Lubricant bio- grease obtained from sugar cane filter cake vegetable oil in Ethiopia

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Abstract

Under the conditions of Ethiopia, there is no previously reported research on the extraction and use of Sugar Cane Filter Cake Oil. The industry has begun the search for environmentally friendly, renewable and less toxic lubricants. Within these, the production of lubricants of vegetable origin is less aggressive for the environment. The objective of this work is to formulate lubricating grease using Sugar Cane Filter Cake oil extracted from sugarcane by leaching process through the use of selective solvent, from Sugar Cane Filter Cake collected from the Wonji sugar factory in Ethiopia. The lubricating grease was formulated by mixing Sugar Cane Filter Cake Oil, sodium soap and molybdenum disulfide as an additive. The physical- chemical properties of the formulated lubricating grease make it possible to classify it as multipurpose and biodegradable, being able to be used in ball bearings, sliding bearings and other general purposes.

Key words: Vegetable oil; Grease; Lubricant; Sugar cane filter cake oil; Physic chemical properties; Tribology.

1. INTRODUCTION

Lubricants, which include organic and inorganic substances capable of reducing friction, lowering the wear of friction surfaces, and preventing damage by scoring, are employed to upgrade the durability and dependability, serviceability and service efficiency of mechanisms, machines and equipment. In addition to reduce friction and wear in the machine element being lubricated under various operating conditions, protect rust and corrosion, prevent dirt water and other contaminants from entering the part being lubricated [1, 2].

Two of the big problems to face at the moment for the humanity, are the energy crisis because of the exhaustion of the fossil fuels and the contamination of the environment due to the different noxious substances that are poured to the rivers, seas, soils and atmosphere [3].

Alternative sources of energy are currently being investigated, as well as the replacement of lubricants of mineral origin , due to the depletion of the main fossil fuel (petroleum) and the polluting effects of this and its derivatives on the environment [3].

Bio Grease features has high load carrying and anti-wear characteristics along with low water washout properties. It is designed for heavy-duty used in a variety of industrial, mobile, and marine applications. Biodegradable greases, which are made from renewable resources, 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 current issue therefore in the world of tribology today is searching for renewable, biodegradable high viscous oils that will satisfy the requirements of lubrication [4, 5].

Research carried out in the last fifteen years to obtain a new product for use in technological processes that have no effect on the environment has revived the vegetable oils. The great advantages of these lubricants are that they are environmental friendly, have low toxicity and a high biological degradation ratio and can be applied to the tribology systems with considerable technological losses, such as transmission chains in the manufacture of wood, in agriculture or in the food industry [3].

A very interesting opportunity for bio-greases has recently occurred due to new legislation, some countries like USA regulation, the 2013 Vessel General Permit (VGP) issued by the United States Environmental Protection Agency. The VGP aims to reduce the environmental impact of ships, barges, tugs and other commercial vessels, protecting American coasts, lakes and rivers [6].

This need for use vegetable oils as lubricants is a very crucial to be solved the problem faced, which leads to the next investigation, so the objective of the research is to formulate lubricating grease using as dispersing medium sugar cane filter cake oil.

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2. MATERIALS AND METHODS

2.1 Raw Material

A sample of sugarcane filter cake was collected from a Wonji sugar factory which is found in Ethiopia located in Oromia region nearest to Adama town which 15km from Adama and110km from Addis Ababa town. Wonji town has a latitude and longitude of 8°27′N 39°17′E with an elevation of 1588 meters above sea level.

2.1.1 Sugarcane filter cake sample preparation

The dried sample was taken to laboratory of chemistry Department in Jimma University, for extraction of oil. The extracted oil e samples of sugarcane filter cake will be cleaned and prepared as shown in figure 1.

Size reduction and balancing the sample for oil extraction

The moisture was removed by placing the sample in a sun for one week from 20°C to 30℃. The dried filter cake was crushed by hand with sieve size of 1mm- 2mm. The sample was sieved with set of sieves sizes arranged in 1mm- 1.4mm, 1.4mm-, 1.8mm, 1.8mm-2mm to obtain sizes, because to investigate the effect of particles size on yield and quantity of oil.

