Utilization of sulphurized palm oil as cutting fluid base oil for broaching process

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Utilization of sulphurized palm oil as cutting fluid base oil for broaching process

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Abstract. Broaching is one of the most severe metal cutting operation that requires the use of cutting fluids formulated with extreme pressure (EP) additives to minimize metal-to-metal contact and improve tool life. Enhancement of EP performances of the cutting fluids can be achieved by addition of sulphur containing compounds that will allow the formation of metal sulfide film that has low shear strength and good antiweld properties and acts as protection layer from wear and seizure. Most of the cutting fluids are mineral oil based. However, as regards to health and environmental issues, reseach on vegetable oil based cutting fluid have been increased recently. This paper reports a study on the sulphurization of palm oil derivatives and its usage as broaching oil. Sulphurization of the palm oil derivative was conducted via non-catalytic sulphurization using elemental sulphur at various composition and under heating of 150-160^{o}C for 3 hr. Broaching oil was made by blending the sulphurized palm oil and additive packages. The performance parameters of the broaching oil that has been observed including load carrying capacity, wear scar diameter, corrosion protection, oxidative stability, and surface finish of workpiece. From this research, it was found that sulphurized FAME based broaching oil has excellent EP properties. The optimum formulation was obtained on composition of sulphurized FAME-mineral oil with 6% wt of sulphur. The result from the test showed that kinematic viscosity of sulphurized palm oil was about 25.3 cSt (at 40 ^{o}C), load carrying capacity was 400 kg_f, and wear scar diameter was 0.407 mm. In addition, it can be concluded that the class of corrosion protection of modified palm oil was 1.b (slight tarnish category), oxidative stability at 160 ^{o}C was obtained for 0.11 hr, and the surface roughness of workpiece was about 0.0418-0.0579 µm. These performances are comparable to commercial broaching oil. By this result, it indicates that sulphurized palm oil is applicable for industrial cutting fluids formulation.

1. Introduction

Metal working fluids consumption is increasing as the consequence of machining industry development. Kline & Company Market Analysis (2012) has reported that 38 million tones of lubricants were globally used and 2.2 million tones of it were allocated for metalworking fluid requirements. Asia pacific was the largest market of metalworking fluid with 42% of total world's consumption. North America, Europe, and another region contributed with 28%, 26%, and 4% of total world's consumption, respectively [1]. Metal cutting in manufacturing industry requires cutting fluids for lubricating and cooling the contact surfaces between the cutting tools and the workpieces, so it will generate a product with good quality and good surface texture [2].

Broaching is one of the metal cutting processes where a toothed tool called a broach is run linearly or rotary and pressed into a surface of the workpiece to effect the cut and to remove material.

Broaching process is categorized as a heavy machining process because it works in extreme pressure and relatively high temperature [3]. The heavy machining processes generally require middle or heavy cutting oil, such as neat cutting oils that contain 90% base oil and have better EP performances [4]. Broaching process requires a cutting fluid that contains highly active sulphur. Most of broaching oils is formulated by blending a low viscosity of mineral base oils, i.e paraffinic, and a highly contained sulphur additive [5]. Since it contains mineral oil, the spills dan the waste oil of the broaching oils are considered as pollutant that can possibly contaminate soil and water. Moreover, directly disposal of the waste oils to the environment is environmentally unsafe [6].

The utilization of vegetable oils as the green raw material for cutting fluid application has been studied by several researchers. One of the main objective research is to increase the poor thermo-oxidative stability properties of the vegetable oil due to the presence of bisallylic protons at its structure, to meet the stability requirement during various tribochemical processes. Another objective is to pursue a cutting fluid that has excellent boundary lubricating properties through strong physical and chemical adsorption with the metal surface.

