



## Research paper

## Impact of a pressurized hot water treatment on the quality of bio-oil produced from aspen

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## ABSTRACT

The bio-oil produced by fast pyrolysis is a genuine alternative to fossil resources. However an improvement of its quality is required in order to improve its application.

To upgrade the quality of bio-oil, Pressurized Hot Water Treatment (PHWT) has been applied on trembling aspen whole wood chips prior to fast pyrolysis process. The pyrolysis was then performed in an auger reactor at the temperature of 723 K. The effects of PHWT on yields, physicochemical properties, and composition of bio-oils were investigated.

Although PHWT does not seem to influence the bio-oil yield, which remains around 56% for both untreated and pre-treated wood, it does improve its quality. The main effect of pre-treatment is the lower water content of the oil obtained from pre-treated wood, which is thus meeting the requirements of ASTM D7544 Standard. Moreover, PHWT appeared to favor the levoglucosan production and to decrease the syringol derivatives content of the bio-oil. The elementary composition revealed an increase of the C/O ratio when the biomass was pre-treated. This is in agreement with the heating value of bio-oil from pre-treated biomass which was found to be higher than that of bio-oil from untreated biomass.

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## 1. Introduction

The search of an alternative to fossil resources as energy and chemicals sources has been a critical issue for many decades. The depletion of petroleum reserves, the growing demand of energy and the negative environmental impact of fossil fuels have urged industries and laboratories to prepare a way-out from the petroleum dependency.

The thermochemical conversion of biomass by fast pyrolysis is one of the most promising alternative technologies [1–5]. Biomass is a renewable, abundant and widespread resource. The most common types of biomass used in the valorization process are energy crops, agricultural, forestry and wood transformation residues. Fast pyrolysis can be defined as a thermal degradation occurring in the absence of oxygen. The products of biomass pyrolysis are a liquid phase called bio-oil, a solid char and non-condensable gases. Their yields depend on process conditions,

reactor design and feedstock types. Fast pyrolysis is operated in such a way to produce mainly the oil. Its main characteristics are [6]:

- moderate and controlled temperature between 673 and 873 K;
- high heating rate and heat transfer;
- short residence time of pyrolysis vapors;
- rapid quenching of gas and aerosols to condense the bio-oil.

Bio-oil is described as a complex mixture of thermally degraded products from holocellulose, lignins and extractives. An overall composition of bio-oil could be cited as consisting of 30% water, 30% phenolics, 20% phenolics and ketones, 10% alcohol and 10% of others compounds [7]. The highly oxygenated compounds are generated mainly by the thermal decomposition of holocellulose. These compounds are mainly responsible for the bio-oil acidity, its low heating value, chemical instability, and immiscibility with conventional fuels. These drawbacks impede their direct application as fuel.

Upgrading the bio-oil quality can be achieved in several ways. Catalytic deoxygenation can be performed *in situ* on pyrolysis

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vapors with an integrated catalytic system or directly on bio-oils by hydrotreating [8]. This would reduce oxygen content and hence increase the heating value [9]. Unfortunately, these techniques require further development and have to fix the coking problem.

Biomass pre-treatment is essential for improving lignocellulosic biomass conversion into fuel. Since the 2000s, the interest in applying these techniques to pyrolysis has grown [10]. Hemicelluloses and ash constituents are known to be partially responsible for the acidity of bio-oil, its low calorific value and instability [8,11–13]. A pre-treatment which selectively removes these constituents from the biomass could therefore improve the bio-oil properties. Indeed, pre-treatments with water, dilute acids or dilute alkalis were applied on wood [14]. Indeed, the chemical composition of the bio-oil is affected by the removal of alkali metals. A study reports that pyrolysis oil produced from biomass washed with deionised water and dilute nitric acid [15] had better physicochemical properties than oil from untreated biomass, especially the viscosity. Moreover, bio-oil from acid pre-treated biomass had high content of anhydrosugars, particularly of levoglucosan, but had also a higher content of water, and a lower pH [12,14]. However, another study affirms that biomass pre-treated with dilute acid, 1% H<sub>2</sub>SO<sub>4</sub>, gave pyrolysis oil with higher yield and better physicochemical properties including a lesser water content and a higher pH than bio-oil from untreated biomass [16].

Hydrothermal pre-treatment, such as Pressurized Hot Water Treatment (PHWT), removes selectively hemicelluloses and ash from wood biomass. The reaction mechanism is similar to a dilute acid hydrolysis [17]. The cleavage of glycosidic bonds causes the depolymerization of hemicelluloses. A partial solubilisation of lignins has also been reported. No cross-linking reactions were reported to occur during the pre-treatment and thus there should not be a thermal resistance issue for the pre-treated biomass [11]. The effect of hydrothermal pre-treatment on bio-oil yield and its chemical and physical properties was studied using eucalyptus and beech wood [11,18]. The pre-treated biomass gave higher bio-oil yields than untreated biomass. Moreover, bio-oil from pre-treated wood had higher heating value and lower water content. As for the dilute acid pre-treatment, the production of anhydrosugars was also significantly enhanced.

