

Investigation of Waste Coffee Ground as a Potential Raw Material for Biodiesel Production

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Abstract- In this study, the utilization of waste coffee ground for biodiesel production was investigated. Waste coffee ground sample was collected from TOMOCA PLC, Addis Ababa, Ethiopia. The oven dried sample was then soxhlet extracted using n-hexane to yield 19.73 % w/w oil. The biodiesel was obtained by a two-step process, i.e. acid catalyzed esterification followed by base catalyzed transesterification using catalysts sulphuric acid and sodium hydroxide respectively. The conversion of waste coffee ground oil in to biodiesel, was about 73.4% w/w. Various parameters that are essential for biodiesel quality were evaluated using the American Standard for Testing Material (ASTM D 6751- 09) and all comply with the specification except acid value. The fatty acid composition of the biodiesel was analyzed by Gas chromatography and the major fatty acids were found to be linoleic acid (39.8%), palmitic acid (37.6%), oleic (12.7%), and stearic acid (7.6%). In addition, preliminary investigation on the solid waste remaining after oil extraction was conducted for possible use as a feedstock for the production of bioethanol. Hydrolysis of the spent of waste coffee ground was carried out using dilute sulphuric acid followed by fermentation using *Saccharomyces cerevisiae*, and resulted in bioethanol yield of 8.3 %v/v. Furthermore, the solid waste remaining after bioethanol production was evaluated for compost (21.9:1 C/N) and solid fuel (20.8 MJ/Kg) applications. The results of this research work give insights on the production of biofuel from waste ground coffee. In addition, the preliminary analysis on the solid waste after the extraction of the oil suggests that it can be used as fuel hence alleviating major disposal problems.

Keywords- Waste coffee ground; Biodiesel; *Saccharomyces cerevisiae*; Bioethanol; solid fuel; compost

1. Introduction

Biodiesel, a mixture of long-chain fatty acids alkyl esters, is a novel energy source that has grown in importance over recent years. In recent years, biodiesel production from vegetable oils and animal fats has gained attention because of its eco-friendly nature. However, biodiesel is more expensive than conventional fuels, which hinders its applications. The major production cost of biodiesel is from its feedstock [1]. To conquer this problem, industries use waste materials to produce low-cost biodiesel [2, 3].

Coffee is one of the largest agricultural products that are mainly used for beverages throughout the world and providing approximately 30.6% of Ethiopia's foreign exchange earnings in 2010-2011 [1, 4]. Ethiopia is currently producing an estimated 9.804 million 60-kg bags that would rank as the third largest coffee producer in the world after Brazil and Vietnam and half of the coffee is consumed by Ethiopians [5, 6].

Waste coffee grounds (WCGs) are the main coffee industry residues with a generation of 6 million tons worldwide [7] and 235,296 tons in Ethiopia. The biodiesel from waste coffee grounds possesses better stability than

biodiesel from other sources due to its high antioxidant content [8, 9]. WCG is also considered an inexpensive and easily available adsorbent for the removal of cationic dyes in wastewater treatments [10]. In this work, the potential of WCG for biodiesel production, its solid by-product after oil extraction for bioethanol production, as well as the second by-product after bioethanol production for solid fuel and compost production was demonstrated.

2. Materials and Methods

2.1. Materials and Chemicals

The WCG (waste coffee Arabica) sample was supplied from TOMOCA PLC coffee shop (Addis Ababa, Ethiopia). All solvents and analytical grade chemicals were obtained from chemistry department (Addis Ababa University, Ethiopia).

