

WET TORREFACTION OF MISCANTHUS – CHARACTERIZATION OF HYDROCHARS IN VIEW OF HANDLING, STORAGE AND COMBUSTION PROPERTIES

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ABSTRACT

Properties of miscanthus hydrochars obtained through wet torrefaction were studied. The process was carried out in three different temperatures – 180, 200 and 220 °C and with four different ratios of water to biomass – 3:1, 6:1, 12:1 and 16:1. The obtained solid products were characterized with respect to their fuel properties. The best results were obtained for the temperature of 220 °C and showed a noticeable improvement in fuel properties – especially grindability and lowered ash content. The influence of water to biomass ratio was not so explicit and while high ratio showed an improvement in all mentioned properties, low ratio allowed to achieve the highest energy yield. The results obtained for miscanthus wet torrefaction and the literature data for dry torrefaction were compared.

Keywords: biomass, wet torrefaction, hydrochars.

INTRODUCTION

Contribution of renewable energy sources in the world energy production is consistently increasing. This is mainly due to two reasons – depleting fossil fuels resources and climate changes. Biomass, as one of the most widely utilized renewable energy sources, has an undoubtedly meaningful impact on production of the renewable energy, not only now, but in all likelihood also in the future [IEA, 2006]. It can be used for various biofuel supply chains, for both thermo- and biochemical processes, like combustion, gasification, pyrolysis, or fermentation [Kaygusuz, 2009]. Combustion is one of the simplest and best-known processes of the aforementioned. Hence, it is commonly used in power plants where biomass combustion and/or co-combustion takes place. However, introducing biomass processing into boiler operation is linked with a few problems. These involve transport and storage of

biomass that has a low volume and energy density and can easily decay. Moreover, biomass pulverization prior to combustion is far more difficult and inefficient when compared to coal, due to biomass fibrous structure [Esteban et al., 2006]. What is more, the biomass high content of alkalis and chlorine causes failures of boilers due to corrosion, fouling, and slagging [Hardy et al., 2012].

A solution to these problems is to seek pretreatment methods which improve biomass as a potential solid fuel for the thermochemical conversion purpose. Two distinct technologies were introduced: dry and wet torrefaction, both of which are based on a mild thermal pretreatment of the biomass.

Dry torrefaction, or just torrefaction, is carried out in a temperature range of 200–300 °C in an inert atmosphere [van der Stelt et al., 2011]. This process was deeply investigated and can be read about in greater detail in other sources [van der Stelt et al., 2011], [Bridgeman et al., 2008],

[Yan et al., 2009]. Although dry torrefaction gives satisfying results considering the increase of calorific value and improvement in grindability and hydrophobic nature of the product [Yan et al., 2009], wet torrefaction (also known as hydrothermal carbonization (HTC)) seems to have a few additional and meaningful advantages. In wet torrefaction, biomass is mixed with water. The process is carried out in a temperature of 180–250 °C [Pala et al., 2014] and under pressure caused by water vapors and evolved gases, reaching even 4.6 MP [Lynam et al., 2011]. Functioning of the HTC reactor is similar to an autoclave – thanks to high saturated pressure, even in a temperature above 200 °C water stays in a liquid phase. It is said that in these sub-critical conditions water has a high concentration of ions which have an influence on biomass transformation like acid or a base catalyst [Lynam et al., 2011]. It was stated that during wet torrefaction the key reactions are hydrolysis, condensation, decarboxylation and dehydration [Funke and Ziegler, 2010]. Biomass HTC results in three products: gases (mostly CO₂), aqueous chemicals (mainly primarily sugars and organic acids) and char (often called HTC biochar or hydro char) [Reza et al., 2013]. More detailed investigation over the nature of HTC was presented in many works [Funke and Ziegler, 2010], [Funke and Ziegler, 2011], [Stemann et al., 2013]. It is important to note that in contrast to dry torrefaction, HTC has the following advantages: it requires a lower temperature, it can be used for biomass with a high content of moisture that normally requires an intensive drying process, and it reduces ash content through washing it out with hot, compressed water [Pala et al., 2014]. Moreover, research shows that wet torrefaction was more successful when considering the improvement of energy densification in comparison to dry torrefaction under similar mass yields [Yan et al., 2009; Pala et al., 2014].

