Bioresource Technology 223 (2017) 115–120

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Treatment of aqueous phase of bio-oil by granular activated carbon and evaluation of biogas production

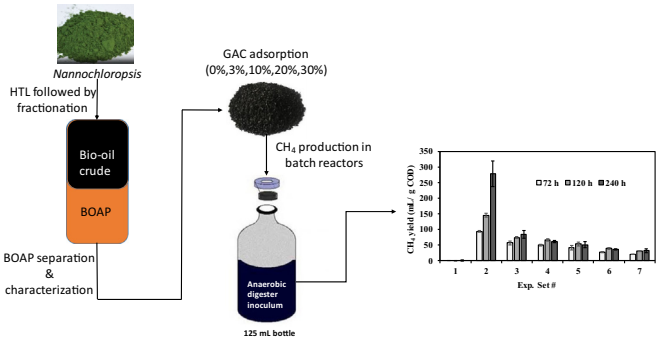
Saravanan R. Shanmugam, Sushil Adhikari *, Zhouhang Wang, Rajdeep Shakya

Department of Biosystems Engineering, Auburn University, Auburn, AL 36849 USA

HIGHLIGHTS

- GAC treatment of BOAP was conducted to remove complex organics.
- GC–MS analysis showed presence of large amounts of nitrogenous compounds.
- Higher COD reduction and CH_4 production were observed in 30% GAC treated BOAP.
- Almost 67% of initial COD reduction is possible using combination of GAC and AD.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 22 August 2016
Received in revised form 29 September 2016
Accepted 2 October 2016
Available online 13 October 2016

Keywords:
Bio-oil aqueous phase
Activated carbon treatment
Methane production
Hydrothermal liquefaction
Algae

ABSTRACT

Hydrothermal liquefaction of wet biomass such as algae is a promising thermochemical process for the production of bio-oil. Bio-oil aqueous phase generated during liquefaction process is rich in complex organics and can be utilized for biogas production following its pre-treatment with granular activated carbon. In our study, use of 30% activated carbon resulted in higher chemical oxygen demand (COD) reduction (53 ± 0.3%) from aqueous phase. Higher CH_4 production (84 ± 12 mL/g COD) was also observed in 30% carbon-treated aqueous phase fed cultures, whereas only 32 ± 6 mL CH_4/g COD was observed in control (non-carbon treated) cultures. The results from this study indicate that almost 67 ± 0.3% initial COD of aqueous phase can be reduced using a combination of both carbon treatment and biogas production. This study shows that aqueous phase can be utilized for CH_4 production.

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1. Introduction

Biomass is one of the most abundant and an important part of a sustainable renewable energy system. Over the past decade, conversion of biomass into liquid fuels has gained a lot of attention as compared to direct combustion (Toor et al., 2011). Thermochemical processes such as pyrolysis (Mahadevan et al., 2016), gasification (Sadhwani et al., 2016), and hydrothermal liquefaction (Shakya et al., 2015) are used in conversion of biomass to liquid fuels. Among these thermochemical methods, hydrothermal liquefaction (HTL) process is mainly attractive to wet biomass feedstocks, such as algae, because it eliminates a need for drying biomass feed, as required in other thermochemical processes such as pyrolysis and gasification (Bridgewater et al., 1999). Due to the presence of large amount of water in the feedstocks, the products generated from HTL process are separated into aqueous phase and organic phase. The organic phase (often called as biocrude) produced from algal biomass using this process is a complex mixture of oxygenated hydrocarbons and nitrogenated compounds

* Corresponding author.
E-mail address: sushil.adhikari@auburn.edu (S. Adhikari).

http://dx.doi.org/10.1016/j.biortech.2016.10.008
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such as aromatics, short chain carboxylic acids, ketones, phenolics, sugars and furan derivatives depending upon the type of biomass (Yanik et al., 2007; Hassan et al., 2009). Complex nature of this bio-oil together with presence of high oxygen levels prevents them from direct utilization as fuel. Currently, the research attention in this area mainly focuses on improving the bio-oil yield from different types of algal species, reducing the char content during HTL process and catalytic upgrading of the bio-oil.

Aqueous phase, on the other hand, contains sugars, furan derivatives, and other water soluble organics formed during the HTL process. The bio-oil aqueous phase (BOAP) thus generated is also a complex mixture comprising several organic compounds. Thus, the BOAP cannot be discharged directly into the water stream due to high level of carbon content of this waste. Hence, proper treatment of this waste needs to be carried out onsite before discharging it out into nearby treatment facilities, waterbodies, or streams. Current research in this area focuses catalytic process to extract energy from BOAP in the form of hydrogen and methane using catalytic gasification, alkanes (ranging from C1 to C6), and polyols such as methanol, ethylene glycol, 1,2-propanediol, 1,4-butanediol (Vispute and Huber, 2009; Elliot et al., 2012). Production of other value-added chemicals as 5-hydrodymethylfurfural, furfural, and phenolics from BOAP is also reported in literature (Abou-Yousef and Hassan, 2014; Elkasabi et al., 2015). Although the energy/carbon recovery from BOAP using these alternatives seems promising, many of these processes are energy intensive and expensive due to the use of expensive catalysts, solvents along with low concentration of chemicals present. For example, catalytic conversion of BOAP involves the use of expensive catalysts such as Ru/C and Pt/Al_2O_3 and also requires high operating temperatures and pressures (Vispute and Huber, 2009). Similarly, production of furan derivatives from BOAP requires use of solvents such as 2-butanone, methylisobutylketone and a selective cation exchange resin (Abou-Yousef and Hassan, 2014). Due to these reasons, an efficient alternative method has to be developed to treat and also to recover energy from BOAP.

