



Using liquid waste streams as the moisture source during the hydrothermal carbonization of municipal solid wastes



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ABSTRACT

Hydrothermal carbonization (HTC) is a thermal conversion process that can be an environmentally beneficial approach for the conversion of municipal solid wastes to value-added products. The influence of using activated sludge and landfill leachate as initial moisture sources during the carbonization of paper, food waste and yard waste over time at 250 °C was evaluated. Results from batch experiments indicate that the use of activated sludge and landfill leachate are acceptable alternative supplemental liquid sources, ultimately imparting minimal impact on carbonization product characteristics and yields. Regression results indicate that the initial carbon content of the feedstock is more influential than any of the characteristics of the initial liquid source and is statistically significant when describing the relationship associated with all evaluated carbonization products. Initial liquid-phase characteristics are only statistically significant when describing the solids energy content and the mass of carbon in the gas-phase. The use of these alternative liquid sources has the potential to greatly increase the sustainability of the carbonization process. A life cycle assessment is required to quantify the benefits associated with using these alternative liquid sources.

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1. Introduction

Hydrothermal carbonization (HTC) is a relatively low temperature thermal conversion process that has been shown to be an environmentally beneficial approach for the transformation of biomass, carbohydrates, and waste streams to value-added products (e.g., Berge et al., 2011; Hwang et al., 2012; Li et al., 2013; Libra et al., 2011; Román et al., 2013; Titirici et al., 2012; Sevilla and Fuertes, 2009). Results from recent research indicate that conversion of municipal solid wastes (MSW) via HTC, particularly wet waste components, is energetically advantageous. As a result of HTC, a solid carbon-rich, energy-dense material referred to as hydrochar is generated. Berge et al. (2011), Lu et al. (2012), Hwang et al. (2012) and Li et al. (2013) report that when carbonizing different MSW components, the majority of initially present carbon remains integrated within the hydrochar and that the hydrochar has an energy content and structure resembling a low-grade coal material. Other advantages associated with using HTC as a waste conversion technique include the formation of a liquid stream that contains appreciable concentrations of valuable compounds (e.g., organic acids, hydroxymethylfurfural (HMF)) and

nutrients (e.g., Berge et al., 2011; Li et al., 2013) and that carbonization has the potential to convert compounds of concern found in waste streams (i.e., pesticides, pharmaceuticals) to less harmful products (Weiner et al., 2013).

An important and required component of HTC is the presence of adequate moisture/liquid. At typical HTC system temperatures, the properties of liquid differ significantly from that at room temperature, ultimately mimicking that of an organic solvent (Akiya and Savage, 2002; Siskin and Katritzky, 2001; Wantanabe et al., 2004). The liquid properties/behavior during carbonization play a key role in the carbonization process, leading to increased saturation concentrations of dissolved organic and inorganic compounds and promotion of ionic reactions. Funke and Ziegler (2010) report that for hydrothermal carbonization to proceed the feedstocks need to be completely submerged. Often, to achieve feedstock submersion, the moisture requirement for HTC is greater than that naturally present in the feedstock, particularly for dry components of MSW, such as paper. It should be noted that carbonization reactions will proceed under limited moisture contents, but the carbon content of the solid materials is reduced and solids yields are increased (Funke et al., 2013).

To date, water (often deionized) is the liquid used as the moisture source in the majority of reported hydrothermal carbonization studies. As concerns associated with water scarcity increase, the

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need for identifying carbonization liquid sources increases. Using liquid waste streams as moisture sources during carbonization would be advantageous. Liquid waste streams, such as municipal wastewater and landfill leachates, represent wastes streams that are plentiful and require extensive treatment prior to their discharge to the environment. In addition, use of these liquid waste streams during HTC may be beneficial to carbonization, potentially increasing carbonization kinetics and enhancing the properties of the generated solids. Stemann et al. (2013) conducted experiments in which carbonization process water was recirculated and report that changes in initial process water quality catalyze dehydration reactions and that organics in the liquid stream are further polymerized, increasing the carbon and energy content of the recovered solids. Catalyst addition to the initial process water has also been shown to positively influence carbonization. Lynam et al. (2012) found that when adding salt to carbonization systems, the energy value of the solids increases. In another study, Lynam et al. (2011) report that the addition of acetic acid and lithium chloride also increase the energy value of the recovered solids.

Using municipal waste streams as the initial moisture source for HTC has not been previously investigated. The purpose of this work is to evaluate the influence of substituting activated sludge and landfill leachate as the moisture source for HTC and to determine their influence on carbonization product characteristics. Laboratory-scale experiments evaluating the influence of using landfill leachate and activated sludge as initial moisture sources during the carbonization of yard waste, paper, and food waste were conducted. Characteristics of the solid, liquid, and gas-phases were measured and used to determine if these alternative moisture sources can be used to increase the sustainability of the HTC process. Statistical analyses were conducted to evaluate whether changes in initial liquid properties results in a statistically significant change in carbonization product characteristics and to assess the relationship between carbonization products, initial liquid source characteristics, feedstock type, and process conditions.

