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OPTIMIZATION OF THE COMPOSITION AND PROCESS VARIABLES FOR THE PRODUCTION OF SEMI-SOLID LUBRICANT FROM JATROPHA CURCAS SEED OIL

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ABSTRACT

In this study, the process (temperature and time) and the composition (base oil, additive, and thickeners) variables were optimized using mat lab statistical tool. The result show that the Jatropha curcas seed oil was suitable for the production of grease. Low temperature and time of production operation favor low viscosity temperature coefficient and high dropping point temperatures which characterizes a good quality grease. The optimum quality was obtained with 20.00g or 5% selected additives, 120.00g or 35% thickener, 240.00g or 60% base oil and produced at temperature and time of 160^0C and 20 minutes respectively. The produced bio grease has BOD (16-20 PPM) making it biodegradable and hence environmentally friendly.

Keywords: Jatropha curcas, Mat lab, semi solid, Lubricant, optimization

1. INTRODUCTION

The over dependence on fossil oil and its products for technological advancement has brought so much trouble to the society in the areas of global warming, mutation and ecological extinction. The emergence of renewable sources of raw material was welcomed and accepted by researchers and engineers as the only way to tackle environmental degradation effectively, as well as reduce the over dependence on fossil oils [1]. The demand for grease continues to increase as the world population is increasing; this has necessitated research interest in the production of goods with natural raw materials for the purposes of resource conservation and environmental sustainability. This is generally called green technology and it has come to substitute fossil based technology in so many areas such as fuel, lubricants, agro-allied materials etc. [2]

The word lubricant, originates from a Latin word “lubricare” meaning to make slippery. It has a principal function of reducing friction and wear between contacting surfaces as well as providing heat transfer action, suspending contaminates, preventing corrosion and rust. The knowledge of lubricant dates from the early history in Egypt, Greece, Rome and Persia, where grease were made of animal fat and calcium, used to lubricate chariot wheels [3]. Grease may be defined as a semi fluid, semi-solid lubricant produced by the dispersions of a thickening agent in liquid-base oil, the addition of chemical substances (additives) is to improve, enhance the desired properties and quality [4]. They are non-Newtonian lubricants which will not flow under their own weight [5].

Some grease are more viscous when worked [6] The bases for blending the components together however depend on the type of grease to be produced by the manufacturer. But generally, base oil takes the highest percentage of blending proportions because it imparts the lubricating ability into the grease structure.[7] This is followed by the thickener, the amount of which determines how hard or soft the grease will be and the additive is added to enhance the final quality of the product. The base oil which the majority of grease available today is made from is petroleum.

This raw material is not renewable and as such will get depleted in near feature as a result of global rise in population, economic and industrial activities. It is therefore very imperative to seek for alternative raw materials as a substitute for base oil in grease production.

Jatropha curcas is a drought resistant perennial plant growing well in marginal /poor soil. It is easy to plant, grows relatively quickly, producing seeds for 50 years [8]. Jatropha plant produces seeds with an oil content of 37% [9]. 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 byproduct are pressed cake, a good organic fertilizer [1]. 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 grows in tropics as well as temperate regions.

In this work, grease was produced from Jatropha curcas seed oils by blending with base oil and the process optimized using MATLAB statistical tool.

2. MATERIALS AND METHODS

Weighing balance, Oven (Memmert UF 450), Tray, Hand hammer. Grinding machine, 500ml sohxlet apparatus, water bath, Beakers. Open cup flash point analyzer, Hydrometer, U-tube viscometer, viscosity index chart. Sensitive weighing balance, Adjustable heating element, stirrer, reactor. Lithium hydroxide, stearic acid, n-Hexane, Sodium thiosulphate, Calcium Sulphate, 1, 2, 3 Benzotriozole. Penetrometer (11045-01), Aluminum block oven, Brookfield viscometer (CAP1000+) plastic cups, heating mantle.

Preparation and extraction of the Jatropher carcus oil

Jatropha curcas seeds were locally collected in Makurdi town, Benue State Nigeria. The seeds obtained were washed and oven dried at 1000C at an hourly interval until a constant weight was obtained. The moisture free seed were ground using a grinding machine; this is to increase the total surface area for leaching operation to take place effectively.

The ground seed was placed inside the soxhlet apparatus, using n-Hexane for the extraction, the extracted oils were taken for analyzed for the following parameters: Density, Flash point, Kinematic viscosity, Viscosity Index and Color.

