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Autohydrolysis of White Birch (*Betula papyrifera*) and Poplar (*Populus*) in laboratory batch digester; characterization of solids for bioenergy and/or subsequent conversions

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ABSTRACT The pretreatment of biomass to subsequently facilitate water removal processes or to reduce hygroscopic properties could be beneficial to maintain optimal calorific value, to stabilize geometry of feedstock particles or to reduce biodegradation susceptibility. Such improvements could be worthwhile for some bioenergy applications or subsequent conversions along a biorefining approach. The present study targets the effect of pretreating White Birch (*Betula papyrifera*) and Poplar (*Populus*) wood chips by autohydrolysis in a 6L batch recirculating digester (M/K^{TM} Systems inc.) operated at temperatures from 140 to 170 °C and over time range between 60 to 180 min.. The effect of these operating conditions were related to three main feedstock characteristics: the calorific value (HHV), the equilibrium moisture content (EMC at 23°C, 50% relative humidity) and the water retention value (WRV). For Poplar, a significant increase of $HHVs_{avg}$ was observed following treatments with respect to non-treated biomass; at temperature conditions from 150-170 °C. For both types of pretreated feedstock, respectively, all EMC values averaged to a single EMC value was significantly lower compared to non-pretreated feedstock EMC value. In addition, significant relationships were obtained between EMC results and severity index variation; for both feedstocks. The treated White Birch resulted in a significant increase of all $WRVs_{avg}$ obtained, compared to the non-treated feedstock.

Keywords: Autohydrolysis, recirculation digester, Calorific value, Equilibrium moisture content, Water retention value.

INTRODUCTION Autohydrolysis of wood chips is recognized as a possible pretreatment step before pulping processes such as Kraft pulping process or chemi-thermomechanical pulping (CTMP) step in pulping industries (Yoon and Heiningen, 2008 and Hou *et al.*, 2014). Kraft pulping, which makes uses of chemicals such as sodium hydroxide (NaOH) and sodium sulfide (Na_2S), leaves a "clean" cellulose for pulping process by removing lignin, but where hemicelluloses are degraded into a mixture of sugar acids hard to separate into value-added products (Yoon and Heiningen, 2008). Chemi-thermomechanical pulping generally consist in a series of steps where chemicals such as sodium sulfite (Na_2SO_3) and sodium hydroxide (NaOH) are impregnated in wood chips in order to subsequently mechanically defiber the wood chips along pulp and paper production (Hou *et al.*, 2014). Prior to Kraft pulping or CTMP, extraction of hemicelluloses at good selectivity level can be conducted using the autohydrolysis pretreatment (where reactants are water and wood chips only). Autohydrolysis is reported as a potential process integration along a forest biorefining approach, where, from hemicelluloses, hexose and pentose sugars can be obtained and converted into a variety of chemicals (Xu *et al.*, 2016, Hou *et al.*, 2014 and Yoon and Heiningen, 2008). Most importantly, autohydrolysis conducted at temperatures below 230 °C was shown to leave the solid and its cellulose fiber in good conditions, therefore subsequent pulping process may be applied in the remaining solids, even more effectively. Because the chemicals are better impregnated, it may lead into better pulp preparation for the pulp and paper industry (Xu *et al.*, 2016 and Garrote *et al*., 2002).

In an integrated biorefining approach it could also be interesting to use the solid product for a co-application beside pulp and paper production. Bioenergy, other solid biobased products or subsequent conversion of the solid could be desired along a biorefining platform. Xu *et al.* (2016) reported that autohydrolysis could partially remove extractives, hemicelluloses and lignin, which in turn, could affect structure, swelling and porosity of wood chips and resulting in a possible improvement fluid capillary effect and capacity of absorbing water. The same authors who treated poplar by autohydrolysis observed a decrease in contact angle (water droplet on treated poplar) for an increasing severity of autohydrolysis conditions. The change in wood chips surface chemical composition was reported as a potential reason of that observation (Xu *et al.*, 2016).