2. Materials and methods

2.1. Liquid source and solid waste material characteristics

Activated sludge and landfill leachate are the liquid waste streams evaluated in this study. Each waste stream was obtained from either a local wastewater treatment plant or a municipal solid waste landfill. The BOD/COD and COD/TOC ratios of the landfill leachate are low, indicating its biodegradability is low. Typical properties of each liquid stream were measured and are reported in Table 1 using the analytical methods described in subsequent sections.

The solid waste materials used in this work include paper, food waste, and yard waste. Before use, the office paper was shredded into 25 by 4 mm strips using a titanium paper shredder (micro-cut shredder, Staples, Inc.). Food wastes were collected from local restaurants. The collected waste was sorted, as reported by Li et al. (2013), and the food component subsequently homogenized with a food-grade blender (Ninja Master Prep Model #: QB900, Euro-Pro Operating LLC). Yard waste is comprised of a mixture of

50% (dry wt.) of grass clippings and 50% (dry wt.) shredded shrubs. The shrubs were chipped using an electric shredder (Chicago Electric Power Tools, Inc., 1.5 in., 14 Amp Shredder). The properties of each of these waste materials are presented in Table 2.

2.2. Batch HTC experiments

All batch carbonization experiments were conducted following procedures previously described (Berge et al., 2011; Li et al., 2013; Lu et al., 2012, 2013; Flora et al., 2013). Briefly, the feedstocks (e.g., paper, food, and yard wastes) were placed in 160-mL stainless steel tubular reactors (2.54 cm i.d., 25.4 cm long, MSC, Inc.) fitted with gas-sampling valves (Swagelok, Inc.). A mass equivalent to 8 g of dry solids was added to all reactors. Moisture was subsequently added to achieve the desired solid material concentration of 20% (dry wt.). The moisture sources evaluated include: (1) deionized (DI) water, (2) landfill leachate, and (3) activated sludge. In addition, control experiments containing only activated sludge and landfill leachate were conducted. All reactors were sealed and heated in a laboratory oven (Heratherm model, Fisher Scientific, Inc.) at 250 °C. The desired in-situ temperature of the reactors was achieved after 90 min. Experiments for each feedstock and moisture source were conducted over three carbonization times (2, 8, and 24 h) to evaluate how reaction time influences carbonization. These sampling times include the period of reactor heating. All experiments were conducted in triplicate.

2.3. Analytical techniques

At each sampling time, reactors were removed from the oven and placed in a cold water bath. Following cooling, the produced gas was collected in either a 1 or 3-L foil gas sampling bag (SKC, Inc.). Gas composition of these samples was analyzed using GC-MS (Agilent 7890). Gas samples were routed through a GS-Carbon-Plot column (30 m long and 0.53 mm id, J&W Scientific). Initial oven temperature was 35 °C. After 5-min, the temperature was increased at a rate of 25 °C/min until a final temperature of 250 °C was achieved. Carbon dioxide standards (Matheson Trigas) were used to determine concentrations in the gas. Results from this analysis were also used to provide qualitative data associated with the composition of the gas stream. Gas volumes were measured with a large volume syringe (S-1000, Hamilton Co.).

The process liquid and solid were separated via vacuum filtration through a 0.45 µm cellulose nitrate membrane filter (Whatman International Ltd.). Liquid conductivity and pH were measured using electrodes (Thermo Scientific Orion). Liquid chemical oxygen demand (COD) was measured using HACH reagents (HR + test kit, HACH Co.). Liquid total organic carbon (TOC) was measured using a TOC analyzer (TOC-Vcsn, Shimadzu). The 5-day biological oxygen demand (BOD) of the liquids collected after the 24-h reaction time was measured using the HACH BODTrak technique (BODTrack II, HACH Co.).

All collected solids were dried at 80 °C. Solid carbon, hydrogen, and nitrogen content (Perkin Elmer 2400 Elemental Analyzer) and energy content (C-200 bomb calorimeter, IKA, Inc.) were measured.

Table 1
Characteristics of liquid sources used this study.

Process water	Total suspended solids (TSS, mg/L)	pH	Conductivity (mS/cm)	COD _{Total} (mg/L)	TOC (mg/L)	BOD (mg/L)
DI water	0	6.85 (0.05)	0.002 (0.0009)	0	0	0
Landfill leachate	88 (6.1)	8.24 (0.09)	12.74 (1.14)	3220 (325)	2169 (125.3)	101 (20)
Activated sludge	1746 (202)	7.02 (0.25)	0.61 (0.12)	1830 (173)	181 (15.1)	404 (83)

Values in parentheses represent standard deviations.

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