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Appraisal of Esters Of Edible Oils For Drilling Mud Formulation

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Abstract

The need to preserve the environment led to the engineering of ester base mud. Ester base mud is dispersible, less toxic and biodegradable as compared to water and oil base muds. Edible oils are readily available oils.
Oils were extracted from edibles: Palm kernel and *Treculia africana.* The oils were characterised for their Physico-chemical properties; and fatty acid content using the GC-FID. Results obtained were utilised to synthesize the esters. The esters were characterised for its properties using the GC/MS and FTIR. 350ml mud for each was with 80/20 ester water ratio with other requisite additives and their attendant rheological properties, electrical stability and retort tests were carried. Control, fossil Diesel Based Mud was formulated and the Ester Based Mud were compared.
Palm kernel and *Treculia africana* yielded 38% and 22% respectively. Similarly, the ester yield of T. *africana* was 96% while P. Kernel amounted to 86%. The better yield was a product of esterification before transesterification. The pH values were greater than 8 an indicator that they would not corrode the drilling equipment. The gel strength of the mud, oil, water and solid content of the mud, and the rheological properties of the mud are acceptable. The electrical stability of OBM was 468 Volts and SBM-PK was 392 Volts. SBM-TA indicated 389 Volts.
The formulated muds were non Newtonian and conform to the Bingham Plastic and the Power Law Models.

Common words: Synthetic Base Mud, SBM, TA-*Treculia africana,* Palm Kernel, PK, Electrical Stability, ES, Gas Chromatography/Mass Spectroscopy, GC/MS.

Introduction

The rotary drilling is the main technique for drilling hydrocarbon wells and drilling fluid its "blood". This fluid travels from (a.) the steel tanks to the mud pump (b.) from the mud pump through the high pressure surface connections to the drillstring, (c.) through the nozzles of the bit and up to the annular space between the drillstring and hole to the surface, and (d.) through the contaminant-removal equipment and back to the suction tank (Bourgoyne jr. *et al.,* 1991).

The functions of drilling mud include (i.) carrying cuttings from the hole (ii.) cooling and cleaning the drill bit (iii.) reducing friction (iv.) maintaining the stability of the bore (v.) maintaining down-hole hydrostatic pressure and (vi.) to damage the formation (Onungwe, 2015 and Queensland, 2013).

Drilling mud is a fluid, usually, a mixture of clay and other additives. The fluid can be aqua (H_2O), oil and recently synthetic. When the mud is mainly composed a specific fluid it is termed base-fluid. Ester base mud is a subset of synthetic mud (SBM). Fundamentally, three factors influence the choice of drilling mud. These are (a.) cost (b.) technical performance and (c.) environment concerns (onepetro.org, 2014).

The issues of cost and technical performance motivate investors to the detriment of the environment save for the regulations of the state or related institutions. The environment deserves more attention because it belongs to the future generation. Hence, oily cuttings generated offshore are disposed of by: (a) shipping them to shore and discarding or treating them there with other wastes; (b) downhole injection (down the annulus or into another well); or (c) cleaning them on site via solvent extraction or distillation and these are expensive to run.

Today, the economics of low hydrocarbon prices and environmental concerns are forcing oil and service corporations to monitor discharges or increasingly to dispose of discharge in an environmentally acceptable manner (Geehan and Mckeen,). To advert the attendant litigations, sanctions, downtime, remediation and other losses associated with the issues of poor formulation, use and disposal of drilling mud, mud engineering is key. This involves the application of science and technology to create specialised and well characterised mud, monitoring at every moment during drilling to determine what is being removed at the surface and what is needed to maintain established specifications.

Ester base mud is a synthetic mud which is one of the innovative research of mud engineers. Ester, an organic compound is obtained from the reaction between carboxylic acid and alcohol. During this reaction, the hydrogen of an acid is replaced by an alkyl from the alcohol. This ester is referred to as synthetic fluid and when it serves as a base-fluid can be called synthetic mud. This reaction is aided by heat and catalyst. SBM is particularly useful for deepwater and deviated hole drilling. By design, they combine the technical advantages of oil base mud with the low persistence and toxicity of water base mud (Onungwe, 2015).

Carboxylic acid can be obtained from oils and fats whose sources include: (1.) vegetable oils (edible and non edible), (2.) animal fat, (3.) waste cooking oil – used oil materials and (4.) algae oils. Each of these have been examined by different researchers and vegetable oils appear more appealing due to their availability and low cost of processing into biodiesel (Bankovic-llic *et al.*, 2012).