Elimination of the bisallylic protons at the chemical structure of the vegetable oil by incorporating sulphur element into the structure of vegetable oil is considered to be able to improve EP performances and also thermo-oxidative stability. The polar group of sulphur is known as active site of molecules that can increase its adsorption on the metal surfaces to form protection layer [7]. EP performances of vegetable oil may also be obtained by the incorporation of phosphate, chlorine, nitrogen and zinc, however, compounds or additives that contain those elements have been considered as the cause of numerous health and environmental concerns [7,8,9]. Meanwhile, sulphur contaning compounds or additives are well-known for their excellent antiwear properties and EP characteristics, low toxicity, and categorized as a non-carcinogenic additive [9,10].

Incorporation of sulphur to the oils can be done by sulphurization method such as sulphur flower reaction (with and without H_2S, aminic, and other suitable catalysts), sulphur chlorination with S_2Cl_2, organic halides with alkalipolysulfides, and mercaptan route. Today's large-scale production technology in general avoids any halogen-containing reaction steps because there are low limits (max 30 ppm chlorine) in the final lubricants that may not be exceeded [11]. Black sulphurization is the simplest sulphurization method. The manufacturing equipment needs to withstand pressure above 1–2 bars (it may even be pressureless).

In this research, palm and its derivatives are sulphurized with sulphur flower/elemental sulphur. The mixture of oil and elemental sulphur is heated above the melting point of sulphur. An uncatalyzed reaction starts to become exothermic above 150–160°C, with the evolution of substantial amounts of H_2S [11]. This research was aimed to produce sulphurized oil that will be use for formulating broaching oil with maximum viscosity of 107 cSt at 40°C [12] using sulphurized palm oil for steel-made process machines. For broaching process, the desire viscosity of broaching oil is low to allow a good penetration of lubricants. This process will lead into the elimination of chips and contaminants from workpiece and working tools rapidly by fluid current. The high level of viscosity will decrease the efficiency of working tools due to the trapped chips [4]. The formulation of broaching oil include the addition of additive packages, such as antioxidant and anticorrosive additives, into the sulphurized oil and also the blending with other oil. The characterization of sulphurized oil are viscosity, sulphur contents, and the spectrum of FTIR. Performance tests for the formulated broaching oil to workpiece and worktool include EP performance, surface finish, corrosion protection level, and thermo-oxidative stability. Furthermore, these performance tests were also done to a broaching oil of commercial products as comparison.

2. Experimental

This research consists of three main parts: preparation of base oil, synthesis of broaching oil, and performance test of broaching oil. The process flow diagram of the research is shown in the Figure 1.

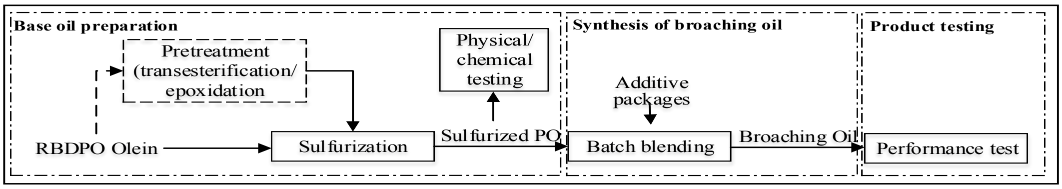


Figure 1. The process flow diagram of the research.

2.1 Base oil preparation and synthesis of broaching oil

Base oil are the products of sulphurization process of RBDPO olein, epoxydized of RBDO olein, FAME and epoxydized FAME. The FAME was prepared by process of esterification of RBDPO olein using methanol and NaOH as catalyst. Epoxydized of RBDO olein and epoxydized FAME were obtain from epoxydation reaction using oxidator hydrogen peroxide formic acid as the catalyst. The sulphurizations were conducted using elemental sulphur of technical grade, which was added into the oil, heated at 150-160^{o}C, stirred at 500 rpm for 3 hr. The condition of temperature, time, and speed rate were remained constant while the quantity of the elemental sulphur was varied at 2, 4, 6, 8, and 10% wt, respectively. The desire sulphurized oil was the high sulphur content with viscosity less than 86 cSt which was considered as the proper viscosity for broaching oil. The selected sulphurized oil was then used in formulation of broaching oils by blending it with additive package which contain sodium tolytriazole as anticorrosion and butylated hydroxy toluen as antioxidant. Mineral base of parafinik HVI 60 was also used as base oil for the purpose of comparison.

