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COMPARATIVE STUDY OF CASTOR, COTTON AND NEEM OIL SAMPLES AS SOURCES GLYCERIN

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Abstract - The study focused on the comparative analysis of castor oil (Ricinus communis), cottonseed oil (Gossypium hirsutum), and neem oil (*Azadirachta indica*) as sources of glycerin. The oil samples were characterized to ascertain their proximate analysis. Castor oil, cottonseed oil, and neem seed oil had saponification values of 182.71 mg/g, 185.06 mg/g, and 193.45 mg/g, respectively. Other physicochemical properties evaluated included acid value, free fatty acid content, iodine value, density, and peroxide value using standard titrimetric and analytical methods. The oil samples are appropriate for the synthesis of glycerin, according to the recorded average saponification values as well as the properties of the oils. Glycerin was synthesized from the oils through transesterification using methanol in the presence of an alkaline catalyst under controlled temperature and reaction time. After the reaction, phase separation was allowed to occur, and the crude glycerin layer was separated, washed, and dried before further analysis. The produced glycerin samples were characterized by determining their kinematic viscosity, moisture content, and chemical composition using standard laboratory procedures and spectroscopic techniques. The presence of heteroatoms (N and O) in each glycerin product indicates that glycerin is a possible raw material for the synthesis of bio-grease. Trimethylolpropane, 4-ethoxy-4-oxobutanoic acid, dihydro-2-methyl-3-furanone, linoleic acids, neopentyl glycol, methyl ester, and pentaerythritol were identified as the chemical constituents of the glycerin. The glycerin made from castor oil, cottonseed oil, and neem seed oil has kinematic viscosities of 928 cP, 931 cP, and 933 cP, respectively. Additionally, the glycerin made from castor oil, cottonseed oil, and neem seed oil had moisture contents of 1.92%, 2.15%, and 2.04%, respectively. Castor oil outperformed cottonseed and neem seed oil in terms of the glycerin yield. The yield was influenced by the increase in oil/methanol ratio, catalyst concentration, temperature, and time. Overall, the results show that all three oils are suitable sources of glycerin, with castor oil giving the highest yield under the studied conditions. The study confirms their potential for industrial glycerin production and related applications.

Keywords: Glycerin, Characterization, oil

1. Introduction

Glycerol is a simple triol molecule that is also known as glycerine or glycerin. It is a sweet-tasting, thick, colorless, odorless, and non-toxic liquid. Lipids called glycerides have the glycerol backbone. It is also frequently employed as a humectant in medicinal formulations and as a sweetener in the food sector. Glycerol is hygroscopic and miscible with water due to its three hydroxyl groups (Christoph et al., 2006). Typically, glycerol comes from plants and animals in the form of triglycerides, which are glycerol esters with long-chain carboxylic acids. These triglycerides can be hydrolyzed, saponified, or transesterified to yield glycerol and the fatty acid derivative. Common plant sources are palm or soybeans. Another source is tallow obtained from animals. Between 2000 and 2004, the United States alone produced 350,000 tons of glycerol annually, out of the approximately 950,000 tons produced annually

in Europe and the United States (Nilles, 2005). By 2010, all member states were required to replace 5.75% of petroleum fuels with biofuels under EU Directive 2003/30/EC. According to a 2006 prediction, supply will surpass demand six times over by 2020, resulting in an excess of glycerol as a byproduct of the manufacturing of biofuel (Christoph et al., 2006). Despite the large-scale production of glycerol from triglycerides, the crude product has a low selling price of as low as US$0.02–0.05 per kilogram in 2011 and is of inconsistent quality (San Kong et al., 2016). Although purification is possible, the procedure is costly. Although glycerol has a low heat value, some of it is burned for energy (Sims, 2011).

2. Materials and Methods

2.1 Raw materials and equipment

Every chemical reagent that is employed is of analytical grade. Ogbete market in Enugu is where the materials and chemicals utilized in this experiment were bought. These consist of distilled water, methanol, magnesium oxide, castor oil, cottonseed oil, and neem seed oil. A stopwatch, measuring cylinders, beakers, an electric heater with a magnetic stirrer, a reflux condenser, conical flasks, an electronic weighing balance, and a Fourier transform infrared (FTIR) spectrophotometer are among the tools and devices utilized.

