

## Characterization of crude glycerol from biodiesel produced from cashew, melon and rubber oils

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### ABSTRACT

The purpose of this paper is to present a report on the characterization of crude glycerol derived from biodiesel processed from cashew, melon and rubber seed oils. Crude glycerol is a co-product from biodiesel production. To make the crude glycerol economically useful, it is imperative to characterize it. Investigative tests were carried out on the vegetable oils and on the crude glycerol that were separated from the biodiesel fuels with a separating funnel. The fatty acid profile was obtained using a gas chromatography analyzer and the physicochemical properties of the glycerol sludge were measured. Some of these physicochemical properties are flash point, ash content and material organic non-glycerin (MONG). Further works can still nutritional, industrial and pharmaceutical qualities and uses. © 2014 Trade Science Inc. - INDIA

### KEYWORDS

Biodiesel;  
Characterization;  
Crude glycerol;  
Material organic non-glycerin (MONG).

### INTRODUCTION

The publication reports the characterization of crude glycerol obtained when biodiesel was produced from cashew, melon and rubber seed oil. Biodiesel is a renewable fuel from natural oils, animal fats and waste cooking oils and it can be used as an alternative fuel for diesel engines. Biodiesel is the product obtained when a vegetable oil or animal fat is chemically reacted with an alcohol to produce fatty acid alkyl esters. A catalyst such as sodium or potassium hydroxide is required. Glycerol is produced as a co-product. The approximate proportions of the reactant and products of the reaction in kilogramme mass are as follows,

100kg of oil +10kg of methanol → 100kg of biodiesel + 10kg of glycerol<sup>[1,2]</sup>.

This implies that for every 100kg of biodiesel produced, the corresponding value of crude glycerol is 10kg. The world production of glycerol has been growing geometrically the last few years as a result of increased production of biodiesel. Crude glycerol is of low economic value because of the presence of impurities. It can be processed by refinement via filtration, demethylation, acidulation, chemical addition and fractional vacuum distillation to produce a vast number of different products for the pharmaceutical, cosmetic, animal feed processing and plastics industries<sup>[3-6]</sup>.

### MATERIALS AND METHOD

Three crude glycerol samples were obtained from the transesterification of the three vegetable oils and

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methanol. 500cm<sup>3</sup> rubber seed oil was measured and transferred into the reaction flask and preheated by the hot plate to the desired reaction temperature before the reaction started. The methanol to oil ratio was 6:1 and 1% wt/wt sulphuric acid catalyst<sup>[7]</sup> was added. The reaction mixture was continuously stirred at a temperature of 65°C. The reaction mixture was poured into separating funnel and was allowed to settle for one hour. After settlement, the top layer which is made up of the methanol water mixture, sulphuric acid and other impurities was removed. The lower layer which is the oil phase is ready for alkali-catalyzed transesterification.

The transesterification reaction for the cashew, melon and rubber seed oil was conducted using a 6:1 molar ratio of methanol to oil and sodium hydroxide catalyst concentration of 1% by weight of the oil at 65°C under reflux condenser with a mechanical stirrer. After two hours reaction time, the mixture was transferred to a 500cm<sup>3</sup> separating funnel. Two distinct layers were formed after allowing the reaction mixture to stay overnight for 12 hours. The top layer is methyl esters and the bottom layer is the crude glycerol which is used for the study. The methyl ester layer was heated at 85°C for 1 hour to remove the methanol.

The ash content was determined by weighing a given amount of it into a crucible and burnt in a furnace. The crucible and its content were allowed to cool down, after which the ash content was obtained by dividing the final weight by the initial weight and then multiplied by 100 to express it in percentage basis. The moisture content in the crude glycerol was measured by using Karl-fisher method (ASTM D6304). The glycerol content analysis was performed in accordance to the AOC3

Ea 6-94 standard. The methanol content in the crude glycerol was calculated as the difference between the initial methanol used in the experiment and the final methanol measured in the methyl ester layer.

