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BIODIESEL PRODUCTION FROM SPENT COFFEE GROUNDS

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Abstract

The residue after brewing the spent coffee grounds is an oil-containing waste material having a potential of being used as biodiesel feedstock. Biodiesel production from the waste coffee grounds oil involves collection and transportation of coffee residue, drying, oil extraction, and finally production of biodiesel. Different methods of oil extraction with organic solvents under different conditions show significant differences in the extraction yields. In the manufacturing of biodiesel from coffee oil, the level of reaction completion strongly depends on the quality of the feedstock oil. This paper presents an overview of oil extraction and a method of biodiesel production from spent coffee grounds.

Key words

waste utilization, coffee waste, spent coffee grounds, biodiesel, FAME, oil extraction

INTRODUCTION

Coffee has been consumed for over 1000 years and is currently one of the most widely consumed beverages around the world. According to the International Coffee Organization, more than 9 million tons of this compound were consumed worldwide in 2015/2016 (10/2015 to 09/2016) (1). Two species are of significant economic importance: Coffea Arabica (Arabica) providing 75% of the world production and Coffea Canephora (Robusta), both provides 25% of the world production. So far, many studies have shown the properties of coffee beverages, such as the antioxidant, antibacterial, antiinflammatory and antiobesity properties, and effects on type 2 diabetes mellitus, amongst several others, but little known is the impact on human and environmental health from its disposal in the environment.

During the extraction of the beverage from coffee powder with hot water, a large amount of residue is produced (SCG –spent coffee grounds), and considering the worldwide coffee consumption, it can be concluded that tons of coffee waste are generated from cafeterias and domestic production (2, 3). SCG are the most abundant coffee by-product (45%) generated

not only in the coffee beverage preparation but also during the instant coffee manufacturing. About 2 kg of wet SCG are obtained from each kg of instant coffee produced, with generation of several million tons worldwide every year (4). SCG contains large amounts of organic compounds (i.e. fatty acids, amino acids, polyphenols, minerals and polysaccharides) that justify its valorization. Earlier innovation explored the extraction of specific components such as oil, flavor, terpenes, and alcohols as value-added products. However, by-products of the coffee bean processing can also be considered as interesting sources of raw materials for different applications, mainly as sorbent for the removal of heavy metals and dyes from aqueous solutions, converted into fuel pellets, as potencial functional ingredients for the food industry, biofuel production and composting. There is an urgent need for practical and innovative ideas of using this low cost materials and exploit its full potential, thus increasing the overall sustainability of the coffee agro-industry (5).

Biodiesel (fatty acid methyl ester – FAME) can be derived from edible oil seed crops such as sunflower, palm, rapeseed, soybean, coconut, etc. which are considered as the first generation biodiesel feedstocks (6). Over 95% of biodiesel was produced from edible oil (7). However, use of such feedstocks for biodiesel production has faced problems as they disturb the overall worldwide balance of food reserves and safety (6). In addition, using the virgin edible oils as reactant can result in the increasing cost of feedstock (7). Therefore, other sources of raw materials suitable for biodiesel production are constantly being sought (e.g., non-edible seed crops, waste cooking oil, animal fats, algae) (8, 9).

In recent years, there has been increased interest in production of biodiesel out of SCG as a sustainable practice for waste reduction. Coffee is the second largest traded commodity worldwide, and the world's coffee production in 2016/2017 is estimated at 9.34 million tons (10). Coffee oil, extracted from coffee grounds and/or defective coffee beans was found to be a high quality and cost-effective feedstock for biodiesel production via transesterification compared to other waste sources (9). It is less expensive, exhibits higher stability (due to its high antioxidant content) and a pleasant smell (9, 11). However, the ester-based biodiesel has many limitations, such as producing the excessive glycerol, low energy content, low oxidative stability and plugging from the tank to the engine. Moreover, biodiesel can be added into unmodified diesel engine just only ca. 7 – 10 wt% because of the molecular oxygen presence in the structure of biodiesel (7).

