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Co-conversion of waste activated sludge and sawdust through hydrothermal liquefaction: Optimization of reaction parameters using response surface methodology

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HIGHLIGHTS

- Co-conversion of wastewater sludge and sawdust was investigated.
- The operating conditions were optimized based on response surface methodology.
- A maximum of 33.7 wt% bio-oil yield was obtained at optimum operating conditions.
- Co-conversion resulted in significant molecular weight improvement of the bio-oil.
- The water-soluble product was used for biogas production by anaerobic digestion.

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ABSTRACT

The present paper examines the co-conversion of waste activated sludge and birchwood sawdust to bio-oil via hydrothermal liquefaction. The purpose of using the sawdust with sludge was to increase the solids concentration using another waste material for possible resource recovery. The operating conditions including reaction temperature, reaction time and solids concentration were optimized based on the response surface methodology for the maximum bio-oil production. A maximum of 33.7 wt% bio-oil yield was obtained at optimum operating conditions of 310 °C, 10 min, and 10 wt% concentration. Comparison of this oil with the oil produced from only sawdust showed a significant improvement in the molecular weight of the bio-oil by having lower molecular weight (hence less viscosity), indicating the presence of lighter components, with a slight decrease in bio-oil yield. The optimized operating condition could be used to effectively co-liquefy waste activated sludge and sawdust with the advantage of producing higher quality bio-oil with respect to molecular weight. The water-soluble product which is the largest fraction of by-products from the co-conversion was tested as a feedstock for biogas production through anaerobic digestion and resulted in 800 ml bio-methane production per 0.816 g of TOC or 2.09 g of COD of this waste stream.

1. Introduction

Growing interest in renewable energies due to shrinking reserves of fossil fuels and climate change concerns have led to extensive research towards gaseous and liquid fuels production from renewable energy resources such as biomass and wastes. Energy generation from municipal and industrial wastes such as wastewater sludge is also an environmentally friendly way to deal with large volume of waste disposal with the additional advantage of eliminating part of the indirect greenhouse gas emissions from energy crops-derived biofuels [1].

Sludge management is one of the most challenging and costly tasks of wastewater treatment plants due to high water content and poor dewaterability. Currently, there are several options for energy recovery from sludge [2], of which the most important ways are biological and thermochemical processes. Hydrothermal liquefaction (HTL) is a thermochemical process where raw sludge with high water content can be heat treated directly in the absence of oxygen at 150–450 °C under pressure (up to 25–30 MPa) [2]. HTL is a promising technology for converting waste biomass with high water content into liquid fuels. It eliminates the need of costly de-watering/drying process that is otherwise required in other

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thermal/thermochemical processes. The remarkable properties of water such as low dielectric constant and high ionic product play important roles as a solvent in liquefaction. The main products of HTL treated sludge are bio-oil, water-soluble products (WSP), char, and gases. The process can be made self-sufficient in energy using a part of the produced oil and char to provide heat for the HTL process and it has been found to be cost-effective compared to incineration [3]. It can also achieve the additional benefit of pathogen reduction meeting the stringent regulation on sludge land applications.

HTL has been used for treating dairy manure by converting high-water-content sludge/bio-solids into value-added products, mainly bio-oil and solid residue (bio-char) [4]. An early study of sewage sludge liquefaction was performed by Kranich and Eralp [5,6]. Sewage sludge was converted to oil at different reaction temperatures in presence of hydrogen as reducing gas and catalysts such as Na_2CO_3, $NiCO_3$, and Na_2MnO_4. The oil yields were less than 20 wt% with water as the reaction medium. A pilot scale study was carried out by Molton et al. where primary and undigested sludge with 20% total solids (TS) were heated at 300 °C and 10 MPa pressure in a continuous reactor with 30 L/h flow rate and hydraulic retention time of 90 min [2,3]. The technology was patented as sludge-to-oil reaction system (STORS) with oil yields ranging from 10 to 20 wt% and char from 5 to 30 wt%. In a more recent work by Vardon et al., bio-oil characteristics of three different wastewater feedstocks (Spirulina algae, swine manure, and digested anaerobic sludge) were compared using HTL conditions of 300 °C, 10–12 MPa pressure, and 30 min reaction time [7]. The bio-oil yield from digested sludge with the total solids of 26% was found to be the lowest (9.4 wt%) compared to other feedstocks. The oil from digested sludge had the highest amount of high boiling point compounds leading to a high molecular weight of 3470 g/mol. Although HTL has been applied earlier to produce energy from sludge, the bio-oil yield is usually very low due to low solid concentration in wastewater sludge and oil with high molecular weight is produced. High molecular weight can result in high viscosity and instability of bio-oils [8]. Complete parametric studies for maximum bio-oil yields and energy recovery are also lacking in literature. In addition, there should be a proper method to utilize the water soluble phase as the largest fraction of the HTL products are distributed in this waste stream as a by-product. While this is a major challenge for HTL operations, there are only a few studies suggesting a possible use for this waste stream. Tian et al. suggested that by using a proper recycling method, the nutrients in the WSP along with the carbon dioxide of the HTL gaseous product can be used for algal biomass production, which can provide another type of feedstock for the production of bio-oil [9].

