



In-situ transesterification process for biodiesel production using spent coffee grounds from the instant coffee industry



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ABSTRACT

Industrial spent coffee grounds (IND-SCG) are a potential non-edible biodiesel feedstock due to their abundant global supply and high oil content. In this study, an *in-situ* transesterification (*in-situ* TE) was developed and scaled up for IND-SCG biodiesel production. Several hurdles must be overcome, including the high acid value, and wide range in particle size of IND-SCG. Washing IND-SCG with methanol reduced its high acid value with negligible loss of oil. Size reduction (0.25–1.68 mm) and an increase of the reaction temperatures (30–60 °C) were found to improve the biodiesel yield significantly. The whole deacidified IND-SCG was processed at 50 °C; and a maximum biodiesel yield of 77% was achieved within 3 h. The process was successfully scaled up for processing 4 kg IND-SCG per batch with a yield comparable to the 30-g scale. The IND-SCG biodiesel met the ASTM biodiesel standard in terms of total glycerin, water content, kinematic viscosity and oxidative stability index (OSI), but its acid value exceeded the standard. A simple process modification using acidic water to neutralize alkaline catalyst during refining step, instead of strong acid, enabled the IND-SCG biodiesel to meet the standard for acid value. The oxidative stability index of the *in-situ* IND-SCG biodiesel was superior to that of the conventional process, probably due to the co-extraction of natural antioxidants.

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1. Introduction

The environmental impact and unreliable supply of petroleum-based fuels continue to spur demand for bio-based transportation fuels such as biodiesel. Biodiesel, also known as fatty acid methyl ester, is mainly produced from vegetable oil. One criticism of the use of vegetable oil for biodiesel production is that it diverts edible oil from the food supply (Yusuf et al., 2011). One projection shows that 80% of biodiesel feedstock will still come from edible vegetable oil in 2020 (OECD-FAO, 2011). Moreover, the cost of this edible feedstock accounts for 70–80% of the total biodiesel production cost (Yusuf et al., 2011). Due to these concerns, there have been growing efforts to identify and use non-edible and inexpensive biodiesel feedstocks (i.e., by-products or waste from other industries).

Spent coffee grounds (SCG), a waste from the coffee industry, has an oil content between 15–20% mass (Kondamudi et al., 2008; Vardon et al., 2013). More than 9 million tons of coffee were consumed in 2014 (International Coffee Organization, 2016); and global coffee production and consumption have been increasing annually. Approximately 50% of the total coffee consumption was from instant coffee (Ramalakshmi et al., 2009). With the availability of more than 4 million tons annually of industrial SCG (IND-SCG) from the instant coffee industry, more than 0.7 million tons of biodiesel could be produced annually. The IND-SCG is therefore an attractive non-edible feedstock for the biodiesel industry.

Several studies have shown that biodiesel of adequate quality can be produced using SCG from local coffee shops via conventional biodiesel production processes at a small scale (<100 g SCG per batch) (Al-Hamamre et al., 2012; Vardon et al., 2013). In conventional biodiesel processes, the SCG biodiesel is synthesized via transesterification (TE) using SCG oil extracted by *n*-hexane solvent (Al-Hamamre et al., 2012; Kondamudi et al., 2008; Vardon et al., 2013). Although this process might be attractive and economical

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at a production scale of 2400 tons SCG day⁻¹ or more (Haas et al., 2004), the amount of SCG from local coffee shops is insufficient for this kind of process. Also, the initial water content in SCG is very high with >70% by mass, which potentially reduces the quality of SCG oil due to hydrolysis during transportation and storage. To prevent oil hydrolysis the wet biomass must be stored frozen and/or dried to a moisture content <5% by mass (Go et al., 2016). However, neither intensive on-site drying nor cold storage during transportation and storage are practical at the scale of coffee-shop SCG.

Use of IND-SCG from instant coffee production is a more promising approach. For example, a single instant coffee plant in Pathum Thani province, Thailand generates 1725 kg day⁻¹ of IND-SCG, dry basis (P. Senawong, personal communication). This steady daily supply of IND-SCG waste may be sufficient to justify development of an on-site biodiesel production unit. However, this amount of IND-SCG is still insufficient for the conventional process (using a solvent-extraction step with *n*-hexane as solvent). An *in-situ* TE process incorporated into the instant coffee plant may be a feasible alternative process. Such a process may be attractive to the instant coffee producers, because on-site biodiesel production will not only add value to IND-SCG, but also reduce the waste management cost.

In an *in-situ* TE process, methanol with an alkaline catalyst is used as the biodiesel reactant and simultaneously as a biodiesel extraction solvent, thereby reducing the size and complexity of biodiesel production systems. The *in-situ* TE process has been successfully used with canola (Haagensohn et al., 2010), soybean (Haas et al., 2004; Haas and Scott, 2007), palm fruit (Jairuob et al., 2013), algae (Suganya et al., 2014), jatropha (Hailegiorgis et al., 2013), cottonseed (Georgogianni et al., 2008; Qian et al., 2008), and castor seed (Pradhan et al., 2012). These studies showed that an *in-situ* TE process may be equal to conventional processes in the yield and quality of biodiesel. In the case of SCG, Park et al. (2016) successfully produced biodiesel from coffee shops' wet SCG via an *in-situ* TE process using acid as the catalyst. However, there are no reports on use of IND-SCG the biodiesel feedstock.