Few works were devoted to the characterization of biomass wet torrefaction products. This includes loblolly pine [Yan et al., 2009], agricultural residues [Oliveira et al., 2013], corn stalk and forest waste [Xiao et al., 2012] or eucalyptus sawdust and barley straw [Sevilla et al., 2011]. However, considering combustion and energy production on a commercial scale, miscanthus is seen as the most promising biomass in the European area [Iqbal et al., 2014; Meehan et al., 2014]. This energy grass is favorable due to its properties, e.g. high yields of dry matter, perennial growth, efficient use of nitrogen and water, and good pest

and disease resistance [Price et al., 2004]. Nevertheless, as biomass, miscanthus still retains the previously mentioned attributes that complicate its utilization. All these challenges seem possible to be overcome if wet torrefaction is introduced.

The objective of this work was to analyze the properties of miscanthus wet torrefaction hydrochar with an emphasis on the attributes that are important for solid fuel processing; these include typical characteristics like heating value, elemental analysis, ash content, but also grindability and resistance to deterioration.

EXPERIMENTAL

Material

Miscanthus in a form of approximately 3 cm length cuttings was kindly provided by Warsaw University of Life Sciences-SGGW in Skierniewice. Prior to the main experiments, biomass was dried in a drying oven until the samples' mass became stable. For the purposes of comparison all of the analyses that were done to characterize products were also conducted on raw miscanthus.

Experimental procedure

Wet torrefaction was carried out with the use of a setup presented in Figure 1. The main element of the setup was a reactor made of stainless steel. The reactor was filled with miscanthus and tap water with different mass proportions. Although demineralized water might have given better results considering the ash transfer, it would as well create more unnatural conditions – in commercial scale wet torrefaction should be carried out on biomass with a high content of moisture obtained from natural sources like rainfalls or ground water, what is more, tap water would be more economically justified. The basic water/biomass ratio was 12:1, but to evaluate the ratio impact on hydrochar properties some additional research with the ratios of 3:1, 6:1 and 16:1 were carried out. The biomass sample mass depended on the water/biomass ratio – for a ratio of 12:1 it was about 20 g. During all the experiments the water level in the reactor was kept constant (about 2 cm from the top of the reactor) while the biomass sample mass was adjusted accordingly to the ratio. After loading the reactor it was immersed in a hot fluidized bed oven. The experiments were conducted at three different temperatures: 180, 200 and 220 °C. The

average heating rate in all the cases was about 35 °C/min. The temperatures inside the reactor and fluidized bed oven were measured using thermocouples (shown in Figure 1). The maximum temperature was limited by the reactor's construction. With temperatures higher than those applied there was a risk that the vapors pressure would exceed 35 bar, which was the maximum safety pressure for the reactor. Moreover, sealing that was used in the reactor's construction didn't allow to rise the temperature to levels much higher than those applied. When a ratio different than 12:1 was applied the research was performed under the temperature of 220 °C. When the reactor reached a definite temperature it was held inside the fluidized bed for 10 minutes. The length of this time was determined by previous experiments and literature data that proved an insignificant influence of the time exceeding 10 minutes on the processed material parameters [Świątek, 2013], [Pala et al., 2014]. The pressure inside the reactor varied from 8 to 25 bar depending on the experimental conditions. The pressure inside the reactor was measured using a manometer.

