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Synthesis and Characterization of Bio-Glycerol from Cameroon Palm Kernel Seed Oil

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Abstract

Bio-glycerol was synthesized from Cameroon palm kernel oil (PKO) through the transesterification procedure. Palm kernel oil extracted from palm kernel seeds using mechanical expression and solvent extraction was purified and characterized by physico-chemical methods and used in the transesterification process to give biodiesel and bio-glycerol. The biodiesel was purified and characterized as reported in previous articles. Our focus in this article is on glycerol, an important by-product of the transesterification process which has potential pharmaceutical, cosmetic and engineering applications. The bio-glycerol was purified by acidification and the purified glycerol was subjected to physical and chemical characterization. The specific gravity of glycerol was obtained as 1.2 kg/L, viscosity at 40˚C gave 1500 cSt and 500 cSt at 100˚C; pH was 7.4; the flash point was 160˚C, and the ASTM color was 2.0 before purification and zero after purification. The sulfur content was 0.016%w/v. This sulfur content is low thus posing no environment threat. The chemical composition of the synthesized bio-glycerol determined using IR spectroscopy and gas chromatography-mass spectrometry (GC-MS) confirmed the known chemical structure of glycerol. The purification and analysis of bio-glycerol is important as it can find applications in the pharmaceutical, cosmetic and food industries *inter alia.*

Keywords

Palm Kernel Seed Oil, Transesterification, Biodiesel, Bio-Glycerol, Physico-Chemical Characterization, Viscosity, GC-MS, Flash Point

1. Introduction

A good fraction of glycerol used for industrial and research applications is produced from fossil fuel sources through various synthetic routes [1]. Owing to the fact that petroleum is a nonrenewable source of energy and fossil fuels are gradually undergoing extinction [2], more research is tilted towards renewable energy sources which have greater technological, environmental and health advantages [3]. In addition, the need to replace fossil fuels is a driving force for the development of renewable fuels from biomass [4] [5] and biofuels are used as substitute for fossil fuels due to their biodegradability, cleaner and low emission of toxic gases [6] [7]. Biomass sources have greater potentials for green energy production and related applications and are able to generate important industrial products such bio-ethanol, bio-methanol and bio-diesel whose production generates bio-glycerol as by-products.

Bio-glycerol is obtained as an important by-product of the transesterification process of vegetable oils for the production of bio-diesel [8] [9]. Bio-glycerol is a polyhydric alcohol which possesses high potential applications in the cosmetic and pharmaceutical industries as well its use as a precursor for the preparation of explosives for constructions and mining applications. Glycerol has manifold applications in various industries such as the pharmaceutical, cosmetic, food, and polymer processing industries [10] [11] [12] [13]. Glycerol can be added to food to improve its water-coating ability and also acts as a solvent for some food additives. In the cosmetics industry, glycerol is used as a soothing and anti-inflammatory agent. In pharmaceutical formulations, lubrication and moisture retention or humectant properties are enhanced by the addition of high purity glycerol. Glycerol is also important in the production of syrups, creams and ointments [14] [15]. Furthermore, glycerol is an antimicrobial preservative and can act as a co-solvent, emollient (in hand sanitizers *inter alia*), humectant, plasticizer, sweetening agent, tonicity agent and solvent. Glycerol is widely used in many pharmaceutical preparations including oral formulations where it acts as a sweetening agent, ophthalmic, topical and parenteral formulations [16]. In topical pharmaceutical formulations and cosmetics, the prime use of glycerol is for its humectant and emollient properties. It is also a viscosity enhancing agent in some pharmaceutical preparations. Glycerol is a plasticizer in film coatings, gelatin capsules and suppositories [17]. Hence, glycerol is a therapeutic agent in a variety of clinical applications. The physical and chemical properties of glycerol enable the molecule to be susceptible to a variety of chemical transformations to different products [18] [19] [20] [21].

Tan *et al.* [22] review the different methods of producing crude glycerol as the major by-product in biodiesel production by transesterification or soap manufacturing by saponification as well as hydrolysis reaction. Azeez *et al.* [23] reported on the physicochemical properties of *Jatropha curcas* seed oil and its use as raw material to synthesize its ethyl ester biodiesel using the base-catalysed transesterification process for thus providing a sustainable strategy and a viable alternative and renewable source for energy generation thus guarding against the over-dependence on fossil fuel and contributing to national energy security and environmental degradation concerns.

