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PHYSICOCHEMICAL EVALUATION AND OIL YIELD ASSESSMENT OF COTTON SEEDS

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Abstract: The study aimed at extracting the oil from cotton seed and determination of the physical and chemical properties of the oil. The method used was soxhlet extraction method. The results obtained showed the following chemical compositions: Moisture content (13.55%), pH (5.01), Ash content (1.5g/100g), Heating value (39.06 MJ/kg), Acid value (10.62mgKOH/g), Iodine value (90.20gI2/100g), Cetane number. (51.6), Density (816.2kg/m3), Specific Gravity (0.915), Saponification value (190.20mgKOH/g), FFA (5.05), Viscosity (72mm2/s) and Carbon content (1.97g/100g). The economic importance of the cottonseed oil include; production of cotton seed meal, improve digestion, oil resistance to oxidation, manufacture of bio-diesel and in paint industry, cooking and salad oil sources and cottonseed meal as organic fertilizers. The physical properties studied were unit mass, unit volume, geometric mean, respectively. Information of these properties could be useful in process machine design. On the other hand, knowledge of the chemical properties is important for biodiesel production.

Keywords: Extraction; Determination; Physiochemical; Oil; Cotton Seed

1.0 INTRODUCTION

Cotton (Gossypium spp) is a warm-climate plant that can be either a shrub or a tree (Malik et al., 2020). Cotton is a perennial plant in its native environment, but for agricultural reasons, it is produced as an annual crop. Cotton bolls are classified as fruits by botanists (Negash et al., 2019). The hirsutum, barbadense, arboreum, and herbaceum species of cotton are the most important domesticated varieties of the plant for commercial use. Through the use of traditional breeding techniques, a large number of distinct varieties of these species have been established in order to produce cotton plants with enhanced agronomic features, as well as improved properties pertaining to cotton fiber and cottonseed (Mengistie et al., 2018).

Cotton seed oil is a type of cooking oil that is obtained by delinting and then decorticating the cottonseed before extraction. Cotton seed may be processed into a number of different products, the most significant of which being cottonseed oil (Aremu et al., 2015). The process of refining also eliminates any elements present with deeper coloring, leaving clear yellow oil in its place (Malik et al., 2020).

The yearly consumption of fats and oils by humans is projected to be 40 million tons throughout the globe, and this need is only growing alongside the expansion of the human population (Shah et al., 2017; Sam et al., 2022b). Because supplies are unable to keep up with demand, there is a shortfall in the availability of oils, which has led to an increase in their prices. Therefore, over the course of the years, concerted efforts have been made to find alternative sources of oils to augment the existing ones

(Udom et al., 2023). In addition, to the greatest extent possible, efforts have been made to find non-edible oil sources for non-edible industrial uses and vice versa, with the goal of reducing the amount of oil that is used for both food and non-food applications (Malik et al., 2020). However, accurate identification of the possible applications of oil extracts requires reliable information regarding the physiochemical properties of the oils. In light of these considerations, the purpose of this study was to first extract oil from cotton seeds (Gossypium spp) and then investigate the physiochemical characteristics of that oil. The goal is to produce baseline data on the oil's quality in order to provide an indication as to the types of applications that would be most appropriate for it.

Few research has been done on the physiochemical characteristics of cotton seed, despite the fact that it is common knowledge that cotton seeds contain a sizeable quantity of oil (Negash et al., 2019). As a result, it is necessary for the swaying to extract oil from this seed and also to establish its physiochemical characteristics in order to assess its culinary, nutritional, and industrial qualities. This research is of immense benefit to the agricultural sectors because it will make them aware of an alternative source of oil for the production of food as well as its physiochemical properties in order to determine their edible, nutritional, and industrial qualities.

2.0 ECONOMIC IMPORTANCE OF COTTON SEED

Shortening, margarine, liquid oils, and other processed fats and oils become key ingredients in food products that are manufactured at home, in restaurants, and by food processors, as it has been claimed that cotton's importance is crucial in many food products (Gerasimidis et. al., 2017). Despite the fact that the majority of food products that are created today were originally conceived using a shortening, margarine, or an oil product that had cottonseed oil as their base ingredient, Cottonseed oil, as opposed to other new sources of oil, is a more ubiquitous source of oil that is used for the operation of the functionality that is preferred for the majority of products. Other applications are: Production of Cottonseed meal; Improve Digestion, Oil Resistance to Oxidation; Manufacture of Bio-diesel and in Paint Industry; Cooking and salad oil sources; Cottonseed meal as organic fertilizer.

