemical elements.

3. Results and discussion

Figs. 3 and 4 pictures some of the agglomerates that were collected from the different gasification experiments of grass pellets and wood pellets. Visual examinations show that the agglomerates exhibit a hollow structure with large surface irregularities. The structures, sizes and shapes of the agglomerates vary widely depending on both the type of biomass and the process temperature. The agglomerates from grass pellets (Fig. 3) are typically larger in size and characterized with ash coated sand particles that are packed loosely together. These agglomerates have a more porous structure compared to the agglomerates from wood pellets, (Fig. 4), which tend to have higher degree of ash sticking and melting on the surface of the sand particles. In addition, the fractions of molten areas in the agglomerates formed from grass pellets tend to increase with higher gasification temperatures. After the gasification of grass pellets in the temperature range $800\text{--}900\text{ }^\circ\text{C}</math>, residual solid$

carbon from the biomass was also observed in a number of samples. This indicates that the ash melting process has taken place during the biomass conversion process in the gasifier.

During the gasification processes, the bed operating temperature and pressure were measured continuously by the set of thermocouples and pressure transducers (T2/P2) installed in the bed of the gasifier. Fig. 5 shows the bed temperature and pressure profiles from the agglomerated bed conditions during gasification of grass pellets and wood pellets at $800\text{ }^\circ\text{C}</math> and $900\text{ }^\circ\text{C}</math>, respectively. The bed temperature and pressure profiles are useful indicators for an early detection of bed agglomeration and for identifying the process conditions where the bed tends to de-fluidize. Previous studies [20,33–37] have reported that formation of agglomerates leads to increased local bed voidage and a subsequently decrease in the fluid flow resistance through the bed. It is often related to the occurrence of “hot-spots” due to combination of temporary fuel feed fluctuations and intensive burning of char residues. It leads to significantly high local temperature over the given operational temperature of $700\text{--}870\text{ }^\circ\text{C}</math> and subsequently intensive melting of ash and formation of large agglomerates. This can cause temporary gas channeling and localized fluidization disturbances. The restricted fluid flow causes bed disturbances with air channelling and de-fluidized zones in the bed. These zones create randomly formed hot spots, and thus a non-uniform temperature distribution that are recognized as large fluctuations in the bed temperature and pressure profiles [31,38]. The presence of complete de-fluidization is detected as a sudden increase in the bed temperature and a decrease in the bed pressure.$$$

The de-fluidized bed conditions are summarized in Table 3. The critical amount of accumulated ash in the bed is calculated based on the biomass ash content, the fuel feed rate and the onset of de-fluidization. The theoretical mass ratio of accumulated ash/bed material is introduced to provide a realistic comparison of the agglomeration tendency for the two different biomasses. The theoretical ash/bed material ratio is expressed as a weight

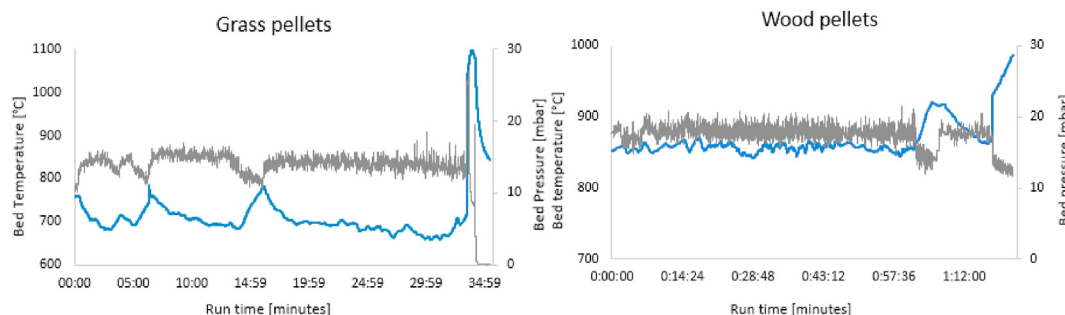


Fig. 5. Bed pressure (grey line) and temperature profile (blue line) observed for de-fluidized bed conditions for (a) gasification of grass pellets in the temperature range $700\text{--}800\text{ }^\circ\text{C}</math>, and (b) wood pellets in the temperature range $800\text{--}900\text{ }^\circ\text{C}</math>.$$

Table 3
Observations of de-fluidization and agglomeration for grass pellets and wood pellets.

	Fuel feed rate [kg/h]	Fluidization ratio	De-fluidization temperature [°C]	Onset of de-fluidization [minutes]	Critical amount of ash ^a [g]	Theoretical ash/bed material [wt %]
Grass	2.7	>2.5	698 (<750)	56	240	10
			750 (700–800)	29	124	5
			898 (800–900)	14	60	3
Wood	2.4	>2.5	815 (700–800)	153	37	1.5
			910 (800–900)	77	19	0.8

^a Assuming no ash leaves the reactor together with the produced gas.

