

6<sup>th</sup>

# Annual Engineering Conference

School of Engineering and Engineering Technology  
Federal University of Technology, Minna.



## Book of Proceedings

**Theme:**

**Engineering and Technology for Wealth  
Creation in the Spirit of NEEDS**

**Date: 15th - 17th June 2005**

Edited by:

**Dr. O. K. Abubakre, Dr. E. N. Onwuka and Dr. B. A. Alabadan**

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**School of Engineering And Engineering Technology,  
Federal University of Technology  
Minna**

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15<sup>th</sup> – 17<sup>th</sup> June 2005 at the New Lecture Hall-1  
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**Theme: Engineering and Technology for Wealth Creation in the Spirit of  
NEEDS**

**Members of the Local Organizing Committee**

*Engr. Dr O.K. Abubakre*  
*Mr. P.N. Ndoke*  
*Dr. E.N. Omwuka*  
*Engr. Dr. B.A. Alabandan*  
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## Preface

The theme of this year's conference, the sixth in the series of the Annual Engineering Conference of the School of Engineering and Engineering Technology, was chosen to be **Engineering and Technology for Wealth Creation in the Spirit of NEEDS**. This theme was chosen in tune with government's policy on economic empowerment. It is envisaged that solutions to the Nigeria's national economic problem will **better be addressed** by Nigerians themselves. It is hoped that the few selected papers in this edition of the **Book of Proceedings** will go a long way in motivating individuals and groups to **improved-economy oriented ventures** that will **serve as the foundation stone** to industrializing the whole nation.

The sub themes of the conference were also chosen with the same objective in mind. The various sub-themes addressed by the various papers presented at the conference are:

1. Produce, Processing and Storage in Agric and Agro-allied Industry
2. Quality Control, Pollution and Environmental Management.
3. Research and Development in Rural Infrastructure
4. Advances in Information Technology.
5. Power Systems Theory and Application.
6. Development of Raw Materials for Economic Empowerment
7. Engineering Education and Training
8. Product and Equipment development for SME's
9. Partnership between Research Institutions and Industry.

A total of seventy (70) technical papers were received on the 9 sub-themes of the conference. The papers received were subjected to peer review and only 38 of them were considered fit for inclusion in this Book of Proceeding. It is the hope of the Local Organizing committee that the various papers published in this book will be of immense contribution to the academic community, the engineering practice in both the public and private sector and to those interested in becoming Job Creators. It is with this in mind that we present this Book of Proceedings of the 6<sup>th</sup> Engineering Conference of the School of Engineering and Engineering Technology of the Federal University of Technology, Minna for your intellectual enrichment and economic prosperity.

*Engr. Dr. O.K. Abubakar*  
(Chairman LOC)

## Acknowledgement

The LOC members express profound appreciation to the Management of the Federal University of Technology, Minna and to the Dean, school of Engineering and Engineering Technology for the moral, financial and material support for the conference. We are also grateful to all the staff of the school in particular and the University Community in general for the wonderful support enjoyed prior to, during and after the conference. We specially thank our reviewers, our donors and all the participants in the conference. Specific mention should be made of the well-wishers of FUT, Minna who made financial commitment to ensure the success of the conference. Such individual and corporate bodies are listed below:

1. An- Nasiha Construction Company, Minna
2. Intercity Bank, PLC Minna
3. PHCN (Shiroro – Generation) Minna
4. PHCN (Hydro Business Unit) Jebba
5. NEPA – Transysco Shiroro, Minna
6. The Controller of Works, FMW, Minna
7. S. E. E. T. FUT, Minna
8. Agric Engineering Department F.U.T. Minna
9. Civil Engineering Department F.U.T. Minna
10. Electrical Engineering Department F.U.T. Minna
11. Chemical Engineering Department F.U.T. Minna
12. Mechanical Engineering Department F.U.T Minna
13. PHCN Minna District
14. Niger State Water Board, Minna
15. Niger State R.E.B., Minna
16. Permanent Secretary, M.O.W., Niger State, Minna
17. Ministry of Transport, Niger State, Minna
18. Engr. A.A. Aliyu, M.O.W., Niger State, Minna
19. Yandalu Bello, M.O.W., Niger State, Minna.



The 6<sup>th</sup> Annual Engineering Conference

**Address Presented by the Vice-Chancellor, Professor Tukur Sa'ad, on the  
Occasion of the Opening Ceremony of the 6<sup>th</sup> Annual Engineering  
Conference, Held in the School of Engineering and Engineering  
Technology, Federal University of Technology, Minna.**

**The Special Guest of Honour,**

**The Chairman,**

**The Guest Speaker,**

**Deputy Vice-Chancellor,**

**Principal Officers of the University,**

**Dean, School of Engineering and Engineering Technology,**

**Deans of Schools & Heads of Departments,**

**Members of Senate Present,**

**Invited Guests,**

**Gentlemen of Press,**

**Ladies and Gentlemen**

I wish, on behalf of the Governing Council of the Federal University of Technology, Minna, the Senate of the University, and the University Community at large, to welcome you to yet another episode of the famous Annual Engineering Conference, which is about the only gathering of its kind in the whole of northern region of Nigeria. It makes me proud to note that this exceptional regular gathering of the technically minded academics is being organized by the School of Engineering and Engineering Technology of our own Institution.

This Annual Conference has been growing from strength to strength, calling the attention of researchers to some practical needs of our developing economy through carefully selected conference themes. Such themes draw from the fertile minds of the intellectuals, bringing out solutions that could enrich our society. This year's theme "Engineering and Technology for Wealth Creation in the Spirit of NEEDS," is our modest contribution to the government's program for the economic empowerment.

It is befitting that such technical conference is being organized and hosted by the School of Engineering of the Federal University of Technology Minna, as it is in keeping with the enabling law establishing the university, which requires it to produce self-reliant graduates who will become wealth creators and employers.

The current administration has, amidst several difficulties, succeeded in moving the University to its Permanent Site. This marks a new and important epoch in the life of this Institution. The Bosso campus has been reasonably decongested, however, there is still a lot of work pending in the Permanent Site. But we shall gradually and systematically scale all the hurdles that lie on our path to bringing this great institution to the height where it belongs. Hopefully by the end of this year, or latest by the first quarter of next year, the movement to Permanent Site will be completed with the movement of the Host School of this conference to the Permanent Site. In other words, the next edition of this conference, God willing, would take place in Gidan Kwano.

We are mindful of the fact that the University is still grossly understaffed, with the academic staff carrying such workload that leaves little or no time for meaningful academic research. However, we are not relenting on our efforts to recruit more qualified hands. Interviews for new academic staff will resume this month; we shall soon, therefore, be in a position to recruit more hands. Our goal remains to continuously strive to create a research environment that enables collaboration with the private sectors both within and outside the country.

We are looking forward to the day when this University would be distinguished by its research achievements. It shall be a great honour. Therefore, let each and every one of us work towards this great day by helping to build the enabling environment and, training our students to be innovation minded.

May I congratulate the Dean and the entire staff of School of Engineering and Engineering Technology for yet another successful Engineering Conference. At the same time, I wish you successful deliberations.

## Welcome Address by the Host, Engr. Prof. F.O. AKINBODE, Dean, School of Engineering and Engineering Technology, Federal University of Technology, Minna.

The Special Guest of Honour.

The Chairman.

The Chief Host, Prof. H.T. Sa'ad, V.C., Federal Univ. of Tech., Minna.

