

Comparative Study of *Thevetia peruviana* and *Jatropha curcas* seed oils as feedstock for Grease production

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Abstract: *Thevetia peruviana* and *Jatropha curcas* seed oils were investigated as feed stocks for grease production. Seven different blends of *Thevetia peruviana* and *Jatropha curcas* seed oil were used for this research. The seeds were obtained locally and oven dried until a constant weight was obtained, *Thevetia peruviana* seeds had 3.91 % moisture content while *Jatropha curcas* seeds had 2.93 % moisture content. The oils were thereafter extracted. *Thevetia peruviana* seeds were found to have 58.5 % oil content while *Jatropha curcas* seeds had 54.6 % oil content. The Chemo-physical tests of the oils indicated that the density of the oils were the same (908.7 kg/m³), and the flash point of *Thevetia peruviana* seed oil was 168°C while that of *Jatropha curcas* seed oil was 122°C; the viscosity at 100°C for both oils fell within the range of 7.2 - 7.9 cSt for 400 pale oil 400p, which is of petroleum origin. The viscosity index values for both oils were very high, hence very stable. *Jatropha curcas* oil had a viscosity index of 214 and *Thevetia peruviana* oil had 202. The grease was produced by cooking a calculated quantity of base oil and lithium stearate in a pot at 200°C with continuous stirring for 30 minutes, the reaction was allowed to cool to 85°C before sodium thiosulphate and calcium sulphonate additives were added to the reactor; further milling was maintained to yield a homogeneous product (lithium stearate grease) which were taken for analysis. Five classes of grease were produced from each oil blend; all the grease samples produced, showed no significant difference at 95 % confidence level among blends, but showed a significant difference among the grease compositions. The viscosity temperature coefficient for all the grease samples was very low, this means that all the oil blends and grease compositions were very suitable for the production of semi-solid lubricant (grease). *Thevetia peruviana* and *Jatropha curcas* seed oil and its blends were found to be suitable for the production of different grades of bio-degradable grease and can effectively substitute 400 pale oil 400p grade of fossil origin for grease production.

Keywords: Grease, production, *Jatropha curcas* seed oil *Thevetia peruviana* seed oil, additives, comparative, study, feedstock.

1. Introduction

Most engineering components fail as a result of the failure or loss of the properties of the lubricants used, this loss of properties is primarily due to high temperature and pressure they are subjected to. There is also high level of environmental pollution and degradation causing the much talked about global warming, economic melt down, mutation and ecological extinction. The use of fossil fuel and petrochemical based lubricant are the major contributors of these problems because of the high emission of carbon and sulphur based oxide. The development and formulation of bio-based grease and products will checkmate these problems. Lubricants exist in the three states of matter; gas, liquid and solid. Any material capable of preventing and controlling wear and tear between contacting surfaces is a lubricant (Benton, 1982). Gaseous lubricants include air and neon. Liquid lubricants include: engine oil, gear oil and water based emulsion used as cutting fluids in metal working operations. Lubricants can also be classed as semi-solid/solid which are made by the dispersion of a thickening agent in a liquid lubricant. Grease can be seen as a fine dispersion of an oil insoluble thickening agent, usually soap in a fluid. Lubricant is basically oil. The soap is made up of fatty acid, saponified with alkali which can be hydrated lime, caustic soda,

lithium hydroxide or aluminum hydroxide. The type of soap used depends on the conditions in which the grease is to be used. Different soap provides different levels of temperature resistance, water resistance and chemical reactivity. Thickening agents include metallic soaps, complex soap and hydrophobic clays (Mang and Dresels, 1975). These play important roles in the overall finishing of greases. Bio based semi solid lubricants intends to substitute petrochemical based grease because the long carbon chains present in the bio oils can be broken down naturally to simpler carbon chains of harmless compounds and the fatty acids are used by microorganisms as a source of energy.

