

## Physical Characterization of Modified Palm Oil with Hybrid Additives Nanofluids

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### ABSTRACT

The common use of petroleum-based metalworking fluids (MWFs) in industry harms the environment and human beings. The biggest disadvantage of RBD palm oil is its low thermal-oxidative stability. As a result, the goal of this research is to create an innovation for palm oil based MWFs. The new creation of modified palm oils (MPOs) has been produced by a transesterification process with palm methyl ester to trimethylolpropane molar ratios of 3.5:1. Later, at a concentration of 0.025wt.%, the MPOs were mixed with single and hybrid additives such as activated carbon (AC) (MPOa), tungsten disulphide (WS<sub>2</sub>) (MPOw) and hybrid AC with WS<sub>2</sub> (MPOaw). The physicochemical parameters of MPOs were studied, including viscosity, and acid value. The physicochemical properties were evaluated as per ASTM standards over a period of 4 weeks to check the state of lubricant and observable changes in the properties during this period. According to the findings, MPOaw shows enhanced thermal (high viscosity index 365-387) and oxidative stability (lubricant storage). Moreover, MPOaw recorded has higher antioxidant properties that can help prevent or slow down the oxidation process, reducing the production of acids and subsequently lowering the acid value (0.14 -0.19 mg NaOH/g). In conclusion, it has been proven that MPOaw has the best performance and the potential to have a positive effect on the industry as a sustainable MWF for machining processes.

## 1. Introduction

Metalworking fluids (MWFs), referred to as lubricants, coolants, or cutting fluids, are extensively applied in many manufacturing and industrial operations. (MWFs) possess significant importance in the reduction of friction and wear between tool and workpiece components that are in proximity and exhibit relative motion. This is achieved by establishing a protective layer between the surfaces,

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hence facilitating load distribution and alleviating pressure created at the interface [1]. The growing worry over environmental and health-related matters has prompted the industrial sector to prioritize the development of biodegradable materials and eco-friendly goods. The depletion of fossil fuel supplies, namely petroleum, has garnered significant interest in research, prompting a move towards renewable and biodegradable sources. Around 85% of MWFs utilized in industrial settings are sourced from petroleum-based oil. This preference is mostly attributed to their commendable lubricating properties, notable stability, and prolonged durability [2].

This awareness helps direct focus towards the search for alternative environmentally friendly, biodegradable, and non-toxic oils that may be used as substitutes for mineral oil in metalworking fluids. Consequently, vegetable-derived oils are currently seen as more feasible options for use as lubricants and metalworking fluids (MWFs) in industrial applications. Numerous research endeavors are currently underway to develop novel cutting fluids derived from a diverse range of readily available vegetable oils from globally [3]. In the present study, palm oil was selected as a primary constituent for the formulation of metalworking fluid. The selection of palm oil as a new renewable energy source was made to facilitate the implementation of eco-friendly machining practices. The potential for Malaysia to create palm oil-based cutting fluid is highly promising, given the country's status as one of the top producers of palm oil and its convenient access to all essential raw materials [4]. Rahim *et al.*, [5] study had noted that palm oil serves as a primary supply of oil in Southeast Asia, alongside petroleum. Therefore, it is crucial to consider the possibility of using palm oil as a functioning lubricant in place of petroleum-based oil in the future.

In order to address the inherent limitations of vegetable oil, such as its susceptibility to oxidation, high friction, elevated viscosity, limited thermal stability, and vulnerability to corrosion, it is imperative to undertake appropriate modifications to the oil prior to its utilisation [6]. Based on the research conducted by Masood *et al.*, [7], it can be deduced that the optimal strategy for improving the performance characteristics of vegetable oils is achieved through the utilisation of chemical modification techniques. Hence, it is essential to improve the characteristics of the oil through modification. A variety of research investigations have been undertaken to investigate the modification of vegetable oil. Jeevan and Jayaram [8] conducted a chemical modification of jatropha and pongamia oil by the technique of epoxidation. The changed oils were subsequently evaluated in terms of their physicochemical qualities and machining capabilities and compared to Mineral Oil. The authors noted that the physicochemical characteristics of modified oils were like those of mineral oil. In their study, Rahim *et al.*, [5] examined several formulations of Modified RBD Palm Olein as a metalworking fluid. Experiments were conducted to investigate the effects of varying ratios of methanol to RBD Palm olein, specifically at ratios of 3:1, 6:1, and 9:1. The researchers found that the formulation with a ratio of 6:1 exhibits exceptional tribological properties, suggesting its potential as a viable substitute for metalworking fluid.