The D.V.C. Federal Univ. of Tech., Minna, Dr. T.Z. Adama

Principal Officers of the University.

Deans of Schools & Heads of Departments.

Invited Guests.

Distinguished Participants.

Chairman and Members of LOC.

Students.

Ladies and Gentlemen.

I am happy to welcome you all again on behalf of the School of Engineering and Engineering Technology, Federal University of Technology Minna, to yet another event of our Annual Engineering Conference. We thank the Special Guest of Honour for sparing time from his busy schedule to come and grace this annual technical event. We also thank the Chief Host, Prof. H.T. Sa'ad, for his moral support.

A renowned University is usually distinguished by its research strength. The research results are the building bricks for today's economy both in the developed and developing countries of the world. In spite of our poor facilities and low staff strength, we are determined to make our environment research oriented. We are strongly convinced that it is through meaningful research that we can create wealth and eradicate poverty from our society. Hence the Annual Engineering Conference chooses its themes carefully. In consciousness of the governmental efforts to eradicate poverty, we have chosen this year's theme to be "Engineering and Technology for Wealth Creation in the Spirit of NEEDS," thus seizing the opportunity of the conference to direct the minds of researchers to bring out diverse solutions for creating wealth from out of our richly endowed environment.

It is our hope that the Engineering Conference shall continue to feature year-by-year, and that its quality shall continue to improve. The research awareness among our colleagues has greatly improved from what it was about six years ago, thanks to our Annual Conference. As we improve ourselves through research efforts, so shall the quality of what we feed to our students equally improve. The conference is beneficial in many ways.

I wish the participants fruitful deliberations and a nice stay in Minna. Once again, welcome and God bless.

# Production and Characterization of Biodiesel from Cotton Seed Oil.

M. Alhassan and A.G. Isah

Department of Chemical Engineering  
Federal University of Technology, Minna, Nigeria  
[mohakusi2003@yahoo.com](mailto:mohakusi2003@yahoo.com)

M.U. Garba

Department of Chemical Engineering  
University of Maiduguri  
Maiduguri, Nigeria

**Abstract-** This paper present result of the research work conducted on the production of biodiesel by transesterification of cottonseed oil (new and waste) with alcohol in the presence of a catalyst (NaOH), with the aim of providing an alternative to fossil fuel which have adverse effect on the environment. To achieve these; batch transesterification process was used, which involved pre-treatment of the cottonseed oil, conversion of the oil into biodiesel, purification of the biodiesel and then its characterization. Using methanol as the alcohol and reaction temperature of 65°C gave the optimum yield of biodiesel with valuable co-product (glycerine), 99.0% and 14.67% for new oil, and 98.33% and 14.0 for waste oil respectively. The analysis of the biodiesel produced from the new oil, shows that it has the following characteristics; specific gravity of 0.859, flash point of 115°C, pour point of -5°C, Cetane index of 50.58, kinematic viscosity of 5.7cst, and sulphur content of 0.0002 %mass. While that obtained from the waste oil are; specific gravity of 0.86, kinematic viscosity of 6.53 cST, flash point of 117°C, pour point of -2°C, Cetane index of 51.22 and sulphur content of 0.0005%mass. These values fall within the range of the standard specifications for diesel fuel (ASTM and National Soy diesel Development Board), and indicate that the biodiesel produced is essentially sulphur free, has high cetane number (less knocking) higher lubricating effect than petrodiesel, and will not cause corrosion of pipes, and can effectively serve as an alternative to petrol diesel. Hence base catalyze trans-esterification method is an effective method for biodiesel production.

## I. INTRODUCTION

Diesel oil is a fuel made from petroleum. It is made up of hydrocarbons boiling in the range of 250 – 350°C and incorporates hydrocarbon molecules containing 11 - 16 carbon atoms. Its uses as a fuel, covers fuel for diesel engine, domestic and industrial heating and fuel oil blending.

A variety of ester-based oxygenated fuels derived from natural, renewable biological sources such as vegetable oils are called “biodiesel” because they possess properties (both physical and chemical) and engine performance that are very close to those of petrol diesel. The process of converting vegetable oil into biodiesel fuel is new and called trans-esterification. Trans-esterification is a chemical reaction whereby an alcohol (usually methanol or ethanol) is used in the presence of a catalyst, such as sodium hydroxide or potassium hydroxide, to chemically break the molecule of the raw renewable oil into methyl or ethyl ester (biodiesel), of the renewable oil with glycerol as a co-product.

Trans-esterification is not a new process; it was conducted as early as 1853, by scientist E. Duffy and J. Patrick. One of the first uses of trans-esterified vegetable oil was for powering heavy-duty vehicles in South Africa before World War II (Roger *et al*, 2003). A successful trans-esterification reaction is signified by the separation

of the ester and glycerol layers after the reaction time. The heavier co-product; glycerol settles out and may be purified for use in pharmaceutical, cosmetics and soap industries. Conversion of cottonseed oil to methyl or ethyl esters (biodiesel) is an example of many chemical processes available to improve biofuel availability in the world.

About 15-20% of the embryo of cottonseed is oil, which is extracted by squeezing and rolling. The oil may be used directly in diesel engines with adjustment to the injection system, but toxic fumes are produced and deposits occurred, which lead to serious engine problems (University of Idaho, 1994). This is the reason why the oil needs to be converted into an alkyl ester. The esters produced are better engine fuel than the crude oil and the glycerol is a valuable co-product. The energy density of biodiesel is about 38 MJ/Kg while that of petrol diesel is 47 MJ/Kg.

Series of research have been carried out on the use and conversion of vegetable oil into biofuel. Roger *et al*, 2003 carried out experiments on rape seed and jojoba oil as fuel for diesel engines. Peterson *et al*, 1983 and Peterson *et al*, 1991 carried out experiment on vegetable oil expression into a high-grade biodiesel fuel, and batch type trans-esterification process for winter rape oil. But it was observed that little has been done so far on the conversion of cottonseed oil into biodiesel.

Virgin/refined cottonseed oil was procured from Sun seed Nig. Ltd. Zaria, followed by conversion of the cottonseed oil (new and used) into biodiesel using the base-catalyzed trans-esterification method, which studies have shown to be the cheapest with very high efficiency. Sodium hydroxide was the base-catalyst used. In addition, the excess alcohol in the products was recovered by distillation and further purification of the products was carried out.

## II. LITERATURE REVIEW

### A. Historical Development of Biodiesel

Cotton (*Gossypium hirsylium*), grows as a perennial crop in the tropics and can attain the size of a small tree. The seeds contain oil and other by-products for use in the manufacture of edible and industrial products. The seeds are oblong in shape, pointed toward one end and large from one quarter to almost one half of an inch of length. The seed coat is dark brown or black. The embryo makes up almost the entire kernel and consists of the cotyledons or seed leaves. Throughout the cotyledons are numerous dark spots that are oil glands or rosin cavity. Until 1900, cottonseed oil was considered essentially inedible because it contained, among other things, gossypol, a bitter pigment produced in glands located throughout the cotyledons (Beryl, Simpson and Molly, 1985 and Weiss, 1983).

Three basic routes to biodiesel production from oils and fats are;

- Base catalyzed transesterification of biolipid (biological oils and fats). This yields about 98% conversion at low temperatures and pressure.

- Direct acid catalyzed trans-esterification of the biolipid

- Conversion of the biolipid into its fatty acids and then to biodiesel.

The reaction between the fat or oil and the alcohol is a reversible reaction and so the alcohol must be added in excess to drive the reaction towards the right and ensure complete conversion (University of Idaho, 1994).

