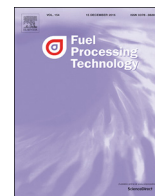




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Research article

Biomass pellet combustion: Cavities and ash formation characterized by synchrotron X-ray micro-tomography

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ABSTRACT

Ash formation during thermochemical conversion of biomass-based pellets influences both char conversion rates and ash-related operational problems. The objective of the present study was to provide detailed insights into changes in fuel and ash properties during fuel conversion. Pellets of poplar wood and wheat straw were used as model biofuels, representing vastly different compositions of ash-forming elements. Pellet samples at different char conversion phases were analyzed by synchrotron-based 3D X-ray micro-tomography, to map and visualize the development of cracks, internal cavities, and ash layers during conversion. The analysis of ash layers was complemented by scanning electron microscopy combined with energy-dispersive X-ray spectroscopy. The results provide new insights into how large cracks and internal cavities are developed already during devolatilization, for example, the poplar wood pellets had a 64% void fraction after the devolatilization stage. As expected, there were large variations between the ash layer properties for the two fuels. A porous, low density, and calcium-rich ash was formed from the poplar fuel, whereas the wheat straw ash was a high-density silicate melt that developed into bubbles on the surface. As the conversion proceeded, the wheat straw ash covered parts of the active char surface area, but without blocking the gas transport.

1. Introduction

Renewable lignocellulosic biomass is expected to become an important energy source in the future, and therefore the need for more biomasses will most probably increase. To fulfill the anticipated increase in biomass utilization, alternative fuel assortments are being introduced to the market, for example, agricultural residues and short-rotation coppices. Pelletization of biomasses is commonly used to lower costs in handling and transports and improve fuel feeding, especially for biomasses with very low bulk density and inhomogeneous structures such as grass, straw, and forest residuals [1]. Agro-based crops generally have higher ash contents and more varying ash compositions than woody biomass [2–5]. Such variations in ash composition and content places high demands on boiler flexibility, especially in the ash handling system, and some of these fuels are known to cause severe operational problems related to ash properties [6–9]. Ash formation and ash transformation during biomass combustion has been the focus of extensive research in recent decades, often with a focus on slag and particulate matter formation. General reaction mechanisms have been proposed, and the concentration of various ash-forming elements can be

used to predict and describe ash chemistry for typical woody fuels [7,10]. However, these predictive models typically have a basis in thermodynamic equilibrium or are based on empirical relationships, and they typically do not describe the ash properties in relation to time and char conversion.

The rate of fuel particle conversion is also of major interest during the thermochemical conversion of biomass. Solid fuels usually undergo thermal conversion in two more-or-less distinct stages – devolatilization (flaming combustion) and the subsequent char oxidation (glowing combustion). The char oxidation reactions are relatively slow and are considered to be the rate-limiting step of the thermal conversion process [11–13]. The conversion rate during oxidation is influenced by a combination of the char properties, including porosity, thermal properties, and chemical reaction kinetics [14]. These char properties are affected by fuel type and process conditions during devolatilization, but transport of gaseous components in and around the burning fuel particle also play an important role in char oxidation [15]. Therefore, the physical fuel properties, e.g. particle size, water content, density, and particle shape, and their development during conversion are important for the resulting conversion rate of fuel particles, such as a fuel pellet

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[13,16,17].

In addition to the relevance of physical properties of fuel particles for the fuel conversion, it has been found that ash layers may form on char surfaces during thermal conversion in studies of the intrinsic reactivity of biomass powders [18–22] as well as larger fuel particles [23,24]. This phenomenon might influence the char conversion rate, especially during the later stages of conversion when the ash comprises an increasing fraction of the remaining fuel. The formed ash layer could limit diffusion to and from the char particle, thus affecting the transfer of oxidant to the fuel as well as the removal of combustion reaction products. The formation of such ash layers during fuel conversion of single fuel particles such as pellets are scarcely described in literature but a couple of studies have been reported [25,26].

Ash enrichment on the surface of chars, produced from pine and switchgrass at different degrees of char conversion, was reported by Wornat et al. [25]. The underlying mechanism for this ash enrichment was a combination of volatile ash-forming elements (e.g. potassium) migrating to the char surface together with conversion of the carbonaceous matrix. For switchgrass, potassium silicates were detected during conversion, but the amount of potassium decreased as the silicates were gradually transformed into calcium–potassium silicates. This observation is in agreement with a mechanism described elsewhere [10,27], and recent findings by Fagerström et al. [26] who also discussed carbonate and oxide formation from silicon poor fuels in single pellet combustion. The effect of ash layer composition was further investigated by Strandberg et al. [21] where it was suggested that char conversion rates could be affected by dense ash layers, often dominated by low-temperature melting alkali-silicates. It was further suggested that such an inhibition is unlikely to occur for woody-type biomass with a high content of calcium and low silicon content. Ash layers with such a composition were found to be highly porous with discernible small particles similar to a powder. The chemical composition was likely dominated by oxides, and possibly carbonates, and this morphology did not exhibit any discernible effect on the rate of fuel conversion [21].