Additionally, adding nanoparticles to MWFs has the potential to significantly enhance their performance. Tungsten disulphide ( $WS_2$ ) has garnered recognition from the scientific community as one of the most superior materials for mitigating friction and facilitating lubrication. It exhibits suitability not only for typical lubrication conditions but also for challenging operational settings, including those characterized by high temperature, high pressure, high vacuum, high load, radiation, and corrosive mediums. Therefore, this material is widely utilized in the domains of aviation, aerospace, and other industries characterized by advanced technology. The microstructure of  $WS_2$  crystals exhibits a hexagonal layered hollow sphere configuration, whereby the surface of the sphere is composed of a network layer consisting of six squares formed by S-W-S molecules. The layers are interconnected by the van der Waals force, a weak binding force that may be readily disrupted between the layers. Additionally, the slide is likely to happen between layers, resulting in a low

friction coefficient. Hence, throughout the lubrication process, the  $WS_2$  particle exhibits a high level of stability and quality at the microscopic scale. Moreover, the  $WS_2$  at the nano-scale exhibits attributes such as elevated density, reduced dimensions, substantial specific surface area, and heightened specific surface energy [9]. Srinivas *et al.*, [10] mentioned that the physicochemical properties of motorbike lubricant dispersed with  $WS_2$  nanoparticle show improvement compared to only lubricant itself. In addition, activated carbon (AC) is a type of carbonaceous material that has undergone treatment. It possesses a consistent surface shape, notable biocompatibility, favorable mechanical strength, and stability [2,11]. According to the preceding investigation conducted by Kaggwa *et al.*, [12], it was observed that the incorporation of activated carbon in nanofluid resulted in a notable augmentation of lubricant viscosity and enhancement of heat transfer efficiency. According to Khoo and Aziz [13], the use of activated carbon (AC) as an addition for lubricants exhibits significant enhancement in the base oil through the amplification of its adsorptive properties. According to Oginni *et al.*, [14], AC possesses a porous structure and surface chemical properties that facilitate its ability to enhance both non-polar and polar adhesion. The efficacy of activated carbon (AC) is contingent upon the surface chemistry of carbon, as highlighted by Reinoso and Alberio [15]. The surface chemistry of carbon has a substantial impact on the adsorptive properties of AC [16].

The investigation of the combined performance of nanoparticles as an additive to lubricants is justified due to the distinct tribological features shown by each nanoparticle. The combination of two separate nanoparticles inside a heat transfer fluid result in the formation of a hybrid nanofluid, which represents an innovative fluid utilised in the field of nanotechnology. The boundary layer flow generated by hybrid nanofluids has garnered significant interest from academics in recent times. The boundary layer refers to the region in which viscosity plays a prominent role, resulting in the formation of the majority of drag experienced by an object immersed in a fluid [17]. In their study, Sidik *et al.*, [18] conducted a comprehensive review on several techniques employed for the creation of hybrid nanofluids, as well as an analysis of their thermal performance. The participants engaged in a discourse on the procedural aspects of creating hybrid nanofluids, as well as the many elements that exert an influence on their performance. The researchers reached the conclusion that hybrid nanofluids exhibit favourable thermal characteristics as a result of the synergistic interaction between two distinct nanofluids. Additionally, it was suggested that additional empirical investigation is required in order to have a comprehensive understanding of the distinctive characteristics of the fluid. The tribological performance of individual nanoparticles and  $SiO_2/MoS_2$  hybrid nanoparticles dispersed in deionized water was investigated by Meng *et al.*, [19] in their study. The samples were subjected to a grinding process lasting 30 minutes in order to assess the extent of change in the friction coefficient. The researchers made the observation that the incorporation of a hybrid lubrication layer, improved adsorption capabilities, and the synergistic interplay among individual nanoparticles resulted in hybrid nanofluids exhibiting exceptional frictional properties. According to the findings of this study, the integration of hybrid nanoparticles into a lubricant base demonstrates a higher level of effectiveness compared to the use of single nanoparticle additives. The inclusion of many nano-additives can frequently be considered the optimal approach due to the potential compensation of deficiencies in one additive by another, as well as the observed synergistic interactions among different types of nanomaterials, resulting in enhanced friction performance. In early 2023, Sabri *et al.*, [20] study on the physicochemical properties of hybrid nano additives added to modified Jatropha oil. They found that MJO with hybrid nano additives improve the viscosity index by 24 %-27%.