Biomass waste generated by the agricultural sector such as palm oil processing factories in Cameroon and other African countries can be absorbed by relevant industries as feedstock to extract vegetable oil for green energy as well as by-products for the cosmetic, pharmaceutical and allied industrial uses [24] [25]. Vegetable oils are potential sources for the production of alternatives to petroleum-based diesel which are esters of fatty acids, the main constituents of vegetable oils. Palm kernel seed oil is not comestible *per se* and a trivial fraction is used in the soap industry. Most of the kernel seeds are therefore dumped with their enormous energy, pharmaceutical and cosmetic potentials.

We report herein, the synthesis and characterization of bio-glycerol obtained as a by-product of the transesterification process of vegetable oils for the production of bio-diesel from palm kernel seed oil obtained from agricultural and industrial waste emanating from palm oil processing industries in Cameroon [26] [27] [28]. Palm kernel seed oil is the oil of choice in this research because it is a nonedible by-product of palm oil processing in which palm kernel seeds tend to accumulate and pollute the environment [29]. The raw materials are readily available and their transformation into useful industrial products will contribute to sanitizing the environment and valorizing industrial waste.

2. Materials and Methods

2.1. Materials

The following materials were used in this research: palm kernel seeds obtained from Widikum sub-division of the North West region of Cameroon, mechanical press for vegetable oil extraction, Soxhlet extractor used for more efficient oil extraction of palm kernel oil from seeds. Potassium hydroxide (99.9%w/v), methanol (99.85%w/v), hydrochloric acid (36%w/v), sulfuric acid (98%w/v) and nitric acid (68%w/v) were obtained from commercial sources and used without further purification.

2.2. Methods

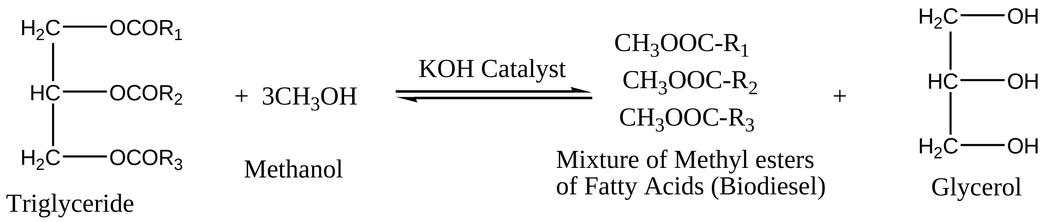
The methods used in this study ranged from oil extraction, purification of the oil, physical methods for oil analysis, chemical methods for oil analysis, synthetic methods for bio-glycerol, methods for testing products, methods for characterizing bio-glycerol, purification methods of synthetic products and by-products and instrumental analytical methods for the characterization of products and by-products.

2.2.1. Extraction of Palm Kernel Seed Oil

Five kilograms of deshelled palm kernel seeds were used produce palm kernel oil using a mechanical press. The mechanically extracted palm kernel oil was purified by warming to 80˚C and allowing it to stand for 2 hours for impurities to settle. The clear supernatant oil was decanted into clean plastic containers and taken to the laboratory for use. The physical and chemical characterization of the palm kernel oil has been previously reported [29].

2.2.2. Synthesis of Bio-Glycerol

Bio-glycerol was synthesized from palm kernel oil using the transesterification process inconformity with the following stoichiometric equation



In the procedure, 500 mL of PKO was transferred into a heating pan and warmed to 60˚C. 118 mL of methanol was transferred into a beaker and 4.6 g of KOH catalyst added. The mixture was magnetically stirred until the methanol completely dissolved the potassium hydroxide. The warm PKO was transferred from the heating steel pan into the reaction vessel (1000-mL round bottom distillation flask) and the catalyst solution was added. The contents of the reaction vessel were swirled vigorously for two minutes so as to form a uniform mixture of oil, reagent and catalyst. The reaction mixture was then subjected to reflux in a water bath at 70˚C for 45 minutes during which the transesterification reaction took place. After this time, the heater was switched off and the reaction mixture allowed to cool. The contents of the reaction vessel were transferred into a separating funnel and the separation of the bio-glycerol and bio-diesel started after 10 minutes.

2.2.3. Recovery of Bio-Glycerol from Reaction Mixture

The separating funnel was allowed to stand overnight for proper separation to take place and the reaction mixture separated into two main layers with a tiny debris layer in between the upper bio-diesel layer (amber colored) and the lower brown colored bio-glycerol layer. The reaction products were carefully separated into different labelled beakers.

