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Production and Characterization of Biodegradable Grease from Neem Seed Oil

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Abstract This paper reports on the production and characterization of biodegradable grease from neem seed oil. The extracted neem oil was purchased from National Research Institute for Chemical Technology (NARICT), Zaria. The base oil (neem oil), thickener (calcium hydroxide and steric acid) and additives were mixed together to form a solid grease. Work penetration, un-worked penetration, dropping point and biodegradability tests were carried out to determine the quality of the biodegradable grease produced. Ammagrease was used as control and standard. The production condition, measurement standards and results were obtained. The grease was produced at 3 different temperatures; 100oC, 160oC and 200oC. The biochemical oxygen demand (BOD) test results were within the acceptable range of 4.0 – 8.0 ppm. The results for the worked penetration level, un-worked penetration level, and the dropping point were within the American Society for Testing Materials (ASTM) standard values of 248, 257 and 180 respectively. This shows that neem oil can be used as a substitute to synthetic/fossil (petroleum) base oil in the production of grease for industrial and commercial uses.

Keywords Biodegradable grease, neem seed oil, characterization, grease.

Introduction

The effect of friction on industrial equipment (machine parts) and on automobiles is a major problem confronting man. As a result, lubricants or greases made from petroleum products were initially developed to limit these dangerous situations. The need to develop as much as possible bio-based materials as industrial and automotive lubricants has increased in recent years. This trend is primarily due to the non-toxic and biodegradable characteristics of vegetable oils that can replace mineral oils as base fluids in grease making [1-2].

Biodegradable greases which are made from renewable resources via vegetable oils provide suitable alternative to the petroleum base greases that are environmentally toxic and non-biodegradable. Vegetable oil-based greases are semi-solid colloidal dispersions of thickening agents (a metal soap) in a liquid lubricant matrix (vegetable oil). The thrust therefore in the world of tribology today is the search for renewable, biodegradable high viscous oils that will satisfy the requirements of lubrication. Neem seed oil can be one of such options in the search.

Grease is a semi fluid of a fluid lubricant, a thickener, and additives. The fluid lubricant that performs the actual lubrication can be petroleum (mineral) oil, synthetic oil, or vegetable oil. The thickener gives grease its characteristic consistency and is sometimes thought of as a "three-dimensional fibrous network" or "sponge" that holds the oil in place. Common thickeners are soaps and organic or inorganic no soap thickeners [2]. The majority of greases on the market are composed of mineral oil blended with a soap thickener. Additives enhance performance and protect the grease and lubricated surfaces. Grease has been described as a temperature-regulated feeding device, when the lubricant film between wearing surfaces thins, the resulting heat softens the adjacent grease, which expands and releases oil to restore film thickness [1].

Neem seed oil is a pure vegetable oil extracted from the fruits and seeds of neem (Azadirachta Indica), a tropical evergreen tree which is native to the Indian subcontinent and has been introduced to many other tropical areas like the West African tropics and particularly, the Northern Nigeria where it's natively called 'Dogonyaro'. Apart from its major use to produce laundry soaps and its medicinal importance, the neem seed oil according to recent studies, can be used as engine lubricants and as replacement for biodiesel if its properties are enhanced [5].

Materials and Methods

Production and characterization of biodegradable grease from neem seed oil

The grease production began with transestrification process. Neem oil was first filtered to remove impurities in the oil. Lye catalyst, sodium hydroxide (NaOH) was mixed with methanol and shake well until the NaOH is completely dissolved in the alcohol. The alcohol-catalyst mixture was then charged into blender, where neem oil was added. The whole mixture was stirred well to obtain a homogenous solution. The blend was then heated to a temperature from 50^{o}C to 70^{o}C. Reaction time was varied from 2 to 24 hours. Samples of the mixture were extracted at one hour interval and the total glycerin level of the ester fraction was measured. The same procedure was repeated several times until the desired amount of transesterified neem oil was obtained (first generation biodiesel production from non-edible vegetable oil and its effect on diesel emissions [4].

A mixture of calcium hydroxide $Ca(OH)_2$, stearic acid in the ratio 1:0.75 and the Neem oil which was thrice the combination of $Ca(OH)_2$ and stearic acid were mixed at a temperature of 100^{0}C in a glass reactor. The temperature was then raised to 200^{o}C for 2 hours in the reactor with a continued stirring until soap foams were formed, when this was attained, the temperature was reduced to 160^{o}C for an additional amount of the base oil and additive to be added. The final mixture was allowed to cool to room temperature. The resulting mixture was roll-milled to obtain the grease. The initial temperature of 190^{o}C was attained so as to enable the base oil to be trapped in the fiber network of the soap. The same process was repeated for sample 2 following the formulation of the sample.

Dropping Point Determination

The experiment was performed at the quality control laboratory of Ammasco international limited. A Pyrex beaker (800ml) was 70 percent filled with base oil (SN900) as a heating medium. A standard grease cup with a hole at the bottom was coated with the test grease. A thermometer was inserted into the cup and held in place with a clamp so that the thermometer never touches the grease. This assembly was placed inside a test tube which was in turn lowered into the oil-containing beaker. Another thermometer was inserted into the oil bath so that it was about the same level as the test tube thermometer.

The oil together with its content was heated by a regulatory heater while being stirred at the rate of 5^{o}C per minute until the temperature was approximately 17^{o}C below an expected dropping point of 85^{o}C. The heating was gradually reduced until the test tube temperature became 2^{o}C less than the oil temperature. The dropping point was the temperature recorded on the test tube thermometer when a drop of grease fell through the hole in the grease cup.