Hence, the objective of this study is to assess the physical characteristics of RBD palm oil that has been modified for use as a metalworking fluid in machining operations. The study focuses on the incorporation of single and hybrid nanoparticles, specifically activated carbon (AC), tungsten

disulphide ( $WS_2$ ) and a combination of AC and  $WS_2$ , as additives in the oil. The physical characteristics were evaluated in accordance with the standard set by the American Society for Testing and Materials (ASTM). In this project, testing was conducted to get the requisite physical parameters of viscosity (as per ASTM D445), viscosity index (as per ASTM D4502), and acid value (as per ASTM D664).

## 2. Methodology

### 2.1 Bio-Based Nanofluids Preparation

In this study, transesterification was used to chemically modify the vegetable-based nanofluids created from palm oil. Refined, bleached, and deodorized palm olein (RBD PO) undergoing the transesterification process with methanol ( $CH_4O$ ) in the existence of 1% (wt/wt) sodium hydroxide (NaOH) as a catalyst to create RBD Palm Oil Methyl Ester (POME). Before pouring the solution into the three-neck round bottom flask containing the heated oil, NaOH was dissolved in the methanol ( $CH_4O$ ). In the water bath, the reaction temperature was kept around  $60^\circ C$  for 2 hours. POME undergo transesterification process with Trimethylolpropane (TMP) with 1% (wt/wt) sodium methoxide ( $NaOCH_3$ ) as a catalyst resulted in the production of modified palm oil (MPO). The reaction was carried out in an oil bath condition at  $120^\circ C$  in a 1000 ml three-neck round bottom flask equipped with a thermometer, sealed with a graham condenser, and sampling port as shown in Figure 1. The condenser is linked to the compressor, which includes a relief valve, vacuum trap, and accumulator.



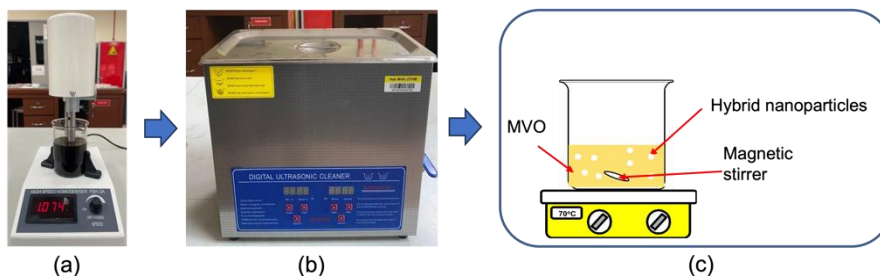
Fig. 1. Set-up of the transesterification process

After that, the MPO obtained was blended with single and hybrid additives. Activated carbon (AC) and tungsten disulphide ( $WS_2$ ) was used as additives. MPO was blended with 0.025 wt.% single additives AC,  $WS_2$  and hybrid additives of AC and  $WS_2$  respectively. All the samples were shown and described in Table 1. As shown in Figure 2, the samples were blended first by using the homogenizer at 10,000 rpm for 30 minutes. Next, the samples were continued blended by using Bandelin HD3200 model ultrasonic homogenizer for another 30 min (at 20 kHz frequency and 200 W). Then, MPO will be completely blended with the additives for 30 minutes at 700 rpm and  $60^\circ C$  using a magnetic stirrer. In the final step, any precipitation or layer separation in the combinations was observed

visually. The performance of the blended MPO mixture will be evaluated using physiochemical testing. The properties of AC and WS<sub>2</sub> are shown in Table 2.

