

ภาคผนวก

PALM OIL-BASED GREASE BLENDING: UTILIZATION OF THE STATISTICAL DESIGN OF EXPERIMENTS (DOE)

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Natural fatty oils such as palm oil, castor oil, rapeseed oil, soybean oil, sunflower oil, lard, and sperm oil have been used as lubricants for years. They are normally called triglycerides of more or less unsaturated fatty esters. This type of base is "biodegradable" and, compare to mineral oils, show excellent tribological properties (low friction coefficient and good wear protection). However, their range of use is limited by lower stability against thermal oxidative and partly inferior cold flow properties, specifically in lubricating grease.

- Their limits can be improved gradually either with appropriate additives or with chemically modified process. In this particular work, an optimal palm oil-based grease blending formula is proposed utilization of the statistical design of experiments (DOE). Input variables are palm oil-based grease structures (% of palm oil, % of thickener and % of additives), type and percentage of each specific additives.

Key words: design of experiments, vegetable grease.

1. Introduction

The design of the experimental method is a typical approach to identify any complex process characteristic efficiently and logically by experiment. It is designed by means of orthogonal array and the experimental results are analyzed using ANOVA (Analysis of Variance) (Moen *et al.*, 1999). An orthogonal array (OA), which is a kind of fractional factorial design with much information, is extracted using a small number of experiments. In comparison with full factorial designs that performs experiments for every combination of parameter level, is a name designated by the fact that all control parameters, are independent to each other (Snee *et al.*, 1985). The initial phase of the research program involved statistically reduction the number of required observations for the entire experiments. The reduction involves the minimization of the number of data points necessary to represent optimum "grease blending" ingredients as a function of "responding" values i.e. anti-wear, anti-oxidation, pour point characteristic.

Table 1. Multipurpose industrial grease specification.

Properties	Specifications
Thickener type	Lithium
NLGI Classification	2, 3
Dropping Point	200°C
Load Carrying properties (4-ball Welded Load)	Greater than 800kg
Timken O.K. Load	45lb
4-ball MWSD (Mean Wear Scar Diameter) (40kg for 1hour) mm	0.52

General multipurpose mineral oil-based grease characteristics is normally included basic grease base oil behavior, thickener type, dropping point, penetration number (NLGI consistency number), anti-wear (AW)/extreme pressure (EP) properties (Pirro and Wessol, 2001). Table 1 shows basic specification of industrial grease commonly available in the market worldwide. Basically, the viscosity and/or viscosity index, thickener type, dropping point and the load carrying capacity are the vital characteristics that should be tested for the grease production process. Table 2 presents a comparison between the properties of mineral base oil and palm base oil. In general, the oxidation and thermal stability of palm oil is significantly inferior to mineral base oil. Hence, in this particular research, the design of experiments is proposed to find the improvement in the blending formula.

Table 2. Comparison chart for mineral base oil and palm base oil.

Properties	Mineral base oil (The closest ISO VG range)	Palm base oil
Viscosity at 40°C (cSt)	ISO VG 46	43
Viscosity index	100 or less	200 or more
Pour point	Less than -10°C	Approx. 9°C
Specific gravity	0.95	0.93

2. Manufacture of grease

The manufacture of a grease, whether by a batch or continuous process, involves the dispersion of the thickener in the fluid and the incorporation of additives or modifiers. This can be done in a number of ways. In some cases, the thickener is purchased by the grease manufacturer in a finished state and then mixed with oil until the desired grease structure is obtained (Moller and Boor, 1986).

In the manufacture of a multipurpose lithium soap grease, for example, based oil, fatty acids are dissolved in a portion of the oil and then saponified with an aqueous solution of lithium hydroxide. This produces a wet lithium soap that is partially dispersed in the base oil and is then dehydrated by heating. After drying, the mixture is cut back with additional oil and additives (optional to this research project as the next step to be carried out in the future work) to produce the desired consistency and formulation characteristics intended of the finished grease (Mortier and Orszulik, 1997).

Table 3. Grease blending structures.

Formula	Palm oil (% by vol.)	12-Hydroxysteric (% by vol.)	Lithium Hydroxide (% by vol.)	Physical characteristic (NLGI No.)
1	70	21	9	extremely hard (>> 6)
2	70	15	15	extremely hard (> 6)
3	70	9	21	too hard (\cong 6)
4	80	14	6	very hard (\cong 5)
5	80	10	10	hard (\cong 4)
6	80	6	14	hard (\cong 3)
7	90	7	3	medium hard (\cong 2 to 3)
8	90	5	5	soft (\cong 1)
9	90	3	7	very soft (\cong 0)

In this work as a basic study on palm based-oil grease manufacture, three main factors, namely, Lithium Hydroxide, 12-Hydroxysteric and palm base oil are selected as main factors. The only response variable at this first stage is the grease consistency. Each main variables has been assigned for three levels. A 100% combination of blending is performed. Table 3 shows blending structures. The response is observed as

its physical property of the grease and it is rated semi-quantitatively, namely, very fluid, fluid, medium, soft, hard, and very hard.

3. Experimental design and approach

As the preliminary tests, reported elsewhere, the particular blended grease has poor oxidation and thermal stability, specially, in low temperature environment and also in severe loading condition. The three process parameters identified as the most critical variables in the generation of optimum quality palm oil-based grease are I) anti-wear additive II) anti-oxidation and III) pour-point depressant. Each factor is investigated at three levels to determine the optimum settings for the blending process. The two levels of these three variables (AW (anti-wear), AO (anti-oxidant), PD (pour-point depressant) adopted in the present experimental work is summarized in Tab.4.

Table 4. The level of the three factors in the experiment.

	AW	AO	PD
Low level (-)	0.5%	0.25%	0.20%
High level (+)	2.0%	0.50%	0.50%

Table 5 to 7 show the combination of full factorial experiments for the grease blending options available. These tables resulted from a 2³ factorial design. The aim of the experiment is to quantify the effects of three factors on the response variable i.e. anti-wear, anti-oxidation and low/high temperature properties of the blended grease. Once the response variables have been quantitatively measured, the ANOVA can then be performed. In this aspect, both main and interaction effects can then be determined statistically.

Table 5. Data on shade of the grease blending option – wear behavior.

AW	AO		PD	
	Low	High	Low	High
Low	xx.xx	xx.xx	xx.xx	xx.xx
High	xx.xx	xx.xx	xx.xx	xx.xx

Table 6. Data on shade of the grease blending option – oxidation sability.

AW	AO		PD	
	Low	High	Low	High
Low	yy.yy	yy.yy	yy.yy	yy.yy
High	yy.yy	yy.yy	yy.yy	yy.yy

Table 7. Data on shade of the grease blending option – temperature stability (Low/hHigh temperature resistance).

AW	AO		PD	
	Low	High	Low	High
Low	zz.zz	zz.zz	zz.zz	zz.zz
High	zz.zz	zz.zz	zz.zz	zz.zz

In this work, two experiments have been proposed. Both were based on factorial experimental designs and were successful in supplying the information desired. However, the validity of these techniques should be verified by more rigorous methods of statistical analysis i.e. ANOVA. This experience supports the view that, when working in an area where obvious improvement in product quality is possible, grease blending for quality improvement in this particular application, a tremendous amount of this potential improvement can be obtained by designing the experiment well and using quantitative analysis techniques to analyze, interpret, and present data.

4. Conclusion

Factorial experimental designs are the most widely used class of design. They have gained their excellent reputation because they are flexible, and easy to construct, to carry out in the laboratory, to analyze, and to present to others. These techniques, factorial planned experiment in particular, allow the analysts to investigate the effects (main and interaction effects) of many variables in a few runs. In some instances the problem will be solved. In other cases the most important variables will be identified for inclusion in subsequent experiments to develop more detailed information such as measurement of interaction and curvature effects and the construction of response surface prediction equations. Another important characteristic of factorial experimental designs is that they can be used to study the effects of both quantitative and qualitative variables.

Acknowledgment

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References

- Moen R., Nolan T.W. and Provost L.P. (1999): *Quality Improvement Through Planned Experimentation*. – New York: McGraw-Hill Companies.
- Moller U.J. and Boor U. (1986): *Lubricants in Operation*. – London: Mechanical Engineering Publications Limited.
- Mortier R.M. and Orszulik S.T. (1997): *Chemistry and Technology of Lubricants*. – Suffolk: St. Edmundsbury Press
- Pirro D.M. and Wessol A.A. (2001): *Lubrication Fundamentals*. – New York: Marcel Dekker, Inc.
- Snee R.D., Hare L.B. and Trout J.R. (1985): *Experiments in Industry – Design, Analysis and Interpretation of Results*. – Wisconsin: American Society for Quality Control.

PALM OIL-BASED GREASE TRIBOLOGICAL CHARACTERISTICS

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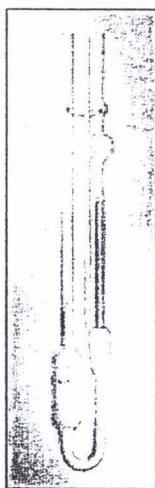
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The pressure for increased use of vegetable oils in lubricants, due to both environmental and health and safety considerations, is likely to continue. In addition, economic trends are likely to make vegetable oils increasingly more attractive than in the past in many areas of the globe. All these factors will probably result in an increase use of vegetable oil as lubricants. In this work, systematic research on palm oil-based grease tribological characteristics assessment will be carried out.

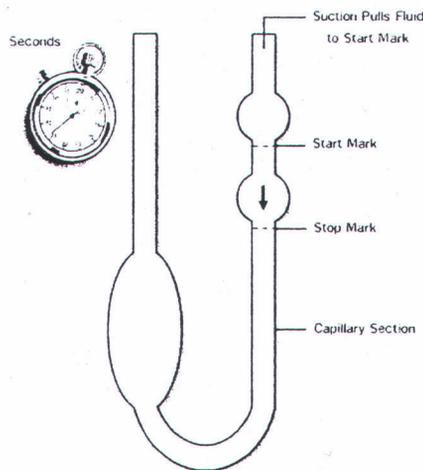
Key words: vegetable grease, tribological characteristics.

1. Introduction

The used of vegetable oils and animal fats for lubrication purposes have been practices for years. With the discovery of petroleum and the availability of inexpensive oils, alternatives became unattractive and were left by the wayside. Attention was refocused on vegetable oils during wartime and oil shortage situations. For example, during World War I and World War II, the use of vegetable oils for fuel, lubricants, greases and energy transfer increased rapidly. Also, the oil embargo of 1973 brought needed attention to alternatives for petroleum oils (Mortier and Orszulik, 1997).



a) Typical viscometer



b) Principal of viscosity measurement

Fig.1. Kinematic viscosity measurement.

In the manufacture of a multipurpose lithium soap grease, for example, based oil, fatty acids are dissolved in a portion of the oil and then saponified with an aqueous solution of lithium hydroxide (Pirro and Wessol, 2001). This produces a wet lithium soap that is partially dispersed in the base oil and is then dehydrated by heating. After drying, the mixture is cut back with additional oil and additives (optional to

this research project as the next step to be carried out in the future work) to produce the desired consistency and formulation characteristics intended of the finished grease (Moller and Boor, 1986). In this work as a basic study on palm based-oil grease manufacture, three main compositions, Lithium Hydroxide (3% by volume), 12-Hydroxysteric (7% by volume) and palm based oil (90% by volume) are blended and used throughout. In this particular work some important tribological behavior of palm oil has been assessed as follow.

2. Tribological characteristics

2.1. Viscosity and viscosity index measurements (ASTM D-445, 1994; ASTM D-2270, 1993)

Kinematic viscosity is the calculated measure of the resistive to flow of a fluid, based on the time in seconds required for a fixed amount to flow through a calibrated capillary tube. For any particular viscometer, the time of flow for a fixed volume of fluid is directly proportional to its Kinematic viscosity. Based on the viscometer calibration constant and the flow time, the viscosity is computed as centistokes. Typical viscometer is shown in Fig.1. Palm oil viscosity at 40°C is approximately at 40 to 43cSt.

Viscosity Index (VI) is an empirical number indicating the rate of change in viscosity of an oil within a given temperature range. A low viscosity index signifies a relatively large change in viscosity with temperature, whereas a high viscosity index shows a relatively small change in viscosity with temperature. The Viscosity Index can be calculated as follows

For $VI \leq 100$

$$VI = \frac{(L-U) \times 100}{(L-H)} \quad (2.1)$$

L – kinematic viscosity at 40°C of an oil of 0 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated, mm^2/s (cSt),

Y – kinematic viscosity at 100°C of the oil whose viscosity index is to be calculated, mm^2/s (cSt),

H – kinematic viscosity at 40°C of an oil of 100 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated mm^2/s (cSt), and

U – kinematic viscosity at 40°C of the oil whose viscosity index is to be calculated mm^2/s (cSt).

For $VI > 100$

$$VI = [((\text{antilog } N) - 1) / 0.00715] + 100 \quad (2.2)$$

where

$$N = (\log H - \log U) / \log Y, \quad (2.3)$$

or

$$Y^N = H/U \quad (2.4)$$

where U – kinematic viscosity at 40°C of the oil whose viscosity index is to be calculated mm^2/s (cSt).

Figure 2 show a comparison plot of viscosities at 40°C and 100°C of palm base oil and industrial hydraulic mineral base oil. Table 1 indicates the Viscosity Index of palm oil in this work.