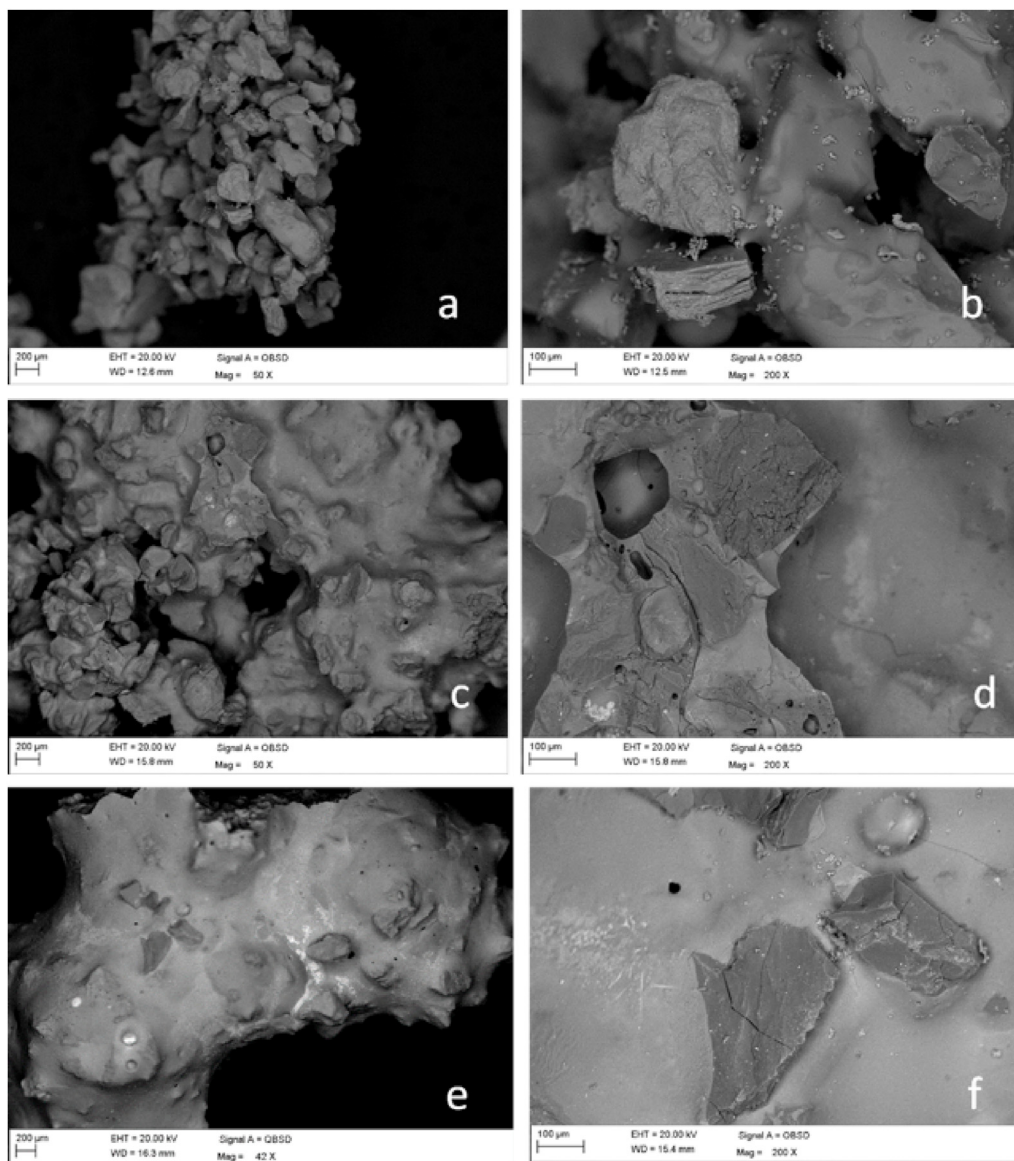


Fig. 6. SEM micrographs at different magnifications of (a) an agglomerate from gasification of grass pellet with de-fluidization temperature at 686 °C, (b) details of agglomerated grains, (c) an agglomerate from gasification of grass pellet with de-fluidization temperature at 750 °C, (d) details of broken part of the agglomerate, (e) an agglomerate from gasification of grass pellet with de-fluidization temperature at 898 °C, and (f) details of the external surface with sand grains embedded in melted fraction.