Experimental design

The experiment was designed to work 12 runs and 2 trials as shown in table 1. The design of experimental was structured this way because the major aim was to optimize grease production. The production entails both compositional and production variables, the composition variables include, Additives, thickeners, and base oils, while the production variables include, the production temperature and the duration (time) of cooking. These variables were used to optimize the process, while the base oil was kept constant throughout the production process Table 2.

Table 1: The design plan for the work with 12 runs and 2 tries

dRE1 = row exch (4,12;purequadractic';tries';2)

Additives	Thickener	Temperature	Time
-1	0	1	-1
0	0	0	0
-1	-1	0	1
0	1	0	-1
0	-1	1	0
1	1	1	1
-1	1	-1	-1
0	0	-1	1
1	0	0	-1
1	-1	-1	-1
-1	0	-1	0
1	1	0	0

Table 2: Composition and production variables prescribed by the design of experiment for production of grease.

S/N	Additives		Thickener		Temp (oc)	Time (min)	Base oil (g)
	%	(g)	%	(g)			
J1.	0	0	35	140	200	20	240
J2.	5	20	35	140	180	30	240
J3.	0	0	30	120	180	40	240
J4.	5	20	40	160	180	20	240
J5.	5	20	30	120	200	30	240
J6.	10	40	40	160	200	40	240
J7.	0	0	40	160	160	20	240
J8.	5	20	35	140	160	40	240

J9.	10	40	35	140	180	20	240
J10.	10	40	30	120	160	20	240
J11.	0	0	35	140	160	30	240
J12.	10	40	40	160	180	30	240
Castrol grease							

Analysis of the oils

The following standards were used for the analysis: Density, ASTM D1278 [10] ; Flash point, ASTM D92 [11] ; Kinematic viscosity at 40^{O}c and 100^{O}c , ASTM D445 [12] ; Appearance and color , ASTM D417 [13] and Viscosity index , ASTM D22070 [14]

Blending of Base Oils

A constant mass (240g) of the base oil was used for the entire production of the grease samples table 2.

Preparation of Thickening Agent.

The thickening agent, Lithium Stearate was prepared through a saponification process following the method described by [15]. Stearic acid was added into a certain amount of base oil and heated with constant stirring to about 80-100°C till the acid was fully dissolved in the base oil. The lithium hydroxide solution was diluted to the concentration of I: 6 of LiOH and water respectively and maintained at 100°C-110°C for 2h to ensure complete reaction. The water produced as a by-product of the reaction is evaporated at a temperature of 160 ^{O}C and allowed for 30 minutes The reaction is shown below:

Stearic Acid + Lithium Hydroxide = Lithium stearate + water

$$C_{17}H_{35}COOH + LiOH \longrightarrow C_{17}H_{35}COOLi + H_2O \quad (2).$$

Preparations of Additives

Two samples of the following additives were used.

1, 2, 3 benzotriazole (8g) + Sodium thiosulphate (20g) **+ Calcium** sulphate (12g)= 40g

1, 2, 3 benzotriazole (4g) + Sodium thiosulphate (10g) ++ Calcium sulphate (6g) =20g

Formulation and Production of Grease

Formulation of twelve (12) different samples of grease was done in accordance to the design of experiment and conditions for each grade as shown in table 3

The grease was produced by cooking the base oil (240g) and the thickener (120g) for forty (40) minutes at 200^{0}c with continuous stirring, this temperature was maintained by using the thermometer and adjusting the heating element; after he stated time, the heat was removed and the stirring continued until the composition was cooled to 85^{0}c, then 20g of additives(selected chemicals) was added, the stirring continuous until the semisolid product was obtained, then stored in a separate container waiting for analysis. Other grease samples were produced based on their corresponding compositions and a production conditions.

Characterization of grease samples

The characterization of the grease includes cone penetration, dropping point temperature, apparent viscosity test and biological oxygen demand.

Determination of the consistency (cone penetration) of the grease

The Penetrometer (11045-01) was used to determine the consistency at M.R.S Apapa Lagos, former Texaco Nigeria. Sufficient amount of grease was put in the grease Jarring cup which was severed in order to eliminate any air entrapped within the grease. The vent cork was released and retightened in order to ensure that appropriate pressure was maintained during the working of the grease. The grease was then subjected to 60 full double strokes of the plunger which was completed in one minute. This is referred to as Grease Working. The plunger was then removed and the grease cup was taken to the penetrometer table where the cone shaft of the penetrometer was released into the grease in the cup for 5 seconds during which it penetrated the grease. The penetration was read off from the penetrometer's indicator. This procedure was then repeated for the remaining samples of the grease. The consistency numbers of the grease were determined from the penetration depth, see table 5.

