

Experimental study on effect of nano Al₂O₃ in physiochemical and tribological properties of vegetable oil sourced biolubricant blends

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In this study, nine different vegetable oil-based blends were prepared from POME, NOME and TOME by blending with SAE20W40 commercial grade engine oil and 1%, 2% Al₂O₃ nano particles in weight percentage. The physiochemical properties viz. density, viscosity, viscosity index and flash point were investigated and tribological properties were investigated by four ball lubricant test under ASTM D4172B standard. The results showcased better outcomes with higher percentage (2%) of nanoparticle addition. Especially, the N102 and P202 blends has shown reduction in friction (13.3% and 12.8% respectively) and reduced wear scar diameter (55.1% and 50.5% respectively) when compared with SAE20W40 commercial lubricant.

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1. Introduction

Fossil fuels are primarily used as energy sources, substantially in today's manufacturing and automotive market scenario. Lubricants, which are dependent on crude oil supplies, are widely used to control the wear and tear damages in equipment. The low biodegradability of crude oil-based lubricants poses a dangerous anthropogenic climatic change, by contaminating the natural resources [1,2]. The toxicity, inadequate disposal, and the rapid degradation, call for the adoption of alternative methods and materials for the same purpose [3]. On the other hand, due to urbanization, infrastructural growth and deforestation, the plant-based resources which has enormous potent to solve these issues have not gained importance. To cope up with this snag, there is an effort to use vegetable oil-based lubricant along with mineral oil lubricant, which can result in decreased usage and can be a better alternative to it. That's not something new to our culture, though. The plant oils and animal fats had been used in ancient times to minimize friction and wear [4]. Also, studies and research in this sector, which is gaining rapid importance, may also take place.

In the same context, vegetable oil-based lubricants are gaining their importance in the industrial practice. They have some unique merits viz. high Viscosity index, high flash and fire points, bio degradability and each to renew [5]. Ergo they have some shortcomings such as poor oxidation and thermal stability and poor cold flow properties. To overcome such issues, chemical treatments like Transesterification [6], Epoxidation [7] can be done. The tribological properties of many vegetable sourced oils viz neem [8], Pongamia [9], Rice bran [9], Palm [9] tamanu [10], jatropha [11], sesame [12] and coconut [13] oils were investigated by various researchers. These treated oils can be used as bio additives with base stocks in order to reduce usage of mineral oil-based lubricants.

Hence, the environmentally friendly lubricants play an important role in today's crude oil crisis. To improve their lubrication characteristics on par or even better than its commercial counterparts, compatible nano particles can be added. It helps to improve service life and quality

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of lubrication thereby results in reduced energy consumption. This is highly possible because of good anti wear and heat dissipation properties of nano particles [14]. Especially in enhancing the tribological properties, the nano particles are used as extreme pressure additives [15] and anti-wear additives [16]. The quantity and size of the nanoparticles are important because the increasing volume may end up in poor tribological behavior. The spherical shape of the nano particles plays a vital role in reducing the friction and wear, since it fills the spaces between the rough asperities of the surface [17].

There are different nano particle additives which are compatible with lubricants. Researches are being carried out in nano particles viz. Al_2O_3 [17-20], SiO_2 [17,18], CuO [21,22], TiO_2 [23] graphene [24] and other nanoparticles [25] around the globe.

Mohammed Kamal Ahmed Ali et.al [17] had done a research based on Al_2O_3 , TiO_2 and hybrid particles dispersion of the two in SAE5W30 oil, under sliding contact friction and wear analysis. They concluded that the dispersion of nano particles in commercial lubricant helped in reduction of fuel consumption. Similarly, Ankit Kotia et.al [18] observed the mechanism of friction and wear in gear oils with Al_2O_3 and SiO_2 nano particles. They found that comparing with SiO_2 nano lubricant Al_2O_3 nano lubricant performed well in reduction of friction and wear of SS 304 material pin which showcases the good anti wear property of Al_2O_3 nano particle.