3.0 HEALTH BENEFITS OF COTTONSEED OIL

3.1 Cottonseed Oil and Cardiovascular Diseases: According to animal research, the consumption of cottonseed oil (CSO) reduces cholesterol levels; nevertheless, the effects of a CSO-rich diet on people have been evaluated in a separate study. (Konuşkan, Yilmaztekinm and Mert, 2015). It has been determined that hypercholesterolemia is primarily characterised by an increase in LDL-cholesterol levels, which is clearly associated with an increased risk of cardiovascular disease.

3.2 Anti-Allergic Response of Cottonseed Oil: Numerous research has been conducted to assess the allergic and toxicological effects of cottonseed oil. Under such conditions, CSO played a crucial part in allergy defence. In accordance with this theory, an investigation on cottonseed oil revealed an adjuvant impact on the humoral reaction, hence inducing a secondary response that favoured the delayed-type hypersensitivity response to ovalbumin. The precise selection of adjuvants can impact the epitope specificity, affinity, class, and/or isotype of an antibody response (Oluwole, Aviara, Umar, & Mohammed, 2015).

3.3 Anticancer activity of cottonseed oil: It has been demonstrated that research on unsaturated fatty acids inhibits the development of cancer cells (Abdelmoez, Abdelfatah, Tayeb, & Yoshida, 2017). While some saturated branched-chain fatty acids have been shown to have anticancer properties, more research is needed. Oleic acid is one of the most abundant fatty acids found in numerous vegetable oils, such as cottonseed oil.

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4.0 FACTORS AFFECTING OIL EXTRACTION METHOD

4.1 Extraction Time: The extraction time should ensure that oil molecules have enough time to diffuse into solvents. However, with the prolongation of extracting time, the reduction of residual oil in meal has been very slow, and the content of non-oil substances in crude oil has increased, and the processing capacity of oil extracting equipment has also decreased accordingly. In actual production, the extracting time should be shortened as far as possible, generally 90-120 minutes, under the condition that the residual oil of meal reaches the target. Under ideal oil material properties and other operating conditions, extracting time was shortened to about 60 minutes.

4.2 Material Layer Height: Material layer height has an effect on the utilization rate and extracting effect of extraction equipment. The production capacity of the same extracting equipment is improved with the increase of the material layer. The amount of meal foam in the mixture decreases, and the concentration of the mixture is also higher. However, the permeability and drip-drying performance of solvents and blends will be affected if the material layer is too high.

4.3 Solvent Oil Extraction Temperature: The solvent extraction temperature has a great influence on the oil extraction rate. With the increase of extraction temperature, the viscosity of oil and solvent decreases, and the thermal movement of molecule increases, so the extraction speed is increased. However, if the extraction temperature is too high, the amount of gasification solvents in the extractor will increase, the pressure will increase, the solvent loss in production will also increase, and the amount of non-oil substances in the extracted crude oil will increase. Generally, the extracting temperature is controlled below the initial boiling point of solvent distillation at about 5℃.

4.4 Permeability of Solvents or Oil Mixture to the Material Layers: The permeability of solvents to the material layer is expressed by the number of kilograms of solvents flowing through the billet surface per square metre per hour. According to the actual production experience, the permeability must be maintained at more than 10,000 kg/h·m2. The larger the permeability, the higher the permeation speed of solvent or mixture through the material layer, the stronger the convective diffusion effect, the smaller the thickness of the interface layer, and the greater the concentration difference between the oil inside and outside the billet, and the stronger the molecular diffusion effect, all of which are conducive to the increase of solvent oil extraction rate.