The oil content in the coffee source varies from 11 – 20 wt%, depending on its types. In defective coffee beans, the oil yield ranges from 10 – 12 wt% on a dry weight basis and SCG contain 10 – 15 wt%. [9] Assuming 16 wt% oil content in SCG, the use of this oil as reactant for biodiesel production could produce a lot of tonnes of biodiesel every year. The coffee oil has a potential for biodiesel production and is considered as a low cost feedstock (7).

Biodiesel production from waste coffee ground oil involves collection and transportation of coffee residue, drying, oil extraction and biodiesel production.

OIL EXTRACTION FROM SPENT COFFEE GROUNDS

The SCG must be dried to eliminate the moisture content before being introduced into the extraction process. Moreover, the SCG drying process will prevent the spoilage and microbial growth (12). For oil extraction SCG drying at: (1) 50 °C (13, 14) or (2) 105 °C ± 5 °C can be used (9, 11, 15). Wet spent coffee grounds (60 wt% moisture) can be used in the direct transesterification (in situ transesterification) (16). Water may occur as unbound and excess moisture or trapped in the microstructure of the solid (bound moisture). The bound moisture content depends on the physical nature of the solid, drying temperature and time and it can be a limiting factor to the drying rate (9). Following haracteristics of SCG can be

determined: content of moisture, total carbon, total nitrogen, protein, ash, cellulose, and insoluble (or Klason) and soluble lignin content (17).

SCG contains high oil content about 11 – 20 wt% on a dry weight basis and the dominant fatty acids of extracted oil from spent coffee ground were linoleic (C18:2), palmitic (C16:0) and oleic (C18:1) whose carbon length suits the production of diesel fuel. [7] The obtained oil yield might depend on different brewing methods of the fresh ground coffee beans, i.e., boiling, drip-filtering or percolating, which all lead to different concentrations of substances in the SCG. In addition, different types of fresh coffee, e.g. Coffee Arabica and Coffee Robusta, have various lipids content and hence, the SCG will also have variable lipid fractions. On the other hand, oil extraction yield from SCG may correlate closely with polarity of the extraction solvent which makes polar solvents extract greater amounts of free fatty acids (FFA) and, in return, greater crude oil yields are achieved. Here, when polar solvents are used, a black gummy material is observed in the extraction flask beside the extracted oil. In case of ethanol, the percentage of this material is 1.02% of the SCG sample. The material could be proteins, carbohydrates, and other compounds produced due to complex formation between fatty acids and carbohydrate breakdown components. Such complexes are likely to cause difficulty in oil extraction (9). The oil yield also depends on moisture content in SCG, particle size, amount of solvent, extraction technology, extraction time, etc.

Several processes can be used for the extraction of oil from spent coffee grounds, such as Soxhlet extraction, Supercritical fluid extraction (SFE), Ultrasound extraction and Microwave extraction. Oil is usually extracted by using organic solvents, but environmental safety rules and increased public health risk make the industry consider alternatives to the organic solvents for use in oil extraction (18). Soxhlet oil extractions are very time consuming, require a large amount of solvent and energy, and have low productivity (19). SFE is a new process of separating one component (the extractant) from another (the matrix) using supercritical fluids as the extracting solvent that eliminates traditional extraction method problems. The extraction is usually from solid particles, but can also be from liquids. SFE can be used as a sample preparation step for analytical purposes, or in a larger scale to either remove unwanted material from a product (e.g., decaffeination) or collect a desired product (e.g., essential oils). Carbon dioxide (CO_2) is the most widely used supercritical fluid, sometimes modified by the cosolvents such as ethanol or methanol. Extraction conditions for supercritical CO_2 are above the critical temperature of 31.85 °C and critical pressure of 78 bar. The addition of modifiers may slightly alter this. CO_2 is a clean, safe, inexpensive, nonflammable, nontoxic, environment-friendly and non-polluting solvent (18, 19, 20). Ultrasound-assisted solvent extraction is an efficient process and was applied in extraction of oil from different seeds such as flaxseed and soybeans. Integrating high intensity ultrasound with two-phase oil extraction processes can enhance the oil extraction processes from oil-containing materials with high moisture contents (20).