percentage of accumulated ash in relation to 2.4 kg initial mass of bed material and provides useful knowledge of the time when agglomerates are formed during the gasification process. Based on this value, the recirculation rate of the bed material can be

calculated in order to avoid bed agglomeration and de-fluidization. On the other hand, for the design and operation of the fluidized bed gasifier for converting ash rich biomass fuels, low operation temperature has been proposed and tested. As reported in previous

work, reducing the operational temperature to an acceptable low value can be an efficient way to mitigate bed agglomeration in order to 1) restrain and avoid migration and release of ash forming elements from the fuel, which further react with each other and the bed material (i.e., silica sand), leading to operational problems related to sintering and agglomeration, 2) reduce accumulation rate of large amounts of inorganic material from the fuel in the bed, and simultaneously by recirculating the bed materials and allowing for more mechanical ash particle downsizing as an effect of prolonged retention times [39].

The findings from the gasification experiments suggest that agglomeration of grass pellets occurs after shorter time and at lower temperatures compared to wood pellets. However, the critical amount of ash in the bed is significantly lower for the wood pellets than for the grass pellets, which indicate that the agglomeration process for grass and wood involves different mechanisms as well as different ash melting behaviour.

3.1. SEM-EDS analyses

At the end of each gasification experiment, agglomerates were discharged together with the bed material and the ash residues for further analyses. Fig. 6(a and b) show the SEM images of an agglomerate discharged from the gasification of grass pellets after de-fluidization at temperature $<750^{\circ}\text{C}$. Fig. 6(a) clearly shows that sand particles agglomerate with a rather loose structure, while in Fig. 6(b) it can be seen that partially molten ash covers the surface of the sand particles that have an intact and smooth surface. The sand particles covered with molten ash are attached to one another. Fig. 6(c-d) show an agglomerate collected from gasification of grass pellets after de-fluidization in the temperature range between 700°C

and 800°C . The agglomerate appears as melted and re-solidified. In comparison to Fig. 6(a), it is rather difficult to identify the individual sand particles that are mainly embedded in a fused fraction, as seen in Fig. 6(d). Fig. 6(e – f) show an agglomerate that was discharged from the gasification experiments in the temperature range between 800°C and 900°C . The scanned agglomerate has clearly gone through significant melting and fuse, resulting in formation of a dense and compact structure. This observation emphasizes the crucial role of the temperature for the formation tendency and the severity of agglomeration as stated by Scala et al. [40].

Fig. 7(a–d) show the SEM micrographs of agglomerates discharged from the bed after de-fluidization during gasification of wood pellet in the temperature ranges between 700°C and 800°C , and 800°C and 900°C s. For both operating temperature conditions, the agglomerates exhibit a dense structure and an intact surface. This indicates that an intensive fuse of the ash from wood pellets causes severe agglomeration, sintering and final embedding of sand particles into melted fractions.

The elemental compositions at selected locations of the different agglomerates were determined by EDS spot analyses. For identifying of the exact spots for the EDS analyses, the chosen locations are marked with numbers in the SEM images presented in Figs. 8 and 9. The results of the EDS spot analyses are reported in the same figures. For agglomerates discharged from the gasification experiments of grass pellets (Fig. 8), the predominant elements on the surface are Si, Ca, K and Al while other elements such as Na and Mg exist in varying amount. Further can P and Fe be found to a lesser extent in some of the EDS spots. Several areas of the agglomerates appear as a smooth and clean surface, where sand particles are completely embedded in a fused layer of ash. Extensive melting of ash on the surface is clearly visible in some of the EDS