2.2 Performance test of the product

Each of the sulphurized oils was measured its viscosity by ASTM D-445 method and the sulphur content by ASTM D-2622 method. Fourier transformation infra-red (FTIR) analysis was carried out to examine the change of functional group of the oil's chemical structure. To examine the degree of unsaturation of the oils was determined by iodine value measurement.

Several parameters of broaching oil performance were examined to obtain the quality of broaching oil. The examinations of EP performance of the broaching oil were conducted by 4-ball test for obtaining wear scar diameter (ASTM D-4172 method) and load carrying capacity-weld point (ASTM D-2783 method). The examination of workpiece's surface finish was carried out by surface roughness tester tool kit. Morphology of the workpiece by scanning electron microscopy (SEM) equipped with energy dispersive x-ray (EDX). Corrosion protection level performance was analyzed by copper strip corrosion test (ASTM D-130), and oxidative stability performance was carried out by induction time measurement with Rancimat-892.

3. Result and discussion

3.1. Sulphurized palm oil characteristic

Sulphurization of the palm oil and its derivatives including RBDPO olein, epoxydized olein, FAME, and EFAME was darkened their color from goldish yellow into brown-blackish proprotional with the addition of sulphur. The rise of sulphur content also caused the increase of the viscosity of the sulphurized palm oil products. Sulphur incorporation of various amount into olein, epoxydized olein, FAME, and EFAME results in various viscosity of sulphurized palm oil as shown in Figure 2. It is apparent that the highest sulphur content with the lowest viscosity can only be obtained by sulphurized FAME. The sulphurization with up to 10 wt% of sulphur content resulted in the sulphurized FAME with viscosity of 33 cSt.

To function as a broaching oil, its viscosity l has to be remained low to allow a good penetration of lubricants. This process will lead into the elimination of chips and contaminants from workpiece and working tools rapidly by fluid current. The high level of viscosity will decrease the efficiency of working tools due to the trapped chips [4]. Base on viscosity consideration, this sulphurized FAME still meet requirement of broaching oil viscosity for effective operation. Another configuration of sulphurized products, especially the sulphurized olein and sulphurized epoxy olein have too high viscosity for effective broaching oil

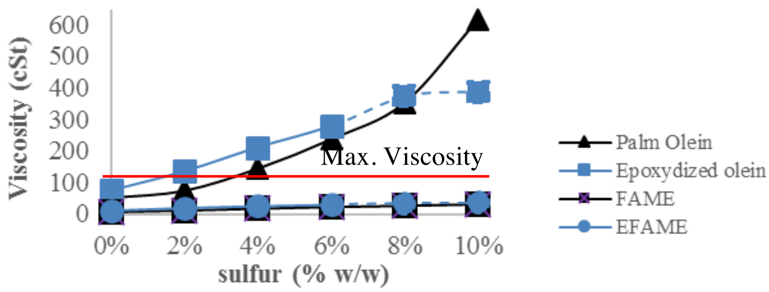


Figure 2. Various viscosity obtained from sulphur incorporation into several palm oil configuration.

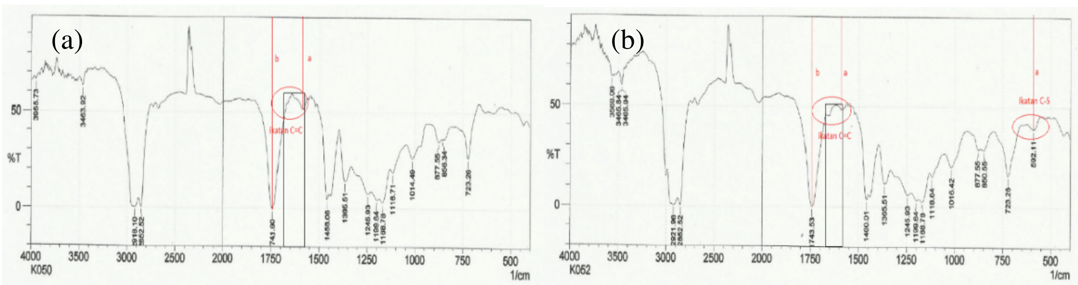


Figure 3. Fourier transform infrared spectrum of FAME: (a) before sulphurization and (b) after sulphurization.

