



Influence of temperature on nitrogen fate during hydrothermal carbonization of food waste



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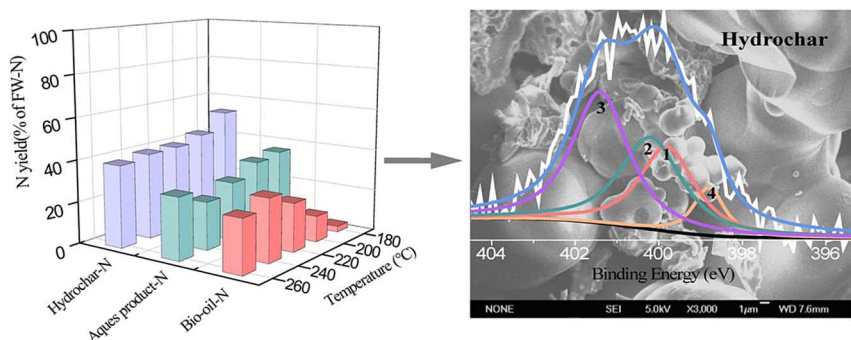
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GRAPHICAL ABSTRACT



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ABSTRACT

The influence of temperature (180–260 °C) on the fate of nitrogen during hydrothermal carbonization (HTC) of food waste (FW) was assessed. The distribution and evolution of nitrogen in aqueous products and bio-oil, as well as hydrochar, were conducted. Results suggested that elevated temperature enhanced the deamination and the highest ammonium concentration (929.75 mg/L) was acquired at 260 °C. At temperatures above 220 °C, the total N in the hydrochar became stable, whereas the mass percentage of N increased. Amines and heterocyclic-N compounds from protein cracking and Maillard reactions were identified as the main nitrogen-containing compounds in the bio-oil. As to the hydrochar, increasing temperature resulted in condensed nitrogen-containing aromatic heterocycles (e.g. pyridine-N and quaternary-N). In particular, remarkable Maillard reactions at 180 °C and the highest temperature at 260 °C enhanced nitrogen incorporation (i.e. quaternary-N) into hydrochar.

1. Introduction

Over the past decades, the increased population of China and rapid development of urbanization has brought significant quantities of

municipal solid waste (MSW). This facilitates the use of appropriate new applications to reduce the related environmental burden, minimize risks to human health and delay energy scarcity (He et al., 2014). The organic fraction of the MSW such as food waste represents an untapped

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resource having great potential for chemical and energy recovery; however, the disadvantages of huge volumes, high moisture and inherently variable composition restrict the direct utilization of MSW. The traditional options for MSW disposal including landfilling, composting and incineration; may lead to severe secondary-pollution such as landfill leachates and gases derived from the high moisture and organic fraction (He et al., 2014). Hydrothermal carbonization (HTC), a new thermochemical conversion technique, is highly attractive due to its superior ability to transform wet biomass into energy and carbonaceous materials (Funke and Ziegler, 2010; Sevilla and Fuertes, 2009a). It was firstly discovered in 1913 by Bergius, who aimed to mimic the natural coal formation process that converted cellulose into lignite-like materials, and was re-discovered more recently (Berge et al., 2011; Libra and Titirici, 2011). The water contained in the biomass or supplied is properly an excellent solvent and reaction medium during HTC, which is generally conducted at relatively low temperature (180–250 °C) under autogenous pressure that is maintained for a couple of hours (Funke and Ziegler, 2010; Libra and Titirici, 2011). However, the transformation and pathways of migration of elements such as N, S, P and heavy metals during HTC, are critical due to their direct relation to the application potential of the solid or liquid products (Heilmann et al., 2014; Xiao et al., 2015; Zhai et al., 2016; Zhang et al., 2017). Among these, the ways that nitrogen is transformed and evolved during the HTC process is of special interest, regarding the use of hydrochar as solid fuels or other materials, because of the close relationship of these processes to the consequent NO_x emissions or applications of N-doped materials (Zhang et al., 2012; Zhao et al., 2013).