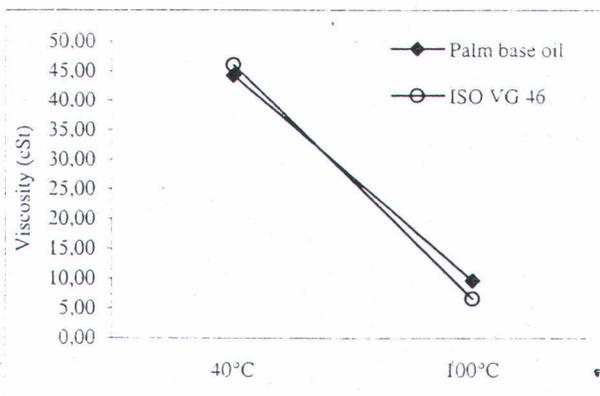


Fig.2. VI of palm and common hydraulic mineral base oil.

Table 1. Viscosity Index of palm and mineral based oil.

	Viscosity @ 40°C	Viscosity @ 100°C	Viscosity Index (VI)
Palm Oil	44.21	9.68	208
Hydraulic Oil	46	6.65	95

As seen in Tab.1, palm oil has a very high viscosity index (VI), 208 versus 95 to 100 for most petroleum oils. Restated, the viscosity of a high VI oil changes less than that of a low VI oil for a given temperature change. The oil's viscosity does not reduce as much when exposed to high temperatures, and does not increase as much as petroleum oils when exposed to cool temperature, to some extent though.

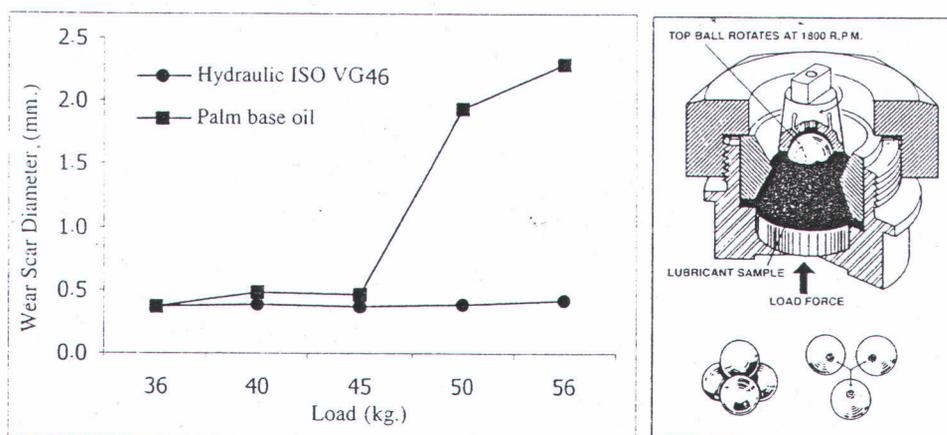


Fig.3. MWSD of mineral-based hydraulic oil and palm base oil.

2.2. Measurement of anti-wear properties (ASTM D-2266, 1991)

2.2.1. Four-ball wear test

The four-ball wear tester consists of 4 balls arranged in the form of an equilateral tetrahedron. The basic elements are 3 lower balls held immovable in a clamp to form a cradle in which a fourth or upper ball is rotated around a vertical axis under prescribed conditions of load and speed. Usually, the balls are made of steel (EN 31 or AISI 52100). The points of contact are lubricated either by grease or oil by immersion in the cup surrounding the four-ball assembly. During the test, circular scars are worn on the surface of the 3 stationary balls; the average diameter of the scars, a measure of wear, depends on the load, speed, duration of

the test and character of the lubricant. Figure 3 shows a plot of MWSD (Mean Wear Scar Diameter) versus load of hydraulic mineral-based oil and palm base oil. It can be seen from Fig.3 that in an extreme case, palm base oil at high load has poorer performance than mineral-based hydraulic oil at similar viscosity. The reason might be because of the blended grease has no AW/EP additives. However, for the sake of mild sliding wear condition of the blended grease in comparison to multipurpose mineral oil-based grease are carried out. Further, in Fig.4, palm oil-based grease and multipurpose industrial mineral oil-based grease wear characteristic is compared. Typical wear scars are shown in Fig.5.

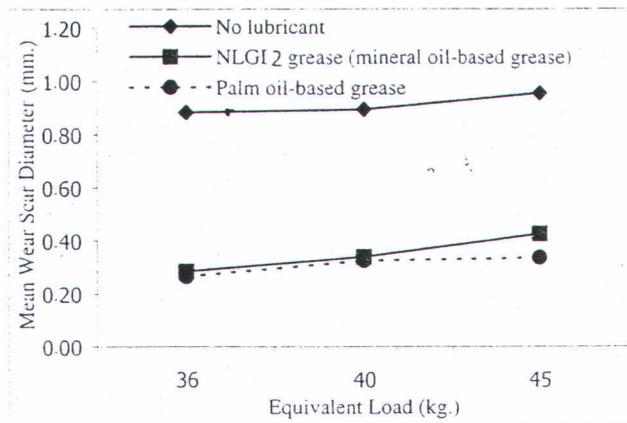
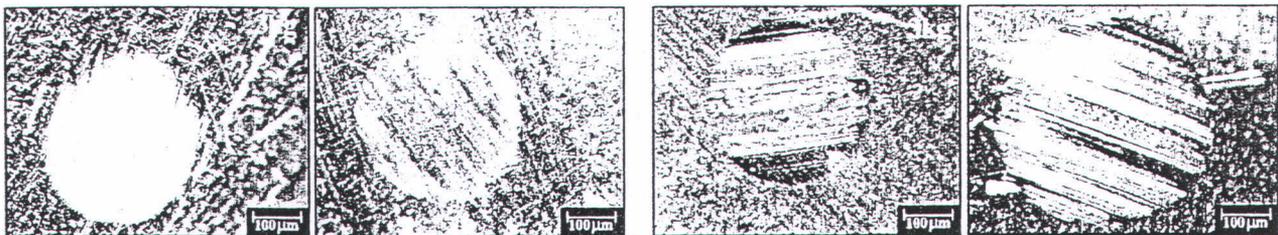


Fig.4. MWSD vs Load of palm oil-based and mineral oil-based greases.



a) Palm oil- based grease.

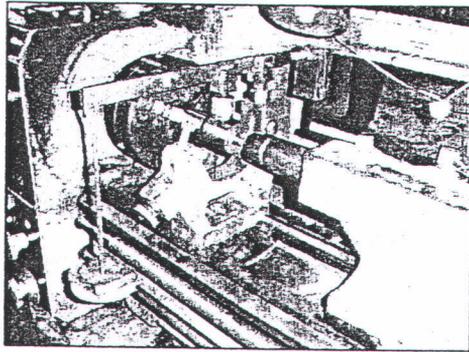
b) Mineral oil-based grease.

Fig.5. Worn surfaces.

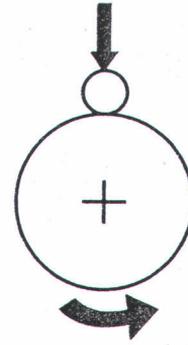
From Fig.4 it can be seen that the size of scar diameter increases with increase applied load both palm grease and mineral oil-based grease. Fig.5 are optical micrographs of the area around the wear scars of the ball specimen at the end of different applied load after a 1min run with palm oil-based grease and multipurpose mineral oil-based grease. It appears, in palm oil-based grease test, to be much smoother, and less material transfer occurred from one ball to another than was found for the wear scars in the mineral oil-based grease lubricated test. This might be because of the no breakdown of the vegetable lubricant film due to their polar property, thus preventing metal to metal contacts. Generally, as vegetable oils can and have been used as lubricants in their natural forms. On the positive side, vegetable oils tend to have excellent lubricity, to some extent, superior than that of mineral oil, specifically in boundary lubrication mode.

2.2.2. Scuffing Ball on Cylinder Lubricity Evaluator (SBOCLE) (ASTM D-6078, 1997)

In addition to the above wear test (four-ball wear tester), SBOCLE is also carried out to get assess to wear characteristic in another extreme condition. A steel ball is loaded against a rotating steel cylinder. A lubricant is fed into a point contact. In this part the comparison between industrial grease (mineral oil-based grease) and palm oil-based grease is carried. Typical arrangement of the wear tester is shown in Fig.6. A plot of MWSD of the steel ball versus load is shown in Fig.7. Typical wear scar of the lower balls are as shown in Fig.8.



a) Wear tester utilization of Lathe.



b) Schematic diagram of wear tester.

Fig.6. Wear tester arrangement.

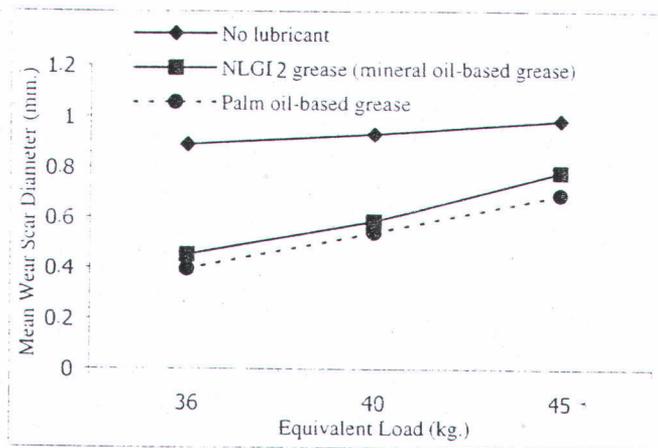


Fig.7. WSD vs Applied load.



a) Palm oil-based grease.

b) Mineral oil-based grease.

Fig.8. Worn surfaces.

From Figs.7 and 8, there is in good agreement with the former wear test (4-ball wear test) as the size of wear scar also increased with the increase of applied load. In addition, the behavior of worn feature of tested balls were similar to the 4-ball test where smoother surfaces are observed for the test with palm oil-based grease lubricated. This may be because of palm base oil has better lubricity property.

3. Conclusion

This study was conducted to investigate the basic tribological behavior of palm oil-based grease. On the positive sides, the high Viscosity Index (VI) and its lubricity under boundary lubrication is not inferior to

that of mineral oil-based grease. Most importantly, vegetable grease are biodegradable, in general are less toxic and reduce dependency on imported petroleum oils, specially for Thailand. These however, it's extreme pressure property yet to be improved, as the blended grease in this work contains no additives.

Acknowledgment

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References

- ASTM D-2266-91 (1991): *Test Method for Wear Preventive Characteristics of Lubricating Grease (Four-Ball Method)*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-2270-93 (1993): *Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-445-94 (1994): *Test Method for Kinematic Viscosity of Transparent and Opaque Liquids*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-6078-97 (1997): *Standard Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)*, vol.05.02, Petroleum Products and Lubricants (II): D 2597 – D 5357.
- Moller U.J. and Boor U. (1986): *Lubricants in Operation*. – London: Mechanical Engineering Publications Limited.
- Mortier R.M. and Orszulik S.T. (1997): *Chemistry and Technology of Lubricants*. – Suffolk: St. Edmundsbury Press.
- Pirro D.M. and Wessol A.A. (2001): *Lubrication Fundamentals*. – New York: Marcel Dekker, Inc.

PALM OIL-BASED CUTTING FLUIDS

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There are no exact figures available as to how many machining operation are carried out without using "cutting fluids" or "coolants". However, there is no doubt that wet machining is applied very much more frequently than dry machining. Also, the oil embargo of 1973 brought needed attention to alternatives for petroleum oils. In addition, the pressure for increased use of vegetable oils in lubricants, due to both environmental and health and safety considerations, specifically on cutting fluids, is likely to continue. In addition, economic trends are likely to make vegetable oils increasingly more attractive than in the past in many areas of the globe. All these factors will probably result in an increase use of vegetable oil as lubricants. In this work, systematic research on palm oil-based cutting fluids characteristics assessment will be carried out.

Key words: palm oil, cutting fluids.

1. Introduction

Owing to the environmental consciousness is increasingly demanded. The direction of cutting fluid consumption is directed to dry cutting condition at its best. One trend in the application of cutting fluids, apart from dry cutting processes, is to move away from flooded coolant of the water-miscible, specially mineral oil based cutting fluids. In recent years, the application of Minimum Coolant Quantity (MQL) system is also reported (Machado and Wallbank, 1997). However, as the mist of coolant may cause lung cancer for the operator which is the main draw back of the application of MQL system, the more appropriate and better biodegradable cutting fluid is needed. Straight mineral oils and synthetic esters have the advantage of requiring less maintenance, a longer service life, often resulting in lower operating and maintenance costs. However, the price per unit is quite high. Vegetable oil exhibit unique features such as very high viscosity index (VHVI), better biodegradable characteristic, lower price, better oiliness property (Honary, 1996). These fluids are well tolerated dermatologically by the operators. In this particular work, a comparison study was performed to evaluate typical mineral neat cutting oil and palm oil-based cutting oils by drilling operation. In addition, other cutting vegetable-based oil characteristics were also reported.

2. Palm oil viscosity and viscosity index characteristic assessments

Kinematic viscosity is the calculated measure of the resistive to flow of a fluid, based on the time in seconds required for a fixed amount to flow through a calibrated capillary tube (ASTM D-445, 1994). Typical viscometer is shown in Fig.1. Palm oil and Refined Palm Oil (RPO) used in this work have their viscosity around $46cSt.$ at $40^{\circ}C$.

