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## ASH MELTING TEMPERATURES FOR BIOMASS FUELS: EFFECT OF FUEL SAMPLE PREPARATION BY ASHING AT DIFFERENT TEMPERATURES

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**Abstract.** In the discussion on biomass as a renewable energy source, its benefits are repeatedly recognised: - reducing greenhouse gas emissions by substituting fossil fuels; - increasing energy security by diversifying the energy mix and reducing dependence on imported fuels; - creating economic prospects by promoting rural development, job creation and valorisation of waste streams. However, the utilisation of biomass in combustion processes faces some challenges. This article emphasises one of them: the ash problem due to the pronounced tendency to form deposits on the heating surfaces. In order to properly design or select a biomass combustion system, the indicators of the tendency of biomass ash to form deposits on the heating surfaces must be adequately determined. Determining ash melting temperatures for biomass is crucial for understanding combustion behaviour and potential problems such as slagging and fouling. Biomass ash melting temperatures are typically determined at 550 °C for biofuels and 815 °C for solid mineral fuels. Some established certification schemes and national regulations favour the preparation of biomass samples by ashing at 815 °C. In this paper, the question is answered whether and how the ashing temperature of the biomass sample influences the characteristic points of the ash melting temperatures. The characteristic ash melting temperatures were determined for different types of biomass (wood chips, soybean and maize straw), where the ash sample was obtained by ashing at three different temperatures (550, 710 and 815 °C).

**Keywords:** wood chips, soybean straw, maize straw, indicators for slagging and fouling

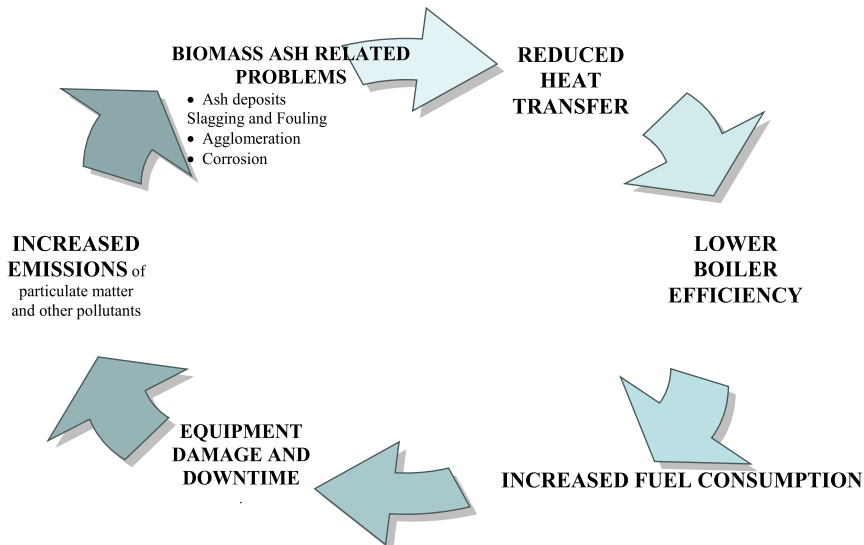
### 1. INTRODUCTION

It is widely recognised that biomass is a significant source of renewable energy that can (at least partially) replace fossil fuels to power a green and sustainable society and accelerate the development of a circular bioeconomy. However, due to its specificity, this energy source also poses implementation challenges. Biomass, in instance, is more

geographically dispersed, has a lower energy density and conversion efficiency than fossil fuels [1]. Furthermore, depending on the biomass, technology and expertise used, certain pollutants are released during the conversion process [2] that are more or less common to all fuels. The utilization of biomass therefore requires complex processes of material handling, pre-treatment and design of the processing facilities.