The objective of the present work was to find the optimum operating conditions for the maximum bio-oil production from wastewater sludge based on experimental design. Successful implementation of this operation can result in optimal and efficient use of wastewater sludge as an energy resource as well as analysis and optimization of this technology as a process of recovering energy from waste for larger applications. Waste activated sludge (WAS) was used as the main feedstock with the addition of birch wood sawdust as the co-feed. There have been a few other studies on co-conversion of different types of feedstock, mostly algal biomass; for example co-liquefaction of swine manure and mixed-culture algal biomass [10], microalgae and macro-algae [11], microalgae and lignocellulosic biomass [12], and liquefaction of mixed culture microalgal strains [13]. There have also been some studies on swine manure/crude glycerol co-liquefaction [14], secondary pulp/paper-mill sludge and waste newspaper liquefaction [15] and co-liquefaction of spent coffee grounds and lignocellulosic feedstock [16]. To the best of our knowledge, this is the first report on co-liquefaction of WAS and sawdust. Co-conversion of sludge with agricultural biomass allows higher percentage of solids and improves the process economy while recovering energy from the waste and influencing the products yields. It also allows for using two waste streams and thus can help resolve waste management and increases the efficiency by converting two renewable resources into sustainable bio-energy products. The operating conditions such as temperature, reaction time, and solids concentration were optimized using experimental design. Although, co-conversion of some wastes streams has been tested earlier in literature, application of experimental design to optimize HTL parameters for the co-conversion of waste activated sludge and birchwood was never evaluated. Based on a previous catalyst screening study performed by the authors [17], potassium hydroxide (KOH) was used as a homogenous catalyst in the process. The properties of the produced bio-oil were determined and compared with the oils obtained from sludge or sawdust by other researchers. The water-soluble phase from the HTL was tested as a feedstock for biogas production through anaerobic digestion which provides a novel process for recovering energy from this major by-product and also makes the co-production of biogas and bio-crude oil from a waste stream feasible.

2. Materials and methods

2.1. Materials

Birch wood sawdust was supplied from a local lumber mill and the waste activated sludge was collected from Adelaide Pollution Control Plant, London, Ontario. The WAS samples were taken from rotary drum thickeners and stored at 4 °C prior to the experiments. The proximate and ultimate analyses results of birch wood sample and characteristics of WAS are presented in Table 1. Potassium hydroxide was purchased from Sigma-Aldrich and used as catalyst as received.

A.C.S. reagent-grade acetone, used as reactor rinsing/washing solvent for product separation, was purchased from Caledon Laboratory Chemicals (ON, Canada) and used as received.

2.2. Experimental setup

Hydrothermal liquefaction experiments were performed in a 100 mL stirred reactor (Parr 4590 Micro Bench top reactor). The

Table 1
Characteristics of birchwood sawdust and waste activated sludge (WAS).

Parameter	Birchwood sawdust	WAS
Proximate analysis		
Volatile matter (VM)[a,b] (wt%)	83.5	62.2
Fixed carbon (FC)[a,b] (wt%)	16.3	14.1
Ash[a] (wt%)	0.231	23.6
Moisture (wt%)	6.49	96.1
pH	–	7.84
Ultimate analysis[a]		
C (wt%)	47.6	38.0
H (wt%)	6.34	5.23
N (wt%)	0	7.20
S (wt%)	0	0.749
O[c] (wt%)	45.9	25.2
H/C	1.59	1.65
N/C	0	0.162
O/C	0.722	0.498
HHV[a,d] (MJ/kg)	16.9	16.0

[a] On a dry basis.
[b] Determined by TGA.
[c] Calculated by difference (100% - C% - H% - N% -S%-Ash%).
[d] Higher Heating Value (HHV) calculated by Dulong formula, i.e., HHV (MJ/kg) = 0.3383C+1.422(H-O/8).