Pretreatment of biomass in order to improve the process of drying or water removal, as well as to reduce hygroscopic properties of wood chips for an optimal calorific value, geometric stability of particle and reduction of biodegradation susceptibility (due to moisture level reduction) is a step which could be worthwhile for solid biofuels or for subsequent conversion of the solid.

The present study targets the effect of autohydrolysis leaching conditions on the solids during the digestion process. Value addition potential of processed solids for bioenergy application or feedstock for subsequent conversions is evaluated along this study mostly for three main characteristics: the calorific value (HHV), the equilibrium moisture content (at 23°C, 50% relative humidity) and water retention value of two biomass types, White Birch (*Betula papyrifera*) and Poplar (*Populus*).

MATERIALS AND METHODS

Biomass Wood chips used along this study are White Birch (*Betula papyrifera*) and Poplar (*Populus*) from two regions of the Province of Quebec, Canada; obtained from John Lewis plant in La Tuque, Mauricie (QC), Canada (harvested by Rémabec inc.) and Fortress Cellulose, Thurso, Outaouais (QC), Canada, respectively. Wood chips dimension classification was done using a Domtar laboratory apparatus, followed by manual removal of bark pieces. Only wood chips of 2-6 mm thickness and surface up to 40X40mm were selected for the leaching digester process study. Wood chips were stored at 4-5 °C until being used for process experiments.

Autohydrolysis-leaching-digestion process The M/K^{TM} Systems inc. laboratory digesters utilized for the experiment are dual 6 liters capacity of reactor per batch. Per experiment, 4000 g of water for 250 g of dry wood chips were used, which corresponds to a water-biomass ratio of 16:1. During the heating phase, initial air contained in the reactor was allowed to escape by using water vapor pressure during quick valve opening.

Equilibrium moisture content For the equilibrium moisture content test, 100g of each wood chips samples (processed or non-processed) were spread on thin layer in aluminum pan and in a controlled environment room at 23 °C and 50% relative humidity (R.H.) for about 10 consecutive days, until reaching stable mass with respect to time. Two samples of about 7.5g were taken from each pan and put at 105 °C for a minimum of 24h, in order to determine their respective humidity content.

Size reduction Wood chips samples remaining from controlled environment drying and left at equilibrium moisture content were used for further characterization. The remaining samples were first milled using Thomas-Wiley Laboratory Mill (Model 4, #3375-E15) setup with a 2mm grid and operated during 1 minute per sample. Milled portion was sieved using a Rotap apparatus for 4min using 60, 45, 20 and 10 mesh size sieves. Each fraction was weighted and kept for subsequent analyses.

Water retention value For water retention value determination (PL-048,1996), 1.2g of the 10-20 mesh fraction was soaked in demineralized water during more than 12 hours. After, samples were placed into cylinders equipped with removable bottom made of wire mesh (140 mesh no.) and adapted to be inserted into a centrifuge apparatus. Samples were centrifuged at 2500 rpm for 12 min and remaining samples humidity content was determined by drying at 105 °C for a minimum 24h. The humidity content corresponded to the water retention value. Each analysis was conducted in duplicates.

Calorific value Higher heating value was determined for each sample in duplicates using the 45-60 mesh fraction obtained from size reduction. About 1 g of sample was used per analysis. Powder material was first pelletized and subsequently analyzed using an automatic Parr bomb calorimeter (model 6400 - Automatic Isoperibol Calorimeter). Humidity content of biomass was also determined in order to get the HHV on a dry basis using Equation 1.

$$HHV_{dry} = \frac{HHV_{humid}}{1-Humidity} \quad (1)$$

Ash content Wood chips were analyzed for their ash content using ASTM method E1755-01. About 0.5g of each sample was heated at 275 °C during 30 min followed by a temperature increase at 575 °C. Temperature was maintained at this level for a minimum of 6 hours.

Statistical analysis All Tested conditions in laboratory digester were conducted once. Therefore comparisons were conducted on a factor basis confounding other specific conditions (For examples: T1 *vs.* T2 for operating times confounded or no treatment *vs.* treatments averaged). Statistical comparison among factor operating conditions were done by analysis of variance and by Student T-test.