When designing or selecting a biomass combustion (and, more broadly, thermochemical conversion) facility, as well as optimizing combustion conditions, it is crucial to understand and predict the behavior of biomass ash. Key issues associated with biomass ash include [3, 4]:

- **Slagging:** molten high temperature ash deposits build up on the furnace walls and on the heat transfer surfaces, hindering heat transfer;
- **Fouling:** unmelted ash deposits at lower temperatures, that accumulate on the surfaces of heat exchangers and also impair heat transfer;
- Biomass ash deposits are often more difficult to remove than coal ash deposits as they have lower porosity and smoother surface, greater strength and viscosity [5];
- **Agglomeration:** formation of larger ash clusters through melting and sticking together of ash components, which can obstruct combustion (“trapping” of the fluidized bed) and the flue gas flow;
- **Alkali-induced corrosion:** chemical reactions between the alkali metals contained in the biomass ash (especially in combination with Cl or S) and the boiler materials that lead to material deterioration.



**Figure1.** Impact of biomass ash-related issues on biomass-based energy systems



This paper addresses these biomass ash-related issues, which have a significant impact on the reliability and efficiency of biomass-based energy systems (see fig.1). For this purpose, two types of biomass were studied, namely two agricultural (soybean and maize straw) and one woody biomass (wood chips). The proximate and ultimate analysis, as well as the chlorine content, were determined for all three biomass samples. In addition, the characteristic ash fusion temperatures (AFT) were determined in relation to the shrinkage starting temperature (SST), the deformation temperature (DT), the hemisphere temperature (HT) and the flow temperature (FT) as well as their dependence on the alkali content (K, Na) of the ash and the chlorine content of the original biomass. The biomass samples were also ashed at three different temperatures to investigate how the ashing temperature affects both the ash melting stages and the carbon, hydrogen, and sulfur content of the ash.

These findings can help industrial users of biomass gain a better understanding of how biomass characterisation, namely its ash, influences combustion optimisation and technology selection. Furthermore, the work answers the testing laboratory's question of whether different ashing temperatures change the indicators for the biomass ash deposition tendency, and if so, for which type of biomass this influence is more pronounced.

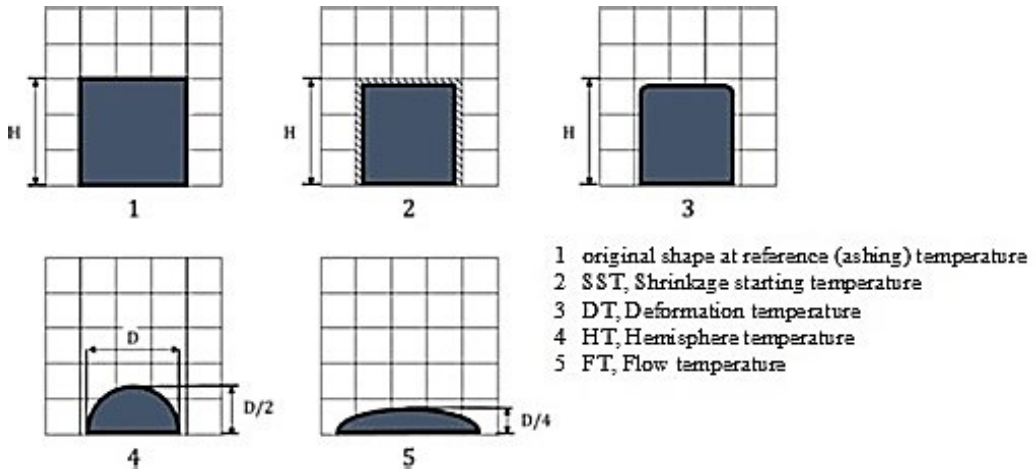
## 2. METHODOLOGY

The biomass samples were prepared according to the standard [6]. All samples were dried in a drying oven (Binder GmbH) at a temperature of 105 °C and then ground with a mill.

Proximate analyzes were carried out in accordance with the standards [7-10]. The ash content of all samples was determined using a muffle furnace (Vecstar furnace) at 550 °C, 710 and 815 °C according to [8]. The analysis is done in triplicate.

The LECO CHN 628 series was used to determine the total N, C and H content in solid biofuels and representative biomass ash samples according to the standard method [11], while the O content was calculated according to [12]. All measurements were carried out in triplicate.