If extraction processes use a solvent, after the extraction had been accomplished, the solvent is distilled off in a rotary evaporator under moderate vacuum. The solvent is then recovered in order to be used in the next batch of extraction (9). The oil quality is then assessed by determining the iodine number, the saponification, calorific values, acid value (AV), and content of water, kinematic viscosity, viscosity index, density and elemental composition (9, 17). Moreover, the heating value of the SCG after extraction is measured. The acid and saponification values are the most important parameters considering fatty acid content in the extracted oil. The AV reflects the total acidity or, in other words, the amount of fatty acids that are free, i.e., not attached to a glycerol backbone. The free fatty acid content is defined as the percentage weight of free acid groups in the oil. FFA have a great influence on the oil quality. High FFA content in the oil increases its susceptibility to oxidation. The

saponification value (SV) is defined as the amount of alkali required to saponify a defined weight of sample. It specifies the content of total fatty acids in oil (free fatty acid and bound fatty acid). The mean molecular weight of the fatty acids in a lipid system as well as the number of ester bonds per gram sample can be derived from the SV (9).

Many studies have been carried out on oil extraction from spent coffee grounds. Either polar and non-polar single solvents or binary solvent mixture have been used, different extraction methods under various conditions (Soxhlet extraction, Supercritical fluid extraction, Ultrasound extraction). Yields of oil depends on used extractions conditions.

Caetano et al. (15) studied the effect of different solvents on the coffee oil yield by using Soxhlet extractor with the condition: 2.5 to 9.5 h of extraction time, the 10 g of dried SCG (at 105 ºC) and 200 mL of solvent were fixed. Therefore, the optimal condition of coffee oil extraction should be studied to reduce the extraction costs, such as type of solvent, ratio of SCG to solvent, and extraction time. The oil extraction rate and the solvent recoverability were determined for hexane, isopropanol, mixtures of hexane/isopropanol in various volume ratios (50:50, 60:40, 70:30, and 80:20), n-heptane, n-octane and ethanol. Results reveal yields of extract in a wide range: from ~ 6.3 – 28.3 wt.%. The extraction was processed at each solvent boiling temperature during 2.5 to 9.5 h being the shortest and the longest extraction time corresponding to hexane/isopropanol mixture (80:20, vol/vol) with oil extraction ~ 19,5 wt% and n-octane (26 wt%), respectively. Isopropanol showed a very good compromise between the capacity of oil extraction (~ 21 wt.%), but with higher variability, and the solvent recovery, followed by the mixture of hexane and isopropanol at a 50:50 (vol/vol) ratio that allowed for high oil extraction (~ 21.5 wt%) but with a lower solvent recovery.

Authors of (9) studied effect of type of solvents with different extraction periods for yield of oil extraction. The dried SCG (60 g) and 250 mL solvents (isopropanol, ethanol, and acetone as polar solvents and toluene, chloroform, hexane and n-pentane as nonpolar solvent) were used in this work for Soxhlet extraction. The yield of SCG oil ranges from 8.60% to 15.28% on a dry weight basis depends on type of solvent and time extraction. The highest yield is 15.28% (in a 30 min extraction time) when hexane is used as solvent. The amount of oil extracted by the polar solvents ranges from 11.43% (in 70 min extraction time with isopropanol extraction solvent) to 12.92% (in 30 min extraction time with acetone extraction solvent).

Extraction of oil from spent coffee grounds was carried out using n-hexane under reflux conditions in (13). 300 mL of solvent was used for 100 g of dried spent coffee grounds (at 50 ºC) for extraction of oil. The maximum yield of oil (15 wt.%) was observed at 45 min.