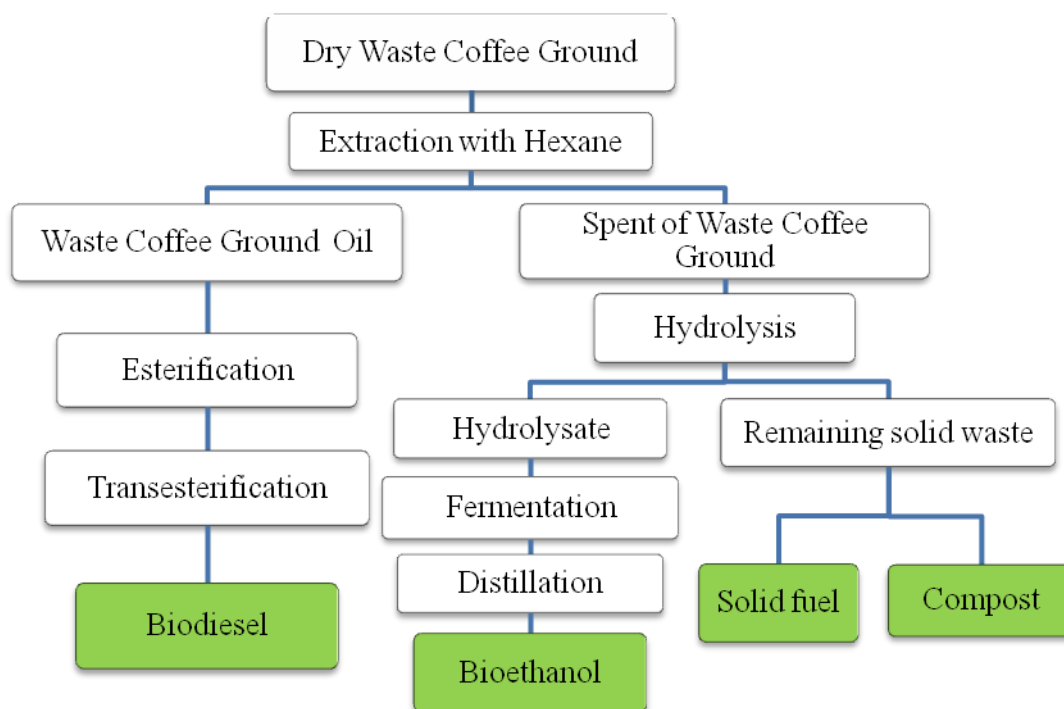


Fig. 1. Schematic representation of the production of waste coffee ground biodiesel that yield high value added by-products: bioethanol, fuel pellets and compost.

2.2. Experimental

2.2.1. Moisture content determination

The moisture content of the waste coffee ground was determined using an oven. The sample was weighed to the nearest 10 g in Petri dishes and then dried at 105 °C. It was then cooled in desiccator over silica gel (0% relative humidity) and reweighed until the constant weight is maintained. The moisture content was determined as in Eq. (1);

$$M = \left(\frac{W_1 - W_2}{W_1} \right) \times 100 \% \quad (1)$$

Where M, W₁, W₂ are moisture content, initial mass and final mass

2.2.2. Waste coffee ground (WCG) oil extraction

500 g of the dried WCG sample was placed in a 4 L round bottom flask of the soxhlet apparatus and percolated

for 6 h using n-hexane as a solvent. The solvent was then removed from the oil using a rotavapor (Buchi RE111 Rotavapor, BUCHI, Flawil, Switzerland). The solvent was reused for subsequent batch of extracting. The oil yield was determined as in Eq. (2);

$$\text{WCG oil content} = \frac{W_o}{W_s} \times 100 \% \quad (2)$$

Where: W_o = weight (g) of oil extracted and
 W_s = weight (g) of sample (dry base)

2.2.3. Physicochemical characterization of WCGs oil

The WCG oil was characterized for its physical and chemical properties like, density (ASTM D1298), kinematic viscosity (ASTM D445), flash point (ASTM D93), cloud point (ASTM D97), peroxid value (ASTM D3703), water and sediment (ASTM D2709), iodine value (EN14111), acid value (ASTM D974) and higher heating value (HHV) (ASTM D240). All experiments were run in triplicate and mean values were reported.