5.0 COTTON SEED OIL EXTRACTION METHODS

Cotton seed oil is a cooking oil made from cottonseed that has been delinted and decorticated. The purified cottonseed is initially run through a series of pressure rolls to create thin flakes, and then the flakes are passed via steam pressure to break the oil cells. Additionally, the flakes are compacted using hydraulic presses. In addition to hydraulic pressing, they are treated in a continual, high-pressure, screw-type expeller. There are various techniques for extracting oil from seeds. The most prevalent methods for cottonseed oil extraction as explained by (Mengistie, Alemu, & Mekonnen, 2018) are: Solvent extraction method; Mechanical Extraction Method; Soxhlet Extraction Method; Hydraulic pressing; Microwave-assisted extraction etc.

6.0 MATERIALS AND METHODS

6.1 Materials: The materials, equipment and chemical used in this research are cotton seed, N-hexane, nitric acid, phenol red indicator, hydrochloric acid (HCl), sodium hydroxide (NaOH), distilled water, sodium chloride (NaCl), ethanol, filter paper, soxhlet apparatus, measuring cylinder, round bottom flask, heating mantle, stop watch, water, hydraulic press and thermometer.

6.2. Sample Acquisition and Preparation: The cotton seed was obtained from HOTORO, KANO, NIGERIA. The seeds obtained was thoroughly cleansed in other to get rid of any agricultural residues and other contaminants.

Figure 1: Cotton seed sample

Figure 2: Sorting of cotton

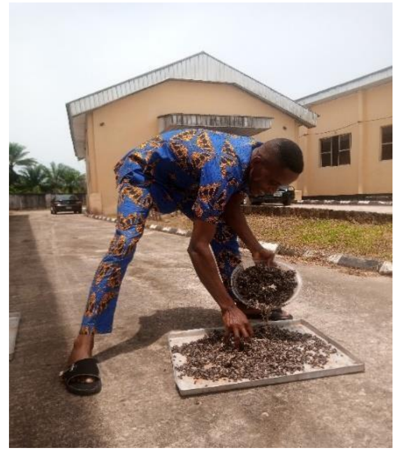

Figure 3: Cleaning of the

6.3 Extraction flow chart

The experimental flow chart for the extraction of cotton seed oil is as presented in Figure 4 below

Collection of seeds
↓
Seeds cleaning
↓
Drying
↓
Cooling
↓
Reduction of size
↓
Weighing of the crushed seeds
↓
Solvent extraction
↓
Cotton seed cake weighing
↓
Solvent recovery
↓
Crude cotton seed oil recovery

Figure 4: Flowchart showing the flow diagram of the preparation process

Figure 5: Soxhlet Extractor

Figure 6: Extraction process

6.4 Procedure for Extraction of Oil: Cleaned samples were dried in an oven in the lab at a temperature of 80°C. This was done due to the fact that a lower percentage of moisture results in a higher amount of oil being produced. Soxhlet extraction method was used, which require the use of an extraction solvent which can be used alone or paired with another solvents. To obtain optimum yield, the best solvent for a particular analyte was critically selected. It involves the process of a repeated washing (percolation) of an oil containing solid in the present of a solvent. This method involves the use of mild conditions, reduction in energy loss and an enhanced oil yield. This method stands on the hypothesis that oil in vegetables is located in the cells and is linked with other macromolecules; high yield of oil can be obtained by partial hydrolysis. At the end of the extraction, hexane was recovered using a distillation setup.

6.5 Determination of Percentage Yield of Cottonseed Oil Extracted: The oil obtained after evaporation was weighed and the oil yield calculated using the formula (Aremu et al., 2006)

Oil yield (%) = $\frac{W_o}{W_p} \times 100$

W_o = Weight of cotton seed oil extracted

W_p = Weight of grinded cotton seed

7.0 PHYSICAL CHARACTERIZATION OF KERNEL NUT

7.1 Geometric diameter: The geometric mean diameter (GMD) was calculated as

GMD = $(abc)^{1/3}$

7.2 Sphericity: The sphericity f, was calculated using the equation prescribed by O'Brien and Wakelyn, (2015) as:

f = $\frac{(abc)^{1/3}}{a}$

7.3 Unit Volume: The unit volume of 20 individual seeds was determined from values of a, b, and c using the formula proposed by Mittelbach (1994):

V = $\frac{abc}{\pi}$

7.4 Projected Area: The projected area was calculated using the equation proposed by Oderinde et al. (2009) as:

$A_p = KV^{\frac{2}{3}}$

Where,

A_p = average projected area (mm^2)

K = constant, 1.21

V = projected volume (mm3)

8.0 CHARACTERIZATION OF EXTRACTED COTTONSEED OIL

According to the procedures outlined by the Association of Official Analytical Chemists, the physical, chemical, and proximate components of the oil was analyzed in order to make a determination (AOAC, 2004).