2.2 Method of Experimentation

2.2.1 Oil sample characterization and density determination

A 100 ml empty bottle's density was measured using an electronic weighing scale. The weight of the oil and container was then noted when the oil was poured into the bottle until it was full. This procedure was repeated, and the density was calculated. The density of each oil sample was determined using the density bottle method and calculated using Equation (1).

$$Density\ (\rho)\ (g/cm3) = \frac{W_2 - W1}{V} \quad (1)$$

Where:

W1 = weight of empty density bottle
W2 = weight of density bottle + sample
V = volume of the density bottle

2.2.2 Saponification Value (S.V.) Determination

A 250 mL conical flask containing 30 mL of each oil sample was mixed with 25 mL of 0.5 M ethanolic potassium hydroxide (KOH) solution. The flask was fitted with a reflux condenser and heated over a water bath for 30 minutes with constant swirling to ensure complete saponification. After heating, the mixture was allowed to cool slightly. A few drops of phenolphthalein indicator were added, and the excess potassium hydroxide was titrated with 0.5 M hydrochloric acid (HCl) until the pink colour disappeared. A blank determination was carried out under the same conditions using distilled water instead of the oil sample. The saponification value was calculated using Equation (2):

$$Saponification(S.V) = \frac{(B-R)^{\times}28.05}{\text{Weight of oil}} \quad (2)$$

where R is the titre value of the actual sample (oil), and B is the titre value of the blank (distilled water).

2.2.3 Free Fatty Acid (FFA) Determination

A 250 mL conical flask was first warmed gently. Forty millilitres (40 mL) of the oil sample was introduced into the flask, followed by the addition of 25 mL of methanol. The mixture was swirled thoroughly to ensure proper mixing. Two drops of phenolphthalein indicator were added, after which the mixture was titrated with 0.14 M sodium hydroxide (NaOH) solution while shaking continuously. The titration was continued until a faint pink colour appeared and persisted for about one minute, indicating the end point. The Free Fatty Acid (FFA) value was calculated using Equation (3):

$$FFA(as\ oleic)\% = \frac{\text{Titre}^{\times}\text{N}^{\times}\ 28.2}{\text{Weight ofsample}} \quad (3)$$

where N is the base's molarity.

2.2.4 Assessing the peroxide value (PV)

A 22 cm3 solution of 10 cm3 acetic acid and 12 cm3 chloroform was mixed with 2.0g of oil. The flask was filled with 0.5 cm3 of saturated potassium iodide. After corking the flask, it shook sporadically for a minute. After adding 30 cm3 of distilled water, the mixture was titrated against 0.1M of Na2SO3 until the yellow color was nearly completely gone. After adding the starch indicator in 0.5 cm3

increments, titration was carried out until the blue hue hardly changed. The identical conditions were used for a blank titration as well. The peroxide value was calculated using Equation (4):

$$PV(meq/Kg)\% = \frac{(S-B)\text{ x N x }1000)}{W} \quad (4)$$

Where: PV = Peroxide value ($meq \cdot kg^{-1}$), S = Volume of sodium thiosulfate solution used for the sample (**mL**), B = Volume of sodium thiosulfate solution used for the blank (**mL**), N = Normality of sodium thiosulfate solution ($\mathbf{mol \cdot L^{-1}}$), and W = Weight of oil sample (**g**)

2.2.5 Calculating the acid value (A.V.)

A 250 ml conical flask was warmed before 40 ml of each oil sample was added. When a vivid pink color was seen that persisted for a minute, two drops of phenolphthalein indicator, one drop of 0.14M sodium hydroxide solution, and vigorous shaking were added.

The acid value was calculated using Equation (5)

$$Acid\ Value\ (mgKOH/g) = \frac{V\text{ x M x }56.1)}{W} (5)$$

The FFA value was determined using this point, which was noted as the end point, as indicated in equation 6:

$$Acid\ Value\ \left(\frac{mgKOH}{g}\right) = \%\ \text{FFA (as oleic) x 1.99} \quad (6)$$

2.2.6 Determination of iodine value

A 250 mL conical flask stopper was filled with 15 mL of the sample, and it was left for precisely 30 minutes. To remove any remaining iodine from the stopper, 10 milliliters of a 15% W/V potassium iodine solution was added to the flask. This was titrated until the sodium turned bright yellow against 0.14M ($Na_2S_2O_3$). After adding 1% starch indicator (2 milliliters), the titration proceeded until the blue hue vanished. The same conditions were used for a blank (distilled water) determination. As shown below, the titre value was recorded and used to determine the iodine value. The iodine value was calculated using Equation (7):

$$Iodine\ value = \frac{(B-R\ )\text{xmolarities of }(Na_2S_2O_3).}{\text{Weight of sample}} (7)$$

B= Titre value of blank (distilled water), R=Titre value for real determination.

2.2.7 Glycerin Production Process

The transesterification process was carried out for glycerin production using MgO as a heterogeneous catalyst. A 100 g oil sample was preheated to 60°C and transferred into a conical flask. Agitation was maintained at 250 rpm for 60 minutes. A prepared mixture containing 0.7 g MgO and methanol at a 6:1 methanol-to-oil molar ratio was added to the heated oil. The reaction mixture was further heated at 75°C for 70 minutes under continuous stirring.