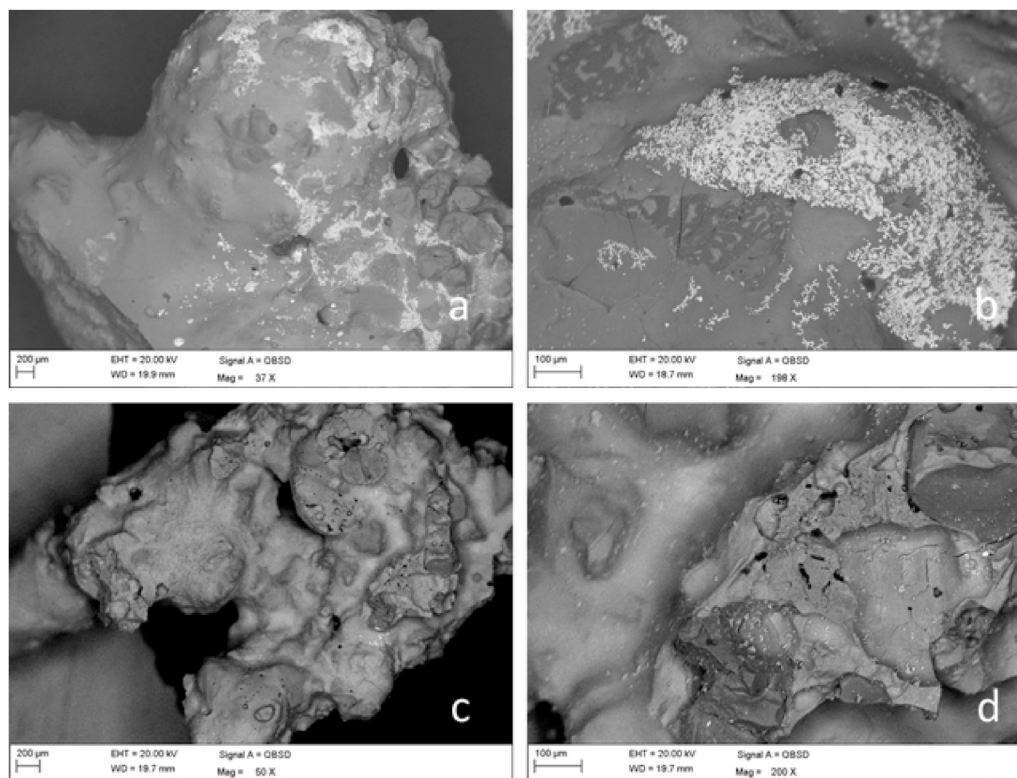


Fig. 7. SEM micrographs at different magnifications of (a) an agglomerate from gasification of wood pellet with de-fluidization temperature at 815°C , (b) details of the external surface, (c) an agglomerate from gasification of wood pellet with de-fluidization temperature at 910°C , and (d) details of broken part of the agglomerate.