Figure 3 shows the FTIR spectrum of FAME before and after sulphurization. The sulphurized FAME is characterized by the appearance of C-S group (592 cm^{-1}). This confirming the incorporation of sulphur into FAME at the reactive site C=C group. Therefore, theoritically the peak height of C=C group (1620-1680 cm^{-1}) should decrease or disappear after sulphurization. From the spectrum, the ratio peak height of C=C to peak height of C=O (1750 cm^{-1}) after sulphurization is lower than before sulphurization, but C=C peak does not disappear after sulphurization. This fact implied that not all C=C group had been replaced by C-S group. This is in agreement with the result of iodine value measurement shown on table 1, where the iodine value is decrease with increasing the sulphur content, but never reach value of zero

Table 1. Quantitative analysis result of sulphurized FAME.

Theoritical Composition		Actual Analysis Result		
FAME (% wt)	S Content (%wt)	S Content (%wt)	Viscosity (cSt)	Iodine Value (g Iod/100 g)
100	0	0	5.7	47.1
98	2	1.9	11.0	42.6
94	6	6.0	23.4	31.8
90	10	10.0	32.6	30.8
85	15	14.1	39.7	25.9
80	20	18.1	46.3	25.0

3.2 The performance of formulated broaching oil

The formulated broaching oil sample are mixture of sulphurized FAME containing sulphur 6% wt (S FAME 6) plus additive package, mixture of sulphurized FAME containing sulphur 10% wt (S FAME 10), mixture of sulphurized FAME containing 6% wt sulphur plus mineral base oil (paraffinic HVI 60) as combined base oil (S FAME+Min) plus additive package. Commercial product of broaching oil that used mineral oil as base oil (Min Commercial), was used as the comparison. Table 2 shows the matrix of performance test.

Table 2. Matrix of broaching oil performance test.

Configuration of Product	Parameter of Performance Test			
	Extreme Pressure	Surface Finish	Corrosion Protection Level	Oxidative Stability
S FAME 6	4 Ball-scar diameter 4 Ball-weld point	Surface rouhgness Workpiece temperature	Copper strip corrossion	Induction time
S FAME 10	4 Ball-scar diameter 4 Ball-weld point	Surface rouhgness Workpiece temperature	Copper strip corrossion	Induction time
S FAME+Min	x x	Surface rouhgness Workpiece temperature	Copper strip corrossion	Induction time
Min Commercial	4 Ball-scar diameter 4 Ball-weld point	Surface rouhgness Workpiece temperature	Copper strip corrossion	Induction time

3.2.1 *Extreme pressure (EP) performance test.*
The EP performance of the formulated broaching oil sample were assesed by the wear scar diameter and weld point obtained from 4-ball test method. EP performance describes the ability of broaching oil to protect surface by avoiding metal-to-metal contact between workpiece and working tool.

As shown in Figure 4, the EP performance of S FAME 6 resembled the Min Commercial with the average scar diameter value of 0,407 and 0,384 mm, respectively, while the scar diameter value of S FAME 10 was at 0,689 mm.

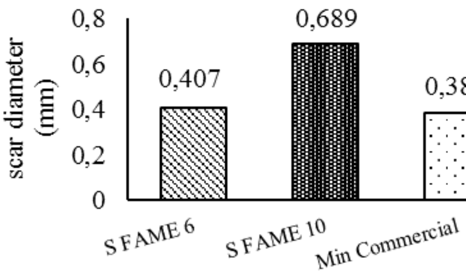


Figure 4. The comparison of wear scar diameter between the samples.

