Full Length Article

Role of surface morphology in bed particle layer formation on quartz bed particles in fluidized bed combustion of woody biomass

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A R T I C L E  I N F O

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A B S T R A C T

The influence of quartz bed particle surface morphology on the bed particle layer and crack layer formation process in fluidized bed combustion of woody biomass was investigated in this work. Bed material samples were collected at different sampling times from the startup with a fresh bed in industrial scale bubbling fluidized bed (BFB) and circulating fluidized bed (CFB) boilers, both utilizing woody biomass. X-ray microtomography (XMT) and scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS) were employed to characterize bed particle layers and crack layers in the samples. Results showed that there is a noticeable difference between the bed layer characteristics over the so-called “concave” and “convex”-shaped morphologies on the bed particle surface with respect to layer formation. The concave areas are mainly covered with a thin inner layer, whilst the convex display a comparably thick inner layer and an outer layer. In addition, 3D images of the particles revealed that the crack layers mainly originate from concave areas where the particle is less protected by an outer bed particle layer in conjunction with cracks in the inner layer.

1. Introduction

Biomasses comprise a wide variety of organic-based fuels which provide renewable energy sources [1–3]. In the boreal region of the world, woody biomasses are prevalent biomass fuels in the energy sector due to the abundance of forest residues [4–8]. Amongst current processes for energy conversion of biomass fuels, fluidized bed combustion (FBC) is one of the most favorable technologies. It provides high combustion efficiency, emission control, and low process temperature [9–12]. However, bed agglomeration is a potential operational challenge in FBC which increases maintenance costs and might cause unplanned process shutdown in severe cases. Moreover, fine bed particle fragments could deposit on furnace walls and in return legs which raises further maintenance and operational concerns [13,14].

Extensive research has investigated the chemical interaction of different bed materials with biomass ash to elucidate mechanisms responsible for bed agglomeration and bed particle fragmentation [13,15–22]. Studies have shown that bed particle layer formation is one of the prevalent agglomeration mechanisms, ordinarily initiated by the formation of an adhesive layer on the bed particles [21,23]. For woody biomass fuels which are typically characterized by high Ca and K content, formation of two layers (i.e., inner and outer layer) with distinct properties and compositions have been identified in the literature for several types of bed particles [24]. In general, the inner layer is homogeneous and is dominated by the main components of the fuel ash and bed material. On the contrary, the outermost layer is a heterogeneous mixture of fine ash particles and accordingly has a composition similar to that of the fuel ash [21,25].

Natural sand, majorly comprising quartz, is commonly used in FBC as it is widely available [21]. In case of utilizing woody biomass as fuel, bed particle layer formation is regulated by the interaction between the two primary constituents of these type of fuels (i.e., K and Ca) and the bed particles [26]. Fate of the remaining constituent elements within the fuel ash is beyond the scope of this study and could be found elsewhere [17,27]. Accordingly, bed layer formation on quartz bed particles is initiated through condensation of gaseous K-species (e.g., KOH), and/or direct reaction of gaseous alkali with the bed material [20,23]. The reaction between gaseous alkali and quartz bed particle surface is relatively fast and occurs in the very first stages of the process [21]. This leads to the formation of a sticky K-rich silicate melt on the bed particle surface. However, since Ca-silicate is thermodynamically more stable at the process temperature compared to K-silicate, Ca from coarse ash...
particles diffuse into the melt, replaces K in the silicate structure and gradually develops the inner layer [17,21]. Increased Ca/K ratio in the inner layer composition generally increases its melting temperature, which reduces the diffusion rate and consequently decreases the growth rate of the inner layer [21,26]. As a consequence, the inner layer serves as a protective layer for the bed particle towards further reaction with alkali. Yet, alkali can still react with quartz bed particles in the proximity of relatively thin inner layers or cracks. Therefore, in these regions a wider area dominated by Si, K and Ca can be identified and it is referred to as the inner-inner layer [26].

