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EXTRACTION, CHARACTERIZATION AND FT-IR ANALYSIS OF OIL EXTRACTED FROM COTTON SEED (GOSSYPIUMHIRSUTUM)

Kamalu Garba Sulaiman,
Department of Chemistry
Yusuf Maitama Sule University, Kano Nigeria.

Aminu Rabi'u Doguwa, Abubakar Suraja Ibrahim and Nasiru Ado Muhammad
Department of Chemistry
Bayero University, Kano Nigeria.

Abstract: **In this study cottonseed oil extracted from Gossypiumhirsutum (Cotton seed) was carried out. The extraction was done using soxhlet method with n-hexane as the solvent. The extracted oil was characterized by different parameters. This includes the acid value, the free fatty acid value, the saponification value, the boiling point, the density, the pH value as well as the FT-IR spectra. The acid value, free fatty acid value, saponification value, and boiling point were found to be 3.903 mg KOH/g, 1.914 mg KOH/g, 193.23 mg KOH/g and 234^{0}C respectively. The density of the oil was found to be 0.928 gcm^{-3}, while the pH value was 5.6. FT-IR analysis was carried out to ensure the quality of the extracted oil. The functional groups detected were sp^2 C-H stretches, sp^3 C-H stretches, a strong band of –C=O of an ester and medium band of –C-O of an ester, this confirmed that the oil was not rancid and my resist rancidity due to the presence of tocopherol. The oil was found to be edible and of numerous potentials domestically and industrially after comparisons were made with values obtained from literature.**

Key words: **Cottonseed oil, Tocopherol, Rancidity, FT-IR (Fourier Transform Infrared Radiation)**

I. INTRODUCTION

Approximately 85% of the fats and oils consumed globally now are vegetable oils (O' Brien, 1998). One of the most significant commercial crops in the world, cotton once served as the primary natural supply of fiber. As the foundation of the textile industry, it has a huge impact on the industrial economy. However, the kernel of cottonseeds contains a sizable amount of oil. Cottonseed oil concentration ranges from 12 to 25%. (Mert et al., 2004). Cottonseed oil is produced as a byproduct and provides a significant portion of the country's oil needs. After soy and sunflower oil, cottonseed oil is the oil that has been used the most.

Many competitions arise between the vegetable plants from which the oil is extracted. Cottonseed oil emanates to be at the top of those competitions. It worsens to the point that, in the 1950s, cotton seed oil was the most popular oil eaten in the United States, but soya bean oil eventually overtook it because of its low production. Despite having a lot of negative effects, consumer services use the oil well since it enhances the flavor of snacks and preserves their taste. It also gives them a wonderful mouthfeel. It might be challenging for many food service organizations to effectively preserve long-lasting food snacks. But cotton seed oil helps to make this easier. This is because using cottonseed oil to cook with improves the flavor of the food (Jones and King, 1993)

Gossypium species provide the cottonseed oil. These species are raised for use as animal feed, cotton fiber, and other products. Cottonseed oil is typically used in cooking oil, salad oil, and vegetable oil combinations (Metin et al., 2003). It is also utilized in the production of margarine, shortening, mayonnaise, and sauces, but to a lesser level (Sekhar and Rao, 2011).

Tocopherol (1), a natural antioxidant, is a key ingredient of cottonseed oil. The amount of tocopherol (1) in oil does, however, considerably decrease after refining. As a result, unrefined cottonseed oil has a higher tocopherol content and is more oxidation-resistant than refined cottonseed oil and soybean oil (Saxena et al., 2011; Sekhar and Rao, 2011). One of the most crucial characteristics of cottonseed oil is its fatty acid makeup (Ping et al., 2009).The proportion of polyunsaturated to saturated fatty acids in cottonseed oil is 2:1. Because it typically contains 70% unsaturated fatty acids, including 18% mono-unsaturated (oleic (2)) and 52% poly-unsaturated (linoleic (3)), and 26% saturated fatty acids (mainly palmitic (4) and stearic (5)), it is referred to be naturally hydrogenated. Without the requirement for extra processing or the creation of trans-fatty acids, these made the oil stable enough for frying (Sekhar and Rao, 2011), despite the fact that trans-fatty acids have been linked to