In order to analyse the studied values (HHV, equilibrium moisture content and water retention value) with respect to temperature and residence time at operating T, these two factors were combined into the severity index (Equation 2). In Equation 2, "t" corresponds to residence time (in minutes) at T operation and "T" to temperature (in °C) (Overend and Chornet, 1987 and Lavoie *et* *al*., 2010). Simple regression or 2nd degree polynomial relationships applied to studied responses (HHV, EMC and WRV) with respect to the severity index were analyzed for their signification levels by ANOVA using JMP® (SAS).

$$S = \log_{10} \int_0^{t(min)} e^{\frac{T-100}{14.75}} dt \qquad (2)$$

RESULTS AND DISCUSSIONS

Higher heating value Firstly, starting by looking at the Birch wood chips and the effect of the treatments on the HHV, all treatments averaged in comparison to the raw feedstock, the HHV does not increase significantly following treatments for all temperatures and times confounded (Figure 1). In addition, no significant difference was observed between averages of results regrouped by temperatures (for time confounded at a same temperature). For specific condition of 150 °C-180min, 160 °C-120min and 170 °C-60min, the HHV obtained are more elevated than HHV of raw wood chips (Figure 1 and Table 1). Nonetheless, replicates on these specific conditions would be needed in order to corroborate this observation, especially since neighboring values obtained at similar severities do not appear more elevated (Figure 1 and Table 1).

HHV results of Poplar wood chips differ from the Birch ones. For all the Poplar wood chips treated between 150 and 170 °C, their average is significantly higher than raw biomass HHV (19.93 *vs.* 19.49 MJ/kg, treated *vs.* non-treated, respectively ; p<0.03). However, no significant difference was observed among all HHV of Poplar chips when 140 °C wood chips are considered within ANOVA calculation. Analyzing the effect of temperature on Poplar HHV among all studied temperatures (at confounded time conditions for a same temperature), only HHV results for conditions combined at 160 °C *vs.* no treatment were significant (p<0.04).

In Figure 2, it can be observed that as severity increases, the HHV of treated wood chips seems to increase as well. Both models are significant at p<0.03 and 0.02 for Birch and Poplar respectively. However, the linear relationship applied to actual experimental values is weakly linear (R^2 of 0.43 and 0.49 for Birch *vs.* Poplar value respectively; Figure 2).

The increase in HHV observed following hydrolysis treatment could be partly due to hemicelluloses released, degradation and losses. Hemicelluloses of Birch and Poplar being formed of components containing oxygen (Girard, 1992 and Royer *et al.* 2010), a loss of these components as well as all compounds segments containing oxygen could then lead to carbon concentration, resulting in a HHV increase.

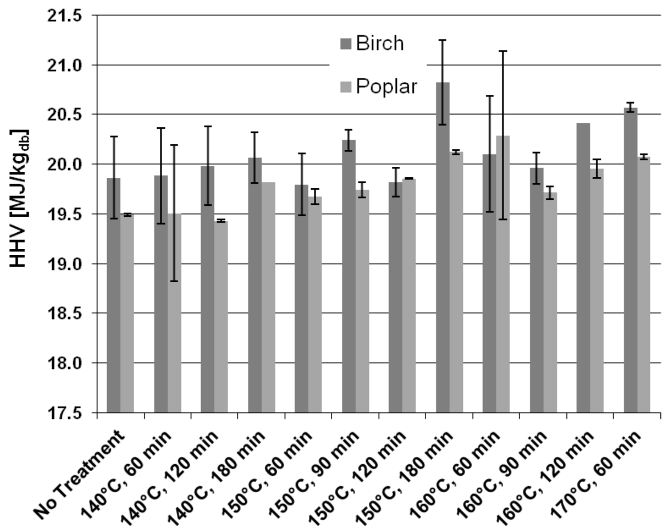

Figure 1. Birch and Poplar higher heating values before and after cooking in M/K™ 6L leaching digesters. (n.b.: Error bars correspond to standard deviation).