The ash fusion temperatures (AFTs) was investigated on a Leitz Wetzlar device for determining ash melting in accordance with the standard [13]. Ash samples are obtained by controlled thermal decomposition of the fuel in a muffle furnace. In this case, as already mentioned, the ash was obtained at three different ashing temperatures. The ash thus obtained is then pressed into a cylinder. The prepared samples are then subjected to uniform heating under oxidizing or reducing conditions, and the AFTs are monitored by measuring the dimensional and shape changes of an ash cylinder as a function of temperature, identifying four characteristic temperature points, as shown in Fig. 2. In this case, the characteristic AFTs were determined in an oxidizing atmosphere, as this corresponds better to real combustion conditions and is more cost-effective. Moreover, the difference between the results obtained under oxidizing and reducing conditions is negligible for samples with low Fe content, which is generally a characteristic of biomass [14].



**Figure 2.** Visual determination of the characteristic AFTs [13]

The alkali content (Na and K) in the ash of representative biomasses was determined using a flame photometer - Sherwood Scientific Ltd. PF360, in accordance with [15]. The chlorine content in biomass samples was determined using a standard method according to [16].

### 3. RESULTS AND DISCUSSION

The physico-chemical composition of biomass is not uniform and varies considerably depending on the plant species, growing conditions (climate, irrigation, soil type/pH, fertilization, distance from the pollution source) and the particular plant part. The process of collecting, transporting, and storing biomass also has a significant impact on its composition. The value of a particular biomass as a fuel is best categorized by a proximate, ultimate analysis and heating value. According to the findings in Table 1, all biomass samples are highly acceptable for burning, considering the moisture content ( $<10\%wb$ ) and the ash concentration ranging from 1.28 to 3.38%. This is supported by the high calorific value of these biomasses. All three biomasses have a pronounced volatility and consequently a lower fixed carbon content than coal, which is a primary attribute of biomass as a fuel. This requires different combustion system designs compared to coal, the fuel most similar to biomass.



**Table 1.** Proximate and ultimate analysis of representative biomass

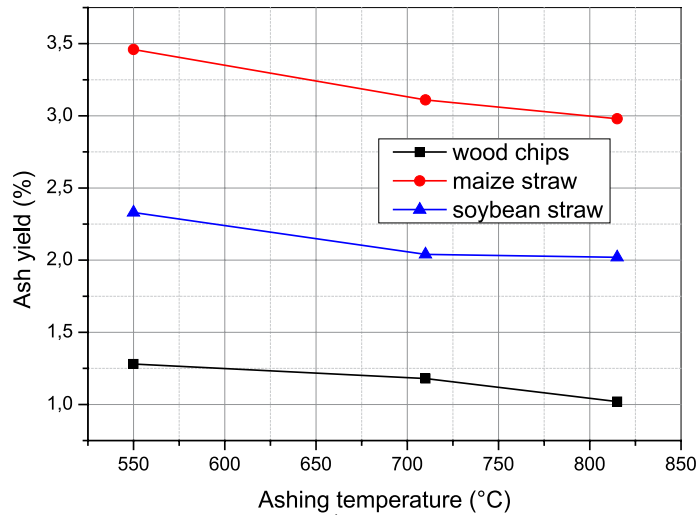
			wood chips	maize straw	soybean straw
Proximate analysis	Inherent moisture	%	8.75	9.75	9.10
	Ash 550°C	% <sub>dwb</sub>	1.28	3.38	2.33
	C-fix		17.49	17.72	15.17
	Volatile		81.23	78.90	81.15
Heating value	High	MJ/kg	19.59	18.05	20.87
	Low		18.36	16.78	19.57
Ultimate analysis	Total carbon		49.57	46.43	47.88
	Hydrogen		5.94	6.20	6.29
	Sulfur	% <sub>dwb</sub>	0.02	0.09	0.13
	Nitrogen		0.22	0.54	0.75
	Oxygen*		42.96	43.36	42.61
	Chlorine	ppm	84	1219	757
	% <sub>dwb</sub>	0.01	0.12	0.08	