Before opening, the reactor was cooled and the gases produced during the process were released and analyzed. The material was further dried in an oven at 105 °C for 24 h and stored for further analyses. Samples were named accordingly to the HTC temperature. If a water/biomass

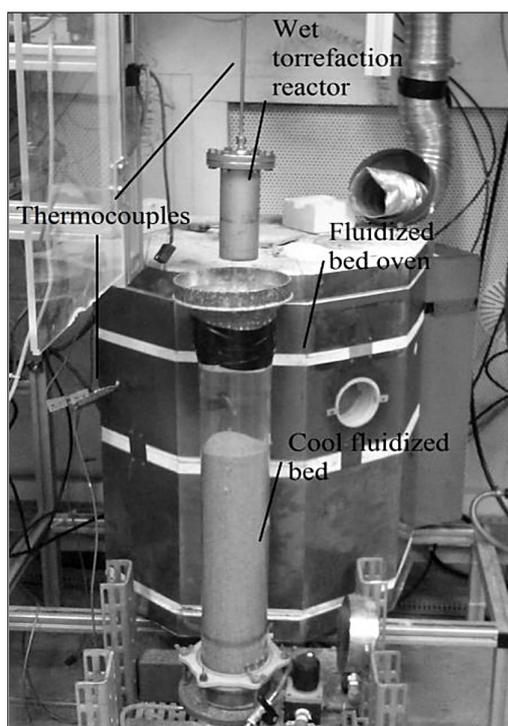


Figure 1. The wet torrefaction setup

ratio different than 12:1 was applied, then the ratio was also given at the end of the sample name.

Product characterization

The ash content analysis was conducted according to ASTM E1755. An elemental analyzer, Flash 2000 Organic Elemental Analyzer (Thermo Scientific), was used for determining C, H, N and O content. Samples with particle size of less than 0.125 mm and weight of approximately 0.5 mg were introduced into the analyzer.

Higher heating value was determined using calorimetric bomb (Ika C2000 Calorimeter system). Dried samples analyzed in the calorimetric bomb weighted from 0,30 to 0,46 g, depending on bulk density, and their particle size was in a range of 125–250 nm.

All of the analyses and experiments mentioned, were repeated at least twice. If the results varied significantly, additional measurements were conducted. All presented results are the average values.

Grindability tests were performed with the use of ball mill Planetary Mill pulverisette 5 classic line (Fritsch). Six big and six small balls, with diameters of 20 and 5 mm respectively, were used. Ground material was sieved with using a sieve shaker for 20 minutes. Sieves of the following mesh were utilized: 63, 125, 250, 355, 500, 800 and 1000 µm.

To identify gaseous products of wet torrefaction, the Micro GC chromatograph C2V-200 coupled with TCD detectors was used.

To determine resistance to biological deterioration and equilibrium moisture content of raw material and hydrochars, the samples were placed in a humidity chamber (Mettler HCP153). Humidity inside the chamber was set on 95% at a temperature of 30 °C. The samples weight was measured every day until it stabilized. To estimate the resistance of the material to biological deterioration traces of mold were sought. It was assumed that the later the mold appeared the more the material was resistant to deterioration.

In order to express severity of the process and energy stored in the processed material, two commonly used indicators [Yan et al., 2009], [Xue et al., 2014] were applied – mass yield (MY) and energy yield (EY). Additionally, ash yield (AY) was calculated as an indicator expressing the amount of ash left in the material after the torrefaction process. The indicators were calculated according to Eqs, (1), (2) and (3):

$$MY = \left(\frac{m_{tor}}{m_{raw}} \right)^{daf} \cdot 100, wt\% \quad (1)$$

$$EY = MY \cdot \frac{HHV_{tor}}{HHV_{raw}} \cdot 100, \% \quad (2)$$

$$AY = MY \cdot \frac{A_{tor}^d}{A_{raw}^d} \cdot 100, \% \quad (3)$$

where: m_{tor} – the mass of torrefied biomass,
 m_{raw} – the mass of unprocessed biomass,
 HHV – higher heating value,
 daf – stands for dry ash free basis,
 A^d – stands for ash content expressed on the dry basis.