Determination of dropping point temperature of grease

Aluminum block oven equipment is used in determining the dropping point of grease samples. This experiment was performed at M.R.S Apapa Lagos, former Texaco Nigeria.

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The cup, cup support and test tube were thoroughly cleaned with mineral spirit to ensure that the cups were free of any residue. Empty test tubes were inserted in every test tube compartment and a thermometer having a range from -5 to 400 ^{0}C was also fixed in the thermometer well of the oven. The oven was turned on and the present temperature was noted. Test tubes and accessories were selected and used to minimize possible wobbling of the thermometer. All components must be at room temperature prior to the test. The grease cup was filled by pressing the larger opening in to the grease to be tested until the cup was filled. The thermometer assembly and depth gage was removed from the tube, the grease cup was placed on the cup support in the test tube and the thermometer assembly was carefully reinserted, no further adjustment was made as the thermometer bulb was now positioned to provide adequate clearance between the tip of the bulb and grease samples in the cup. Tilting of the cup was avoided to prevent erroneous value. When the first drop of material fell free from the cup orifice and reached the bottom of the test tube, temperature value is read on the thermometer and were recorded to the nearest degree as the dropping point temperature of the grease.

Apparent Viscosity

Broke field viscometer equipment was used for the determination of apparent viscosity. Grease samples were put into the 100ml glass cup to about three quarter the glass volume using a spatula, the glass cup containing the grease sample was placed in a heating mantle, thermometer was used to take the temperature of the grease samples, the tip of the thermometer did not touch the bottom of the cup, immediately the temperature reached +5 ^{0}C of the desired temperature, the sample was removed from the heating mantle and was taking to the Broke field viscometer for the sample reading. The desired temperatures were 40 ^{0}C and 100 ^{0}C respectively. Spindle number 4 and speed 30 rev/min was selected for this test, the heated sample was positioned in such a way that the spindle of the viscometer was well sunk in to the grease sample, the viscometer was switched on and was maintained on hold for about a minute for stability before turning it to sample and immediately digital values starts to display on the screen, any value that appears twice repeatedly was recorded as the Apparent Viscosity of that sample. For each grease sample the test was done at 40^{0}C and 100 ^{0}C. The viscosity temperature coefficient was also evaluated from the corresponding values of apparent viscosities at 100^{0}C and 40^{0}C respectively.

Test for Biodegradability

The test was carried out at the research laboratory of the Chemical Engineering Department of the Enugu University of Science and Technology. Biochemical oxygen demand (BOD) is the amount of oxygen used by microorganism as they decompose the organic matter in a given sample over a period of time and at a particular temperature [7]. This test was done as described by [7]

Table 3: Empirical composition of components of developed grease and their associated production variables for oil

S/No	Base Oil (g)	Stearic Acid (g)	Lithium Hydroxide (g)	H_2O Evaporation (g)	Additives (g)	Temp (0c)	Time (min)
J1.	240	137.102	11.592	8.694	0	200	20
J2.	240	137.102	11.592	8.694	20	180	30
J3.	240	117.516	9.936	7.452	0	180	40
J4.	240	156.688	13.248	9.936	20	180	20
J5.	240	117.516	9.936	7.452	20	200	30
J6.	240	156.688	13.248	9.936	40	200	40
J7.	240	156.688	13.248	9.936	0	160	20
J8.	240	137.102	11.592	8.694	20	160	40
J9.	240	137.102	11.592	8.694	40	180	20
J10.	240	117.516	9.936	7.452	40	160	20
J11.	240	137.102	11.592	8.694	0	160	30
J12.	240	156.688	13.248	9.936	40	180	30

3. RESULT AND DISCUSSION

Table 4: chemo- physical properties of *Jatropha curcas* Seed oil

Properties	Result
Density (Kg/m^3)	907.5
Flash point (^{0}C)	127
Kinematic Viscosity at 40^0 C (cSt)	30.40
At 100 ^{0}C (cSt)	7.22
Appearance and color	Clear
Viscosity Index	215

Chemo-Physical properties of the oil

Table 4 presents the result of the chemo-physical properties of Jaropha curcas seed oil. The density is 907.5Kg/M^3, the high density is advantageous in semi-solid lubricant production. The flash point is (127 ^{0}C), it simply means that it can withstand high temperature operations. This high flash point may not be very important in the production of grease, because other components of grease may affect this property [16].