2.2.4. Two-Step Biodiesel Production Process

2.2.4.1. Acid-catalyzed esterification

Esterification was carried out to pre-treat the FFA for Transesterification. The WCG oil was heated to 60 °C to homogenize the oil. The reaction was conducted in a 1 L two necked round-bottomed flask attached with a reflux condenser and thermometer placed in an oil bath. The oil is mixed with methanol (a molar ratio of alcohol to free fatty acids of 20:1) and significant quantities of H₂SO₄ (10 wt% of total fatty acids content). The reactor was stirred at about 600 rpm, at temperature of 60 °C for 2 h [11]. Then the reaction product mixture was poured into a separating funnel and allowed to settle for 24 h. The top layer which is comprised unreacted methanol and water was removed. The acid pre-treatment loss was calculated as in;

$$APL = \frac{\text{weight of pretreated oil}}{\text{Weight of crude oil}} \times 100 \% \quad (3)$$

Where; APL= Acid Pre-treatment Loss

2.2.4.2. Base-catalyzed transesterification

The transesterification reaction of WCG oil was carried out in a 1 L round-bottomed glass flask, with anhydrous methanol in molar ratio methanol to oil 6:1 at 60 °C for 2 h, using sodium hydroxide (NaOH) as catalyst in amount 1% (w/w). The reaction product was then poured into a separating funnel for glycerol and methyl ester separation, allowed to settle for 24 h [12]. After the two phase separations, the excess alcohol in each phase was removed by vacuum distillation at 90 °C placed on oil bath.

2.2.4.3. Purification

The methyl ester was purified by washing gently with warm (55 °C) deionized water to remove residual catalyst, glycerol, methanol and soap. To neutralize the remaining soaps, a small amount of sulphuric acid (H₂SO₄) was used in the second washing. The washed methyl ester was then dried over anhydrous sodium sulphate (Na₂SO₄). The dried methyl ester was then bottled and kept for characterization studies.

2.2.5. Characterization of WCG Biodiesel

The biodiesel esters were characterized for their physical and chemical properties using accepted standards. Density (15 °C) (ASTM D1298), Kinematic viscosity(40 °C) (ASTM D445), Gross calorific value (ASTM D240), Cloud point (ASTM D97), Iodine number (EN14111), water and sediment (ASTM D2709), Ash content (ASTM D874), acid value (ASTM D974), Carbon residue (ASTM D189), flash point (ASTM D93), Distillation, 90% recovery (ASTM D86) and Copper corrosion (ASTM D130) were used.

2.2.6. Fatty acid composition of WCG methyl ester

Fatty acid composition of the synthesized alkyl ester was determined by gas chromatography. Gas chromatograph (DANI GC 1000) equipped with flame ionization detector (FID) was employed during fatty acid determination. The GC was calibrated by injecting standards at varying concentrations. 1µL of the sample was injected in to GC, equipped with a capillary column of EC TM-5 (25 m x 0.53 mm x 1µm). The oven starting temperature was 50 °C and kept for 2 minutes. Then 15 minutes hold time with heating rate of 4 °C /minute to 250 °C. Nitrogen at (1 mL/minute) was used as carrier gas at a flow rate of 1.25 bars was adjusted.

2.2.7. Production of Bioethanol from the solid waste remaining after oil extraction of WCG (Spent of WCG)

Spent of WCG was hydrolyzed by refluxing, a solid to liquid ratio of 1:10, using dilute sulphuric acid (each of 1, 2 and 3 molar) and distilled water for 15 minutes at a temperature of 90 °C. Then the liquid fraction of the hydrolysate sample was cooled, filtered with suction filtration and adjusted to PH 5 by adding concentrated NaOH and H₂SO₄. Fermentation was then carried out using yeast (*S.cereviciae*) for 24 h at temperature of 30 °C [13]. Finally ethanol was separated from the fermented sample by fractional distillation and the concentration was analyzed with FTIR spectroscopy (spectrum 65 PerkinElmer, UK).

2.2.8. Determination of the quality of the solid ground remaining after Bioethanol production for compost and solid fuel

The major quality properties of the solid waste remaining after Bioethanol production were analyzed using standard test methods like calorific value (MJkg⁻¹), total nitrogen (w%), total carbon (w%) and proximate analysis (w%).