Viscosity Index (VI) is an empirical number indicating the rate of change in viscosity of an oil within a given temperature range. A low viscosity index signifies a relatively large change in viscosity with temperature, whereas a high viscosity index shows a relatively small change in viscosity with temperature (ASTM D-2270, 1993). The viscosity index can be calculated as follows:

For $VI \leq 100$

$$VI = \frac{(L-U) \times 100}{(L-H)} \quad (2.1)$$

where L – kinematic viscosity at 40°C of an oil of 0 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated, mm^2/s (cSt); H – kinematic viscosity at 40°C of an oil of 100 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated mm^2/s (cSt), and U – kinematic viscosity at 40°C of the oil whose viscosity index is to be calculated mm^2/s (cSt).

For $VI > 100$

$$VI = [((\text{antilog } N) - 1) / 0.00715] + 100 \quad (2.2)$$

where

$$N = (\log H - \log U) / \log Y \quad (2.3)$$

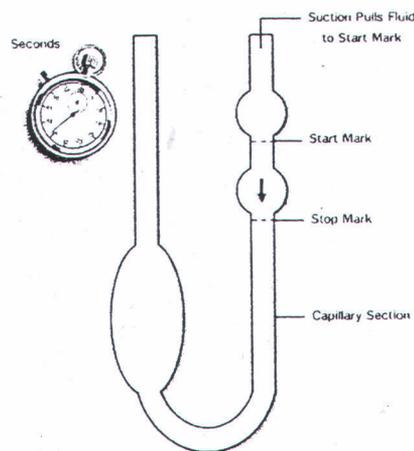
or

$$Y^N = H/U \quad (2.4)$$

where Y – kinematic viscosity at 100°C of the oil whose viscosity index is to be calculated, mm^2/s (cSt).



a) Typical viscometer tube.



b) Principal of viscosity measurement.

Fig.1. Kinematic viscosity measurement.

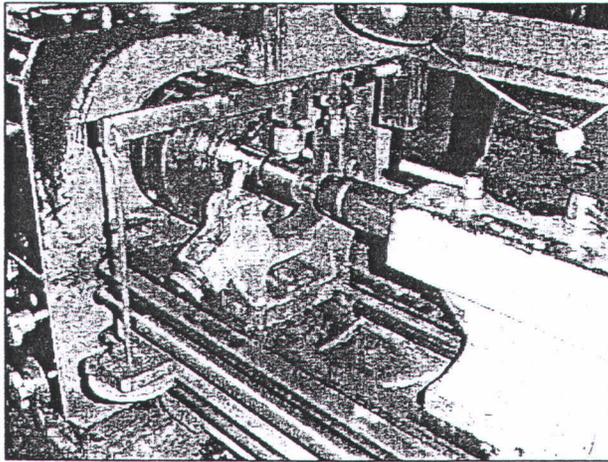
Typical viscosity and viscosity index characteristics of commercially available neat cutting oil as compare to palm oil, Refined Palm Olein (RPO) and the mixing oil between RPO and coconut oil (50%: 50%) is shown in Tab.1.

As can be seen from Tab.1, palm oil possess the highest value of VI (212.3) as compare to other oil tested so far in this work. Restated, the viscosity of a high VI oil changes less than that of a low VI oil for a given temperature change. The oil's viscosity does not reduce as much when exposed to high temperatures, and does not increase as much as other oils when exposed to cool temperature, to some extent though.

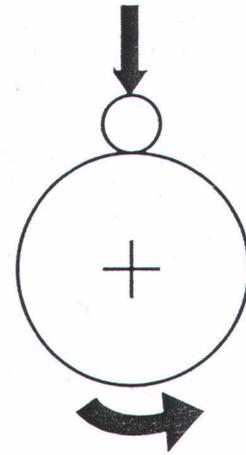
Further analysis is carried out to study the anti-wear characteristics of the palm –based oil used in this work. These include palm oil, RPO and RPO:coconut oil.

Table 1. Vicosity and Viscosity Index in comparison.

	Viscosity @ 40°C	Viscosity @ 100°C	VI
Industrial Neat Cutting Oil	32.0	7.5	114.2
RPO: Coconut	33.5	7.1	181.8
Palm Oil	45.4	9.9	212.3
RPO	45.5	9.4	196.0



a) Wear tester in action.



b) Schematic diagram of wear tester.

Fig.2. Wear tester arrangement.

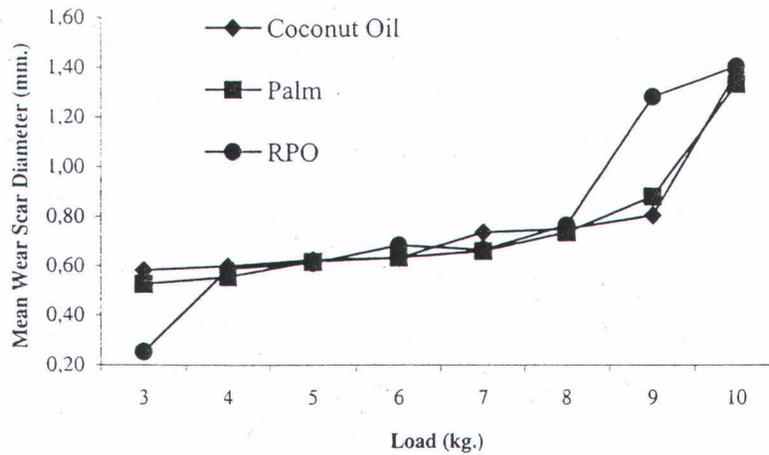


Fig.3. MWSD vs applied load.

3. Assessment for palm oil anti-wear property

The Scuffing Ball on Cylinder Lubricity Evaluator (SBOCLE) (ASTM D-6078, 1997) was performed. In short, a steel ball is loaded against a rotating steel cylinder. A lubricant is fed into a point

contact. In this part the comparison between three of the above palm oils are compared. Typical arrangement of the wear tester is shown in Fig.2. A plot of Mean Wear Scar Diameter (MWSD) of the steel ball versus applied is shown in Fig.3.

In general, palm oil is more or less exhibits similar anti-wear behavior to other oil tested. In this particular context, palm oil was selected to be only a candidate to perform a drilling test in comparison its performance with a commercial neat cutting.

4. Drilling test

A comparison test on a commercial reference neat cutting oil product was tested in comparison with a straight palm oil without additives. All operation were carried out on rectangular specimen, at a thickness of 10mm, made of low carbon steel (SM 490C), utilising a vertical drilling machine. The specimen was not moved between subsequent operations. The positioning accuracy was within a range of 0.1mm. The specimen was immersed in cutting oil reservoir containing 3 litre of cutting fluid. The cutting speed was fixed at 1400rpm. The feed was at 100m/min. throughout. Before the test, every tools was cleaned with appropriate solvent, checked for burrs or breaks on the cutting edge visually and dried in dry air. The characteristics of worn drilling bit were examined and evaluated. Generally, the drilling bit wear mechanisms can be classified into several types. These types of wear are: outer corner wear, flank wear, margin wear, crater wear and chisel edge wear. These are illustrated in Fig.4 together with chipping of the lip. The dominant types of wear which result in drill failure and breakage are: chisel wear, outer corner wear, flank wear and margin wear. The effect of each these four types of wear from both cutting fluids will be described and discussed below.

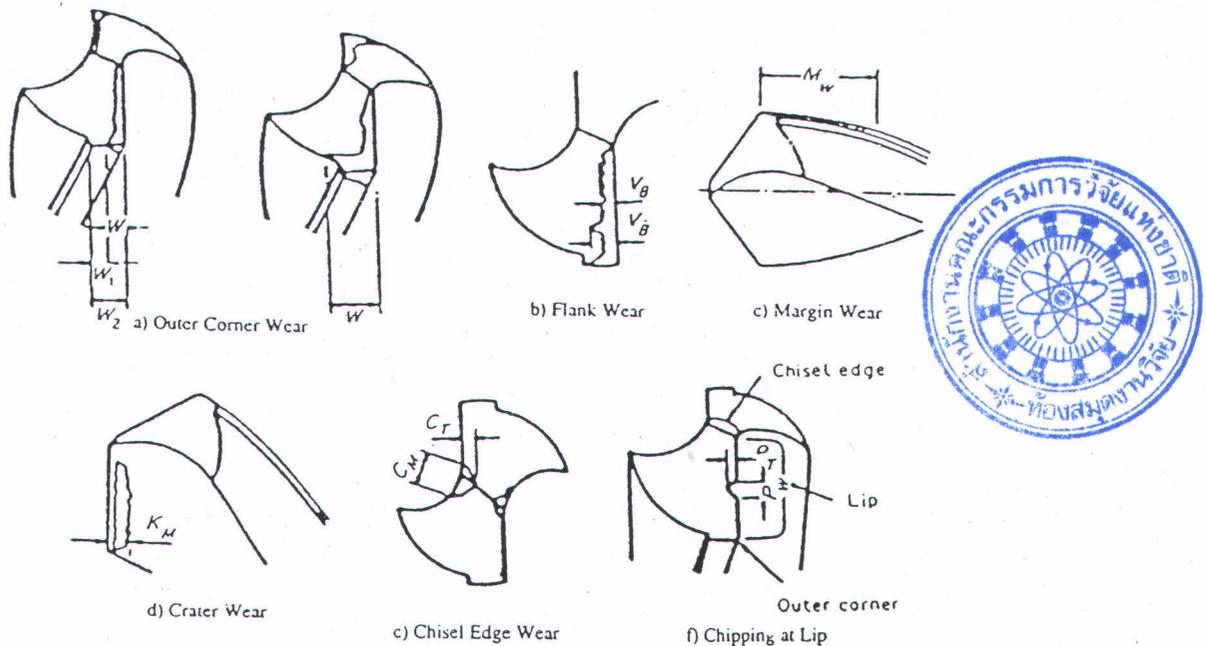


Fig.4. Common wear mechanisms of drill bit (El-Wardany *et al.*, 1996).

A result from the drilling wear test can be seen in Fig.5. In comparison, the worst case with the worn drill bit assessment was for the test without cutting fluid. Severe adhesive wear, another word "Build Up Edge": BUE, has been observed.

This can be evaluated as the flank wear of the drill bit. In contrast, the test with commercial neat cutting oil showed the least wear of the drill bit. This implies that with proper based-oil and appropriate additives was the major factor for better drill bit performance. The anti-wear performance of straight palm oil

was in between. This can be deduced that if proper selection of additive package is employed, there is a feasibility to blended a palm-based neat cutting oil for industrial use.

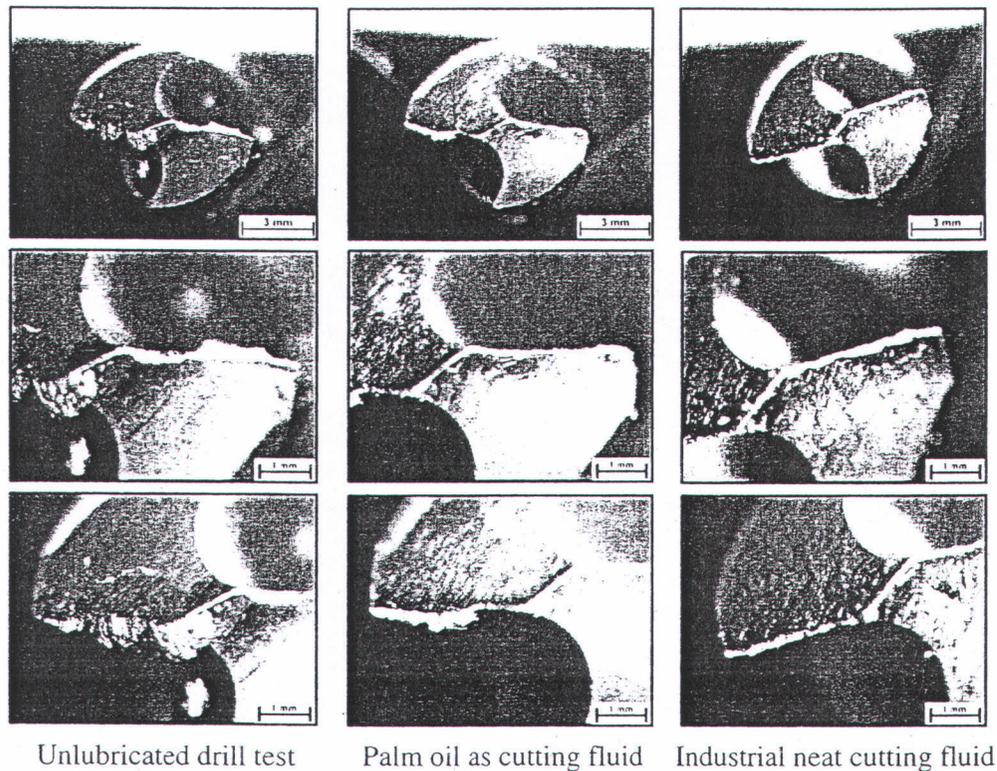


Fig.5. Typical worn drill bit with different cutting fluids.

5. Conclusion

A study concerning testing palm oil as cutting fluids has been carried out. A sequence involving viscosity, VI , anti-wear assessment and also drilling performance analysis was performed. Drilling could be used as a screening test for BUE reduction property of a cutting fluid. On the positive sides, the palm oil possess the highest VI and its lubricity under boundary lubrication is not too far inferior to that of mineral-based oil. Most importantly, vegetable oil is biodegradable, in general are less toxic and reduce dependency on imported mineral-based oil. These however, its anti-wear and extreme pressure property yet to be improved, as the palm-based cutting oil used in this work was a straight palm oil.

