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FORMULATION OF LUBRICATING GREASE FOR AGRICULTURAL EQUIPMENT APPLICATION

FORMULASI GEMUK LUMAS UNTUK APLIKASI PERALATAN PERTANIAN

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ABSTRAK

Formulasi gemuk lumas ramah lingkungan dengan bahan dasar minyak jarak untuk peralatan dan mesin pertanian telah dikembangkan. Untuk meningkatkan sifat tribologi dari minyak dasar ditambahkan beberapa jenis aditif yaitu sterically hindered phenolic sebagai antioksidan (AO), liquid mixture of amine phosphate sebagai aditif tekanan ekstrim (EP), dan disodium sebacate sebagai aditif corrosion inhibitor (CI). Tujuan dari studi ini adalah memperoleh formula gemuk lumas untuk peralatan dan mesin pertanian yang mempunyai ketahanan oksidasi yang tinggi, kemampuan pelumasan dan pencegahan korosi yang baik, serta tahan terhadap air. Hasil uji fisika kimia dan unjuk kerja menunjukkan bahwa spesifikasi dari produk gemuk lumas ini sebagai berikut : NLGI grade adalah 2, Dropping point 192.5^{o}C, Unworked dan worked penetrations masing-masing 255 mm/10 and 307 mm/10, Korosi bilah tembaga adalah 1a, serta Diameter scar adalah 0.57 mm. Hasil uji gemuk lumas yang dikembangkan masih memenuhi spesifikasi, terutama untuk aplikasi pada peralatan dan mesin pertanian.

Kata Kunci: *Formula gemuk lumas, minyak jarak, peralatan dan mesin pertanian.*

ABSTRACT

The formulation of environmentally friendly greases based on castor oil have been developed for agricultural equipment application. In order to improve base oil and tribological performances, several additives such as sterically hindered phenolic type antioxidants (AO), liquid mixture of amine phosphate as an extreme pressure (EP) and disodium sebacate as a corrosion inhibitor (CI) additive were added. The objective of this study is to develop a lubricating grease formula for agricultural equipment applications having high oxidation stability, good wear and corrosion protection, and excellence in terms of water resistance. The results of physical-chemical and performance examination show that the product developed has the following specifications: NLGI grade is 2, dropping point is at temperature of 192.5^{o}C, unworked and worked penetrations are 255 mm/10 and 307 mm/10 respectively. The copper strip corrosion is 1a, and scar diameter is 0.57 mm. The physical-chemical characterics of the grease developed still meet specifications, especially for agricultural equipment application.

Keywords: Lubricating grease formula, castor oil, agricultural equipments.

I. INTRODUCTION

In recent years, pollution and environmental health have become increasingly important as public issues. In the area of lubrication, concern is focused on the issues relating to a large proportion of lubricants lost in the environment as, 50-60% of the synthetic lubricants directly comes into contact with soil, water and air, which are a potential threat to ecosystems (Nagendramma and Kaul 2012).

With respect to the impact that lubricants exert on the environment, every year millions of tonnes of engine, industrial and hydraulic oils leak into the ground or waterways, or are disposed of into the environment. Mineral based oils can contaminate groundwater for up to 100 years, which may inhibit tree growth and be toxic to aquatic life (Martin-Alfonso et al. 2011).

Lubricants based on mineral and synthetic oil are widely used in industries. They consist predominantly of hydrocarbons but also contain some sulphur and nitrogen compounds with traces of a number of metals. Due to their inherent toxicity and non-biodegradable nature petrochemical based lubricants pose a constant threat to ecology and vast ground water reserves (Mandakar et al. 2013).

There is wide interest in environmentally friendly products derived from vegetable oils with a high content of oleic acid. They are considered to be potential substitutes to conventional mineral oil-based products. Vegetable oils have advantages such as good lubricity, biodegradable nature, there are non-toxic, have low volatility and a high viscosity index compated to mineral oil. They have some disadvantages namely poor oxidation and thermal stability, and poor cold flow properties. Attempts have been made to improve the stability of vegetable oils via transesterification, selective hydrogenation and epoxidation (Mandakar et al. 2013).