3. Results and Discussion

3.1. Oil Content of Waste Coffee Ground (WCG)

Waste coffee ground sample (WCG) collected from TO.MO.CA PLC(Addis Ababa, Ethiopia) was dried in an oven (moisture content 57.6% w/w) and the oil was extracted with hexane using a soxhlet apparatus. The oil yield was found to be 19.73% w/w on dry weight basis, which is relatively higher than those reported for WCR (10-15% w/w) in the literature [1, 13]. Higher oil yield (28.3% w/w) has been obtained when WCR was extracted with 1:1 mixture of Isopropanol and hexane [14]. It is noteworthy that the oil yield of WCG in this study is higher than other oil seeds such as, olive (17%), soybeans (18%), cottonseeds (14%), and corn (3.4%) [15]. The variation in the oil yield could be

attributed to differences in variety of coffee, solvent type and cultivation climate.

3.2. Physicochemical Characteristics of the Extracted WCG oil

WCG oil that was utilized as a feedstock in the production of biodiesel was characterized to determine its physicochemical properties. The results obtained on the properties of the oil were compared with that of American Standard for Testing Materials (ASTM D 6751). The standard values and the results obtained are summarized in “Table 1”.

Table 1. Characterization of the oil extracted from waste coffee grounds

Property	Units	Test Methods	Limits	Results
Density (15 °C)	g /cm ³	D1298	0.86–0.90	0.9228
Kinematic Viscosity(40 °C)	mm ² /s	D 445	1.9– 6.0	35.50
Gross calorific value	MJ/kg	D 240	Report	37.88
Cloud point	°C	D 97	Report	10
Iodine Value	gI2/100g	EN14111	120 max	75.94
Water and sediment	% volume	D2709	0.050 max	0.025
Acid value	Mg	D974	0.8 max	14.65

The oil was found to be very acidic and the saponification value was also high as in “Table 1” to be directly converted into biodiesel without pre-treatment. In order to avoid a higher degree of oxidation and occurrence of hydrolysis reactions [16] in the process of transesterification, the oil was first esterified using Sulfuric acid as a catalyst. Flash point of the oil is very high, making it better suited for biodiesel production with respect to safety during storage and transportation.

3.3. Biodiesel Yield of WCG oil

The %FFA of the WCG oil decreased from 7.33% to 0.9% after three consecutive esterification steps. After esterification, the oil pretreatment loss was found to be 8.67

%w/w based on the initial sample of WCG oil. The pretreated oil was transesterified using the optimal condition of 6:1 methanol to oil molar ratio, 1 wt% as catalyst concentration (NaOH), 2 h reaction time and reaction temperature of 60 °C to give biodiesel conversion rate relative to WCG oil 73.4 %w/w and acid pretreated WCG oil 80.4 %w/w.

3.4. Waste Coffee Ground Biodiesel Fuel Properties

The biodiesel produced from WCG oil was analyzed for its fuel properties as in “Table 2”. It was found out that the fuel properties of the waste coffee ground biodiesel are within the range set by the American Standard ASTM D6751 except for the acid value.

Table 2. Fuel Properties of the oil extracted from waste coffee ground

Property	Units	Test Methods	Limits		WCR oil methyl ester
			ASTM D6751	EN 14214	
Density (15 ⁰ C)	g /cm ³	D1298	-----	0.86 – 0.9	0.8915
Kinematic viscosity(40 ⁰ C)	mm ² /s	D445	1.9 -6.0	3.5-5	5.26
Gross calorific value	MJ/kg	D240	-----	-----	38.4
Cloud point	°C	D97	Report	-----	14
iodine value	gI2/100g	EN14111	-----	120 max	73.41
water and sediment	% volume	D2709	0.05 max	500 mg/kg	<0.01
Ash content	W%	D874	0.02 max	0.02 max	0.0123
acid value	mg KOH/g	D664	0.5 max	0.50 max	0.78
Carbon residue	% mass	D189	0.05 max	0.3max	0.033
flash point	°C	D93	93.0 min	120 min	222
Copper corrosion	Max.	D130	No. 3 max	No. 1 max	1a
Distillation, 90% recovery	°C	D86	360°Cmax	-----	349

3.5. Fatty Acid Composition of Waste Coffee Ground Oil Methyl Ester

Fatty acid components of WCG biodiesel was determined by gas chromatography analysis (GC). The

analysis showed the presence of C16-C18 and two unidentified methyl ester of fatty acids (Fig. 2). WCG biodiesel consists of both saturated and unsaturated methyl esters, with linoleic acid (39.8%), palmitic acid (37.6%), oleic (12.7%) and stearic acid (7.6%) comprising 97.7% of the total composition.

