



Influence of the particle size on the release of inorganic trace elements during gasification of biomass pellets



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HIGHLIGHTS

- On-line analysis of hot gas by Molecular Beam Mass Spectrometry.
- Main gaseous species were $^{34}\text{H}_2\text{S}^+$, $^{36}\text{HCl}^+$, $^{74}\text{KCl}^+$, $^{58}\text{NaCl}^+$, $^{60}\text{COS}^+$ and $^{63}\text{PO}_2^+$.
- Release of KCl and H_2S decreased with decreasing pellet size for herbaceous fuels.
- Release of HCl increased strongly with decreasing pellet size for herbaceous fuels.
- Secondary reactions have significant influence on the amount of the released species.

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ABSTRACT

In the present work the release of inorganic trace elements as a function of the particle size of different biomass pellets was investigated. Therefore, three different sample sizes of three kinds of biomass (wood, miscanthus and straw) were gasified in an electrical heated furnace at 800 °C. The sample sizes were: Half cylinders with a particle size of 3 mm × 6 mm, irregular fragments with a particle size of 2–4 mm, and powder with a particle size of ≤ 0.54 mm. Online hot gas analysis was done by Molecular Beam Mass Spectrometry. The focus was set on the alkali metals K and Na and on the nonmetals S, Cl and P. Qualitative and quantitative analysis of the mass spectra were done. Main gaseous species were $^{34}\text{H}_2\text{S}^+$, $^{36}\text{HCl}^+$, $^{74}\text{KCl}^+$, $^{58}\text{NaCl}^+$, $^{60}\text{COS}^+$ and $^{63}\text{PO}_2^+$. The data clearly showed the correlation of the size of the biomass samples on the release behaviour. It is shown that several secondary reactions have a significant influence on the release of inorganic elements.

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1. Introduction

Due to the prospected increasing usage of renewables in energy generation [1], fast growing and annual crops (e.g. miscanthus and straw) become more and more importance as energy source. Especially the thermochemical conversion becomes more and more interesting, because of the possibility of co-generation of alternative fuels and chemicals. Straw is an abundant agricultural byproduct and the interest in using straw to generate energy is growing in the EU. However, in comparison to the classic renewable biomass wood, the herbaceous crops straw and miscanthus contain high amounts of inorganic species, e.g. chlorine, sulphur, phosphorus, alkali metals, and silica. The release of inorganic

species can give rise to several problems in power plants. Phosphorus and sulphur species can cause increased catalyst deactivation [2–4]. Alkali metal species can cause fouling, slagging and corrosion [5,6]. Alkali metals can also cause defluidisation of the bed material in fluidised bed gasification due to the formation of eutectics with a low melting point. Several studies underlined that the particle size significantly affects the release of inorganic species, especially from a quantitative point of view [7–9]. It was shown that secondary reactions of the released species with unburned fuel or ash can have significant influence on the release. Especially, the amount of the release is related to the contact time of the gaseous species with the remaining fuel material. Nevertheless, there are still many open questions, especially regarding more detailed information of the influence of biomass particle size under gasification conditions. Therefore, the objective of this study was to investigate the influence of particle size on the release of K, Na, P, Cl, and S species during gasification of biomass.

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2. Experimental

2.1. Preparation and analysis of the biomass samples

Three kinds of biomass were used in the experiments: wood, miscanthus and straw. The biomass was delivered pelletised and archived up to the time of the study under dry conditions at room temperature. Samples of three different sizes of each of the three biomass were investigated. Half cylinders with a particle size of 3 mm × 6 mm were prepared by cutting the cylinders along the 6 mm diameter section and with a radius length of 3 mm. Irregular fragments with a particle size of 2–4 mm were obtained by crushing the pellets. The fragments achieved were then screened to obtain the fraction of interest. Powder with a particle size of ≤0.54 mm was obtained using an IKA laboratory mill to chop the samples into pieces which were screened afterwards to obtain the particle size of interest.

The ultimate analysis of the biomass under investigation was performed by standard analytical methods by the central division of analytical chemistry (ZCH) of the Forschungszentrum Jülich. Inductively coupled plasma optical emission spectroscopy (ICP-OES) was used for chemical analysis of the biomass samples (Model TJA-IRIS) (Table 1). Chlorine was analysed by Cl-Ion chromatography (IC) after soda extraction. Carbon, hydrogen, nitrogen and sulphur were analysed by CHNS-analyzer (system LECO). The results are given in Table 1. The differences between the three size fractions were within the variance of the biomass composition.

2.2. Thermochemical conversion of biomass and characterisation of inorganic trace species in the product gas

The thermochemical conversion of the biomass was carried out in an electrical heated high temperature tube furnace. Vapour K-, Na-, Cl-, P-, and S-species in the product gas were detected by Molecular Beam Mass Spectrometry (MBMS). A schematic of the experimental setup is given in Fig. 1. The experimental setup has proven its reliability in our group for more than 10 years and already been described in detail elsewhere [10,11]. Therefore, only sufficient detail to allow the work to be reproduced is provided and only relevant modifications are described.