After completion of the reaction, the mixture was transferred into a separating funnel and allowed to settle undisturbed for 24 hours at ambient temperature to facilitate complete phase separation. Extended settling time promoted gravitational separation between glycerin and the ester phase. The glycerin layer was then carefully decanted into a PET bottle. The glycerin yield was calculated as a percentage of the initial oil feed volume.

2.2.8 Characteristics of the Glycerin

1. Functional Groups of the Glycerin (FTIR)

To identify the glycerin's functional groups, a Fourier transform infrared (FTIR) spectrophotometer (Cary 630, Agilent Technologies USA) was employed. Over a broad spectrum of spectra, the FTIR spectrophotometer gathered data with great spectral resolution. The raw data was transformed into the real spectrum using a mathematical procedure called the Fourier transform. To identify the proper functional groups, analysis of the different FTIR-produced peaks was done.

2. Chemical Compositions of the Glycerin using GCMS

A chemical analysis of the glycerin was carried out using a gas chromatography-mass spectrophotometer (GC–MS) (Agilent Technologies model 7890A and 5977B MSD). The gas chromatography-mass spectrophotometer integrated the capabilities of gas chromatography (GC) and mass spectrometry (MS) for the detection of compound identification and quantification in a test sample of each glycerin. The glycerin separated into different compounds when heated in the GC. The heated materials were sent through an inert gas column filled with nitrogen. The divided materials flew into the MS as they came out of the column hole. The

chemicals in the glycerin were identified using the MS.

3. Determination of Physico-chemical properties

The physico-chemical properties of the produced sample were evaluated to determine its quality and conformity with standard specifications. The parameters analyzed and the corresponding methods used are presented in Table 1.

The ASTM D7042-04 standard was followed in determining the kinematic viscosity. A sample was injected into a digital automatic viscometer analyzer set to 40 °C for analysis.

After a known amount of the sample was carefully weighed in an aluminum weighing dish, it was dried in a closed oven at 40 to 45°C for about 48 hours to maintain a steady weight. The sample was weighed after 45 hours, re-oven-tested after 48 hours, and then weighed again.

The sample is deemed dry when the weight difference is less than 2%. The moisture content in the sample was then determined by weighing the dried sample. Moisture content is thus calculated as:

$$Initial\ weight\ of\ the\ sample\ (wet) = \text{Xgm}$$

After drying the sample;

$$Final\ weight\ of\ the\ sample\ (dry) = \text{Ygm}$$

$$Moisure\ content\ (Z\ \%) = 1 - \frac{\text{Y}}{\text{X}}\ X\ 100 \quad (8)$$

2.2.8 Process Variables' Impact on Glycerin Yield

The effects of temperature, reaction time, catalyst concentration, and methanol-to-oil ratio on glycerin yield were evaluated by varying one parameter at a time while keeping the others constant. After each reaction, the mixture was allowed to settle for a fixed period to ensure phase separation. The glycerin layer was separated and weighed. Glycerin yield was calculated as:

$$Glycerin\ Yield\ (\%) = \frac{\text{Mass of glycerin obtained}}{\text{Initial mass of oil}} x\ 100 \quad (9)$$

3 Results and Discussion

3.1 Features of the Composition

3.1.1 **Characteristics of the oil samples**

The physico-chemical parameters of the oils are displayed in Table 1. It illustrates differences in the oil samples' characteristics (acid, free fatty acid, density, saponification, iodine, and peroxide readings). Castor oil, cotton seed oil, and neem seed oil had saponification values of 182.71 mg/g, 185.06mg/g, and 193.45 mg/g, respectively. The oils can be used to produce glycerin because of their saponification properties. This is the case because a higher saponification value results in the formation of soap (saponification) as opposed to glycerin or methyl ester, as may be the case. According to earlier research (Onyegbado et al., 2002; Minodora et al., 2010; Koh and Ghazi, 2011; Janajreh et al., 2015), very high oil saponification values promote soap formation rather than the expected product (Miyuranga et al., 2013; Farooq et al., 2013; Esonye et al., 2019; Hundie and Akuma, 2022). The assertion that the oils are suitable for the synthesis of glycerin is supported by additional values of the oils' characteristics, including their acid, free fatty acid, iodine, density, and peroxide levels.

Table 1: Physico-chemical properties of the oil samples

Properties	Castor oil	Cotton seed oil	Neem seed oil
Density (g/cm^3)	0.889	0.884	0.887
Saponification value (mg/g)	182.71	185.06	193.45
Acid value (mg/g)	3.46	3.32	3.40
Free fatty acid (%)	1.73	1.66	1.70
Iodine value (g/100g)	31.98	35.12	33.26
Peroxide value (meq/kg)	0.44	0.39	0.42

3.2 Characterization of the palm fruit bunch

The proximate analysis of the palm fruit bunch shows moderate moisture (9.87%), low protein (3.76%), moderate crude fibre (10.22%), low crude fat (2.31%), and high ash content (15.45%) (Table 2). These values indicate its potential suitability as a biomass material for

industrial and agricultural applications. The results show that the palm fruit bunch contains moderate moisture content, which supports better storage stability. The low protein and fat contents indicate that it is not a nutrient-rich feed material but mainly a structural biomass. The moderate fibre and high ash content suggest its suitability for bioenergy production and soil amendment applications.