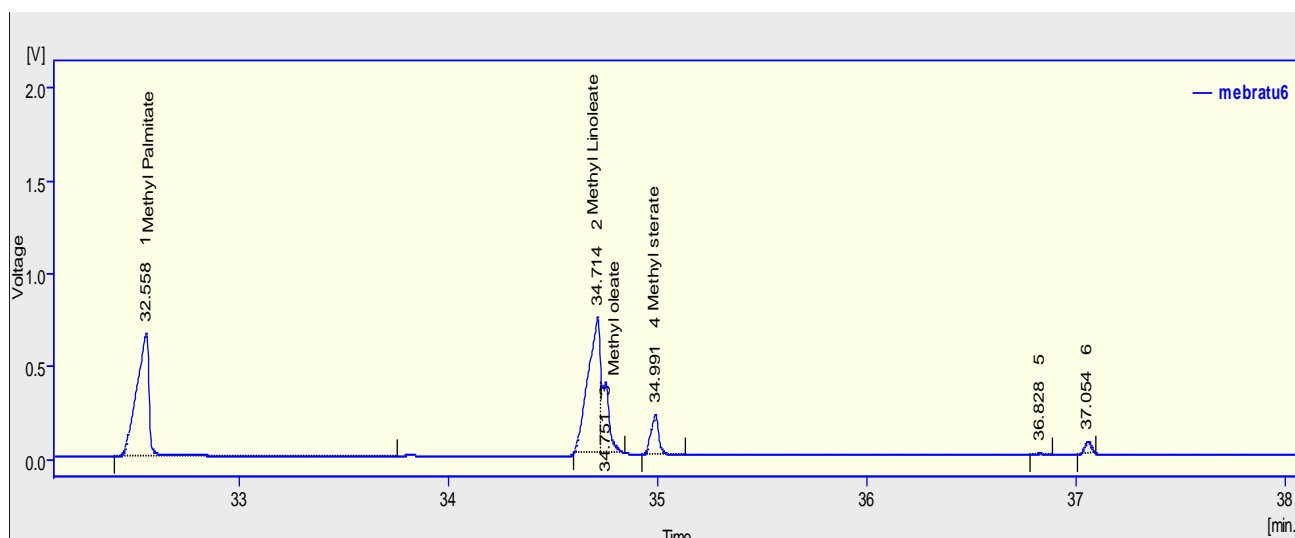


Fig. 2. Gas chromatogram profile of fatty acid methyl ester of WCG

3.6. Bioethanol Yield From Solid Waste Remaining After Oil Extraction of Waste Coffee Ground (Spent of WCG)

The Spent of WCG after oil extraction was analyzed for its ethanol content. Hydrolysis of the spent with dilute H₂SO₄ and subsequent fermentation with yeast yielded ethanol.

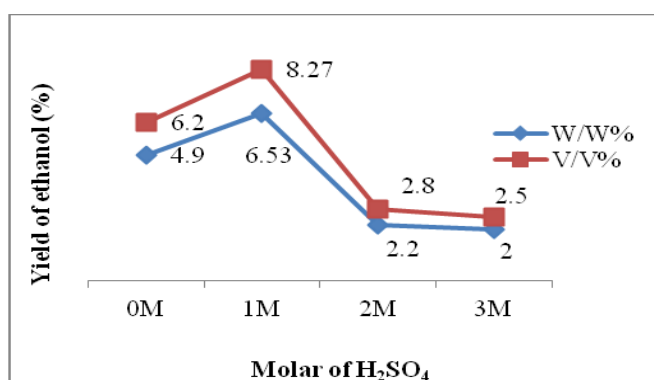


Fig. 3. Bioethanol yield (%) of the solid waste after oil extraction (spent of WCG)

Dilute sulfuric acid (each of 1, 2 and 3 molar of H₂SO₄) and distilled water were used for hydrolysis, which resulted in bioethanol yield (%v/v) of 8.3, 2.8, 2.5 and 6.2 respectively. The maximum bioethanol yield (8.3 %v/v) was achieved at 1Molar of sulfuric acid as in “Fig. 3”.

