



Full Length Article

Direct emulsification of crude glycerol and bio-oil without addition of surfactant via ultrasound and mechanical agitation

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ARTICLE INFO

Keywords:

Bio-oil
Crude glycerol
Emulsion
Ultrasound agitation
Mechanical agitation

ABSTRACT

This study investigates the possibility of directly preparing emulsions from crude glycerol and bio-oil by ultrasound and/or mechanical agitation without addition of surfactant. It has been found that the approach is feasible when the loading level of crude glycerol is controlled to ensure soap content being about 1.0% in the emulsion to avoid droplets re-coalescence caused by surfactant insufficiency or overloading. Increasing ultrasound amplitude and agitation time can improve the emulsion stability as a result of moderate temperature raise induced by ultrasound treatment. After ultrasound agitation (40% amplitude, 4 min), the temperature raised to ~80 °C. The emulsion prepared under such condition has stability (~9.0 h) similar to the emulsion prepared by mechanical agitation at 80 °C. A maximum stability of ~15.0 h can be achieved if the emulsions have a suitable soap content (~1.0%) under a combined emulsification process (ultrasound at 40% amplitude for 4 min followed by mechanical agitation for 2 min). The superior of combination of ultrasound and mechanical agitation can be attributed to both effective droplet breakdown by ultrasound agitation and reduction in droplet re-coalescence chance by followed mechanical stirring. Additionally, the prepared crude glycerol/bio-oil emulsion fuels have similar or even better properties compared to the bio-oil. Accelerated ageing has insignificant influence on emulsion stability if soap content is ~1.0% in the emulsion, and the fuel properties of the aged samples showed only minor changes (including slight decrease in viscosity and slight increase in water content but little change in acidity).

1. Introduction

Rapid growth of biodiesel industry has led to a large surplus of glycerol as a by-product [1–5] of which the utilization will bring significant economic and environmental benefits [4,6–8]. Despite of the strategies of mixing glycerol with bio-oil or bio-oil water soluble fraction to prepare fuel blends for stationary applications (e.g. combustion in boilers) [9–11], emulsification of immiscible glycerol and bio-oil can be also a promising alternative strategy for glycerol utilisation [12], in consideration of the potential surfactant roles of some components in crude glycerol (e.g. soap) or even bio-oil (e.g. phenolates [13]). It was reported [12] that with the assistance of suitable surfactant (Span80), the emulsions prepared from bio-oil and pure glycerol could stay stable for over 12 h under optimum mechanical emulsification conditions. However, the presence of impurities such as water, salt or catalyst in crude glycerol, the emulsion stability could significantly reduce to less than one hour due to severe adverse influence on phase separation of bio-oil [12]. Soap, which is another key impurity in crude glycerol and can act as surfactant, surprisingly imposes strongly negative effect on

glycerol/bio-oil/Span80 emulsion system, as a result of surfactant overloading in the system [12]. Therefore, considering the emulsifying ability of soap, it may be possible to prepare emulsion from crude glycerol and bio-oil directly, without addition of extra surfactant.

Emulsification can be achieved by various agitation processes including mechanical agitation (e.g. in a rotor-stator system), ultrasound agitation (e.g. using ultrasound bath or transducer) or membrane emulsification [14–16]. Mechanical emulsification is widely used in both laboratory and industry applications, while ultrasound and membrane emulsification systems are mainly used in laboratories [14,17]. Ultrasound emulsification was initially reported in 1927 by Wood and Loomis [18], and attracted much research and industrial interests afterwards, especially in cosmetic, pharmaceutical and food industries [17,19,20]. It is accepted that ultrasound can create cavitation phenomena that is responsible for effective breakup of droplets to produce stable emulsions [19]. Recently, ultrasound emulsification was adapted to prepare diesel or bio-oil based emulsion fuels, and its effectiveness was compared with conventional mechanical emulsification [17,19,21–25]. Ultrasound emulsification of a water/kerosene system

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Table 1
Composition of crude glycerol (CG) formulated in this study (wt%).

	Glycerol	Soap	Water	NaOH	Methanol
CG1	79.0	10.0	4.0	2.0	5.0
CG2	79.0	5.0	4.0	2.0	10.0

produced smaller droplet sizes and thus more stable emulsions compared to mechanical emulsification [19]. It was also reported [25] that compared to ultrasound emulsification alone, combination of ultrasonic and mechanical produced more stable bio-oil/diesel emulsion. Therefore, this study investigates the preparation of emulsions from bio-oil and crude glycerol by ultrasound, mechanical or a combination of these two methods. Fuel properties of the emulsion fuels will also be investigated such as viscosity, surface tension, acidity and ageing characteristics.

2. Experimental section

2.1. Materials

Bio-oil was purchased from a commercial supplier, who produced the bio-oil at 500 °C by fast pyrolysis of pine wood biomass. Analytical grade glycerol and sodium oleate (hereafter referred to as soap) were bought from Sigma; analytical methanol and sodium hydroxide were purchased from Chem-supply. Two crude glycerol samples (CG1 and CG2) were formulated according to the composition listed in Table 1.