An extensive literature on a great variety of vegetable oils and chemically modified derivatives proposed as biodegradable alternatives to mineral and synthetic lubricant oil has been reported in the last years. They outline additional benefits as base materials for the manufacturing of lubricating greases (Abidakun and Koya, 2013; Cermak et al. 2013; Gryglewicz et al. 2013).

New technology regarding environmental protection has been developed and applied in all industries. There is an increasing demand for environmentally compatible lubricants, particularly in areas where they can come into contact with water, food or people. However, there is no universal definition used to determine environmental friendliness. Biolubricants are often, but not necessarily, based on vegetable oils. They can also be synthetic esters which may be partly derived from renewable resources i.e. the hydrolysis of fats and oils to produce the constituent fatty acids. They can be made from a wider variety of natural sources including solid fats and low grade or waste materials such as tallows. Genetically-modified vegetable oils, such as high-oleic sunflower and rapeseed, are also beginning to find use in applications where higher oxidative stability is needed (Honary Lou and Richter Erwin 2011).

No machinery is so uniquely suited to use vegetable oil based lubricants as agricultural equipment. Since this equipment is particularly close to the environment, the lubricant can easily come into contact with the soil, ground water, and crops. The opportunity exists to create a continuous cycle in which the agricultural equipment is lubricated by oil from a plant growing in the field being cultivated by that same equipment. The agricultural equipment are widely used and are mostly hand tractor, power sprayer, tresher, and rice milling unit. The agricultural equipment consists of moving parts that are in close contact with water, so it needs to be lubricated properly to prevent rust. Lubricating grease is a common type of lubricant used in this agricultural equipment. When used, grease will probably be spilled out into the environment. Since, the base oil is mostly supported by mineral oil which has disadvantageous properties such as poor biodegradibility, and is toxic and carcinogenic, consequently the amount of mineral base greases used contributes significant pollution to the agricultural environment (Erhan Sevim 2012).

The environmental impact has to be reduced by developing environment friendly grease base oil made from non toxic materials such as Castor Oil (Ricinus Communis L). Biodegradable grease effectively operates under a wide temperature range and provides superior lubricity and adherence to surfaces. It also delivers performance properties comparable to petroleum products in areas such as: oxidation stability, high flash point, wear and surface protection. This justifies using natural oil to be a lubricants base oil instead of mineral oil, as it has excelent biodegradibility and is non toxic (Ulfiati Ratu et al. 2011).

The main goal of the present study is to obtain a lubricating grease formula for agricultural equipment applications that have high oxidation stability, good wear and corrosion protection, as well as excellent water resistance.

II. METHODOLOGY

A. Development of Three Formulations of Greases

In order to get the best specification of greases, three formulations are developed base on trial and run method. These three formulas are processed with some variation in operating conditions. The product of the greases are then characterized according to ASTM methods and select the grease products based on the best physical-chemical properties of the greases.

B. Materials

The base oil used in this study is commercially available castor oil (Refined Bleached Deodorized Castor Oil, RBDCO) produced by PT. Kimia Farma, Semarang. The castor oil has the following physical and chemical properties: The viscosities at 40°C and at 100°C are 265.3 mm^2/s and 20.07 mm^2/s respectively; total acid number is 0.970 mgKOH/g; wear scar diameter is 0.49 mm; and iodine number is 85.94.

In the present study, sterically hindered phenolic type antioxidants such as [pentaerythritol tetrakis (3-(3,5-di-tert-butyl-4-hydroxyphenyl) propionate)] was used. To modify the natural properties of base oil and to fulfill the requirements for the lubricating grease, the extreme pressure (EP) additive of a liquid mixture of Amine Phosphates, and corrosion inhibitor (CI) additive of disodium sebacate were added. These additives have been approved by US Food and Drug Administration (FDA) as an ingredient for use in lubricants with incidental food contact for use in and around food processing areas. In addition, lithium-12-hydroxy-stearate formed by neutralization of 12-hydroxy-stearic acid in base oil with lithium hydroxide, is the world wide used thickener for multipurpose lithium greases. The molecular structures of the materials are exhibited in Figure 1. The formulas for bio-lubricating grease are shown in Table 1.