The aim of this project was to evaluate the influence of PHWT on the bio-oil quality produced from forest biomass. The pyrolysis system used for this study was an auger reactor. The optimal fast pyrolysis temperature, 723 K, was determined in our previous study by Py-GC/MS analysis [19]. The other operational parameters, such as auger speed, and particle size were fixed according to preliminary experiments. The pre-treatment applied to aspen biomass was PHWT at mild severity in order to maximize the removal of alkali and hemicelluloses while minimizing lignins solubilization [20]. To our best knowledge, it was the first time that these two promising technologies, PHWT and fast pyrolysis in auger reactor, were combined. Yield and quality of bio-oil produced from untreated and pre-treated aspen, and production of anhydrosugars were studied.

## 2. Experimental

### 2.1. PHWT biomass production

Trembling aspen (*Populus tremuloides*) chips with bark used for this study were produced from Copeaux de la Vallée sawmill located in Amqui, Quebec, Canada. The trees were harvested at the forest management unit UAF 111-61 (48°22'N 66°41'W) located in the Quebec provincial administrative region of Gaspésie—Îles-de-la-Madeleine (Canada) and stored outdoor before chipping. The age of the trees ranged from 25 to 30 years. The wood chips were

received oven dried to 8% moisture content and subjected to a PHWT in a 2 dm<sup>3</sup> stirred pressure reactor (Parr 4522) at 468 K. Each PHWT was conducted on 150 g (dry basis). To produce enough feedstock for the subsequent fast pyrolysis, eleven batches were treated under these conditions. The solid residues were collected, washed with hot water (1 dm<sup>3</sup>), and air dried. A severity factor, *S*, was calculated taking into account the time and the pre-treatment temperature. Thus, for PHWT performed at 468 K, the severity factor, *S*, was 4.6, as determined in our previous study [20]. A chemical analysis and a proximate analysis were performed on both untreated and pre-treated aspen.

### 2.2. Grinding of untreated and pre-treated biomass

Tests were performed on 100 g of untreated and pre-treated wood chips. Biomass samples were dried overnight prior to experiments. The average moisture content measured by IR method was 2% for both pre-treated and untreated aspen. The grinding was carried out in two steps by a cutting mill (SM 300, Retsch). First, a pre grinding step was performed using a bottom sieve with an aperture size of 4 mm. Then the biomass was ground with a 2 mm grid. The cutting bars were attached to a rotor the speed of which had been set at 25 Hz.

The particle size distribution of untreated and pre-treated wood was determined by a sieve shaker. The sieve sizes were:

- 3.35 mm, 2 mm, 1 mm, 0.85 mm, 0.425 mm, 0.25 mm for the pre grinding step
- 1 mm, 0.85 mm, 0.425 mm, 0.25 mm, 0.125 mm, 0.075 mm for the grinding step

The measures of particle size distribution were duplicated for untreated and pre-treated aspen.

### 2.3. Pyrolysis system

Prior to fast pyrolysis process, pre-treated and untreated aspen chips were oven dried overnight. Thereafter, they were ground in a mill (SM 300, Retsch) following the procedure of section 2.2 in order to obtain a particle size less than 2 mm. The moisture content of the milled aspen was measured by IR determination (MB 23, Ohaus).

The fast pyrolysis was performed on pre-treated and untreated aspen with a lab-scale auger reactor, as shown in Fig. 1. Heat was supplied by two external electric furnaces which cover the reactor. Once the pyrolysis temperature was reached, the auger system was purged by nitrogen, with flow of 0.1 dm<sup>3</sup> s<sup>-1</sup>, during 1200 s. After that, about 700 g of milled whole wood aspen were fed into the reactor by a screw feeder. The auger speed was fixed at rpm 0.3 Hz according to preliminary experiments on driving the biomass through the reactor. The bio-char was collected by gravity in a specific vessel placed at the end of the reactor. The solid residence time was 210 s. A nitrogen flow of 0.06 dm<sup>3</sup> s<sup>-1</sup> was kept to maintain an inert atmosphere inside the reactor and to expel the pyrolysis vapors in 6 s. The vapors were cooled by a two condenser system. The first one was cooled with tap water while the second condenser was cooled at 263 K. The bio-oil was mainly recovered in the first condenser. The temperatures were measured along the pyrolysis system by six thermocouples. The pyrolysis temperature of 723 K was determined by the combination of the data from thermogravimetric [20] and from the Py-GC/MS [19] performed on pre-treated aspen in our previous studies. The fast pyrolysis of untreated aspen was duplicated.

For the mass balance calculations, the bio-oil and bio-char collected were weighted and the non-condensable gases (NC gas)

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