The thrust of this work is to make use of bio-based oils to produce grease of comparative efficiency with those made of synthetic oils. *Thevetia peruviana* oil is obtained from *thevetia peruviana* seed; the plant is a dicotyledon which belongs to the family of *aponaceae*, it is a composite ever green shrub, it has a milky sap, it is native to West-indies, Mexico and Brazil, it is known as Yellow Oleander (Ibiyemi *et al*, 2002). The plant is perennial, a shrub reaching 3 to 3.9 metres, the leaves are linear narrow, sword like, the flower is yellow flute (some species are pink in colour), which develop to a fruit which has a pair of follicle. It has one to four compartments

each containing a seed. The fruit, when unripe is hard and green, but gradually turns black as it ripens. It is propagated by seed and stem, the plant fruits ten out of the twelve months of the year, the seed contains about 60-64 % oil on dry matter basis. The plant produces white latex (sap) that is highly poisonous, the seed is also poisonous. The plant thrives very well in all the climatic and vegetation belts of Nigeria, readily found in all the states: Rivers, Sokoto, Borno, Benue, Kwara, etc. (Ibiyemi, 2008). *Thevetia peruviana* oil is not edible; it maintains its properties at 220 °C, which makes it an important choice for engineering base oil (Ibiyemi *et al.*, 2002) while *Jatropha curcas* is a drought resistant, perennial plant, growing well in marginal /poor soil. It is easy to establish, grows relatively quickly, producing seeds for 50 years. The oil can be combusted as fuel without being refined. It burns with clear smoke free flame, tested successfully as fuel for simple diesel engine, the oil is not edible. The by product of pressed cake is a good organic fertilizer; oil also contains insecticide. The trees are deciduous, shedding the leaves in the dry season. Flowering occurs during the wet season. The seeds mature about three months after flowering. It is still uncertain where the plant originated from, but it is believed to be Mexico and Central America. It has been introduced to Africa and Asia and is now cultivated world

wide. It grows in the tropics as well as temperate regions. It is a small tree or shrub with smooth gray bark, which exudes a whitish colored, watery, latex when cut, it is poisonous.

Extensive research has been made on *Thevetia peruviana* seed oil, the oil was studied for its thermal stability, the oil was heated to 180, 200 and 220 °C over a period of 15 hours and there was no significant difference in the five parameters (iodine value, acid value, peroxy values, saponification value, and amount of polar compounds) studied. The study proved that the seed oil would be stable to heat at those temperatures. This property also justifies the use of the seed oil in the making of lubricants and greases (Ibiyemi *et al.*, 2008). In this study, research and emphasis have been focused on the suitability and properties of grease produced from *Thevetia peruviana* and *Jatropha curcas* seed oils. Works have been recorded for the use of other biodegradable oils for the production of greases. The works of Awoyale *et al.* (2011) reported the successful production and utilization of black date oil as biodegradable lubricant.

2. Materials and Methods

Thevetia peruviana and *Jatropha curcas* seeds were locally collected from Makurdi town, Benue State of Nigeria. The seeds

obtained from the fruits and capsules were washed and oven-dried at 100°C at an hourly interval until a constant weight was obtained. The moisture-free seeds were ground using a grinding machine and oil extracted from it using Soxhlet apparatus. The rotary evaporator was used to recover the solvent (n-hexane). *Thevetia peruviana* seed oils and *Jatropha curcas* seed oils have similar extraction processes. The moisture – free *Thevetia peruviana* seeds were ground with a grinding machine. The finely ground grain were put into the thimble in an extraction chamber of sohxlet apparatus. Two hundred millilitres (200 ml) of normal hexane was charged into the round bottom flask (500 ml) of the Soxhlet apparatus and mounted in the heating mantle of the apparatus and was heated to between 65-100°C in order to prevent loss of hexane vapour because the boiling point of n-hexane is 69°C. The evaporating n-hexane was condensed into the thimble by the condenser where it leached the oil out of the paste. The extracted oil was taken for analysis and characterization at A-Z

Petroleum Products Limited Nnewi, Anambra state of Nigeria for densities, flash points, pour points, kinematic viscosities, appearance/colours, viscosity indexes, and cloud points. These are some of the parameters with which base oils are identified as recommended by National Lubricating Grease Institute (NLGI) and American Oil Analytical Chemists (AOAC, 1984).

The extracted oils were blended into seven grades and labeled A,B,C,D,E,F and G at the proportions of 100:0, 0:100, 20:80, 80:20, 40:60, 60:40 and 50:50 respectively (See Table 1). Each blend of oil was used to produce five grease types with different thickener and additive composition. Thirty five grease samples were produced and characterized at A-Z Petroleum Product Limited, Nnewi, Nigeria. Mechanical application was carried out using the counting gears of a fuel dispensing machine and the bearing of a simple grinding engine. Each blend was used to produce grease with same empirical composition of components as shown in Table 2.