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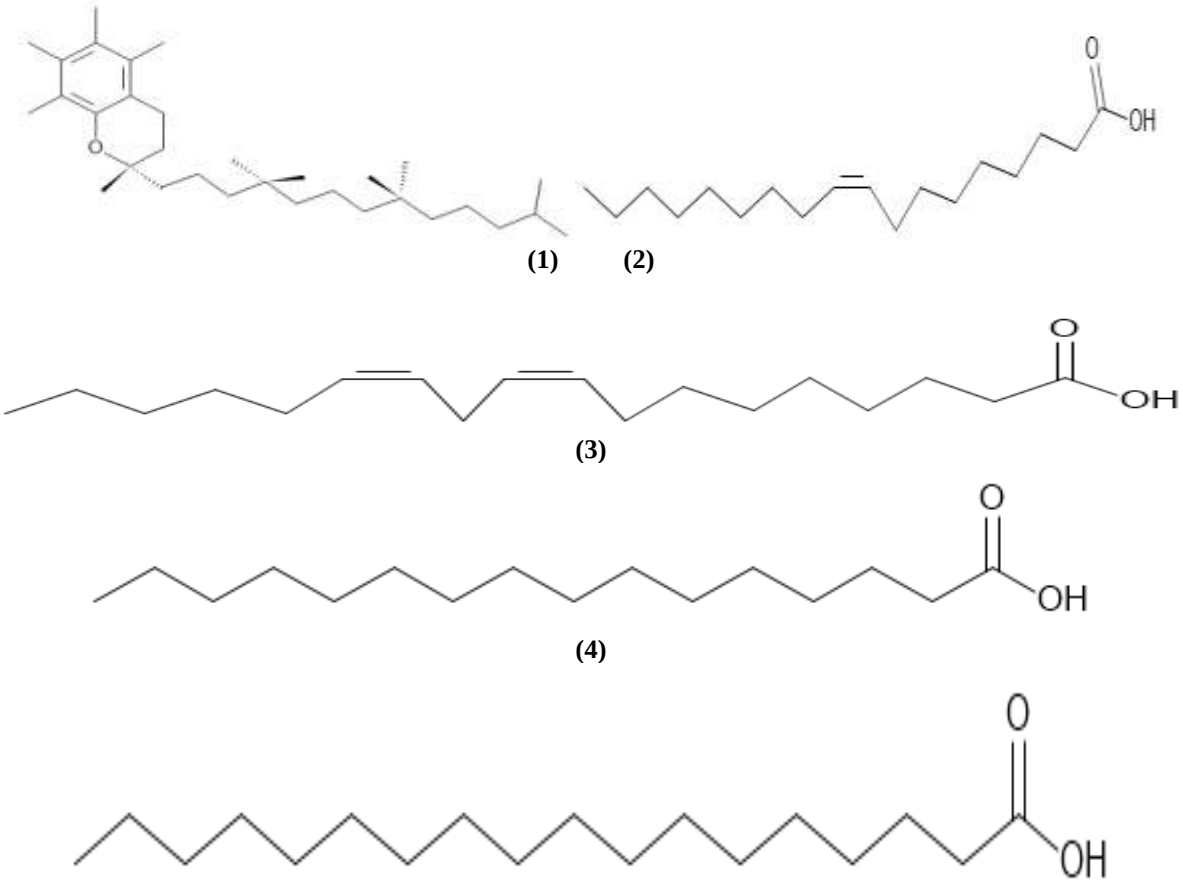
heart disease. The fatty acid composition and saponification value of cottonseed oil, like other vegetable oils, typically determine its quality. The amount of the fatty acid and saponification value together with the oil yield varies depending on the specie from which the oil is extracted, ecological conditions of the processing region and the storage conditions of the oil (Reddy and Aruna, 2009)