There are several co-benefits to using an *in-situ* TE process at an instant coffee plant, which are (1) the heat waste from the brewing and drying processes in instant coffee production can be used in an on-site *in-situ* TE process; (2) SCG has a high content of antioxidants that would be co-extracted by the methanol used in the *in-situ* TE process (Naidu et al., 2008a), thereby enhancing the oxidative stability of the IND-SCG biodiesel; (3) the defatted IND-SCG after the process has a high energy content, thus can be used for heat and steam generation; and (4) the *in-situ* TE process might be applied as a pretreatment step for bioethanol fermentation. The lipid removal can enhance the glucose hydrolysis in the defatted SCG (Kwon et al., 2013; Rocha et al., 2014).

One of the challenges of using SCG or its oil in TE is its high acid value (free fatty acid; FFA) between 7 and 13 mg KOH g⁻¹ oil (Al-Hamamre et al., 2012; Vardon et al., 2013). This results from the roasting and coffee brewing process, as well as from the wet storage, and is a challenge for IND-SCG as well. This high acid value neutralizes the alkaline catalyst used during the *in-situ* TE process; hence lowering biodiesel yield. Therefore, pretreatment to reduce the acid value of SCG (deacidification) is a very important step in developing a successful *in-situ* TE process. Another problem of IND-SCG is that the coarse, roasted matrix of the IND-SCG particles makes a portion of the oil inaccessible for *in-situ* TE; a better understanding is needed of the effect of IND-SCG particle size on the performance of the process at different process temperatures. One last concern is the lack of understanding of the kinetics of the *in-situ* TE process when used with IND-SCG.

Therefore, the objectives of this study were to develop an *in-situ* TE process that produces acceptable quality IND-SCG biodiesel

while taking into consideration the effect of process temperature, particle size, and reaction time; and to then scale up the process to 4 kg IND-SCG per batch with acceptable biodiesel yield and quality.

2. Materials and methods

2.1. Materials

IND-SCG was kindly provided by Jacobs Douwe Egberts TH Ltd, located in Pathum Thani province, Thailand. They processes coffee beans (*Coffea robusta*) grown in Chum Pon province, Thailand. Analytical grades of methanol, *n*-hexane, and NaOH, H₂SO₄ (98%), and phenolphthalein (1%) in methanol were purchased from VWR (Chicago, IL, USA). 1,1-diphenyl-2-picrylhydrazyl (DPPH), KOH, ethanol (95%) and butylated hydroxyanisole (BHA) were procured from Sigma-Aldrich, Chemical Co. (St. Louis, MO, USA).

2.2. IND-SCG pretreatment process (bench scale)

2.2.1. Drying, grinding, and fractionation

The coffee extraction process uses pressurized water at 125–175 °C. The IND-SCG had 75% initial water content by mass and was sun dried for three days to 30%. The sun dried IND-SCG was further dried in a hot air oven at 105 °C overnight. The dried IND-SCG was fractionated into five different size ranges using US sieve sizes No. 12 (1.68 mm), 16 (1.19 mm), 18 (1.00 mm), 40 (0.42 mm) and 60 (0.25 mm). Due to negligible amount of the fraction of particle size <0.25 mm, it was combined with the fraction 0.25–0.42 mm. The IND-SCG fraction that could not pass through the US sieve size No. 12 was ground using a Kassel food blender (model KBL-550, China) and sieved again through the five different sieve sizes. The five fractions from the grinding step were combined with the corresponding fractions from the initial sieve step, and were reported on a percentage mass basis and used in the subsequent process.

2.2.2. Deacidification

After the sieving and size reduction steps, each IND-SCG fraction was washed with methanol to reduce the high FFA content. Thirty six grams of IND-SCG were suspended in 120 mL methanol in a 500 mL three-neck round bottom flask. The suspension was mixed for 1 h using a 15.7 rad s⁻¹ mixing speed by a two-blade propeller at an extraction temperature of 45 °C. The slurry was then filtered through a Whatman #4 filter paper (Cole-Parmer; Vernon Hills, IL) under vacuum at 6.0–6.2 kPa. Methanol 15 mL was used to rinse the deacidified IND-SCG (DSCG) in the three-neck round bottom flask and filter paper. Finally, the DSCG were dried in a hot air oven at 75 °C overnight, and used in the *in-situ* TE process shown in Fig. 1.

2.3. In-situ TE (small scale)

Sodium methoxide (alkaline catalyst) solution was prepared by dissolving 2.25 g NaOH in methanol and adjusting the volume to 250 mL. Thirty grams of the selected size of DSCG were suspended in 105 mL of sodium methoxide solution using a three-neck round bottom flask as a reactor. The suspension was mixed for 3 h with a mixing speed of 15.7 rad s⁻¹ using a two-blade propeller at the selected temperature between 30 and 60 °C. After 3 h of operation time, catalyst in the liquid fractions was neutralized by adding 2.76 g of H₂SO₄ (Haas et al., 2004), which was calculated based on the stoichiometry of the NaOH and H₂SO₄ reaction. The slurry was then filtered through a Whatman #4 filter paper under vacuum at 6.0–6.2 kPa. The defatted IND-SCG in the glassware and filter paper were rinsed with 15 mL methanol. The filtrate from the *in-situ* TE process and rinse were pooled together and called the “liquid fraction” as exhibited in Fig. 1. This liquid fraction contains fatty acid

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