Table 1: Composition of base oil blends for grease production

Blends	Sample	<i>T. peruviana</i> (g)	<i>J. curcas</i> (g)	Total (g)
A (100:0 %)	1	300	0	300
	2	284	0	284
	<i>T. peruviana:</i>	264	0	264
	<i>J. curcas</i>	252	0	252
	5	228	0	228
B (0:100 %)	1	0	300	300
	2	0	284	284
	<i>T. peruviana:</i>	0	264	264
	<i>J. curcas</i>	0	252	252
	5	0	228	228
C (20:80 %)	1	60	240	300
	2	56.8	227.2	284
	<i>T. peruviana:</i>	52.8	211.2	264
	<i>J. curcas</i>	50.4	201.6	252
	5	45.6	182.4	228
D (80:20 %)	1	240	60	300
	2	227.2	56.8	284
	<i>T. peruviana:</i>	211.2	52.8	264
	<i>J. curcas</i>	201.6	50.4	252
	5	182.4	45.6	228
E (40:60 %)	1	120	180	300
	2	113.6	170.4	284
	<i>T. peruviana:</i>	105.6	158.4	264
	<i>J. curcas</i>	100.8	151.2	252
	5	91.2	136.8	228
F (60:40 %)	1	180	120	300
	2	170.4	113.6	284
	<i>T. peruviana:</i>	158.4	105.6	264
	<i>J. curcas</i>	151.2	100.8	252
	5	136.8	91.2	228

	1	150	150	300
G (50:50 %)	2	142	142	284
<i>T.peruviana:</i>	3	132	132	264
<i>J.curcas</i>	4	126	126	252
	5	114	114	228

Table 2: Empirical composition of components of developed grease

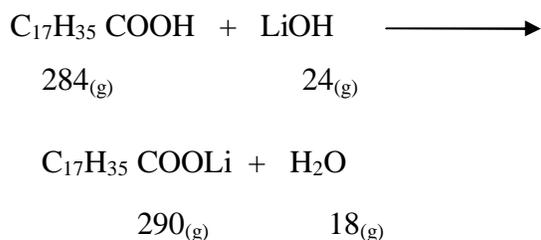
Sample	Base oil (g)	Stearic acid (g)	Lithium hydroxide (g)	Water evaporated (g)	Additive (g)	Total (g)
1.	300	78.344	6.624	4.968	20	400
2.	284	97.93	8.28	6.21	16	400
3.	264	117.516	9.936	7.452	16	400
4.	252	137.102	11.592	8.694	8	400
5.	228	156.688	13.248	9.936	12	400

2.1 Preparation of Thickening Agents and production of grease

Thickening agents' preparation is carried out by saponification reaction which is a process of reacting fatty acids with metals/metallic hydroxides to produce soap and water at a temperature of about 160°C. The water

produced as a by-product of the reaction is evaporated due to very high reacting temperature of the mixture. The soap produced under this manner is referred to as thickening agent in the lubrication industry. In this study, the thickening agent used was lithuim stearate (lithuim soap) which is a simple soap.

Lithium stearate was produced as a result of the reaction between lithium hydroxide and stearic acid. The stoichiometry is as shown:



Formulation of five different samples of grease was done in accordance to NLGI specifications for each oil blend. For each sample, proportions of base oil and lithium stearate (thickening agent) were varied. Table 3 shows the different proportions of components of the samples of the grease produced. Different quantities of additives were added to these formulations. The base oil and lithium stearate mixtures were cooked at temperature 200°C with continuous stirring. The mixtures were reacted with respect to the proportions as shown in Table 3 while Table 2 shows the empirical composition of the basic components of the developed grease samples. From literature the combination of sodium thiosulphate (0.5 – 6 %), calcium

sulphate (0.2 – 3 %) and 1,2,3-benzotriazole (0.2 – 3 %) is an ideal composition of additives for grease production. But for the purpose of this research, only sodium thiosulphate and calcium sulphonate were used as additives at the ratio of 7: 3 as 1,2,3-benzotriazole was not commercially available.

The mixture of the base oil and the lithium stearate after being thoroughly cooked and blend for 30 minutes at 200°C was allowed to cool to 85°C before the additive was added to the blend; the mixture was continuously stirred until the temperature reached room temperature of 35°C. At room temperature, further milling of the mixture was done until a homogenous substance was obtained. The homogeneous substance obtained was the lithium stearate grease which was then taken for further analysis and characterization. The analysis and characterization of the developed grease samples were based on ASTM specifications. The analysis carried

Table 3: Proportions of Components of Grease Samples

Sample	Lithium Stearate (g)	Base oil (g)	Additive (g)	Total (g)
i.	80=20%	300=75%	20=5%	400
ii.	100=25%	284=71%	16=4%	400
iii.	120=30%	264=66%	16=4%	400
iv.	140=35%	252=63%	8=2%	400
v.	160=40%	228=57%	12=3%	400