(1) **(2)** **(3)** **(4)** **(5)**

Fig 1. Structures of Some Fatty Acids Extracted from Cottonseed (Gossyipiumhirisutum)

When refined, cottonseed oil has a moderate flavor and a light golden hue. However, the refinement procedure determines the color's intensity. It has a comparatively high smoke point for frying. The oil had a density ranging from 0.917gcm-3 to 0.933gcm-3 and a smoke point of roughly 4500F. The amount of tochopherol (1), which helps keep the oil stable and forms compounds that extend the oil's shelf life, was high. As a result, this substance was frequently employed by producers to lengthen the shelf life of their refined cottonseed oil.

The fatty acid composition of cottonseed oil is composed of 26% saturated fatty acid and 70% unsaturated fatty acid (18% mono-unsaturated and 52% poly-unsaturated). With 95% saturated fatty acids and 2% unsaturated fatty acids (1.5% mono-unsaturated fatty acids and 0.5% poly unsaturated fatty acids), cottonseed oil's composition alters when it is fully hydrogenated (Jones and King, 1996). To get a comparable outcome with other oils, cottonseed oil does not need to be hydrogenated. Some small glands in the cotton seed produced the poisonous yellow polyphenol gossypol (6). In figure 2 below, its structure is depicted. This substance's ability to adapt promotes insect resistance. The three keys of refining, bleaching and deodorization in obtaining a finish product eliminates the gossypol level in the oil. Ferric chloride is often used to decolorize cottonseed oil (Yatsuet al., 1970)

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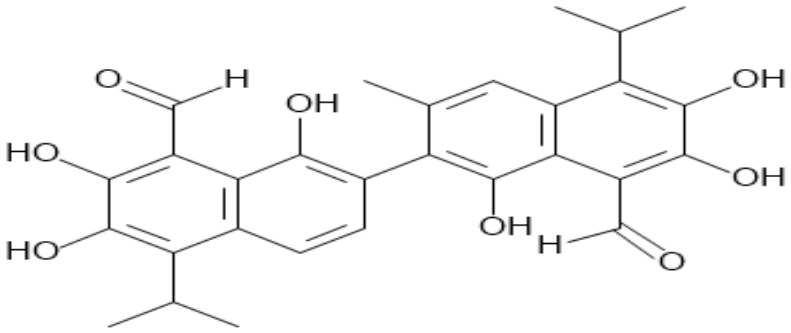

(6)

Figure 2; Structure of a Compound Found in Cottonseed Glands.

The main objectives of this study is to extract cottonseed oil from cottonseed (Gossypium hiristum), to perform a physicochemical examination on the extracted oil, and to offer scientifically reliable data on the chemical makeup of the recovered oil as a fatty acid-containing substance. The study also included the measurement of the extracted oil's acid value, free fatty acid value, saponification value, boiling point, density, pH, and other properties as well as the Fourier Transform Infrared (FT-IR) Spectrometer's detection of the functional groups present in the oil.

II. EXPERIMENTAL METHOD

The cottonseed oil used was extracted by solvent extraction adopting soxhlet as the main extraction technique and n-hexane was the solvent used in other to ensure efficient extraction. The cottonseed was obtained at FalgorenDaji,Doguwa Local Government, Kano State. All reagents used in the analysis were of analytical grade. The procedures and formulas used are bulleted below:

- **Percentage Yield**: - From the mass of the oil extracted, the percentage yield of the oil was calculated from the formula below. And was tabulated at table 1 in the next chapter

Percentage yield of oil= $\frac{\text{mass of oil obtained}}{\text{mass of ccottonseed used}} \times 100\%$..... (i)