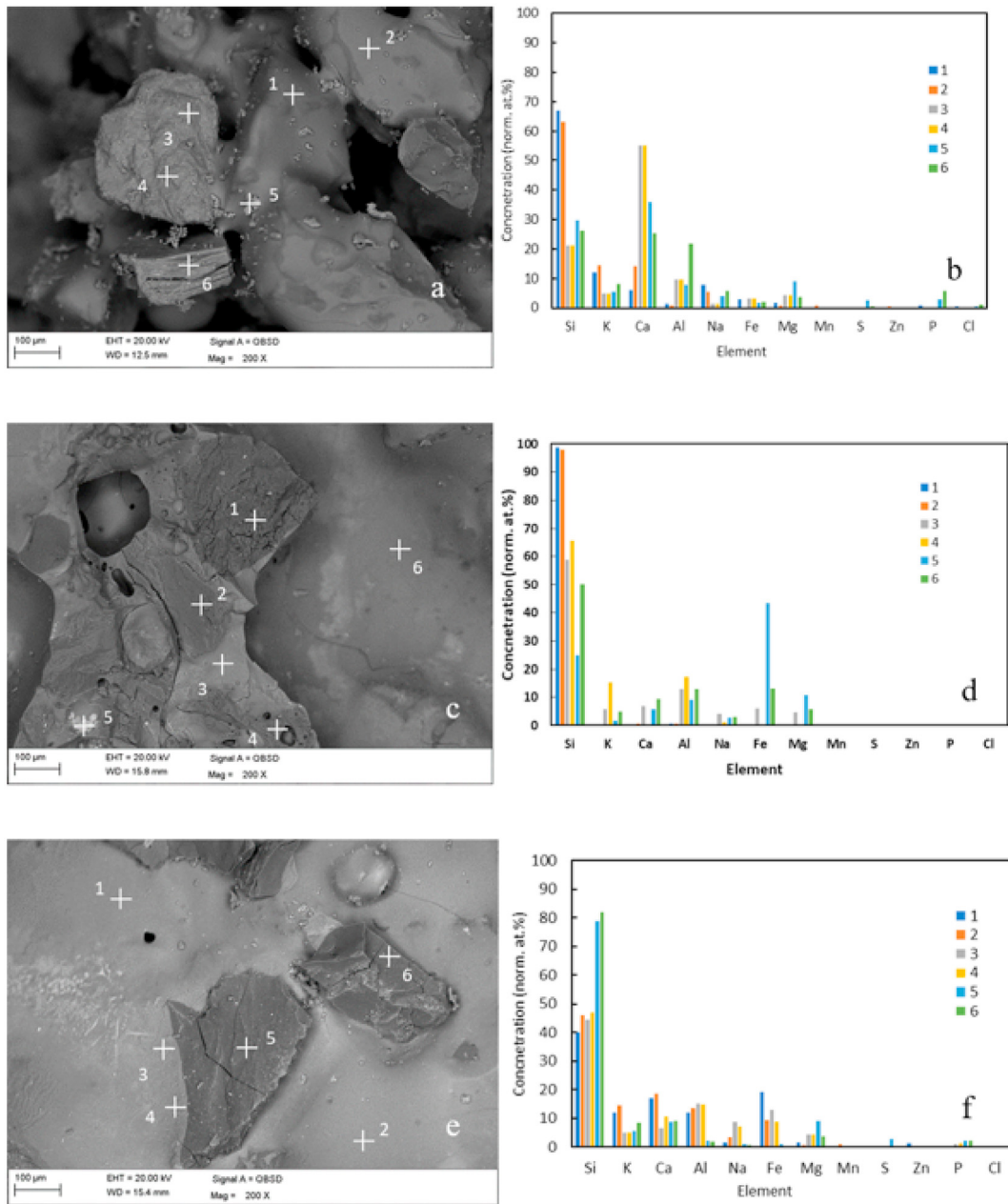


Fig. 8. SEM-EDS analysis agglomerate from gasification of grass pellet with de-fluidization temperature at 686 °C ((a) and (b)), at 750 °C ((c) and (d)) and at 898 °C ((e) and (f)).

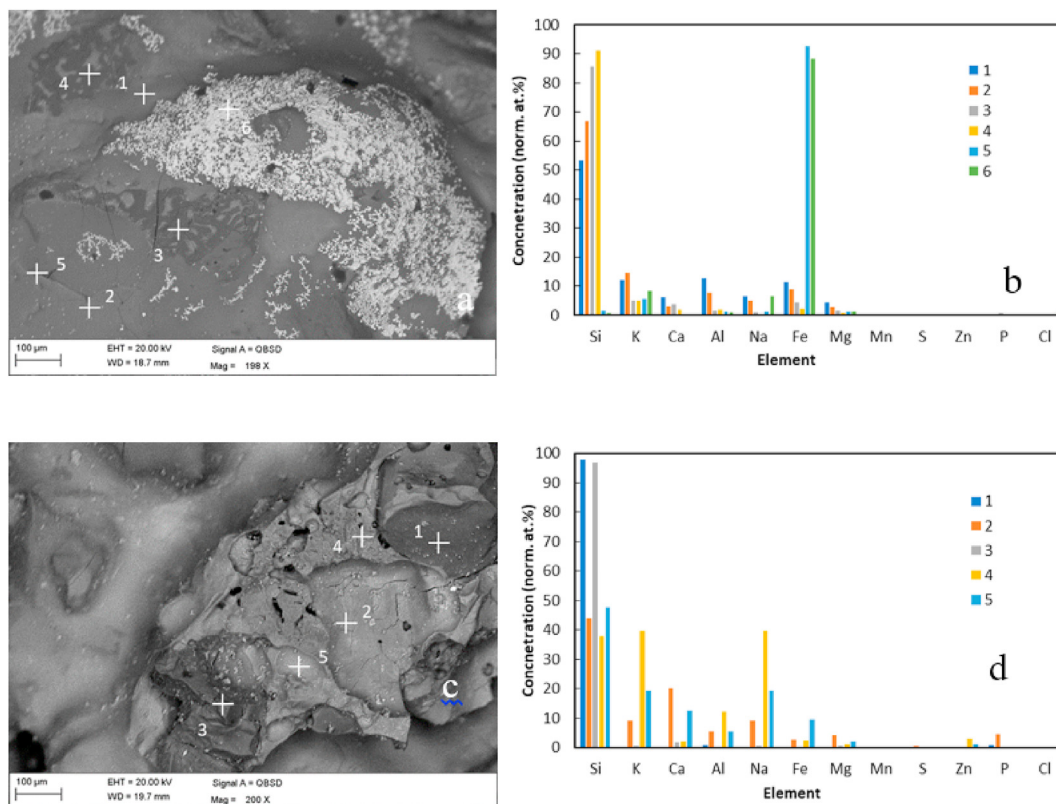


Fig. 9. SEM-EDS analysis on agglomerate from gasification of wood pellet with de-fluidization temperature at (a) 815 °C and (b) 910 °C.

spots and suggest the surface to be a layer of solidified molten ash. This can be clearly seen in the SEM micrographs in Fig. 8. Fig. 8 (a) shows the presence of finer particles glued to the smooth surface, which may be a result of the deposition of non-molten ash particles.

