



# Phases' characteristics of poultry litter hydrothermal carbonization under a range of process parameters



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## HIGHLIGHTS

- Hydrochar produced at 250 °C resembles sub-bituminous coal.
- Liquid-phase's high nutrient level indicates possible use as fertilizer.
- CO<sub>2</sub> is the main gaseous component, with presence of CH<sub>4</sub> and H<sub>2</sub>S.
- HTC was not affected by high solid-to-water ratio of up to 1:3.
- Energy balance favors HTC of sludge with high solids concentration.

## ARTICLE INFO

### Article history:

Received 28 June 2016

Received in revised form 8 August 2016

Accepted 9 August 2016

Available online 10 August 2016

### Keywords:

Hydrothermal carbonization

Solid fuel

Liquid fertilizer

Energy efficiency

Gas emission

## ABSTRACT

The aim of this work was to study the hydrothermal carbonization of poultry litter under a range of process parameters. Experiments were conducted to investigate the effect of HTC of poultry litter under a range of operational parameters (temperature, reaction time, and solids concentration) on the formation and characteristics of its phases. Results showed production of a hydrochar with calorific value of 24.4 MJ/kg, similar to sub-bituminous coal. The gaseous phase consisted mainly of CO<sub>2</sub>. However, significant amounts of H<sub>2</sub>S dictate the need for (further) treatment. The process also produced an aqueous phase with chemical characteristics suggesting its possible use as a liquid fertilizer. Temperature had the most significant effect on processes and product formation. Solids concentration was not a significant factor once dilution effects were considered.

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## 1. Introduction

Worldwide production of poultry meat surpasses that of beef and veal and is growing at a faster rate than pork meat (United States Department of Agriculture Foreign Agricultural Service, 2015). Most of the produced manure and litter are applied to agricultural lands prior to or after treatment, and can lead to water, soil and air pollution (Whitely et al., 2006). Common poultry-litter treatments include composting, anaerobic digestion and direct combustion, but these are not without problems (Kelleher et al., 2002; Edwards and Daniel, 1992), and alternatives are needed.

Hydrothermal carbonization (HTC) is a fairly new technology that can potentially treat wet organic matter such as animal manure and litter efficiently with minimal environmental pollution. HTC is a process that converts wet organic matter into a C-rich solid product referred to as hydrochar, as well as aqueous and gaseous products (Libra et al., 2011). HTC takes place under a typical temperature range of 180–250 °C, autogenous pressure, reaction times varying from minutes to several hours (Funke and Ziegler, 2010), in a low oxygen media. Under these conditions, water stays in a liquid state, and there are various changes in its properties, such as decreased density and dielectric constant, and increased ion dissociation constant (Peterson et al., 2008). These changes can result in lowering of the feedstock's O and H content through dehydration and decarboxylation reactions (Funke and Ziegler, 2010). HTC has been considered for various purposes, such as production of nanostructured materials, C sequestration, production of adsorbent materials, soil amendment, energy production,

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nutrient recycling, and waste treatment (Libra et al., 2011). To date, however, only a relatively small number of feedstock types have been investigated in recent HTC studies, such as agricultural wastes (Hoekman et al., 2011; Reza et al., 2013), sewage sludge (Danso-Boateng et al., 2015; He et al., 2013; Zhao et al., 2014), municipal solid waste (Berge et al., 2011), algal residues (Levine et al., 2013), and wet animal manure (Heilmann et al., 2014; Oliveira et al., 2013). Moreover, current information is still in its infancy, with many research and methodological gaps.

The aqueous phase has been characterized focusing on the presence of organic compounds such as 5-hydroxymethylfurfural (5-HMF), furfural, propionic acid, acetic acid, and guaiacol (Becker et al., 2014; Danso-Boateng et al., 2015) which can be used as precursors to the production of bio-fuels and bio-based chemicals. Only a few studies have investigated its direct use, such as for bio-gas production (Danso-Boateng et al., 2015; Mumme et al., 2011; Oliveira et al., 2013) and as a nutrient source for algal biomass production (Levine et al., 2013).

The gas formed during HTC consists mainly of CO<sub>2</sub> with minor fractions of CO, CH<sub>4</sub> and H<sub>2</sub>, and traces of CmHn (Funke and Ziegler, 2010). The gas composition does not vary significantly with the carbonization of different types of feedstock (Berge et al., 2011). The amount of gaseous product has been found to increase with rising temperature. Simultaneously, the CO fraction decreases while those of CH<sub>4</sub> and H<sub>2</sub> increase (Funke and Ziegler, 2010). Identified gases of environmental concern are furan, H<sub>2</sub>S, NO<sub>2</sub>, NO, and NH<sub>3</sub> (Danso-Boateng et al., 2015).