Broaching oil sample using S FAME 10 gave the highest wear scar diameter, while the lowest wear scar diameter is mineral base broaching oil of Min Commercial. This results agree with the experiment's result reported that higher sulphur content increased the load carrying capacity decreased the wear protection level of broaching oil [13]. The exessive sulphur content of broaching oil will accelerate both the wear scar rate and corrosion rate [14]. Another factor considered to have contribution to the rate of wear (scar diameter) is oxygen containing compounds in the broaching oil. Wear scar diameter reflects the oxidation rate during the examination. Oxygen containing compounds contribute the oxidation of the ball's surface. This process cause the surface to brittle. Thus the wear scar rate becomes higher. Mineral based broaching oil does not have O bonds on its structure. Therefore, the oxidation can be minimized [15].

The morphology of scar diameter from the samples is shown in Figure 5. It shows that the formation of scar are generally similar, but the diameter size are different. The wear scars are affected by the chemical reaction between the metal and sulphur or so called corrosive wear effect. The excessive sulphur content and the metal-to-metal friction will trigger the corrosion on the metal surfaces, leading to the brittleness of the suraface. The most common corrosive compounds that generally attack metals are hydrogen sulfide (H_2S), sulphur dioxide (SO_2), chlorine (Cl_2), nitrogen dioxide (NO_2), as well as oxygen (O_2) [16]. Among those elements, sulphur is the most corrosive compound. The result shows that the commercial broaching oil may use EP additives of non-sulphur compound, allowing the product to form the lowest wear scar diameter.

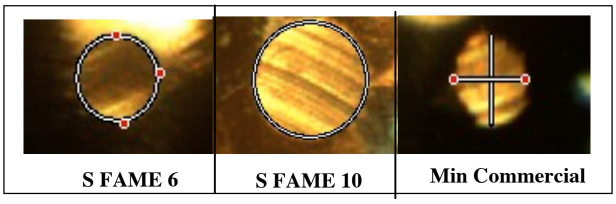


Figure 5. The comparison of wear scar diameter and morphology among the samples.

Load carrying capacity performance test was carried out by weld point examination. The higher the weld point, the better the load carrying capacity of the lubricants. Figure 6 shows the picture of weld phenomena of the lower balls of 4-ball tester after the weld test. Figure 7 depicts the result of weld point of broaching oil sample whereas the sample of S FAME 10 shows the highest weld point load of 800 kg_f. On the other hand, both sample of S FAME 6 and Min Commercial has the same value of 400 kg_f. The best load carrying capacity performance was obtained by broaching oil having highest sulphur content (10% wt sulphur), since the formation of salt-metal layer sulphur containing compound was thought to enhance the anti-weld properties and reduce the friction coefficient. The oil with a high load carrying capacity would be applicable for heavy duty metal cutting process.

Figure 6. Weld phenomena.

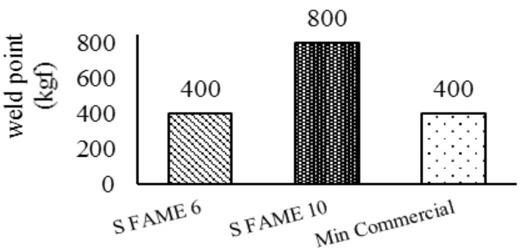


Figure 7. Weld point of the samples.

3.2.2 Surface finish of workpiece performance

The performance of surface finish can be observed from the surface roughness, the cooling level of lubricated workpiece, and the morphology of workpiece after cutting process. The surface roughness is an arithmetic average (R_a) of the peak height and the valley depth at the surface profile, commonly expressed in unit μm. Figure 8 shows the roughness of the workpieces after cutting process at certain cutting speed.

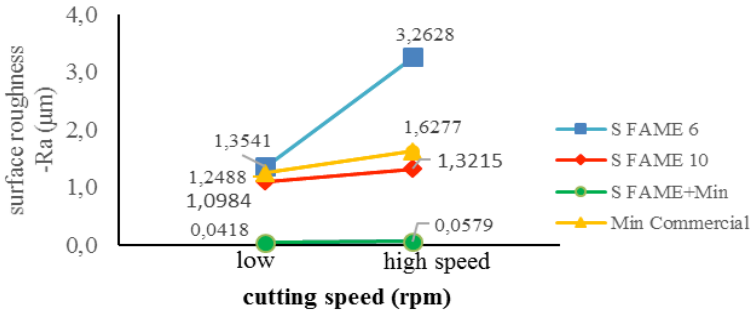


Figure 8. Surface roughness profile of workpiece compared to cutting speed level.