The reason that lower bioethanol yield obtained with distilled water hydrolysis in comparison to acid hydrolysis could be due the carbohydrate fraction (hemicellulose and cellulose) of spent of WCG can be depolymerised into sugars which act as a primary carbon source for the microbial biocatalysts for the production of ethanol by acid hydrolysis [17, 18]. Further increase in molarity of sulfuric acid beyond 1 molar of sulfuric acid resulted low bioethanol yield. This decrease in bioethanol yield may account for the further sugar degradation that occurred under the severe acidity. In general, this result indicates that extreme acidity had an adverse effect on sugar conversion of spent of WCG [19]. The obtained result of this study (65.3 g/kg) was lower than

the result (110g/kg) as in [20] on the original WCG (before oil extraction) without optimization. Spent of WCG has high ethanol yield compared to the waste materials as in [21] on beet waste (2.15%) and banana peels (1.90%), and [22] on Chrysanthemum waste degradation (0.45 wt %) but lower yield as compared with Sugar Molasses 53%v/v using *Saccharomyces Cerevisiae* [23] and 59.1g/l using immobilized enzymes [24]. Therefore, this result shows that the spent of WCG has still the potential to use as source of bioethanol.

3.7. Solid Fuel and Compost from the WCG After Bioethanol Production

Analysis of the solid waste remaining after Bioethanol production of spent of WCG for solid fuel and compost was carried out. “Table 3” below showed the result of the quality parameters analyzed.

Table 3. Characteristics of wcg after bioethanol production for solid fuel and compost

property	Units	Solid waste remaining after Bioethanol
Gross calorific value	MJkg ⁻¹	20.8
Proximate Analysis	Fixed Carbon	w % 17.59
	Moisture	w % 4.5
	Ash	w % 2.04
	Volatile matter	w % 75.87
Total nitrogen	w %	2.30
Total carbon	w %	50.43

All the % values are on a dry weight basis, except the moisture content.

Measurement of the calorific value of WCG after bioethanol production provided the result of 20.8 MJ/kg (expressed per dry weight of solid) as in “Table 3”. In particular, It has high calorific value compared to the conventional biomass as in [25], like bagasse (7.7-8 MJ/Kg),

rice husks (14 MJ/Kg), coffee husk (16 MJ/Kg) and wood (8.4- 17). Ideal waste coffee grounds for the soil need a carbon to nitrogen ratio (wt) of 20:1 [26]. The carbon to nitrogen ratio after the bioethanol production process was found to be 21.92:1. This C/N ratio of solid waste after bioethanol production of spent of WCG met the ideal carbon to nitrogen ratio as in [26].

4. Conclusion

The aim of this study was to evaluate waste coffee ground remaining after brewing coffee as a potential alternative feedstock for biodiesel production. Around 19.73 %w/w of the WCG oil was extracted and characterized. The obtained WCG oil was chemically converted to fatty acid methyl ester with 73.4%w/w biodiesel yield via two-step reaction process. The WCG oil biodiesel produced was characterized and has fuel properties that met the latest ASTM D 6751 standard except for acid value. GC analyses indicated that the waste coffee residue biodiesel comprised of saturated (45.2%), unsaturated (52.5%) and two unidentified (2.3%) esters.

The result of the study showed that the highest bioethanol yield of 8.3 %v/v was obtained with 1 molar H₂SO₄ from spent of WCG, which is significant. Finally, the quality property of solid waste after bioethanol production were characterized and found to be it still has the potential to use for solid fuel and compost.

Therefore, WCG can be potentially used as a raw material for biodiesel and bioethanol production on a commercial scale. This can supplement the Ethiopian energy sector approximately by 10.32 and 4.38 million gallons of biodiesel and bioethanol respectively by sequential utilization of the same WCG per year. The results of this work may suggest a new insight to production of biofuel from waste materials.

Acknowledgements

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