Acknowledgment

The Thailand Research Fund (TRF) has supported this work.

References

- ASTM D-2270 (1993): *Practicing for calculating viscosity index from kinematic viscosity at 40°C and 100°C.* – vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-445 (1994): *Test method for kinematic viscosity of transparent and opaque liquids.* – vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.

- ASTM D-6078-97 (1997): *standard test method for evaluating lubricity of diesel fuels by the scuffing load ball-on-cylinder lubricity evaluator (SLBOCLE)*. – vol.05.02, Petroleum Products and Lubricants (II): D 2597 – D 5357.
- El-wadany *et al.* (1996): *Tool condition monitoring in drilling using vibration signature analysis*. – International Journal of Machine Tools Manufacturing, vol.36, No.6, pp.687-711.
- Honary L.A. (1996): *An investigation of the use of soybean oil in hydraulic system*. – Bioresource Technology Journal, vol.56, pp.41-47.
- Machado A.R. and Wallbank J. (1997): *The effect of extremely low lubrication volumes in machining*. – Wear Journal, vol.210, pp.76-82.

PALM OIL AS HYDRAULIC FLUIDS

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The used of vegetable oils and animal fats for lubrication purposes have been practices for years. With the discovery of petroleum and the availability of inexpensive oils, alternatives became unattractive and were left by the wayside. Attention was refocused on vegetable oils during wartime and oil shortage situations. For example, during World War I and II, the use of vegetable oils for fuel, lubricants, greases and energy transfer increased rapidly. Also, the oil embargo of 1973 brought needed attention to alternatives for petroleum oils. In addition, the pressure for increased use of vegetable oils in lubricants, due to both environmental and health and safety considerations, is likely to continue. In addition, economic trends are likely to make vegetable oils increasingly more attractive than in the past in many areas of the globe. All these factors will probably result in an increase use of vegetable oil as lubricants. In this work, basic research on palm oil-based hydraulic fluid properties assessment will be carried out.

Key words: palm oil, hydraulic fluid.

1. Introduction

Some industrial applications impose demands on lubricants and thus require very stable oil. Typically, stable oils are more expensive and need to be specially formulated and blended. Other applications are less demanding, and for those the oil may be formulated with off-the-shelf products. Due to the environmental impacts of used mineral-based industrial oils, there is an increasing need to develop alternatives. Substitution of any of the industrial lubricants with vegetable oil will prove to be of significant economic and environmental advantage. Economically, disposal of the used oil will be less expensive as the vegetable-based industrial oil could be disposed of safely with minimum expense. In addition, prices of mineral - based industrial oils vary based on the application and requirements. This provides opportunities to add value to some of the vegetable oils i.e. palm oil in this particular paper. Environmentally too, the used of vegetable-based industrial lubricants will be less harmful to the ecological system, and in certain applications they could be helpful to the environmental as well. There are also other benefits to vegetable oils, such as bring safer for human-beings when exposed to the oil or its fumes. If even a small portion of the annual industrial oil consumption are substituted by the palm-based oils, the results will have significant economic impact on the Thai agricultural industry. However, the real potential will be limited by the extent to which manufacturers of industrial lubricants are economically and environmentally enticed to the use of palm oils. Considering the price of palm oil and the environmental regulations for conventional industrial lubricants, it is not too optimistic to estimate that the market for palm oil-based industrial lubricant will be much larger to some extent.

Current efforts in the research and development of vegetable-based industrial lubricants seem to be concentrated on the use of canola, rapeseed, sunflower, soybean oil and other oils, and even fish oil (Kassfeldt and Dave, 1997; Honary, 1996; Christensen and Bimbo, 1996; Krzan and Vizintin, 2003). In this particular paper, the purpose of the study was to carry out a fundamental study in order to identify the potential of palm oil for use in hydraulic system.

2. Palm oil general characteristic assessments

2.1. Viscosity and viscosity index

Viscosity is the property of a liquid that determines its resistance to flow, resulting from its internal friction among the molecules. In oils, viscosity is affected by temperature, increasing as the temperature drops and decreasing as it rises (ASTM D-445, 1994). On top of the viscosity value, the Viscosity Index (*VI*) is generally calculated. The *VI* is an empirical number indicating the rate of change in viscosity of an oil within a given temperature range. A low viscosity index signifies a relatively large change in viscosity with temperature, whereas a high *VI* shows a relatively small change in viscosity with temperature (ASTM D-2270, 1993).

2.2. Pour point

The pour point of a petroleum oil is the lowest temperature at which the oil will pour or flow under prescribed conditions when it is chilled without disturbance at a fixed rate. To determine pour point, a sample is cooled in a test jar under specified conditions; the temperature is observed in increments of 5°F until no movement is apparent on the surface of the oil when the test jar is held in a horizontal position for 5 seconds. This temperature is recorded as the solid point. By definition the pour point is 5°F above this temperature (ASTM D-97, 1993).

Table 1. Summarised viscosity/*VI* and pour point values of palm-based oils vs industrial hydraulic oil.

	Viscosity @ 40°C	Viscosity @ 100°C	<i>VI</i>	Pour Point
Hydraulic ISO VG 46	49.5	7.5	114.2	-15
RPO: Coconut	33.5	7.1	181.8	19
Palm oil	45.4	9.9	212.3	15
RPO	45.5	9.4	196.0	12

2.3. Anti-foam characteristic

It is important to evaluate the foaming tendency of oils. Poor anti-foaming properties lead to serious problems in high oil agitation situations, such as high speed gearing, high-volume pumping and splash lubrication (ASTM D-3519, 1982). Foaming contributes to overflow loss, inadequate lubrication, potential cavitation and mechanical failure.

Table 2. Quantitative assessment of anti-foam test in comparison between vegetable oils vs hydraulic oil.

	RPO: Coconut oil (1:1)	Palm oil	RPO	Industrial hydraulic ISO VG 46
F_m (cm)	$5.2 - 4.5 = 0.7$	$4.7 - 4.5 = 0.2$	$5.0 - 4.5 = 0.5$	$4.8 - 4.5 = 0.3$
F_r (cm)	$5.0 - 4.5 = 0.5$	$4.7 - 4.5 = 0.2$	$4.9 - 4.5 = 0.4$	$4.7 - 4.5 = 0.2$

Note:

$$F_m = M - I$$

F_m = Maximum foam height

M = Maximum total height at zero time

I = Initial height

F_r = Residual foam after 5 minutes

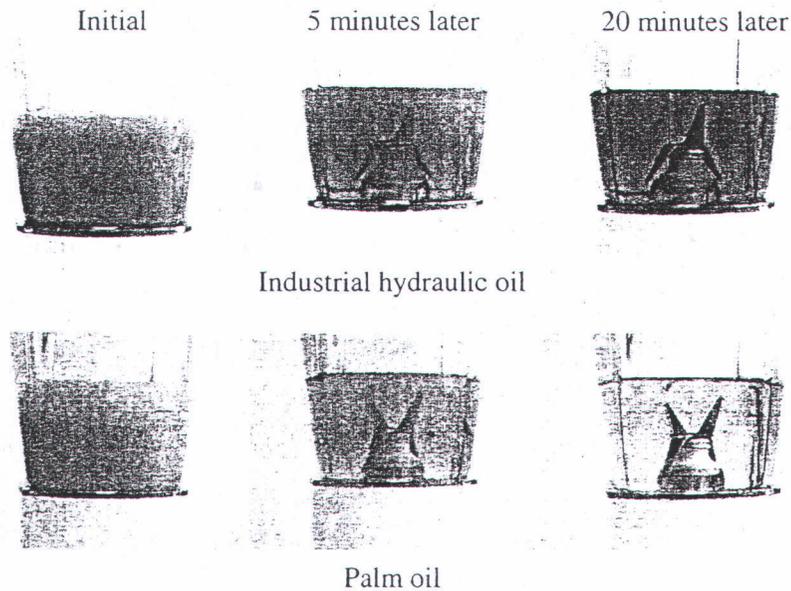


Fig.1. Qualitative assessment of anti-foam (Blender) test in comparison between palm oil vs industrial hydraulic oil.

2.4. Demulsification property

To ensure proper lubrication and to avoid corrosion of polished steel surfaces, it is important to evaluate the demulsibility properties of hydraulic oil and also medium to high viscosity circulating oils. It is a method that measures the ability of an oil to separate from water under static conditions (ASTM D-2711, 1993).

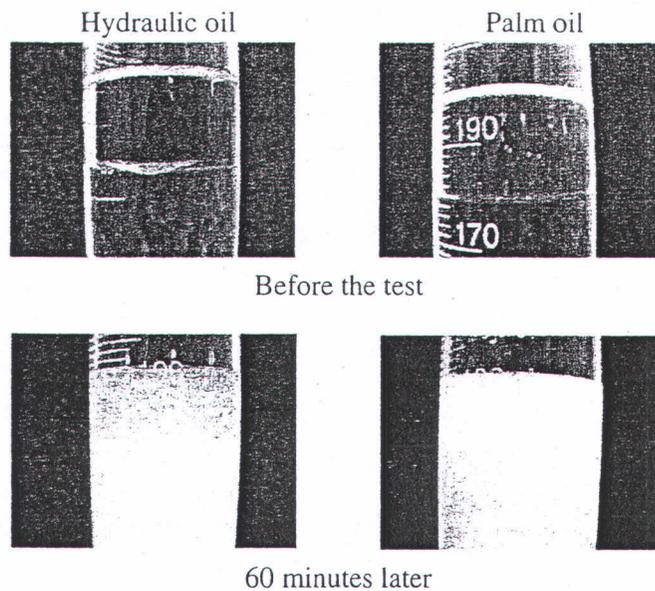


Fig.2. Demulsification property in comparison between industrial hydraulic oil vs palm oil.

2.5. Seal compatibility characteristic

This method covers the procedure for measuring physical properties of elastomer seals in the form of O-rings after exposure to industrial hydraulic fluids and thermal aging. The measured properties are then

compared to the physical properties of elastomer seals that have not been exposed to the industrial hydraulic fluids and thermal aging. The changes in these properties form a basis for assessing compatibility when these changes are compared against the suggested limits (ASTM D-6546, 1994).

Table 3. The change in volume as the O-ring get exposed to tested oil after 2 weeks.

	Weight in air (g)		Weight in water (g)		% Volume change
	Before	After	Before	After	
Palm oil	0.4230	0.3838	0.0775	0.0756	-0.0107
R.P.O	0.3916	0.3945	0.0754	0.0772	0.0035
R.P.O.: Coconut oil (1:1)	0.3861	0.3797	0.0732	0.0736	-0.0217
Hydraulic ISO VG 46	0.3940	0.3872	0.0757	0.0711	-0.0069

Note:

$$\Delta V, \% = \frac{(M_3 - M_4) - (M_1 - M_2) \times 100}{(M_1 - M_2)}$$

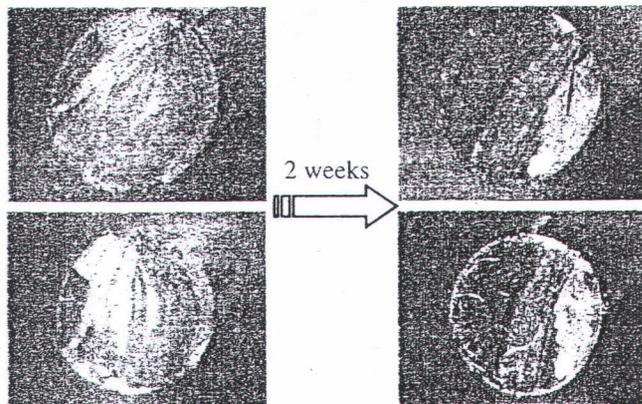
$\Delta V, \%$ = the change in volume or shrinkage

M_1 = initial mass of specimen in air (g)

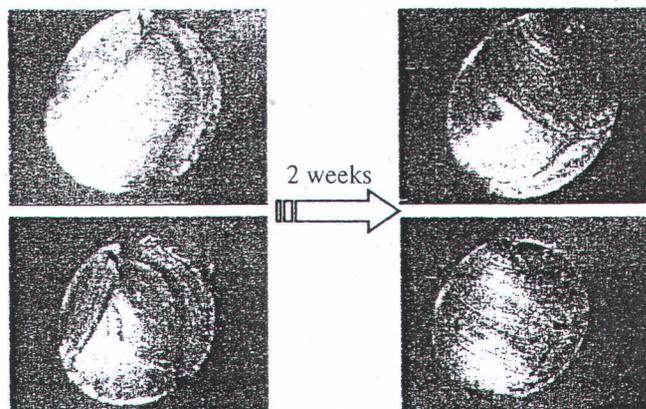
M_2 = initial mass of specimen in water (g)

M_3 = mass of specimen in air after immersion (g) and

M_4 = mass of specimen in water after immersion (g)



Industrial hydraulic ISO VG 46

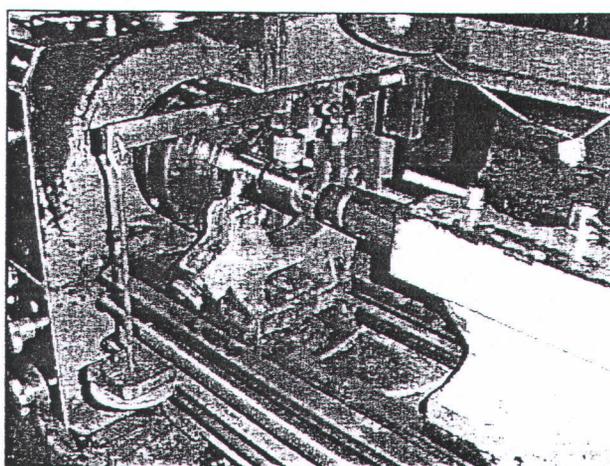


Palm oil

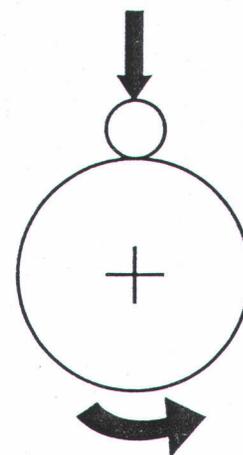
Fig.3. Seal compatibility test in comparison between Industrial hydraulic oil vs palm oil.