Operationally, ester base mud performance has been reported to equal that of conventional oil base mud. Some differences remain, however, which on the one hand give them desirable environmental attributes, but on the other hand may pose some limitations on handling and use. SBM is more expensive than conventional OBM but if thoroughly formulated and properly handled, it pays off on the long run.

(Amorin *et al.*, 2015) noted that the initial cost of formulating SBM compared to OBM may be doubled but the cost of containment, hauling, and disposal of OBM after use is quite high compared to SBM. The use of cheaper local Synthetic Base Fluid (SBF) products and its allowed discharge at drilling sites offsets its initial cost of formulations, thus transportation and disposal costs are saved.

Ester base mud is highly environmental friendly because micro-organisms can easily break down and this speeds up its rate of decomposition. They are biodegradable, non toxic and non carcinogenic unlike the use of diesel and mineral oils in oil base mud.
(Bankovic-llic et al, 2012) observed that edible oils are the main resources for world biodiesel production (more than 95%) noted that edible oils are unlike non edible plants is that contain high content of free fatty acids (FFA) or non esterified fatty acid (NEFA) which increases the cost of biodiesel production.

During this process, alcohol is reacted with oil (triacylglycerol) to produce fatty acid alkyl ester and a by product, glycerol. The main factors affecting trans-esterification reaction and produced ester yield are: (1.) the molar ratio of alcohol and oil, (2.) type of alcohol, (3.) type and amount of catalyst, (4.) reaction temperature, (5.) pressure, (6.) time, (7.) mixing intensity (8.) free fatty acid – FFA content and (9.) water in oils.[5]

Equation of Reactions

1. **Esterification**

$$ROH_{(l)} + R' - COOH_{(l)} \underset{}{\overset{H_2SO_4}{\rightleftarrows}} R'COOR_{(l)} + H_2O_{(l)} \text{ --(i.)}$$

2. **Transesterification**

$$ROH_{(l)} + R'COOR_{(l)} \overset{NaOH}{\rightleftarrows} R'COOR''_{(l)} + C_3H_8O_{3(s)} \text{ ---(ii.)}$$

Scores of feedstock for the production of biofuel abound, but the most appropriate ones should not compete with food, have high yield per unit area of growth and require a minimum input (water, fertilizer, energy) to grow (Babatunde *et. al.*, 2013). Seeds chosen for this research were: Palm Kernel and *Treculia africana* meets these conditions.
The samples were collected at the University of Ibadan Botanical Garden, dried, de-husked and pulverised. Oils were extracted using n-hexane as solvent and the Soxhlet Extractor equipment at the University of Ibadan, Petroleum Engineering Department.

Characterisation for the Physico-chemical properties of both the oils and the esters were conducted at the Kappa Biotechnology. Laboratory, Ibadan. Preceding transesterification were the Methylation and Gas Chromatographic analysis of the oils at the University of Ibadan Multidisciplinary Laboratory, Ibadan, to determine their Fatty Acid Composition using internal standard of GC-FID. Esterification and transesterification of the oils were carried out at Forestry Research Institute of Nigeria (FRIN), Ibadan. Transesterification was carried out using oil/methanol ratio of 1:6 and about 1% weight of catalyst. The catalysts used were liquid sulphuric acid (H_2SO_4) for esterification and (NaOH) sodium hydroxide for transesterification. The magnetic stirrer bar was set at about 1000 rpm for laboratory barrel (350 ml) of oil sample.

Fourier Transform Infrared Spectroscopy, FTIR and the Gas Chromatography/Mass Spectroscopy, GC/MS of samples were analysed at the University of Ibadan, Ibadan and Obafemi Awolowo University, Ile-Ife respectively for the purpose of identifying their bonds and functional groups; and composition and possible structures, in that order. Esters were analysed for their Physico-chemical properties.

Laboratory mud was formulated using ester/water ratio of 80/20 with – 3g of organophilic clay, 3g of lime, 10g of barite and 5g of emulsifier. Rheological, gel strength, pH, electrical stability and oil-water-contents analyses were determined using the Rheometer, pH meter, Electrical stability and Baroid Retort Kit in that order.