Such detailed information concerning ash formation in terms of physical properties as well as ash layer composition and distribution is partially implemented in fuel conversion models [24,28,29]. However, numerical conversion models often either disregard [30,31] or simplify [32,33] ash layer formation and related effects on fuel conversion. Besides a possibly inhibiting ash layer, fuel particle conversion rates are dependent of the active char surface and thus on pore development in the char. The formation of cracks, cavities, and pores is expected to deviate between compressed biomass particles and uncompressed biomass particles. However, most models available for fuel conversion for thermally thick particles have been developed for uncompressed biomass particles [29–36], and yet they are considered to be valid for predicting the conversion of compressed particles like pellets and briquettes even though the detailed mechanistic information is only approximated. Only a few studies suggest fuel conversion models specific for biomass pellets [37–39] and new empirical information is required to enable refinement of such models.

The objective of the present study was to provide new detailed and time resolved information on char morphology and the development of cavities and ash layers during the combustion of biomass pellets. The char residues were analyzed using synchrotron-based X-ray micro-tomography, giving 3D density information describing the development of cracks and cavities in the char during conversion, as the formation of ash. This was complemented by energy-dispersive X-ray spectroscopy (SEM-EDS), providing elemental composition for the ash. Two different lignocellulosic biomass pellets were used for the study, with wheat straw representing grassy fuels with silicon and potassium dominating the ash-forming elements, and poplar representing woody fuels with ash dominated by calcium and potassium. Char samples were prepared by interrupting and quench cooling combustion at varying degrees of fuel conversion in a setup designed for single-pellet isothermal

Table 1

Fuel properties and composition of pellets from poplar wood and wheat straw.

	Poplar pellets	Wheat straw pellets
Moisture (wt%)	7.9	8.5
Ash (wt% _{d.b.} ^a)	2.2	4.3
Volatiles (wt% _{d.b.})	81.0	77.5
Main elements (wt% _{d.b.})		
C	49.5	47.5
H	6.0	5.8
N	0.3	0.4
O	42	42
Main ash-forming elements (mg kg _{d.b.} ⁻¹)		
Cl	75.0	2090
S	384.0	717.0
Si	211.0	9460
Al	31.2	145.0
Ca	6010	2860
Fe	55.4	117.0
K	3070	8450
Mg	554.0	767.0
Mn	9.0	22.2
Na	19.9	42.1
P	873.0	638.0
Zn	42.3	6.8
Ash fusion temperature (K)		
ST ^b	990	970
DT ^c	1610	1120
HT ^d	> 1820	1350
FT ^e	> 1820	1370
Pellets bulk density (kg m _{d.b.} ⁻³)	640	570
Pellets durability (%)	99.1	96.4

^a d.b. dry basis.

^b Shrinkage starting temperature.

^c Deformation temperature.

^d Hemisphere temperature.

^e Flow temperature.

thermogravimetric analysis (large-sample TGA).

2. Material and methods

2.1. Fuels

In this study, two types of lignocellulosic biomass pellets were used. The first consisted of stems from poplar (a mixture of stem wood and bark from two clones, both hybrids of *Populus maximowiczii* × *Populus nigra*, harvested after growing for 7 years in a short-rotation coppice field near Straubing, Germany), and the second was wheat straw (*Triticum aestivum*, originating from Thüringen, Germany). The weight of the single pellets used for the experiments was 600 mg ± 5%, and the diameter was 6 mm. The compositions of ash-forming elements differed significantly between the two biomasses, with the wheat straw ash being dominated by silicon and potassium, and the poplar ash consisting mainly of calcium and potassium (Table 1). The pellets were analyzed by standard fuel analysis, including moisture content (EN 14774/15414), ash content (EN 14775:2009/15403:2011), content of volatile matter (EN 15148:2009/15402:2011), main elements C, H, N, O (EN 15104:2011/15407:2011), detailed analysis of main ash-forming elements (EN 187185, 187177, and ICP-AES analysis of elements dissolved in LiBO₂/HNO₃), and pellet properties (ISO 17831-1:2016, ISO 17828:2015, and ISO 18846:2016). The ash-melting behavior was determined according to CEN/TS 15370-1:2006.

2.2. Combustion experiments

Combustion experiments were carried out in a laboratory-scale single-pellet thermogravimetric reactor as described in detail in previous studies [16,26,40] and visualized in Fig. 1. The reactor consists of

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