After heavy duty machining process, the surface roughness of workpiece increases as the cutting speed rises. In general, broaching operation at lower lower speed cutting speed would result in better surface finish quality. Figure 8 shows that performance of all samples were relatively the same on the lower cutting speed. However, at higher cutting speed, the sample wf S FAME 6 has the poorest performance. The finest surface roughness value was given by sample of S FAME+Min at both lower and higher cutting speed. These results demonstrated that cutting speed level has a significant influence on the heavy duty metal cutting process, where the lower cutting speed level will generate better surface roughness value than the higher cutting speed level. The surface finish is affected by lubricating properties and cooling performance of the cutting oil. The poor lubricating properties of cutting fluid will accelerate wear of the working tool, thus resulting in the low quality of product [4,17]. Cooling performance of oil is needed to lower temperature or remove heat generated when cutting at high speed. Figure 9 shows the workpiece final temperature during cutting process plotted versus cutting speed level.

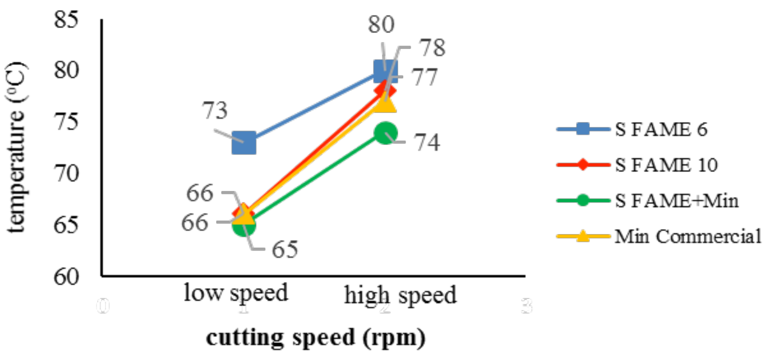
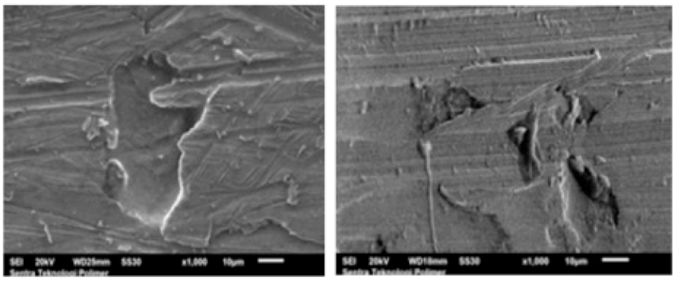


Figure 9. The final temperature profile of workpiece compared to the cutting speed level.

It is clearly seen from Figure 9 that the higher cutting speed level will increase the temperature of workpiece. Most of the sample show the final temperature are about the same in the range of 75-77 C^{o}, with the lower temperature given by the sample of S FAME+Min. It concluded that the surface finish performance of the formulated broaching oil resembles the commercial product.

Micro-structure observation of the surface of the cut workpiece by SEM is shown in Figure 10. Samples of S FAME 10 and Min Commercial shows relatively deep crater-like scratch lines. The crater-like scratch on the surface of S FAME 10 sample is a bit deeper than that of Min Commercial sample. This is might be due to the effect of corrosive wear stimulated by the excessive sulphur of the S FAME 10 sample.



a) b)

Figure 10. Morphology of the workpieces lubricated (1000x magnification)
(a) Formulated broaching oil (S FAME 10) and
(b) Commercial broaching oil (Min Commercial)

To evaluate the elemental composition of the workpiece cut surface, the EDX analysis was done. The results of EDX analysis are shown in Figure 11 and the summary is available in Table 3.

