



Effects of solvent participation and controlled product separation on biomass liquefaction: A case study of sewage sludge



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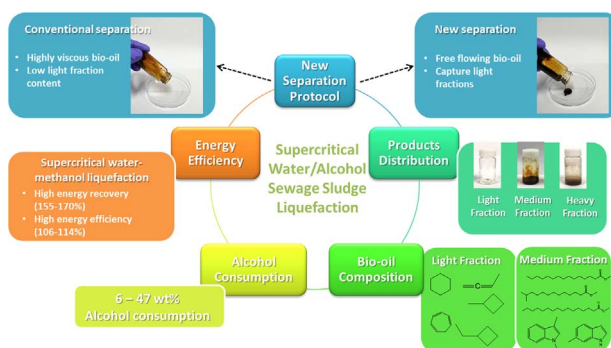
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HIGHLIGHTS

- Bio-oil was produced using liquefaction of sewage sludge in water-alcohol mixture.
- A sophisticated separation method was developed to capture light fractions.
- 10–25 wt% increase in bio-oil yield resulted because of captured light fractions.
- Amount of alcohol participated into reaction was 6–47 wt% depending on reactions.
- Considering light fractions/alcohol participation, energy efficiency was 106–114%

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:

Sewage sludge
Liquefaction
Bio-oil
Supercritical fluids
Separation

ABSTRACT

In this study, the effects of product separation on the distribution of liquid products and the energy efficiency of sewage sludge liquefaction in supercritical alcohol and supercritical alcohol–water mixtures were investigated. While considering alcohol participation in the liquefaction reaction (6–47 wt%), the effects of process parameters such as temperature (300–400 °C), residence time (10–120 min), concentration (9.1–25.0 wt%), and type of supercritical fluid (water, methanol, ethanol, water–alcohol mixture) on the yield and properties of bio-oils were examined. Accounting for alcohol participation and product separation allowed the bio-oil yield, energy recovery, and energy efficiency to be newly defined. Application of the new separation protocol developed in this study realized a 10–25 wt% increase in bio-oil yield because light fractions were efficiently captured. When supercritical methanol was used, the light fractions consisted primarily of methylated short-chain esters, whereas ketones and alcohols were the major species when supercritical ethanol was used. Liquefaction at 400 °C and 20 wt% sewage sludge in a mixture of water–methanol (80:20, v/v) resulted in a bio-oil with a high calorific value (35.8 MJ kg⁻¹), achieving 155% energy recovery and 106% energy efficiency. Computational fluid dynamics (CFD) analysis of bio-oil combustion conducted in a commercial boiler demonstrated that cofiring with a mixture of petroleum heavy oil and bio-oil resulted in a high firing temperature of 1570 °C and a heat transfer rate, which were comparable to that obtained from conventional heavy oil firing.

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1. Introduction

Biomass liquefaction, which utilizes a high-temperature and high-pressure solvent to decompose solid biomass into liquid bio-oil, is one of the most promising techniques for the conversion of high-water-content biomass (e.g., sewage sludge [1–3], macroalgae [4–7], microalgae [8,9], lignocellulosic biomass [10–15], lignin [16–18], and animal manure [19,20]) into liquid fuel. As highly energy-intensive and costly drying steps can be omitted, this technique allows the development of energy-efficient biomass-to-fuel process [21,22]. In contrast to fast pyrolysis, some degree of deoxygenation occurs during liquefaction, which could enhance the calorific value of the bio-oil produced, even without the use of external catalysts and molecular hydrogen. Despite these advantages, recovery of the produced bio-oil from the solvents used for liquefaction is quite complex, and various approaches, including liquid–liquid extraction, vacuum drying, and combinations of the two, have been examined under various operating conditions. In some cases, water remaining in the bio-oil phase can be removed using a highly energy-intensive vacuum drying method. However, some fractions of bio-oil produced during liquefaction (e.g., light fractions and water-soluble oil) cannot be completely recovered using the simple separation methods, leading to low bio-oil yields and low energy recovery. The product separation issue is highly critical when the high-water-content biomass is used as the liquefaction feedstock. Typically, a large amount of water-soluble oil is produced during liquefaction of high-water-content biomass, and thus, to understand the liquefaction behavior, it is very important to find a suitable method to recover, or at least to quantify, the water-soluble oil fraction. Considering that the product yield and energy efficiency of an energy conversion process is highly dependent on the choice of product separation method and protocol, it is crucial to employ well-designed and controlled separation techniques to achieve a complete understanding of liquefaction mechanisms and ultimately develop cost-effective and energy-efficient biomass conversion techniques.