2.6. Anti-wear performance assessment

In addition to the above properties, the anti-wear characteristic is also performed. The Scuffing Ball on Cylinder Lubricity Evaluator (SBOCLE) is carried out to get assess to wear characteristic in an extreme conditions (ASTM D-6078, 1997). In this part, the comparison between several vegetable-based oils and commercial industrial hydraulic oil is presented. Typical arrangement of the SBOCLE wear tester is shown in Fig.4. In general, a steel ball is loaded against a rotating steel cylinder. A lubricant, hydraulic oil in this context, is fed into a point contact. A plot of the Mean Wear Scar Diameter (MWSD) of the steel ball versus applied load is shown in Fig.5.



a) Wear tester in action.



b) Schematic diagram of wear tester.

Fig.4. Wear tester arrangement.

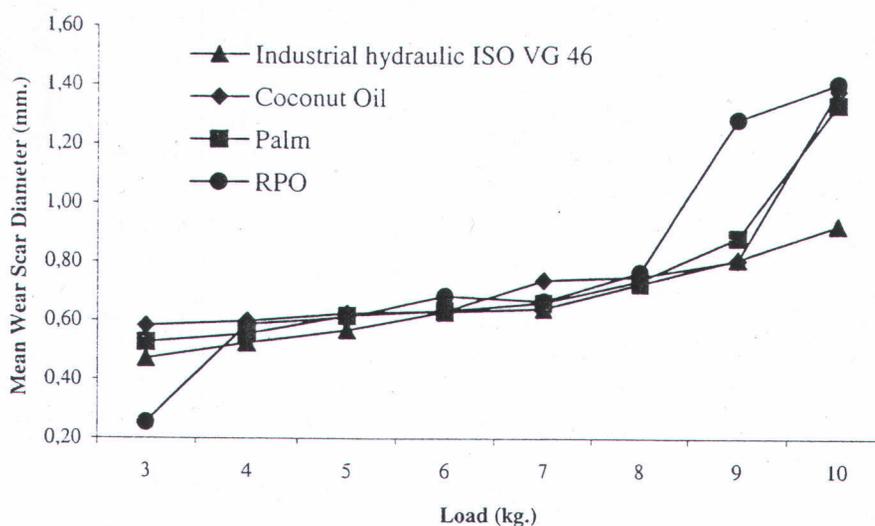


Fig.5. A plot of MWSD versus applied load.

3. Discussion and conclusion

In general, the viscosity and VI of vegetable-based oil is less dependent on temperature and pressure than the viscosity of mineral-based oils. This means that the VI of vegetable-based oil compare well with that

of mineral-based oils, even those with additised mineral oils. Other major properties such as anti-foam, seal compatibility and demulsification, in comparison, in general be considered not inferior to mineral oil based hydraulic fluid. However, the pour point property of the vegetable-based oils tested so far in this work were quite high when compare with that of mineral oil based hydraulic fluid. This can be improved either with proper dosage of pour point depressant or based-oil winterization process. The lubricating properties of vegetable oil remain good and wear is slight, to some extent as can be seen in Fig.5, as long as vegetable oil avoids oxidation, since the oxidation products are caustic acids and sediments that cause wear. However, as in this work, the vegetable oils used were without anti-oxidation additives. Hence, this inferior property can be avoided by using suitable anti-oxidant additives or by utilising "epoxidation process".

On the basis of the results, vegetable-based oils proved from the tests to be not inferior to mineral oil based hydraulic fluid. So vegetable oil with proper additives seems to be adaptable for hydraulic use.

Acknowledgments

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References

- Christensen T.E. and Bimbo A.P. (1996): *Fish oil for use as hydraulic fluid*. – Bioresource Technology, vol.56, pp.49-54.
- Honary L.A. (1996): *An investigation of the use of soybean oil in hydraulic system*. – Bioresource Technology Journal, vol.56, pp.41-47.
- Kassfeldt E. and Dave G. (1997): *Environmentally adapted hydraulic oils*. – Wear Journal, vol.207, pp. 41-45.
- Krzan B. and Vizintin J. (2003): *Tribological properties of an environmetally adopted universal tractor transmission oil based on vegetable oil*. – Tribology International, pp.1-7.
- ASTM D-2270 (1993): *Practicing for calculating viscosity index from kinematic viscosity at 40°C and 100°C*. – vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-2711 (1993): *Standard test method for demulsibility characteristics of lubricating oils*. – vol.05.02, Petroleum Products and Lubricants (II): D 1949-D 3601.
- ASTM D-3519 (1982): *Standard test method for foam in aqueous media (Blender test)*. – vol.05.02, Petroleum Products and Lubricants (II): D 2597 – D 5357.
- ASTM D-445 (1994): *Test method for kinematic viscosity of transparent and opaque liquids*. – vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-6078 (1997): *Standard test method for evaluating lubricity of diesel fuels by the scuffing load ball-on-cylinder lubricity evaluator (SLBOCLE)*. – vol.05.02, Petroleum Products and Lubricants (II): D 2597 – D 5357.
- ASTM D-6546 (1994): *Standard test methods for and suggested limits for determining compatability of elastomer seals for industrial hydraulic fluid applications*. – vol.05.03, Petroleum Products and Lubricants (III): D 3602 – latest.
- ASTM D-97 (1993): *Test method for pour point of petroleum oils*. – vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.

PALM OIL BASED COOLANT FOR METALWORKING

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Cutting oils and coolants are used to reduce friction and wear when cutting or forming metal. As well, they lubricate the cutting area, remove contaminants and prevent corrosion. There are two broad categories of conventional metalworking fluids: neat and water based. Neat cutting oils can be derived from animal, petroleum, vegetable or synthetic sources; however most are petroleum based. Having achieved success in Europe, vegetable oil based metal working oils are now being introduced in other parts of the world. This because vegetable oil cutting fluids are more environmental friendly, less of a health hazard, and superior in "boundary-condition" lubrication mode, they could become the based oil of choice for coolant formation. In this work, the performance of palm oil based coolant was evaluated in comparison with the reference commercial coolants. The aim of the test was to evaluate mineral and palm oil based coolants characteristics. In addition of specific combinations of function formulae, work piece quality and tool wear were compared.

Key words: coolant, metalworking, palm oil.

1. Introduction

Owing to increasingly tight environmental and production cost constraints, one trend in the application of cutting fluids is to move away from mineral oil based to vegetable oil based coolant. Vegetable oils exhibit unique features such as a high natural lubricity; great solvency power and very high viscosity indices (Mang and Dresel, 2001). In addition, these fluids are well to related by the operator's skin.

Despite countless attempts in manufacturing research to avoid cutting fluids, the present state-of-the-art technologies do not seem to assure that cutting fluids will be entirely phase out in the near future. The research in this field should go in the direction of both reducing their environmental impact and optimizing cutting fluid efficiency as well as reduction of quantity usage (Upton, 2000). In this context, cutting fluid based on vegetable oils and ester qualify as potential candidates to replace mineral-based products, because they are almost entirely biodegraded and well compatible with Minimum Quantity Lubrication (MQL) technology.

2. Methods

Currently, the efficiency of cutting fluids is evaluated in many different ways, that are characterized by a variety of different methods and evaluation parameters. Traditionally, cutting fluid performance is evaluated with basic tribological tests such as four-ball, TIMKEN, FALEX, tapping torque and others (Belluco and De Chiffre, 2001; John *et al.*, 2004). It is generally agreed that basic wear testers are poorly correlated with the behaviour of a cutting fluid in actual machining, whereas the closer to manufacturing process, the better correlation with actual production should be expected. Among the machining operations that can be used for cutting fluid fluid evaluation, drilling qualified as potentially interesting. In connection with the development of palm oil based cutting fluids, this study presents performance evaluation carried out in drilling low carbon steel workpiece with high speed steel drilling bit.

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A set of palm oil based coolants as well as two commercial reference products were tested. The performance of five cutting fluids, three of which of palm oil basestock, was tested with respect to tool wear, chip characteristic and surface quality of the workpiece. All tests were carried out on SM490C steel plates. Tool wear characteristic were visually assessed on a photo-micrograph. While chip and surface quality of the drilled holes were evaluated visually and qualitatively. Table 1 shows cutting fluid descriptions used in this work.

Table 1. Cutting fluid descriptions: basic characteristic of test.

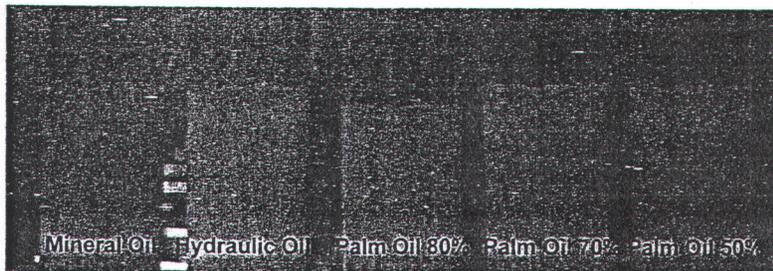
Type of cutting fluid	Base oil	Emulsifier
Palm oil based (80%)	Palm oil 80%	20%
Palm oil based (70%)	Palm oil 70%	30%
Palm oil based (50%)	Palm oil 50%	50%
Hydraulic oil based	Hydraulic oil 80%	20%
Mineral oil based	Commercial coolant	
All cutting fluids were emulsified with water at a ratio of 20:1 (water: cutting fluid)		

Preparation of emulsions

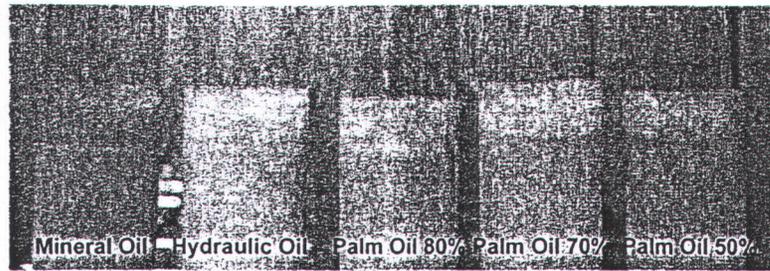
The stability of the emulsion system depends on the size of the droplets and concentration of both water and emulsifier used during emulsion formation. In order to preliminary produce an emulsion, a blender was used at a rotational speed of 500rpm for 10 minutes. Because the volume of the sample was found to influence the emulsification effectiveness, a minimal volume was placed in a closed chamber to present turbulence during blending and prevent loss of material during homogenization. After emulsification, the samples were kept at room temperature. Emulsion stability was judged by the appearance and by observing phase separation, if any, during storage. Figure 1 illustrates the tested cutting fluid appearances before and after emulsification process. It can be seen that they exhibited more or less the same appearances. However, mineral oil based coolant showed a bit more stable of solvency of the emulsion.



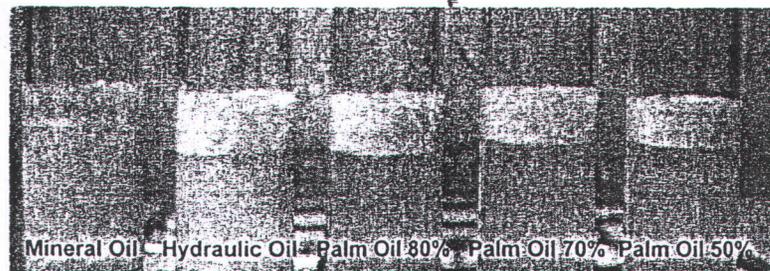
a) Before emulsification.



b) Immediately after emulsification.



c) 30 minutes after emulsification.



d) 24 hours after emulsification.



Fig.1. Appearance comparison of emulsions as time went on.

Test for rust formation

The ability to provide corrosion protection is very important for metalworking fluid. The emulsions were evaluated for rust formation on cast iron. The test for rust formation was conducted as follows. Two grams of cast iron chips were immersed in the emulsion and kept there for 10 minutes in a water glass. The container was covered. After 10 minutes, the solution was removed by tilting the water glass. The rust preventive effect was observed visually after 24 hours. A rating of 10 points corresponded to no appearance of rust. Figure 2 shows the appearance of all the tested coolants which were all rated as 10 points.