Figure 1 Raw filter cake after Drying

Determination moisture (specific humidity) content of filter cake sample

It was used 50g, 100g, and 150g of the cleaned sample was weighed and dried in an oven at 105°C and the weight was measured every 2hrs. The procedure was repeated until a constant weight was obtained.

The percentage moisture in the filter cake was calculated using the following Equation:

$$\text{Moisture } \% = \frac{W1-W2}{W1} \times 100\% \quad (1)$$

Where: W1 = original weight of sample before drying; W2 = Weight of sample after drying

2.1.2 Oil extraction

Leaching extraction (Solvent extraction)

In the research Reinosa [9], is obtained that the sugar cane filter cake oil has five time more antioxidant content (6%) than other vegetable oil like soy bean (1.5%), olive (0.5%) , sun flour (2%) approximately. There are presents, in sugar cane filter cake oil, some of important fatty acid to use oil as lubricant such as oleic and linoleic it was obtained. It is demonstrated that the sugar cane filter cake oil have good properties to production lubricant and good protect metals against corrosion.

The oil used during the research was supplied by the chemistry laboratory of Jimma University, where carried out the extraction process by leaching to using hexane as solvent, from plantations of sugar cane of Wonji Sugar Factory of Adama, Oromia Region of Ethiopia.

Solvent extraction is used for oil production from filter cake, because solvent extraction has higher oil yield when compared to other like mechanical extraction method for low content of oil. Experimental work was conducted using cold

hexane solvent for extraction of oil, because hexane has high extraction ability when compare to other solvents and another component like wax and resin inside the sugar cane filter cake are not soluble or has lowest solubility in cold hexane [8].

Samples preparation

The ratio of solid to solvent that was used in this experiment (Figure 2 a) was 1:5, thus 500 ml of solvent was added to the beaker containing 100 g of filter cake powder. A stirrer in the bottom beaker was used to mix up the powder with the solvent thoroughly. During this process, the mixture was kept in cold weather the beaker in to the cold water with ice to control the temperature less than 20 ^{o}C. The solvent-oil mixture in the beaker was collected at the end of the extraction process.

Organic solvents for oil extraction Hexane (99% of purity) were used as organic solvents to leach out oils from the insoluble solid structure of the filter cake. During the selection of organic solvents, the solid to solvent ratio was maintained as 1:5 for the oil extraction. Temperature and mixing time were kept constant at less than 20^{o}C and 3 hours respectively, according to the suggestion parameters reported by Martínez [8], amount of 500 ml of cold hexane (less than 20 ^{0}C) was put into 1000ml container. 100g of the sample was placed in the beaker and was mixed with the dissolvent. This is continued for 3 hours. The experiment was repeated by placing the same amount of the sample again by varying particle sizes (1mm- 1.4mm, 1.4mm- 1.8mm, 1.8mm-2mm) respectively. The solvent was separated and recovery to be used again by rotary vacuum evaporator (figure 3), the weight of oil extracted was determined.

Fig. 2 Extraction of oil from filter cake by using dissolvent extraction method a) Leaching extraction Cold Solvent & Sugar cane filter cake oil b) Sugar cane filter cake oil extracted

Fig. 3 Rotary vacuum evaporator

2.2 Experimental procedure to obtain lubricating grease

Grease Manufacturing Process [10], [11], [12], [13]

Manufacturing of grease is carried out either in pressurized vessels or open cooking kettles. Greases can be made in either by Batch Process or In-line/ Continuous Process.

Experimental procedure used in this research to produced grease.

1. 100 g of filter cake oil puts into the 200 mL beaker and heat to 60 ° C.
2. Mix with electric stirrer as shown in Figure 4
3. 2g of sodium hydroxide was added, continued starry up to get 90 ° C and kept the stirred for approximately 1 hours for saponification.
4. The mixture was stirred and heated up to a temperature of 160 ° C for 30 minutes and then cooled to 90 ° C.
5. Molybdenum disulfide (MoS_2) additives added with the weight of 5%.
6. Raise and lower the stirrer to homogenize the whole dough.
7. Check pH throughout the period by adding hydroxide.

8. Add 30 mL of oil if required.
9. The stirring and heating was continued at temperature of 90° C for next 30 minutes
10. Continue stirring from top to bottom moving the whole dough until cool to room temperature (25℃) and stored for future analysis.