### B. Trans-Esterification Reaction

The alcohol/catalyst mixture is charged into a closed reaction vessel and the oil or fat is added. The reaction mixture is kept just below or above the boiling point of the alcohol (around 65°C for methanol and 78°C for ethanol) to speed up the reaction. Recommended reaction time varies from 1-5 hours (Peterson *et al*, 1991), and some authors recommend the reaction to take place at

room temperature (Noureddini and Zhan, 1997) Excess alcohol is normally used to ensure total conversion of the fat or oil to its esters. Care must be taken to monitor the amount of water and free fatty acids in the incoming oil or fat. If the free fatty acid level or water level is too high it may cause problems with soap formation and the separation of the glycerin. Acid-catalyzed and base-catalyzed trans-esterification reactions also known as acidolysis and alcoholysis are explained by different mechanisms (Noureddini and Zhan, 1997). With base catalyzed reactions, the basicity of the oxygen of the alcohol appears to be increased so that it attacks the carbonyl carbon directly. Lower activation energies are observed in base catalyzed reactions than in acid catalyzed ones (Noureddini and Zhan, 1997).

#### C. Catalysts Used

The catalyst used in trans-esterification of vegetable oil or animal fats and oils is sodium hydroxide (NaOH; Caustic soda), or potassium hydroxide (KOH). These alkalis are hygroscopic, i.e. they absorb water from the atmosphere. So fresh lye (NaOH or KOH) must be obtained and containers kept tightly sealed always. After weighing, the lye is added to the methanol as quickly as possible. These alkalis (NaOH and KOH) absorb carbon (VI) oxide from the atmosphere and become carbonated if not stored properly. Fresh lye is almost translucent. Carbonated lye can still be used, but with an added 25% of the calculated amount of lye needed. NaOH is cheaper and more readily available than potassium hydroxide (KOH). It usually comes in grades, flakes and 5mm pearls or half-pearls are 96-97% pure, small pearls (1-2mm) are 99% pure, but more expensive. Ones with lower percentage purity will not work for biodiesel. KOH is not as strong as NaOH; anhydrous grade flake purity is usually about 92%, while half-pearl 85%. It requires 3.5g of NaOH or 1.40253g of KOH per liter of oil to transesterify new oil (virgin, uncooked). Used oil (waste

vegetable oil, WVO) required more bases than new oil to catalyzed the neutralization of the free fatty acids (FFA'S) formed in the cooking oil, which can slow or stop the trans-esterification process. Titration is carried out to determine the FFA content and how much bases will be required. The extra base act as a neutralizer and convert the FFA's to soap, which drops out with the glycerin layer (Noureddini and Zhan 1997).

#### D. Quantity of Methanol/Ethanol Required

The stoichiometric quantity of methanol is the amount needed to convert triglycerides (fats and oils) into methyl esters (biodiesel); it provides the "methyl" portion of the methyl esters. An excess of methanol is needed to push the conversion process (reaction) towards completion. Without the excess methanol, the ester and the glycerol react to provide an equilibrium mixture which is less than desirable and the process runs out (reach equilibrium) before all the triglycerides are converted to esters, resulting in to poor fuel that doesn't combust well and can be corrosive. The excess methanol acts more like a catalyst. It encourages the process but does not become part of the final product and can be recovered afterwards. Fats and vegetable oils have been found to require different stoichiometric amount of methanol for their trans-esterification, typical value ranges from 11-13% by volume of oil. This depends on the type of fatty acid residues present in the fat or oil. However, excess has been found to be between 60% and 100% of the stoichiometric amount, so as said above if the stoichiometric amount of methanol to be used is 12.6%, therefore for 126 ml per litre of oil, the excess would range between 76 ml and 126 ml bringing the total amount of methanol to be used as 200-252 ml per litre of oil

For ethanol, a relationship developed by Peterson C.L. et al (1983) was used. It is given as

$\text{EtOH} = 0.2738 \times \text{oil}$ ,  $\text{KOH} = \text{oil}/85$  Where  $\text{EtOH}$  = amount of ethanol in litre, oil = desired amount of oil in litre. The excess amount of ethanol required has been taken care of in this relationship.

### III. EXPERIMENTAL METHODOLOGY

Standard methods of testing and characterization of hydrocarbons were used as recommended by ASTM (Annual book of ASTM, 1987, University of Idaho, 19994), and the procedures followed accordingly.

#### A. Production Process

500 ml of cottonseed oil was poured into a beaker and warmed gently to a temperature of 120°C (Make your own biodiesel 2 n.d); 100 ml of it was taken and mix with measured with catalyst/alcohol mixture containing 0.35g of sodium hydroxide pellets and 20ml of methanol which was thoroughly mixed to ensure complete dissolution of the pellets and was then transferred into biodiesel reactor (1l conical flask) stopper, and stirred vigorously for 1, to 3 hours using a magnetic stirrer for different runs. After the reaction time, the mixture was then centrifuged; producing two phases, methyl ester and crude glycerol. Crude glycerol, the heavier brownish liquid collected at the bottom while clear amber yellow methyl ester (biodiesel) was withdrawn (decanted) from the top. Each run of the trans-esterification reaction was carried out at room temperature and at 65°C. The same procedure was repeated using 27.38ml of ethanol at room temperature and at 78 °C for both new and waste oil. The basis of chosen this temperatures bellow the boiling point of the respective alcohols was not to allow the evaporation of the alcohols involved and to obtained optimum yield of Biodiesel since reaction tend to proceed faster at higher temperatures.

#### B. Pretreatment of Waste Oil

Solid bodies were filtered off using a mesh filter of 5µm. The oil was washed with hot water in a separating

funnel. It was allowed to settle for 1 hour and decanted. The oil was then heated again and allowed to settle for some minutes and decanted for the second time.

#### C. Titration

One gram of NaOH was dissolved in 1l of distilled water. In a 250ml beaker, 1ml of the dewatered used cottonseed oil was dissolved in 10ml pure isopropyl alcohol. The beaker was warmed gently and stirred until all the oil dissolved and the mixture turned clear. Two drops of phenolphthalein solution was then added. Using a graduated syringe, 0.1% NaOH solution was added drop by drop to the oil-alcohol-phenolphthalein mixture, stirring all the time until the solution turns magenta for some seconds. The number of millimeters of 0.1% sodium hydroxide solution used was recorded.

### IV. CHARACTERIZATION OF BIODIESEL FUEL PRODUCED

The analysis of the resulting biodiesel was carried out in other to compare its property with that of fossil diesel fuel. The procedure for the various test carried out are shown below and the results are tabulated in table 1 and Table 2.

#### A. Density

The mass of the oil was measured using pyknometer, hence the density of the oil was calculated using the formula;

$$\rho_i = \frac{m_1 - m_o}{V_i} \quad (1)$$

Where,  $m_o$  is the mass in gram of the pyknometer or density bottle.

$m_1$  is the mass in gram of the pyknometer filled with water.

$V_i$  is the volume in ml of the oil in the pyknometer at a temperature  $t$ .

### B. Refractive Index

The refractive index was determined by placing a drop of the oil sample on the glass slab of the refractometer and the adjustable arms of the meter were adjusted to give reading. It was determined at a temperature of 15°C.