2.2. Emulsification and stability test

Bio-oil and crude glycerol (CG) were mixed at different CG concentrations (referred to as “BOCG-x%” where x is the concentration of CG in the mixture). The mixtures were then subjected to different agitation processes as shown in Fig. 1. Ultrasound emulsification was achieved using an ultrasonic processor (Model: VCX 500; power: 500 W, frequency: 20 kHz) with a standard solid probe. Considering the influence of probe tip position in the liquid on ultrasound emulsification [26], the sample volume and the distance of probe tip in the sample were kept constant to ensure the reproducibility and comparability [27]. Mechanical emulsification was conducted using a T25 digital homogeniser (with 25N-10G dispersing element) at a speed of 3500 rpm for 2 min if not stated otherwise. The stirring speed and time were chosen based on our previous study [12]. The temperature of the mechanical agitation process was controlled by a water bath at designated temperatures (45, 65 and 80 °C). Ultrasound–mechanical

emulsification was processed in sequence of ultrasound agitation of the sample followed by mechanical stirring. Emulsion stability was examined via a method developed previously [12]. Briefly, an emulsified sample was immediately transferred into a sealed glass tube and observed at most a 0.5 h interval under torch light until inhomogeneous state (settling or floating) of the sample takes place. Stability is defined as the stable time during which there is no visible settling or floating of emulsion droplets.

2.3. Sample characterization

A rheometer (model: Haake Mars II) configured with a cone/plate sensor (C35/4 Ti) was employed to measure the viscosity and characterise the thixotropic behaviour of the bio-oil and selected emulsion samples at 25 °C (controlled by a Haake TC501 thermocontroller). The shear rate was firstly raised from 0 to 1000 s⁻¹, hold for 30 s and then reduced to 0 s⁻¹. Viscosity was taken as the average value obtained at the shear rate of 1000 s⁻¹. The surface tension and density of the bio-oil, glycerol, and selected stable bio-oil/glycerol emulsions were determined at room temperature (25 °C) by a surface tensiometer (KSV Sigma 701) and a volumetric cylinder, respectively, with standard deviations less than 1% according to previous methods [10]. Elemental compositions of the samples were determined by a CHN/O analyser (model: PerkinElmer 2400 series II) and the higher heating value (HHV) of samples were calculated based on the elemental composition according to a method reported previously [28]. Following a method reported previously [10], accelerated ageing tests were conducted at 80 °C for 24 h and the water content and total acid number (TAN) of the samples before and after ageing were analysed by a Karl–Fisher titrator (model: Mettler V30) and an acid–base titrator (model: MEP Oil Titrim plus 848), respectively.

3. Results and discussion

3.1. Preparation of crude glycerol/bio-oil emulsion by ultrasound agitation

Depending on feedstock and biodiesel process, crude glycerol can be produced with different compositions [5,29,30]. According to a previous report, composition of major impurities in crude glycerol can be categorized into three main groups based on with or without water addition for separation of biodiesel and crude glycerol, and acidification process for crude glycerol purification. For those with acidification purification or water addition for biodiesel and crude glycerol separation processes, the resulted two crude glycerol types generally have high content of water and catalyst or salt being over 10% and 5% respectively [9]. Preliminary experiments showed those crude glycerol

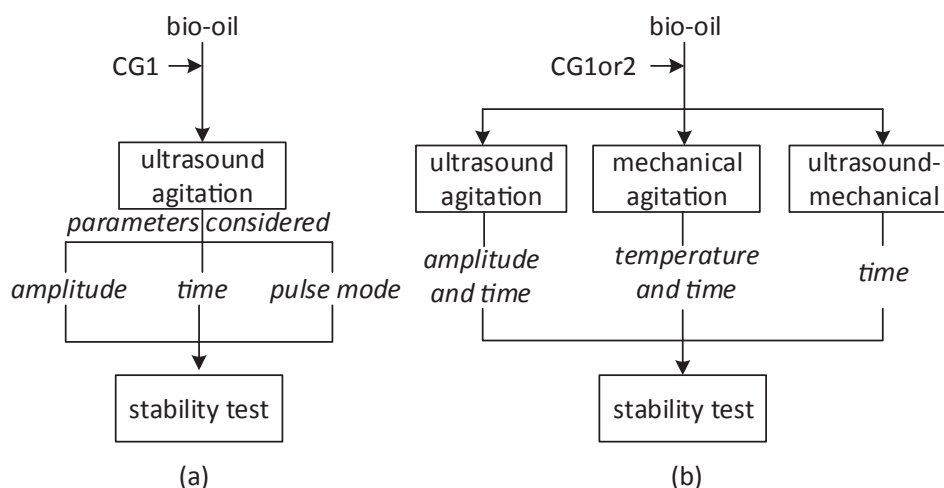


Fig. 1. Emulsification processes of crude glycerol/bio-oil samples considered in this study.

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