Results and Discussions

Oil yield expressed in percentage: There seems to be divergent reports on percentage oil yield for instance, P. Kernel oil data gives between 40% and 48%. Hence, the 38% oil yield of this analysis is acceptable. The oil yield of *Treculia africana* was approximately, 22% this is good as literatures gave a range of 14 – 21.92% oil yield.

Table 1
Physico-chemical properties of the oil samples

Parameter	TA	PK
Density (g/ml)	0.88 ± 0.05	0.96 ± 0.05
Flash Point (°C)	172.00 ± 1.41	242.67 ± 2.49
Pour Point (°C)	14.33 ± 1.70	4.67 ± 0.47
Acid Value (mg KOH)	1.27 ± 0.10	1.67 ± 0.13
Saponification Value (mg/g)	123.27 ± 0.57	145.83 ± 0.29
Iodine Value (mg/100g)	74.53 ± 0.21	86.37 ± 0.17
Moisture Content (%)	1.50 ± 0.16	0.93 ± 0.13
Viscosity (CentiStokes)	196.17 ± 0.40	497.47 ± 0.13
Kinematic Viscosity	215.57 ± 0.21	546.80 ± 0.47

Table 1 shows the Physico-chemical properties of the oil samples. Palm kernel oil is denser than that of *Treculia africana* as their values were: 0.96 and 0.88 g/ml respectively. The same phenomenon applies for their flash points 242.67 and 172.00 °C . This table also presents the pour points of *Treculia africana* and Palm kernel oil as 14.33 and 4.67 °C respectively – an indication that palm kernel pours more at cold temperature.

Table 2
Sample versus the Volume of n-hexane

Sample	Vol. of n-hexane
Treculia africana	2220 ml
Palm Kernel	4400 ml

From table 2, it can be observed that Palm kernel sample consumed more solvent, 4,400ml as compared to 2,220 ml of *Treculia africana.* This is because unlike the later, palm kernel sample was soaked in the solvent for about 24 hours and was manually extracted before the Soxhlet Extractor was used to distil it. The essence was to aid better oil recovery but this method was not efficient.

Ester yield expressed in percentage: *Treculia africana* had 96% ester yield while the Palm kernel sample yielded 86% the variation in the yield may be attributed to the esterification process that preceded the transesterification of the former against the Palm Kernel. This should be thoroughly examined with respect to cost.

Table 3
Physico-chemical Properties of the Biodiesel

Property (°C)	Syn-TA	Syn-PK	Diesel
Flash Point	303.6 ± 1.5	174.6 ± 1.5	90.5 ± 2.0
Cloud Point	17.3 ± 1.1	5.6 ± 00	5.5 ± 0.5
Smoke Point	141.6 ± 2.5	141.6 ± 2.0	179.6 ± 2.0
Pour Point	4.6 ± 1.2	-3.0 ± 0.0	9.6 ± 0.5

Table 3 gives the flash points of the esters as 303.6 °C and 174.6°C for synthetic *Treculia africana* and Synthetic Palm Kernel respectively. These values indicate that these esters can be handled at higher temperature without the tendency of igniting when compared to conventional OBM which is measured 90.5 °C. The cloud points are 17.3 °C for Syn-TA, 5.6 °C for Syn-PK and 5.5 °C for diesel while the smoke point 141.6 °C for Syn-TA, Syn-PK for 141.6 °C and for 179.6 °C. In the same vein its pours are 4.6 °C, -3.0 °C and 9.6 °C for the Syn-TA, Syn-PK and diesel respectively and it means that at cold temperatures Syn-PK pours when compared among the three followed by 4.6 °C.

Table 4
Temperature Versus Viscosity

Temp (°C)	Syn-TA	Syn-PK	Diesel (CentiStokes)
40	40.73 ± 0.25	61.56 ± 0.15	37.33 ± 0.35
100	31.9 ± 0.30	42.56 ± 0.20	26.30 ± 0.31

the relationship is not always linear, generally, an increase in temperature results in a decrease viscosity. A critical examination of these reveals that Syn-Ta, diesel and Syn-PK decreased by 8.33, 11 and 19 centistokes respectively.

Table 5
Temperature Versus Density

Temp (°C)	Syn-TA	Syn-PK	Diesel(g/ml)
40	0.8678	0.8822	0.8281
100	0.8353	0.8514	0.7952

Similarly, temperature increase decreases density as shown in table 5.