### C. Viscosity Test

Some amount of the oil was drawn into the stem of the Cannon-Ubbelohde viscometer (with Stokes constant of 0.03) to the mark above the upper bulb of the viscometer. The time taken for the meniscus to fall between the upper and the lower mark of the bulb through the capillary tube of the viscometer was recorded. The viscosity was determined using the following expressions:

$$\text{Kinematic viscosity} = \text{Time of fall} \times \text{Stokes constant} \quad (2)$$

The measurement was carried out at 40°C.

### D. Specific Gravity Determination

A known volume of the biodiesel fuel was weighed and the weight recorded, and the same volume of water was also weighed and the weight recorded. The ratio of the weight of the oil to the weight of water gives the specific gravity of the Biodiesel oil.

### E. Flash Point Test

Pensky-Martens (ASTM D93) method was used to determine the flash point of the Biodiesel fuel. The test cup was filled to a specific level with a sample of Biodiesel. The temperature of the sample was increased rapidly at first and then slowly as the flash point is approached. At 200°C a small test flame was passed across the cup, with a smooth continuous motion of the test flame across the cup. At a point the vapor above the surface of the testing sample was ignited with the aid of the test flame and the temperature at this point was noted and was recorded as the flash point. The test is continued

until at temperature was reached where the flame causes the oil sample to ignite and burned consciously for 5sec. the temperature at which this occur is at the fire point.

### F. Pour Point Test

The Biodiesel was poured into the test jar to the appropriate level. The cork into which the thermometer was inserted tightly closed the test jar. The position of the cork was adjusted and the thermometer fits the cork tightly. The thermometer and the cork were set coaxial and the thermometer bulb was immersed such that one end of the capillary was 3mm below the surface of the oil. The oil was heated without stirring to 48°C and maintained at this temperature. The fuel was then cooled to 35°C (95°F) in water bath. A jar ring was placed around the testing jar, 25mm from the bottom. The test jar was inserted into the ice jacket. The jacket was supported by the test jar in a vertical position in the cooling bath. After preliminary heating, the sample was cooled at a specific rate and examined at interval of 3°C for flow characteristics. The lowest temperature at which movement of the biodiesel was observed was recorded as the pour point.

## V. DISTILLATION CHARACTERISTICS

Heat was applied to the distillation flask contents gradually and the initial boiling point (IBP) was observed and recorded, with the tip of the condenser away from the walls of the graduated cylinder. The graduated cylinder was moved immediately. So that the tip of the condenser touches its inner wall. The heating was regulated so that the time from initial boiling point to 10% by volume recovered and the temperature was read on the thermometer and recorded. The heating was continuously regulated so that the uniform average rate of condensation for 10%-99% recovered was obtained. In the interval between the initial boiling point and ends of the distillation, all volumes in the graduated cylinder and all



thermometer readings corresponding to them were recorded. The end point, which is the final boiling point (FBP), was observed and recorded. While the condenser tube continues to drain into the graduated cylinder, the volume was measured accurately and recorded. After the flask has been cooled, its contents were poured into a 5ml-graduated cylinder. The flask was allowed to drain until no appreciable increase in the volume of liquid in the 5ml-graduated cylinder was observed. The value obtained for the percent recovery was added to the percent residue and the total recovery was obtained.

#### A. Sulphur Test

The sulphur content of the biodiesel produced was determined using ASTM.D 2622 method (Annual book of ASTM, 1987). The sample was placed in an X-ray beam and the intensity of the sulphur X-ray fluorescence was measured.

#### B. Copper Strip Corrosion Test

For this test, the ASTM.D 130 detection of copper corrosion from petroleum products by the copper strip tarnish test method was used. A polished copper strip was immersed in the biodiesel sample for 3hours at 100°C and then removed and washed. The condition of the copper surface was qualitatively rated by comparing it with standards.

#### C. Ash Content

The biodiesel sample (of known weight) was placed in a crucible, ignited and allowed to burn. The carbonaceous residue was heated further in a furnace to convert the entire carbon to carbon dioxide and all the mineral salts to oxides (ash). The ash was then cooled and weighed.

#### D. Water Content

A known weight of the Biodiesel sample was heated at a constant temperature of 100°C in an ovum for 50mins and weight was taken after every 10mins. The process was repeated until a constant weight was obtained. After every 10mins the sample was removed and placed in the desiccators for 20 minutes to cool the oil. The sample was then removed and re-weighed. The percentage water content in the oil was then calculated.

#### E. Cetane Index

Cetane index of a diesel fuel is a measure of the tendency of the fuel to ignite spontaneously. In the Cetane index scale, high values represent fuels that ignite readily and therefore perform better in diesel engines. It was calculated using equation below as recommended by ASTM D 4737(Annual book of ASTM, 1987).

$$CCI = 45.2 + (0.0892) (T_{10N}) + [0.131 + (0.901) (B)] [T_{50N}] + [0.0523 - (0.420) (B)][T_{90N}] + [0.00049][(T_{10N})^2 - (T_{90N})^2] - (107)(B) + (60)(B^2) \quad (3)$$

Where: CCI = calculated Cetane index, D = Density at 15°C, DN = D - 0.85, B =  $[e^{-(1.5)(DN)}] - 1$ , T<sub>10N</sub> = 10% recovery temperature, T<sub>10N</sub> = T<sub>10</sub> - 215, T<sub>50N</sub> = T<sub>50</sub> - 260, T<sub>90N</sub> = 90% recovery temperature, and T<sub>90N</sub> = T<sub>90</sub> - 310

## VI. EXPERIMENTAL RESULTS

The results of various analysis conducted on biodiesel oil production and their characterization are presented in Table 1 to 3

TABLE 1 PRODUCTION OF BIODIESEL FROM NEW AND WASTE OIL AT VARIOUS CONCENTRATION AND TEMPERATURE

PARAMETERS	Using 0.35g NaOH, 20ml methanol at 28°C		Using 0.35g NaOH, 20ml methanol at 65°C		Using 0.84g of NaOH, 27.38ml ethanol at 28°C		Using 0.84g of NaOH, 27.38ml ethanol at 78°C	
	N	W	N	W	N	W	N	W
	Amount of oil Used (ml)	100	100	100	100	100	100	100
Amount of Bio-Diesel produced (ml)	98.25	95.67	99.00	98.33	97.50	94.00	98.50	97.00
Amount of Glycerin produced (ml)	12.63	11.37	14.67	14.00	8.33	6.00	14.60	13.67
Alcohol recovered (ml)	5.40	6.30	4.54	5.10	7.35	7.95	6.00	6.70
Process efficiency (%)	96.90	94.42	98.51	97.86	88.85	84.75	93.50	92.14

TABLE 2 COMPARISONS BETWEEN THE COST OF PETROL DIESEL AND BIODIESEL

MATERIAL	QUANTITY (L)	COST (₹)
Petrodiesel	1	60
Biodiesel	0.9900	88.9
Glycerin	0.1467	293.4
Cottonseed oil	1	120