**Table 1**  
 Sample of MPO with nanoparticles

Sample	Description	Weight of nanoparticles (wt.%)
MPO	MPO	-
MPOa	MPO + AC	
MPOw	MPO + WS <sub>2</sub>	0.025
MPOaw	MPO +( AC + WS <sub>2</sub> )	



**Fig. 2.** Mixing Process of MRPO with nanoparticles; (a) Homogenizer (b) Ultrasonic vibration (c) Stir process

**Table 2**  
 Physical properties of activated carbon and tungsten disulphide

Properties	Activated carbon (AC)	Tungsten disulphide (WS <sub>2</sub> )
Appearance	Black powder	Blue-grey powder
Density (g/cm <sup>3</sup> )	3.3	7.5
Melting Point (°C)	1772	1250
Solubility	Insoluble in water	Slightly soluble
Size (nm)	<100	<100
Thermal expansion coefficient (10 <sup>-6</sup> /K)	3.6	10

## 2.2 Physical Testing

### 2.2.1 Kinematic viscosity and viscosity index (VI)

The kinematic viscosity of the samples was determined in accordance with the ASTM D445 standard using a portable viscometer known as the Viscolite 700. The examination was conducted at the Fuel Analysis Laboratory, Faculty of Mechanical and Manufacture Engineering (FKMP), Universiti Tun Hussein Onn Malaysia (UTHM). Viscosity refers to the measure of a fluid's internal resistance to flow, specifically in the context of oil. The selection of an appropriate lubricant is crucial in ensuring smooth and efficient flow and distribution during the machining process. The experimentation involved the utilisation of oil, which was subjected to a range of temperatures spanning from 30°C to 100°C. A 10 mL sample of lubricant was poured into a beaker. When the lubricant sample covered the grooved spindle on the shaft, the kinematic viscosity value was noted. The determination of the VI was performed using the calculation method outlined in the ASTM D2270 standard. The samples were subjected to a comparative analysis with the benchmark Synthetic Ester (SE).

### 2.2.2 Acid value

As per ASTM D664, the test was conducted using the titration process to obtain the acid value. The solution used for titration was 0.1 N NaOH. 4g of the sample was added to 2 g of 2-propanol in a conical flask and heated to 60 °C. Five drops of phenolphthalein were added to the mixture as an indicator. The titration procedure was begun and until the sample solution turned pink and lasted 30 seconds. To determine the acid value, the volume of 0.1 N NaOH titrated was measured, noted, and calculated. The procedure was repeated three times, with the average from each run being recorded.