Table 2: Proximate analysis of the palm fruit bunch

Composition	Palm fruit bunch
Protein (%)	3.76
Moisture content (%)	9.87
Crude fibre (%)	10.22
Crude fat (%)	2.31
zAsh (%)	15.45
Carbohydrate (%)	58.39

3.3 Characteristics of the Glycerin

3.2.1 Functional groups of the glycerin

Figures 1–3 show the FTIR spectra of glycerin derived from castor, cottonseed, and neem seed oils. All spectra exhibit a broad and intense band at approximately **3200–3500 cm⁻¹**, attributed to O–H stretching vibrations, confirming the presence of hydroxyl functional groups characteristic of glycerol. Peaks within **2850–2950 cm⁻¹** correspond to C–H stretching, while bands observed around **1000–1150 cm⁻¹** are assigned to C–O stretching vibrations. The absence of strong ester carbonyl peaks (~1740 cm^{-1}) indicates effective hydrolysis/transesterification and successful separation of glycerin from the parent oils. The spectral profiles are consistent with reported FTIR signatures of glycerol, supporting its structural identity and suitability for bio-based applications such as bio-grease synthesis.

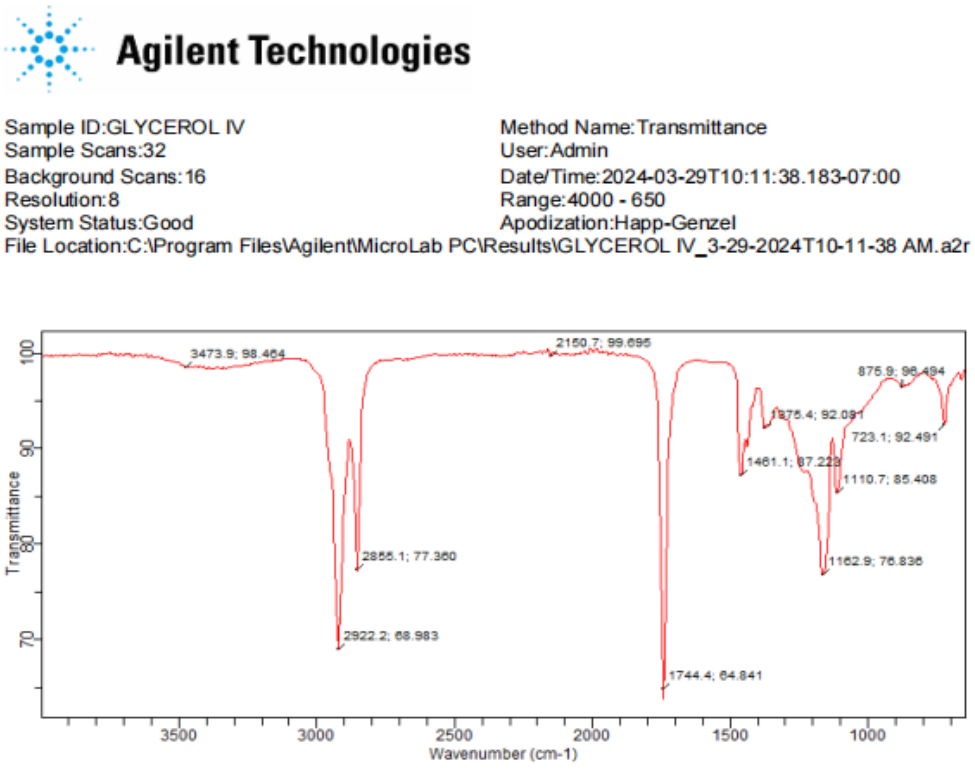

Figure 1: Spectrum of glycerin from castor oil

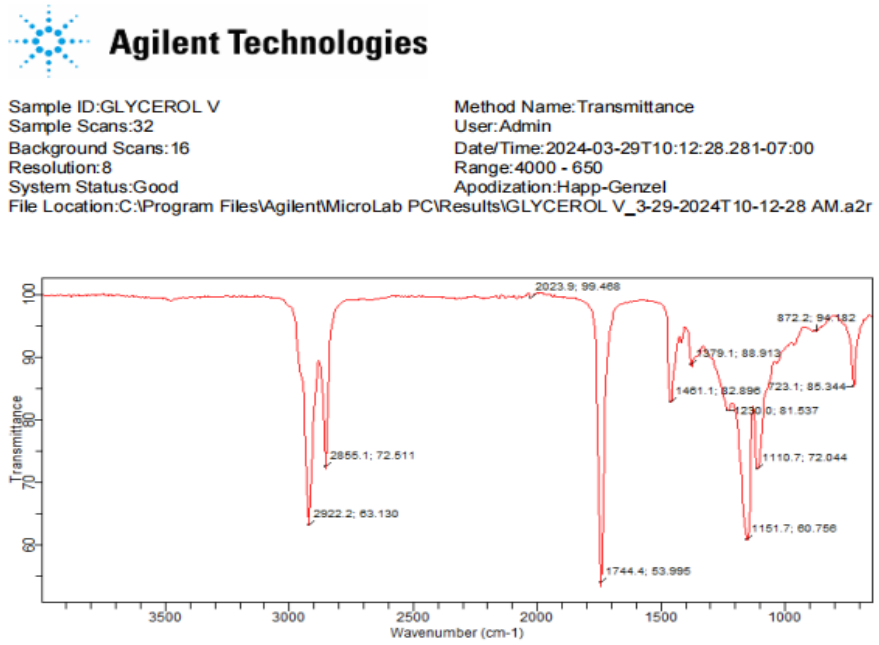

Figure 2: Spectrum of glycerin from cotton seed oil

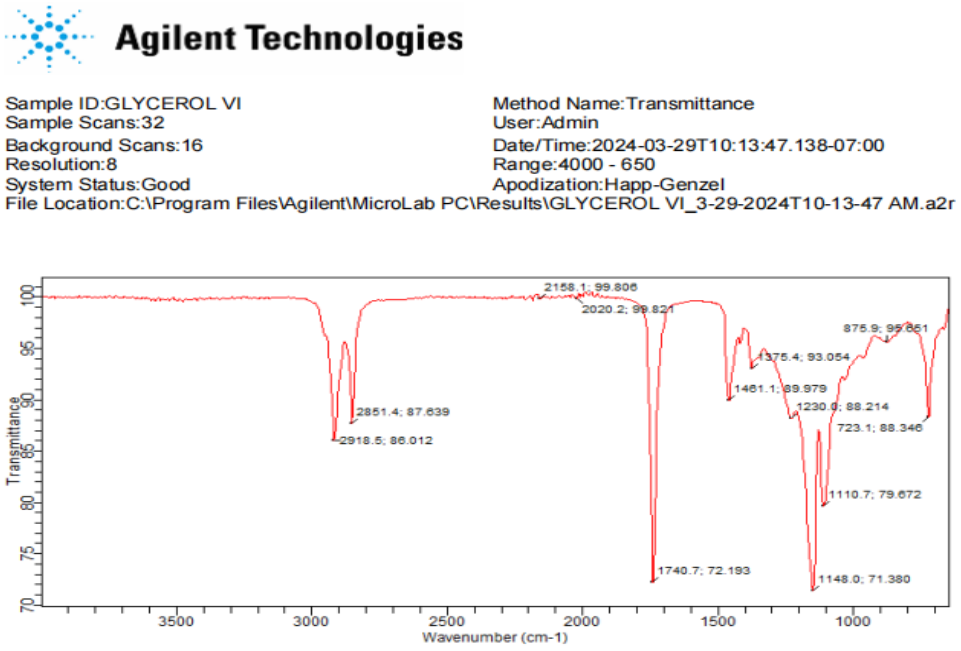

Figure 3: Spectrum of glycerin from neem seed oil

3.2.2 Composition of the glycerin

Figures 4 and 6 display the chromatograms of the castor oil, cotton seed oil, and neem seed oil that were generated using glycerin, respectively. The chemical constituents of the glycerin were acquired using a gas chromatography mass spectrophotometer (GCMS). Trimethylolpropane, 4-ethoxy-4-oxobutanoic acid, dihydro-2-methyl-3-furanone; linoleic acids, neopentyl glycol, methyl ester and pentaerythritol were identified as the chemical constituents of the glycerin. It shows that obtained glycerin is appropriate for the synthesis of bio-grease (El-Adly et al,2015; Chilakamarry, 2021; Ben et al, 2022).