Table 6
Fatty acid content of *Treculia africana* Oil

Type of fatty acid		Mean Percentage
C15:0	Saturated Pentadecyclic acid	30.17
C15:1		1.76
C18:0	Saturated Stearic acid	68.08

The fatty acid profile of *Treculia africana* oil as in table 6 shows that saturated stearic acid is dominant with 68.08% and saturated pentadecyclic acid 30.17%.

Table 7
Fatty acid content of Palm Kernel Oil

Type of fatty acid		Mean Percentage
C08:0	Saturated Caprylic acid	1.11
C10:0	Saturated Capric acid	3.83
C11:0	Saturated Undecyclic acid	52.01
C12:0	Saturated Lauric acid	1.32
C13:0	Saturated Tridecylic acid	19.16
C15:0	Saturated Pentadecyclic acid	10.86
C16:0	Saturated Palmitic acid	3.49
C18:0	Saturated Stearic acid	2.98
C18:1n9t	Unsaturated Elaidaic acid	1.08
C18:3n3	Unsaturated Linolenic acid	0.36

On table 7, contrary to the result published by (Wikipedia, 2014) that lauric acid is the most prominent fatty acid in this oil, this result shows that saturated undecyclic acid ranks highest. The reason could be attributed to the GC-FID analysis in which C17 which ideally appears at low concentration in vegetable oils took was significant in the result. This can be further explained by the presence of C17 in the GC standard.

Fourier Transform Infrared Spectroscopy (FTIR)

Most of the peaks below fall 3000cm^{-1} wavelength, an indication of alkyl group (organic molecules).

Comparing the peaks of these spectra, the two samples peak between 3500 – 3200 cm^{-1} and having O – H stretch, H – bonded with the alcohol functional group. Also, between 3400 – 3250 cm^{-1} wavelength was the N – H stretch bond and primary, secondary amines, amides.

Common among them is C – H stretch bond with the alkanes as its functional group and this appears between 3000 – 2850 cm^{-1}. Common among is the C = O stretch and this exists between 1750 – 2850 cm^{-1} and has esters, saturated aliphatic as its functional group. 1500 – 1400 cm^{-1} lies C – C stretch (in – ring) with an aromatic functional group. Similarly, the 1370 – 1350 cm^{-1} has C – H rock bond, alkanes as a functional group and finally between 1300 – 1150 cm^{-1} is C – H (-CH2X) alkyl halides as its functional group. 900 – 675 cm^{-1} has C – H "oop" aromatics functional group and finally 725 – 720 cm^{-1} goes with the C – H rock bond, alkanes as functional group.

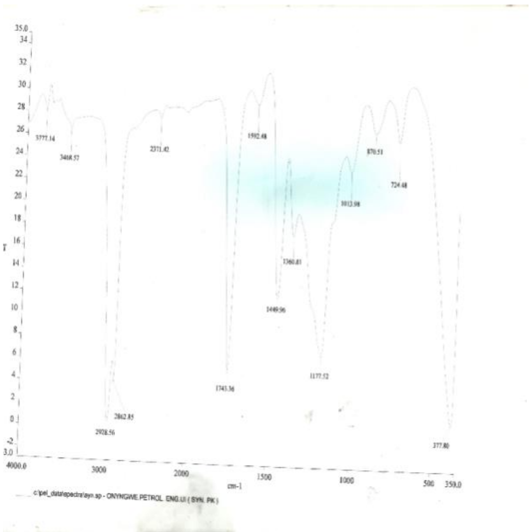

Fig. 2. Syn-Palm Kernel FTIR Spectrum

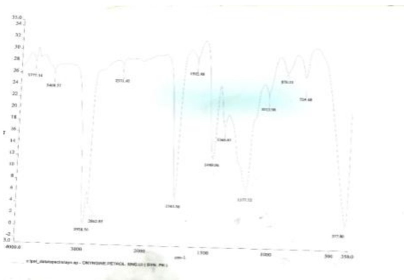

Fig. 1. Syn-*Treculia africana* FTIR Spectrum

Table 8

Rheological properties of Palm Kernel Ester Base Mud

RPM (DIAL READING)	SBF-PK @ 30°C	SBF-PK @ 50°C	SBF-PK @ 70°C	SBF-PK @ 90°C
600	20	18	17.5	16
300	12	11	11	10
200	9	9	9	9
100	6.5	6	6	7
60	5	5	5	5
30	3.5	4	4	4
6	2.5	3	3	3
Apparent Viscosity, cP	20	18	17.5	16
Plastic Viscosity, cP	8	7	6.5	6
Yield Point, lb/100 sq ft	4	4	4.5	4