8.1 Determination of moisture content: A known weight of fresh cotton seed was dried at 8 hours for 60 °C. The sample was cooled and reweighed. The percentage of moisture was calculated as shown (Sam et al., 2022a) below:

%moisture content = $\frac{100(M_1 - M_2)}{M_1}$

Where: M_1 is the weight of sample before drying

M_2 is the weight of sample after drying

8.2 Determination of Density: The density of cotton seed oil was determined using a density bottle. The bottle was first washed, cleaned and weighed. The bottle was filled with distilled water at room temperature and weighted. The distilled water was discarded and the bottle dried, the citrus peel oil was poured into the bottle and the weight was taken.

8.3 Specific Gravity: The specific gravity was determined using a specific gravity bottle. The dried empty specific gravity bottle was weighed and recorded. Distilled water was poured into the specific gravity bottle and weighed. The specific gravity bottle was emptied, dried, filled with cotton seed oil and weighed. The density of water was calculated and the specific gravity of the oil was obtained by dividing the density of the oil by the density of water.

8.4 Determination of Viscosity: The viscosity of cotton seed oil is a measure of the fluid's resistance to flow (Usoh et al., 2023). This was determined using an NDJ-5S viscometer.

8.5 Determination of Acid Value: 50ml of ethanol and 1ml of phenolphthalein was mixed, 10g of cotton seed oil was added to the mixture and stirred. The mixture was heated and stirred till a clear mixture was obtained. 0.1M of NaOH was prepared and titrated against the oil mixture until a pink color appeared.

Acid value = $\frac{V \times N \times 40}{W_s}$

Where: V is the volume of NaOH required to neutralize the oil solution

N is the normality of NaOH

Ws is the weight if the oil sample to be use

8.6 Determination of Free Fatty Acid: In determining the free fatty acid value was determined by dividing it by 1.42 which is a standard value when using NaOH.

8.7 Determination of Peroxide Value: This was determined using the equation;

Peroxide value = $\frac{V \times N \times 1000}{W_s}$

Where V is the volume of sodium thiosulphate required to neutralize the oil mixture.

N is the normality of sodium thiosulphate

Ws is the weight of the oil sample to be use

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8.8 Determination of Saponification Value: 0.1M of hydrochloric acid was prepared. 2.2g of oil added to 25ml of potassium hydroxide and heated in a water bath for 30minutes for proper mixing, 3 drops of phenolphthalein was added to the oil mixture. The acid was titrated against the oil mixture till it became colorless.

$$saponification\ value = \frac{56.1 \times V \times N}{W_s}$$

Where: V is the volume of HCL required to neutralize the oil solution
N is the normality of the acid
Ws is the weight of the sample

8.9 Ash Content: The ash content was determined.

$$\%\ ash = \frac{W2 - W1}{W} \times 100$$

Where: W_2 - Final weight of dish and ash,
W_1- weight of dish
W- weight of pectin sample

9.0 RESULTS AND DISCUSSION

9.1 Physical Properties of Cotton Seed

Table 1: Physical Properties of Cotton Seed

S/N	Major Diameter (mm)	Intermediate diameter (mm)	Minor diameter (mm)	GMD (mm)	AMD (mm)	Sphericity	Unit volume (m^3)	Projected area (m^2)
1	10.00	0.70	0.40	1.40	3.7	0.14	0.89	1.11
2	0.80	0.50	0.20	0.43	0.5	0.53	0.03	0.10
3	0.90	0.70	0.40	0.63	0.67	0.70	0.08	0.22
4	0.80	0.60	0.30	0.52	0.57	0.66	0.05	0.15
5	10.00	0.70	0.40	1.41	3.70	0.14	0.89	1.11
6	0.70	0.50	0.20	0.41	0.47	0.59	0.02	0.09
7	0.90	0.50	0.40	0.56	0.60	0.63	0.06	0.18
8	0.90	0.70	0.40	0.63	0.67	0.70	0.08	0.22
9	10.00	0.60	0.30	1.22	3.63	0.12	0.57	0.83
10	0.80	0.50	0.20	0.43	0.50	0.54	0.03	0.10
MEAN	3.58	0.6	0.32	0.77	1.50	0.48	0.27	0.41
SD	4.43	0.09	0.09	0.41	1.50	0.24	0.37	0.42