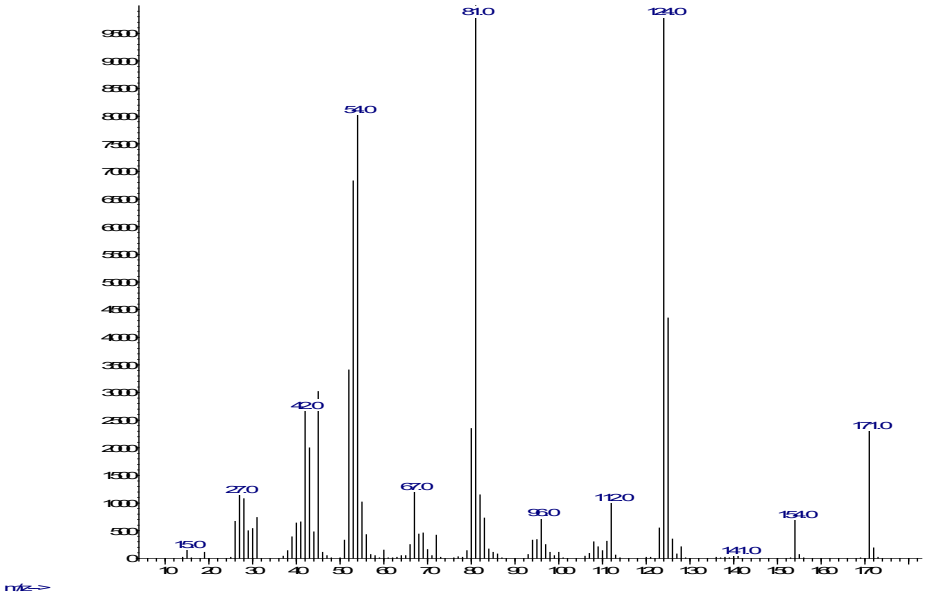

Figure 4: Chromatogram of the glycerin produced from castor oil

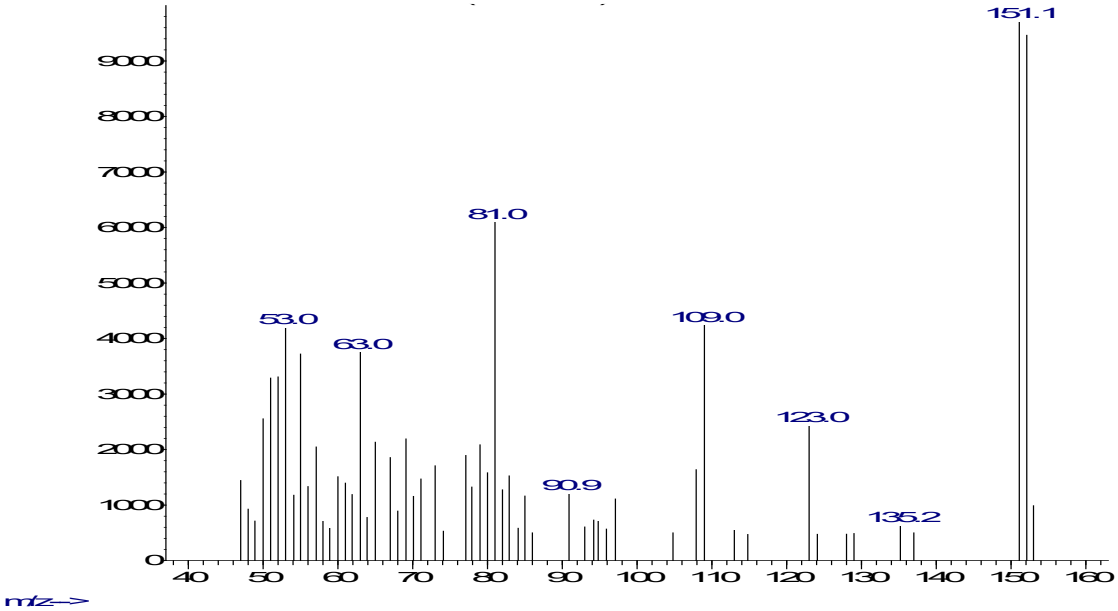

Figure 5: Chromatogram of the glycerin produced from cotton seed oil

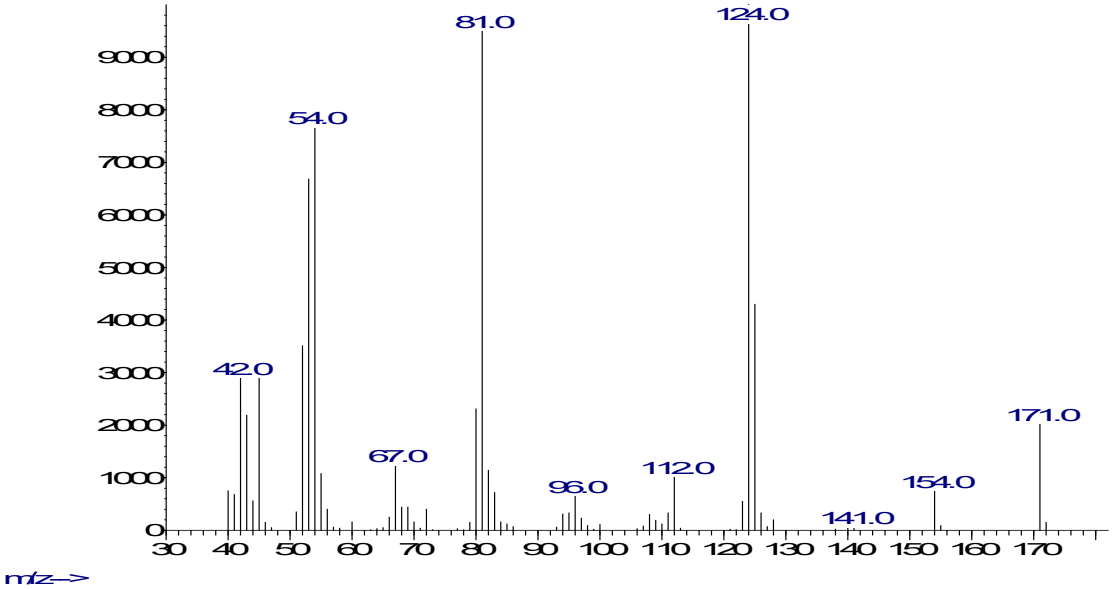

Figure 6: Chromatogram of the glycerin produced from neem seed oil

4.2.3 Viscosity and moisture content of the glycerin

The glycerin samples made from castor oil, cotton seed oil, and neem seed oil have kinematic viscosities of 928cP, 931cP, and 933cP respectively (Table 3). Additionally, the glycerin samples made from castor oil, cotton seed oil, and neem seed oil have moisture contents of 1.92%, 2.15%, and 2.04%, respectively. These findings demonstrate high quality of glycerin (Chilakamarry, 2021; Ben et al, 2022).

Table 3: kinematic viscosity and moisture contents glycerin

Property	Castor oil	Cotton seed oil	Neem seed oil
Kinematic viscosity	928cP	931cP	933Cp
Moisture content	1.92%	2.15%	2.04

4.3 Process factors' impact on the production of glycerin

Figures 7–10 illustrate how process factors affect the glycerin yields. According to the graph in Figure 7, the glycerin yield rose as the oil/methanol ratio climbed until it reached its maximum and then started to fall. A similar pattern can be seen in Figures 8 through 10. In terms of the value of the glycerin yield, castor oil outperformed cotton seed and neem seed oils. The composition of the raw material utilized may be responsible for high glycerin production values (Raqeeb and Bhargavi, 2015; Mostafa and Gelareh, 2016; Kächele et al, 2019; Vávra et al, 2021; Ayatullahi et al, 2023). This indicates that the composition of the raw materials affects the levels of the transesterification products (glycerin and methyl ester).