## 3. Results

### 3.1 Kinematic Viscosity and Viscosity Index (VI)

Figure 3 shows the data for kinematic viscosity of all samples from temperature 30°C until 100°C. From the graph, the kinematic viscosity of all samples decreased with increased in temperature. This trend shows that temperature plays an important role in kinematic viscosity. According to the findings of Sabri *et al.*, [21], it was observed that an increase in temperature led to an accelerated dispersion of molecules in the lubricant samples. Furthermore, there are significant cohesive forces between the molecules of a liquid, which are considerably closer together compared to those of a gas. The cohesion of the liquid decreases as the temperature increases, resulting in a reduction in viscosity [22]. This finding was also supported by the study from Sukkar *et al.*, [23] as they mentioned that the viscosity of the nano lubricant decreased with increasing of the temperature. From Figure 3, it can be seen that the kinematic viscosity of SE was the highest from temperature 30°C until 80°C compared to MPOs. However, at a temperature 100°C, the kinematic viscosity of SE was seen to be the lowest, measuring 5 mm<sup>2</sup>/s. This observation demonstrates a significant decrease in the kinematic viscosity of SE as the temperature decreases, which is associated with a low VI. The experimental results indicate that the differences in kinematic viscosity of nanofluids are comparatively less at elevated temperatures ranging from 80°C to 100°C, in contrast to lower temperatures. This is because at high temperatures, molecular forces weaken and molecular bonds are more easily broken [24]. The presence of unreacted methyl ester in MPOs contributed to their low viscosity when compared to SE [6]. High viscosity of SE can be attributed to the presence of various additives, such as viscosity improvers, anti-wear agents, and corrosion inhibitors, which have been incorporated into the lubricant [25]. In addition, it has been shown that the incorporation of single and hybrid nanoparticles into MPO leads to an increase in kinematic viscosity when compared to MPO without the inclusion of nanoparticles. This phenomenon arises due to the presence of nanoparticles, which leads to the formation of larger nano-clusters that impede the flow of fluid layers in a stacked configuration. As a result, the intermolecular forces experience an increase, leading to a closer proximity of the layers and thus elevating the viscosity [24]. Among MPO with nanoparticles additives, MPO with hybrid additives, MPOaw shows the higher kinematic viscosity from 30°C to 100°C.

Figure 4 illustrates the VI of each sample for 4 weeks storage, which was established by calculating the kinematic viscosity values at temperatures of 40 °C and 100 °C. The VI of a lubricating fluid offers valuable information on the degree to which the fluid's viscosity was affected by variations in temperature. A high VI signifies that the fluid's viscosity exhibits a generally consistent behaviour in response to temperature fluctuations, whereas a low VI suggests a significant change in viscosity. In Figure 4, it can be observed that the MPO had a larger value of VI in comparison to the SE. During the 4-week storage period, there was a significant improvement in the VI, with an increase ranging from 34.6% to 58.5% compared to SE. The formation of a lengthy molecular chain and the increased mass

of the TMP ester are factors that lead to a greater VI. In week 1, the VI of MPO with nanoparticles additives were higher than MPO itself. The VI of MPOa was 358, MPOw was 361, and MPOaw was 365. This is due to the addition of nanoparticles additives that have low thermal expansion coefficient that affected the VI of each sample. The trend of VI result for all samples were same throughout 4 weeks storage period. Among MPOs with nanoparticles additives, MPOaw, MPO with hybrid nanoparticles shows higher VI and improved the VI of MPOa and MPOw by 2% and 1.1% respectively. The synergetic effect of AC and WS<sub>2</sub> with low thermal expansion coefficient  $3.6 \times 10^{-6}/K$  and  $10 \times 10^{-6}/K$  respectively affect the overall thermal expansion coefficient of MPO thus resulting in higher VI of the sample. Nanoparticles with a low thermal expansion coefficient offer enhanced thermal stability to oil and contribute to the maintenance of a larger thermal network [26]. This result shows that the addition of hybrid nanoparticles additives improved the VI of MPO compared to MPO with single nanoparticle additives. The use of a hybrid AC (activated carbon) in conjunction with WS<sub>2</sub> (tungsten disulfide) showcases the enhancement of properties in vegetable-based oil through the incorporation of two separate additives. In a study conducted by Moghaddam and Motahari [27], it was shown that the viscosity of a base oil containing two additives was higher compared to the viscosity of the base oil without any additives.