Several researchers have investigated the distribution of nitrogen containing components during HTC of biomass waste such as microalgae or sludge, and reported the potential pathways of chemical evolution (Gai et al., 2015; He et al., 2015b). Hydrothermal deamination is regarded as the major way to release ammonia from biomass with high protein content. Yu et al. investigated the distribution of nitrogen in the products from hydrothermal processing of low-lipid microalgae. The results suggested 65%–70% of nitrogen in the feedstock could be transformed into water-soluble compounds once the temperature employed was higher than 220 °C and residence time was longer than 10 min (Yu et al., 2011). Ma et al. and Zhang et al. investigated the hydrothermal treatment of antibiotic mycelial dregs and antibiotic biomass residue. The results suggested that the nitrogenous content in the solid product could be lowered to below 5.6 wt% and 6.0 wt%, respectively, at 200 °C for 30 min, compared with feedstock for which the goal was largely the conversion of organic-N to ammonia (Ma et al., 2015; Zhang et al., 2014). Coupled with the decomposition of protein-N from biomass waste, the nitrogenous forms in hydrochar also went through various changes. He et al. investigated the N-evolution in sludge-derived hydrochar and reported that the inorganic N in the solid product could be reduced by dissolution. They also found that increase of the temperature led to enhanced deamination of the protein in the sludge, while higher pyridine-N content formed in hydrochar than in the raw feedstock (He et al., 2015b). Kruse et al. investigated the nitrogen conversion in carrot greens, the algae *Chlorella pyrenoidosa* and straw. The results showed that the chemical nature of nitrogen depended much on the biomass feedstock. The nitrogen was not completely removed from hydrochar when the feedstock included some beech wood impregnated with cysteine due to the Maillard reaction, by which nitrogen was incorporated into the hydrochar structure (Kruse et al., 2016). Falco et al. studied the addition of glucose to microalgae during HTC and found that both the addition of glucose and high temperature enhanced the conversion of pyrrole-N into more stable nitrogen groups, namely quaternary-N and pyridine-N, due to improved hydrothermal severity and reactions between protein and glucose (Falco et al., 2012). Thus, the N-forms in the HTC products and corresponding transformation behaviours rely much on the severity of the hydrothermal process and on the components of the feedstock. However, because the carbohydrate and protein makeup most of the FW, the

sufficient carbohydrate such as starch and glucose or the derivatives may have effect on the protein conversion during HTC. Hence, the N fate during the HTC process may be different from that of traditional biomass wastes such as sludge and microalgae, which have mainly protein as N source. Based on the aforementioned studies, knowledge regarding N conversion during HTC of FW remains inadequate. The relationship between distribution and the conversion of N-containing products during HTC has rarely been studied. Mechanistic insight into the HTC pathways of N-containing compounds is important in order to understand the evolution of N-forms for better utilization of food waste for fuel or functional materials.

In light of FW as a self-sufficient carbon and nitrogen source, the present study aimed to investigate the influence of temperature on the fate of N-forms during HTC of FW. The distribution of N-containing components in aqueous products, in bio-oil and in hydrochar was evaluated in relation to temperature. Nitrogen-containing compounds in the aqueous products and bio-oil were systematically analysed, while the nitrogen functionalities in the hydrochar were evaluated using FTIR, XPS and solid state ¹³C NMR to investigate the potential evolutionary pathways of elemental N. Thus, tracking the effect of temperature on N-evolution during HTC is critical for better control of the N species. It also provides alternative strategies for the application of food waste as a renewable source.

2. Materials and methods

2.1. Materials

The FW used in this study was obtained from the local transfer device of Hunan University in Changsha, China. Visual observation indicated that the raw feedstock consisted of mainly cooked food (e.g., meat, rice, seafood, vegetables, noodles and gravy), condiments (e.g. salad dressing, ketchup) and chopsticks. Due to processing limitations, the plastics and bones in the FW were separated out. The feedstock was thoroughly homogenized and then stored at 4 °C (no longer than 24 h) to avoid the microbial contamination before the HTC experiments. The chemical characteristics of the FW are given in Table 1.

2.2. HTC

The HTC was performed using a 500 mL 316 stainless steel reactor equipped with a PID controller and auto-stirrer. For each run, around 20% of FW (received solid, as prepared in Section 2.1) was filled in the reactor. The reactor was then heated using a 3.0 kW electric furnace at an approximately rate of 4 °C/min to reach a final temperature in the range 180–260 °C (in increments of 20 °C) and maintained for 60 min with stirring at 100 r/min. Upon cooling, the reaction mixture, which consisted of liquid product and solid residue, was completely removed from the reactor and separated employing a vacuum filtration apparatus. The solid residue was dried at 105 °C overnight until reaching constant weight. Subsequently, the solid samples were weighed and milled to produce particles less than 200 μm in size for further analysis. The liquid product was filtered through a 0.45 μm PTFE filter membrane to prepare the filtrate.

Table 1
Characteristics of the FW.

Proximate analysis (wt%, db ^a)			Ultimate analysis (wt%, daf ^b)					N/C atomic ratio (%)
VM	FC	Ash	C	H	O ^c	N	S	
72.6	15.9	11.5	41.96	8.01	35.04	3.45	0.07	0.07

^a db, dry basis.

^b daf, dry and ash-free basis.

^c Calculated by difference.

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