\*Oxygen = 100% - Ash 550°C - Carbon - Hydrogen - Nitrogen; dwb - Calculated on dry weight basis

The organic compounds contained in biomass consist mainly of carbon (C), hydrogen (H), oxygen (O) and nitrogen (N) with traces of chlorine (Cl) and sulfur (S). Again, all three samples show a typical characteristic of biomass as a fuel - they have a significantly higher content of O, but a lower Cl and S content (compared to coal). While a high O is disadvantageous as it represents internal ballast, does not burn but facilitates combustion and displaces other combustible compounds, a low N, Cl and S content is beneficial for pollutant emissions.

After determining that all biomass samples were suitable for combustion, the effects of different ashing temperatures on the ash yield (Figure 3) and the characteristic ash melting temperatures (Table 2) were investigated.

As can be seen from Fig. 3, the ashing temperature has influence on the ash yield, whereby the test results indicate that the ash yield decreases with increasing ashing temperature due to the release of volatile compounds and the change in mineral composition. The effect is greatest for maize straw (ash yield decreases by 0.48 %) and lowest for wood chips (0.26 %). Furthermore, this effect is more pronounced for straw samples at lower temperatures (550-710°C), whereas for wood chips in the higher temperature window (710-815°C).



**Figure 3.** Ash yield of biomass samples (wood chips, maize straw and soybean straw) at different ashing temperatures calculated on dry mass

Laboratory AFT tests estimate melting behavior of the ash and is therefore an essential indicator of the slagging and fouling tendency. In addition, higher AFTs are preferred, especially the first two characteristic temperatures, as the ash is then less sticky and less prone to forming deposits.

**Table 2.** The characteristic ash melting temperatures at different ashing temperatures of representative biomass samples

temperature [°C]	wood chips			maize straw			soybean straw		
	550	710	815	550	710	815	550	710	815
SST	1089	1125	1200	1015	1050	1085	1160	1170	1200
DT	1154	1230	1280	1075	1090	1095	1260	1290	1300
HT	1420	1345*	1360*	1190	1160	1170	>1400	>1400	>1400
FT	1425	1350	1365	1220	1190	1200	>1400	>1400	>1400

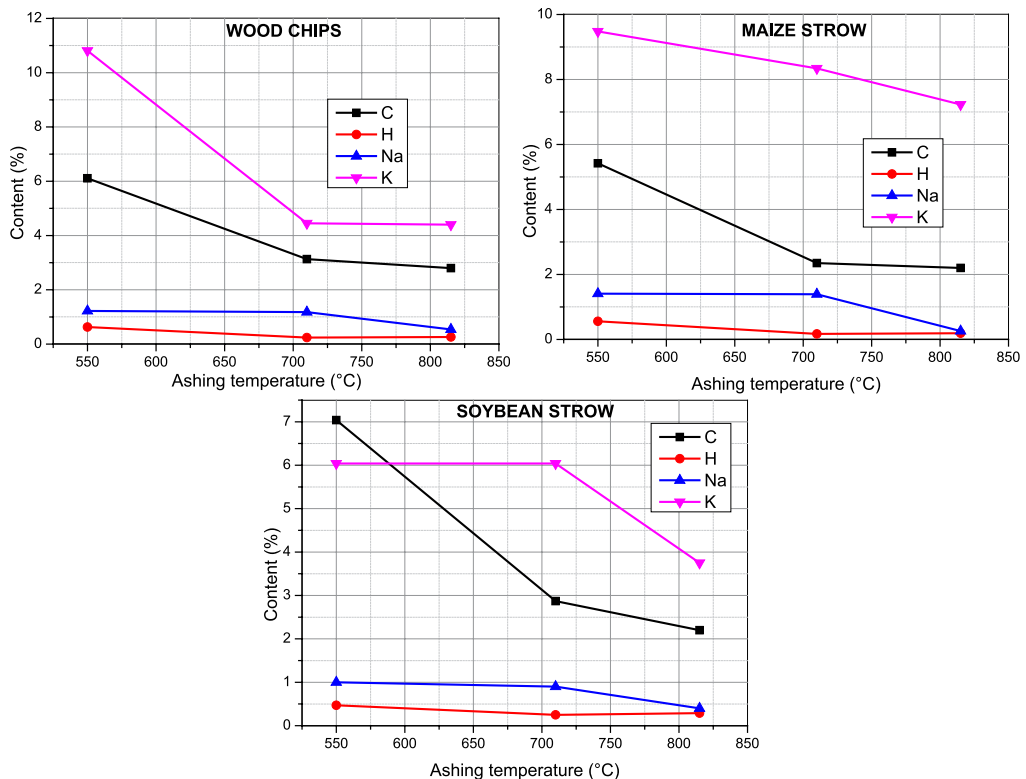
\* The HT of these samples could not be evaluated due to the instantaneous deformation of the sample, so a difference of 5°C is assumed for the calculation of the indicator, as was the case for the sample obtained at an ashing temperature of 550°C.