Whereas the inner and inner-inner layers are formed through chemical reaction between the ash forming elements and the bed particle, the outer layer is formed by deposition of coarse ash particles onto the bed particle surface and provides an auxiliary source of Ca for the inner layer. Upon further transport of Ca via solid phase diffusion from the outer layer to the bed particle surface, more Ca-rich silicates, namely Ca$_2$SiO$_4$ and Ca$_2$SiO$_5$, start to form in the inner layer [21,26]. Formation of calcium silicates with higher number of Ca in their chemical structure, introduces microstructural stress which produces inward cracks in the inner layer. These cracks potentially provide new routes for K-species to reach and react with the bed core and consequently originate crack layers [14]. Crack layers have similar composition to the inner-inner layer and accordingly exhibit the same appearance in SEM images [28]. The crack layers can spread through the bed particle, connect together, and eventually break down the bed particle into smaller fragments [13,14,29]. The fragments are covered with the sticky K-rich silicate which can increase the bed agglomeration risk. Furthermore, entrainment of these sticky finer particles might cause deposition in cyclones, furnace walls and return legs which consequently impose additional maintenance costs [13,16]. Therefore, complete bed replacement on a weekly basis has been recommended in order to avoid aforementioned issues [14].

Recent studies have shown that along with the chemical composition of bed material, surface morphology of the bed particle could also alter the bed particle layer characteristics [28,30]. A previous study on quartz bed particles utilized in fluidized bed combustion of woody biomass revealed surface irregularities between the inner layer and quartz bed particle surface [14]. In a recent work, non-uniformity in bed particle layer thickness was observed at different surface morphologies on K-feldspar and olivine bed particles utilized in a dual fluidized bed gasifier [30]. Moreover, it was noticed that chemical composition of the bed particle layer differs at convex and concave shaped areas on the bed particle surface. In another study on quartz and K-feldspar bed particles taken from two different types of fluidized bed combustion units utilizing woody biomass, signs of different tendencies towards bed particle layer formation at different regions on the bed particle surface were reported [28].

Inertial impaction serves as the primary mechanism responsible for conveying coarse ash particles to solid surfaces in combustion [31]. Capture efficiency of the bed particles portrays the tendency of these particles to remain adhered to the solid surface following their impact [32,33]. Studies have illustrated that the rate of inertial impaction and the capture efficiency of the ash particles during the ash deposit formation process are significantly affected by the gas flow properties, geometry of the target surface and its angular position during the impact. [31]. This means that certain regions/morphologies on the bed particle surface could provide a higher possibility for the impaction of Ca-rich ash particles striking the bed surface, which may affect the bed particle layer formation process. Therefore, alongside the chemical reactions involved in the fuel ash/bed particle system, surface morphology might be taken into consideration to better understand the bed particle layer and crack layer formation process.

The main objective of this study is to investigate the role of surface morphology in bed particle layer characteristics, i.e., layer thickness and composition, for quartz bed particles in fluidized bed combustion of woody biomass. Moreover, a detailed study of the crack layer formation process and its association with surface specifications of the bed particle is also aimed in this work. Results from this study can provide a better understanding of the bed particle layer and the crack layer formation process in association with the surface morphology of the bed particles. This could be valuable in selecting bed particles for FBC reactors with appropriate particle morphologies that may reduce the risk for bed agglomeration and bed particle fragmentation.

2. Materials and methods

Quartz bed particles from a full-scale 30MW$_{th}$ bubbling fluidized bed (BFB$_{30}$) and a full-scale 90MW$_{th}$ circulating fluidized bed (CFB$_{90}$) were studied in this work. The bed materials utilized in each conversion process were taken from a different mineral deposit in Sweden. Bed particle samples were collected at different time intervals after complete bed change. The two reactors were operated with a typical woody biomass mixture and, consequently, similar fuel ash composition. Bed particles acquired at different ages were analyzed via SEM/EDS and X-ray microtomography in order to investigate the bed particle layer and the crack layer formation processes over the operation time.

2.1. Materials

2.1.1. Bed materials sampling from the full-scale BFB$_{30}$

The boiler utilized a typical softwood-based woody biomass mixture (10% bark, 30% logging residues, 40% wood chips) as fuel and was fed with 20 tons of natural sand (80% quartz and the remainder mainly consisting of feldspar microcline and albite) as bed material with a bed removal rate of less than 3 wt%. About 1 kg of bed particle samples were collected at 1, 3, 5, 13 and 23 days after complete bed change.

2.1.2. Bed materials sampling from the full-scale CFB$_{90}$

The boiler was operated with softwood sawdust as fuel and natural sand (~80% quartz and the remainder consisting of feldspar and smaller shares of mica) as bed materials and 10 wt% bed removal rate. Bed particle samples (~1 kg) were collected at 1, 5, and 13 days after complete bed change.