Table 5: Results of grease from (100% *Jatropha curcas* seed oil).

S/N	NLGL Consistency no.	Cone Penetration (C.P) unworked penetration (0.1mm)	Dropping Point (D.P) ^{o}C	Apparent viscosity at 100^{o}C (cSt)	Apparent viscosity at 40^{o}C (cSt)	Viscosity Temperature Coefficient (V.T.C)	Biological oxygen demand (BOD) Ppm
J1	2	271	125	2722	3902	2	19
J2.	2	280	160	2753	3312	0.1687	16
J3.	2	269	118	2839	4001	0.2904	17
J4.	3	235	164	2769	3298	0.1602	18
J5.	2	281	160	2763	3320	0.1679	20
J6.	3	240	163	2813	3390	0.1702	17
J7.	3	248	123	2742	3980	0.3110	18
J8.	2	279	159	2752	3320	0.1711	19
J9.	2	271	161	2863	3409	0.1601	17
J10.	2	268	160	2817	3390	0.1691	16
J11.	2	270	118	2805	4002	0.2990	18
J12.	3	237	162	3283	3980	0.1752	17
NIS Standard	000 – 6	265- 295	200- 260	0.8- 200	1- 2000	0.1023	2

The kinematic viscosity at 100 ^{0}C is 7.22 cst, this simply means that it resists flow naturally and it's values fell within the range 7.2-7.9 cst for 400 pale oil 400p grade, which is one of the grade base oil derived from petroleum origin used for production of lubricants. Viscosity index is 215 this property is important in lubrication, it indicates oils which changes viscosity easily with temperature change; a low number indicates oil which changes viscosity rapidly with temperature, while a high viscosity index value indicates oil which change viscosity little when heated or cooled, slow change in viscosity of an oil when heated or cooled is very important in lubrication so that it is not too thick when cold or too thin when hot [17]. The oil is bright and clear.

Characterization of Grease Samples

The results of the dropping point temperature, cone penetration depth, apparent viscosity and viscosity temperature coefficient, biological oxygen demand for all the grease samples J1 to J12 produced are presented in Table 5. The results shows that the consistency and cone penetration meet the standard specification for a good quality grease. The dropping

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point temperature for all the samples ranges from 118 - 164 °C which are not up to 200- 260Oc for standard quality [18, 19]

Effect of Additives on the probability plot of viscosity temperature coefficient of all the greases produced from 100% Jatropha curcas seed oils

Figure 1 shows the effect of different quantities of additive on a grease sample,(0.00g) Zero gram additives presents viscosity temperature coefficient higher than 0.3, while 20.00g and 40.00g additives presents viscosity temperature coefficient results that are almost the same, about 0.17. The viscosity of a lubricant generally decreases with decrease in density which occurs when temperature increases; viscosity temperature coefficient is a product of the apparent viscosity of grease at two distinct temperatures, the smaller the viscosity temperature coefficient, the smaller the relative change in viscosity with temperature and the smaller the relative change in lubricant viscosities with temperature, the better the performance of the lubricant when subjected to great temperature changes experienced in practice, the lower the viscosity temperature coefficient, the higher the viscosity index and the better the performance of the lubricant. [20].