The data from the EDS analyses representing the agglomerates discharged from the gasification experiments of wood pellets (Fig. 9) show that the agglomerate surfaces are enriched in Si and K. The ash-forming elements Ca, Na and Fe are present to very varying degrees, depending on which spot is being examined. Only small amounts of Al and Mg are observed on the surface of the agglomerates.

The present SEM characteristics and EDS data presented in Figs. 8 and 9, illustrate the agglomeration phenomena in varying degrees. The analyses indicate that there are large differences in structure according to type of fuel, as well as differences within agglomerates derived from the same fuel. Large variations are also seen in the presence of the different ash forming elements. From the SEM-images, it is clearly visible how the majority of the agglomerates contain a large number of particles clustered together in porous lumps and with a rough surface structure. The surface irregularities make the images become more blurred and lighter in colour at the points where the surface has several height differences.

Figs. 10 and 11 show the SEM images and the corresponding EDS analyses of cross-sectioned agglomerates formed during the gasification of grass pellets and wood pellets. The SEM-EDS analyses were carried out to reveal the different agglomeration mechanisms. The results clearly show that there are two distinguished zones, indicated by dark grey color and light grey color. The EDS analyses show that the dark grey zones are sand particles with Si as the main component, and that the light grey zones are sand particles dominated by Si, K, Ca and Al. The sand particles are surrounded by visible

molten ash (black color). The three most abundant elements in agglomerates from grass pellets are Si, K and Ca. On the other hand, the composition of the molten fraction of the agglomerates from wood pellets are more complex, where Si, K, Ca, Al and Na are identified as the main elements. These observations are in good agreement with the initial fuel ash compositions shown in Fig. 1. The SEM-EDS results revealed that both the gasification temperature and the ash composition influence on the agglomeration mechanisms and consequently the severity of the agglomerate formation.

During the gasification process, the transformation chemistry of the grass and wood ash can be considerably different with considering absolute content of total ash and relative concentration of critical ash forming elements like Si, K and Ca. The ash content of grass pellet is 9.5 wt % that is around 15 times higher than that of wood pellets. It indicates that more ash will be formed during conversion of grass pellets with given time and fuel feeding rate. The comparisons of the biomass ashes and the agglomerates in Fig. 12 show that the ash from both wood pellets and grass pellets are rich in Si, K, Ca. However, the wood pellet ash contains around 3 times higher amount of Ca than grass pellet ash. Whereas the grass pellet ash contains two times here higher K than that of wood pellet ash. With considering large amount of Si and K of grass pellets, low temperature melting K-silicates might preferably form during conversion, leading to melt-introduced bed agglomeration. On the other hand, more Ca–K–silicates might form during conversion of the wood pellets, which often have higher melting temperatures. This assumption is approved by ash melting tests on the studied grass and wood pellet ash. The initial ash melting temperature of the studied grass pellets ash is about 1026 °C, whereas the initial ash melting temperature of wood ash is 1283 °C [41]. Similar findings have been reported in literatures with focusing on ash sintering and agglomeration behaviours of herbaceous and woody biomasses [42,43]. The agglomerates collected from gasification of