Mark A. Kedzierski [20] investigated the viscosity and density modifications in pololester based lubricant with various combinations of Al_2O_3 dispersion. Based on his research, he had created a model for correlating the various parameters viz. nanoparticle mass fraction, its diameter, viscosity, temperature and surfactant mass fraction. He concluded that, viscosity of nanolubricant changes based on temperature and surfactant mass fraction. Ehsan-o-llah Etefaghi et.al [21] researched on dispersion of CuO particles in SAE20W50 engine oil to find the effective dispersion percentage. They found that 0.2% wt of CuO in oil was found to be the best sample because it showcased the best flash and fire point values without much change in its viscosity. Ankit Kotia et.al [22] had discovered that viscosity, thermal conductivity and particle volume fraction have a correlation with CuO nanoparticles and SAE 68 hydraulic oil.

Rajeev Nayan Gupta and Harsha [26] studied the behavior of CCTO and ZDDP nanoparticles in castor oil lubricant in four ball wear tester and found that 0.25% and 1% addition for both additives improved the friction and wear resistance. Ke Li, et.al [27] found that the addition of 2% cellulose nanocrystals in polyalphaolefin biolubricant has reduced the COF by 30%. Similarly, a reduction in friction coefficient (15%) and wear scar diameter (11%) were achieved by Zulkifli .et.al [23] with addition of TiO_2 nanoparticles with Palm oil based TMP ester. SS Nair et.al. [28] inferred that addition of rod shaped ZnO nanoparticle in Sesame oil biolubricant reduced the frictional coefficient by 24.04% and wear scar diameter by 13.74%.

The addition of nanoparticles would be quite beneficial in terms of lubricity in vegetable oil-based methyl ester blends. The presence of Al_2O_3 nanoparticles in two distinct proportions has a significant influence on the blends. This study focusses on the influence of nano Al_2O_3 particles in the physiochemical and tribological properties of nine different biolubricant blends for the first time.

2. Materials and Methods

2.1. Nano - biolubricant preparation

The three biolubricant blend samples were shortlisted by prior tests. To prepare the oil samples, the biolubricant blends of Neem oil Methyl ester, Pongamia oil Methyl ester and Tamanu oil methyl esters were obtained by transesterification of Neem, Pongamia and Tamanu oils. After the preparation of biolubricant samples, Al_2O_3 nanoparticle was added in two proportions of 1% wt and 2% wt of biolubricant samples and stirred thoroughly. Morphology and size distribution of Al_2O_3 nanoparticles were done with SEM imaging as shown in fig 1(a,b). The six samples of Nano – biolubricant were prepared as shown in Table 1. Along with those samples, SAE20W40 oil was also chosen as a reference sample to compare the performance of nano – biolubricants.

Ducom Instruments made the four-ball lubricant tester used in this evaluation, which is shown in figure 2(a) and 2(b). The upper ball is held in place by a steel collet, while the other three

balls are held securely in a cup that holds the lubricant. The balls are made of AISI 52100 Chrome – alloy steel with a diameter of 12.7 mm. Its hardness ranges from 64 to 66 HRC. The tribotester is connected to a computer and a data collection device. The real-time testing data is recorded using Ducom's Tribodata software. A force sensor, a load cell, and a speed sensor are used in the device to provide information to the data acquisition system. The tribotester can rotate at speeds of up to 3000 rpm and can handle loads of up to 10000 N. The test standards ASTM D2783, IP239, DIN51350 – 02, ASTM D4172, ASTM D5183, DIN51350 – 03, can be carried out, with ASTM D4172 B being the preferred research standard for this evaluation. The test parameters are listed in the table below (2).

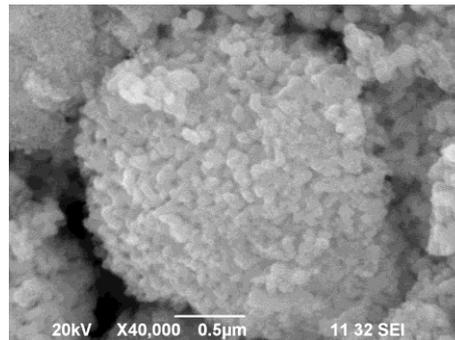


Fig. 1. (a) Nano Particles (Al_2O_3) 40kX times Magnification.

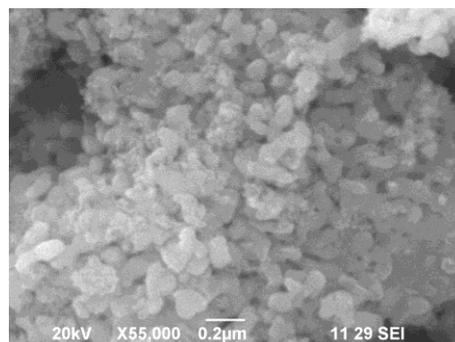


Fig.1. (b) Nano Particles (Al_2O_3) 55kX times Magnification.