2.2.4. Purification of Synthesized Bio-Glycerol by Acidification

The synthesized bio-glycerol was purified by acidification. In this procedure, 50 mL of crude bio-glycerol was measured into each of three 100-mL beakers. The pH in each beaker was adjusted to 1.0 by adding a mixture of 3.0 M solution of nitric acid (HNO_3), 3.0 M solution of hydrochloric acid (HCl), and 2.0 M solution of sulfuric acid (H_2SO_4) drop by drop. The content of each beaker was allowed to settle and the upper layer (pure bio-glycerol) was decanted into a separating funnel. The bio-glycerol was then neutralized with 2.0 M KOH and then decanted. Water impurities in the product were removed by heating the product to 110˚C for 20 minutes. The glycerol was allowed to cool and then filtered to remove some inorganic precipitates. The pure but colored glycerol was passed through a column of activated carbon or charcoal to decolorize, deodorize, and remove some metal ions through adsorption. The pure bio-glycerol was then characterized by determining its vital physical and chemical parameters.

2.2.5. Determination of pH of Bio-Glycerol

The pH of purified bio-glycerol was determined with the aid of a pH meter of serial number 246179 supplied by Crison Instruments S.A. Riera Principal, 34-36 E- 08328- Barcelona. The pH meter was calibrated, and the electrode dried and then dipped into a beaker of bio-glycerol liquid to obtain a stable pH value after the stabilization time.

2.2.6. Determination of Specific Gravity (SG) of Bio-Glycerol

The specific gravity of the bio-glycerol was determined using the density bottle method. The mass of the empty density bottle was measured. Then the density bottle was filled with the newly synthesized bio-glycerol and its mass and contents taken. The mass of the bio-glycerol alone was obtained by subtraction. The specific gravity was then computed from the density formula as follows.

$$\text{S.G.} = \frac{\text{mass}}{\text{mass of equal volume of water}} \tag{1}$$

2.2.7. Determination of ASTM Color of Crude and Purified Bio-Glycerol

In principle, colour indicates the degree of purity of an oil sample. The determination of ASTM D1500 colour was done for bio-glycerol as follows.

50 mL of sample was measured and transferred into a pour point cylindrical jar and 50 mL of distilled water filled in a similar glass jar and placed in the reference compartment of the colorimeter. The sample container was placed in the other compartment of the colorimeter (SETA Lovibold, Stanhope—SETA). The containers were covered to exclude all external light and the machine was turned on to compare the colour of the sample with that of the standard. The sample and reference were viewed through a microscope type of window and a knob in the colorimeter was adjusted until the sample colour matched the colour of the standard. As the colours matched, a figure appeared on a screen and was read off as the ASTM colour.

2.2.8. Determination of Kinematic Viscosities of PKO Bio-Glycerol

The kinematic viscosity of bio-glycerol from palm kernel oil was determined at two temperatures 40˚C and 100˚C using ASTM D445. This test method measures the time for a volume of liquid to flow under gravity through marks on a calibrated glass Cannon-Fenske capillary viscometer. The viscometer was cleaned with organic solvent and dried with the aid of a suction pump. The viscometer was then loaded with bio-glycerol with the aid of a suction pipette. The viscometer was inserted into the viscometer holder and placed in constant-temperature viscometer bath at 40˚C and at 100˚C respectively. A time lapse of 30 minutes was allowed for thermal equilibrium to be attained between the fluid in the bath (filled with water for 40˚C or with glycerin for 100˚C) and that of the bio-glycerol sample in the viscometer. Using the suction pipette, the bio-glycerol sample was raised up the narrow tube above the marks. The time for the meniscus of bio-glycerol took to flow between the upper and lower marks was measured. The measurement was made thrice to obtain readings in seconds whose average was reported using the formula

$$\text{Viscosity}, \eta = Ct \tag{2}$$

where C is the viscometer constant and t is the average flow time.

2.2.9. IR Spectroscopy of Palm Kernel Oil Bio-Glycerol

Bio-glycerol analysis by IR spectroscopy was carried out on the Fourier Transform Infrared Spectrometer manufactured by Agilent Technologies, model number Cary 630. The wavelength range in wave numbers of the IR spectrophotometer was 600 to 400 cm^{-1}.

