



Full Length Article

Direct transesterification of spent coffee grounds for biodiesel production



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HIGHLIGHTS

- A one step process to directly making biodiesel from SCGs without solvent extraction.
- 17.08 wt.% of coffee biodiesel yield, oil-to-biodiesel conversion rate of 98.61 wt.%.
- The optimal reaction condition: 12 h; 70 °C with 20 wt.% H₂SO₄ impregnated catalyst.

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ABSTRACT

Studies of spent coffee grounds (SCGs) as a potential biodiesel feedstock in recent years mostly started from solvent extraction to obtain coffee oil, and then converted it into coffee biodiesel in two steps, acid esterification followed by alkaline transesterification. This paper presents a direct transesterification (*in situ*) method that produces biodiesel from SCGs without the need for oil extraction and esterification steps. Prior to the direct transesterification, SCGs were impregnated with sulfuric acid as the catalyst for subsequent direct transesterification, and moisture was removed from the impregnated SCGs. The effects of H₂SO₄ concentration, reaction time, and reaction temperature on biodiesel yield were investigated. The coffee biodiesel yield (wt.% of dried SCGs) reached 17.08 ± 0.70 wt.% under the optimal condition of 70 °C, 20 wt.% sulfuric acid and 12-h reaction time, which was equivalent to an oil-to-biodiesel conversion rate of 98.61 wt.%. 28.87 ml methanol/g oil was used in the *in situ* method. Compositional analysis of fatty acid methyl esters (FAME) indicated that C16:0 (methyl palmitate) and C18:2 (methyl linoleate) were the major components of the coffee biodiesel.

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1. Introduction

The continued use of fossil fuels for centuries worldwide depletes the finite supply and raises concerns over greenhouse gasses (GHGs) and exhaust emissions. As a result, the demand for biodiesel as an alternative to petroleum diesel in the United States has increased significantly [1]. As reported by the United States Environmental Protection Agency (U.S. EPA), the use of B20 (20% biodiesel blended with 80% of petroleum diesel by volume) can decrease particulate matter emission by approximately 10.1% in comparison with that from 100% petroleum diesel. In addition, the emission of carbon monoxide and hydrocarbons can decrease 21.1% and 11.0%, respectively [2].

In recent years, there has been increased interest to produce biodiesel out of spent coffee grounds (SCGs) as a sustainable practice for waste reduction. Coffee is the second largest traded commodity worldwide [3], and the world's coffee production in 2016/2017 is estimated at 9.34 million tons based on the USDA's report. During this period, coffee consumption within the United States was 1.48 million tons [4]. Up to 0.91 g of SCGs can be generated per gram of SCGs, which is a significant rate of waste generation [5]. Research has been conducted to reutilize the SCGs as biodiesel feedstock [6–8], which is summarized in Table S1. These studies first extracted coffee oil from SCGs by solvent extraction (such as hexane, or hexane/isopropanol mixture), and some studies also conducted solvent recovery [9–12]. Coffee oil was also utilized for bio-hydrotreated diesel production and different catalysts were studied [13]. The reported coffee oil yield mostly ranged from 8.6 wt.% to 16.2 wt.% [14–17], while a few reported more than 20 wt.% [17,18], and two reported less than 8 wt.% [13,19]. Additionally, the effect of different solvents on the yield of coffee oil

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was studied based on polarity and quantity [14,17]. It was reported that coffee oil extraction using the Soxhlet apparatus had a higher yield than using a flask under reflux [20]. Also, hot extraction enhanced oil extraction compared to cold extraction [21]. Besides, ultra-sonication applied during coffee oil extraction could increase oil yield as well as lower the solvent usage [22]. The acid values of SCGs oil typically ranged from 6.50 to 16.59 mg KOH/g oil [14,16,18,23], while high acid values of 118.40 and 40.00 mg KOH/g oil were also reported [12,17]. The reason for the difference in SCGs oil content and acid value may be related to the different sources and heterogeneous nature of SCGs (i.e. variations in particle sizes, types of beans, and roasting time, etc.) [24]. The difference may also be explained by the difference in the conditions used for the extraction (different solvents and extraction temperatures) [19]. A two-step process, acid esterification followed by alkaline transesterification is usually performed for high acid value (greater than 2 mg KOH/g) biodiesel feedstock [25,26]. Compositional analysis indicated that the major fatty acid methyl esters (FAME) of coffee biodiesel are C16:0 (Methyl palmitate) and C18:2 (Methyl linoleate) [9,11,18,24,27].

The three-step process (solvent extraction, esterification, and transesterification) to produce biodiesel from SCGs can be costly. Also, the traditional three-step process may be susceptible to increased frequency of operational parameter adjustment (and consequently increased risk of system shutdown), caused by the heterogeneity of the SCGs' composition. Direct transesterification, or *in situ* transesterification, is a biodiesel production method which combines oil extraction, esterification, and transesterification into one step. The direct transesterification method has been practiced on several biodiesel feedstocks, such as algae [28–30], soybean [31], and *Jatropha* [32] etc. [33,34]. A review paper [34] indicated that although both alkaline and acid catalysts were suitable for the reaction, acid catalysts should be used when the FFA (free fatty acid) concentration of the feedstock is higher than 2.0 mg KOH/g oil. For direct transesterification, most studies used a basic catalyst (sodium hydroxide, potassium hydroxide, or sodium methoxide) due to reduced corrosiveness, lower reaction time, and lower amount of catalyst as compared with the acidic process (e.g. H_2SO_4) [29–31].