2.2 Analysis and characterization of grease

out included, the cone penetration consistency test (ASTM D-217), dropping point of lubricating grease over wide temperature range test (ASTM D-2265), copper strip corrosion test (ASTM D-130) and apparent viscosity (ASTM D1092). The Equation for the dropping point test is $DP = ODP + ((BT - ODP)/3)$ (7)

3.0 Results

Table 4: Oven - dried *Thevetia peruviana* and *Jatropha curcas* seeds characteristics

Time (minutes)	Mass of <i>Thevetia peruviana</i> (kg)	Mass of <i>Jatropha curcas</i> (kg)
0	11.24	14.65
60	10.99	14.44

Where: DP - dropping point; ODP - Thermometer reading when first drop reaches the bottom of the test tube; and BT - block temperature when the drop falls.

2.3 Mechanical application

Grease of grade 2 was applied on the bearing of a grinding engine and grease of grade 1 was also applied on the counting gears of a fuel dispensing machine. The bearing and the gears were observed for two weeks for lubricity, flowability, caking and other visible defects.

120	10.83	14.29
180	10.81	14.23
240	10.80	14.22
300	10.80	14.22
360	10.80	14.22

% moisture content for *Thevetia peruviana* = 3.91; % moisture content for *Jatropha curcas* = 2.93

Table 5: Percentage Oil Extracts

	<i>Jatropha curcas</i>	<i>Thevetia peruviana</i>
Qty. of seeds used (kg)	10.7	10.8
Qty. of oil extracted (kg)	5.85	6.32
Oil yield (%)	54.67	58.518

Table 6: Chemo- physical Properties of *Jatropha curcas* and *Thevetia peruviana* Seed oils

Properties	Test methods	<i>Jatropha curcas</i> Oil	<i>Thevetia peruviana</i> Oil
Density (Kg/m ³)	ASTM D1298	908.7	908.7
Flash point (°C)	ASTM D92	122	168
Pour point (°C)	ASTM D97	-5	8
Kinematic Viscosity at 40 ⁰ C (cSt)	ASTM D445	30.59	35.47

Properties	and	ASTM D445	400 pale oil 400p	Bright and	<i>Jatropha curcas</i>	Bright	<i>Thevetia peruviana</i>
At 100 °C (cSt)			7.23				7.84
colour							
Flash point (maximum) °C			210			122	168
Viscosity Index	ASTM D2270		214			202	
Pour point (minimum) °C			-9			-5	8
Cloud point (°C)	ASTM D2500		3			12	
Kinematic Viscosity at 100 °C			7.2-7.9			7.23	7.84

Table 7: Mechanical Application test of the Produced Grease

Grease grade	Application	Wear	Texture	Visible Defect	Rust	Oil leakage
1	Gear	none	normal	none	None	None
2	Bearing	none	normal	none	None	None

Table 8: Comparison of Base oil 400 pale oil 400p grade with *Jatropha curcas* and *Thevetia peruviana* seed oils

(c.St)			
Viscosity Index	75	214	202
Appearance	Bright and Clear	Bright and Clear	Bright and clear

4. Discussion

Tables 4 and 5 are the percentage moisture content and the percentage oil yields of the two oil seeds studied, the moisture content of *Thevetia peruviana* seeds (3.91%) is higher than that of *Jatropha curcas* seeds (2.93 %). The difference could be as a result of the thick shell covering the nuts and the size of the seeds. Also the moisture contents of seeds are influenced by time of harvest, duration of exposure to sun drying and maturity of the seeds before harvesting/plucking. The percentage oil yield is higher in *Thevetia peruviana* seeds than in *Jatropha curcas* seeds, *Thevetia peruviana* seeds oil yield was found to be 58.5 %, a value lower than the expected value of 64 % as presented by Ibiyemi *et al* (2002). *Jatropha curcas* seeds oil yield stood at 54.6 %, this value is also lower than the expected of 63 % (www.jatrophabiodiesel.org). The difference