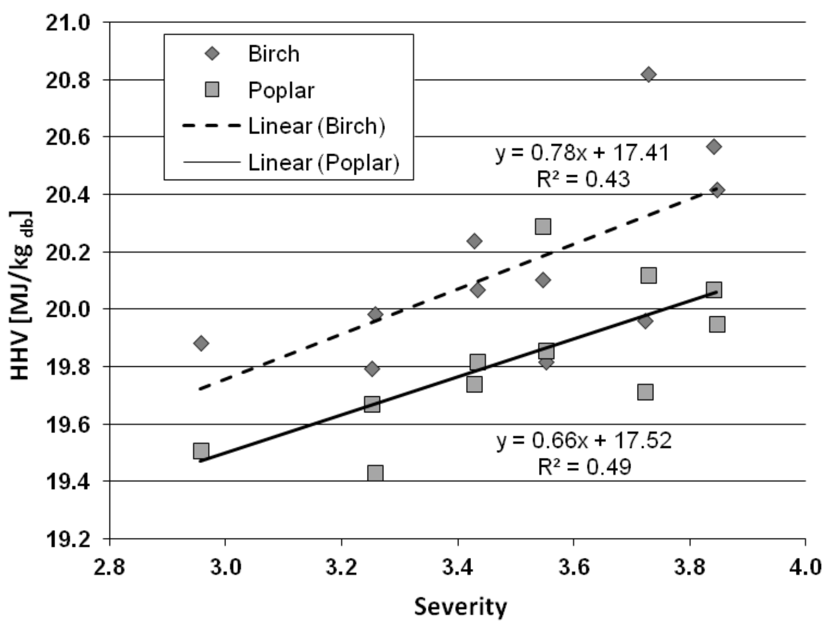

Figure 2. Birch and Poplar wood chips HHV in function of severity operating conditions in leaching digesters.

Equilibrium moisture content and water retention value Equilibrium moisture content results (at 23 °C and 50% R.H.) for Birch and Poplar seem to decrease as temperature and operating times increased (Figure 3). On the other hand, water retention value showed almost the inverse of this behavior, at least for some specific conditions, especially for Birch (Figure 4). These two observations are analysed in more details with statistical analysis in the following sections.

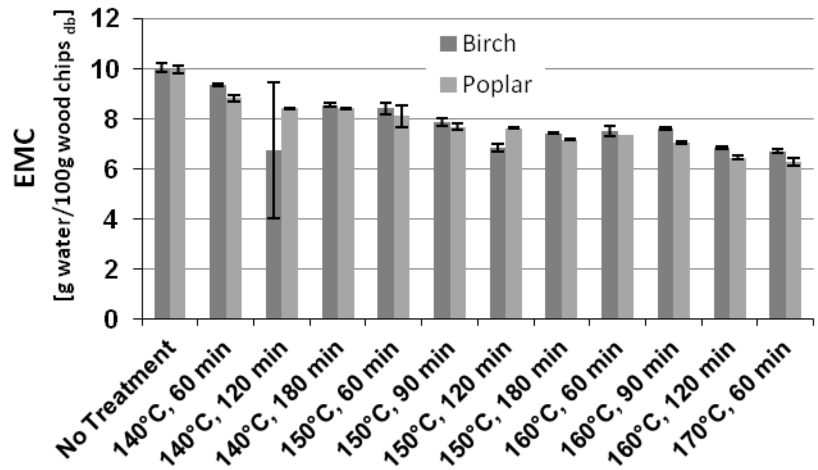

Figure 3. Equilibrium moisture content (at 23 °C, 50% R.H.) of Birch and Poplar wood chips processed by autohydrolysis in M/K™ 6L leaching digesters. (n.b.: Error bars correspond to standard deviation).