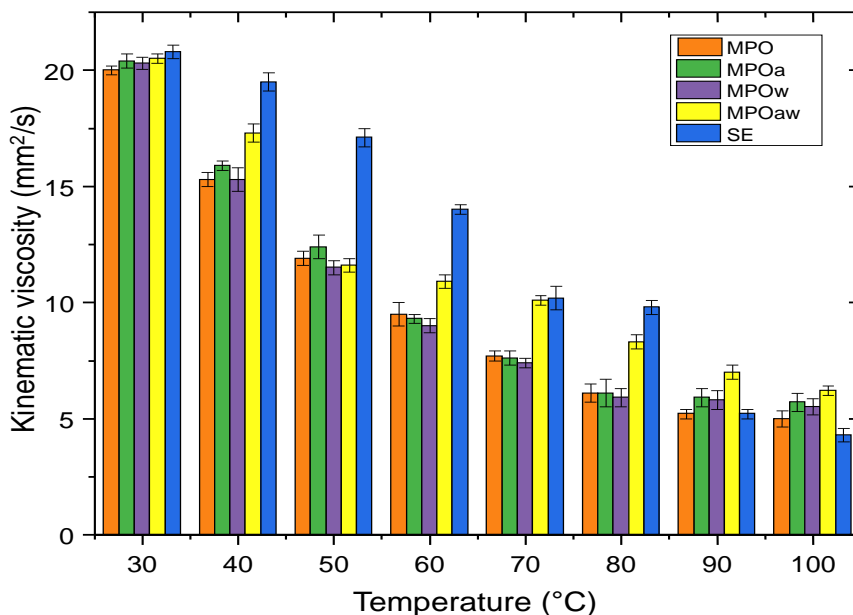


Fig. 3. The kinematic viscosity data from 30 °C to 100 °C

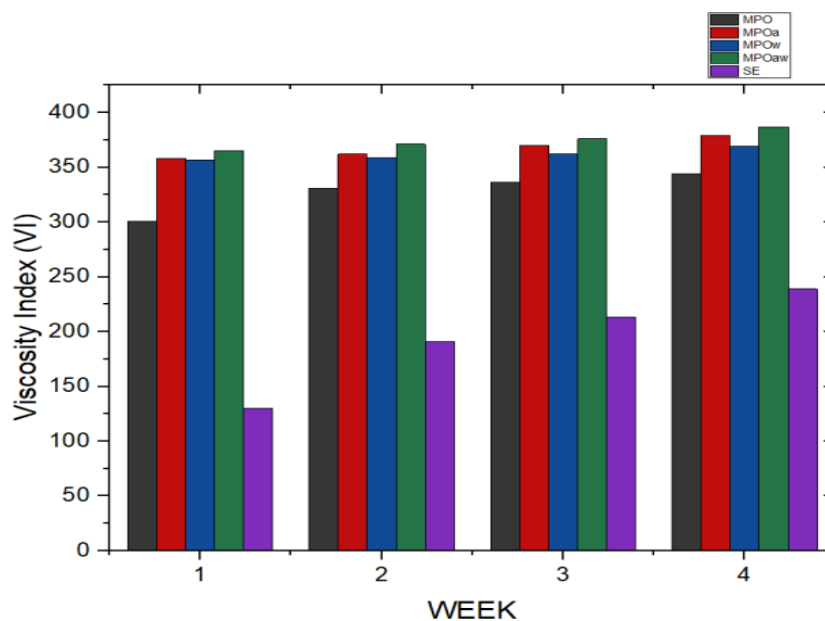


Fig. 4. Viscosity index (VI) of samples for 4-weeks storage

### 3.2 Acid Value

The acid value is a measure of the concentration of free fatty acids (FFAs) in the oil, which plays a significant role in determining the quality of the lubricant feedstock. Additionally, it indicates the quantity of potassium hydroxide (KOH) or sodium hydroxide (NaOH) in milligrams required to achieve neutralisation of the fatty acids present in a 1g sample of oil. Moreover, it was predicted that the acid value of the machining lubricant would be below 0.5 mg NaOH/g. Figure 5 shows the acid value of all samples for 4-weeks storage. The acid value for SE was the highest from week 1 to week 4 at 0.55mg NaOH/g to 0.59mg NaOH/g compared to MPOs samples. The observed phenomenon might be attributed to the presence of a double bond in the fatty acid chain, which leads to an increase in rigidity. Consequently, this hinders the close arrangement of fatty acid chains [21]. The incorporation of nanoparticles into MPOs leads to enhanced acid value in comparison to the MPO. The MPOaw, which incorporates hybrid nanoparticle additions, has the lowest acid value of 0.14 mg NaOH/g. The lower acid value contributes to higher oil quality due to its ability to prevent corrosion as well as the formation of gum and sludge [28]. Following this, the MPOw, which contains single nanoparticle additives, demonstrates an acid value of 0.15 mg NaOH/g, while the MPOa exhibits a higher acid value of 0.2 mg NaOH/g. Based on the observed results, it can be inferred that the introduction of nanoparticles has had a notable impact on the acid value of MPO. Specifically, the acid value of MPOaw, MPOw, and MPOa exhibited reductions of 39.1%, 34.8% and 13%, respectively. The decrease in acid value suggests that modified oils will have a longer shelf life [29].