Only a few studies that have investigated HTC of poultry manure were identified, and these were limited in scope with respect to the influences of temperature, reaction time, and solids concentration on product formation and characteristics. Heilmann et al. (2014) investigated HTC of poultry manure at 200 °C, 225 °C, and 250 °C for 1, 2, and 3 h, with solids concentrations of 5, 10, and 15%, focusing on the elemental composition and metal content of the hydrochar and aqueous phase. Oliveira et al. (2013) investigated HTC of poultry manure at 180 °C for 4 h with 15% solids, focusing on the composition of the hydrochar, aqueous and gaseous phases, as well as on the calorific value of the hydrochar. Ekpo et al. (2016) performed HTC of poultry manure at 250 °C for 1 h with a solid-to-water ratio of 1:9, and focused on the composition of the hydrochar and aqueous phase. Ghanim et al. (2016) analyzed HTC of poultry litter at temperatures ranging from 150 to 300 °C and reaction times of 30, 120 and 480 min at a solid-to-water ratio of approximately 1:8.5, focusing on physicochemical hydrochar characteristics. The existing fragmented information prevents a comprehensive understanding of the factors influencing the HTC process and the three phases' characteristics.

The present study was conducted on a higher solid-to-water ratio than previously investigated, due to poultry litter's natural low moisture content and energy-efficiency considerations. The principal objectives of this research were to investigate the effect of HTC of poultry litter under a range of operational parameters (temperature, reaction time, and solids concentration) on the phases' formation and characteristics. Specifically: (i) a mass balance was conducted for C and N, (ii) potential use of the aqueous phase as fertilizer was evaluated, and (iii) an energy balance was calculated to explore the relevance of this practice as a sustainable environmental solution.

## 2. Materials and methods

### 2.1. Feedstock

Poultry litter from a broiler farm in the Negev region of Israel was collected. The litter contained feces, urine, bedding material,

and feathers. The feedstock was dried at 105 °C for 24 h, and aggregates were crushed using a mortar and pestle and then sieved through a No. 8 mesh. The dried and homogenized feedstock was stored in a desiccator prior to HTC experiments.

### 2.2. Experimental design

Although there are no standards for experimental HTC setups, most HTC studies have been performed with a solid-to-water ratio of around 1:5 or higher (up to 1:20), and only a few have used values closer to 1:3 (Li et al., 2013). Dried feedstock was mixed with double-distilled water to produce sludge with solid-to-water ratios of 1:3 and 1:5. The experiments were conducted in two types of reactor, one for characterization of the hydrochar and aqueous phase, and the second for characterization of the gaseous phase. The first set of reactors consisted of 50-mL stainless-steel tubular cylinders rated to withstand anticipated temperatures and pressures. Each reactor consisted of a 27-mm diameter stainless-steel pipe, nipple and end caps. One reactor was equipped with a temperature probe to provide a representative measurement of the temperature inside all of the reactors. The reactors were heated by immersion in preheated Paratherm (Conshohocken, PA) HR heat-transfer fluid. The second type was a 400-mL stainless-steel Parr (Moline, IL) reactor equipped with gas-sampling valves and a temperature probe, heated by a heating mantle. The experiment was conducted at temperatures of 250 °C, 220 °C, 200 °C and 180 °C, and reaction times of 5, 30 and 60 min. The reaction times did not include the period required for the reactors to reach the desired temperature, which ranged from 12 to 20 min for the 50-mL reactors, and from 30 to 45 min for the 400-mL reactor. After the required time, the 50-mL reactors were placed in an ice bath to quench the reaction. All combinations of temperature, reaction time and solid-to-water ratio were conducted in triplicate. Solid and aqueous phases from the 50-mL reactors were separated by vacuum filtration using a 0.70- $\mu$ m glass-fiber filter. Gas samples from the 400-mL reactor were collected into sampling bags with 0.5-L capacity (SKC Inc., Eighty Four, PA).

### 2.3. Phase characterization

#### 2.3.1. Hydrochar and litter

The wet hydrochar collected following filtration was weighed and oven-dried at 105 °C for 24 h, and then weighed again to determine the hydrochar yield (recovered mass of the initial dry poultry litter mass). Poultry litter and hydrochar organic matter were determined by combustion in a muffle furnace at 450 °C for 6 h (Hue and Evens, 1986). Elemental composition of C, H, N and S was determined with a FlashEA<sup>TM</sup>1112 CHNS-O Analyser (Thermo Fisher Scientific Inc., UK). The O content was calculated as the remaining component after subtraction of C, H, N, S and ash (ASTM-D3176, 2015). Elemental C was confirmed using the Walkley–Black method (Soil and Plant Analysis Council Inc., 1999). Higher heating values (HHV) for the untreated biomass and the hydrochar products were calculated based on the unified HHV correlation for fuels which takes into account C, H, N, S, O and ash % concentration by mass on a dry basis (Channiwala and Parikh, 2002). The data were used to calculate energy yield and energy densification. The untreated biomass and hydrochar were also analyzed by Fourier transform infrared (FTIR) spectroscopy with a Nicolet 6700 Thermo equipped with a diamond smart ATR holder (Thermo Fisher Scientific Inc., UK) in the range of 4000–650 cm<sup>-1</sup> through 36 scans. Spectra were corrected for background transmittance by subtracting the spectrum obtained with an empty holder.

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