The same extraction technology as in (13) also was used by authors in (14). They used low-boiling organic solvents such as n-hexane, ether, and dichloromethane to extract the oil from the coffee particles. The saturation point was observed at 45 min for hexane extraction. The pH of the oil extracted from different solvents was observed to be different due to the variation in the FFA amounts. More polar solvents extracted greater amounts of FFA and, in turn, more crude oil yields (13.4% hexane, 14.6% diethyl ether, and 15.2% dichloromethane), which caused a decrease in pH.

In this (12) study, the optimization of two parameters: (1) solid to solvent ratio and (2) extraction time of the oil extraction process from the dried SCG after brewing coffee were investigated by using the response surface methodology (RSM). The results showed that the 14.75 wt% of calculated yield of coffee oil from the predicted model was obtained, when the optimal condition: the 1:22.5 g/g of mass ratio of dried SCG to hexane and 30.4 min of extraction time under the 30 °C of room temperature was used. The model was verified by the experiment, the 14.68 wt% experimental yield of coffee oil was achieved after passing the extraction process under the optimal condition.

Another studies (18, 19, 20, 21, 22) report results for ultrasound oil extraction (or ultrasound-assisted extraction) from SCG. Results of supercritical fluid extraction of coffee has been reported in the literature, either in green (23, 24), roasted (23) or spent (18, 25, 26, 27) forms. Another type is microwave extraction or microwave-assisted extraction (18, 22).

BIODIESEL PRODUCTION FROM SPENT COFFEE GROUNDS

Transesterification of oils with alcohol is the best method for biodiesel production. Different types of alcohols such as, methanol, ethanol, propanol and butanol have been used. Transesterification reaction can be either carried out via non-catalytic or catalytic processes. Non-catalytic transesterification reaction is slow and normally needs high pressures and temperatures to be completed. The utilization of different types of catalysts improves the rate and yield of biodiesel. There are several kinds of catalysts that can be used to produce biodiesel. It can be categorized into three main categories, i.e. homogeneous catalyst, heterogeneous catalyst and biocatalyst (enzyme) (28). Generally the transesterification reaction involves some critical parameters (e.g., reaction temperature and time, free fatty acid content in the oil, water content in the oil, type of catalyst, amount of catalyst, molar ratio of alcohol to oil, type or chemical stream of alcohol, use of co-solvent and mixing intensity) which significantly influence the final conversion and yield (8).

Biodiesel production involves a transesterification, a reversible reaction with three successive steps, where triglycerides are converted to diglycerides, diglycerides to monoglycerides, and monoglycerides to esters and glycerol (Fig. 1). From this reaction, it results in three moles of fatty acid monoalkylester (biodiesel) and a mole of glycerol as a by-product (8, 17, 29).

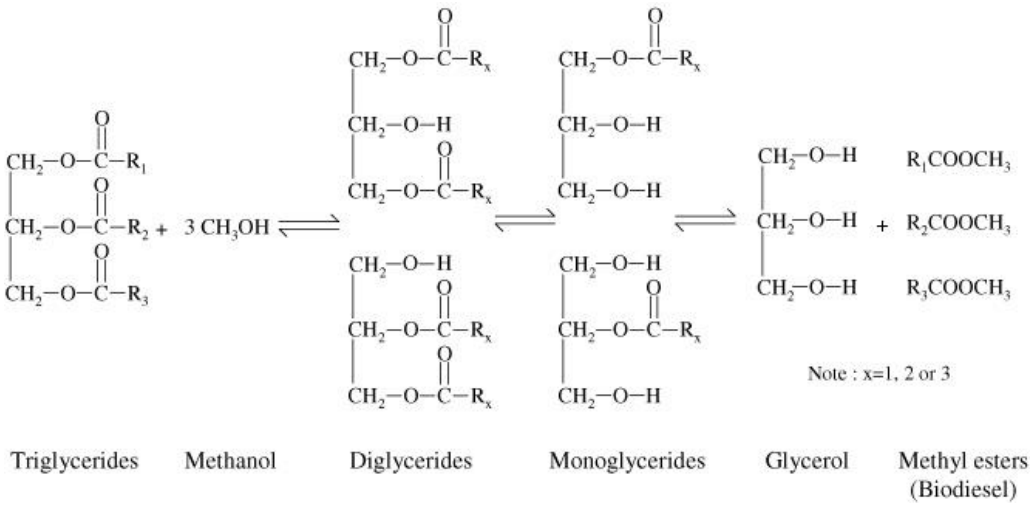

Fig. 1 *Reactions in the transesterification of a triglyceride* (29)