in value could be as a result of plant species, ecological factor, processing methods, or experimental error. Tables 6 presents the results of the chemo-physical properties of *Thevetia peruviana* and *Jatropha curcas* seed oils; the density of the two seed oils were the same 908.7 kg/m³. The flash point of *Thevetia peruviana* seed oils (168 °C) was higher than that of *Jatropha curcas* seed oils (122 °C). It means that *Jatropha curcas* is more volatile than *Thevetia peruviana*. Oil with very high flash point can withstand high temperature operations and safe for handling and haulage. However it is of limited significance as far as grease lubrication is concerned because the additives, thickeners, viscosity improvers play a major part in increasing or reducing the flash point of the lubricant (Spencer, 2002). The pour point of *Jatropha curcas* seed oils (-5 °C) was lower than that of *Thevetia peruviana* seed oils (8 °C). This suggests that *Jatropha curcas* oil can

withstand extreme cold condition more than *Thevetia peruviana* oil; this is an indication that *Jatropha curcas* seed oils will thrive better in temperate region and can transverse across regions without been affected by cold climates unlike *Thevetia peruviana*. It will have an easier start up than the later (Spencer, 2002). The kinematic viscosity at 100 °C for *Thevetia peruviana* seed oils (7.84 c.St) was higher than that of *Jatropha curcas* seed oils (7.23 c.St), it means that *Thevetia peruviana* seed oils resist flow more than *Jatropha curcas*. The low value of kinematic viscosity of *Jatropha curcas*, justifies its unsaturated nature and its ability to be combusted as fuel without been refined in a simple diesel engine (Nouredimi *et al* 1992 and www.jatrophabiodiesel.org). The kinematic viscosity at 100 °C for both *Thevetia peruviana* seed oils (7.84 c.St) and *Jatropha curcas* seed oils (7.23 c.St) fell within the range (7.2 - 7.9 c.St) for kinematic viscosity of 400 pale oil 400p which is one of the grades of lubricating base oil derived from petroleum origins as shown in Table 8.

Viscosity index of *Jatropha curcas* seed oil is 214 which is higher than that of *Thevetia*

peruviana (202). A low number indicates oil which changes viscosity rapidly with temperature, whereas, a high number indicates oil which changes viscosity little when heated or cooled; for both oils the viscosity indexes were very high (Bennett, 2004), which means that both seed oils are very stable with temperature fluctuations, *Jatropha curcas* seed oils is more stable. A lubricant should change its viscosity as little as possible so that it is not too thick when cold thereby making start-up to be very difficult and not too thin when hot, thereby running off the surface (Harris, 1990 and Spencer, 2002). The viscosity index of 400 pale oil 400p is 75; this base oil is of fossil origin. The brightness and clarity of both seed oils were superb.

5. Conclusion and Recommendation

The results of the analysis clearly shows that all the oil blends are suitable for the production of different grades of bio-degradable grease. The produced samples of grease are non corrosive, hence it can be used in different material surfaces and

engineering application. The seed oil content of both *Thevetia peruviana* and *Jatropha curcas* are relatively high. The grease produced did not have any significant difference in the quality among the blends. From the data and results obtained, grease samples of pure *Jatropha curcas* base oil has the highest dropping point, it can withstand higher operating temperatures and hence the best grease blends. The quantity of additives in the grease is best between 5 – 4 % of the total mass of grease. The dropping point temperature of these grease produced met the regular grease standards (80 – 200 °C). The viscosity temperature coefficient for all the grease samples are very low and the viscosity index of both seed oils are very high, this means that all the oil blends and grease compositions are very suitable for the production of semi-solid lubricant (grease), The use of these products in engineering application was also successful. *Thevetia peruviana* and *Jatropha curcas* seed oils can be used to substitute 400 pale oil 400p grade (petroleum base) for grease production. The two seed oils studied can therefore be registered as natural sources of bio lubricants which no doubt will find uses in aquatic Engineering and other applications.

The indispensability of their application in the modern demands for bio based products can no longer be ignored as the urgent needs for technological shift from fossil fuels continue to be an imperative.

Other test such as load carrying capacity (Timken test) D2509, wear preventive characteristic (Four ball test) D2266, evaporation loss of lubricating grease and oil D972 are also important, but some of these tests are not performed locally, an institute in Ghent Belgium runs most of these tests. Therefore further work to involve the aforementioned test should be carried out. The combination of sodium thiosulphate, calcium sulphonate and 1,2,3 Benzotriazole was recommended in literature, but only sodium thiosulphate and calcium sulphonate was used due to the non-availability of 1,2,3 benzotriazole within the country, HKN chemical limited India will make it available if at least a ton of the product was demanded, this additive could improve the dropping point and quality of grease, these three chemical additives should be used (Ozaki *et al*, 2003). These grease samples should further be subjected to different

severe operating conditions in engineering

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