Sampling intervals for the two processes were selected based on the previous observations on the bed particle development over time in the two conversion processes [21]. Information regarding the bed materials, fuel ash compositions, and operating conditions from the sampling campaigns are given in Table 1 and Table 2. More details on the operating conditions during the sampling campaigns can be found elsewhere [14].

2.2. Analysis techniques

2.2.1. Scanning electron microscopy (SEM) / energy dispersive spectroscopy (EDS) analysis

Bed materials acquired under normal operating conditions from both conversion processes were sieved to the typical bed particle size of the commercial CFB and BFB boilers (i.e., bed particle size between 200 and 500 μm and 500–850 μm, respectively). In order to prepare the samples for SEM/EDS analysis, bed particles from each process were encased in epoxy resin blocks which were dry-polished afterward to expose cross-sections of the bed particles. The cross-sections were then analyzed via scanning electron microscope (SEM) using a JSM-IT300 (JEOL, Japan) equipped with a backscattered electron detector (BSE) operated at low-vacuum mode (100 Pa). Elemental analysis of the bed particle layers was conducted using a X-Max 80 energy-dispersive X-ray spectroscopy detector (EDS; Oxford Instruments, UK) with the same SEM. 10 quartz bed particles were examined via SEM/EDS analysis for each bed sample. Given the continuous replacement of a small fraction of the bed material, only those particles with the largest cross-sections and with the thickest inner layers were chosen to ensure the representativeness of the selected bed particles for each sample age. 4–5 EDS spots were evenly
spread around the particle for elemental analysis of the outer layer, the inner layer, and the inner-inner layer. To minimize the errors associated with the spatial resolution, EDS spots were selected at the middle of the bed particle layers. 2–3 spots at the middle of crack layers were also subjected to EDS analysis for each bed particle.

2.2.2. X-ray microtomography (XMT) analysis

X-ray microtomography is a non-destructive imaging method where the result is a 3D anatomic map of the interior, based on the densities of the internal features of the material. Based on these results, visualizations and quantitative analysis of structure can be carried out on the full 3D data volume as well as on 2D cross-sections through the volumes, for direct comparison with SEM/BSE/EDS data. Quantitative analysis is carried out using dedicated software for 3D image analysis which allows analysis of for example volume fractions, size distributions and grain morphologies and facilitates volumetric analysis of the interaction between the ash forming elements and the bed material [28]. Areas in the sample that have higher densities (i.e., comprise heavier elements) exhibit higher attenuation than those containing lighter elements [35]. Analogous to SEM/BSE images, the higher the density, the brighter the area appears in the XMT images. However, unlike SEM/EDS, results from XMT analysis do not support any chemical compositional data from the sample. XMT images of the bed particles enables measurements of bed layer thickness around the entire particle (i.e., at different surface morphologies) and facilitates volumetric analysis of the interaction between the ash forming elements and the bed material [28].

In this work, a Zeiss Xradia 620 Versa XMT (Carl Zeiss X-ray Microscopy, Pleasanton, CA, USA) with a maximum spatial resolution of 0.5 μm and the minimum voxel size of 40 nm was used for X-ray microtomography analysis of quartz bed particles taken from the two conversion processes. Depending on the required information from each XMT scan, two sets of scanning were conducted at two different spatial resolutions. To measure the overall bed particle layer thickness (i.e., inner and outer layer) along with observing bed particle layer distribution at different surface morphologies, samples containing 150–250 bed particles taken at different bed material ages of the two processes were scanned at 3.5 μm spatial resolution of which around 40 typical quartz bed particles were selected and analyzed for each time interval. Another set of scans with higher resolution (1.3 μm for BFB and 1.0 μm for CFB) were executed for 1–2 typical particles from each sample to observe cracks in the inner layer and to study distribution of the inner and outer layer on the bed particle surface, separately.

To analyze the results, reconstructed XMT data were exported to Dragonfly Pro software (Version 2022.2 developed by ORS, Montreal, QC, Canada) for 3D visualization and quantitative analysis. Single-color maps with varying brightness were employed to show different phenomena (e.g., inner layer, outer layer) whereas the bed particle layer thickness as measured by XMT was visualized using color gradient images. More details on the scanning procedure, reconstruction method and data analysis utilized for examining quartz bed particles studied in this work could be found elsewhere [28].

