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Simultaneous production of biocrude oil and recovery of nutrients and metals from human feces via hydrothermal liquefaction

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ABSTRACT

Hydrothermal liquefaction (HTL) is a thermochemical process specifically suitable for treating wet wastes. This study investigated its potential for the production of biocrude oil and the recovery of nutrients and metals from human feces via HTL. Specifically, the effects of temperature (260 °C, 300 °C, 340 °C), retention time (10 min, 30 min, 50 min) and total solid (TS) content (5%, 15%, 25%) were studied. The maximum liquefied fraction was 87.89% and the highest biocrude yield reached 34.44% with a higher heating value of 40.29 MJ/kg. Experimental results showed that 54% of carbon in the human feces was migrated to the biocrude oil while 72% of nitrogen was released to the aqueous phase. In addition, most of heavy and alkaline-earth metal elements in the human feces, including Ca (89%), Mg (81%), Al (88%), Fe (72%) and Zn (94%) were distributed in the solid residue, whereas K (89%) and Na (73%) were mainly dissolved into the aqueous phase. This study demonstrated that the efficient degradation of human waste via HTL without any pretreatment and its potential for the valorization in biocrude oil as well as separated nutrients and metals.

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1. Introduction

Wet human feces is generated in a range of 116–200 g/person/d in China [1]. Currently, 1 billion people still use open defecation while another 2.5 billion people do not have improved sanitation facilities [2]. Human feces contains an array of pathogens, that can cause waterborne diseases if being released into the environment without proper treatment [3,4].

On the other hand, human feces could be regarded as a renewable feedstock for different purposes. Composting of human feces has been an effective method for killing pathogenic bacteria and balancing the ratio between carbon and nitrogen [4]. In addition, anaerobic digestion is frequently used to treat feces for biogas production [5]. However, both composting and anaerobic digestion require a long time to stabilize the feces [3,5]. In comparison, pyrolysis is a much faster process, but it is under high temperatures (>400 °C) and requires an energy-intensive drying process of the wet waste [6]. Hydrothermal liquefaction (HTL) allows for the direct conversion of wet waste into biocrude oil at temperatures of 200–350 °C and pressures of 5–20 MPa, which has recently drawn increasing attention [7]. It is important to point out that HTL also kills all the pathogens in the fecal feedstock due to the high temperature treatment. Characteristically, HTL is different from oil extraction and pyrolysis in two distinct aspects: (1) there is no need to dry the feedstock, making HTL especially suitable for wet biomass with a total solid (TS) content of 10–25% [8]; (2) not only lipids but also proteins and carbohydrates can participate in HTL conversion to produce biocrude oil [7,9]. Previous study showed that swine manure was successfully converted to biocrude oil, and the highest oil yield was 24.2% with a higher heating value (HHV) of 36.05 MJ/kg [10]. HTL could also reduce negative environmental impacts associated with swine manure, because it completely deactivated the antibiotic resistant genes and destroyed the natural pathways of antibiotic resistant genes to the environment [11].

HTL of human feces could weaken its pollution to the environment as well as provide a value-added outlet for human feces. This study focused on the HTL of human feces for the production of biocrude oil as well as recovery of nutrients and metals. The objectives of this study were twofold: (1) to investigate the degradation of

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human feces and its potential for the production of biocrude oil via HTL; (2) to study the elemental distribution (including organic elements and metal elements) in HTL products: biocrude oil, aqueous phase, gases and solid residue.

2. Materials and methods

2.1. Feedstock characterization

Fresh human feces was obtained from a latrine in a suburb of Beijing, China. It was homogenized through stirring for 20 min and then stored in a refrigerator at −20 °C prior to use. The methods for proximate and biochemical analysis of the feedstock were performed as previously described [12]. The characteristics of the human feces in this study (Table 1) were similar to the literature [3].

The HHVs of human feces and biocrude oil were calculated according to the Dulong formula [13]:

$$\mathrm{HHV}\ (\mathrm{MJ/kg}) = 0.3383\mathrm{C} + 1.422(\mathrm{H} - \mathrm{O}/16)$$

where C, H and O were the weight percentages of carbon, hydrogen and oxygen in the feedstock and biocrude oil, respectively.

Total nitrogen (TN) of the aqueous phase was calculated by Eq. (1):

$$\mathrm{TN}\ (\mathrm{g/L}) = \frac{N_{\mathrm{feedstock}} - N_{\mathrm{biocrude\ oil}} - N_{\mathrm{solid\ residue}}}{V_{\mathrm{aqueous\ phase}}} \quad (1)$$

where $N_{feedstock}$, $N_{biocrude\ oil}$ and $N_{solid\ residue}$ were the mass of nitrogen in the human feces, biocrude oil and solid residue, respectively. $V_{aqueous\ phase}$ was the volume of the aqueous phase. It was assumed that there was a negligible amount of nitrogen formed in the gaseous products [14].