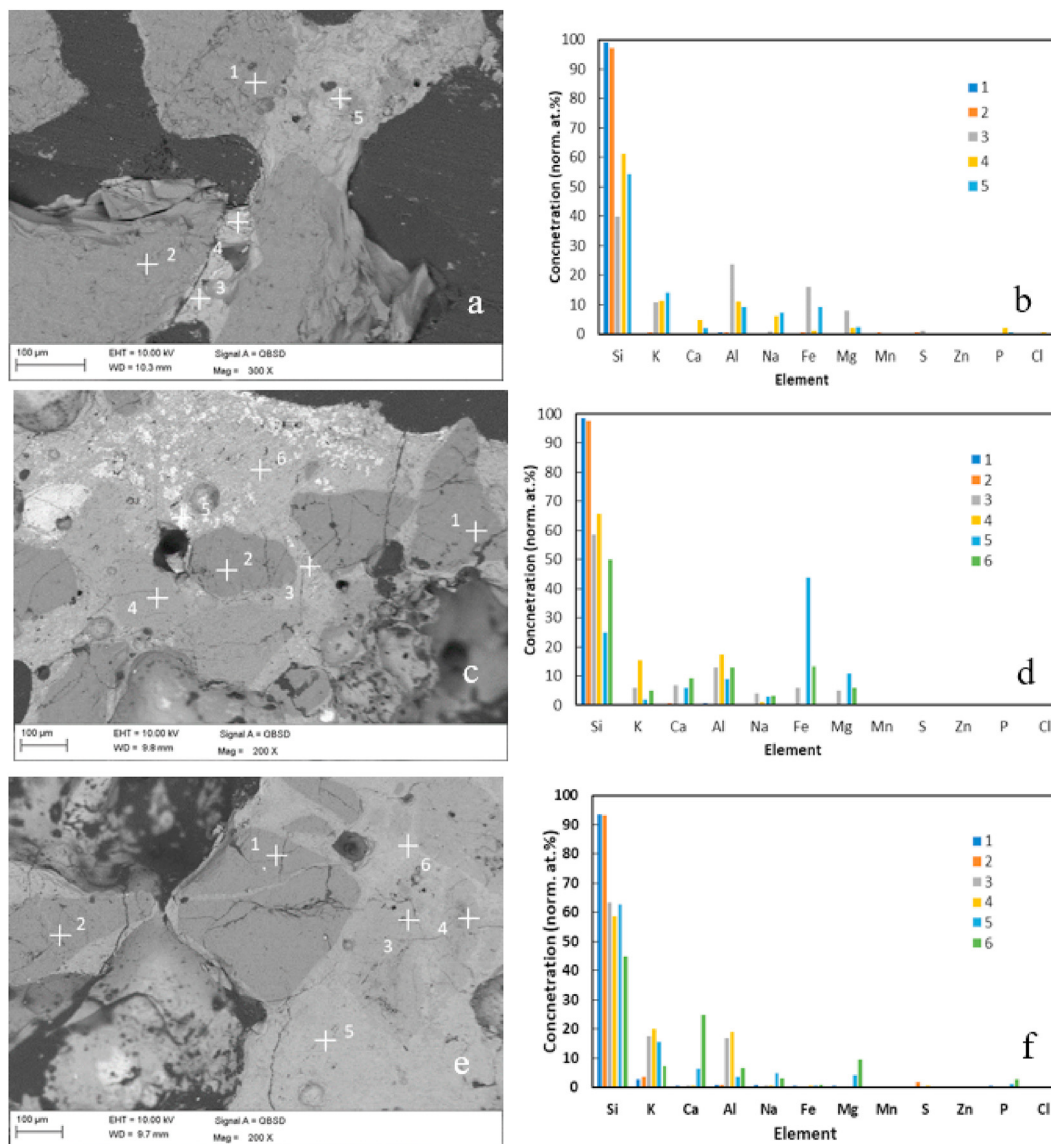


Fig. 10. SEM-EDS analysis of an agglomerate from gasification of grass pellet with de-fluidization temperature at 686 °C ((a) and (b)), at 750 °C ((c) and (d)) and at 898 °C ((e) and (f)).

grass pellets have evidently large content of K and high content of Si, indicating formation of K-silicates as discussed in section 3-1. On the other hand, the agglomerates from gasification of wood pellets contains mainly Si, Ca, Al and minor amounts of K, implying presence of K–Ca/Al-silicates. Fig. 12 shows that chemical composition and formation mechanisms of agglomerates during biomass gasification process can considerably related to ash content and composition of initial fuel.

4. Conclusions

In the present work, agglomeration of bed particles during fluidized bed gasification of grass pellets and wood pellets was studied. The chemical analyses of the fuels show significant differences in the distribution of the ash-forming elements in the fuels. High Si, high K and low Ca content characterized the grass pellets, while wood pellets are rich in Ca.

For both grass pellets and wood pellets, agglomerates were found after ended experiments. The pressure and temperature profiles indicated degree of bed disturbance due to ash

agglomeration for both fuels. Agglomeration conditions appears as instabilities in the bed profiles with a sudden change in temperature and/or bed pressure. The observations of the de-fluidized beds suggest that compared to wood, agglomeration of grass pellets occurs after shorter de-fluidization time and at lower de-fluidization temperature. The findings also show that the critical amount of accumulated ash in the bed is higher for the grass pellets than the wood pellets, indicating that different mechanisms as well as ash melting behaviour are involved in the agglomeration processes. The critical amount of accumulated ash varied from 3 to 10% by weight for the grass pellets and from 0.8 to 1.5% by weight for the wood pellets during the gasification experiments.

The agglomerates examined by SEM showed a hollow structure, with several areas that are apparent to be fused and re-solidified. An EDS detector was used to analyse the compositions of elements on the surface of the bed materials and the agglomerates. The results showed that the layer on the bed particles was dominated by Si and Al from the bed material and Si, K and Ca from the biomass.