RESULTS AND DISCUSSION

Temperature influence

According to the presented results, a clear tendency is noticeable (Table 1). With temperature increase mass yield gradually decreases. This is similar to results obtained by other researchers [Funke and Ziegler, 2010; Xiao et al., 2012]. Another parameter which correlated with the temperature is HHV. As expected, on the basis of scientific literature [Pala et al., 2014; Xue et al., 2014], with a temperature increase higher heating values also increased. However, this growth was insignificant until the temperature of 220 °C, when it reached about 7% changing HHV from approximately 18.8 to 20.1 MJ/kg. With a rapid decrease of mass yield and slow growth in HHV according to the temperature the energy yield also decreased reaching a level of 0.74 in 220 °C.

The HHV increase resulted from changes in a chemical composition. As presented in Table 2, the carbon content increased from 47.10 to 50.11%. At the same time oxygen content decreased from 46.62 to 43.53%. These results are partly consistent with the fact that the gas chromatograph detected only CO₂ in the emerged gases (excluding N₂ and O₂ from air) with no CO, H₂

or CH₄. Changes in hydrogen and nitrogen were infinitesimal and they showed no clear tendency, however, it should be noticed that hydrogen could have been released within condensable compounds. Moreover, the last statement may also apply to other elements (C, O), especially considering wet torrefaction products like acetic acid and other organic acids [Lynam et al., 2011]. With this composition O/C ratio changes from 0.74 to 0.65, and H/C ratio changes from 1.54 to 1.46. Although these changes are noticeable, the final results place the HTC product nowhere closer to the more carbonaceous materials like peat or lignite when considering the van Krevelen Diagram [Pala et al., 2014].

Some interesting results were obtained due to the ash content analysis (Table 1). The ash content decreased with the temperature. Moreover, this decrease was significant and for the temperature of 220 °C it reached 53 %, changing the ash content from 2.11 to 1.00 %. When matched with the mass loss, it results in the ash yield reaching 0.33. Although no investigation of ash composition was conducted, some promising results, which involve washing out of potassium, were presented elsewhere [Reza et al., 2013].

Equilibrium moisture results (Table 1) were in concord with the information that HTC changes material structure into a more hydrophobic one, but in our case the equilibrium moisture content decreased in a very limited range, from 20.42 to 19.28%. This might be explained by a limited decomposition of the feedstock's components – during the decomposition hydrophilic COOH⁻ and OH⁻ groups are removed.

Resistance to biological deterioration did not change in the case of raw material and material processed in 180 °C and 200 °C – in all of these cases traces of mold appeared after 8 days. For the material obtained in 220 °C no mold was noticed up to 12 days. No further observations were conducted due to the moisture chamber malfunction. It is important to note that these observations

Table 1. Results of the analyses for raw and processed material

Samples	MY	HHV [MJ/kg]	ED	EY	A ^d [wt%]	AY	EM	RtD [days]
Raw	1.00	18.799	1.00	1.00	2.11	1.00	20.42	8
180	0.94	19.119	1.02	0.96	1.39	0.61	20.24	8
200	0.82	19.251	1.02	0.84	1.34	0.52	19.28	8
220	0.70	20.099	1.07	0.75	1.00	0.33	19.46	>12

ED – energy densification as a ratio of HHV_{tor} to HHV_{raw}, A – ash, AY – Ash yield, EM – equilibrium moisture, RtD – resistance to biological deterioration, d – on dry basis

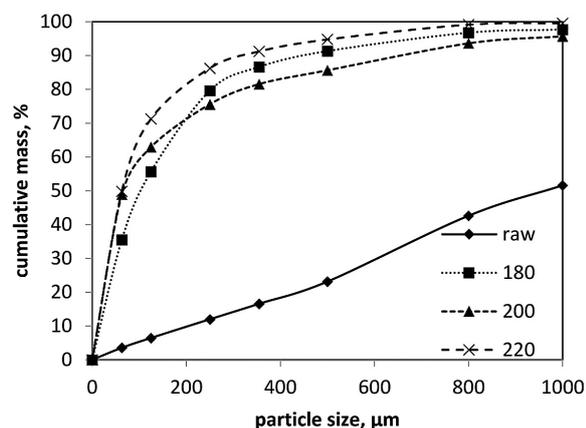
Table 2. Ultimate analysis with O/C and H/C atomic ratios