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As shown in the Table 1, the average major, intermediate and minor diameters of cotton seeds are 3.58, 0.6 and 0.32 mm, respectively. The geometric mean diameter was found to be 0.32 mm. These values are relevant to the design of sorters, screens and containers. An evaluation of average sphericity was found to be 1.50. This value is higher compare to the values reported by Nkafamiya, et al. (2007) for sumac fruits (0.76). The high sphericity value of cotton obtained is important in the selection of handling principles such as conveying and grading. These properties can be equally utilized in the containerization of this crop. Thus, information herein provided is essential for the design of equipment, for handling, transport; process and storage of the seeds.

9.2 Proximate Composition of Cotton Seed

Table 2: Proximate Compositions of Experimental Cotton Seed Oil and Diesel

S/N	Parameters	Cottonseed oil	Diesel (ASTM)
1	pH	5.01	5.60
2	Ash content (g/100g)	1.50	0.08
3	Heating value (MJ/kg)	39.06	41.5
4	Acid value (mgKOH/g)	10.62	2.00
5	Iodine (gI_2/100g)	90.20	88.00
6	Cetane	51.60	42.00
7	Density (kg/m^3)	816.20	861.00
8	Specific gravity	0.915	0.82
9	Saponification (mgKH/g)	190.20	140.00
10	FFA mg/g	5.05	0.90
11	Viscosity mm^2/s	72	3.80

9.2.1 Percentage oil yield: The oil yield content of n-hexane extract of cotton seed was found to be 66.78%. This result was a little higher than oil yield of petroleum ether extract of cotton seed reported by Zerihun (2018).

9.2.2 Moisture content: The moisture content of the extracted cottonseed oil was found to be 13.55%. This value is relatively high compared to the result obtained by Özarslan, (2002) on cotton seed. This suggests that the cottonseed oil may have been extracted using methods that did not completely remove the water content. This may also depends upon the maturity and quality of seeds. The moisture contents of seed determine the ability of all seeds to be stored well (Zerihun et al., 2018)

9.2.3 Relative density: The relative density obtained for the cotton seed oil (0.816 g/cm^3) was slightly in agreement with the specified value reported (ASTMD445), which range from 0.860 to 0.90 for biodiesel.

9.2.4 Acid value: The acid value of cottonseed oil is a measure of the free fatty acid content present in the oil. The acid value of the cottonseed oil in this study was determined to be 10.62 mg KOH/g. This high value indicates that the oil contains a relatively high amount of free fatty acids, which may be due to the presence of impurities or extraction techniques. High acid value shows that the oil may not be edible but may be useful in the production of paints, liquid soap, and shampoos. The high acid value of cottonseed oil also suggests high levels of hydrolytic and lipolytic activities in the oil, making it a better candidate for conversion to biodiesel using the two-stage processes of esterification and trans-esterification to reduce the formation of soap.

9.2.5 Iodine: The iodine value is an important measure of the degree of unsaturation of the fatty acids in the oil. In the case of cottonseed oil, the iodine value is 90.20 gI/100g, which indicates that the oil is relatively high in unsaturated fatty acids. Oils with high iodine values are generally more reactive and prone to oxidation, which can affect their stability and shelf life. However, the high iodine value of cottonseed oil also makes it suitable for certain applications such as in the production of paints and varnishes. According to Aremu et al. (2006), oils with iodine values less than 100 gI2/100g of oil are non-drying oils. Oils with such characteristic may, therefore, be useful as raw materials in the manufacture of vegetable oilbased ice cream (Oderinde, 2009).