- **Determination of Free Fatty Acid (FFA):-** 1 g of oil sample was accurately weighed with an electric weighing balance in a conical flask. To this sample, a 10 cm^3 neutralized 95% ethanol was added with addition of few drops of prepared phenolphthalein indicator. This mixture was then titrated with prepared 0.1 M NaOH solution, with constant shaking until a pink color was observed. This color lasted for 30 seconds.The percentage free fatty acid was calculated from the equation below (Admusa, 1984):

Free Fatty Acid (FFA)= $\frac{V \times M \times 2.82}{\text{weight of samle (g)}} \left(\text{mg}\frac{\text{NaOH}}{\text{g}}\right)$..... (ii)

- **Determination of Acid Number/ Value:**1 cm^3 of prepared phenolphthalein indicator was added to a mixture of 25 cm^3 diethyl ether and 25 cm^3 in a conical flask. 1 g of the oil sample was added to the mixture after neutralized by 0.1 M KOH. This mixture was then titrated with prepared 0.1 M KOH with constant shaking until a pink color was obtained. This color lased for about 115 seconds (Admusa, 1984).

The acid number/ value was calculated from the formulae below

Acid value = $\frac{(vb - va)\ X\ 5.61}{\text{weight of sample used}} \left(\text{mg}\frac{\text{KOH}}{\text{g}}\right)$ ..... (iii)

- **Determination of Saponification Value:**1 g of oil was weighed using a plastic dropping pippete in a conical flask. 25 cm^3 of prepared alcoholic potassium hydroxide solution was transferred into the conical flask. The mixture was transferred into a refluxing condenser attached to round bottom flask which was placed over a hot water bath. The mixture was stirred constantly to about an hour (Admusa, 1984).

The flask was then removed and to it, 1 cm^3 of phenolphthalein indicator was added. This mixture was then titrated while still hot with prepared 0.5M HCl (Hildith and William, 1964)

Saponification value = $\frac{(vb - va)x26.05}{\text{weight of oil}}$ ---------- (iv)

- **Density Determination:** The mass of the empty bottle was measure using the calibrated weighing balance. This mass was recorded (M_1). A measuring cylinder was used to measure a specific volume of the oil sample. This value was labelled (V). Finally, the measured oil was then transferred into the density bottle and the mass of the filled density was measured and recorded as (M_2).

The density of the oil was calculated from the formula below:-

Density = $\frac{\mathbf{M}}{\mathbf{V}}$ **(gcm^{-3})**..... ... . (v)

- **Determination of pH:**The pH was measured using a digital pH meter (Jenway model). The meter was standardized with prepared buffer solutions of known pH values i.e. pH of 4.0, 6.8, and 9.18. The electrode was then attached to the meter and was quickly dipped

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in the oil sample and measurement from the meter was taken after the readings became sable. The electrode was rinsed with a distilled water before each reading was taken. This was repeated three times and the average was taken. The average of these three readings gives the actual pH of the oil sample (APHA, 1992).

- **Determination of Boiling Point:** The boiling point of the oil was determined by putting a thermometer into a beaker containing the boiling oil. The temperature at which bubbles started appearing was recorded. This marks the boiling point of the oil.
- **FT- IR Analysis:** the sample was taken to the laboratory and subjected to FT-IR. The spectra obtained was recorded and displayed in figure 5 of this paper.

III. RESULTS AND DISCUSSION

The values obtained from the physicochemical analysis of the oil sample were summarized at table 1 below.

