



Research article

Accelerating wet torrefaction rate and ash removal by carbon dioxide addition

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ABSTRACT

In this study, CO₂ was employed as an environment friendly catalyst for wet torrefaction of fresh Norwegian forest residues. The effects of CO₂ addition on the yield and the properties of hydrochar were experimentally studied and compared with the base case, wet torrefaction in N₂. The results showed that wet torrefaction in CO₂ and N₂ has similar effects on the improvement in the fuel properties of biomass. However, CO₂ was found to improve the reactivity of forest residues during wet torrefaction, producing the same hydrochar yield at less severe conditions compared with N₂. The hydrochars produced in CO₂ had lower heating value and energy yield, but better grindability and hydrophobicity, compared with those in N₂. More importantly, CO₂-enriched wet torrefaction can remove up to 60–69% of the ash content in the forest residues, compared to only 14–26% for wet torrefaction in N₂.

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1. Introduction

Wet torrefaction (WT) involves hot compressed water for treatment of biomass in the temperature range of 180–260 °C [1–3] to produce hydrochar, a hydrophobic solid fuel with enhanced fuel properties and better grindability compared with the raw biomass [1–4]. Although the terminologies are different, the concept of WT can be found in the literature under other terms such as hydrothermal carbonization, hydrothermal conversion or hydrothermal treatment. Because of employing water as reaction medium, WT is therefore very much suitable for wet feedstocks, which include forest residues, wet agricultural wastes, sewage sludge, and aquatic energy crops. In addition to the main solid product (hydrochar), WT also produces approximately 10 wt.% (on dry feedstock basis) of liquid by-products including acetic acid, formic acid, lactic acid, glycolic acid, levulinic acid, phenol, furfural, hydroxymethylfurfural (HMF), and sugars, which can be further treated for the production of biogas, liquid fuels and/or valuable chemicals [3,5]. Recently, the number of WT studies has been increasing. Apart from the improvements in the heating value, hydrophobicity and grindability of the hydrochars, these studies highlighted the capacity of washing out ash elements from biomass fuels via WT to produce “clean” solid biomass fuels with regard to inorganic elements. In addition, the

compressibility and durability of hydrochar are better than those for the raw material [6–8].

A key point in WT as well as other hydrothermal conversion technologies is that the water should be maintained in the liquid phase in order to avoid energy loss in form of latent vaporization heat. In other words, the operating pressure must be higher than the saturated pressure of water at the reaction temperature. However, to withstand such high pressures, the reactor wall must be thick enough, which obviously increase the investment cost and operational risks of the technology at industrial scale. Therefore, enhancing the hydrothermal reaction rate by catalysts is becoming an essential option for commercial processes because it can help lower the reaction temperature of the processes, which can reduce both the heating energy consumption and the operating pressure. Recently, it can be found in the literature a few hydrothermal treatments supported by some types of catalyst including acids [9–12] (citric, acrylic, sulfuric, and acetic) and salts [13] (chloride, propionate, acetate, lactate, and formate). These studies reported a significant reaction rate improvement when catalyst is added. For example, the addition of acetic acid or lithium chloride to hydrothermal treatment of loblolly pine decreases the solid yield but increases its heating value [9]. Moreover, calcium chloride and lactate were found to help in decreasing the pretreatment pressure [13].

Although the catalysts can accelerate the reaction rate even at a low temperature, the handling of these catalysts can be a challenge as they are corrosive chemicals, especially at elevated temperatures. Moreover, the use of catalysts is associated with some environmental and post-processing issues such as waste water treatment and catalyst recycling,

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which potentially increase the operating cost. An alternative technique that has been explored by Hunter and Savage [14,15] is the addition of carbon dioxide (CO₂) to hydrothermal processes. In an aqueous medium, CO₂ reacts with water to form carbonic acid, which promotes an acid catalyzed hydrothermal reaction. Moreover, due to the volatile nature of CO₂, the catalyst recovery is almost complete at no cost. Similar to other catalysts, the addition of CO₂ was found to enhance the hydrothermal reaction [15–17]. However, most studies on CO₂-enriched hydrothermal treatment focused on a hydrolysis process which aims at maximization of the water-soluble products (e.g., sugars) yield for chemicals production. So far, there is no work dealing with WT in CO₂ for solid fuel production that can be found in the literature. Therefore, the effects of CO₂ addition on the WT process as well as the fuel and physical properties of the hydrochar have not been fully understood yet, which motivates the need of this study.

The present study aims at employing CO₂ as an environment friendly catalyst for WT of fresh Norwegian forest residues. The effects of CO₂ addition on the yield and the properties of hydrochar were experimentally studied and compared with WT in N₂, which serves as the base case. For this purpose, WT was carried out at different temperatures (175, 200, 225 °C) and in two gas media, N₂ and CO₂. Direct comparisons of the solid and energy yield, fuel and physical properties as well as the morphology of the hydrochars produced in the two different gas atmospheres are presented.