Furthermore, it was observed that the acid value exhibited a significant increase for all samples throughout the course of a 4-week storage period. This phenomenon can be attributed to the inherent hydrolysis reaction of the fatty acid. According to Bruun *et al.*, [30], the augmentation in acidity is dependent on the processes of hydrolysis and oxidation. Therefore, over a duration of 4 weeks, the highest acid value of 0.58 mg NaOH/g was reported by SE. Based on the findings from week 4, it was seen that MPOaw exhibited lowest acid value measurements in comparison to MPOs at 0.19 mg NaOH/g. Nevertheless, all the recorded results for MPOs were lowered than the recommended acid value threshold for machining, which is 0.5 mg NaOH/g.



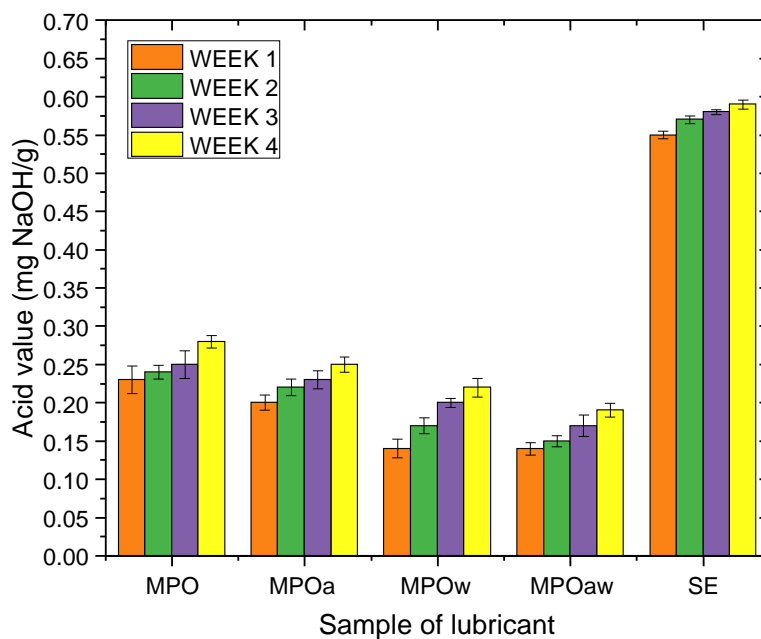


Fig. 5. Acid value data for all samples for 4-weeks storage

#### 4. Conclusions

The VI of MPOaw was higher compared with other samples. The expansion and uncoiling of the polymer chains of hybrid additives help to maintain the lubricant's viscosity at higher temperatures, thus improving its VI. Moreover, MPOaw has the lowest acid value compared with other MPOs samples, due to the combination of hybrid additives can increase the lubricant's alkalinity and buffer its pH, minimizing acid-induced degradation and may help it to minimize the increase of free fatty acid in the samples for a longer period. Due to the addition of hybrid additives in the lubricants may enhance the barrier properties of coatings, reducing the permeability of corrosive agents, such as moisture, oxygen, or chemicals, to the copper surface. Lastly, it has been proven that MPOaw has the best performance as the MWFs compared to other samples.

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