3. Results and discussion

3.1. Bed particle layer characteristics

3.1.1. Nearly fresh bed particles

Fig. 1 exhibits XMT images of nearly fresh typical quartz bed particles and portrays the surface morphology of the bed materials utilized in the two studied conversion processes. Considering the continuous partial bed replacement in the two boilers, bed particles from the samples taken after 1 day with no bed layer were contemplated as nearly fresh ones. As evident in Fig. 1, the particle surface is not smooth but entails concaves and convexes. These two distinct surface morphologies were thoroughly scrutinized in this study in order to investigate potential variations in bed particle layer formation associated with different morphological characteristics on the bed particle surface. The bed particles utilized in the two conversion processes studied in this work originate from different sources. Accordingly, it could be observed that the surface morphology of the quartz bed particle taken from the BFB and the CFB boilers display considerably different properties. The former could be described as bed particles with a smoother surface and rounded edges, while more convex and concaves and an irregular surface texture could be observed on the latter.

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**Table 1**

<table>
<thead>
<tr>
<th>conversion process</th>
<th>primary airflow at full load (nm³/s)</th>
<th>bed cross-section (m³)</th>
<th>fluidization velocity (m/s)</th>
<th>bed weight (ton)</th>
<th>operating bed temperature (°C)</th>
<th>average bed particle size (μm)</th>
<th>bed consumption (w% of the bed/day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BFB 30 MW&lt;sub&gt;in&lt;/sub&gt;</td>
<td>5-6</td>
<td>−20</td>
<td>1.1</td>
<td>20</td>
<td>800–880</td>
<td>700</td>
<td>less than 3</td>
</tr>
<tr>
<td>CFB 90 MW&lt;sub&gt;in&lt;/sub&gt;</td>
<td>−12.5</td>
<td>−15</td>
<td>3.4</td>
<td>20</td>
<td>780–850</td>
<td>300</td>
<td>10</td>
</tr>
</tbody>
</table>

**Table 2**

<table>
<thead>
<tr>
<th>conversion process</th>
<th>ash content (% dry substance)</th>
<th>K</th>
<th>Na</th>
<th>Ca</th>
<th>Mg</th>
<th>Fe</th>
<th>Al</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cl</th>
</tr>
</thead>
<tbody>
<tr>
<td>BFB 1.8 ± 0.51</td>
<td>0.11</td>
<td>0.010</td>
<td>0.44</td>
<td>0.040</td>
<td>0.12</td>
<td>0.019</td>
<td>0.12</td>
<td>0.027</td>
<td>0.023</td>
<td>&lt;0.01</td>
<td></td>
</tr>
<tr>
<td>CFB 0.8 ± 0.15</td>
<td>0.076</td>
<td>0.001</td>
<td>0.293</td>
<td>0.032</td>
<td>0.011</td>
<td>0.015</td>
<td>0.036</td>
<td>0.019</td>
<td>0.013</td>
<td>&lt;0.01</td>
<td></td>
</tr>
</tbody>
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**Fig. 1.** XMT 3D image of nearly fresh quartz bed particles taken from the BFB and the CFB after 1 day from the complete bed change.
3.1.2. Aged bed particles in the process: bed particle layer definition and characteristics

Figs. 2 and 3 show backscattered SEM images from typical cross-sections of quartz bed particles taken from the BFB and the CFB boilers at different ages along with the corresponding 2D image from XMT scanning. Magnified SEM images of the bed particle layers are exhibited in Figs. 4 and 5 for the bed particles taken from the BFB and the CFB boilers, respectively. Comparing images from SEM and XMT shows a resemblance between the appearance of different regions in a bed particle in both characterization techniques. The evolution of the bed particle layer throughout the particle age could be simultaneously observed as a lighter periphery around and within the bed particles in both SEM and XMT pictures. However, given that the results from SEM/EDS contain compositional data, they could be used to aid identification of the bed particle layers depicted in XMT images.

Overall findings from the bed particle layer characteristics at
different ages observed in this work were in-line with the results from a previous study on the particles taken from the exact same bed samples [14,21]. At the early stages of the two conversion processes (day 1–3), formation of an initial layer was observed as a homogeneous brighter periphery around quartz bed particles (Fig. 2a,b, Fig. 3a). In particles older than 3 days, an outer layer was distinguished from the inner layer as a heterogeneous and slightly darker perimeter (Fig. 2c-e and Fig. 3b,c). In addition to the inner and outer layer, a so-called inner-inner layer was identified in both SEM and XMT images in the proximity of thin inner layers or cracks in the bed particle. Crack layers were also noticeable in the images from day 3 and deployed through the bed particles over time (Fig. 2c-e and 3b,c). Axial cracks in the inner layer could also be noticed in the particles of the same age which were proposed in previous studies to be the initiator of the crack layers [14].