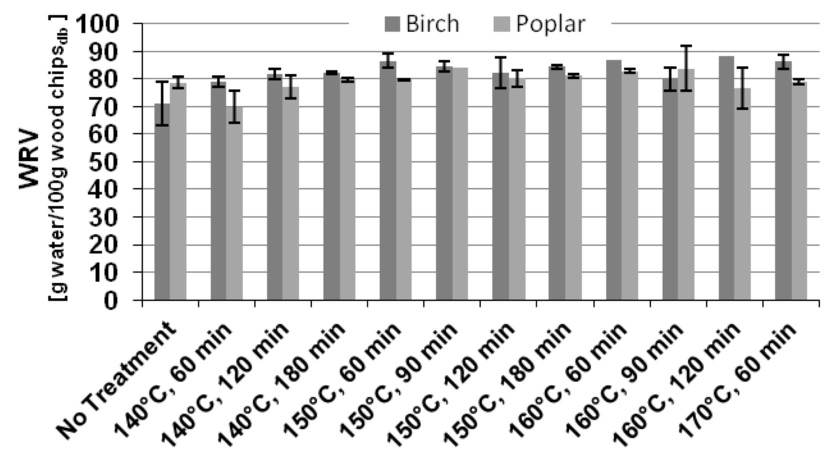

Figure 4. Water retention values of Birch and Poplar wood chips processed by autohydrolysis in M/K™ 6L leaching digesters. (n.b.: Error bars correspond to standard deviation).

Equilibrium moisture content and water retention value - Temperature effect. Concerning equilibrium moisture content (determined at 23 °C, 50% R.H.), the average value of all treated solids is significantly lower than the EMC of raw wood chips and this, for both wood types studied (7.6 *vs.* 10.0 and 7.6 *vs.* 9.9 g water/100 g wood chips $_{d.b.}$, for treated *vs.* raw, Birch and Poplar respectively); significant at p<0,00015 and 0.0025 respectively. In addition, in this case, only for Poplar, significant differences are found among operating temperatures investigated during the process (for time tested confounded). These significant differences where EMC decrease for a temperature increase are found between 140-170 °C, 140-160 °C, 150-170 °C, 140-150 °C et 150-160 °C and are significant at values of p<0.0015, 0.0015, 0.015, 0.02 and 0.05 respectively.

The biomass hygroscopic properties involved in the EMC, is generally explained by the functional groups creating hydrogen bound with water. The hemicelluloses contain these molecular sites (or functional groups) (Célino *et al.*, 2014). Conversions in leaching digesters which enhances hemicelluloses losses and the release of some functional groups could then explain a lower humidity content found at equilibrium after drying at 23 °C, 50% R.H..

As mentioned in introduction of this section, an inverse behavior of the water retention value with respect to EMC may be observed for Birch in Figures 3 and 4. In case of all digester treatments water retention values averaged for Birch specie, the average value is significantly higher than raw wood chips 83,8 *vs.* 71,1 g water/100g of chips, processed *vs.* raw respectively at p<0,00045. This difference does not appear in case of Poplar. Nothing was observed neither when comparing treatments regrouped by temperatures (for all time confounded), for Birch and Poplar. This increase in water retention value observed for the Birch wood chips could be consistent with observations made by Xu *et al.* (2016) where autohydrolysis pretreatment led to a decrease in contact angle (i.e.: water droplet sinking more on treated wood chips at higher severities).

Concerning the water retention value enhanced for Birch following autohydrolysis treatment, in terms of thermochemical bioenergy technical applications (i.e.: gasification or combustion), the inverse situation could have been desired. Drying being an energy intensive treatment, mechanical dehydration pre-treatment (e.g.: centrifugation) targeting the removal of maximum possible water content could be desired. Continuing the investigation of the wood chips characteristics following similar leaching treatments would then be relevant, especially if a condition leading to a lower water retention value than raw biomass was found (such as at around 140 °C-60min for Poplar where WRV appears lower than non-treated biomass; Figure 4). Equilibrium moisture content did decrease after treatment (all treatments confounded). It is however possible to note from the actual difference between EMC and water retention values that mechanisms leading to these two respective properties seem different and could be investigated further.