C. Manufacture of Lubricating Greases

Lubricating greases can be made in either a batch or through a continuous process. In this study, the biolubricating grease was produced by a batch process carried out in a 40 kg capacity pressurized grease kettle (Figure 2) in which the castor oil as base oil, additives, and thickener were blended at 90°C. The steps were as follows:

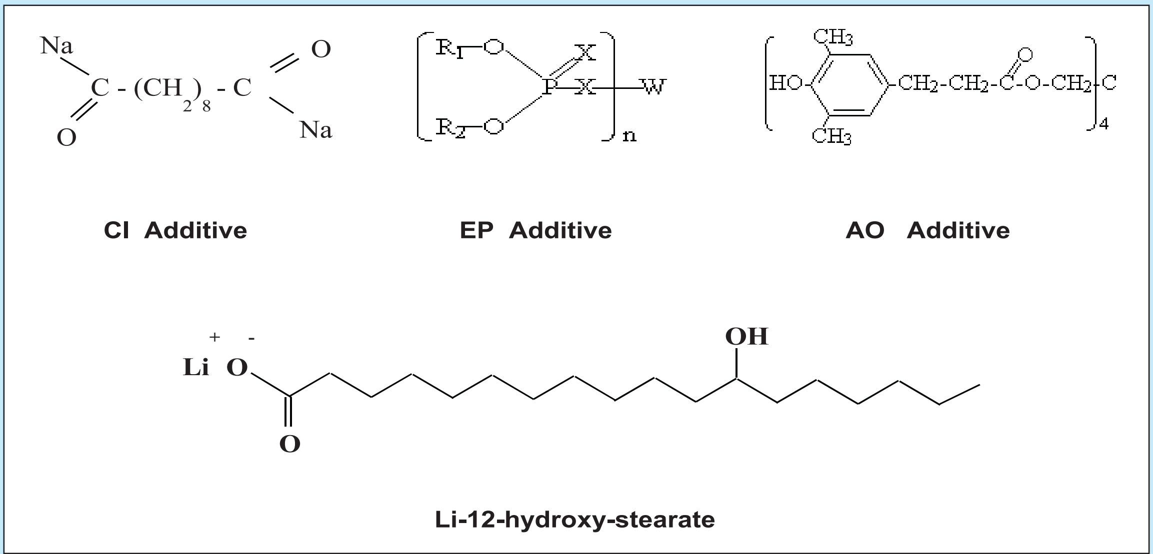

Figure 1
Molecular structure of materials.

Table 1
Formula of Bio-Lubricating Grease

Formula	Composition (mass%)						
	LiOH	12-HSA	AO-A	EP-A	CI-A	Water	Castor Oil
1	0.8	10.0	0.2	0.5	0.5	5.0	83.0
2	1.2	15.0	0.2	0.8	0.8	5.0	77.0
3	1.2	15.0	0.2	0.8	0.8	5.0	77.0

Figure 2
Pressurized Grease Kettle.

Pours 12-hydroxystearic acid and a half of base oil into pressurized grease kettle, heat up to 90°C for 30 minutes. Dilute LiOH in steel container with water, then pours LiOH solution into a pressurized grease kettle while stirring until the soap is formed. Pour the additives into the small kettle, add base oil and heat up to 40^{o}C while stirring until the additives are disolved**.** Slowly add the additives solution into the pressurized grease kettle then cool the temperature down to 80°C and slowly add part of remaining base stock, continue heating and stirring at 80°C with 150 RPM until the soap (lithium-12-hydroxystearate) is completely homogenous. Homogenization time varies and is up to 2 hours.