Un-Worked Penetration Test

The experiment was also carried out at Ammasco International Limited. Two samples of the neem oil grease and the control sample (Ammagrease) were subjected to penetration test. They had their different penetration levels measured using penetrometer by filling in a standard grease cup, cylindrical in shape with 50ml capacity with a little disturbance, the surface was smoothened and placed on the penetrometer assembled and pressed for five seconds during which a special grease-cone on the assembly has its tip just touching the level of the grease surface at the start. The distance dropped for each sample were read from the dial indicator of the penetrometer and recorded.

Worked Penetration Test

This test was also carried out at Ammasco International Limited. The worked penetration test was carried out in the same manner with that of the Un-worked penetration test but the difference was that the grease samples were worked for 60 double strokes in the standard grease worker cup. In this method, the disturbance of the grease was standardized by the prescribed working process and is more reliable than Un-worked penetration test because the grease has been subjected to a work load of double strokes over a wide range of temperatures.

Test for Biodegradability

The test was carried out at the research laboratory of Chemical Engineering Department. 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. For this test, the standard period and temperature required were five days and 20^{o}C respectively. 10ml of the produced grease labeled 1, 2 and 3 were mixed with 100ml pond water which contains microorganisms in 250ml incubation bottles each and this bottles do not allowed the passage of light through it. Another incubation bottle was also filled with 10ml of mineral grease (Control); 1ml each of iron (ii) chloride, phosphate buffer, calcium chloride and magnesium sulphate were mixed in 1 liter of distilled water. The dilute water prepared was added to each of the incubation bottles for about two hours at room temperature from which the dissolved oxygen was measured using an oxygen meter. The incubation bottles were kept at the same temperature of 20^{o}C for five days after which the dissolved oxygen was recorded. The differences in the oxygen dissolved was calculated and recorded.

Results and Discussion

It was observed in the production process that the product of the mixture of base oil (neem oil), thickener (calcium hydroxide), additives and stearic acid and continuous stirring in the reactor changed as the reaction progresses from melt to syrup. The product color also changed from dark brown to yellow signifying the reaction and formation of the final product. It was also observed that the ratio of base oil to calcium hydroxide (thickener) matters a lot. The higher the quantity of the thickener as compared to that of base oil (neem oil), the more difficult will be the formation of grease [3].

Table 1 shows a summary of weights and percentage compositions of the neem oil, calcium hydroxide, additive and steric acid used in the production of the grease samples.

Table 1: Mixing Ratio of materials for grease production

Grease Sample	Sample 1	Sample 2
Mass of neem oil A (g)	75.00	70.00
Mass of calcium hydroxide B (g)	18.00	10.00
Mass of steric acid C (g)	12.90	6.50
Mass of additive D (g)	3.00	2.75
Percentage ratio of A to (B+C+D) (%)	68.9:31.1	78.4:21.6

Grease consistency/penetration is the resistance to deformation by an applied force; this depends on the type and amount of thickener used and the viscosity of the base oil. Table 2 shows the values (in mm) obtained from the un-worked and worked penetration tests as compared to the amount of thickener in Table 1 for each sample. A significant difference between un-worked and worked penetration indicates poor shear stability [2].

The two prepared grease samples from Table 2 have fallen within the NLGI grade (4), thereby classified as semi hard type of grease according to the National Lubricating Grease Institute (NLGI).

Table 2: Un-worked and Worked penetration tests results

S/N	Grease sample	Un-worked penetration (0.1mm)	Worked penetration after 60 strokes(0.1mm)
1	Ammagrease (control)	248	257
2	Sample 1	171	198
3	Sample 2	194	205

The dropping point is the temperature at which the lubricating grease becomes fluid enough to drip. The dropping point indicates the upper temperature limit at which lubricating grease retains its structure, not the maximum temperature at which grease may be used. Dropping point depends to a large extent on the type of thickener used.

It was observed from Table 3 that the lubricating grease produced which was made of calcium soup thickener, has dropping point of 82^{o}C – 126^{o}C which differs from the values obtained for the commercial grease (Ammagrease) of 190^{o}C. The variation is dependent on the type of thickener used. Ammagrease is a Lithium base grease.

Table 3: Dropping point of the produced grease samples

S/N	Grease Samples	Dropping Point (^{o}C)
1	Ammagrease (control)	180
2	Sample 1	82
3	Sample 2	126

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 the oxygen consumption after five (5) days was greater in ppm than that of two (2) hours. In addition, there was a higher oxygen consumption in the prepared grease samples (sample 1 and 2) as compared to that of the commercial sample (Ammagrease).The above implies higher rate of biodegradability of the prepared grease, hence, make it an environmentally friendly product.

Table 4: Biochemical Oxygen Demand

Grease sample	Amount of oxygen after 2hours (ppm) (x)	Amount of oxygen after 5days (ppm) (y)	Biochemical Oxygen Demand (BOD) (ppm)
Ammagrease (control)	7.60	7.49	6.0
Sample 1	7.0	6.3	21.0
Sample 2	6.7	6.2	15.0

Conclusion

Biodegradable grease from neem oil was produced and characterized successfully. The produced grease samples (sample 1 and 2) were classified as semi-hard as defined by the National Lubricating Grease Institute (NLGI) of the United State of America. The grease samples produced after being subjected to the necessary tests (especially biodegradation) was found to be far better than the commercial grease used as controls, as its BOD is higher than that of the control sample. The dropping point value depends on the type of thickener used and was found to have increased from 82^{o}C to 126^{o}C as the mass of calcium hydroxide decreases from 18.00g to 10.00g. Therefore, the vegetable oil (neem seed oil) used is a good renewable source for biodegradable grease production.

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