The Material organic Non Glycerin (MONG) is a combination of all organics in the crude glycerol sample that are not glycerol or glycerin. This includes methanol, fats and oils, other organic solvents like sodium hydroxide catalysts remains, impurities such as soaps and other matters dissolved in it, metal and glycerides and several others. The Material Organic Non Glycerin was calculated using the following formula Material Organic Non Glycerin (MONG) = Total content of 100% - Glycerin content % - Ash content % - Water content %.

The flashpoint of the crude glycerol was determined using close cup method in relation to the technical limits established by ASTM D93 while the viscosities were carried out on a Koehler Model K-233 Oil bath making use of Cannon Fenske Routine Viscometers in accordance with ASTM D445 protocols and the heats of combustion were measured on a Parr 1341 Oxygen Bomb Calorimeter in agreement with ASTM D240 standard<sup>[8]</sup>.

The macro elements (Na, K, Ca, Mg, P and S) were determined by using the Teledyne Fuelpro Biodiesel Metals Analyzer. The Fatty acid profile of the oils and the crude glycerol were analyzed using Gas Chromatography method<sup>[9]</sup> in accordance with ASTM D6584. A gas chromatography model 6890 powered with HP ChemStation Rev. A09.01 [1206] software was used. The carrier gas was Nitrogen and the mechanical device has column dimensions of 30m x 0.25mm x 0.25µm with an inlet temperature of 250°C and Column Type HP INNO Wax.

TABLE 1 : Characterization of crude glycerol

S/No.	Test	Unit	Measurement Method	RESULTS		
				Cashew Glycerol	Mellow Glycerol	Rubber Glycerol
1	Ash content	%		11.12	11.77	11.52
2	Moisture or water content	%	Karl Fisher	1.98	2.11	2.05
3	Glycerol content	%	AOCS Ea6-94	36.68	39.50	43.81
4	Material organic Non Glycerin (MONG)	%		50.22	46.62	42.62
5	Total Content Analysis	%		100.00	100.00	100.00
6	Flash point	°C	ASTM D93 (Closed Cup)	30.9	32.7	32.00
7	Viscosity	mm <sup>2</sup> /s	Cannon Fenske Routine Viscometer	9.17	7.34	11.00
8	Heat of combustion	MJ/Kg	Parr 1341 Oxygen Bomb Calorimeter	20.00	20.76	20.86

## RESULTS AND DISCUSSION

Laboratory tests were carried out on the cashew, melon and rubber seed oils and on the unrefined glycerol. The measurements that were performed are the ash content, moisture content, glycerol content and material organic non glycerin (MONG). The total content analysis was calculated. Other measurements that were also carried out are flash point, viscosity, heat of combustion and macro elements analyses as shown in TABLE 1.

**TABLE 2 : Metal and non-metal concentration level for cashew seed oil (CSO), melon seed oil (MSO) and rubber seed oil (RSO)**

S/No	Material	Units	CSO	MSO	RSO
1	Sodium, Na	Ppm	4.50	2.70	8.90
2	Potassium, K	Ppm	2.80	5.30	4.20
3	Calcium, Ca	Ppm	3.40	15.90	4.70
4	Magnesium, Mg	Ppm	28.50	2.40	1.80
5	Phosphorus, P	Ppm	40.60	15.80	8.00
6	Sulphur, S	Ppm	21.00	26.20	23.50

There was a little variation in the ash content and moisture or water content for the three glycerol but marked variation in the glycerol content of the three glycerol's as well as the difference between the materials organic non glycerin. It is important to note that there was a significant difference in the macro element screening test results for the three vegetable oils. This may be due to the nature of the soil they were grown.

## CONCLUSION

The properties of the by-products of biodiesel, the crude glycerol produced from the three vegetable oils were characterized. This can enhance the production, purification and future development of crude glycerol into economically useful products.

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