The results presented in Table 2 show that SST and DT increase with increasing ashing temperature for all biomass ash samples. However, the values of HT and FT for the ash of wood chips and maize straw are higher for the ash obtained at an ashing temperature of 550 °C than for the ash obtained at higher temperatures. At the same time, HT

and FT for all ashes obtained at 710°C and 815°C increase with the ashing temperature. Soybean straw has the best properties in terms of high all AFT points.

In an attempt to explain the differences in the characteristic AFTs of the ash obtained at different ashing temperatures, a partial elemental analysis of the ash was performed, focusing on the determination of the residual combustible matter and the content of alkali metals as the elements most influential for the behavior of the ash during combustion.

An elemental analysis of the ash was carried out to determine if unburned hydrogen, carbon and sulfur were present in the ash samples of representative biomass obtained at different ashing temperatures (Figure 4). Although the ashing method using a muffle furnace at 550°C, 710°C and 815 °C is used as standard to prepare ash samples for characterization [8], the results from Fig. 4 show that unburned C and H are still present in the samples prepared in this way.



**Figure 4.** H, C, S, Na and K content in ash samples obtained at different ashing temperatures

The elemental analysis revealed no nitrogen or sulfur content in any of the ash samples. The C and H content in biomass ash is significantly affected by the ashing temperature, with higher temperatures resulting in lower content. As the ashing temperature increases, residual organic matter burns more completely, leading to a decrease in total



carbon and hydrogen in the ash. In all biomass ash samples, this effect is more pronounced at temperatures of 550-710°C.

The ashing temperature has an obvious influence on the alkali content in the ash samples (see Fig. 4) and consequently on the AFT, with higher temperatures often leading to increased volatilization of alkali metals and potential changes in ash slagging/fouling tendencies. Potassium reduction is more pronounced in wood chips and maize straw at 550°C -710°C, whereas it is more pronounced in soybean straw in the temperature window 710°C -815°C. Sodium, which is significantly lower than potassium, decreases more intensively in the temperature interval 710°C -815°C (Fig. 4). Analysis of the diagram in Figure 4 and the AFT values in Table 2 clearly shows that a higher alkali content, especially potassium, leads to lower ash melting temperatures, which are in accordance with the trends observed by [14, 17].

#### 4. INDICATORS FOR THE ASH TENDENCY TO FORM DEPOSITS ON THE HEATING SURFACES

In addition to the AFT test, a number of indicators have been developed to assess the tendency of ash to form deposits. Some of them, which are consistent with the analyzes presented in this paper, are listed in Tab. 3, and the values of the indicators for the representative biomass ashes are shown in Tab. 4.