D. Analytical Methods

The quality of lubricating greases were analyzed by the American Society for Testing and Materials (ASTM) method. ASTM D217 method was applied for measuring the consistency of lubricating grease**.** These procedures for unworked, worked, and prolonged worked penetration are applicable to greases having penetrations between 85 and 475, in other word for greases with consistency numbers between NLGI 6 and NLGI 000. The determination of the dropping point of lubricating grease was carried out by the ASTM D566 method**.** This test method is useful to assist in the identification of greases for establishing and maintaining bench marks of quality control. The results are considered to have only limited significance with respect to service performance as dropping point is a static test. The determination of the corrosiveness to copper used the ASTM D130 method. The copper strip corrosion test is designed to assess the relative degree of corrosivity. However, the four-ball wear-test method (ASTM D2266) was used to determine the relative wear-preventing properties of greases under the test conditions and if the test conditions are changed, the relative ratings may be different.

In this study, commercially available multipurpose grease was used as a reference. It represents a grease for agricultural equipment. Indonesia has a National Standard for grease SNI 7069.15-2008, which is mandatory as the grease specification standard. Accordingly, the physical and chemical characteristics of the greases should reffer to the SNI standard.

III. RESULTS AND DISCUSSION

A. Formulation Results Grease Specification

Batch production is the most common manufacturing method for preparation of the greases. The batch process is a simple method where an homogenizing process is easy to control and as a result, molecular contact between the molecules of the reactants can be optimized.

The homogenizing process or milling is very important in the manufacturing of the greases. The appropriate process will produce a uniform crystal and gel structure that will not change when the grease is used. Homogenizing the grease will break down the solid particles or fibers and will disperse the resultant small particles in the liquid. It also breaks up lumps, eliminates grain and produces a smooth product. Homogenizing of certain types of greases will stiffen the grease producing a lower penetration value. Homogenizing can improve texture and “brighten” a grease’s appearance. In many cases this homogenizing process is carried out at temperatures greater than 200°F (93°C). After homogenizing, the grease is cooled further, deaerated and packaged (Honary Lou and Richter Erwin 2011).

In this study, preparation of the greases was conducted with several kinds of formulas and differences in operating conditions. Generally, not all of the formulas developed have physical and chemical characteristics which meet the specification requirements. Therefore, only three greases formulas as well as the operating condition of the process were developed. The compositions of the three formulas and operating condition of the process are shown in Table 1 and Table 2 respectively. The results of characteristic examination of the three greases formulas are listed in Table 3. Moreover, the commercially multipurpose grease used as reference was also analyzed by a similar ASTM method as used for examination of three developed greases. The results of this analysis are shown in Table 4.

According to Table 1, the composition of Formula 2 is similar to Formula 3, however, the homogenizing time of Formula 3 is longer than either Formula 1 or Formula 2. Formula 1 and Formula 2 were homogenized in 60 minutes, whereas Formula 3 in 120 minutes (see Table 2). As a result the performance of Formula 3 is better than the other formulas.

The formulation and processing of greases will determine their structure and physical properties. Therefore, the homogenizing process has a strong influence on the quality of the greases. The greases generally have several desired characteristics, such as: consistency, stability to shear, surface affinity, thermal stability, flow or viscosity, thixotropy, syneresis, texture and appearance, and water resistance (Honary Lou and Richter Erwin 2011).

Table 2
Operation condition of formulation

Formula	Saponification			Homogenizing		
	stirring speed	Temp (°C)	Time (min)	stirring speed	Temp (°C)	Time (min)
1	100 rpm	90	30	150 rpm	80	60
2	100 rpm	90	30	150 rpm	80	60
3	100 rpm	90	30	150 rpm	80	120

Table 3
Test results of formulated bio-lubricating grease

No.	Characteristics	Unit	Test results		
			Formula 1	Formula 2	Formula 3
1	Unworked Penetration	mm/10	352	262	255
2	Worked Penetration	mm/10	352	351	307
3	NLGI	-	0 - 1	2 - 1	2
4	Dropping Point	°C	190	184.5	192.5
5	Copper Strip Test		1b	1a	1a
6	Color	-	off white	off white	Off white
7	Fourball Wear	mm	0.95	0.69	0.57

Table 4
Test results of commercially multipurpose grease

No.	Characteristics	Unit	Test Results	
			CLG-1	CLG-2
1	Unworked Penetration	mm/10	273	224
2	Worked Penetration	mm/10	267	233
3	NLGI	-	2	3
4	Dropping Point	°C	306	348
5	Copper Strip Test	-	1a	1a
6	Color	-	light brown	gray
7	Fourball Wear	mm	0.85	0.80