Anaerobic digestion (AD) is an efficient method to treat organic wastes and obtain bioenergy in the form of methane (CH_4) (McCarty et al., 2011). AD is carried out by several groups of microorganisms that are involved in hydrolysis, acidogenesis, acetogensis and methanogenesis. The final step in AD results in formation of CH_4 and carbon dioxide (CO_2). Proper utilization of BOAP could generate additional energy generation in the form of biogas-CH_4 generation. Owing to the complex nature of organics in BOAP, some form of pre-treatment needs to be carried out before conducting AD. Granular activated carbon (GAC) has been proven to be an excellent option for removal of broad range of synthetic organic compounds present in drinking water sources and industrial wastewaters (Neeko and Fatemi, 2013). To the best of our knowledge, studies directed towards treatment of BOAP produced via HTL and its evaluation for biogas production has not been conducted in the past. Hence, the objectives of this study were (i) to evaluate the effect of GAC on chemical oxygen demand (COD) removal of BOAP, and (ii) to examine biogas production from both GAC-treated BOAP and BOAP generated during HTL of algae.

2. Materials and methods

2.1. Materials

Algae sample of *Nannochloropsis* used in HTL process was obtained in the form of slurry from Reed Mariculture Inc. (Campbell, CA) and stored at 4 °C until used. The biochemical composition of the algae species is as follows: carbohydrates (9), protein (63) and lipids (18), all in wt.%. Both proximate and ultimate analyses of this algal species were previously reported elsewhere (Shakya et al., 2015). Ultrapure (Type 1) water (Synergy Ultrapure Water Systems, EMD Millipore) was used for conducting HTL experiments.

2.2. Activated charcoal treatment

BOAP obtained from phase separation of bio-oil was subjected to GAC treatment. Granular GAC (BET surface area – 289 ± 15 m^2/g) purchased from Sigma-Aldrich was used for the BOAP adsorption studies. The moisture content (on wet basis), volatile solids content (on dry basis), fixed carbon content (on dry basis) and ash content (on dry basis) of GAC were found to be 8.17 ± 0.2%, of 8.00 ± 0.6%, of 61.1 ± 2.0% and 30.9 ± 1.4%, respectively. The particle density of GAC is about 7.27 ± 0.6 (g/cm^3). The average pore diameter, pore volume and particle size distribution of GAC was found to be about 2.6 nm, 0.188 g/cm^3 and 0.72 ± 0.22 mm, respectively. Adsorption studies were conducted at 25 °C in an orbital shaker rotating at 100 rpm. Varying levels of GAC at 0%, 3%, 10%, 20% and 30% (w/w) were added to 20 mL BOAP samples and this mixture was left shaking for 24 h. In order to determine optimum time for absorption, samples were withdrawn at regular time intervals and analyzed for the total organic carbon (TOC) and total nitrogen (TN) contents. Collected samples were then centrifuged at 7500 rpm for 20 min to separate the solids. This supernatant is then filtered using a 0.45 μm polypropylene membrane (GE Osmonics, MN) filters and stored at 4 °C until further used. There was negligible amount of TOC reduction of BOAP in all GAC additions after 1 h (data not shown) so we decided to use 1 h for absorption with GAC.

2.3. Inoculum source and experimental methodology

The inoculum used for conducting AD experiments was obtained from Jackson Pike wastewater treatment facility (Columbus, Georgia, USA). The inoculum was procured from large anaerobic digester generating methane for electric grids. The inoculum had an initial pH of 7.8, total suspended solids about 54,000 mg/L and volatile suspended solids about 30,000 mg/L. The inoculum was collected in two jerry cans (15 US gallons) and stored at 4 °C until used. The VSS and TSS analyses of the inoculum were once again conducted at Auburn University using Standard methods (APHA, 2010) before the start of experiment. The basal medium used for conducting biogas production experiments in this study was adapted from literature (Weigant and Lettinga, 1985). All reagents used for basal medium preparation were analytical grade. The inoculum and basal media was mixed thoroughly using a magnetic stirrer and purged continuously with N_2 (Airgas, Auburn, AL, >99% purity) to achieve anaerobic conditions.

Experiments were conducted in small 125 mL serum bottle batch reactors with 55 mL working volume. The serum bottles were capped tightly with the help of silicone rubber septa and aluminum caps. The batch reactors had an initial inoculum volatile suspended solids (VSS) concentration of 3000 ± 100 mg/L. This is equal to 4260 ± 142 mg COD/L (using a conversion factor of 1.42 g COD/ g VSS). BOAP and GAC-treated BOAP was used as substrates in this experiment at a substrate (COD) loading of 1 g/L. Pure glucose (1 g/L) was used as a substrate control to compare the methane production with BOAP fed cultures. The substrate to inoculum ratio was maintained at 0.24 (calculated using 1000 mg COD/L substrate and 4260 mg COD/L inoculum concentration) in all experimental sets (Table 1). Separate culture control (no substrate addition) was also conducted to analyze any background gas production obtained from inoculum and basal media. Volume of culture, basal media and substrate (added on COD basis)

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