\*The cost as at Oct 2004

TABLE 3. CHARACTERIZATION OF BIODIESEL PRODUCED

PARAMETERS DETERMINED	Standard values			Experimental values	
	Diesel fuel	Biodiesel	Low sulphur diesel oil	N	W
Density at 15°C g/ml	0.82-0.88	0.87-0.89	0.83-0.86	0.860	0.87
Refractive index at 15°C				1.479	1.46
Specific gravity at 15°C	0.82-0.87			0.859	0.86
Viscosity kinematic at 40°C(Cst)	1.6-1.65	4-7	3.2	5.700	6.53
Ash content % mass	0.01max	<0.01		0.006	0.007
Sulphur content %	0.05max	0.01max	0.03max	0.0002	0.0005
Water content % mass	0.05	<0.05	<0.05	0.009	0.04
Flash point °C	>52	>100	>60	115	117
Cu strip corrosion (3hrs at 100°C)	No 1 strip	No 1 strip	No 1 strip	No 1 strip	No 1 strip
Distillation characteristics IBP (°C)				130.75	126.50
FBP (°C)				334	336.50
% Total recovery	90	>98	>98	98.68	98.30
% Residue				0.73	1.15
% Loss, New oil = unused – refined oil	0.1-1.5			0.60	0.55
Pour point °C	-10 to -0.01	<-10		-5.00	-2.00
Cetane index (calculated)	40min	48-52	45	50.58	51.22

Key:

W = waste oil ie oil that has been used for cooking, frying etc

N = new oil

Standard values from (Geyer et al, 1984, Fuel characterization, n d and Vegetable oil Yields characterization n d )

## VII. DISCUSSION OF RESULTS

Biodiesel was produced from cottonseed oil (refined and used) using a base catalyzed transesterification process. Temperatures and concentrations of the reacting mixture influenced the rate of the conversion. It could be observed from table 1 that at temperatures close to the boiling point of the alcohol (i.e. 66°C), a higher yield of biodiesel and glycerin were obtained, 99.0% and 14.67% for new oil, and 98.33% and 14.0 for waste oil respectively. Similarly, the overall process efficiencies were higher at high temperatures. The table also shows that using methanol as the solvent gave higher

yield of biodiesel at all temperatures than ethanol. However, these could be attributed to the fact that higher temperatures significantly increase internal energy and that reactivity decreases as molecular weight increases down the homologous series.

The result of characterization presented in table 3; shows that the densities and specific gravities of biodiesel produced are (0.86g/ml and 0.859) for new oil and (0.87g/ml and 0.86) for waste oil which are within the range of standard values of (.82-0.88)g/ml and (0.82-0.87)respectively for diesel fuel,(0.88)g/ml for biodiesel and (0.83-0.86)g/ml for low sulfur diesel fuel signifying that the biodiesel produced will

have a better lubricating effect on the engine parts of compression ignition engine (Weiss, 1983).

The flashpoint of biodiesel produced was: 115°C and 117°C for new and waste oil respectively. These values are within the minimum range of: >52°C for a typical diesel fuel given by ASTM, >100°C for a typical biodiesel provided by the national soy diesel development board for biodiesel and >60 °C for a typical low sulphur fossil diesel. This higher value of flash point is advantage over fossil diesel in the event of crash (i.e. safety) in handling (Oil Seeds, n.d). The pour point for biodiesel produced from new and waste oil were found to be -5°C and -2°C respectively which is within the range of standard specification of (-10 to -0.01) for diesel fuel, and (>-10 ) for biodiesel which is an indication that the biodiesel can support low temperature operability. Similarly, the biodiesel produced passed the copper strip corrosion test at No: 1 strip, meaning it will not cause corrosion of pipe (Oil Seeds, n.d). The Cetane numbers were calculated using equation 3 recommended by ASTM D 4737. The values obtained were 50.58 and 51.22 for biodiesel produced from new and waste oil respectively. This values fall within the standard specification of (48-52) recommended by ASTM D975 for biodiesel signifying that the biodiesel is of high ignition quality (less knocking. Distillation characteristic of a product signifies its purity, and from table 3, the distillation recovery of the biodiesel produced is 98.68% and 98.30% for new and waste oil respectively. The residue was 0.73% and 1.15% and loss of 0.60% and 0.55% for new and waste oil respectively, these values are within the range of standard specification, which implies that the biodiesel is of high purity (Annual book of ASTM, 1987, Weiss, 1983).

Table 2 shows that the cost of 0.99l of biodiesel is ₦88.9 while for 0.1467l of glycerine is ₦293.4. The

cost of producing the same liters of biodiesel and glyceride from 1 litre of cotton seed oil is ₦ 225.3, comparing this with the selling price of the two product (₦ 382.3) shows that the process is profitable. Finally, the environmental and ecological advantages of biodiesel should be given more emphasis rather than the economic advantage.

## VIII. CONCLUSION

Production and characterization of biodiesel from cottonseed oil was performed, analysis carried out on the biodiesel produced show that it is essentially sulfur free, has a higher Cetane rating (less knocking), higher lubricating effect than petrol diesel and will not cause corrosion of pipes. Thus the biodiesel can effectively serve as an alternative to petrol diesel without modification to diesel engines and that the environmental and ecological advantages of biodiesel should be given more emphasis rather than its economic importance. Hence, the base catalyzed trans-esterification process using ethanol is an effective method for biodiesel production.

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# Production and Characterization of Alum from Ahoko Clay, Kogi State, Nigeria

Nku C.T., Edoga M.O. and Kovo A.S.  
Department of Chemical Engineering,  
Federal University Of Technology, P.M.B. 65. Minna, Niger State  
kovoabdulsalami@yahoo.com

**Abstract** - The aim of this paper is to produce Alum using local raw material. The chosen raw material is Ahoko clay and the process of production of alum includes beneficiation, calcinations and dealumination. 2000g of kaolin clay was soaked in deionised water for 72hrs and the mixture was sieved using 70micron sieve. Sodium bi carbonate and sodium hexametaphosphate was added to the clay mixture for beneficiation. The clay obtained was dried for 24hrs at 45°C and was retained as beneficiated clay. The beneficiated clay was further calcined at 600°C for 3hrs to convert the kaolin to meta-kaolin. The alum was produced via the process of dealumination. This involved treating the calcined clay with 60% of concentrated sulphuric acid ( $H_2SO_4$ ) in 340<sup>cm</sup><sup>3</sup> of solution immersed in a thermostated water bath at 1002°C. The filtrate was crystallized yielding the alum cake. The chemical analysis of the clay showed that the percentage composition of its basic constituents as  $SiO_2$ -73.18,  $Al_2O_3$ -14.53,  $Fe_2O_3$ -0.70,  $CaO$ -1.35,  $mg$ -0.81,  $SO_3$ -0.80 and  $CaCO_3$ -2.41 respectively. The titrimetric analysis of the synthesis alum showed that it reduced total hardness in water from 432ppm to 320ppm.

**Key word:** Kaolin clay, Alum, Beneficiation, Dealumination

## I. INTRODUCTION

Alum is an organic colourless to white crystalline substance which occurs naturally as the mineral kalunite. It is a hydrated double salt that contains aluminum

sulphate and potassium aluminum sulphate by treating bauxite or kaolin since they contain alumina with sulphuric acid to yield alum cake. Alum is also hydrated double salts containing two  $SO_4^{2-}$  cations, one triply charged anion and twelve molecules of water of crystallization. The crystal of one alum is usually isomorphous with the crystal of the others (Liptrot, 1985). Alums are isomorphic (that is, they have the same crystal pattern and very similar lattice dimensions). Alum grows large crystals from saturated solution at constant temperature and the crystals of alum are usually large having octahedral structural. Alums only differ from other types due to the addition of some elemental ions to form double salt known as aluminum sulphate. Alum is used in a number of industries. It is used in textile industry. It is also used in making paper to work smooth and opaque to ink in paper industry and serves as a major coagulant in the treatment of water.