Table 5
Physical chemical characteristics specification performance for industrial extreme pressure lubricating grease (15)

No.	Characteristics	Unit	Range min	Range max	Test Method
1	Penetration	mm/10	1)		ASTM D-217
2	Dropping point	°C	170	---	ASTM D-566
3	Water washout, at 80°C 2)	mass %	---	15	ASTM D-1264
4	Oil separation 2)	mass %	---	10	ASTM D-1742
5	Rust protection 2)	---	pass		ASTM D-1743
6	Wear scar diameter (fourball wear)	mm	---	0.6	ASTM D-2266
7	Extreem pressure, weld point	kg	250	---	ASTM D-2596
8	Kandungan logam & unsur lain	mass %	3)		ASTM D-4628/AAS
9	Texture and color	---	Conform to produsen specification		visual
10	Thickener type	---	Conform to produsen specification		

Note :
1) conform to NLGI
2) record
3) conform to produsen spesification and existence additive content

SNI 7069.15:2008

In this study, the homogenizing process was carried out at temperature of 80°C and a steering speed of approximately 150 RPM under variations of time. The appearance characteristics of the greases are different, and it depends on the homogenizing time. It is noted that the sequential process of homogenizing is very important, and the appropriate process will produce a uniform crystal and an exellent gel structure of greases that will not change when the greases are being used.

B. Greases Quality Control

Consistency

A simplified system was established by the National Lubricating Grease Institute (NLGI) for rating the consistency of grease. NLGI has carried out a consistency classification system for greases and is accepted as an international industrial standard. The NLGI classification system is based on cone penetration of the worked grease (60 strokes) at 25°C. The lower the penetration, the harder the grease, and the higher the NLGI class.

Consistency is defined as the condition of material standing together or remaining fixed in union, i.e. its resistance to movement or separation of the constituent parts. Grease consistency is important for both type of application (ability required to stay put, seal and lubricate) and method of application (dispensing method). For lubricating greases the consistency is usually determined by cone penetration, i.e. the penetration depth of a standard cone under prescribed conditions of weight, temperature and time. In service greases often become softer due to the mechanical shear of the thickener structure. Hence, the softening effect can be temporary (Thixotropy) or permanent. The ability of a grease to maintain its consistency in service is one parameter determining its service life. Resistance of a grease to mechanical shear can be evaluated by measuring penetration before and after a defined number of cycles in a grease worker equipment (Honary Lou and Richter Erwin 2011).

Figure 3 shows that the values between unworked penetrations and worked penetrations of bio-lubricating greases (F1, F2 and F3), are considerably different, except Formula 1 (F1). It is because the NLGI of Formula 1 is lower (0-1) than the other formulas. Formula 2 (F2) has the same composition as Formula 3 (F3), however, the consistency is different. It indicates that the effect of homogenizing time is critical.

In comparison with laboratory test results of commercially multipurpose greases (Table 4), the greases consistency are excellent, and the values of unworked penetrations and worked penetrations are not significantly different.

Instead of the homogenizing process, the compatibility between base oil and thickener determines the strength of chemical bonding of those substances. Therefore, selection of suitable thickener shoud be carried out carefully.

Dropping Point

Dropping point levels depend mostly on the thickener type, but could also vary considerably due to variations in raw materials and manufacturing process and thus should be used as a quality control standard. A dropping point test result may be used as an indication of the maximum temperature a grease can be exposed to, but in practice operating temperatures should be kept well below it. In the laboratory the dropping point is expressed as the temperature, at which the first drop of grease/oil is extruded from a sample under prescribed conditions.

Lithium-soap grease handles extremes of temperature quite well, which makes them highly suitable for both high and low temperature application. They have a dropping point of approximately 177°C, and can be used continuously at temperatures of 149°C. Basically, lithium-soap greases have very good stability; good water resistance, and are also readily pumpable (Honary Lou and Richter Erwin 2011).