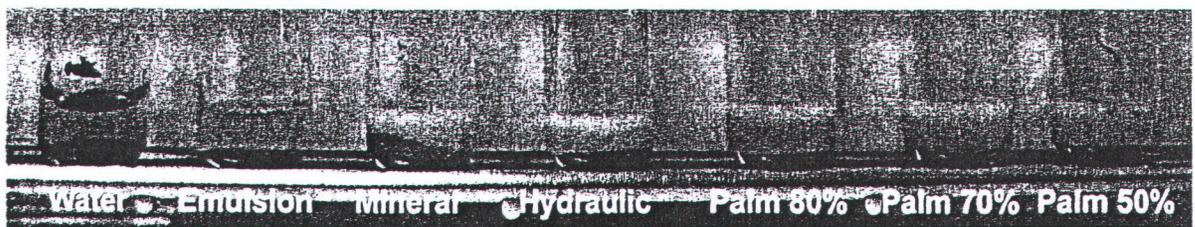


Fig.2. Typical appearance of all tested cutting fluids.

From Fig.2 above, none of the emulsions studied show any rust formation within 24 hours. In this present investigation, all the emulsions tested show excellent anti-rust properties.

Drilling test experiments

In this study, the production of shallow holes with a set of five different coolant formulation was used in a drilling test. This provides information on the performance of coolants which can be reflected by the observation of chip characteristics, tool wear mode/mechanism and surface roughness of workpieces. The objective of this part was to determine the optimum concentration of the palm oil based coolant and the

emulsifier. Drilled holes were produced with an 5mm diameter HSS drilling bit. A set of ten holes, each 30mm deep, were made at a cutting speed of 2200rpm and feed rate of 40mm/min. with flood coolant method. Figure 3 shows typical appearance of drilling bits used during the test series.

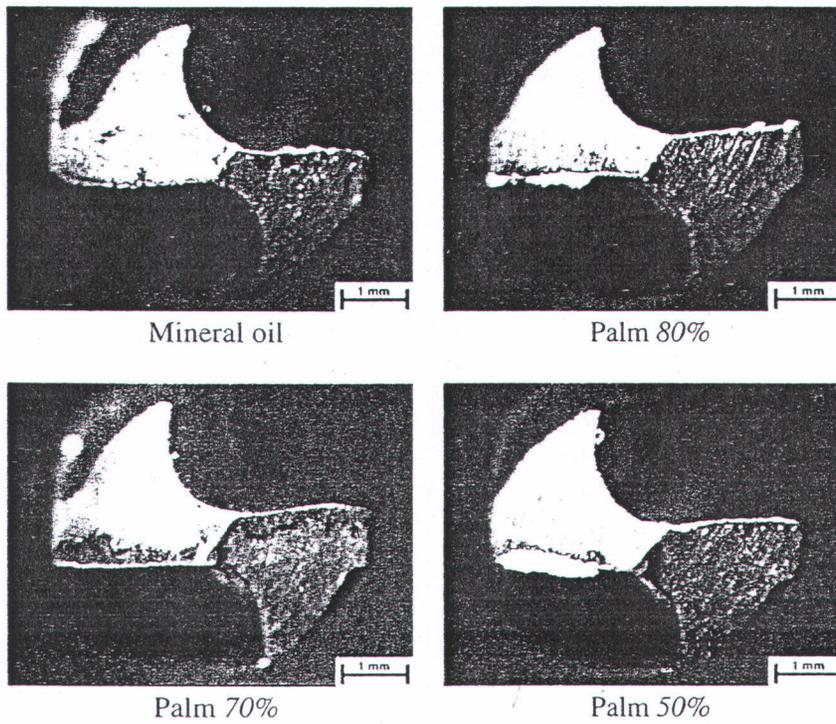


Fig.3. Drill bit appearance from each tests.

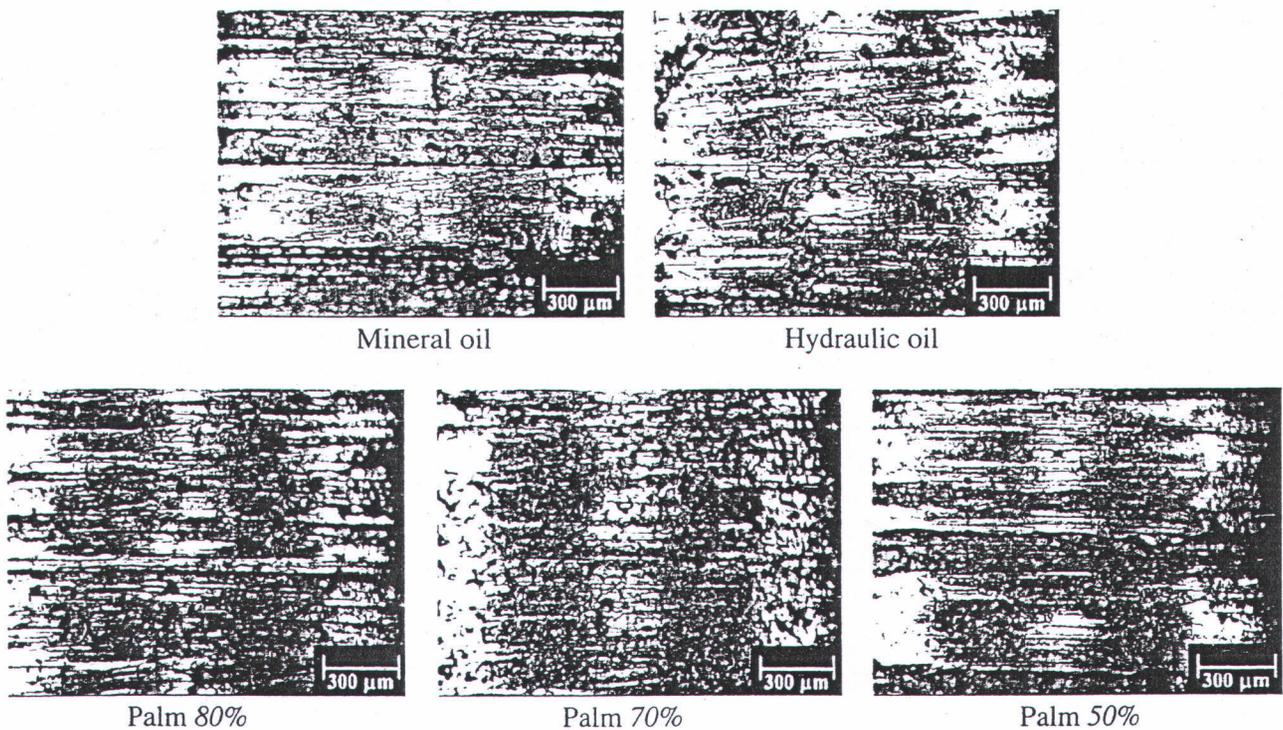


Fig.4. Typical workpiece quality appearance from each tests.

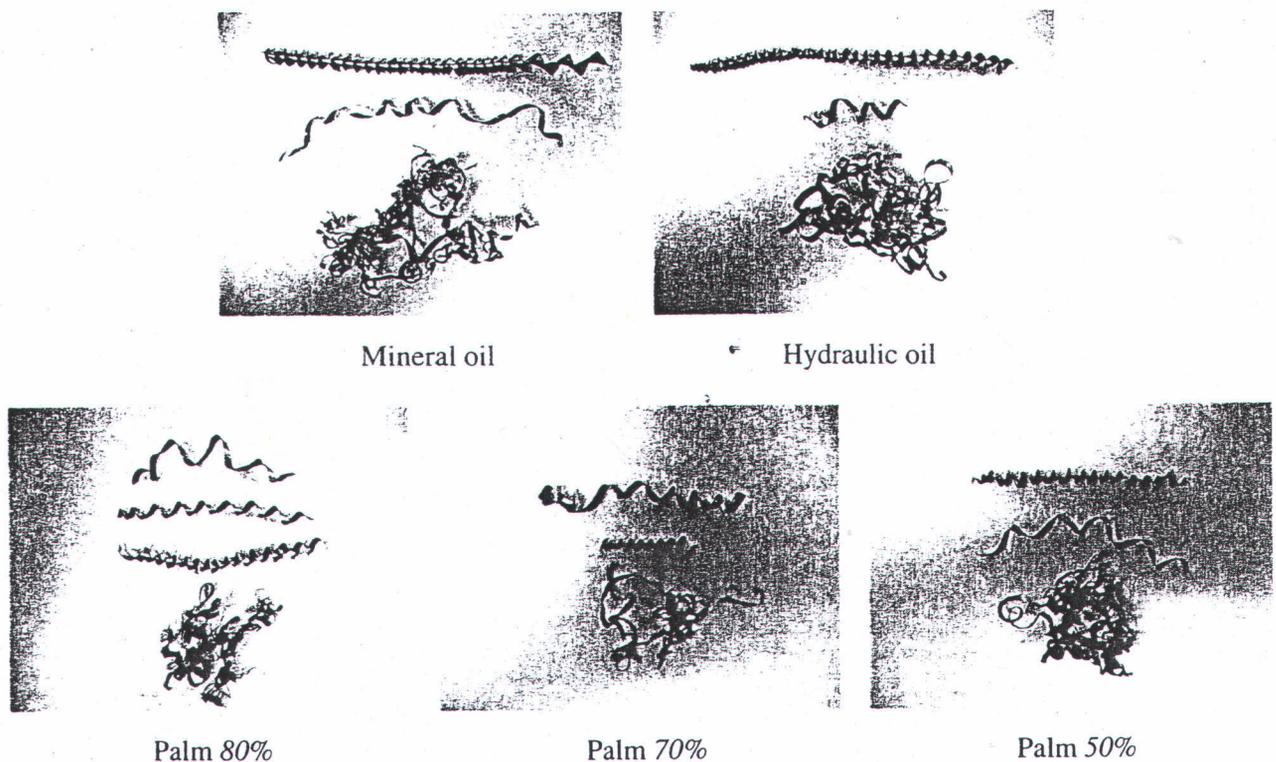


Fig.5. Typical swarfs appearance from each tests.

Note that the drill bit from reference commercial cutting fluid showed the least of Build Up Edge (B.U.E.) while the palm oil based cutting fluid at concentration of 50% palm oil had the largest B.U.E. in comparison. One of the reason may come from that all of the palm oil based coolants contain no additive, except emulsifier at appropriate level which depend on particular blending. From Fig.3, it seems that the concentration of palm oil at 70% provided the best performance among the palm oil based coolants. However, it can be seen from Figs.4 and 5 that the appearances of both surface finish and swarf from all tests are more or less in similar patterns.

3. Conclusion

A study concerning testing cutting fluids with quantitative and qualitative assessments of emulsion stability, rust prevention property, tool wear, surface finish and chip feature from hole making operation on low carbon steel specimens were carried out. According to the results of the present investigation, palm oil based cutting fluids that were developed and tested could achieve at least equal to the reference commercial mineral based oil cutting fluid in drilling operation.

Acknowledgement

This work was financed by the Thailand Research Fund (TRF).

References

- Belluco W. and De Chiffre L. (2001): *Testing of vegetable-based cutting fluids by hole making operations.* – Lubrication Engineering, vol.1, No.8, pp.12-16.

- John J., Bhattacharya M. and Raynor P.C. (2004): *Emulsions containing vegetable oils for cutting fluid application*. – Journal of Colloids and Surfaces A: Physiochem. Eng. Aspects, vol.237, pp. 141-150.
- Mang T. and Dresel W. (2001): *Lubricants and Lubrication*. – Federal Republic of Germany: Wiley-Vch GmbH, Weinheim.
- Upton D.P. (2000): *Optimization of cutting fluid performance*. – Int. J. Prod. Res., vol.38, No.5, pp.1219-1223.

PALM BASED OIL ANTI-OXIDATION BEHAVIOUR IMPROVEMENT: UTILIZATION OF EPOXIDATION PROCESS

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The pressure for increased use of vegetable oils in lubricants, due to both environmental and health & safety considerations, is likely to continue. In addition, economic trends are likely to make vegetable oils increasingly more attractive than in the past in many areas of the globe. All these factors will probably result in an increase use of vegetable oil as lubricants. In this work, systematic research on palm based oil anti-oxidation characteristic assessment was carried out. Mainly, the utilization of "epoxidation process" has been exposed. The key influencing epoxidation parameters on the response variable "anti-oxidation characteristic" has been assessed and reported.

Key words: epoxidation, palm oil.

1. Introduction

Until now, in Europe rapeseed and sunflower oil as lubricant base oils have been in the forefront. However, the disadvantages of these type of base oils are: oxidative, hydrolytic and thermal stability of these base oils is not adequate to allow their use in circulating system. In addition, high-performance additives for vegetable oils have not undergone sufficient development, especially those which are eco-toxicologically safety and ecologically harmless (Mang and Dresel, 2001). Hence, in this work, the application of chemical reaction process in anti-oxidation behaviour improvement for the palm based oil is proposed. It has been accepted that vegetable oils generally have weakness in their oxidative stability because they have fewer number of double bonds in their chemical structure when compared to those of mineral based oils. Epoxidation is one of the most important double bond addition reactions (Okieimen *et al.*, 2002). In the case of unsaturated fatty acid esters, it is often performed using the performic acid method. At present the vegetable oil epoxides are used in PVC and stabilisers. Furthermore, they are also used to improve the lubricity in lubricants. Because of their good lubricity and high oxidation stability in comparison to palm oil, pure epoxidised palm oil should also be used as a lubricant base fluid (Adhvaryu and Erhan, 2002).