2.2.10. Determination of the Chemical Composition of Bio-Glycerol by GC-MS Analysis

The chemical composition of the synthesized bio-glycerol was determined using the combination of gas chromatography and mass spectrometry analytical techniques. The analysis was carried out using the GC-MS—QP2010Plus Gas Chromatography-Mass Spectrometer with helium gas as the mobile phase. In this procedure, 2 mL of the bio-glycerol sample was taken into the sample vial which was fitted into the sample rag manually and then subsequent operations were electronically controlled. A rinsing vial was used to clean and rinse the injection needle as a precaution against sample contamination. The gas chromatograph treated the sample first and then conveyed it to the mass spectrometry spectrometer for identification.

2.2.11. Determination of the Sulfur Content of Bio-Glycerol from Palm Kernel Seed Oil

The sulfur content of bio-glycerol was determined using the technique of energy-dispersive X-Ray Fluorescence Spectrometry in accordance with the analytical method of ASTM D4294 and accomplished with the help of the Sulfur-in-Oil Analyzer-SLFA-2800 machine. The prepared sample cell was placed such that the transparent window film was facing downwards into the X-ray detector compartment, and then it was closed. The results for sulfur contents were expressed as %v/v.

3. Results and Discussion

3.1. Bio-Glycerol Synthesis

Figure 1 shows the synthesized bio-glycerol as the lower layer while the upper layer is bio-diesel. In general, most of bio-glycerol is obtained as a by-product of bio-diesel production [26] [27]. Glycerol is a versatile product that can act as a raw material for various industrial applications and goes a long way to promoting green manufacturing processes [28].

3.2. Physico-Chemical Parameters of Bio-Glycerol

The results of some relevant physico-chemical properties of the synthesized bio-glycerol are shown in **Table 1** below.

Figure 1. Synthesized bio-glycerol (lower layer) and methyl esters (upper layer).

Table 1. Physico-chemical parameters of Bio-glycerol.

Parameter	Value	Literature Value [30] [31]
Specific Gravity	1.20	1.26
ASTM Color	1.50	1.20
Viscosity @ 40˚C	1500 cSt	648 cSt
Viscosity @ 100˚C	500 cSt	276 cSt
Flash point	160˚C	176˚C
pH	7.4	7.0 - 7.5

From Table 1, the specific gravity of bio-glycerol is high showing that it has a higher mass per unit volume than water. This value is closed to that reported by previous researchers. A small volume of the product is thus relatively heavier compared to equal volumes of water or fluids with lower values of specific gravity [32]. The specific gravity is important in designing mixers to produce the required torque and horsepower needed to properly mix fluids during processes of glycerol transformation. When the specific gravity is high, there must be a corresponding high torque in order to produce good results. If specific gravity considerations are not made, the mixer will not be appropriately optimized with attendant unpredictable mixing results leading also to motor damage in extreme cases [33]. The viscosity values of bio-glycerol are really high at the two temperatures studied (40˚C and 100˚C). These values are relatively high probably due to the contribution of impurities in the crude bio-glycerol. Viscosity values determine the forces that have to be overcome when fluids are used in lubrication and during transportation in pipelines. Viscosity controls liquid flow rates in processes like spraying, injection molding, surface coating and related applications [34]. The slight ASTM colour difference translates the lower impurity content. The product is thermally stable by virtue of its high flash point and does not pose a threat of a fire hazard during usage. The pH is near the neutral zone which makes it safe for transport or processing in metallic containers with little risk of corrosion and equipment damage. The pH value obtained for bio-glycerol falls within the range reported in the literature.

3.3. IR Analysis of Bio-Glycerol

The Infrared (IR) analysis of the synthesized bio-glycerol is shown in **Figure 2** below. The chemical identity of glycerol is propan-1,2,3-triol having three hydroxyl groups responsible for the hygroscopic nature and water solubility of the product [35].

From the IR spectrum of bio-glycerol, the peak that occurs at 3304 cm^{-1} indicates the presence of O-H bending groups. The absorption peaks at 2922 and 2855 cm^{-1} fall in the absorption range for C-H stretching in the hydrocarbon component of the bio-glycerol. The absorption peaks at 1110.7 cm^{-1} indicate the presence of C-C, C-O and C-H bonds in carbonyl compounds, alcohols and esters [36] [37]. The above data collectively confirm the presence of all the functional groups of bio-glycerol from palm kernel oil.

3.4. GC-MS Analysis of Synthesized Bio-Glycerol

A more robust method for chemical identification is the GC-MS procedure and technology. The gas chromatographic and mass spectrometric analyses of bio-glycerol revealed that the substance is made up of propan-1,2,3-triol molecules with other impurity molecules as shown by the mass spectrum in **Figures 3-6** bellow.