Figure 4 shows that the values of dropping point of formulated bio-lubricating greases (F1, F2 and F3), are commonly higher than the data exhibited in the literature (177oC) particularly for lithium-soap grease. The dropping point of Formula 3 (F3) is higher than for Formula 2 (F2), whereas their chemical compositions are quite similar. This phenomenon is mostly due to the different homogenizing time. In comparison with specification of SNI 7069.15:-2008 (Physical and Chemical Characteristics Specification Performance for Industrial Extreme Pressure Lubricating Grease) (see Table 5), dropping points of formulated bio-lubricating greases are still on specification. Therefore, the formulas

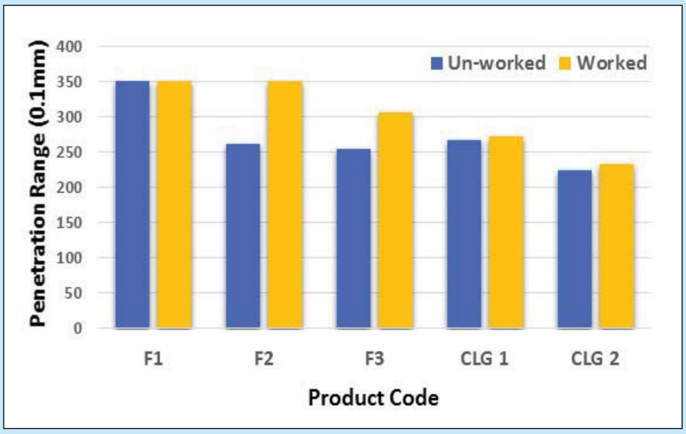

Figure 3
Consistency of grease.

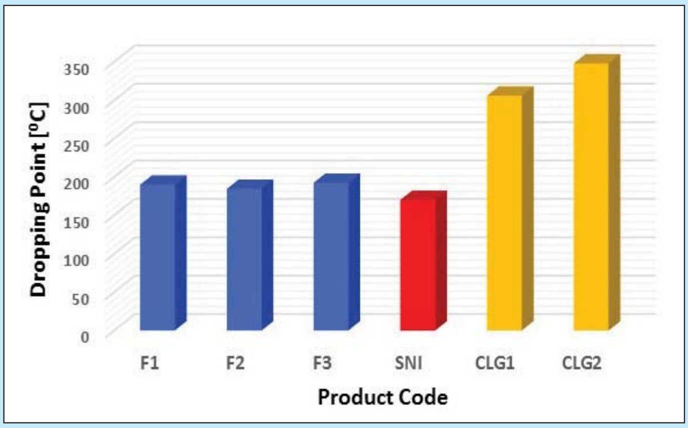

Figure 4
Dropping point of grease.

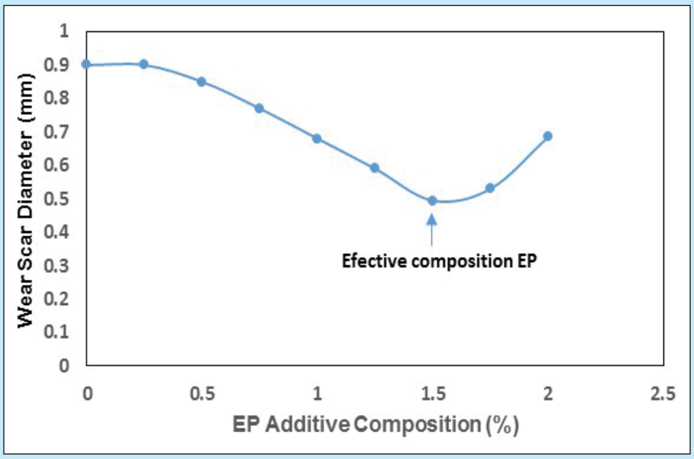

Figure 5
Efective composition of EP additive

are readily accepted for industrial lubricating grease. On the other hand, according to Table 4, commercially multi-purpose grease (CLG1 and CLG2), have dropping points which are mostly much higher than formulated bio-lubricating greases. The commercial greases have a higher dropping point, since the greases base is mineral oil.