10-sec. Gel, lb/100 sq ft.	2.5	3	3	3
10-min. Gel, lb/100 sq ft.	3	3.5	3.5	3.5
ES, Volts @ 50°C				392

Table 9

Rheological properties of *Treculia africana* Ester Base Mud

RPM (DIAL READING)	SBF-TA @ 30°C	SBF-TA @ 50°C	SBF-TA @ 70°C	SBF-TA @ 90°C
600	17.5	15	13	10
300	9	8	8	6
200	7	6	6.5	5
100	4	4	5.5	4
60	3.5	3	4	3
30	3	2.5	3.5	2.5
6	2.5	2	2.5	2
Apparent Viscosity, cP	17.5	15	13	10
Plastic Viscosity, cP	8.5	7	5	4
Yield Point, lb/100 sq ft	0.5	1	3	2
10-sec. Gel, lb/100 sq ft.	2.5	2	4	2
10-min. Gel, lb/100 sq ft.	3	2.5	5	2.5
ES, Volts @ 50°C				389

Table 10

Rheological properties of Diesel Base Mud

RPM (DIAL READING)	OBM @ 30°C	OBM @ 50°C	OBM @ 70°C	OBM @ 90°C
600	27.5	26	20.5	13
300	16	17	14	9
200	13	14	12	7.5
100	9	9.5	9	6

60	7.5	8	8	5
30	5.5	7	6.5	4
6	4	4	4.5	3
Apparent Viscosity, cP	27.5	26	20.5	13
Plastic Viscosity, cP	11.5	9	6.5	4
Yield Point, Ib/100 sq ft.	4.5	8	7.5	5
10-sec. Gel, lb/100 sq ft.	3.5	3.5	4	3
10-min. Gel, lb/100 sq ft.	4.5	4.5	4.5	3.5
ES, Volts @ 50°C				468

Considering tables 8, 9 and 10, it can be observed that OBM has the highest viscosity followed by SBF-PK and finally by SBF-TA. With increase in temperature, SBF-TA deteriorated more while those SBF-PK and OBM appears to be at equal space.

Table 11

Sample/Parameter	OBM	Syn-PK	Syn-TA
n @ 30°C	0.70	0.74	0.95
K @ 30°C	84.04	61.72	12.27
n @ 50°	0.61	0.71	0.75
K @ 50°C	193.16	66.99	14.04
n @ 70°C	0.55	0.66	0.69
K @ 70°C	231.27	73.43	89.69
n @ 90°C	0.53	0.68	0.74
K @ 90°C	168.44	73.43	45.46