The basis for the preparation was on two parameters which were percentage weight and temperature.

The weight ratio of the base oil, sodium hydroxide and **small additives (**Molybdenum disulfide). 100:2:0.05; percent of mass respectively.

2.3 Friction test

The experiment was carried out in the Energy Research Center of Las Tunas University, was analyzed the samples sugar cane filter cake grease base sodium from Ethiopia.

Steps for friction test [15]**:**
The performance of these friction tests comprises the following steps:

1. Mount the preassembled metal specimen under ASTM G77-98 onto the support arm and tighten with the Allen wrench until it is aligned with the ring.
2. On the tribometer shaft, the ring with the dimensions approved by ASTM G77-98 is placed opposite the end of the pulley.
3. Turn on the electric motor.
4. Carefully assemble the support arm with the specimen on the shaft built for it.
5. Lubricate the ring with a known lubricant.
6. Place the weight decided on and adjusts the arms to make a lever and the probe to contact the ring.
7. Connect the terminals of the Network Analyzer to the power supply of the electric motor to immediately collect the data provided by the motor depending on the task you are doing.
8. With a tachometer, measure the revolutions delivered by an electric motor.
9. With a pre-set laser thermometer, the ring temperatures are measured at various times during the test.
10. After 10 minutes, the measurements are recorded again and the motor is stopped.
11. The probe is massaged to remove the material loss by mass difference.
12. For the next test, if you are changing the lubricant, grind the surface of the ring to remove any traces of lubricating film.
13. Repeat the same cycle until step 11

3. RESULT & DISCUSSION

The grease produced using different ratios of base (filter cake) oil, sodium hydroxide and additive was tested for penetration test (ASTM D-217), dropping point (ASTM D-566), corrosion resistance (ASTM D-130), EP (ASTM D2266), critical load (ASTM D-2596-97), oxidation stability (ASTM D-942), copper strip test (ASTM D-408), and etc. in order to determine the best sample produced.

3.1 Characterization of the extracted sugarcane filter cake oil

The physical properties of the extracted sugar cane filter cake oil such as specific gravity at 25℃, density, dynamic viscosity, kinematic viscosity, PH value was analyzed using standard procedures and chemical properties such as acid value, saponification value and iodine value also determined.

The oil mass extracted (figure 2 b) from Sugar cane filter cake oil shown table 1 carried out an experiment in the chemistry laboratory of Jimma University in Ethiopia, from 100g of filter cake processed.

Tabla.1 Mass extracted oil from Sugar cane filter cake.

Samples	Oil (g)
1	4.5
2	4.3
3	4.0
4	5.2
5	4.0
6	5.1

The extraction rate as it can see, obtained from the table 1, was around 4.5% of oil in this research; the extraction carried out for filter cake in Ethiopia are similarly to the result obtained in other country like Cuba, R.M.Torres [8], in Cuba; if it can be considered the sugar cane filter cake oil density around 896.1 kg/m^3, similar too with reported value [5, 8].

It was reported in the research P.D.R.Castaneiras [7] , R.M.Torres [8], O.R.Espinosa [9] some properties of sugar cane filter cake oil that have not significant difference with the values obtained in this research.

Table 2 Some properties of the sugar cane filter cake oil

Properties	Magnitude

Acidity index (mg KOH / g oil)	23
Saponification index (mg KOH / g oil)	145
Iodine content (g iodine / 100 g oil)	83
Content of unsaponifiable matter (%)	20
Density, (kg / m 3) ASTM D 1298	896.1
Kinematic viscosity at 40 oC, cSt ASTM D 445	75*
Kinematic viscosity at 100 ° C, cSt ASTM D 445	14*
Viscosity index (VI)*ASTM 2270	197

*Viscosity of oil with 0.5% of content of sugar cane filter cake wax [7]

3.2 Sodium Grease

Sodium greases, as reported by G.E. Totten [14] have a dropping point about 175 °C and it can be operated at 121℃, are used in reasonably high temperature operations, have an excellent protection against rust and it can be mixed with other greases to produce higher quality grease, it has better adhesive properties than calcium grease. It is, however, still recommended for certain heavy-duty applications and well-sealed electric motors.