Recently, biomass liquefaction in various organic solvents (e.g., methanol [1,2], ethanol [1,2,23–25], acetone [1], tetralin [26], and 2-methyltetrahydrofuran (MeTHF) [26]) has been proposed to enhance the yield and calorific value of bio-oil. The sewage sludge liquefaction and product separation procedures employed in previous studies are summarized in Table S1. In particular, the use of supercritical alcohols in biomass liquefaction has received considerable attention because of the unique physicochemical properties (e.g., liquid-like density, gas-like diffusivity, low viscosity, and rapid rates of mass and heat transfer) and reactivity (e.g., hydrogen generation [27,28], alkylation [29,30], hydroxyalkylation [31,32], deoxygenation [33,34], and esterification [35]) of supercritical alcohols. For example, compared with sub- or supercritical water (sub- or scH_2O)-based liquefaction, the use of supercritical ethanol (scEtOH) in the liquefaction of sewage sludge resulted in higher bio-oil yields of 65–75 wt% [1]. Further, compared with scH_2O -based liquefaction, liquefaction with supercritical methanol (scMeOH) produced a bio-oil with a higher calorific value (35.0 MJ kg^{-1}) [2] and a similar bio-oil yield (67 wt%). In addition, the use of scH_2O –supercritical alcohol mixtures has been demonstrated to produce high-calorific-value bio-oils (up to 37.3 MJ kg^{-1}) [2,24,36]. More recently, the use of various rinsing/recovery solvent after liquefaction and various types of salts (K_2CO_3 , Na_2CO_3), acids (HCOOH , CH_3COOH) and metal-supported catalysts (MoO_3 - $\text{CoO}/\gamma\text{-Al}_2\text{O}_3$, Ru-C, CuSO_4 , ZnSO_4 , CoSO_4 , FeSO_4) in the sewage sludge liquefaction were conducted to enhance conversion and bio-crude yields [36,37]. Although previous studies have demonstrated the great potential of supercritical alcohols in biomass liquefaction, the fundamental roles of alcohol under various liquefaction conditions are still unclear; for example, the “simple” question of how much alcohol participates in sewage sludge liquefaction has not been addressed in previous studies. The uncontrollable loss of bio-oil fractions (e.g., light fractions [24,38]

and water-soluble oils [2,24,38]) using conventional product separation methods makes it difficult to understand the degree of alcohol participation during liquefaction. For example, in our previous study of sewage sludge liquefaction [2], almost 100% conversion of organic matter in the sewage sludge was achieved, but the total yield of organic products (= bio-oil + gas + residual heavy bio-oil fraction and coke in solid residue) was about 60 wt%. As listed in Table S1, various separation methods and conditions have been tested to recover liquid products; thus, the degree of product loss is different in each study, which makes it difficult to compare the results of previous studies. Therefore, to gain a fundamental understanding of the role of solvents in biomass liquefaction, it is necessary to develop a suitable separation method that can minimize the loss of organic products.

Herein, sewage sludge liquefaction in hot-compressed water and alcohol is revisited to elucidate the importance of product separation by employing a new separation protocol. The choice of sewage sludge as the liquefaction feedstock is based on the following reasons. (1) There is considerable need to develop an alternative to conventional sewage sludge treatment methods, such as agricultural use, landfilling, and incineration, as the presence of various toxic substances (e.g., pathogens and heavy metals) and their unpleasant odors [39–41] often cause serious environmental and health issues [42]. Conventional sewage sludge treatment methods suffer from leaching of toxic compounds, accumulation of hazardous compounds, and air pollution [43]. Furthermore, landfilling is currently restricted by land limitations and government regulations [1,44]. (2) Dewatered sewage sludge contains approximately 80 wt% water, with some fraction bound to the surface of organics or trapped inside microorganisms, which make it difficult to treat sewage sludge using conventional thermal methods (e.g., rotary kiln drying and pyrolysis). (3) Considering that sewage sludge contains a high amount of renewable organic matter (in the form of carbohydrates, proteins, lipids, and nucleic acids) with a calorific value of up to 20 MJ kg^{-1} (dry basis) [45], it is highly desirable to develop an appropriate method to convert the organic matter into sustainable biofuel that could be utilized as a combustion or transportation fuel [46,47].

This study attempted to address the following questions concerning sewage sludge liquefaction. (1) If a nonnegligible amount of alcohol participates in the sewage sludge liquefaction reaction, how much alcohol is consumed? (2) Should the amount of alcohol participating in bio-oil production be considered when calculating the bio-oil yield and the energy efficiency of the liquefaction process? (3) Can light fractions and water-soluble oils be recovered completely or partially using a more sophisticated separation protocol? (4) What chemical species are in the light fractions, and are these fractions valuable feedstocks for chemicals or fuels? Herein, for the first time, the amounts of alcohol consumed during sewage sludge liquefaction in supercritical alcohol under different operating conditions were quantified using a newly developed separation protocol. Light and heavy fractions, which were considered as loss in most of the previous studies, were recovered, quantified, and analyzed in this study. Based on the alcohol consumption and product distribution, new definitions of bio-oil yield, energy recovery (ER), and energy efficiency (EE) were proposed. The comparison with the conventional separation method with the new separation method developed in this study was conducted, and the products distribution and properties of bio-oil produced under different liquefaction conditions were discussed in detail. To examine the beneficial roles of scMeOH in the liquefaction of sewage sludge, scMeOH – scH_2O mixtures with a low methanol content (10–30 vol%) were tested. In addition, by analyzing the combustion and heat transfer characteristics in a commercial capacity boiler (with a capacity of 100 MW_e) by computational fluid dynamics (CFD), we demonstrate for the first time that bio-oil produced from sewage sludge can partially replace conventional heavy fuel oil from petroleum refining for the generation of electricity.

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