Sample	C ^{daf} [wt%]	H ^{daf} [wt%]	N ^{daf} [wt%]	O ^{daf} [wt%]	O/C	H/C
Raw	47.10	6.04	0.24	46.62	0.74	1.54
180	47.56	6.12	0.21	46.11	0.73	1.54
200	48.43	6.09	0.24	45.24	0.70	1.51
220	50.11	6.09	0.27	43.53	0.65	1.46

C – carbon, H – hydrogen, N – nitrogen, O – oxygen, O/C – oxygen to carbon atomic ratio, H/C – hydrogen to carbon atomic ratio, daf – on dry ash free basis.

did not follow any normative or widely accepted method, they were simple visual examination and their purpose was to give an overall insight into the changes in wet torrefied material resistance to deterioration.

Figure 2 summarizes the results of the sieve analysis. As it is shown, the grindability of torrefied biomass changed significantly. This showed that due to wet torrefaction the fibrous structure of biomass was broken down and thus the grinding process might have been carried out more efficiently and at a lesser cost. Although these results seem very promising, they could be more reliable by implementing more appropriate and standardize method, like Hardgrove Grindability Index (HGI), and comparing them with coal grindability analysis [Bridgeman et al., 2010]. In addition, some inconsistency might be seen in results obtained for sample torrefied in 200 °C. It seems reasonable and intuitive that the dependence be-

**Figure 2** Sieve analysis for wet torrefaction of miscanthus in different temperatures

tween temperature and grindability should be linear, thus the sample obtained in higher temperature should give a higher contribution of lesser particles, however, examples of samples obtained in 180 and 200 °C are in opposite to this statement. It is hard to explain, especially when more detailed research states differently [Bridgeman et al., 2010], and probably might be caused by not perfectly fitted methodology. Nevertheless, presented results clearly show a great improvement in grindability of biomass due to wet torrefaction.

Water to biomass ratio influence

Results summarizing the influence of biomass/water ratio are presented in Table 3. It is shown, that due to limited washing out caused by the water scarcity, the ash content increased with reducing the ratio. Nevertheless, the overall conclusion is that applying the ratio of 6:1 or greater resulted in washing out ash and decreasing the ash content. The other way round, when the ratio was too low, about 3:1, almost no ash was washed out ($AY = 0.97$) and the ash content increased due to mass reduction.

The mass yield of the processed material increased with a decreasing ratio. Matching this with the higher heating value, which did not change significantly with the water/biomass ratio, the energy yield is the highest for the ratio of 3:1.

The equilibrium moisture analysis showed a clear tendency for increased water capacity when the water to biomass ratio was low. This might be the result of increased porosity due to a greater amount of emerging gases.

Table 3. Results of the analyses for different water to biomass ratios

Samples	MY	HHV [MJ/kg]	ED	EY	A ^d [wt%]	AY	EM	RtD [days]
220-3:1	0.80	20.212	1.08	0.87	2.54	0.97	25.10	12
220-6:1	0.79	20.103	1.07	0.84	1.58	0.59	23.64	8
220-12:1	0.70	20.099	1.07	0.74	1.00	0.33	19.46	>12
220-16:1	0.68	20.130	1.07	0.72	1.25	0.40	16.13	>12

Considering resistance to biological deterioration, the results show that in the case of 3:1 ratio the material lasts a little longer without any traces of mold than the raw material, but still it was sooner than in the case of 12:1 and 16:1 ratios. The sample that was processed with a ratio of 6:1 showed no improvement when compared to raw material.

The sieve analysis (Figure 3) showed that the grindability of the material was improved in each case. The highest improvement was achieved for a ratio of 16:1. Ratios 16:1, 12:1 and 6:1 show similar contribution of dust with a particle size less than 63 μm, but with a larger mesh size the ratio of 6:1 gives much worse results – even when compared to the ratio 3:1. In this case, inconsistency in water to biomass ratio and grindability dependence doesn't have to be caused by chosen methodology but might be actually a result of applied water/biomass ratio. This case requires more detailed research to be solved and clarified.