Copper Strip Corrosion

Since agricultural equipment is particularly close to the environment, the lubricant can easily come in contact with water, part(s) of equipment need proper lubrication to prevent rust and corrosion.

The laboratory examination result of formulated bio-lubricating grease (see Table 3) shows that the level of copper strip corrosion of Formula 1 (F1) is 1b, in contrast with the other formulas which are 1a. As shown in Table 1, Formula 1 contains 0.5 mass% of corrosion inhibitor (CI), whereas the CI content in Formula 2 and 3 are 0.8 mass% respectively. As the concentration of corrosion inhibitor increases, the resistance to degree of corrosivity increases considerably. Consequently the level of copper strip corrosion increases from 1b to 1a. SNI 7069.15:-2008 (Table 5) shows that rust protection has to be recorded, and the lowest level according to this standard is 1a. As a result, proper addition of corrosion inhibitor additive as required should occur.

Wear Scar Diameter

The level of the wear scar diameter is dependent on the Homogenizing time. Figure 5 shows that wear scar diameter is decreasing with an increase in EP additive composition.

As shown in Table 1, concentration of EP additive in Formula 1 (F1) is only 0.5 mass%, whereas Formula 2 (F2) and Formula 3 (F3) contain 0.8 mass% of EP additive, respectively. Enhancing the concentration of EP additive, may reduce the wear scar diameter of a material. It is probably due to the mechanical action. Figure 6 shows that Formula 2 and 3 (F2 and F3) have different wear scar diameter, eventhough their compositions are similar. The Formula 3 (F3) has wear scar diameter which is better than the Formula 2 (F2).

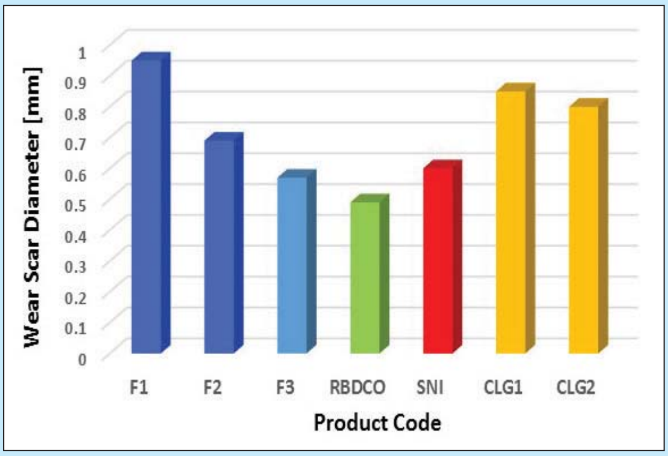

Figure 6
Wear scar diameter of grease.

According to SNI 7069.15:-2008 the maximum wear scar diameter acceptable for industrial grease is 0.6 mm. However in Figure 6, the wear scar diameter of Formula 2 (F2) and Formula 3 (F3) are 0.69 mm and 0.57 mm, respectively. The compositions of those formulas are quite similar, but the time of the Homogenizing process is considerably different. In addition, the Homogenizing time also contributes to the reduction in wear scar diameter. The wear scar diameter on Formula 3 is much better than that listed in SNI 7069.15:2008 specification (Table 5).

IV. CONCLUSION

Lubricating grease is a common type of lubricant used in agricultural equipment. Lubricating grease based on vegetable oil has superior lubricity and stronger adherence to surfaces than mineral oil.

The three formulas F1, F2 and F3 have been developed with some variation in operating conditions. Formula 3 (F3) including its operating condition, is the most preferable formula and process. The values of consistency, dropping point and wear scar diameter of F3 are better than other formulas. Moreover, all these characteristics still meet the SNI 7069.15:2008 specification.

Lubricating grease Formula 3 (F3) that has been developed has the following specifications : NLGI grade is 2, dropping point is at the temperature of 192.5oC, unworked penetration and worked penetration are 255 mm/10 and 307 mm/10 respectively, copper strip corrosion is 1a, and scar diameter is 0.57 mm. The test results obviously show this grease has fulfilled the specification for agricultural equipment application and meet the standard requirement.

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