Formula of compound: $C_3H_8O_3$, Molecular Weight: 92. Alternative names to the compound include; Glycerin, 1,2,3-Propanetriol, Glycerol, Glycerine, Glyceritol, Glycyl alcohol, Glyrol, Glysanin, Osmoglyn, Propanetriol, and Trihydroxypropane.

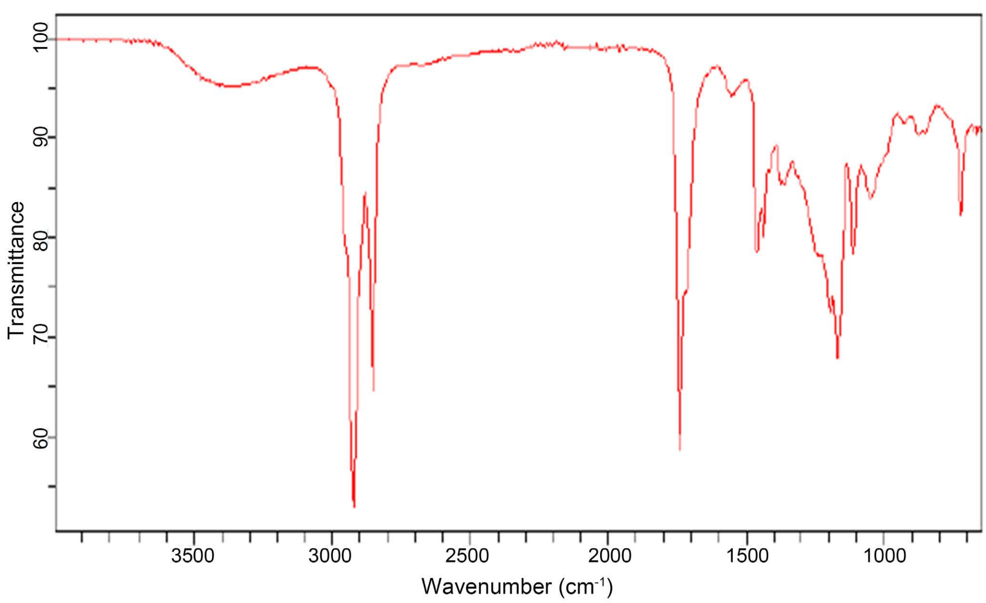


Figure 2. IR spectrum of bio-glycerol.

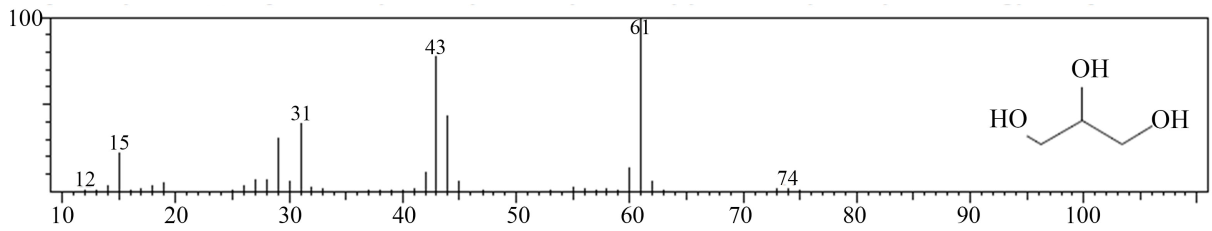


Figure 3. GC-MS spectrum of bioglycerol.

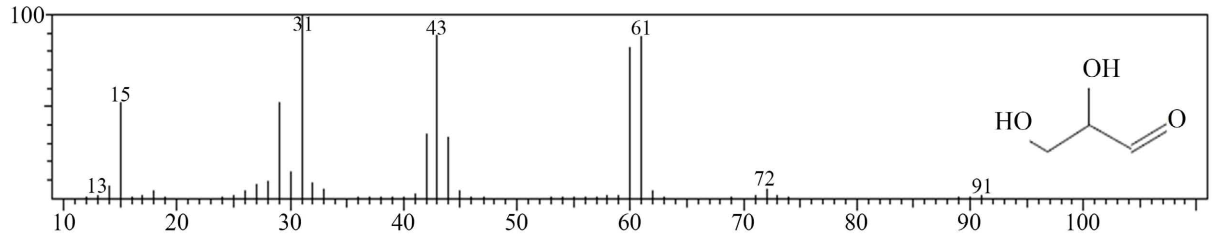


Figure 4. GC-MS spectrum of glyceraldehyde (an impurity).