Table 1. Composition of Nano – biolubricant samples.

S.No	Sample	Vol. of Base oil for 1 litre (in ml) (SAE20W40)	Biolubricant additive	Volume of Biolubricant additive (ml)	Nanoparticle % weight
1	N101	900	NOME	100	1
2	N102	900	NOME	100	2
3	P201	800	POME	200	1
4	P202	800	POME	200	2
5	T301	700	TOME	300	1
6	T302	700	TOME	300	2
7	SAE20W40	1000	-	-	-

Table 2. Wear test parameters (ASTM 4172B).

Test Parameters	Load (N)	Speed (RPM)	Duration (Mins)	Temperature (oC)
Range / values	40	1200	60	75



Fig. 2. (a). Four ball tester
(b) Four ball tester sample desk.

3. Results and Discussion

3.1. Physiochemical properties

The physiochemical properties of nano – biolubricant samples were tested for various physiochemical properties such as density, viscosity, viscosity index, Flash point and fire point. The results are compared below.

3.1.1. Density

Since any equipment has to pump the lubricant to prevent wear and tear in moving components, the density of a biolubricant is critical. Temperature has an impact on the density of oil. And, over time, the wear debris that contaminates oil reduces the density of the oil, which affects the lubricant's efficiency [29,31,32]. The density values observed by ASTM D792 specifications are lower than SAE20W40 oil in this evaluation, which is found to be beneficial in reduction of friction and wear at elevated temperatures. This is due to the added mass density of Nano Al_2O_3 particles which contributes to the increased values of Vegetable oils-based samples than SAE20W40. Especially N102 and T302 samples were found to have about 13% more than the mineral oil lubricant.

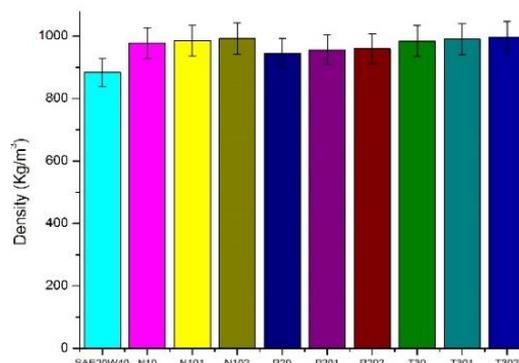


Fig. 3.1. Density of oil samples.

3.1.2. Viscosity

The resistance of a fluid to shear load is defined as its viscosity. It is an essential property of any liquid, particularly lubricants, that determines its suitability for use against friction and wear [29 - 32]. The viscosity is measured at 40°C and 100°C using the ASTM D455 standard. At 40°C, viscosity of all the Nano - biolubricant samples are marginally increase as the concentration of nano Al_2O_3 particles increase. The same behavior was found by Mahara et.al [34]. Ergo, at 100°C, nearly all oils are on par with viscosity of SAE20W40 oil.

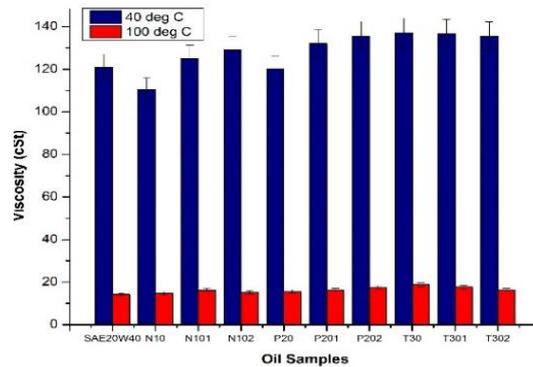


Fig. 3.2. Viscosity of oil samples (at 40°C and 100°C).

3.1.3. Viscosity Index

The viscosity index is a vital measure of lubricating liquids that predicts the change in viscosity as a function of temperature. Since any friction generates heat, and lubricants are designed to reduce friction and wear, this is a very significant and desirable property of lubricants. As a result, it should be able to maintain its lubricity at higher temperatures. The obtained viscosity index values of Nano-biolubricants blends are higher than SAE20W40 oil, which is an important property of vegetable oils, according to literature [30 - 32]. T301 and P201 oils, in particular, have higher viscosity index that is 30% higher than normal SAE20W40 oil.