Equilibrium moisture content and water retention value in function of severity index.

Expressed in function of severity index ("S" value), the values of EMC and water retention can then be analyzed with respect to one parameter regrouping temperature and time. In case of Birch, the application of a simple regression (linear model) to EMC as well as to water retention value in function of S leads in both cases in poor linear relationship; 0.49 *vs.* 0.25 respectively (Figure 5). However, in case of EMC, despite the fact R^2 value is really low (0.49), the linear model shows a decrease of EMC in function of increasing "S", which is significant at p<0.02. In case of Poplar, a linear model applied to EMC in function of S leads to a decrease in EMC values in function of the severity index "S" (Figure 6). The model has a R^2 of 0.88 and is significant at p<0.001. This result supports the significant differences for equilibrium moisture content with respect to raw biomass and between common operating temperature conditions averaged (time effect confounded); results presented in previous section.

Still for Poplar, but for water retention values, the simple regression applied to values obtained leads to very poor R^2 (0.26) and linear model is not significant (Figure 6). Nonetheless, a 2nd degree polynomial adjustment leads to a R^2 of 0.81 and the model is significant at p<0.0015. Following the 2nd degree polynomial model, the water retention values would increase up to maximum severity where it would start to decrease (from S=3.84 and 3.85; corresponding to 170 °C-60min et 160 °C-120min, respectively). In addition, it should be noted that at "S" = 2.96 (corresponding to 140 °C-60min), this minimum "S" value among tested conditions is the only value which is lower than the initial biomass; only being observed in the case of Poplar. It would be relevant to replicate that specific condition along future tests in order to confirm whether this observation holds. In case where it would hold, this could be of technical application interest, especially as a pretreatment before mechanical dehydration.

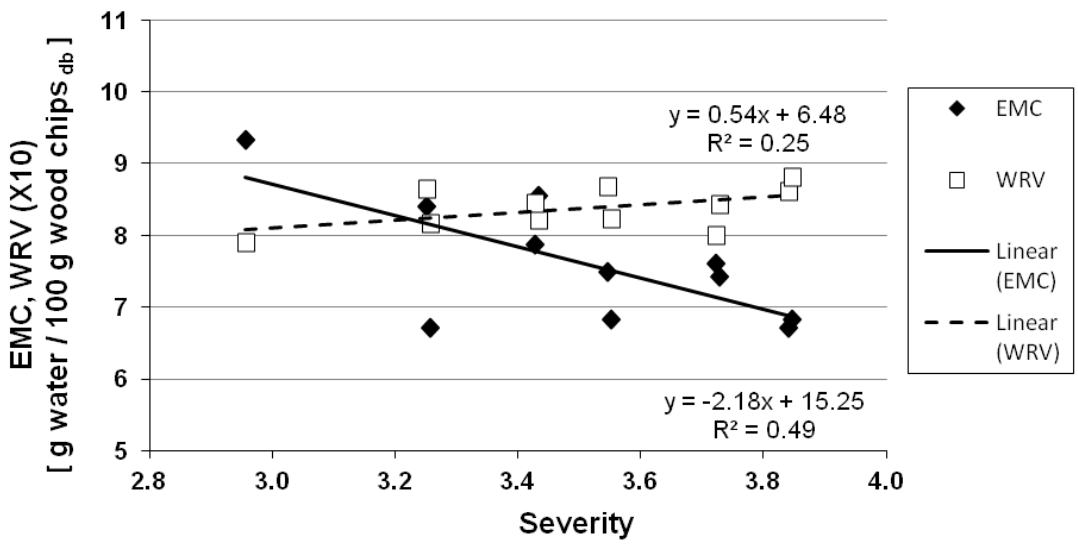

Figure 5. EMC (at 23 °C; 50% R.H.) and water retention values for Birch samples in function of severity index "S" (Table 1).