**Table 3.** The calculations and ranges for indicators of the ash tendency to form deposits on the firing side of heating surfaces

Indicator	Calculation	Range	Tendency to form deposits	Reference
Cl [%]	-	<0.2	low	[18]
		0.2-0.3	medium	
		0.3-0.5	high	
		>0.5	extremely high	
TA, total alkalis [%]	$\text{Na}_2\text{O}+\text{K}_2\text{O}$	<0.3	low	[19]
		0.3-0.4	medium	
		>0.4	high	
AFI, Ash fusibility index [°C]	$(4 \text{ SST} + \text{HT})/5$	>1342	low	[18]
		1232-1342	medium	
		1052-1232	high	
		<1052	extremely high	
DT [°C]	tab.3	>1100	low	[18]
		900 -1100	medium	
		<900	high	



**Table 4.** Indicator values for selected biomass ash samples

		Na <sub>2</sub> O %	K <sub>2</sub> O %	SST °C	HT °C	Cl %	TA %	AFI °C	DT °C
wood chips	550	1.64	12.98	1089	1420	0.01	14.62	1155.2	1154
	710 °C	1.59	5.35	1125	1345	0.01	6.94	1169	1230
	815	0.73	5.29	1200	1360	0.01	6.02	1232	1280
maize straw	550	1.90	11.39	1015	1190	0.12	13.29	1050	1075
	710 °C	1.88	10.2	1050	1160	0.12	12.08	1072	1090
	815	0.35	8.69	1085	1170	0.12	9.04	1102	1095
soybean straw	550	1.35	7.26	1160	1400	0.08	8.61	1208	1260
	710 °C	1.21	7.26	1170	1400	0.08	8.47	1216	1290
	815	0.54	4.47	1200	1400	0.08	5.01	1240	1300

From the results of Table 4, it can be concluded that the chlorine content shows the lowest tendency and the DT test the mildest tendency for the biomass samples analyzed. Depending on the DT, the ashes show either a low (wood chips and soybean straw) or a medium tendency (maize straw), while the total alkali shows the most extreme - high potential for the formation of deposits for all biomass ashes analyzed. The AFI indicator is particularly remarkable. It has been empirically developed to favor the initial temperature of deformation, i.e. the shrinkage starting temperature (SST), while taking into account HT as the initial temperature of extensive ash melting. The AFI shows that ashes obtained at higher ashing temperatures are less prone to deposit formation.

## 5. CONCLUSION

The ash fusion temperatures observed in the laboratory are commonly used to assess the tendency of fuels to form ash deposits. Although laboratory conditions do not always perfectly reflect conditions in industrial boilers, experimental AFTs are still a preferred tool for predicting potential slagging and fouling problems in real combustion applications. This study investigates whether varying the ashing temperature during sample preparation has an influence on the measured AFT values and, consequently, the prediction of ash deposition tendencies on heating surfaces. Based on the tests carried out, the following conclusions were drawn:

- Ashing temperature affects AFTs. SST and DT increase with increasing ashing temperature for all biomass ash samples. The HT and FT values for the ash of wood chips and maize straw are higher for the ash obtained at an ashing temperature of 550 °C than for the ash obtained at higher temperatures. At the same time, HT and FT for all ashes obtained at 710°C and 815°C increase with the ashing temperature.



- The ashing temperature has a significant influence on the ash yield –
- the ash yield decreases as the ashing temperature increases.
- The ashing temperature has an influence on the alkali content in the all ash samples – the alkali content decreases as the ashing temperature increases.
- As the ashing temperature increases, residual combustible matter burns more completely, leading to a decrease in the total carbon and hydrogen content in the ash. For all biomass ash samples, this effect is more pronounced at ashing temperatures of 550 and 710°C.
- Of the indicators used, the indicator based on the Cl content of the biomass is the mildest and all ashes show a low tendency to form deposits according to it. The situation is similar with DT, which shows only a medium tendency to deposit for maize straw ashes, while the others show a low tendency. The strongest is TA, where all ashes show a high tendency to deposit. The AFI indicator provides perhaps the most realistic prediction. Ashes obtained at ashing temperatures of less than 815 °C are more likely to form deposits.
- Of all biomass ash samples examined, maize straw ash showed the greatest tendency to form deposits.

The composition of the biomass ash, like the composition of the biomass, varies greatly and is determined by a variety of causes. Therefore, the results refer exclusively to the biomass samples examined in this work.

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