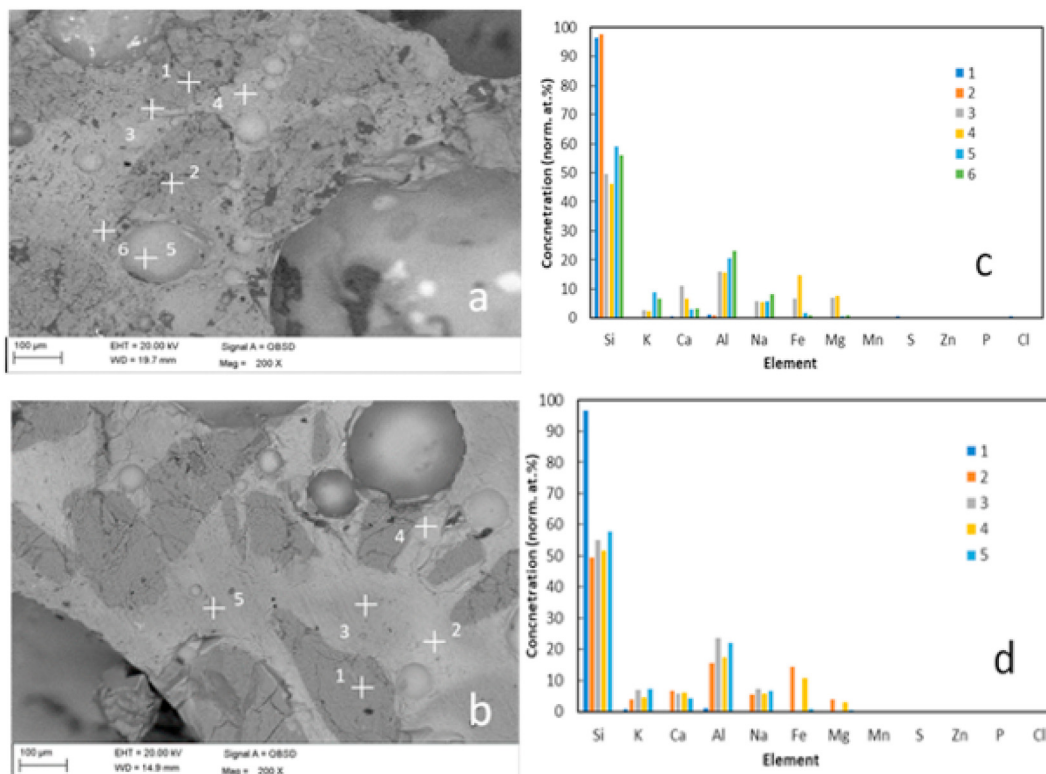


Fig. 11. SEM-EDS analysis an agglomerate from gasification of wood pellet with de-fluidization temperature at 815 °C ((a) and (c)) and at 910 °C ((b) and (d)).

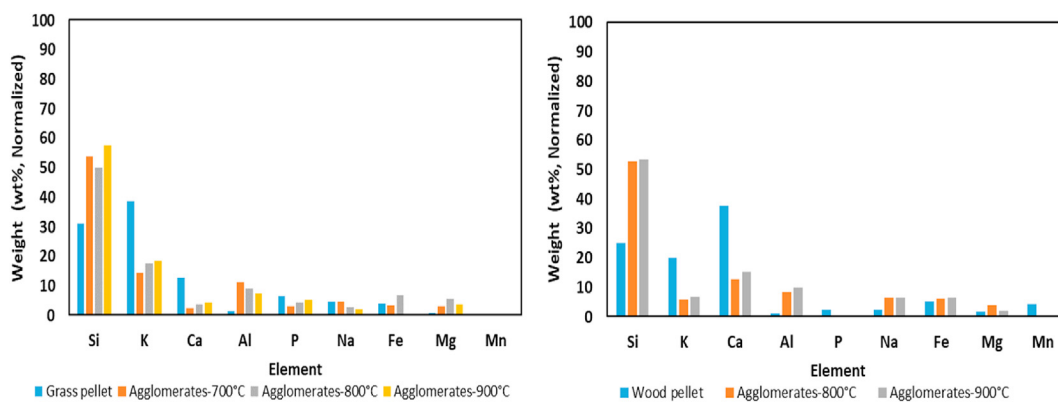


Fig. 12. Comparison of elements in fuel ash and agglomerates from grass pellets (left) and wood pellets (right).

Credit author statement

Nora Furuvik: Conceptualization, Methodology, Writing – Original draft preparation. **Rajan Jaiswal:** Methodology, Investigation, Writing – Reviewing and editing. **Lian Wang:** Conceptualization, Methodology, Investigation, Writing – Reviewing and editing. **Rajan Thapa:** Resources, Supervision, Writing – Reviewing and editing. **Marianne Eikeland:** Resources, Supervision, Writing – Reviewing and editing. **Britt Moldestad:** Conceptualization, Supervision, Resources, Writing – Reviewing and editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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