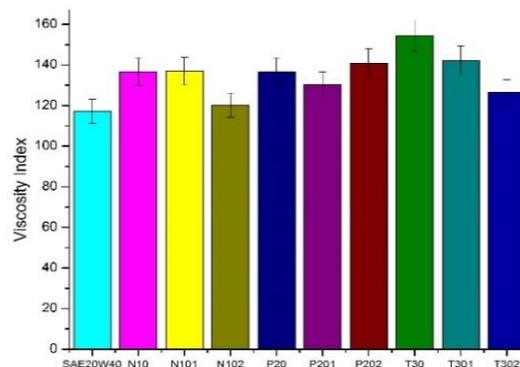


Fig. 3.3. Viscosity Index of oil samples.

3.1.4. Flash point

The flash point of a lubricant refers to its ability to withstand heat before becoming an ignitable mixture. It is carried out using a Cleveland open cup tester in accordance with ASTM D92 [30 - 32]. It is observed that, the addition of Al₂O₃ nanoparticles has improved the flash point values of N10, P20 and T30 oils which are highly desirable because it is a good indication of fitness to lubrication. The superior thermal properties of Al₂O₃ which attributed to the improvisation of flash point characteristics of vegetable oil methyl ester-based blends. Among all the blends, T302 showcased a flash point value which is on par with commercial SAE20W40 oil.

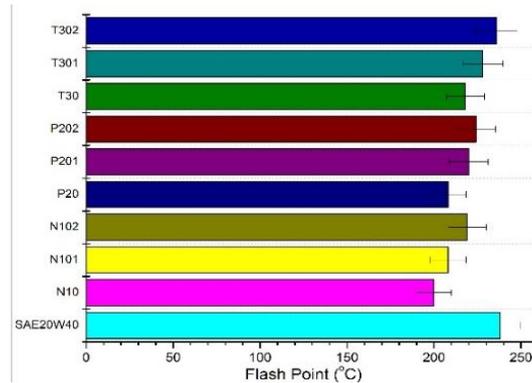


Fig. 3.4. Flash point of oil samples.

3.1.5. Friction and Wear Analysis

The results of the experiments conducted by four ball tester under test standard ASTM D4172B were discussed in this section.

3.1.6. Coefficient of Friction

In all the Coefficient of Friction plots, the curves initially increase and then decreases, which is because of the point friction changing to surface friction. As the frictional coefficient is the identification of energy consumed by the frictional surfaces, in turn reduces the wastage of power.

3.1.7. With Neem oil Methyl ester blends

The COF values of NOME blends were shown in figure (3.5). When compared with SAE20W40 oil, the N10 performed better and with addition of 1% and 2% Al_2O_3 particles, the friction coefficient values are lesser which showcases a lesser friction induced between the surfaces. Generally, Neem oil has high content of Oleic fatty acid and it has longer carbon chains [39], which might have attributed along with the rolling friction mechanism of spherical Al_2O_3 nanoparticles [33]. Without nanoparticles the COF curve seems to increase first and decreased but the plot seems to have an unsteady pattern. With addition of 1% Al_2O_3 nanoparticles, it is evident that the COF values of N101 are little higher, but it follows comparatively steady pattern, which is due to the ability of nanoparticles to fill the worn-out cavities [38, 41]. The N102 curve which has 2% Al_2O_3 nanoparticles which shows a matured friction behavior by relatively steady pace with and the friction coefficients are reduced by 13% from the SAE20W40 oil and 7% lesser than the 10% NOME blend with Al_2O_3 nanoparticles.

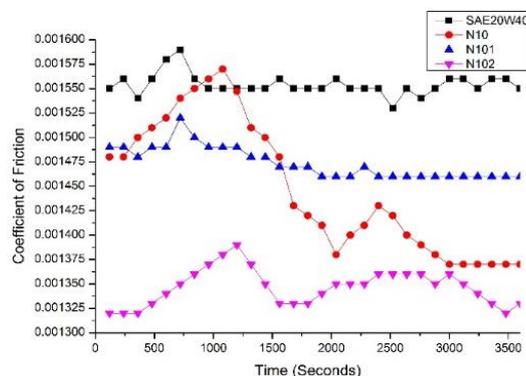


Fig. 3.5. COF comparison of NOME blends.