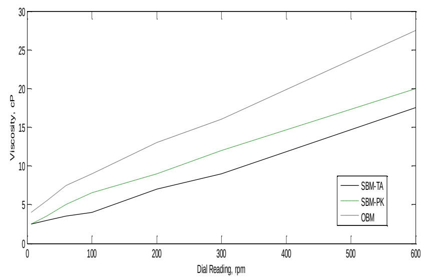

Fig. 3. Dial Reading Versus Viscosities of the Muds at 30°C

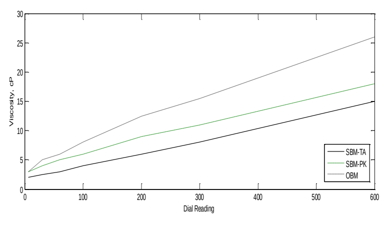

Fig. 4. Dial Reading Versus Viscosities of the Muds at 50°C

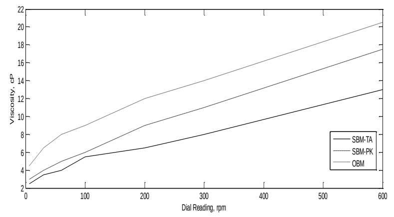

Fig. 5. Dial Reading Versus Viscosities of the Muds at 70°C

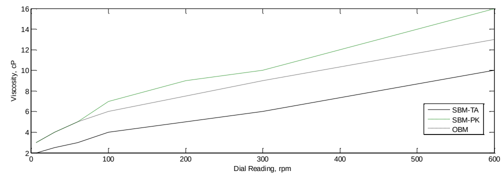

Fig. 6. Dial Reading Versus Viscosities of the Muds at 90°C

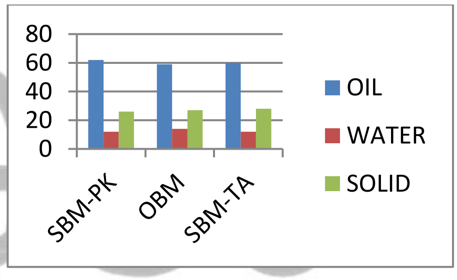

Fig. 7. Oil, Water and Solid Content

Conclusions and Recommendations

4.1 Conclusions

1. The oil yield of the pulverised samples were 28% *for* Palm Kernel and 22% for *Treculia africana*.
2. Ester yield for *Treculia africana* was 96% while Palm Kernel was 86% The better yield resulted from two-steps process of esterification (pre-treatment) preceding transesterification.
3. OBM had a pH of 8.10 at 28.9°C, SBM-PK indicated 8.20 pH at 28.4°C and SBM-TA 8.05 pH at 29.1 and 33.7°C. The alkalinity of the formulated muds means that they would not corrode (damage) downhole equipment.
4. The muds aged were at 30, 50, 70 and 90 degrees Celsius each exhibited variation in their deformation with fossil or

convention diesel based mud most stable.

5. Measurements show that the OBM has 468 Volts, SBM-PK was 392 Volts and SBM-TA indicated 389 Volts.

6. The retort analysis of the formulated muds show that SBM-PK has the highest oil 62%, SBM-TA has 60% while OBM's oil content is 59%. Their water content vary thus: SBM-PK and SBM-TA are both 12% each, OBM possess 14%. SBM-TA has the highest solid content of 28%, next is OBM – 27%, while 26% is for SBM-TA.
7. The formulated muds conform to the Bingham Plastic and Power Law Models and have acceptable rheological properties.

4.2 Recommendations

1. Further research should be carried out on the muds' toxicity, dispersibility and biodegradation to ascertain their true effect on the environment.
2. The mud should be subjected to field test to determine its applicability for drilling operations.
3. The mud were aged at 30°C, 50°C, 70°C and 90°C and the feasibility of older muds may not be guaranteed. Hence, the need to utilise advance technology to age the mud at elevated temperatures to ascertain its true behaviour before a recourse to field application.
4. High pressure high temperature equipment should be used to determine the mud filtration property.

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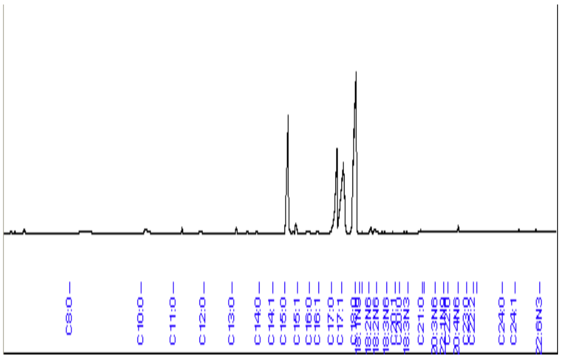
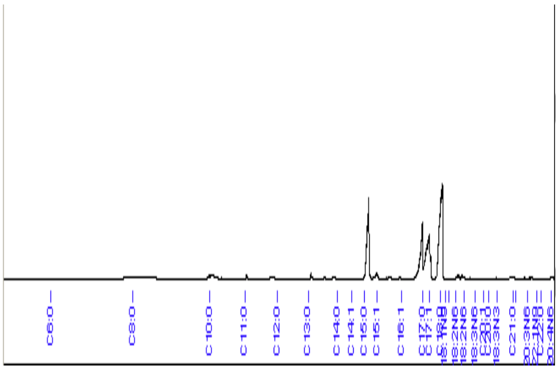
Appendices