Fig. 3. Backscattered SEM images (a1)-(c1) and 2D X-ray microtomography images (a2)-(c2), of typical cross-sections of quartz bed particles found in bed samples taken from the CFB at 1 (a), 5 (b), and 13 days (c) after complete bed change.

Fig. 4. Magnified backscattered SEM images of typical cross-sections of quartz bed particles found in bed samples taken from the BFB at 1 (a), 3 (b), 5 (c), 13 (d) and 23 days (e) after complete bed change, showing bed particle layers.
Elemental analysis of the bed particle layers and the crack layers (excluding oxygen) showed that, after 3 days the inner layer mainly comprised Si (41.0 ± 15.0 mol%) and Ca (47.4 ± 9.3 mol%) while the outer layer was primarily dominated by Ca (40.2 ± 10.3 mol%), Si (32.0 ± 13.0 mol%) and Mg (11.7 ± 6.0 mol%). Comparing the elemental analysis results over the particle age for both BFB and CFB samples illustrated that the Ca/K ratio increased over time due to substituting K with Ca in the inner layer in a counter diffusion process. Elemental analysis of the inner-inner layer showed that excluding oxygen, after 3 days these regions mainly comprised Si (70.2 ± 3.4 mol%), K (12.1 ± 1.6 mol%) and Ca (8.3 ± 2.7 mol%), with an increasing profile for K and Ca and a constant concentration of Si over time. Crack layers in quartz bed particles taken from both conversion processes were also found to be dominated by Si (71.5 ± 4.0 mol%), K (11.8 ± 1.5 mol%), Ca (7.2 ± 3.0 mol%), after 3 days from the complete bed change. These characteristics agree with previous findings [14,21]. Analogous composition of the crack layers and the inner-inner layer explains their near-identical appearance when observed with SEM/BSE as well as with XMT.

Fig. 5. Magnified Backscattered SEM images of typical cross-sections of quartz bed particles found in bed samples taken from the CFB at 1 (a), 5 (b), and 13 days (c) after complete bed change, showing bed particle layers.

(a)  
(b)  
(c)

Fig. 6. 3D XMT image of the overall bed particle layer thickness measurement for quartz bed particles taken from the BFB at 1 day (a), 3 days (b), 5 days (c), 13 days (d) and 23 days (e) after complete bed change.

(a)  
(b)  
(c)  
(d)  
(e)
3.1.3. Thickness distribution of the bed particle layer

XMT images in Fig. 6 show the overall bed particle layer thickness measurement for typical quartz bed particles taken from the BFB process at different ages. Fig. 7 shows the same information for quartz bed particles acquired from the CFB boiler. Monitoring all cross-sections attentively through XMT scanning for both conversion processes revealed that the initial layer is not homogeneously established over the whole particle surface. After 1 day from the startup, there were regions on the bed particle surface where the bed particle had not reacted with the fuel ash and consequently a bed particle layer had not been formed. Those areas could be noticed as black spots in Fig. 6a and Fig. 7a. It is also possible to observe these regions in Fig. 8 where the inner layer is separated from the bed particle core and is visualized in 3D for quartz bed particles taken after 1 day from the BFB and the CFB boilers. The red color in this figure shows the inner layer and the white regions (similar to the black spots in Fig. 6a and Fig. 7a) are parts of the bed particle surface where the bed particle layer has not been formed yet to an extent visible with XMT.

Following the layer development pattern over the bed particle age for both processes (Fig. 6b-e & Fig. 7b-c), it could be noticed that the layer thickness growth has not the same rate throughout the bed particle surface. The overall bed particle layer (i.e., inner and outer layer) is thicker at convex areas on the bed particle surface and comparatively thinner in concave regions. 2D XMT images also revealed that in both conversion processes, convex areas were covered both with the inner and the outer layer while concaves were mainly covered with the inner layer. In some concave areas, the inner layer was completely detached from the bed particle surface which is demarcated with yellow dashed circles in Fig. 9. This might be due to the higher microstructural stress in the tighter space within the concave regions. In addition, formation of cracks in the inner layer could facilitate detachment of the bed particle layer as it deteriorates the uniform structure of the inner layer and makes it vulnerable towards the external forces exerted on the bed surface (i.e., collision and erosion). In regions where the bed particle surface was relatively smooth, both inner and outer layers appeared to be uniformly distributed on the bed particle surface (Fig. 10). Considering the approximate size range of the ash particles (>1–3 μm), the eddy effect is not applicable in this case. Instead, it is likely that the deposition process is primarily governed by inertial impact of the coarse Ca-rich ash particles. Accordingly, the difference in the bed particle layer growth rate in concave and convex shaped regions could be contributed to the lower supply of the ash forming matter in the concave areas as the majority of the coarse ash particles are considerably large and consequently the ash deposition is much lower in these regions compared to that of the convex areas.