Figure 4 Electrical stirrer

Figure 5 Sugar Cane Filter Cake Grease

A lubricating grease, as shown in figure 5, was obtained with the following properties:

Colloidal stability: 14.4%. Ability of the grease to retain solidly in the reticulum of the spatial structure, the oil of which it is composed.

Acidity: 2.3 mg of KOH / g: The acid character of a lubricant is determined by the presence of acid substances in oil.

Consistency: grade 2. Consistency gives a measure of average dispersion-medium thickener ratio (vegetable oil-soap). For the formulated grease the consistency is medium, ASTM worked (60 strokes) penetration at 25 °C tenths of a millimeter, the penetration worked at 25 ° C is between 265-295 ("normal" grease).

Copper Corrosion: 1year, copper corrosion tests are intended to determine the ability of the oil to attack soft metals, such as copper, lead.

This index is made by introducing sheets of copper in grease at a temperature of 100 ° C and for 3 hrs; gives a measure of the corrosiveness of the grease.

The physical appearance of the grease shows a black color, with a smooth and homogeneous texture.

The magnitude of the penetration worked locates this grease with a degree of consistency NLGI Grade 2, that is to say it is a very soft grease, recommended to be used for its degree of consistency in bearings of bearing and sliding bearing, centralized systems, lubrication of gears open and in total loss systems.

The dropping temperature is 150 °C and the copper corrosion test was 1year, without corrosive action on the Copper sheet.

The results of the physicochemical properties are as follows:

In the case of penetration, the repeatability according to ASTM 217 states that for duplicate results obtained by the same operator under the same test conditions, the difference will not exceed 7 units, in the test performed the difference was up to 5.

For the drip temperature, repeatability according to ASTM 566 states that for duplicate results obtained by the same operator under the same test conditions, the difference will not exceed 7 ° C, in the test performed the difference was up to 2 °

Copper corrosion was determined by taking 10 samples of grease, in all cases the corrosion result was the same degree 1year.

The physicochemical properties determined, described above, make it possible to conclude that lubricating grease was obtained for mechanical purposes. The consistency grade 2 allows to be used for its degree of consistency in bearings of bearing and sliding bearing, centralized systems, and lubrication of gears open and in total loss systems.

3.3 Relation of the temperature with the time and friction coefficient

The test of this properties was carried out in the Research Center of Las tunas University, with the block and ring tribometer show in the following figure 6.

In this case was analyzed the sugar cane filter cake grease (base sodium soap) and comparison with a mineral grease mineral (Mineral_ Grease_ Li _ MoS_2) base lithium soap (Lisan 2m) supply for CUBALUB one of enterprise specialized in lubricant and certify this grease as standard characteristics was analyzed.

Fig. 6 Tribometer Block and Ring Research Energy Center, Las Tunas, Cuba.

3.3.1 Temperature

In the research S. Rani [16], is obtained that the tribological properties of vegetable oil are always superior to mineral oils at room temperatures. The vegetable oils are highly biodegradable, less toxic and renewable lubricant base oil which needs to be developed by adding suitable additives. In this research as shown figure 7 and 8 the vegetable oil grease have a better behavior than mineral one.

The comparison between sugar cane filter cake and a mineral lithium grease showed that the temperature increase between ring and block tribometer show that the sugar cane filter cake grease have better behavior than mineral one, the temperature was stabilized around 48 ^{0}C (figure 8) for vegetable oil grease, while the temperature stabilized at 70 ^{o}C (figure 7) for the mineral grease base lithium, in both case the starting to stabilized the temperature occur after 25 minutes approximately; this result that the sugar cane filter cake grease with additive MoS_2 has more contribution to energy conservation and more protection to wear of surface in contact is demonstrated.

