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Products evolution during hydrothermal conversion of dewatered sewage sludge in sub- and near-critical water: Effects of reaction conditions and calcium oxide additive

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ABSTRACT

In the present study, hydrothermal conversion (HTC) of dewatered sewage sludge (DSS) under sub- and near-critical water has been performed to investigate effects of reaction temperature and pressure, moisture content of DSS, and calcium oxide (CaO) additive on evolution profile and characteristics of gas, solid, and liquid products. Although energy recovery rate decreased with increasing temperature and pressure, significant decarboxylation and dehydration reactions led to hydrochars with best fuel quality at 320 °C. High moisture content favored decarboxylation reaction but reduced H₂ and CH₄ yields. Compared to that in the absence of additive, H₂ yield increased almost 6-fold at 380 °C and Ca/C molar ratio of 0.2, resulting in 58% H₂ and 26% CH₄ in final fuel gas. The results suggested that mineralization of heteroatomic compounds and dissolution of metals or mineral elements occurred during HTC. Under higher temperature and pressure, heavy metals or mineral elements were prone to be immobilized whereas dehalogenation became more distinct. CaO additive not only facilitated hydrolysis and deamination of organic compounds but also accelerated further fixation of inorganic elements and anions. Copyright © 2015, Hydrogen Energy Publications, LLC. Published by Elsevier Ltd. All rights reserved.

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Introduction

The management and disposal of sewage sludge (SS) generated from wastewater treatment plants is problematic due to high moisture content and pollutants (e.g. pathogens, heavy metals, and persistent organic pollutants) together with unpleasant smells therein [1]. Nevertheless, SS is rich in organic matter and nutrients, thus it is regarded as an alternative energy source. In order to efficiently harness energy from SS with great volume reduction, thermochemical conversions (e.g. gasification and pyrolysis) have been extensively investigated [2,3]. Among them, hydrothermal conversion (HTC) technology represents a more sustainable technique to convert wet SS to biofuels since prior thermal drying process is eliminated under high temperature (200–600 °C) and high pressure (5–40 MPa) conditions [4,5].

Currently, research is widely focused on supercritical water gasification (SCWG) of SS for H₂ rich gas production [6–8], hydrothermal liquefaction of SS to generate bio-oil [9–11], and hydrothermal carbonization for hydrochar solid fuels production [4]. Most of the research is application oriented and thus only certain aspects were considered. For instance, the effect of moisture content on direct SCWG of SS has been explored [8]. Fuel characteristics and combustion behavior of solid hydrochars, removal of N or S precursors, as well as NO emissions during combustion after hydrothermal carbonization were investigated towards cleaner solid fuel production [4,12]. In addition, transformation behaviors and environmental risk of heavy metals in solids have been extensively discussed for land application [10,11,13]. Moreover, simultaneous extraction, decomposition, and stabilization processes during HTC could result in mineralization and migration of inorganic elements in SS [14]. Although heavy metals were likely to be accumulated and redistributed in solid residues after sub- and supercritical water (SCW) treatment [10,13], the distribution of heavy metals in liquid phase is also of great importance for post-treatment and reclamation of the processing effluents.

Apart from heterocyclic N and S precursors, trace amounts of halogenated substances and/or perfluorinated compounds (PFCs) can be found in SS during wastewater treatment. It has been reported that a typical cyclic perfluoroalkyl surfactant was efficiently decomposed in sub- and SCW in the presence of iron catalyst [15]. Besides, desulfurization of organic sulfides was found in SCW via free-radical reactions [16]. Hwang et al. [17] indicated that chlorine in precursors could be converted into soluble chloride compound after subcritical water treatment. Yet little effort has been devoted to the destruction of the heteroatomic compounds during HTC of SS. Calcium oxide additive has been used to facilitate water–gas shift reaction (WGS) to increase hydrogen yield during SCWG of lignite [18] as well as catechol and starch [19], and catalytic aqueous phase reforming of glycerol [20]. Furthermore, Ca(OH)₂ was found to inhibit toxic pollutants formation during hydrothermal gasification of biomass waste [21]. These results indicated that Ca-based alkaline could be economical conditioner for effective HTC of SS.

More recently, subcritical water treatment of SS under lower temperature hydrothermal condition (200–380 °C,

2–22 MPa) has attracted great attention due to its lower energy consumption and safer operation. However, the gasification process (i.e. gas yield and H₂ purity) for SS under this range has not been well studied. Besides, characteristics of liquid products are unspecified, especially the behavior of heteroatomic compounds, heavy metals, mineral elements and their interactions. Moreover, little attention has been paid to the effects of Ca-based alkaline on products distribution and migration of heteroatoms (such as nitrogen, sulfur, chlorine, and fluorine) which are essential information to make this promising technology commercially viable.

In order to conceptualize the reaction chemistry in HTC of SS under low temperature and pressure, effects of operating conditions and moisture content on evolution characteristics of gas, solid, and liquid products were examined in this study. Furthermore, CaO additive was employed in near-critical water to act as CO₂ absorber via carbonation. The reaction chemistry was systematically interpreted in terms of products distribution and environmental pollutants migration.

Experimental

Materials

Dewatered anaerobically digested sewage sludge (DSS) with a moisture content of 82.5 wt.% was collected from Ulu Pandan Water Reclamation Plant in Singapore. Before being used as feedstocks in low temperature/pressure HTC process, DSS sample was stored at 4 °C. The main characteristics of DSS are shown in Table 1.

HTC procedures

HTC experiments were carried out in a 1 L stainless steel (SS 316L) fixed-head batch autoclave reactor (Parr Instrument Co., USA) with a maximum operating temperature and pressure of 500 °C and 35 MPa, respectively. Detailed configuration of the HTC system has been described elsewhere [22]. Three sets of experimental runs were conducted as shown in Table 2. For set 1 (Run 1–10), various operating temperatures and pressures were employed. Typically, 182 g of DSS containing approximately 150 g of water was loaded into the reactor vessel and sealed. Under stirring at 500 rpm, the vessel was heated up to the temperature and pressure in Table 2. After 20 min reaction time, the heating mantle was removed and the vessel was quenched rapidly to room temperature via internal cooling water circulator. Gaseous products were gently released to Tedlar gas bags through the gas outlet valve for further analysis. Subsequently, the vessel was opened for solid and liquid products separation and collection. Liquid products were filtered through 0.45 μm PTFE membranes and kept in glass vials at 4 °C before analysis. The solid residues were carefully collected and oven dried at 105 °C overnight. The overall schematic procedure is depicted in Fig. 1. The dry solids were ground into less than 0.5 mm fine powders and termed hydrochars hereinafter. The hydrochars were denoted as HC-T, where T stands for the reaction temperature.

For set 2 (Run 11–13), the feedstocks were 182 g of DSS and a specific amount of CaO (see Table 2). This set of experiments

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