Table 1; Physicochemical Analysis Results

	Results	**Results Obtained from the Literature**
Acid number (mgKOH/g)	3.903	3.76(OkonkwoandOkafor,2016)
Freefatty acid (mgNaOH/g)	1.914(asoleic acid)	1.88(OkonkwoandOkafor,2016)
Saponification Value (mgKOH/g)	193.23	193.310.31(OkonkwoandOkafor,2016)
Density(gcm^{-3})	0.928	$0.917gcm^{-3}$to$0.933gcm^{-3}$ (JonesandKing,1996).
pH value	5.6at32^{0}C	
Boiling point(^{0}C)	234	232^{0}C(JonesandKing,1996).
Color	Dark-brown	Dark-brown(OkonkwoandOkafor,2016)
Yield	46.05g(18.42%)	12–25%(Mertetal.,2004)
Taste	Unpleasant	pleasant(OkonkwoandOkafor,2016)

Table 1 above showed the physicochemical analysis of cottonseed oil from the Gossypiumhiristum. The chemical properties of an oil are the most important features that determines the condition of the oil (Nzikouet al., 2009). The oil sample's physicochemical investigation revealed that it was dark-golden in color and tasted unpleasant because the oil was not refined, it had a strong hue and an awful flavor. The free fatty acid value and the acid value, which were compared with those acquired from the literature as cited from the table 1 above, were found to be 1.914 mg NaOH/g and 3.903 mg KOH/g, respectively. These low readings indicated that the oil is edible and will not go rancid over time (both oxidatively and hydrolytically) (Akubor, 2008). The cottonseed oil's saponification value was determined to be 193.23 mg KOH/g, which was within the range found in the literature. It was reported that oils with low free fatty acid value usually have high saponification value which is in accordance with the result obtained. The cottonseed oil's distinctive fatty acid is revealed by the saponification number (Aremu et al., 2006). This shows that cottonseed oil is suitable for making soap. Figure 5 below depicts the equation for the saponification reaction which is chemically known as a hydrolysis reaction. The cotton seed oil's pH was determined to be 5.6. This might be connected to the

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many pesticides used on cottonseed and other significant components found in oil kernels, like the polyphenolic molecule gossypol. When compared to the industry standard of 2320C, the boiling point was discovered to be 2340C.The degree of oil purity was indicated by how closely the obtained boiling point matched the benchmark. The oil was determined to have a density of 0.928 gcm-3. It was compared and was within the range of the usual density value of cotton seed oil from 0.917gcm-3 to 0.933gcm-3 as with most oils, it floats over water due to the less density compared to that with 1.013 gcm-3 of water (Jones and King, 1996). Furthermore, a good oil output was discovered. This could be as a result of the extraction method utilized and the extraction solvent.