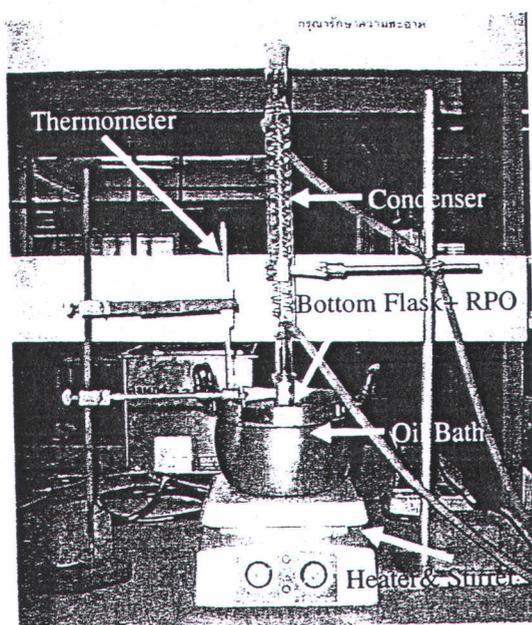
2. Methods

Epoxidation of palm oil was carried out by using peracetic acid. In a typical experiment, a known amount of palm oil was placed in a 500ml flask containing the required amounts of peracetic acids, condenser, thermometer and a stirrer. The flask was allowed to attain the reaction temperature in a thermostat bath (controlled to better than $\pm 2^\circ\text{C}$). The stirring rate of a stirrer was controlled so that the oil in the mixture was finely dispersed. The reaction was monitored by withdrawing aliquots of the reaction mixture at various time intervals into large excess of cold water in a separatory funnel. The aqueous layer was drawn off and the oil layer was washed successively with warm water until it was acid free. The level of epoxidation in the oil was determined using the measurement of Iodine value (Wijs method). Test apparatus was set up as shown in Fig.1. Table 1 shows typical characteristics of palm oil used in this work.

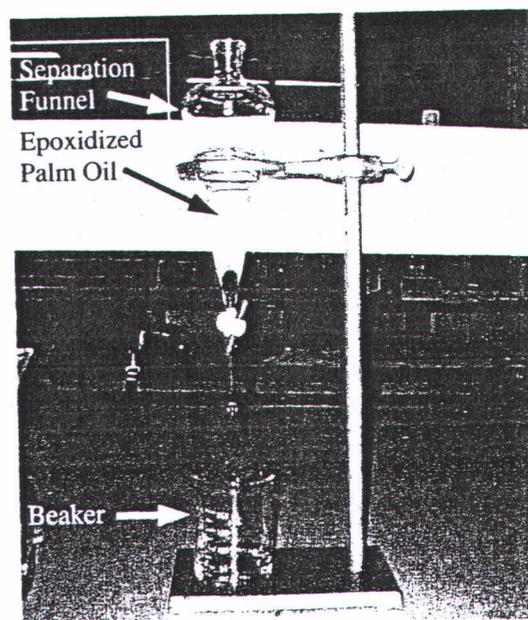
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Table 1. Physio-chemical characteristics and fatty acid profile of palm oil.

Parameters	Values
Pysio-chemical characteristics	
Specific gravity (21°C)	0.898
Viscosity (21°C) cSt	88.6
Flash point (°C)	167
Viscosity Index (VI)	150+
Iodine value (Wijs method)	55
Fatty acid and composition (%)	
Palmitic acid	43.9
Oleic acid	39.0
Linoleic acid	9.5
Stearic acid	4.9
Myristic acid	1.3
Lauric acid	0.9
Caprylic acid	0.1
Capric acid	0.1



a) Epoxidation process.



b) Epoxidised palm oil separation process.

Fig.1. Experimental set-up for palm oil epoxidation process.

Kinetics of epoxidation

The chemistry of epoxidation process may be explained in terms of the following reaction process:
Formation of Peracetic acid



Effect of temperature on epoxidation process

From Fig.3 above, as noticed that the optimal Peracetic acid level is at 40%. This concentration is picked up and kept constant throughout the following experiments. The rate of epoxidation of palm oil by Peracetic acid generated, at 40% concentration, at various temperatures are shown in Fig.4. The results show that the initial increase in the extent of epoxidation with reaction time reached maximum value at 55°C (for epoxidation at 35, 45, 55, 65 and 75°C). The reductions in the levels of epoxidation observed for reaction at 65 and 75°C are low and was highest at 55°C. These results suggest that optimum levels of epoxidation (at 40% Peracetic concentration) could be attained at moderate reaction temperatures (50 to 60°C) at which epoxidation degradation would be minimal.

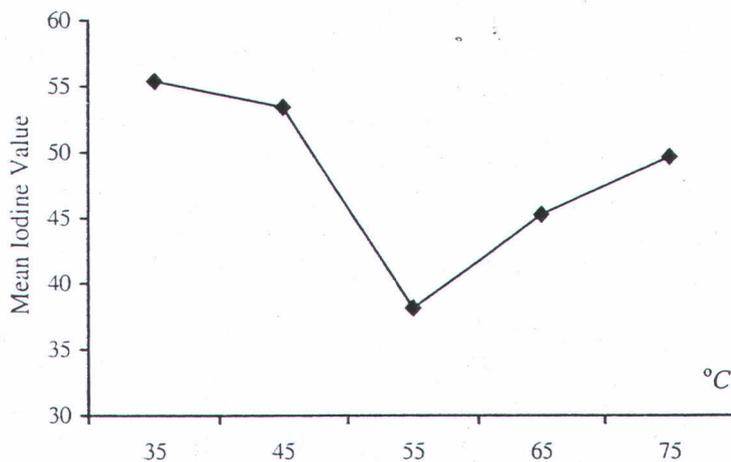


Fig.4. Effect of temperature on epoxidation of palm oil (at 40% concentration of Peracetic acid).

Effect of epoxidation processing duration

Figure 5 shows an effect of test duration on the level of Iodine value. These results suggest that optimum levels of epoxidation could be attained at moderate epoxidation duration (8 hours) at which epoxidation reaction would be minimal.

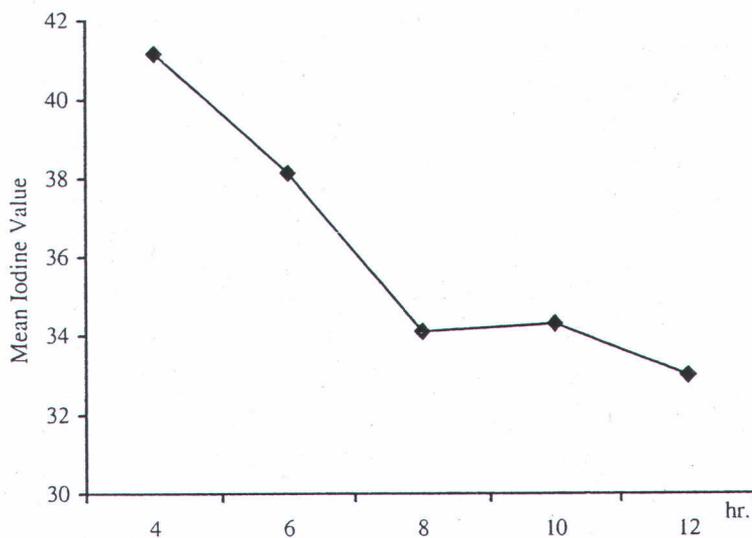


Fig.5. Effect of epoxidation reaction time on Iodine value.

Effect of stirring speed on epoxidation process

The rate of epoxidation of palm oil at various stirring speeds are shown in Fig.6. The results show that at some particular speed the extent of epoxidation with reaction time reached optimum value. The reductions in the level of Iodine value, in another word "epoxidation", observed for speed of 400rev/min.

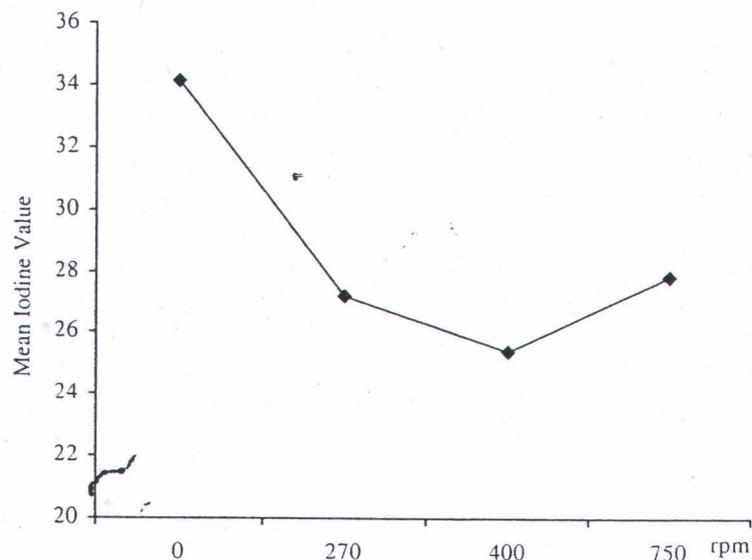


Fig.6. Effect of stirring speed on Iodine value of epoxidised palm oil.

4. Conclusion

Vegetable oils have particularly good tribological properties. Due to their specific structure, they combine good boundary friction lubricity and general wear protection with stable viscosity-temperature behaviour and very low evaporation. In addition, they are easily biodegraded. All-in-all, they are excellent raw materials for the formulation of ecologically friendly lubricants. All their highly positive physical features are countered by a few limitations, the most important of which is the inadequate aging resistance of these products, another word the oxidation stability or anti-oxidation characteristic. As a result they are either not, or only sometimes suitable for circulation lubrication systems. The optimization of vegetable oils refers to specific technical properties such as: hydrolytic stability, foaming, viscosity, evaporation and oxidation stability etc.

In this paper epoxidation process for the modification of palm oil is covered. Success in this area should reduce the dependence on petrochemical raw materials and create new synthesis processes. The challenge for research in the field of lubricants will be to improve certain characteristics of vegetable oils without impairing their excellent tribological and environmentally-relevant properties. This implies the utilisation and sustainment of the natural chemistry of vegetable oils to a high extent.

Acknowledgement

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References

Adhvaryu A. and Erhan S.Z. (2002): *Epoxidized soybean oil as a potential source of high-temperature lubricants*. – Industrial Crops and Products, vol.15, pp.247-254.

Mang T. and Dresel W. (2001): *Lubricants and Lubrication*. – Federal Republic of Germany: Wiley-Vch GmbH, Weinheim

Okieimen F.E., Bakare O.I. and Okieimen C.O. (2002): *Studies on the epoxidation of rubber seed oil*. – Industrial Crops and Products, vol.15, pp.139-144.

PALM OIL-BASED GREASE RHEOLOGICAL PROPERTIES

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Vegetable oils have a long and continuity history of use in lubricants, even though modern lubricants are predominantly based on mineral oil owing to cost and performance considerations. However, recently there has been increased interest in extending the use of vegetable oils in lubricants, driven mostly by environmental as well as health and safety issues and also arising from changes in economic and supply factors; there is, specially in Thailand, a plentiful supply of palm oils domestically.

Most of the published research works have been mainly focused on tribological characteristics of lubricating greases. In fact lubricating grease rheological property assessment is among the most complex systems studied by industrial rheologists. In this particular work some preliminary rheological properties of the palm oil-based grease will be reported.

Key words: vegetable grease, rheological properties.

1. Introduction

In the manufacture of a multipurpose lithium soap grease, for example, based oil, fatty acids are dissolved in a portion of the oil and then saponified with an aqueous solution of lithium hydroxide. This produces a wet lithium soap that is partially dispersed in the base oil and is then dehydrated by heating. After drying, the mixture is cut back with additional oil and additives (optional to this research project as the next step to be carried out in the future work) to produce the desired consistency and formulation characteristics intended of the finished grease (Mortier and Orszulik, 1997). In this work as a basic study on palm based-oil grease manufacture, three main compositions, Lithium Hydroxide (3% by volume), 12-Hydroxysteric (7% by volume) and palm base oil (90% by volume) are blended and used throughout. In this particular work some important rheological behaviors of palm oil has been assessed as follow.

Table 1. NLGI grease grade as per penetration depth.

NLGI number	ASTM worked penetration ($10^{-1} mm$)	Appearance at room temperature
000	445 – 475	very fluid
00	400 – 430	fluid
0	355 – 385	semi-fluid
1	310 – 340	very soft
2	265 – 295	soft
3	220 – 250	medium hard
4	175 – 205	hard
5	130 – 160	very hard
6	85 – 115	extremely hard

2. Rheological properties

2.1. Grease penetration and consistency (ASTM D-217, 1994)

Penetration, with respect to a lubricating grease, is the depth (in tenths of a millimeter) that a standard cone penetrates a sample of the grease under prescribed conditions of weight, time, and temperature. The depth of penetration denotes its resistance to deformation, also known as its consistency. The deeper the penetration, the softer the grease and the lower its NLGI (National Grease Lubricating Institute Standard) grade as shown in Tab.1. Typical penetrometer is shown in Fig.1.

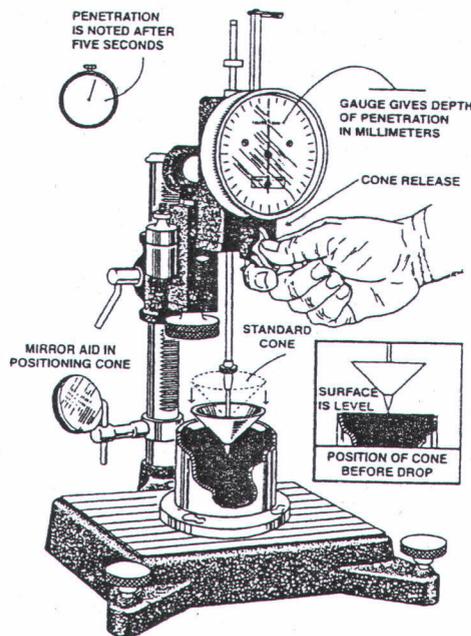


Fig.1. Typical set up of penetrometer.