9.2.6 Saponification: The saponification value of cottonseed oil was found to be 190.20 mgKOH/g, indicating a relatively high molecular weight of the fatty acids in the oil. This value is important in determining the potential applications of the oil, as oils with high saponification values are generally used in the production of soaps, detergents, and other cleaning products. Comparing the saponification values of cottonseed oil with those of other oils, the results indicate that the saponification values are higher than those of Persea americana oil (35.76 mgKOH/g) (Adaramola et al., 2016), argan oil (190.88 mgKOH/g) (Gecgel et al., 2015), and olive oil (97.94 mgKOH/g) (Borchani, 2010).

9.2.7 pH: The pH of the cottonseed oil was determined to be 5.01. The pH of cottonseed oil is generally acidic, which is expected since oils are generally acidic due to the presence of free fatty acids. This can influence its stability and suitability for different applications. For example, acidic oils are more prone to oxidation, which can lead to rancidity and a shorter shelf life.

9.2.8 Ash content: The ash content of the cottonseed oil was found to be 1.5 g/100g. In this case, the ash content can be attributed to the presence of minerals and other inorganic compounds that are present in the cottonseed. The ash content of an oil can influences its nutritional value and suitability for different applications. For example, oils with high ash content may not be suitable for use in certain food applications due to their impact on taste and texture. (Esuoso 1998).

9.2.9 Heating value: The heating value of the cottonseed oil was determined to be 39.06 MJ/kg. Heating value is a measure of the energy content of a substance, typically expressed in units of energy per unit mass (Edet et al., 2022). The heating value of cottonseed oil indicates its potential as a fuel source, with higher heating values being desirable for this application.

The heating value of cotton seed biodiesels (39.06 MJ/Kg) was comparable to those of soybean oil biodiesel (38.1 MJ/Kg) (Rashid et al., 2009), but greater than neem seed oil biodiesel (35.2 MJ/Kg) (Sekhar et al., 2009). The heating value is an important parameter for estimating fuel consumption; the greater the heat of combustion, the lesser the fuel consumption (Knothe et al., 2008). The lower values of heating value of cotton seed biodiesel, and conventional biodiesels in general, when compared to that of petro diesel might be due to higher oxygen content and lower carbon-to-hydrogen ratio in the former than in the later.

9.2.10 Cetane number: The cetane number of cotton seed oil was determined to be 51.60, which is a measure of its ignition quality as a potential biofuel. A higher cetane number indicates better ignition quality and good cold start properties, which can minimize the formation of white smoke. While cottonseed oil is not typically used as a diesel fuel, its high cetane number of 51.60 indicates its potential as a biofuel, as it is well above the minimum value of the international biodiesel fuel standards ASTM D6751 (40 minimum) and EN 14214 (51 minimum).

9.2.11 Free fatty acid: The free fatty acid content of the cottonseed oil was determined to be 5.05mg/g. Free fatty acids are a measure of the amount of unbound fatty acids present in the oil, which can influence its quality and stability. High levels of free fatty acids can indicate that the oil has undergone hydrolysis or other forms of degradation, which can affect its nutritional value and suitability for different applications.

9.2.12 Viscosity: The viscosity of the cottonseed oil was found to be 72mm²/s. Viscosity is a measure of a fluid's resistance to flow, with higher viscosity indicating greater resistance. The viscosity of an oil can influences its suitability for different applications, such as in the production of lubricants or as a fuel source. Oils with high viscosity may be more suitable for use as lubricants, while oils with lower viscosity may be more suitable for use as fuel.

The kinematic viscosity of cotton seed oil is above the limits of Thailand ASTM (1.9-8.0 mm²/s) fuel standard. This value was found to be greater when compared to that of neem oil (44.00 mm²/s) (Sekhar et al., 2009) and coconut oil (43.30 mm²/s) (Alamu et al., 2010), but higher than that of Jatropha curcas oil (17.00 mm²/s).

10.0 CONCLUSION

The major, minor, geometric mean diameter and sphericity were observed to be 3.58 mm, 0.60 mm, 0.32 mm, 0.77 mm, and 0.48 mm, respectively. These properties provided is essential for the design of equipment, handling, transport; process and storage of the seeds.

The study further analyzed the extraction and characterization of oil from cottonseed. The percentage oil yield of the n-hexane extract was found to be 66.78%, which is higher than other oil-yielding seeds like linseed and soybean. The moisture content of the extracted cottonseed oil was 13.55%, which is relatively high. The proximate and other physiochemical properties of the cotton seed oil were analyzed.

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