3.2. Crack layer characteristics

As shown in Fig. 2b-e and Fig. 3b-c, the crack layers were first observed in day 3 and gradually developed further and deployed through the bed particles over time. Nevertheless, the examination also revealed the presence of physical cracks in the bed particles, which may potentially stem from factors such as fluctuations in temperature throughout the process, erosion/collisions among bed particles, and the loading of bed materials.

Crack layers can split the bed particle into smaller fragments which cause deposition and produce operational problems in the downstream facilities. This could be contributed to the fact that alkali-silicate-rich crack layers tend to have less mechanical strength compared to the unreacted parts of the bed particle core. As a result, the particle becomes more vulnerable to fragmentation within the crack layers when exposed to the impact forces in the fluidized bed process. A typical quartz bed particle utilized in the BFB process for 23 days is utilized to demonstrate the degree of potential fragmentation in a single bed particle and to provide a scale for comparing fragments’ size with the original particle in Fig. 11. To do so, bed particle layers (inner and outer layer, inner-inner layer and crack layers) are segmented and removed from the bed particle. The bed particle and the unreacted parts are shown in Fig. 11 in citrine and teal colors, respectively.

In a previous work it was suggested that the crack layers are formed by diffusion of alkali species through the cracks in the inner layer [14]. To further develop this proposed mechanism for the crack layer formation, 3D images of quartz bed particles taken from both conversion processes were examined from different angles/cross sections to trace the path of the crack layers in the bed particle over time. Using both 3D images and 2D cross sections acquired from XMT analysis of quartz bed particles taken from the BFB and CFB process, it was noticed that the crack layers are mainly found in connection to the concave regions on the bed particle surface where the overall layer is thinner and/or the layer is detached from the bed surface (Fig. 12). These results suggest that cracks in the inner layer could bring the gaseous alkali in contact with the particle core only where the overall bed particle layer is still thin, and no outer Ca-rich protecting layer has been developed. Besides, as mentioned above, formation of the cracks in the inner layer could expedite the detachment of the inner layer in the surface concaves, leaving the bed particle unprotected against further reaction with gaseous alkali.

The phenomenon can be observed more clearly in Fig. 13 where the inner layer and the outer layer of a typical quartz bed particle taken after 23 days from the BFB boiler are segmented and shown separately along with a cross-sectional view of the same particle. As could be observed in Fig. 13a, in a convex area, cracks in the inner layer can potentially provide a route for alkali to penetrate into the bed core and initiate crack layer formation. Howbeit, observing the same area in Fig. 13b indicates

Fig. 7. 3D XMT image of the overall bed particle layer thickness measurement for quartz bed particles taken from the CFB at 1 day (a), 5 days (b) and 13 days (c) after complete bed change.

7
that these routes are blocked by the outer layer which protects the bed particle from further access of the alkali to the bed core in that area. Fig. 13c reveals a cross-sectional view of the same region revealing the bed core precisely under the above-mentioned convex area. Despite the existence of cracks in the inner layer, no crack layer has been formed under this convex, which demonstrates a shielding effect by the outer layer. On the other hand, crack layers can be noticed in connection to the concave areas on the bed particle surface.

3.3. Comparison of quartz bed particle layer characteristics for the BFB and the CFB boilers

Despite similarities in bed particle layer characteristics of quartz bed particles taken from the two conversion processes, unique behaviors were observed for each. The differences in bed particle layer characteristics are demonstrated in Fig. 14 where the inner layer (red); the outer layer (green); and the inner-inner and crack layers (yellow) are isolated from the rest in two cross-sections of typical quartz bed particles taken from the BFB and CFB processes. In this figure, the inner-inner layer is analyzed together with the crack layers as both have similar composition and consequently have the same appearance in XMT images. 3D images of the inner and the outer layer formed on the exact same particles shown in Fig. 14 are portrayed separately in Fig. 15.