The gasification reactor consisted of a 21 mm diameter alumina tube, placed in a furnace working through four heating zones

independently adjustable of each other. The reactor was made of alumina to prevent reactions between conversion products and the reactor walls and to allow thermal cracking of organic species at high temperatures. Before the MBMS inlet there was an additional heater used to prevent the condensation of gaseous species. The end of the reactor was coupled to the sampling orifice of the MBMS device, to sample the high-temperature gasification products. The orifice protruded into the furnace, to maintain an elevated temperature to prevent condensation of gas-phase species on the tip of the orifice. The gas and sample inlet consisted of a brass flange sealed to the cold zone of the reactor. The furnace was maintained at a constant temperature of 800 °C in the reaction zone as shown in Fig. 1. The 1500 °C zone was necessary to thermally crack organic matter. The temperature at the end of the reactor was maintained at 1100 °C to prevent gaseous species of interest from condensation. The experiments were performed at atmospheric pressure. The gas mixture consisted of He/0.2%O₂. The low oxygen content was necessary to simulate gasification like conditions during the thermochemical conversion of biomass as shown by Porbatzki et al. [12]. The overall flow rate was 3.0 l/min (STP). The residence time of conversion products in the reactor before reaching the MBMS was in the range of 0.1–0.2 s.

A typical experimental run consisted of the following steps. At the start of the experiment a platinum boat loaded with 140 mg biomass was attached to the end of a 6 mm diameter alumina rod and inserted into the air cooled end of the heated flow channel. The gas mixture was fed into the reactor and the background signal was detected by MBMS. After about 20 s the sample boat was placed in the 800 °C hot reaction zone by the horizontally displaceable alumina rod. The heat up of the biomass sample could not be directly determined. However, the heat up of the close environment of the sample was determined by a platinum/platinum-rhodium thermo couple which was placed close to the biomass sample. The heat up of the thermocouple occurred with about 300 °C/s. The thermocouple was made of a type S element (wire of 0.5 mm diameter) which was housed in a corundum capillary tube (4 mm diameter). The thermocouple wire extended the corundum capillary tube for about 2–3 mm. It is concluded that the heat up of the thermocouple is retarded in comparison with the environment which is caused by several effects, e.g. heat transfer and heat capacity differences of the wire and the capillary. However, it can be concluded that the heat up of the biomass particles environment occurred faster. Furthermore, it can be concluded that devolatilisation temperature of the particles was reached shortly after sample insertion. Additionally, the heat up time depends on the particle size and increases with increasing particle size. In conclusion, devolatilisation temperature was reached shortly after sample insertion and the high temperature gasification product gas flowed to the end of the reactor. All parts downstream the reaction zone were kept over the condensation point of the vapour K-, Na-, Cl-, P-, and S-species. The reactor was coupled to the MBMS. A schematic of the MBMS is given in Fig. 1. The reaction products entered the MBMS through a nozzle with 0.3 mm in diameter. The MBMS consists of three differentially pumped chambers. Due to immediately supersonic free jet expansion of the hot gas into the first high vacuum chamber (10⁻² mbar), the species are cooled down below room temperature in microseconds, attain free molecular flow and therefore, form a molecular beam. The core of the free jet expansion is extracted by a conical skimmer of 1 mm diameter and directed into the third chamber. There, a hot filament emits electrons with an electron energy of 50 eV and an emission current of 1 mA. Every 10⁻⁴–10⁻³ molecule is ionised by electron impact. After passing the deflector, the ions are filtered in a quadrupole mass analyser and detected by an off axis electron multiplier. The amplified signal is recorded by a computer and software package as a function of time and

Table 1
Chemical composition of the biomass.

	Wood	Miscanthus	Straw
<i>Proximate analysis (wt.%)</i>			
Ash	0.34	3.3	11.6
Moisture	8.1	6.8	7.3
<i>Ultimate analysis (wt.%, as received)</i>			
C	46.4	45.2	40.3
H	6.05	5.86	5.45
N	0.20	0.67	0.59
O	45.7	43.5	40.6
S	0.02	0.11	0.15
P	<0.005	0.051	0.077
Cl	0.012	0.222	0.364
K	0.029	0.720	1.700
Na	<0.005	0.028	0.023
Ca	0.083	0.340	0.130
Mg	0.009	0.077	0.068
Fe	<0.005	0.032	0.037
Al	<0.005	0.051	0.047
Si	0.018	0.560	3.300
<i>Elemental ratio (on a molar basis)</i>			
K/Si	1.2	0.9	0.4
K/Cl	2.2	2.9	4.3
Ca/S	3.3	1.8	0.9

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