## II. EXPERIMENTAL PROCEDURE

Beneficiation of the clay is the first step towards the petroleum of alum. 2000g of the clay was soaked in four liters of deionised water for 72 hours; stirring was done at periodic interval using the magnetic stirrer. The mixture was passed through a 70micron sieve to remove the particle above 70micron sieve. Four liters of 2:8 sodium bicarbonate and sodium hexametaphosphate solution was

added to the filtrate for deflocculation. The mixture was left to settle for eight hours after which it was decanted, centrifuged and washed thoroughly with deionised water to remove the excess deflocculants. The clay was then dried for 24 hours in the electric oven at 45°C. The beneficiated clay was calcined at 600°C for three hours to put the clay in a more reactive form.

40g of the calcined clay was poured into solution containing 240cm<sup>3</sup> of 60% sulphuric acid concentration. The mixture was stirred vigorously to prevent the formation of coagulated mass at the base of the flask after which the flask was inserted in a thermostated water bath that had attained a constant temperature of 102°C. When dealumination process has been complete in an interval of 20 minutes after cooling, the filtrate was put in an evaporating dish, placed on a regulated hot plate and was heated to crystals, as soon as crystal are formed, heating was stopped and the crystal allowed to cool. The crystal obtained is aluminum sulphate (Alum). The chemical composition of the raw clay used in the production of the dealuminated clay was determined using the x-ray fluorescence (XRF). The effect of the synthesized alum and commercial alum on land water was determined by titration in order to know the effectiveness of the produced alum

### III. RESULT

The result of chemical composition of crude clay and calcined clay presented in Table 1 and 2 respectively.

TABLE 1 CHEMICAL COMPOSITION OF CRUDE KAOLIN CLAY

Chemical constituent	Percentage composition of crude clay
SiO <sub>2</sub>	73.18
Al <sub>2</sub> O <sub>3</sub>	14.53
Fe <sub>2</sub> O <sub>3</sub>	0.70
CaO	1.35
MgO	0.81
SO <sub>3</sub>	0.08
CaCO <sub>3</sub>	2.41

TABLE 2 CHEMICAL COMPOSITION OF CALCINED CLAY

Chemical parameter	Percentage composition of calcined clay
SiO <sub>2</sub>	73.22
Al <sub>2</sub> O <sub>3</sub>	7.48
Fe <sub>2</sub> O <sub>3</sub>	0.27
CaO	1.12
MgO	0.65
SO <sub>3</sub>	0.87
CaCO <sub>3</sub>	0.25

TABLE 3 X-RAY FLUORESCENCE ANALYSIS OF DEALUMINATED CLAY WITH 50% H<sub>2</sub>SO<sub>4</sub>

Chemical parameter	Percentage composition of dealuminated clay
SiO <sub>2</sub>	66.34
Al <sub>2</sub> O <sub>3</sub>	2.51
CaO	1.33
MgO	0.67
SO <sub>3</sub>	17.40
CaCO <sub>3</sub>	2.20

TABLE 4 TITRIMETRIC ANALYSIS OF THE EFFECT OF SYNTHESIZED ALUM ON HARD WATER

Time (mins)	Total hardness	Calcium hardness	Magnesium hardness
0	432.00	225.00	207.00
10	335.00	184.00	151.00
20	334.00	183.00	151.00
30	334.00	183.00	151.00
40	320.00	180.00	139.00
50	320.00	180.00	139.00

TABLE 3: TITRIMETRIC ANALYSIS OF THE EFFECT OF SYNTHESIZED AND COMMERCIAL ALUM ON HARD WATER

Time (mins)	Total hardness	Calcium hardness	Magnesium hardness
0	432.00	225.00	207.00
10	255.30	160.20	90.10
20	250.20	160.20	90.00
30	250.20	160.20	90.00
40	250.20	160.00	90.00
50	250.20	160.00	90.00

#### IV. DISCUSSION

The chemical analysis of the raw kaolin was carried out and the percentage composition of the constituents showed that kaolin contain  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{SO}_3$  and  $\text{CaCO}_3$  in the amount 73.18, 14.53, 0.70, 1.35, 0.81 and 2.41 respectively as table 3.1. Also the chemical analysis of the calcined clay was carried out and the chemical composition in percentage includes  $\text{SiO}_2$ -73.22,  $\text{Al}_2\text{O}_3$ -7.48,  $\text{Fe}_2\text{O}_3$ -0.27,  $\text{CaO}$ -1.12,  $\text{MgO}$ -0.65,  $\text{SO}_3$ -0.87,  $\text{Na}_2\text{O}$ -0.25 and  $\text{CaCO}_3$ -2.0.

The effect of commercial and synthesized alum on hard water was analysed. It was observed in table that the total hardness of water which was 432.00(ppm) at time zero (0) was reduced to 335.00 (ppm) at time 10minutes.

Also, the total hardness was reduced from 335.00 (ppm) to 334.00(ppm) at time 20 and 30minutes respectively and from 334(ppm) to 320 at time 40 and 50minutes respectively by using the synthesized alum. Also the calcium hardness which was 225(ppm) at time (0) has reduced to 184(ppm) to 183(ppm) at time 10minutes and from 183(ppm) to 180.50(ppm) at time 40 and 50minutes respectively using the synthesized alum.

Furthermore, the above results can be compared with the effect of commercial alum on hard water as shown in table 3. The alum also showed that total hardness of water at 432(ppm). The commercial alum reduced the hardness to 250.30 at time 10mins, notices from 250.30 to 250.20 (ppm) at time 20 and 30minutes respectively; also

reduction of total hardness, took place at 250.20 to 250(ppm) at time 40minutes and 50minutes respectively. Meanwhile, the commercial alum reduced the calcium hardness from 225(ppm) to 160.20(ppm) at time 10minutes reduction of calcium took place at 160.20(ppm) to 160.00(ppm) at time 30, 40 and 50minutes respectively.

The result of the analysis of the effects of commercial and synthesized alums on water showed that the synthesized alum is within the range of standards for alum production and has quality effect on hard water. Since the total hardness of a sample of hard water with total hardness of 432(ppm) was dractically reduced to 320(ppm). Alum reduces hardness in water because it reduces the pH of solution which means that as the pH is reduced it tends to decrease from alkalinity to acidity, since alum is acid forming compound that causes hardness in water such as calcium ion and magnesium ion are alkaline, as such when the pH of the water is reduced from alkaline to acidic, the hard water ions tends to be affected by this phenomena as such the carbonated and magnesium ion in the water are reduced thereby reducing the hardness of water. Furthermore, it was noticed that when the synthesized alum was added to turbid water, it was noticed that the alum reduces turbidity by 89-97% within few hours of dosage at 1—30mg/litre (ppm).

#### V. CONCLUSION

The developed alum from kaolin showed the characteristic feature of Alum used in the treatment of hard water. The raw material used in the production of alum contain,  $\text{SiO}_2$ -73.18%,  $\text{Al}_2\text{O}_3$ -14.53%,  $\text{Fe}_2\text{O}_3$ -0.7%,  $\text{CaO}$ -1.35%,  $\text{MgO}$ -0.81%,  $\text{SO}_3$ -0.08%, and  $\text{CaCO}_3$ -2.41%. It is proven that the alum produce reduces the hardness of water. Total hardness was reduced from 432(ppm) to 250(ppm) using the commercial alum while total hardness was reduced from 432(ppm) to 320(ppm) using the synthesized alum.



Finally, the synthesized alum was tested on turbid water and a good result was achieved i.e. coagulation took place after a short period of time.