In this work, a simpler penetrometer is used and it is calibrated with the penetration depth of the standard penetrometer. Typical arrangement of the test apparatus is shown in Fig.2. A good correlation is achieved as shown in Fig.3. It can be depicted that the consistency of blended palm grease used in this work is approximately between NLGI No.2 and 3.

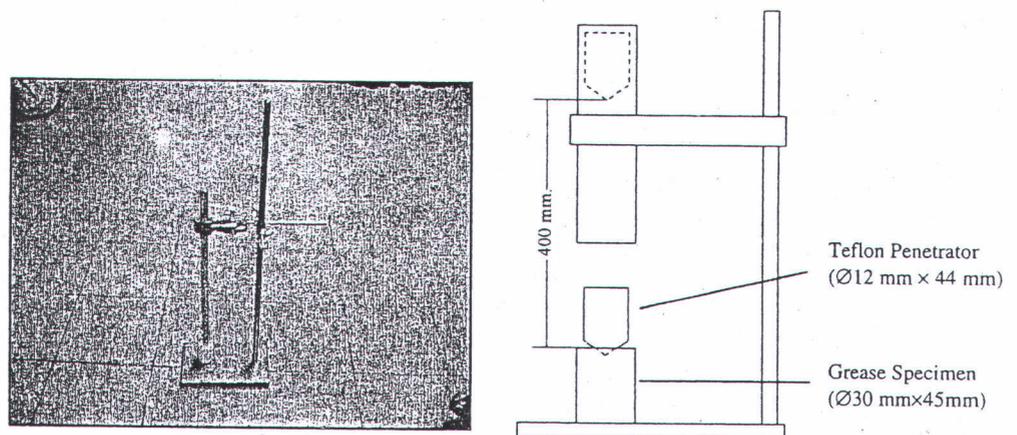


Fig.2. Arrangement of test apparatus.

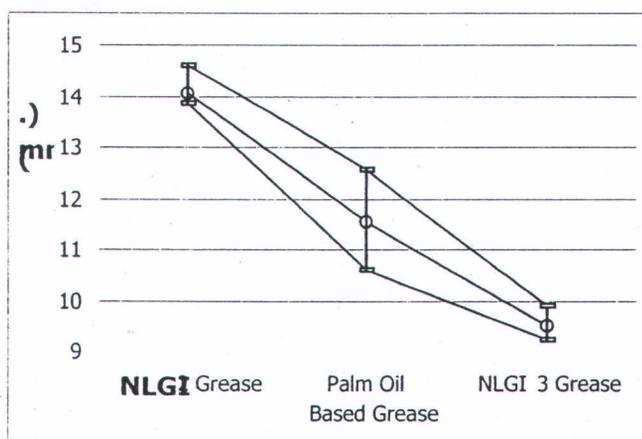


Fig.3. A plot of a penetration depth of the modified test.

2.2. Grease dropping point (ASTM D-2265, 1994)

The dropping point is a qualitative indication of the grease's heat resistance. It is used by suppliers for quality control. It provides an indication of the type of thickener but cannot be correlated with field performance as this is only a static test. It offers an important guideline in grease selection for a given application. Arrangement of grease dropping point as per ASTM D-566 is shown in Fig.4. By performing the dropping test, in comparison with multipurpose mineral oil-based grease with the blended palm grease in this work, the dropping point is within similar range where mineral oil-based grease has its dropping point around 155 to 160°C .

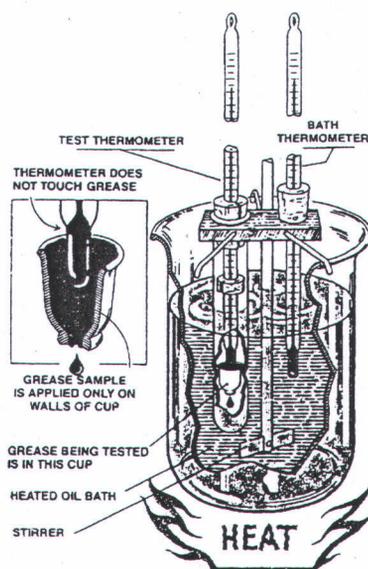


Fig.4. Grease dropping point test setup.

2.3. Water spray-off resistance (ASTM D-1264, 1993)

The test is used to evaluate the ability of grease or residual compound to adhere to a metal surface when subjected to an intense water spray. A stainless steel panel is coated with a $1/32$ " film of grease, weighed, and then sprayed with a high velocity water spray for 5 minutes, held 12 " from the panel surface. The panel is dried and weighed to determine the weight of the grease washed away. The result is given in percent spray-off. A superior grease or residual compound will have a low, 5% or less spray-off. Others

range as high as 98%. Typical arrangement of modified test and test results are shown in Fig.5 and Tab.2 respectively. It can be noticed from Tab.2 that the palm oil-based grease has performed inferior to those from mineral oil-based grease for the water spray-off resistance.

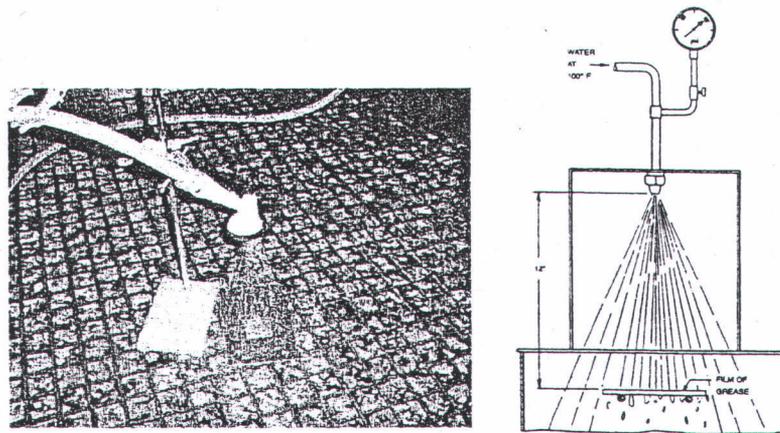


Fig.5. Modified test apparatus arrangement.

Table 2. Water Spray-off resistance comparison between multipurpose industrial grease (mineral oil-based grease) and palm oil-based grease.

	Weight before test	Weight after test	Weight of SS 400 plate	% Grease weight loss
NLGI 2	110.623	110.525	110.379	40.16
NLGI 3	116.073	116.031	115.850	18.83
Palm oil-based grease	114.028	113.891	113.732	46.28

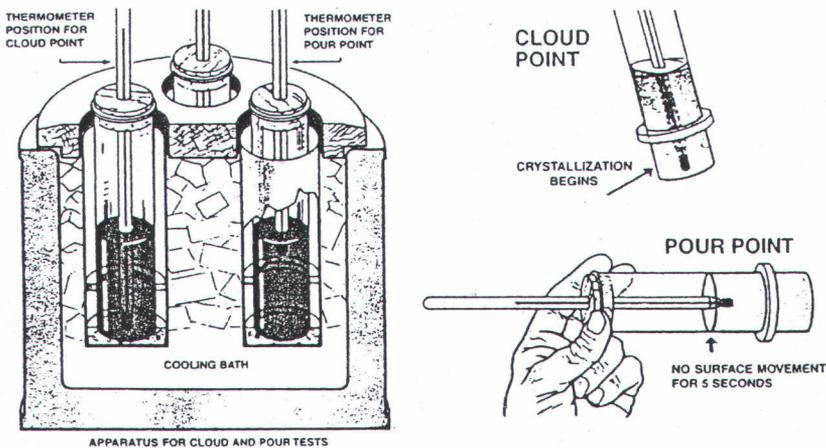


Fig.6. Apparatus for pour point test.

2.4. Pour point of base oil (ASTM D-97, 1997)

The pour point of a base oil is the lowest temperature at which the oil will pour or flow under prescribed conditions when it is chilled without disturbance at a fix rate. To determine pour point, a sample of oil is cooled in a test jar under specified conditions; the temperature is observed in increments of 5°F until no movement is apparent on the surface of the oil when the test jar is held in a horizontal position for 5

seconds. This temperature is recorded as the solid point. By definition the pour point is $5^{\circ}F$ above this point. Apparatus arrangement as per ASTM D97 in pour point determination is shown in Fig.6. Typically, from the pour point test, the palm oil used in the current work has a pour point of $10^{\circ}C$ where common mineral based oil at similar viscosity perform better for the pour point at around less than $-10^{\circ}C$. Hence, the need for pour point depressant will be needed for the blending of palm oil-based grease.

2.5. Corrosion preventive properties of lubricating grease (ASTM D-1743, 1994)

The method evaluates the corrosion properties of grease that may be used in conjunction with rolling contact bearings, either in storage or during idle periods under wet conditions. New, cleaned and lubricated steel balls (AISI 52100 or EN 31 steel) are coated evenly. After the balls are exposed to water, they are stored for 48 hours at approximately $25^{\circ}C$ at ambient relative humidity. After cleaning, the balls are examined for evidence of corrosion. Typical result in comparison between a multipurpose industrial grease (mineral oil-based grease) and palm oil-based grease is shown in Fig.7. It can be noticed that the blended palm grease performs not worst than mineral oil-based grease, at least at similar controlled conditions in this work though.

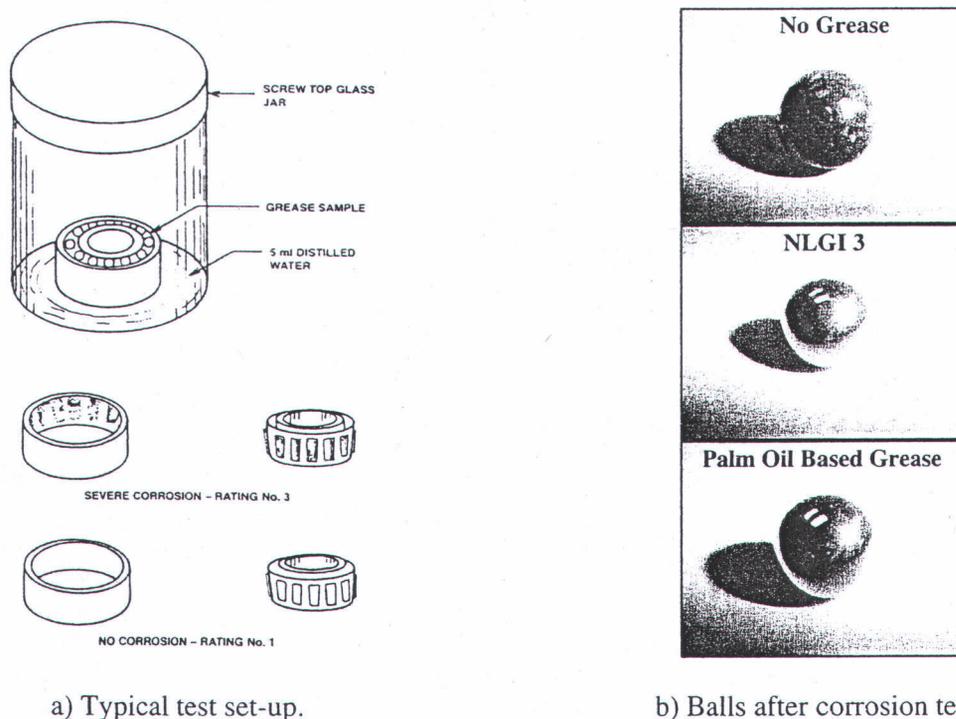


Fig.7. Corrosion resistance of palm oil-based grease versus multi-purpose industrial mineral oil-based grease.

3. Conclusion

Blended palm grease can have several advantages and disadvantages when considered for industrial and machinery lubrication. On the positive side, palm oils have high viscosity index and good lubricity under boundary lubrication, lower price than imported petroleum oils and their biodegradability (Pirro and Wessol, 2001). On the negative sides, specifically for their rheological properties i.e. high pour-point (the temperature at which oil loses fluidity and does not flow). However, this problem can be addressed by either winterization or blended with proper type and quantity of pour point depressants (Moller and Boor, 1986). Proper additive packages and/or other group of thickeners may have to be considered to enhance the water washing ability, dropping point characteristics and also anti-wear and extreme pressure properties.

Acknowledgement

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References

- ASTM D-1264-93 (1993): *Test Method for Determining the Water Washout Characteristics of Lubricating Greases*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-1743-94 (1994): *Test Method for Determining Corrosion Preventive Properties of Lubricating Greases*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-217-94 (1994): *Test Method for Cone Penetration of Lubricating Greases*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-2265-94a (1994): *Test Method for Dropping Point of Lubricating Greases Over Wide Temperature Range*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- ASTM D-97-93 (1993): *Test Method for Pour Point of Petroleum Products*, vol.05.01, Petroleum Products and Lubricants (I): D 56 – D 2596.
- Moller U.J. and Boor U. (1986): *Lubricants in Operation*. – London: Mechanical Engineering Publications Limited.
- Mortier R.M. and Orszulik S.T. (1997): *Chemistry and Technology of Lubricants*. – Suffolk: St. Edmundsbury Press.
- Pirro D.M. and Wessol A.A. (2001): *Lubrication Fundamentals*. – New York: Marcel Dekker, Inc.