2. Materials and methods

2.1. Feedstock collection and preparation

The feedstock used for this study is Norway spruce and birch branches of 2–2.5 cm in diameter. The fresh branches were collected from a local forest in Trondheim, Norway. The bark was completely removed from the collected branches to avoid possible interferences caused by impurities/contaminants and composition differences between the core and the bark. The branches were then cut into slices (3–4 mm thick), washed with water and stored in a climate cabinet (series VC³ 0100 of Vötsch Industrietechnik) to maintain the moisture content. Typical fuel properties of the feedstock including moisture content, proximate and ultimate analyses as well as higher heating value are presented in Table 1. The proximate analysis was performed according to ASTM standards: E871, E872 and D1102 for moisture content, volatile matter and ash content, respectively. The ultimate analysis was determined by an “EA 1108 CHNS-O” elemental analyzer (Carlo Erba Instruments). The higher heating value (HHV) was calculated according to a correlation proposed by Channiwala and Parikh [18].

2.2. Wet torrefaction procedure

The WT procedure were carried out as reported in our previous study [1], brief information is extracted here. The system employed for WT experiments consists of a 250 ml Parr reactor model 4651, a ceramic furnace (Parr 4923EE) connected to a temperature controller (Parr 4838EE). Distilled water was used as reaction media. The ratio of

dry feedstock over water was 1:5 (wt:wt). Before every torrefaction run, the furnace without the reactor was heated for 30 min to a preset temperature. At the same time, the reactor was loaded with fresh branches, closed, sealed, and purged with compressed gases (N₂ or CO₂) for 10 min. The reactor was pressurized by amounts of N₂ or CO₂ until it reached a pressure of 70 bar at WT temperatures for all experiments. It was then placed in the preheated furnace operating at the maximum power, giving a heating rate of approximately 12 °C per min. The holding time was 30 min for all experiments, counted from the time at which the reactor temperature reached the WT temperature to the end point when the reactor was replaced from the furnace and submerged in an ice bath for cooling. When the reactor cooled to room temperature, the pressure was gradually released and the reactor was opened for collection of the products, which were in the solid and liquid phases. The solid products (hydrochars) were separated from the mixture by filtration using a filter paper. After separation, the collected hydrochars were dried at 103 ± 2 °C for 24 h and the dried hydrochars were then stored in a desiccator filled with silica gel for further analyses.

2.3. Assessment methods

2.3.1. Assessment of grindability via specific grinding energy

Specific grinding energy (SGE) of the forest residues was determined using an IKA MF 10 cutting mill (from IKA®-Werke GmbH & Co. KG) equipped with a 1 mm bottom sieve. The grinder was connected to a current input module NI 9203 (from National Instruments Corporation) to record the electrical current during grinding. A LabView program was used for data acquisition. The energy consumption was calculated and logged to a file every 2 s. The grinding energy was determined by integrating the power curve during the grinding period. The power of the mill under no-load condition was measured and subtracted from the power of grinding the samples. Finally, the data was normalized to the initial sample weight to obtain the SGE.

2.3.2. Assessment of hydrophobicity via equilibrium moisture content

Powder collected after grinding was sieved by a Fritsch Analysette 3 Pro vibrator to select the particles smaller than 250 µm for the moisture up-take test. The selected powder was dried further at 103 ± 2 °C for 24 h to completely remove any moisture up-taken during the grinding period. Approximately 2 g of tested sample was spread on a glass Petri dish. Then, the dish with sample was placed in a climate cabinet (series VC³ 0100 of Vötsch Industrietechnik) operated under controlled conditions of 20 °C and 90% relative humidity. The mass changes of the tested samples due to the moisture up-take were recorded for the total test period of one week. The up-taken moisture was calculated as shown below:

$$MC_i(\%) = \frac{m_i - m_0}{m_0} \times 100\%, \quad i = 1, 2, \dots, 7 \quad (1)$$

where MC_i is the moisture content of the tested sample on the i th day; m_0 and m_i are the mass of the initial dry sample and mass recorded on the i th day, respectively.

Table 1
Feedstock characteristics.

Type of feedstock	Moisture content ^a	Proximate analysis ^b			Ultimate analysis ^c					HHV ^d
		Ash	VM	FC	C	H	O	N	S	
Spruce	49.96 ± 2.34	0.68 ± 0.05	85.18 ± 0.26	14.14 ± 0.15	51.31 ± 0.14	6.22 ± 0.09	42.35 ± 0.06	0.122 ± 0.01	<0.02	20.84 ± 0.07
Birch	56.31 ± 1.93	0.64 ± 0.04	89.74 ± 0.32	9.63 ± 0.09	48.55 ± 0.17	6.19 ± 0.06	45.09 ± 0.14	0.165 ± 0.03	<0.02	19.63 ± 0.06

VM: volatile matter; FC: fixed carbon; HHV: higher heating value.

^a wt.%, wet basis.

^b wt.%, dry basis.

^c wt.%, dry and ash free basis.

^d MJ/kg, dry and ash free basis.

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