Appendix A



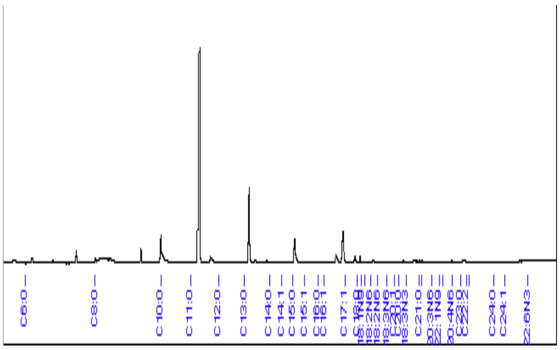
First Fatty Acid Chromatogram of

Treculia africana oil



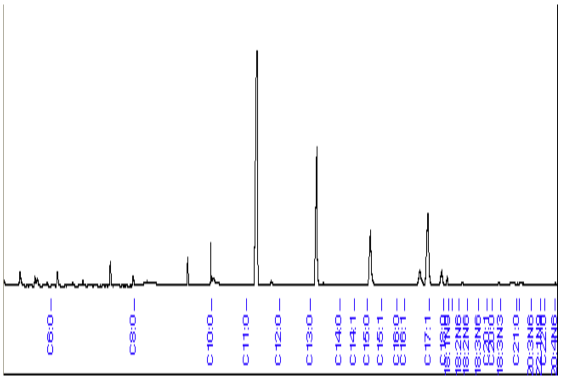
Second Fatty Acid Chromatogram of
Treculia africana oil

Appendix B



First Fatty Acid Chromatogram of

Palm Kernel Oil

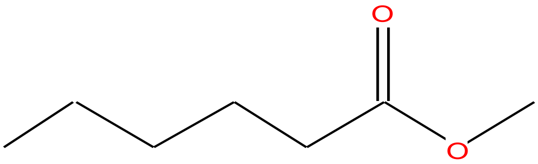


Second Fatty Acid Chromatogram of

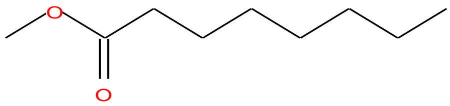
Palm Kernel oil

Appendix C

Structures in Palm Kernel Ester



Hexanoic acid, methyl ester



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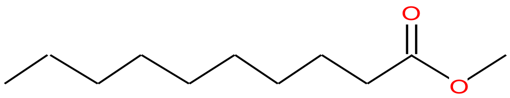
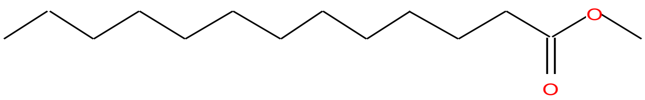
Octanoic acid, methyl ester