The *in situ* process of SCGs was recently investigated [35], using chloroform as co-solvent at 95 °C, and sulfuric acid as the best catalyst with a concentration of 92% (wt. H_2SO_4 /wt. wet SCGs). This demonstrated the feasibility of the *in situ* process with SCGs, and offers room for improvement such as temperature, solvent use, and sulfuric acid dosage.

This study presents the use of the direct transesterification method to directly obtain biodiesel from SCGs without the separate solvent extraction and esterification steps (Fig. 1). The effects of H_2SO_4 impregnation, reaction time, and reaction temperature on biodiesel yield were investigated in this study.

2. Materials and methods

Materials: The SCGs used in the present work were collected from a local coffee shop. Solvents used, such as methanol, hexane, and isopropyl alcohol are all HPLC grade (Fisher Scientific). The titration indicator, 1 w/v% phenolphthalein in 50% ethanol, was prepared from a phenolphthalein powder in the laboratory. Potassium hydroxide pellets ($\geq 85\%$) were purchased for titration. Reagents were purchased from Fisher Scientific.

The direct transesterification process was conducted in a Soxhlet apparatus. Both the distillation flask and extractor were wrapped with heating tapes to maintain the desired reaction temperature. SCGs were first impregnated with the catalyst for direct esterification. Since the acid value of our coffee oil (6.18–6.94 mg KOH/g oil) is higher than 2 mg KOH/g oil, sulfuric acid (Certified ACS Plus, 95.0 to 98.0 wt./wt.%) was selected as the catalyst instead of KOH to avoid saponification. SCGs (100 g dry weight) were impregnated by various concentrations of sulfuric acid (5, 10, 15, and 20% of dry SCGs weight). Then, 200 ml deionized water was added for homogenous coating. H_2SO_4 concentration during impregnation was 0.22–0.27 M. The slurry was heated to 70 °C and stirred for 3 h, after which the slurry was completely dried in an oven at 105 °C for 24 h.

50 g of impregnated SCGs in dry basis were loaded into a cellulose thimble. 250 ml of methanol was placed in a distillation flask. After direct transesterification, the reaction mixture (excess methanol, glycerin, and coffee biodiesel) was transported into a rotary evaporator (Buchi Rotavapor® R11 evaporator) for methanol recovery at 70 °C and 25.4 mmHg vacuum. Thereafter, the liquid (biodiesel and glycerin mixture) was transferred into a separatory funnel (Fisher Scientific, 60 ml). 20 ml of pre-heated (80 °C) deionized water was then added to the funnel and the mixture was settled for at least 30 min for glycerin separation [36]. Afterwards, the bottom glycerin layer and mid layer of washing water were drained out. The wash process was repeated until the pH of the wash water became the same as that of the initial deionized water. As a final step, after the washing water was drained, the washed biodiesel was placed into a centrifuge (Thermo Scientific, Sorvall ST 8) to separate any remaining impurities (5000 rpm for

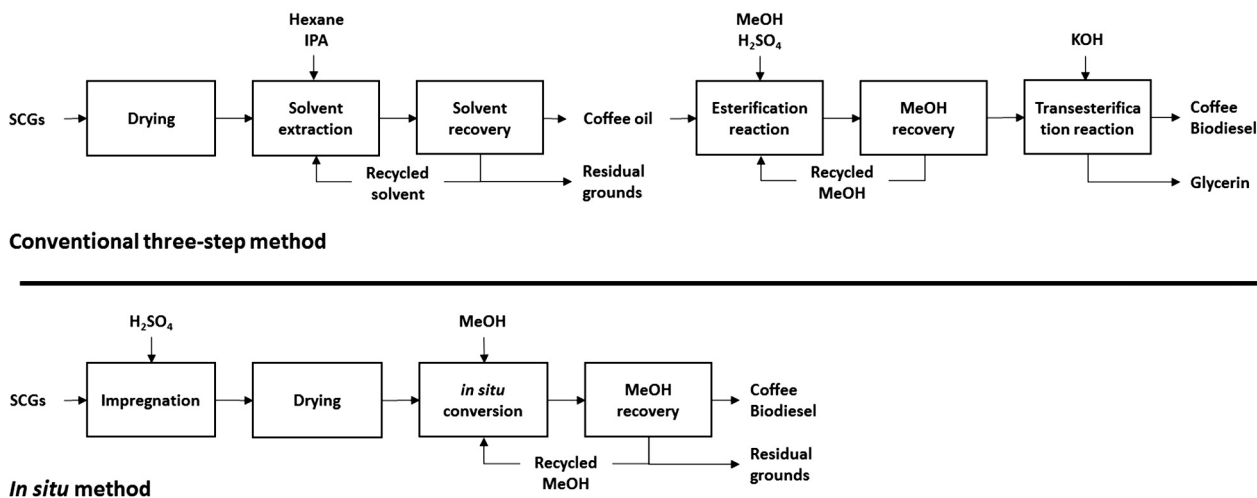


Fig. 1. Flow diagrams of conventional and direct transesterification methods of biodiesel production from the SCGs.

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