In the manufacturing of biodiesel from different type of oil, the level of reaction completion strongly depends on the quality of the feedstock oil (9). The methods of biodiesel production from SCG can be devided:

- *One step alkali-catalyzed transesterification*
- *Two step transesterification*
  - *Acid-catalyzed pretreatment*
  - *Alkali-catalyzed transesterification*
- *In situ transesterification*

Schemes of method biodiesel production from spent coffee grounds are shown on Fig. 2.

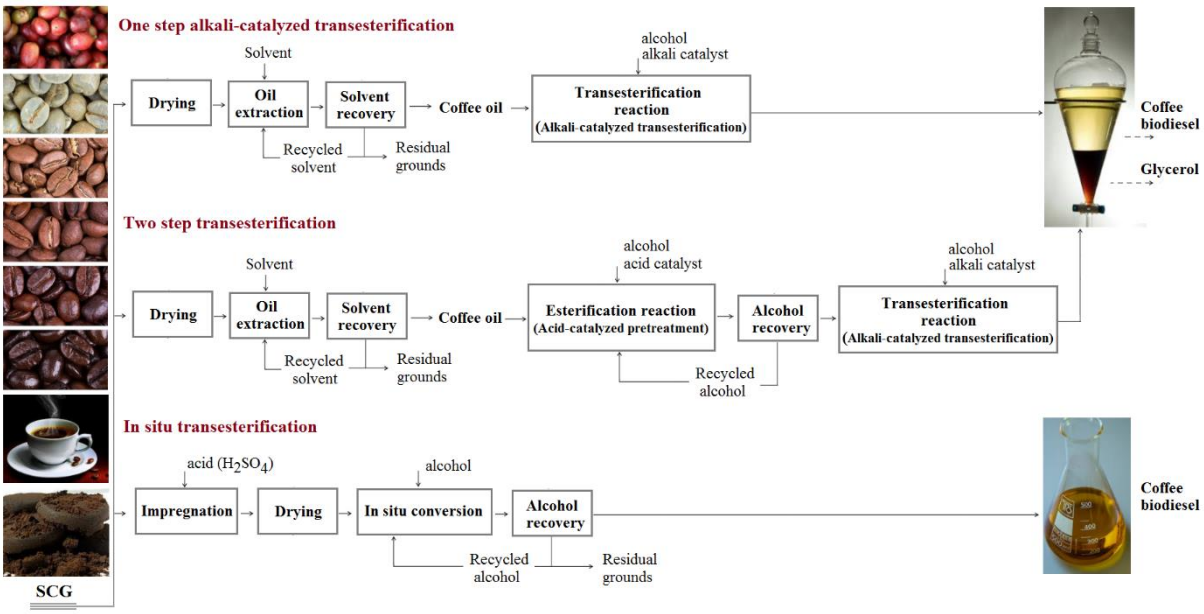

Fig. 2 *Schemes of method biodiesel production from spent coffee grounds*

One step alkali-catalyzed transesterification

The one step alkali-catalyzed transesterification is reaction of alcohol (usually methanol) and triglyceride under an alkali (base) catalyst. The alkali-catalyzed process has a one limitation – is very sensitive to the purity of reactants (to both water and free fatty acids) (30). The presence of water may cause ester saponification under alkaline conditions. Also, free fatty acids can react with an alkali catalyst to produce soaps and water. Saponification not only consumes the alkali catalyst, but also the resulting soaps can cause the formation of emulsions. Emulsion formation creates difficulties in downstream recovery and purification of the biodiesel (16, 30, 31).