To conclude, it was shown that two extreme ratios are profitable but each in a different way. With a small water to biomass ratio (3:1) material showed the highest EY but also high ash content and equilibrium moisture. On the other hand, the material that was torrefied with a high water to biomass ratio showed the lowest energy yield but

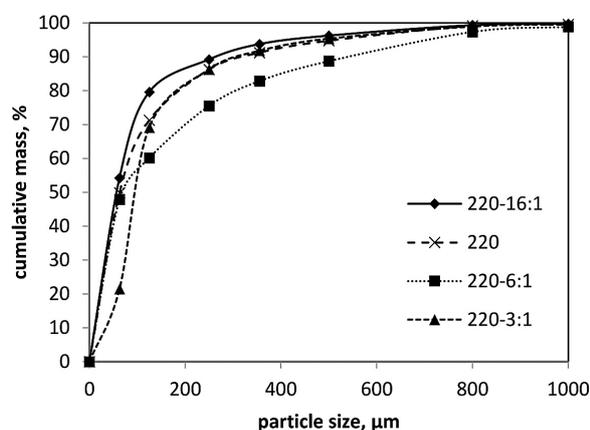


Figure 3. Sieve analysis for wet torrefaction of miscanthus with different water to biomass ratios

the greatest improvement, considering ash content, equilibrium moisture, or resistance to deterioration. The ratio did not influence the higher heating value in a noticeable way. The sample with a ratio of 6:1 showed the poorest quality.

Comparison of wet and dry torrefaction of miscanthus

Table 4 summarizes results mentioned in this article and those obtained by [Xue et al., 2014] in similar conditions. The results showed that applying dry torrefaction allows reaching a higher mass yield. On the other hand, hydrochar showed better properties with respect to HHV, elemental composition and ash content, even though the material was processed in the temperature 10 degrees lower. Furthermore, the processed material had a different moisture content. Although dry torrefaction requires a drying process before the material can be processed further, wet torrefied material must be dried after the process. In our research the obtained material had moisture content at a level of 80% (on a wet basis), making the drying process essential. However, it is worth to mention that the moisture content of freshly harvested miscanthus may reach 70% [Meehan et al., 2014]. On the whole, it is hard to say which of these processes is favorable and this is not the goal of this work. The choice of the process should be determined by the properties desired to achieve and some deep economic analysis including the cost of heating for the process purpose and drying costs.

CONCLUSION

Wet torrefaction process has shown to be a promising way for solid biomass improvement. It results in energy densification, ash content and equilibrium moisture reduction, increased resistance to biological deterioration and improved grindability, but to achieve these changes a temperature of minimum 220 °C is required. One of

Table 4. Comparison of chars obtained due to dry and wet torrefaction

Sample	MY	HHV ^d [MJ/kg]	EY	A ^d [wt%]	Ultimate analysis [wt%]			
					C ^d	H ^d	N ^d	O ^d
Raw*	1.00	18.360	1.00	4.47	47.63	6.19	0.40	45.78
230*	0.92 ^{daf}	19.101	0.96 ^{daf}	4.35	48.68	6.13	0.37	44.82
Raw	1.00	18.799	1.00	2.11	47.10	6.04	0.24	46.62
220	0.70	20.099	0.74	1.00	50.11	6.09	0.27	43.53

* – results obtained in work of Xue et al. [Xue et al., 2014].

the most significant improvements, which dry torrefaction lacks, is a great reduction of ash content. The authors believe that implementing higher temperatures should produce better results that would make the product resemble coal more and at the same time, allow for ash content reduction. Another important factor, that had a meaningful impact on the processed material during wet torrefaction, is the water to biomass ratio. The results showed that decreasing this ratio resulted in an increased energy yield but at the same time other parameters, like grindability, ash content or equilibrium moisture became worse than in a case of a high ratio. Optimization of this parameter should be a subject of further and more detailed investigation.

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