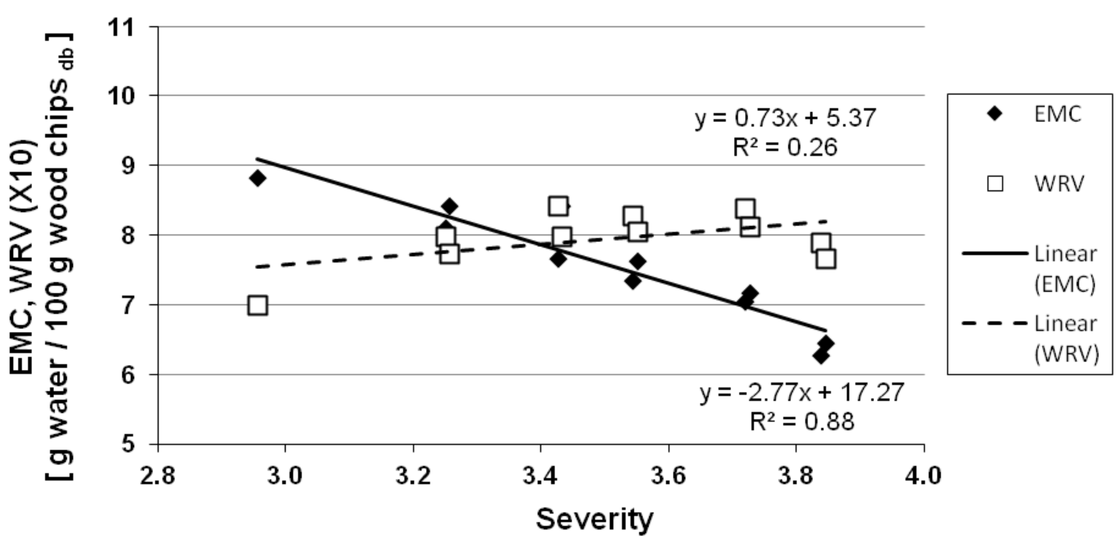

Figure 6. EMC (at 23 °C; 50% R.H.) and values of water retention for Poplar samples in function of severity index "S" (Table 1).

Ash Content Ash content results obtained for raw Birch and Poplar wood chips were <1%; therefore below detection limit. As a result, ash analysis were not conducted on solids treated by leaching digester. Other biomass types of plant sections having initial higher ash content targeted for bioenergy application and facing ash thermochemical problems (clinkers, fooling, slagging), should then be investigated in some of the actual conditions studied for similar characteristics (EMC, WRV, HHV and ash content); especially since ash content was shown to be significantly reduced within that type of reactor before (Pilon *et al.*, 2014).

Table 1. Results for HHV, EMC and WRV values for Birch and Poplar samples.