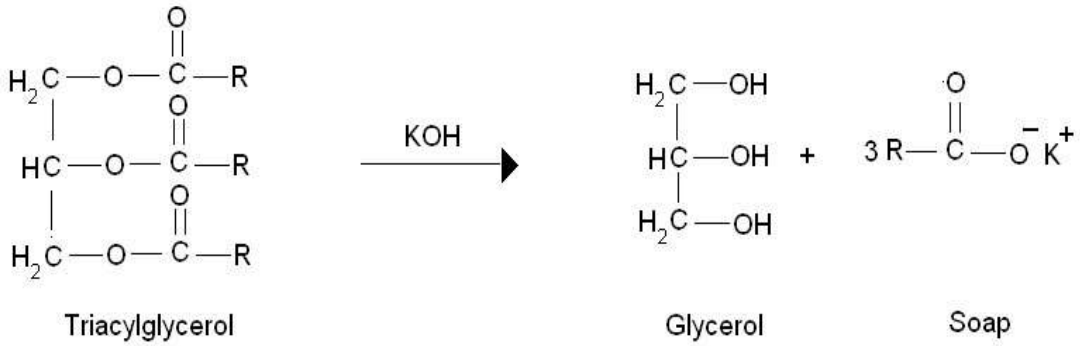
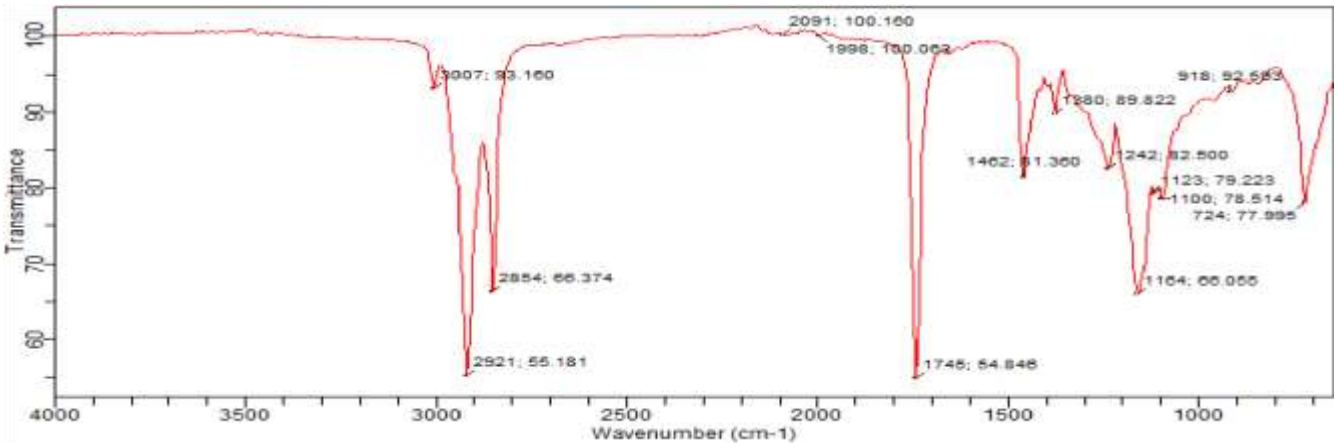


Figure 4; Soap formation from saponification reaction

Detection of Functional Groups

Although the IR-spectra do not give fully information about the structure of a compound but it provides a molecular fingerprint by showing the functional group peaks which can be used to explain the identity of the molecule present in the oil sample. Oil contains triacylglycerol (triglyceride) as the main component which is obtained from the reaction of free fatty acids in the oil with glycerol. The triacylglycerol also known as triglycerides contains an ester linkage. Hence they are esters in functional group. As shown in figure 5 below, the weak narrow band at 3007 cm^{-1} i.e. just above 3000 cm^{-1} is an indication of unsaturation in the triglyceride present (from unsaturated free fatty acid oleic acid or linoleic acid). The strong and medium band observed at 2931 cm^{-1} and 2854 cm^{-1} respectively were stretching of sp^3 –C-H bond from a methylene and methyl carbon respectively. The strong narrow band at 1745 cm^{-1} was an indication of aliphatic ester carbonyl carbon with less substitution around the carbonyl hence confirms the ester of fatty acid (triglyceride). The fingerprint region shows a medium narrow band at 1164 cm^{-1} of C-O bond of an ester. Other fragment peaks also contributes to the characterization of the molecule present in the oil. The absence of broad band around 3300 cm^{-1} was also an indication of no oxidation reaction on the oil to alcohols or organic acids. Hence the purity of the oil can be chemically guaranteed.

Figure 5; FT-IR spectrum of the extracted cottonseed oil



IV. CONCLUSION

In conclusion, the results were reliable from a scientific standpoint. Due to the oil's resistance to rancidity, the results for the acid value and free fatty acid value are favorable for the oil's edibility and storage stability. The saponification value result indicates that the oil is suitable for making soap. However, the fatty acid found in the oil gives it significant potentials in terms of its digestibility, storage ability, and appropriateness for making soap.

Its recommend that more researches on cottonseed oil should be developed to investigates more of its eminent potentials in scientific fields such as biodiesel productions and non-resistant insecticide productions. It is also recommend that more researches should be made on the pharmacological activity of the oil contents especially the chroman nucleus alpha-tocopherol otherwise called vitamin E which was widely known natural anti-oxidant

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