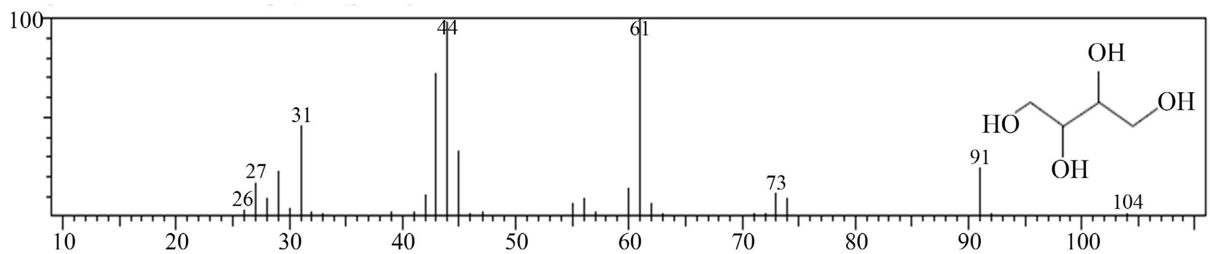


Figure 5. GC-MS spectrum of 1,2,3,4-btanetetrol (an impurity).

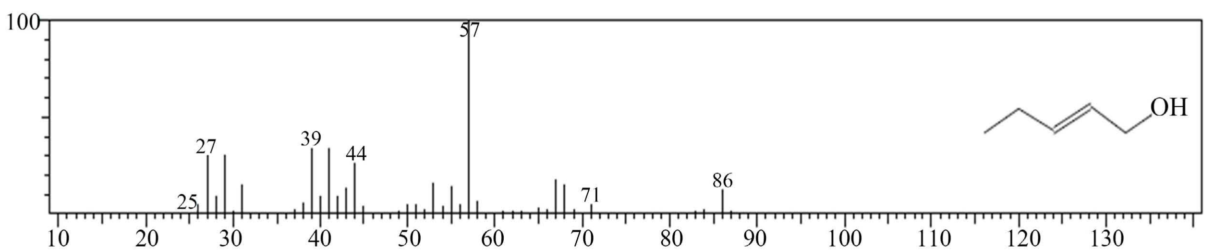


Figure 6. GC-MS spectrum of 2-pentenol (an impurity).

Other molecules that featured in the GC-MS analysis of glycerol which may be derived from glycerol included.

1) Glyceraldehyde

Formula of compound: $C_3H_6O_3$, Molecular Weight: 90. Other names of the compound include; Propanal, 2,3-dihydroxy, and DL-Glyceric. Glyceraldehyde may be produced from glycerol through oxidative dehydrogenation. This molecule may result due to partial oxidation of the glycerol molecule in the reaction medium.

2) 1,2,3,4-Butanetetrol

Formula of compound: $C_4H_{10}O_4$, Molecular Weight: 122. Other names of the same compound include; Erythritol, and l-Threitol. This impurity molecule is due the chain extension reaction caused by the addition of a methanol molecule to the glycerol structure.

2-Penten-1-ol

Formula of compound: $C_5H_{10}O$, Molecular Weight: 86. Another name of the compound include is (2E)-2-Penten-1-ol. The impurity molecules are may be obtained from glycerol and from other molecular fragments in the transesterification medium through oxidative and slight polymerization processes in the reaction medium.

4. Conclusion

Crude bio-glycerol was obtained through transesterification of palm kernel seed oil (an agricultural waste). The crude bio-glycerol was purified and characterized. The results of the physical and chemical characterization indicate that bio-glycerol is a potent raw material that can meet the needs of various industries in Cameroon and beyond. Research in green energy production especially biodiesel has led to the expansion of industries producing biodiesel with attendant accumulation of bio-glycerol since 10%w/w of the bio-diesel produced is crude glycerol obtained as by-product. Apart from the existing industries that currently make use of glycerol, the development of more innovative technologies for the conversion of crude glycerol to new product needs to be encouraged in Cameroon and beyond. Hence, obtaining bio-glycerol from palm kernel seed oil further enhances industrial waste minimization, environmental sanitization and cost-effective manufacturing in Africa in general and Cameroon in particular.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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