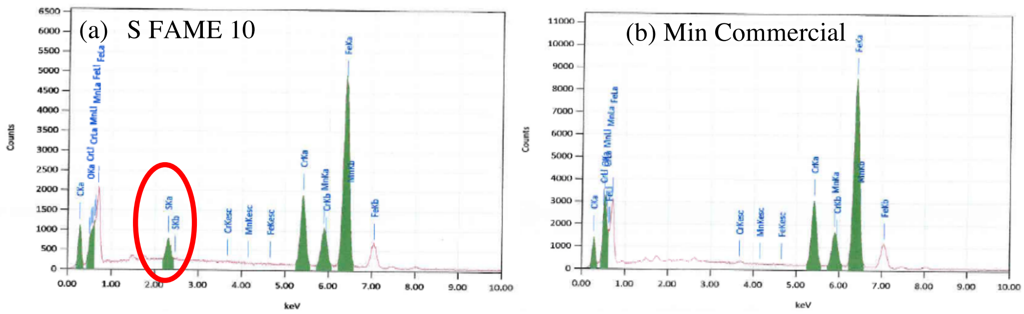


Figure 11. EDX spectra of the workpieces lubricated by (a) formulated broaching oil (S FAME 10) and (b) commercial broaching oil (Min Commercial).

Table 3. EDX results of cutted workpiece.

	Elemental composition (wt%)							
	C	O	Al	S	Cr	Mn	Fe	Mo
Formulated broaching oil (10% w/w S)	14.82 ± 5.08	2.02 ± 0.57	0.3 ± 0.53	0.53 ± 0.91	12.82 ± 0.83	7.22 ±0.53	60.13 ± 4.22	2.16 ±2.16
Commercial broaching oil	6.83 ± 4.37	6.17 ± 4.39	-	-	13.21 ± 1.31	7.34 ±1.06	66.46 ±5.87	-

As shown in Table 3, elemental sulphur is found on the cut workpiece surface lubricated by the S FAME 10 oil. On the other hand, it is not found in the surface lubricated in Min Commercial oil. This result confirmed that the sulphur containing compound in the formulated broaching oil adsorbed in the surface of workpiece and the adsorbed sulphur upgraded the surfcace's load carrying capacity performance whereas, Min Commercial oil contain non-sulphur additives.

3.2.3 Corrosion protection level performance

Elemental sulphur of sulphur EP additives will degraded and form sulfide metals on boundary lubrication condition, when the rise of local temperature is high [18]. Under boundary lubrication, the metal-to-metal contact between the working tool and the workpiece increase the temperature around the surface, leading to the degradation of sulphur containing compound and the formation of highly reactive metal sulfide. The sulphur will be highly active and then be able to stimulate metal corrosion, color alteration, and layer formation on the surface of metals, especially the yellow metals, such as copper and its alloys. Table 4 shows the corrosion test result of copper strip that lubricated by various broaching oils.

Table 4. Broaching oil corrosivity level.

Property	S FAME 6	S FAME 10	S FAME+Min	Min Commercial
Corrosivity level	1.b (dark orange)	4.a (dark grey)	1.b (dark orange)	1.a (light orange)

As shown in Table 4, S FAME 10 sample is the most corrosive broaching oil, as it caused the sample strip to darken into class 4.a, a corrosion category that is equal to dark grey-colored copper strip. On the other hand, the corrosivity of S FAME 6 and S FAME+Min are class 1, equal to dark orange-colored copper strip. The Min Commercial is in class 1.a, slight tarnish with the light orange-colored copper strip. Except the S FAME 10, the 3 samples meet the requirements of lubricant corrosivity level for general use at maximum class 2.a, moderate tarnish category with the red wine-colored copper strip [19].