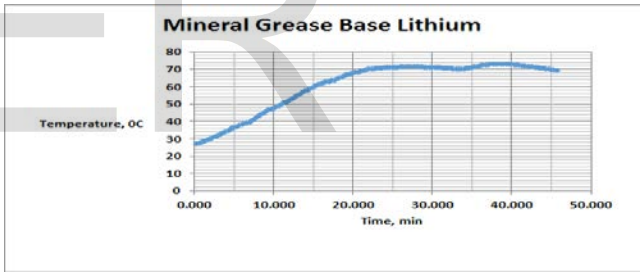

Fig. 7 Mineral _ Grease_ Li _ MoS_2 Friction coeficiente vs time for Load= 123N

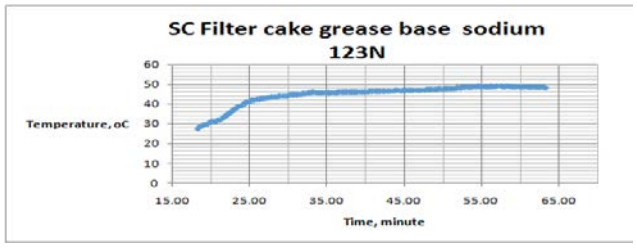

Figure 8 SC_ Filter Cake Grease_ Na_ MoS_2 Friction coeficiente vs time for Load=123N

3.3.1 Friction coefficient

At the same time, the friction coefficient showed in figure 9, the friction coefficient decrease in both case with the temperature, as in the temperature variation with the time after around 65^{o}C for the mineral

grease and around 48^{0}C for sugar cane filter cake grease is observed certainly tendency to keeping constant, it can see that there are certainly concentration of values for both case.

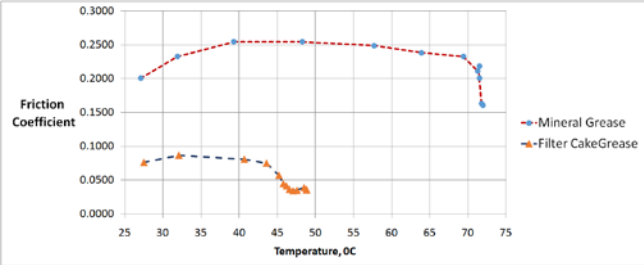

Figure 9 Friction coeficiente vs temperature for Load= 123N

At the same time, the friction coefficient showed in figure 9, at the beginning increase for both case, after 40 ^{o}C decrease and 33 ^{o}C for mineral grease and vegetable oil is stabilized at 0.25 and 0.08 respectively. For both, after reach temperature around 65^{o}C for the mineral grease and around 48^{0}C for sugar cane filter cake grease, similar as in the temperature variation with the time (figure 7 and 8), the friction coefficient is observed certainly tendency to keeping constant, it can see that there are certainly concentration of values for both case.

As in figures 9 is observed that the sugar cane filter cake grease showed less than friction coefficient that mineral one. Theses has correspondence with temperature behavior with the time (figure 7 and 9), there was more heat generated in the friction system with mineral grease where temperature stabilized around 70 ^{0}C and the friction was higher in relation with sugar cane filter cake grease where temperature stabilized at 45^{0}C after 48 minutes approximately; that confirm the sugar cane filter cake grease show better lubricant properties that mineral grease tested.

4. CONCLUSIONS

1. Lubricating grease was formulated using the vegetable oil extracted from the sugar cane filter cake; as saponification reaction with sodium and potassium hydroxide. The physic - chemical properties of the formulated lubricating grease allow it to be classified as multipurpose grade 2 lubricating grease and can be used in bearings, slides and other general purposes.
2. The formulated grease (SC_Filter Cake Greas_ Na_ MoS_2) has a Grade 2 NLGI consistency, a 150 ^{o}C dripping temperature and a degree of copper corrosion 1year, recommended for lubricating bearing and sliding bearings, as well as centralized systems, open gears and total loss systems .
3. The temperature behavior show in both case the starting to stabilized the temperature occur after 25 minutes approximately; for the sugar cane filter cake grease with additive MoS_2 the temperature stabilized at less than that mineral one, so it is shown that this has a greater contribution to the protection of the wear of the surfaces in contact and the conservation of energy.
4. At the preliminary study of friction coefficient carried out in this research, show tendency to increment in the first time with the temperature, and in certainly interval of the temperature decrease slightly with the increase of the temperature; also as it is possible to be observed from a certain value of temperature the friction coefficient tends to stabilize: for the mineral grease around 45^{o}C and for the sugar cane filter cake grease around 4^{0}C. This decrease must be influenced by the contents in both cases of high pressure additive (MoS_2). It was also observed that sugar cane grease shows lower friction coefficients for the same test conditions than mineral grease.

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