Conditions			Yield		Higher heating value		Equilibrium moisture content (at 23 °C; 50% R.H.)		Water retention value	
Temp. (°C)	Time (min)	Severity index	Birch (g solids/ 100g chips$_{d.b.}$)	Poplar (g solids/ 100g chips$_{d.b.}$)	Birch (MJ/kg$_{d.b.}$)	Poplar (MJ/kg$_{d.b.}$)	Birch (g water/100g chips$_{d.b.}$)	Poplar (g water/100g chips$_{d.b.}$)	Birch (g water/100g chips$_{d.b.}$)	Poplar (g water/100g chips$_{d.b.}$)
No treat.	--	0	100.0	100.0	19.87 ± 0.42	19.49 ± 0.01	10.02 ± 0.17	9.94 ± 0.16	71.08 ± 7.88	78.63 ± 2.12
140	60	2.96	97.7	98.3	19.88 ± 0.48	19.51 ± 0.68	9.33 ± 0.04	8.81 ± 0.14	78.96 ± 1.68	69.89 ± 5.63
140	120	3.26	90.4	94.8	19.98 ± 0.39	19.43 ± 0.01	6.72 ± 2.72	8.40 ± 0.03	81.67 ± 1.74	77.17 ± 4.13
140	180	3.43	92.4	92.2	20.07 ± 0.26	19.82 ± 0.00	8.54 ± 0.10	8.40 ± 0.03	82.12 ± 0.38	79.68 ± 0.68
150	60	3.25	93.6	92.3	19.80 ± 0.31	19.67 ± 0.08	8.40 ± 0.23	8.10 ± 0.43	86.51 ± 2.57	79.73 ± 0.21
150	90	3.43	95.0	89.6	20.24 ± 0.10	19.74 ± 0.08	7.86 ± 0.17	7.66 ± 0.13	84.47 ± 1.68	84.04 ± 0.09
150	120	3.55	86.6	88.6	19.82 ± 0.14	19.86 ± 0.00	6.82 ± 0.16	7.62 ± 0.01	82.35 ± 5.53	80.29 ± 3.03
150	180	3.73	79.7	85.4	20.82 ± 0.43	20.12 ± 0.02	7.42 ± 0.05	7.16 ± 0.03	84.28 ± 0.83	81.11 ± 0.62
160	60	3.54	86.8	86.1	20.10 ± 0.58	20.29 ± 0.84	7.48 ± 0.21	7.34 ± 0.02	86.83 ± 0.18	82.74 ± 0.71
160	90	3.72	79.6	84.9	19.96 ± 0.16	19.72 ± 0.06	7.60 ± 0.07	7.03 ± 0.07	79.93 ± 4.10	83.75 ± 8.08
160	120	3.85	87.6	80.3	20.42 ± 0.00	19.95 ± 0.09	6.82 ± 0.04	6.44 ± 0.07	88.10 ± 0.00	76.52 ± 7.37
170	60	3.84	78.9	81.6	20.57 ± 0.05	20.07 ± 0.03	6.70 ± 0.05	6.26 ± 0.14	86.17 ± 2.51	78.81 ± 0.89

Legend: Temp.: Temperature, treat.: Treatment, d.b.: dry basis, R.H.: relative humidity. N.b.: "±" is standard deviation.

CONCLUSIONS This study about the autohydrolysis using laboratory leaching digester, an apparatus usually used in pulp and paper industry, was investigated within another or a complementary application; i.e. for bioenergy application or other subsequent various transformations ("biorefinery" approach).

Concerning the HHV of Poplar wood chips, the average of all treatments done from 150 to 170 °C compared to raw Poplar chips HHV was found to be significantly higher; 19.93 *vs.* 19.49 MJ/kg, treated *vs.* raw, respectively; p<0.03. This difference is not statistically significant (for $\alpha = 0.05$) when 140 °C treatments are considered among the treatments' solid HHV averaged. Nonetheless, in case of Birch HHVs, no significant difference was observed for the same comparisons. In addition, linear relationships applied to higher heating values (HHVs) in function of severity index leads to linear models (significant at p< 0.03 and 0.02 respectively) where HHV would increase in function of "S". However, both models have poor R^2 values (0.43 and 0.49 respectively).

All treated solids EMC (at 23 °C; 50% R.H.) values averaged (from 140 to 170 °C, all time confounded) were significantly lower than EMC of their respective raw wood chips (7.6 *vs.* 10.0 and 7.6 *vs.* 9.9 g water/100 g chips $_{d.b.}$, for treated *vs.* raw, Birch and Poplar respectively); significant at p<0,00015 and 0.0025 respectively. A linear relationship applied to EMC values in function of "S" shows that EMC decreases in function of "S" increases, for both tree species; in both cases it is significant at p<0.02 and 0.001, with R^2 of 0.49 and 0.88 for Birch and Poplar respectively.

For the water retention values, only Birch WRV averaged together (all treatments confounded) presented a significant difference with respect to raw biomass (83.8 *vs.* 71.1 g water/100g of treated Birch chips, transformed *vs.* raw; at p<0,00045).

Further investigation of conditions where observations were made, such as for the lower value of WRV at 140 °C- 60 min compared to raw biomass, could be relevant to corroborate these results as well as for potential mechanical water removal. In addition, further investigation of the use of biomass initially really moist and having high ash content would also be relevant; especially within a bioenergy or potentially for subsequent transformations and applications into other biobased products.

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