In general, the red and the yellow areas could be interpreted as regions where silicate in the bed particle has reacted with the ash forming elements. However, the formation process and consequently the chemical composition of the two areas are different. The inner layer (red area) contains Ca and low concentrations of K with low reaction rates, whereas the inner-inner layer (yellow area) is dominated by K-silicates. The outer layer (green area) composition is on the other hand dominated by Ca-rich ash particles deposited on the bed particle surface.

The inner layer is distributed more uniformly around quartz bed particles taken from the BFB while the inner layer on the bed particle taken from the CFB boiler was thick on the convex regions and thin in the concaves. The outer layer is also distributed more uniformly in the bed particle taken from the BFB compared to the outer layer of the bed particle utilized in the CFB boiler. Additionally, the outer layer in the latter is thinner probably due to the high erosion rate in the CFB boiler. Therefore, cracks in the inner layer are less frequent in the inner layer of the quartz bed particle taken from the CFB compared to that in the BFB where there is a higher reserve of Ca in the outer layer to diffuse into the inner layer and change its structure. Lower degree of coverage provided
Frequency of the cracks in the inner layer for quartz bed particle utilized in the BFB process could be observed from the surface view of the inner layer (Fig. 13a).

The difference in the uniformity of the bed particle layers in the two conversion processes can, in addition to process-related parameters, result from different surface morphologies of the fresh bed materials utilized in the two boilers. As mentioned above, the growth rate of the bed particle layer differs at convex and concave areas. Correspondingly, the smoother surface of quartz bed particles utilized in the BFB process resulted in a more uniform distribution of the bed layers while higher frequency of the concaves in quartz bed particles used in the CFB boiler reduced the uniformity of the bed particle layers.

The average volume fraction of the crack layers compared to the entire volume of the bed particle was measured and compared for quartz bed particles utilized in the two conversion processes in another work [27]. The results showed that after 13 days, the average volume fraction of the crack layers to the entire volume of the bed particles was 32.1% for quartz bed particles utilized in the CFB boiler while the fraction was only 19.3% for quartz particles taken from the BFB boiler. This also proves that solely formation of the cracks in the inner layer is not enough to initiate crack layer formation as the frequency of the cracks in the inner layer was observed to be lower in the CFB compared to that of the BFB. In fact, the protective role of the outer layer could be more determinative in formation of the crack layers. Accordingly, higher frequency of the concaves in quartz bed particle utilized in the CFB

Fig. 10. XMT 2D view from smooth parts of the surface of typical quartz bed particles taken from (a) the BFB at 13 days and (b) the CFB at 13 days after complete bed change.

Fig. 11. 3D XMT image of a typical quartz bed particle taken from the BFB after 23 days from the complete bed exchange: the bed particle (left), unreacted parts of the bed core (right).
Fig. 12. Cross section from the 3D XMT image from a typical quartz bed particle taken from: (a) BFB reactor at 23 days and (b) CFB reactor at 13 days after complete bed change. The red arrows point the concaves and association of the crack layers to this morphology. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 13. 3D XMT image of a typical quartz bed particle taken from the BFB after 23 days from the complete bed exchange: (a) the inner layer, (b) the outer layer, (c) cross-sectional view.
boiler, combined with the thinner inner layer in these regions and absence of the outer layer, had made the bed particles utilized in this conversion process more vulnerable towards crack layer and inner-inner layer formation compared to quartz bed particles used in the BFB.

3.4. General discussions and practical implications

Overall, it was observed that bed particle layer has a higher growth rate at the convex-shape regions for quartz bed particles taken from both conversion processes. In another word, thicker overall layer (inner and outer layer) could be observed over time at the convex shaped regions on

Fig. 14. Bed particle layer segmentation masks of typical quartz bed particle cross sections taken from the BFB and the CFB after 13 days after complete bed change.

Fig. 15. 3D XMT images from the inner layer and the inner-inner layer (red) and the outer layer (cyan) of typical quartz bed particles taken from the BFB and the CFB boilers after 13 days from the complete bed exchange. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)
the bed particle surface compared to the concaves. Consequently, a thick inner and an outer layer exist on the convexes while concaves are mainly covered with only a thin inner layer. Since deposition of the ash forming matter is essential for bed particle layer formation, this behavior could be explained by more effective impact of the ash forming matter on the convex shaped regions [31]. Spatial disturbance of compounds with different sizes in very close vicinity of the bed particle surface might have an effect in different bed particle layer formation at different surface morphologies. In this sense, the relatively large Ca-rich ash particles could not effectively reach, and subsequently react with the concave shaped areas while K-species in the gas phase can easily access these narrow regions.