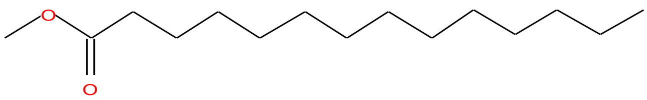


Decanoic acid, methyl ester



143

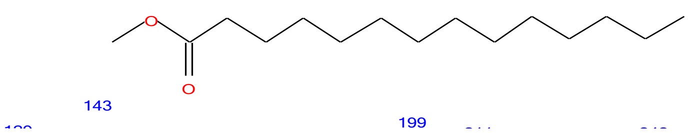
Tridecanoic acid, methyl ester



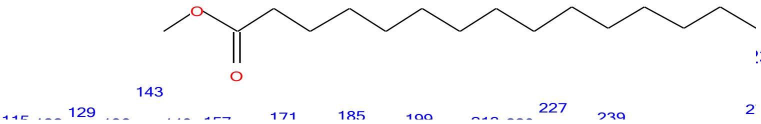
143

199

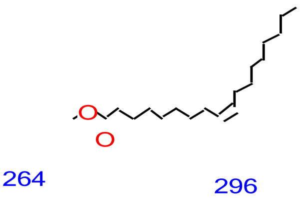
Methyl tetradecanoate



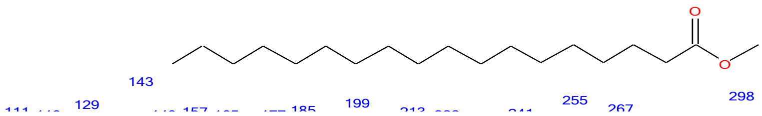
Methyl tetradecanoate



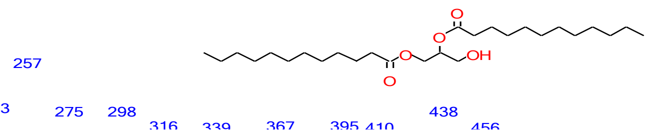
Hexadecanoic acid, methyl ester



9-Octadecenoic acid(Z)- methyl ester

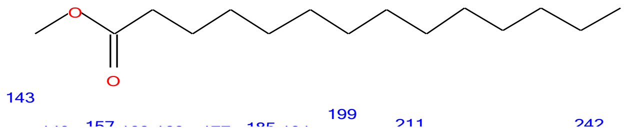


Methyl stearate

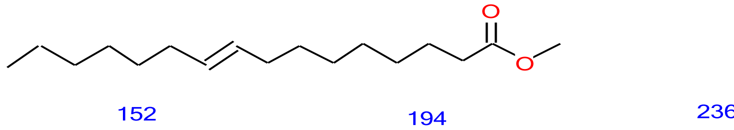


Dodecanoic acid, 1-(hydroxymethyl)-1,2-ethanediyl ester

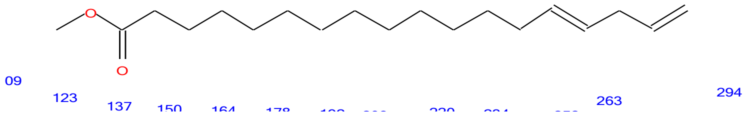
Dodecanoic acid, methyl ester



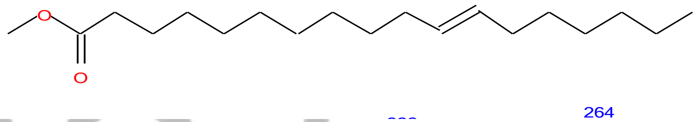
Methyl tetradecanoate



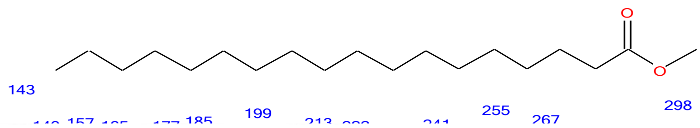
Methyl hexadec-9-enoate



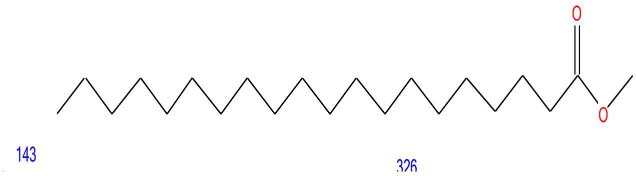
14, 17-Octadecadienoic acid, methyl ester



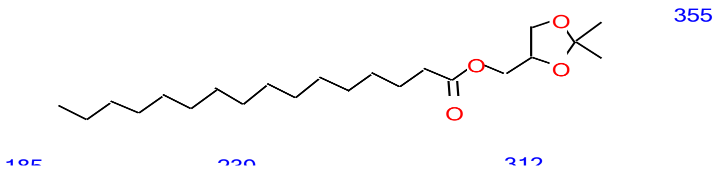
11-Octadecenoic acid, methyl ester



Methyl stearate



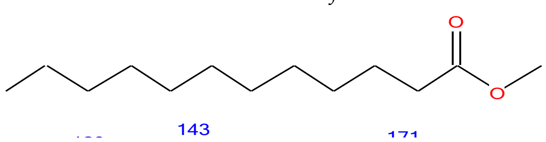
Eicosanoic acid, methyl ester

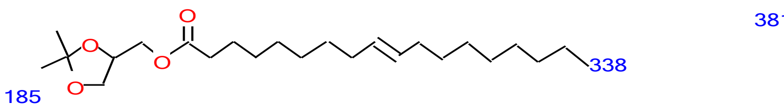


Hexadecanoic acid, (2,2-dimethyl-1,3-dioxolan-4-yl) ester

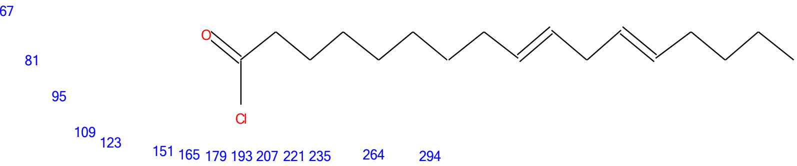
Appendix D

Structures in *Treculia Africana* Ester





Oleic acid (2,2-dimethyl-1,3dioxolan-4-yl) methyl ester



9, 12-Octadecadienoyl chloride, (Z,Z)-