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## INTRODUCTION

The castor bean plant *Ricinus communis* native to tropical Africa, cultivated in several varieties for the oil content. The leaves, it is also grown in tropical and subtropical areas such as central Asia, Tanzania, Brazil and southern Kazakhstan to name a few. It occurs as a perennial or annual plant and considered as drought resistant crop in India. The stalked leaves consist of unusual eight radiating pointed leaflets, and prominent central vein. Many varieties are green and inconspicuous but pink or

bluish. Production, purification, and revelation of castor oil were carried out using Soxhlet apparatus and petroleum spirit as solvent. The extraction was done at constant temperature using various weight of the sample. The gummi, neutralisation, and bleaching reagent reduced the extracted oil. Both physical and chemical properties were evaluated. The refractive index (RI) showed that the oil was not drying oil and so it could be used as a lubricant. Protective coating for roofing paper of the extracted oil was studied against the ASTM specification of 31.5°C. Other physical and chemical analysis carried out on the oil as compared to ASTM standards, confirmed that the property of the oil was within specification.

# Production, Purification and Evaluation of Castor Oil from Castor Bean Seeds

A.G. Isah, M. Alhassan  
Department of Chemical Engineering  
Federal University of Technology, Minna, Nigeria  
[ag\\_isah@yahoo.com](mailto:ag_isah@yahoo.com)

M.U. Garba  
Department of Chemical Engineering  
University of Maiduguri  
Maiduguri, Nigeria

**Abstract** - Production, purification, and evaluation of castor oil were carried out using soxhlet apparatus and petroleum spirit as solvent. The extraction was done at constant temperature using various weight of the sample. Degumming, neutralization, and bleaching refined the extracted oil. Both physical and chemical properties were evaluated. The iodine value (84.86) showed that the oil was non drying oil and so it could be used as a lubricant, protective coating. The boiling point of the extracted oil was 316°C as against the ASTM specification of 313°C. All other physical and chemical analysis carried out on the oil as compared to ASTM standard, confirmed that the property of the oil was within specification.

## I. INTRODUCTION

The castor bean plant, *ricinus communis* native to tropical African cultivated in several varieties for the oil found in the leaves. It is also grown in tropical and subtropical areas such as central Asian, Tanzania, Brazil and southern Kazakhstan to name a few. It occurs as a perennial or annual plant and considered as drought resistant crop in India. The stalked leaves consist of unusual eight radiating pointed leaflets, and prominent central veins. Many varieties are green and inconspicuous but pink or

red in pigmented varieties. Many stamens are near the base and branching pistils are near the top flower. The soft spines contain attractive mottled seeds (Moshkin, 1986).

Castor bean contains 50 -55 % of oil and the extraction of oil from seed is done in similar manner to most other oil seeds (Weiss, 1983). The seed are collected when ripe as capsules dry, they open and discharge the seed. The seed are then cleaned, decorticated, cooked and dried prior to extraction. Cooking is done to coagulate protein, which is necessary to permit efficient extraction, and to free the oil for efficient pressing it is done at 80°C under airtight conditions. After drying the material at 100°C to moisture content of approximately 4 - 5 %, the extraction can be done by varieties of processes or combination of processes. Processes such as continuous screw press expeller, hydraulic press and solvent extraction are employed. The first stage of extraction is pre-pressing using high-pressure continuous screw press expeller which usually consists of barrel containing a stainless steel helical screw (Fellow, 1996).

Extracted oil is filtered and collected in a settling tank. The material discharged from the press called cake which contains 8 - 10% oil is crushed into coarse meal, and subjected to

solvent extraction with solvent such as hexane, heptane, and petroleum spirit e.t.c. After extraction the solvent is removed by distillation, and the resulting oil is processed in similar manner as oil from pressing steps (Weiss, 1971).

Once the oil has been extracted from the seed it is necessary to remove any impurities from the oil. The oil is essentially a pure triglyceride, and contains almost 90% glyceryl ricinoleate. It is the ricinolic triglyceride that is needed in order to produce high quality castor oil that could be used for chemical reaction. Common properties of castor oil include high density and viscosity. These properties are exploited when refining the oil. The process of refining a crude oil includes settling, degumming, bleaching, neutralization and deodorization of the oil (Nawar, 1996).

The uses of castor oil have changed over the years. Sixty years ago castor oil was used for medicinal purposes and as general industrial lubricant. Soon after, chemical engineers were able to produce derivatives of the oil that were of even more benefit to man sulfonated castor oil, or Turkey red oil, was the first synthetic detergent to be produced after which ordinary soap and other forms of oil became important for treatment of leather, industrial lubricants and other industrial uses. Today chemical engineers have come up with many uses of castor oil together with its derivatives such as polyamide, (Nylon 11), engineering plastics, coating, inks, and emulsifiers. Castor oil has even made its way into cosmetics industries and related products. The medicinal purposes include its use as a soothing emollient for dry skin and other skin diseases. Obviously castor oil and its derivatives have

become important commodities and items of interest to chemical industries (Vignolo and Naughton, 1991).

## II. EXPERIMENTAL METHODS

### A. Preparing bean for extraction

The castor bean seed contained some foreign material and dirt's which were separated by hand picking. They were then dried under the sun, until their capsules split and shed their seeds. The seeds were decorticated manually to remove the coats. The shells were separated from the nibs using tray to blow away the shells in order to obtain clean seeds. The cleaned seeds were cooked under airtight condition at a temperature of 80 – 90°C. This was done to coagulate the protein, which was necessary to permit efficient extraction.

### B. Determination of Moisture Content

500g of the seed were weighed. This was dried in an oven at 100°C for 7 hours and the weight taken after every three hours. The process was repeated until a constant weight was obtained. At three hours interval the sample was removed from the oven and placed in the desiccators for 20 minutes to cool. The sample was then removed and re-weighed. The percentage moisture content in the seed was calculated.

### C. Experimental Procedure

35g of the sample was placed in a thimble and about 250ml of petroleum spirit was poured into a round bottom flask. The apparatus was heated at about 80 – 90°C and allowed for 2 hour continuous extraction. The experiment was repeated with different weights of the sample i.e. 35.5g, 30g, 25g and 20g. The solvent was distilled and percentage of oil extracted was calculated.

#### D. Refining of Extracted Oil

##### (i) Preparation of Clay

The clay was ground and sieved using sieve machine of mesh 250-micrometer size, the clay was then mixed with water to remove dirt and stone particle. The clay was activated; 2M hydrochloric acid was added to clay slurry in a round bottom flask. The mixture was boiled for 1 hour 30 minutes at a temperature of about 120°C after which the mixture was washed with distilled water to remove the acid. The recovered clay was dried in the oven for 2 hours at a temperature of 100°C and later ground.

##### (ii). De-gumming

The extracted oil was de-gummed by addition of boiled water. The mixture was allowed to stand in a separating funnel for 5 minute with agitation and allowed to cool. The gum and water was removed. The procedure was repeated to remove the gum completely from the oil.

##### (iii). Neutralization

About 50g of de-gummed oil was poured into a beaker and heated at 90°C with 35ml of 1M NaOH which was constantly stirred. This was transferred into a separating funnel and allowed to stand for 2 hour. The soap formed was separated from the oil. Hot water was added repeatedly to oil solution until the soap in the solution was removed leaving the neutral oil which was later poured into a beaker.

##### (iv). Bleaching

The neutralized oil was poured into a beaker and heated to about 80°C. 7g of the activated clay was poured into a beaker; the mixture was stirred for 30 minutes. The temperature was allowed to rise to 100°C for 30 minutes. The content was then filtered hot.