Even though there are routes provided by the cracks in the inner layer for further penetration of gaseous alkali, having the outer layer on the convex regions of the bed surface protects the bed particle in these areas from further reaction with alkali. However, in the concave regions where there is no protective outer layer, gaseous alkali penetrates through cracks formed in the inner layer and thereby initiate crack layer formation. Given that higher erosion in the CFB reduces the thickness of the outer layer (or the layer is completely detached), it is more likely that cracks in the inner layer are exposed for interaction with flue gases. Quartz bed particles utilized in studied CFB process therefore contain more crack layers and wide inner-inner layers close to the surface of the bed particle. A schematic representation of the bed particle layer formation on quartz bed particles is portrayed in Fig. 16.

Results from this work are valuable in industrial applications for the selection of suitable bed material qualities and also for understanding how operation time could be impacted by bed particle morphologies. It’s important to consider that bed materials with identical chemical composition but different surface morphologies may exhibit varying rates of layer formation. This consideration is particularly relevant for CFBs, where attrition is more pronounced compared to BFBs. The results here suggest that the combined protective effect of a continuous Ca-rich inner layer with a largely coherent outer layer dominated by bed ash composition is noticeable. The largest interaction with bed material with alkali from the fuel occurs where there are cracks in the inner layer and there is no outer layer reducing the exposure of these cracks to gaseous alkali, which then may penetrate into the bed particle and form a crack layer. If both the inner layer and the outer layer are continuous, as is largely the case for smoother parts of bed particles in BFB, there is very low degree of interaction. It may be equally beneficial to use smooth particles in CFBs, as even though the outer layer is removed there would be fewer concave areas with a weaker inner layer protecting the bed particle core. The present study therefore suggests that particles with smoother surfaces might have lower operational problems, as it is the protective properties of Ca-containing inner and outer layer that prevents extensive crack layer formation. Crack layers may cause breakage of the bed particles into fine fragments which are covered with alkali silicate. These fragments can aggravate bed agglomeration and/or result bed deposition in cyclones and return legs. Therefore, given the variation in surface properties observed among bed materials originating from different mineral deposits, selection of bed particles with more favorable surface morphology can noticeably mitigate these operational problems. Surface morphology of bed materials can also be influenced by the way in which the bed particles are extracted, such as whether they are subjected to crushing or maintained in their native form.

Given the findings derived from this work which underscore the role of surface morphology in bed particle layer formation, it is prudent to consider extending research efforts to explore the potential influence of additional geometric and morphological properties of bed particles, such as particle size, sphericity, and roundness, to further enrich our understanding of bed particle layer formation.

4. Conclusions

Bed particle layer and crack layer formation in quartz bed particles
utilized in fluidized bed combustion of woody biomass were examined in this study through 3D and 2D XMT analysis in combination with SEM/EDS characterization of the bed particles taken from two different conversion processes. The main findings from this work are as follows:

- Regardless of the conversion process (BFB/CFB), different tendencies toward bed particle layer formation at different surface morphologies results in the formation of a thicker inner layer and an outer bed particle layer in the convex shaped regions on the bed particle surface, while in concave areas mainly a thinner inner layer is formed.
- The bed particle layer grows faster on the convex areas on the bed particle surface compared to the concave areas. Consequently, the bed particle layer is comparably thicker in the convex areas of the old bed particles.
- Areas with both a continuous inner layer and an outer layer display the least tendencies for crack layer formation.
- If cracks in the inner layer are present, an outer layer may still prevent formation of the crack layers.
- Crack layers mainly form in concave areas where there is both cracks in the inner layer and an outer layer is missing, thereby enabling gaseous alkali reactions with the quartz bed particle core.

CRediT authorship contribution statement

Ali Valizadeh: Conceptualization, Methodology, Investigation, Formal analysis, Visualization, Writing – original draft. Nils Skoglund: Conceptualization, Methodology, Writing – original draft, Writing – review & editing, Supervision, Funding acquisition. Fredrik Forsberg: Formal analysis, Writing – review & editing. Henrik Lycksmal: Formal analysis. Marcus Ohman: Conceptualization, Resources, Writing – review & editing, Supervision, Project administration, Funding acquisition.

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.

Data availability

Data will be made available on request.

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