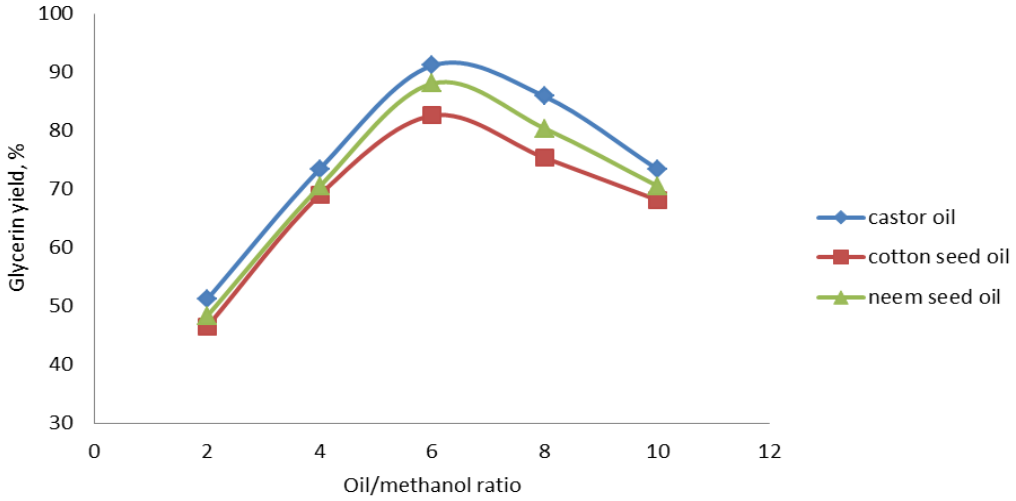

Figure 7: Relationship between glycerin yield and oil/methanol ratio

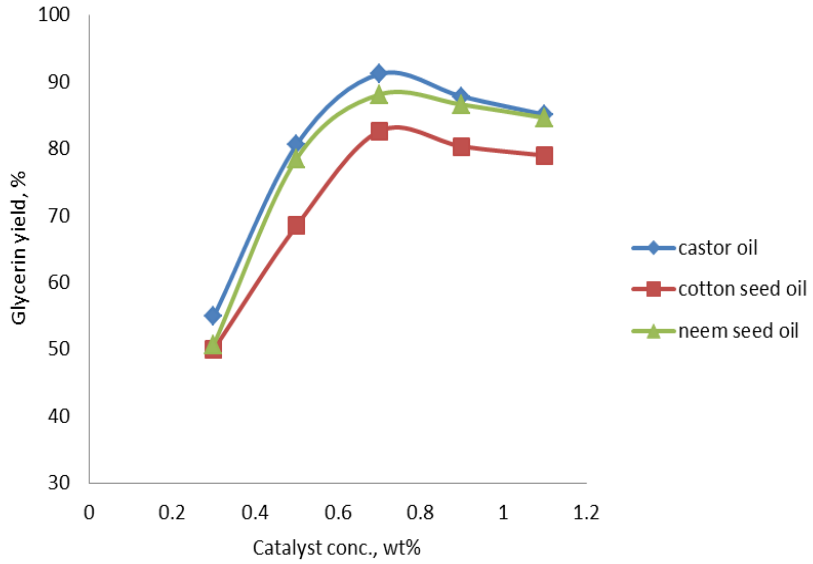

Figure 8: Relationship between glycerin yield and catalyst conc.

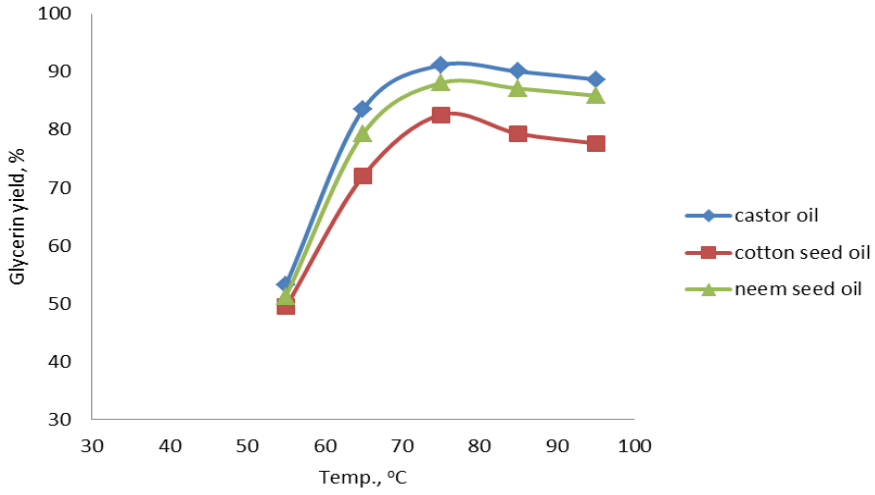

Figure 9: Relationship between glycerin yield and temperature

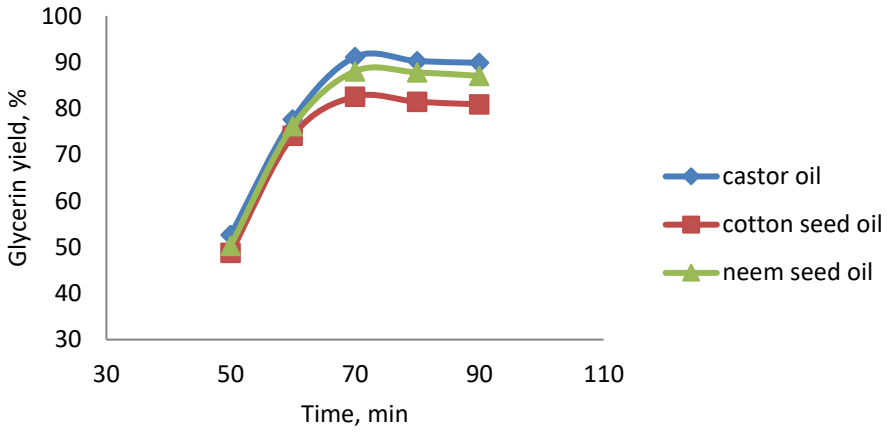

Figure 10: Relationship between glycerin yield and time

4. Conclusion

The physico-chemical properties of castor, cottonseed, and neem seed oils confirmed their suitability for glycerin production based on their saponification, acid, iodine, density, and peroxide values. Transesterification with MgO produced glycerin with viscosities of 928–933 cP and moisture contents of 1.92–2.15%, indicating effective conversion. FTIR and GC–MS analyses verified characteristic glycerol functional groups and identified compounds suitable for bio-grease applications. Castor oil gave the highest yield, with yield influenced by oil/methanol ratio, catalyst concentration, temperature, and time. The study demonstrates the feasibility of producing quality glycerin from the selected oils for industrial use.

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