The activity of sulphur depends on the sulphur chain in the molecules, where the activity of monosulfide and disulfide are moderate to soft metals than the pentasulfides [11]. Sulphurization of FAME would possibly produce the sulphur bridges structure (polysulfides) that will affect the corrosion protection level performance of yellow metals. The elemental sulphur will be more effective on the extreme pressure condition where the high corrosive metals do not exist. The higher content of sulphur EP additives in the lubricants will generate the highly active type of sulphur as same as the elemental sulphur [20]. The higher the ontent of sulphur in S FAME 10 originated from elemental sulphur will make it as one of the sulphur-active type lubricants. Therefore, the formulation of broaching oil that exceeds 6% wt is not recommended for yellow metal-contained cutting process as it will cause corrosion on the workpiece.

3.2.4 Oxidative stability performance.

Oxidative stability properties are essential to the product's storage and handling for it relates to the shelf life and the usage of broaching oil commercially. The results of oxidative stability performance test are shown in Figure 12.

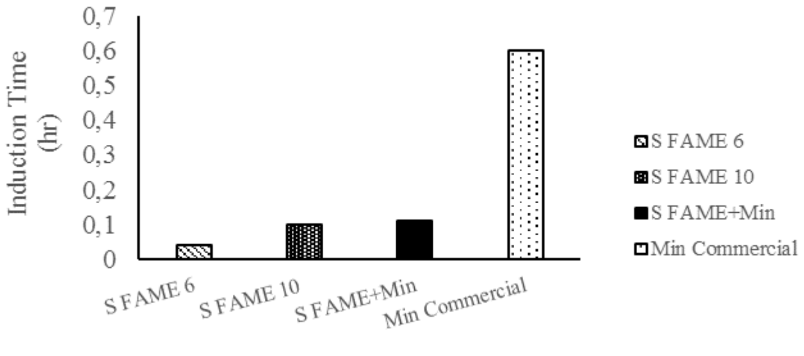


Figure 12. The oxidative stability.

It is clearly showed in Figure 12 that the oxidative stability of the formulated broaching oils are inferior to the Min Commercial broaching oil, with the their inducton time is still far shorter than that of the Min Commersial oil. These results indicate that the oxidative stability problem of the green oil based lubricants has not been overcome yet. This is might be due to the partial conversion of bisallylic protons on the sulphurized lubricant could not be carried out completely. This uncomplete convertion of C=C was confirmed by the result of iodine value that is still in the range of 25–30 g/100 g sample, even with the sulphur addition of 10% wt. Mineral oil based lubricants has more thermo-oxidative stability performance at elevated temperature compared to the green oil based lubricants, as it contains less unsaturated than the green base oil lubricants which contains unsaturated and polar group, such as C=C group, carbonyl group (C=O) and hydroxyl group (O-H) that are susceptible to oxidation.

From Figure 12, it is also interesting to notice that the variation of sulphur in the samples generates various oxidative stability, where the higher sulphur content of broaching oil S FAME shows better oxidative stability compared to the lower sulphur content of broaching oil S FAME 6. This might be due to the more conversion of C=C bonds to C-S bonds or S-S bonds, the higher the stability of the oil. However, excessive sulphur can also attack antioxidant agent in the lubricants [21]. Therefore, the optimum amount of sulphur that will not inhibit antioxidant performance should be passed over. The antioxidants have a good synergy with the sulphurized products that contain inactive type of sulphur. Meanwhile, a long chains of sulphur bridges in the polysulfides are thermally less stable than short sulphur bridges, where sulphur is linked to the carbon atom of the raw material [11]. From the test results, it might be possible that the polysulfide chains formed as a result of sulphurization reaction, and leading to the poor oxidative stability performance.

4. Conclusion

From the result obtained, it concluded that sulphurized FAME from palm oil is the best for broaching oil formulation with low viscosity and high EP performance or high content of sulphur. Addition of sulphur into palm oil or its derivatives by sulphurization produced sulphurized FAME containing more than 6%wt of sulphur. The higher the sulphur content the better the EP performance of the sulphurized products. Excessive sulphur content cause corrosion and britleness of metal surface and accelate wear rate. Broaching oil formulated by sulphurized FAME containing 6%wt sulphur and mineral oil EP additive showed the EP performance as good as the broaching oil of commercial product.

Acknowledgement

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