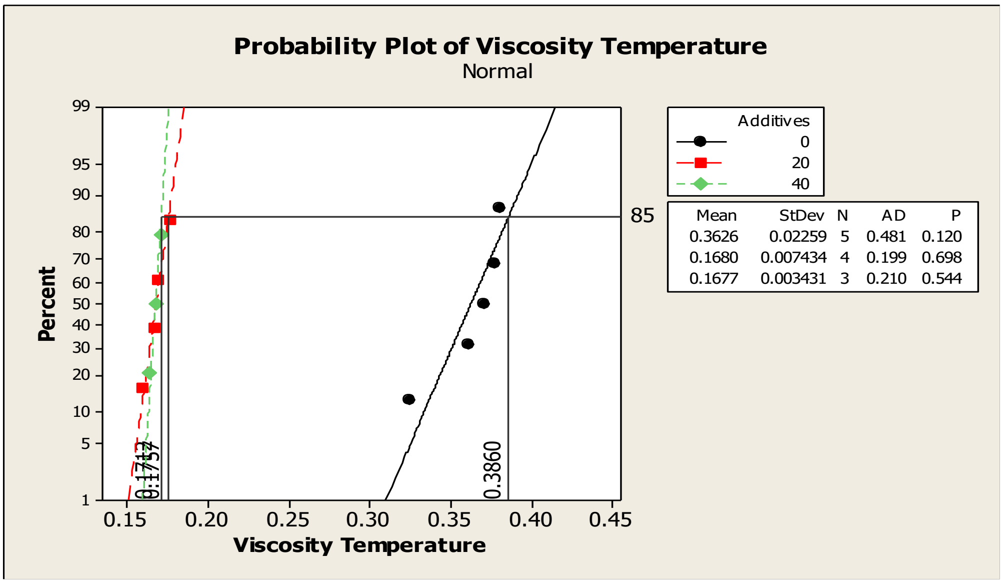

Figure 1: Effect of Additives on the probability plot of viscosity temperature coefficient of all the greases produced from 100% Jatropha curcas seed oils

Effect of Additives on the Dropping point temperature property of all the greases produced.

Figure 2 shows the effect of additive on the dropping point temperature of the grease sample, zero gram of additive yielded grease sample with the lowest dropping point temperatures, while 20.00g and 40.00g additives yield grease sample with almost the same high dropping point temperatures respectively. From the results, it was quite clear that the production variables (temperature and time) does not have any impact or effect on the properties of grease. It will be more profitable to select the least temperature 160^{0}C and the least cooking time 20 minutes. The thickener is only responsible for the thickness or the softness of the grease.

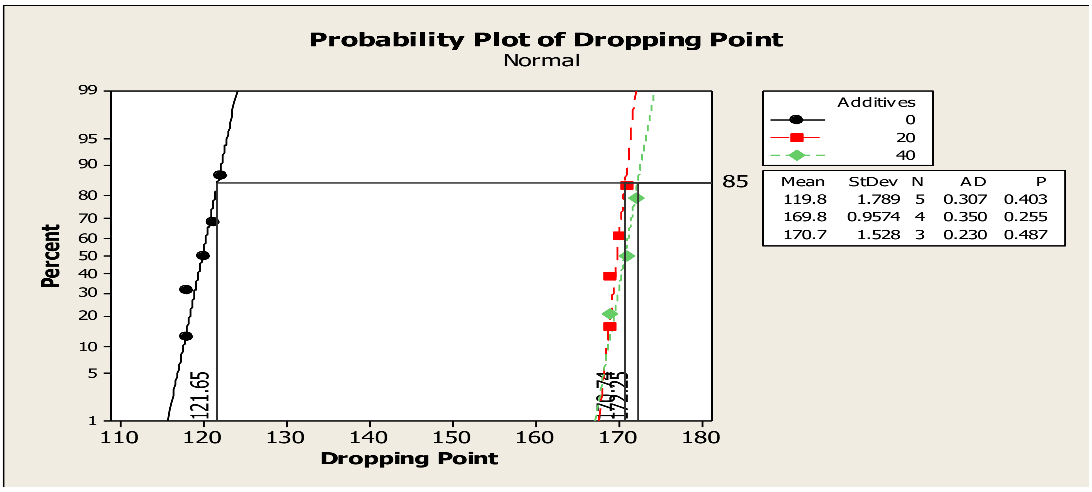

Figure 2: Effect of Additives on the probability plot of dropping point temperature of all the greases produced.

The BOD test is a chemical procedure for determining the action(s) of microorganisms including bacteria in using up the oxygen in a body of water. The BOD test results in Table 5 shows that there was a higher oxygen consumption in the prepared grease samples (16- 20 ppm) as compared to that produced from petroleum products. This implies that there was a high oxygen consumption in the prepared grease samples hence high rate of biodegradability and thus more environmentally friendly products.

4. CONCLUSION.

The base oil used was suitable for the production of grease. Low temperature and time of operation favor low viscosity temperature coefficient and high dropping point temperatures which characterizes a good quality grease. The optimum quality was obtained with 20.00g or 5% selected additives, 120.00g or 35% thickener, 240.00g or 60% base oil and produced at temperature and time of 160^0C and 20 minutes respectively. The grease is biodegradable hence more environmentally friendly.

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