#### H. Evaluation of Extracted Castor Oil

##### (i) Determination of Iodine Value

The method specified by International standards organization (ISO) 3961 (1986) was used. 0.4g of the sample was weighed into a conical flask. 20ml of carbon tetrachloride was added to dissolve the oil and 25ml of dam's reagent was added to the flask using a pipette in a fume chamber. Stopper was inserted into the flask and the content was vigorously swirled. The flask was then placed in a dark for 2 hour. At the end 20ml of aqueous potassium iodide and 120ml of water was added. The content was titrated with 0.1M sodium thiosulphate solution until the yellow colour almost disappeared. Few drop of 1% starch indicator was added and the titration continued until blue coloration disappears at constant shaking.

##### (ii) Determination of pH Value

30ml of the sample was poured into a cleaned dried beaker and 13ml of hot distilled water was added to the sample and stirred to 5°C slowly.

The pH probe was standardized with buffer solution and the probe inserted into the oil, the pH value was then determined.

##### (iii) Determination of Saponification Value

3g of the sample was weighed into a conical flask, 30ml of 0.1M ethanolic potassium hydroxide was added to the sample. The mixture was allowed to boil for 70 minutes with constant shaking. A reflux condenser was then placed on the flask containing the mixture. Few drop of phenolphthalein was added to the warm solution and then titrated with 0.5M hydrochloric acid until the pink colour of the indicator just disappeared. The same procedure was used for other samples and for standard solution.

##### (iv) Determination of Acid Value

2ml of diethyl ether was mixed with 25ml of ethanol in a 250ml beaker. The mixture was poured on 10g of oil in a 250ml conical flask and few drop of phenolphthalein indicator was added to the mixture. The mixture was titrated with 0.1M NaOH with constant shaking until a dark colour was observed and the volume of 0.1M NaOH ( $V_n$ ) used noted.

*(v) Determination of Specific Gravity*

Density bottle was used to determine the density of the oil. A cleaned, dried bottle of 25ml capacity was weighed ( $M_0$ ). It was filled with the oil then the bottle and its content and re-weighed to give ( $M_1$ ). The oil was substituted with water of the same volume and re-weighed to give ( $M_2$ ).

*(vi) Determination of Refractive Index*

Refractometer was used in determining the refractive index of the oil, few drop of the sample was placed on glass slide of the refractometer. Water at 300°C was circulated around the glass slide to keep its temperature uniform. The refractometer was viewed through the eyepiece; the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer to the scale pointed on the refractive index. This procedure was repeated and the mean value was taken.

*(vii) Determination of Viscosity*

The sample was filtered through a sintered glass (fine mesh) to eliminate solid materials in the liquid oil. The viscometer was charged with the oil by inverting the tube's thinner arm into the oil and suction force was drawn up to the timing mark of viscometer. The instrument was then turned to normal vertical position.

The viscometer was placed into a holder and inserted into a constant temperature bath set at 27°C. A period of 5 minutes was given to allow the sample attain the same temperature of the bath. The suction pressure was applied to the thinner arm to draw the liquid level above the upper timing mark. The afflux time the oil as it flows freely from the upper timing mark to the lower timing mark, and the reading was recorded.

III RESULT AND DISCUSSION OF RESULT

A. Results

TABLE 1 PERCENTAGE VOLUME OF OIL EXTRACTED

Weight of sample		Weight of oil extracted (g)	% oil extracted
Before extraction (g)	After extraction (g)		
35.5	28.87	8.63	24.31
30.0	21.68	8.32	27.73
25.0	16.45	8.55	32.2
20.0	13.10	6.9	34.5

TABLE 2 COMPARING THE PHYSICAL PROPERTIES OF REFINED OIL AND STANDARD VALUE.

Property	Refined castor oil	Standard value
Viscosity at 25°C	6.5847st	6.3 - 8.8st
Refractive index 27°C	1.469	1.473 - 1.477
Specific gravity	0.9584	0.958 - 0.968
Boiling point	316°C	313°C

TABLE 3 COMPARING THE CHEMICAL PROPERTIES OF REFINED OIL AND STANDARD VALUE.

Property	Refined castor oil	Standard value
Iodine value	84.86	81 - 91
Acid value	0.861mg NaOH/g of oil	0.4 - 4 mg NaOH/g of oil
Saponification value	182.73mg KOH/g of oil	176 - 187 mg KOH/g of oil
pH value	6.3	-

### B. Discussion of Result

The result obtained for moisture content was 4.7%. This shows deviation from report of Weiss (1983) who reported the moisture content of the range of 2.3 – 3.7% for some varieties. This result may depend on the rate of drying or the variety of the seed. The result obtained for the percentage oil content for different weights of sample extracted within the same period showed that the smaller the sample the higher the percentage of oil extracted. For instance with 30g, 24.3% of oil was obtained, with 20g, 34.5% of oil was obtained. The result was tabulated in Table 1. The total percentage oil content was found to be 32.1%. This result shows deviation from the literature value of 50 – 55%. This may depend on the variety of seed used; hence different varieties contain different percentage oil content. It may also depend on the method of extraction. Fellows in 1996 reported that the most efficient method of extraction was screw press expeller or combination screw press expeller and solvent extraction, which yielded more oil.

Table 3 shows the comparison between physical properties obtained during research work and the standard value (ASTM). The viscosity value obtained was 6.5847st. This falls within the range of the standard value found in the literature 6.3 – 8.8st found.

The refractive index obtained during the research work was 1.469, this result deviate a little from the result reported in Table 3 for standard value which was 1.473 – 1.477. This deviation may be as a result of impurity present in the oil. The result of specific gravity obtained during the research work was 0.958, this result fall within the range of the standard value 0.958 – 0.968 shown in table 3. The boiling point obtained was 316<sup>o</sup>C; the value shows deviation from the standard value, which was

reported to be 313<sup>o</sup>C., this deviation may be as a result of some impurities contained in the oil.

Table 2 shows the comparison between the evaluated chemical properties obtained during the research work and the standard value, reported in the literature. The iodine value obtained was 84.86 compared to the result of the standard value, which was 81 – 91. This result is within the range and reflects the amount of iodine that can be absorbed by unsaturated acid. The oil could be classified as non-drying oil, since the iodine value is lower than 100. This unique property makes it possible for castor oil to be used as lubricants and hydraulic break fluids.

Saponification value obtained was 182.793mg KOH/g of oil. The value still falls within the range of standard value, which was reported in Table 3 to be 176 – 187 mg KOH/ g of oil.

The acid value obtained was 0.86mg NaOH/g of oil. This value is higher than the standard value. This may be as a result NaOH used during the refining.

### IV. CONCLUSION

The percentage of oil content extracted was 32.1%. This result is far below the value found in the literature. Combined method of solvent extraction and continue screw press expeller, yield more oil from the seed. The castor oil extracted is evaluated; it gives viscosity value of 6.5847st, specific gravity of 0.9584, refractive index of 1.469 at 27<sup>o</sup>C, boiling point of 316<sup>o</sup>C, acid value of 0.861 iodine values of 84.86, and saponification value of 182.79. Most of these values fall within the range of the standard specification values of ASTM except boiling point. Since the oil extracted is not solvent free, it cannot be used for cosmetics.

However, the refined castor oil could be suitable for industrial purposes to produce other material that does not have direct

contact with the skin e.g. lubricating grease, printing ink, hydraulic fluid etc.

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