The limits of FFA content regarding the possibility to conduct a one step alkali-catalyzed transesterification range from 0.5% FFA (corresponds to an acid value of 1 mg KOH/g) to 1% FFA (corresponds to an acid value of 2 mg KOH/g) (9, 10, 31).

Two step transesterification

The problem with processing low cost oils and fats (waste cooking oil and fats, SCG oil) is that they often contain large amounts of free fatty acids that cannot be converted to biodiesel using an alkali catalyst (31, 32). Therefore, FFA should be initially converted to ester in a first step, by reaction of the oil with alcohol (usually methanol) using an acid catalyst (acid-catalyzed pretreatment). The acid catalysts should be used when the FFA concentration of the feedstock is higher than 2.0 mg KOH/g oil (FFA content < 1% by weight) (9, 10). In the second step (alkali-catalyzed transesterification) the remaining triglycerides are transesterified with alcohol by using alkali catalyst to produce methyl esters and glycerol. With this approach, it is possible to achieve the maximum possible conversion rate (9).

The acid catalysts are too slow to be practical for converting triglycerides to biodiesel. However, acid catalysts appear to be quite effective at converting FFA to esters and this reaction is fast enough to be. Thus, an acid–catalyzed pretreatment step to convert the FFA to

esters followed by an alkali–catalyzed step to convert the triglycerides should provide an effective and efficient method to convert high FFA feedstocks to biodiesel (31).

In situ transesterification

Biodiesel can be produce also by direct transesterification (in situ transesterification). Since the wet in situ transesterification process of SCG integrates oil extraction, esterification, and transesterification into one step, it allows to directly produce biodiesel from SCG and save the production cost. Also it is an eco-friendly way to recycle the municipal waste and utilize it as renewable energy. In summary, the in situ transesterification method is able to produce biodiesel from SCG that is comparable in quality with the conventional solvent extraction method. This process is much simpler (10, 16).

For in situ transesterification, most studies used an alkali 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) (10).

In biodiesel production is also necessary used purification steps after the transesterification in order to ensure the product complies with stringent international standards and specifications. (21). For biodiesel characterisation is possible evaluated the visual appearance of biodiesel (color and physical state), acid and iodine values, water content, reaction yield, and methyl ester content, density, kinematic viscosity, cetane number, flash point, cloud and pour points, etc.

CONCLUSION

Coffee processing generates significant amounts of residues from which valuable compounds can be extracted. These residues represent about 80% of the total weight of grounds. A particular by-product of coffee processing is spent grounds, a by-product of the coffee brewing operation that represents 10% of the total weight of the fresh grain. SCG can be used for more applications (bioethanol production, as composting material, as a sorbent, as a source of natural phenolic antioxidants, atc.). Also are a valuable product for biodiesel production, because contains high oil content (about 11 – 20 wt% on a dry weight basis). Production of biodiesel from SCG involves collection and transportation of coffee residue, drying, oil extraction and biodiesel production. Yields and quality of biodiesel depends on of SCG (quality, moisture content, particle size), oil extraction method (Soxhlet extraction, Supercritical fluid extraction, Ultrasound extraction, Microwave extraction), used extractions conditions (type of solvent, amount of solvent, extraction technology, extraction time) and on method of biodiesel production. The results of studies showed that using different methods of extraction resulted in significant differences in the extraction yields. Extract oil is carried out usually using organic solvents, but environmental safety rules and increased public health risk are making the industry to consider alternatives to the organic solvents for use in oil extraction. Some extraction method is cleaner, safer, more inexpensive, less nontoxic and more environment-friendly. It is very important choose the right method for oil extraction and biodiesel production with maximal yields and minimum negative environmenal impact.

Each year, a several thousand tons of spent coffee grounds are disposed of. Yet, these waste products have the potencial to fill an urgent global need: a need for alternative fuel sources. Biodiesel is also a viable and promising solution to the environmental pollution which results from conventional fuels combustion and to the increasing world energy demand. The use of SCG as one of the various feedstocks for biodiesel production can be considered as a solution to these problems.

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