2.2. Experimental procedure

An orthogonal experimental design was applied to investigate the effects of the HTL operational parameters, including temperature (260 °C, 300 °C, 340 °C), retention time (10 min, 30 min and 50 min) and TS (5%, 15% and 25%) on the production of biocrude oil from human feces. The experiments were performed in a 100 mL stainless steel batch reactor (Model 4598, Parr Instrument Company, USA) with a heating furnace and a stirrer. The maximum operating temperature and pressure of the reactor are 500 °C and 35 MPa, respectively. The heating rate was 8–10 °C/min. The separation of the products was carried out as previously described [15].

Table 1
Proximate analysis, biochemical composition and organic elemental analysis of human feces.

Parameters	This study	Literature [3]
Proximate analysis (%)		
TS	14.89 ± 0.03	19.6 ± 3.8
Ash (based on dry biomass)	11.49 ± 0.24	17.0 ± 1.3
Biochemical analysis (%)		
Protein	34.68 ± 0.63	–
Lipid	14.01 ± 0.41	–
Cellulose	22.42 ± 0.53	–
Hemicellulose	2.48 ± 0.06	–
Lignin	5.66 ± 0.26	–
Non-fibrous carbohydrate*	9.26 ± 0.39	–
Organic element analysis (%)		
C	47.77 ± 0.19	42.4 ± 1.3
H	7.19 ± 0.03	6.9 ± 0.9
N	6.13 ± 0.12	5.9 ± 1.0
O[a]	33.83 ± 0.31	43.1 ± 3.1
HHV(MJ/kg)	20.38 ± 0.09	18.1 ± 2.2

[a] O (%) = 100-C (%)-H (%)-N (%)-Al (%)-Ca (%)-Mg (%)-Zn (%)-Fe (%)-K (%)-Na (%).

2.3. Analytical methods

Organic elemental components (i.e., Carbon, Hydrogen and Nitrogen) of the feedstock and biocrude oil were determined using an elemental analyzer (CE-440 Elemental Analyzer, Exeter Analytical, Inc. USA). Fourier transform infrared tests were carried out using FT-IR spectroscopy (Nicolet 6700, Thermo Fisher Scientific, USA). ^{1}H nuclear magnetic resonance (NMR) was performed using a JNM-ECA600 (JEOL Ltd. Japan) spectrometer to determine the functional groups in the biocrude oil. Acetone was used as the solvent. The ^{1}H spectra of biocrude oil were acquired at 600 MHz with a 90° pulse angle.

Total carbon (TC), total organic carbon (TOC), total phosphorous (TP) and pH values were determined as previously described [16]. Chemical oxygen demand (COD) of the aqueous phase was measured using a water quality analyzer (DR 2800, HACH, USA).

Metal elements (i.e., Al, Ca, Mg, Zn, Fe, K and Na) were measured through an inductively coupled plasma optical emission spectrometer (ICP-OES, iCAP6300, ThermoFisher, USA). Gaseous products were analyzed using a gas chromatograph (SP-6890, Rainbow Chemical Instrument Co., LTD, China) with a capillary column (TDX-01, inner diameter: 0.25 mm) and nitrogen as the carrier gas at 50 ml/min. The measured gases were CO_2, H_2 and CH_4.

The liquefied fraction was determined based on dry feedstock using Eq. (2) and the yields of the biocrude oil, solid residue, gases and aqueous phase were calculated using the methods as in a previous study [17].

$$\text{Liquefied fraction}\ (\%) = 100 - \frac{\text{Mass of solid residue}}{\text{Mass of dry feedstock}} \times 100 \quad (2)$$

3. Results and discussion

3.1. Production of biocrude oil from human feces

Liquefied fraction was an important parameter for evaluating the performance of HTL, the maximum liquefied fraction was 87.89% (Fig. 1), obtained at 340 °C, a 30 min retention time and a 5% TS. It meant that nearly all the organic components in the human feces were liquefied and degraded because the rest is the ash content of 11.49% in human feces, illustrating the high performance of HTL on the treatment of human feces. In addition, the biocrude yields varied from 25.09% to 34.44%, where the highest biocrude yield (34.44%) was obtained at 340 °C, at a 10 min retention time and a 25% TS. In comparison, the highest HHV (40.65 MJ/kg) of the biocrude oil was achieved at 340 °C, at a 30 min retention time and a 5% TS, suggesting that the condition for the highest oil HHV was not consistent with that for the highest biocrude yield. The products under these two conditions were chosen for further elemental analysis.

The biochemical composition of the feedstock has a great effect on the biocrude yield [12,18]. The conversion rate of carbohydrates to biocrude oil was 0.001/min, in comparison to 0.28/min for proteins and 0.33/min for lipids at 350 °C during the HTL of algae [19]. The high contents of proteins and lipids (total 48.69%) in the human feces in this study led to a relatively high biocrude yield. In addition, the analysis of the significance (Fig. S1) indicated that the TS content and reaction temperature were important factors for the production of biocrude oil while the retention time was an insignificant parameter. The results were similar to a previous study which used algal biomass from Dianchi Lake as the feedstock [20].

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