3.1.8. With Pongamia oil Methyl ester blends

A similar trend was found in POME blends also, since the addition of Nano Al_2O_3 particles resulted in reduced friction, which is evident when comparing the COF values in Fig (3.6). Pongamia oil methyl esters which has most higher carbon chains (Behenic and Lignoceric) which attributes to the reduced frictional properties [40]. With addition of nanoparticles, it has become even better. Al_2O_3 is generally known for its superior load bearing property [41], hence the addition of it improves frictional resistance which is apparent from the graphs. As the percentage of nanoparticle increases, there is a decrease in COF values. Also, the P202 plot shows a gradual decrease of COF values which indicates the Al_2O_3 nanoparticles filling the pores [34] and which acts as a spherical bearing thereby it results in reduced COF values. When compared to SAE20W40 oil, P201 has 5% lesser values and P202 possess even more reduced values (around 13%) of COF, which are respectively 3% and 11% lesser than P20 blend which has no nanoparticles.

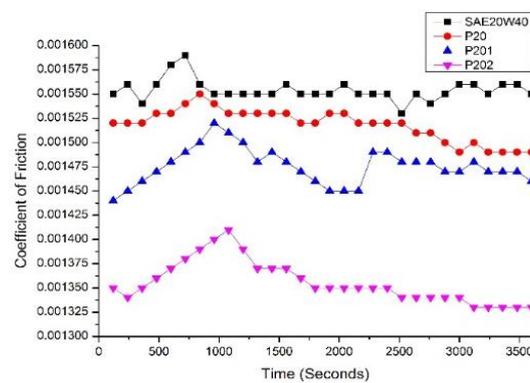


Fig. 3.6. COF comparison of POME blends.

3.1.9. With Tamanu oil Methyl ester blends

In TOME blends also the frictional coefficients of 1% and 2% blends were found to be higher than SAE20W40 oil and 30% POME added SAE20W40 blend which is shown in fig (3.7). As the concentration of Al_2O_3 is introduced with 1%, there is an increase in the COF values. This may be attributed because of the higher viscosity index advantage of T30 oil sample over T301 and T302. Also, the increased density values of T30, T301 and T302 oil samples would have dissolved the thin protective layer [33] which is responsible for friction reduction. Hence in TOME blends, the addition of 10% and 20% Al_2O_3 results in adverse effects in friction characteristics. Yet, when comparing with SAE20W40 oil, T301 blend showcases 9% lesser friction and T302 has shown 3% lesser frictional coefficient values. The T30 sample without nanoparticle additives showcased around 11% more frictional resistance than SAE20W40 oil.

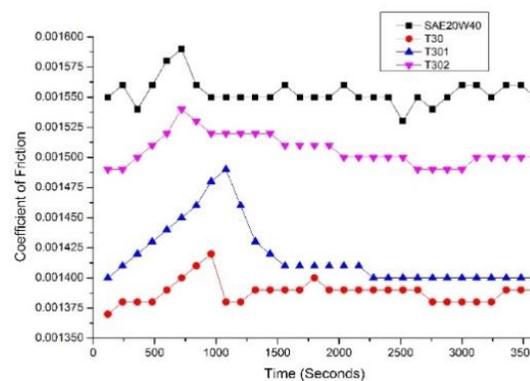


Fig. 3.7. COF comparison of TOME blends.

3.1.10. With all samples

The overall comparison of the coefficient of friction values reveals that the addition of Nano Al_2O_3 particles had influenced significantly in reducing the friction between the contacting ball surfaces which is shown in figure 3.8.

The Frictional coefficient values of N102 was found to be least than all the other blends. When comparing with unblended SAE oil it has 13.3% lesser COF value which is found to be an appreciable friction reduction. And it is observed that all the other oil blend samples namely P202, T30, T301, N10, P201, N101, T302, P201 also exhibited superior friction reduction of 13%, 11%, 9%, 7%, 5%, 5%, 3% and 2% respectively as compared with SAE20W40 oil. In both POME and NOME blends the addition of 1% and 2% nano Al_2O_3 resulted in improvisation of frictional resistance. The TOME blends react in an opposite sense, as the nanoparticles are added, there is a reduction in its performance against friction, which is mainly due to the agglomeration of nano particles [33, 35] occurs with the denser T30 oil results in collapse of boundary layer lubrication.

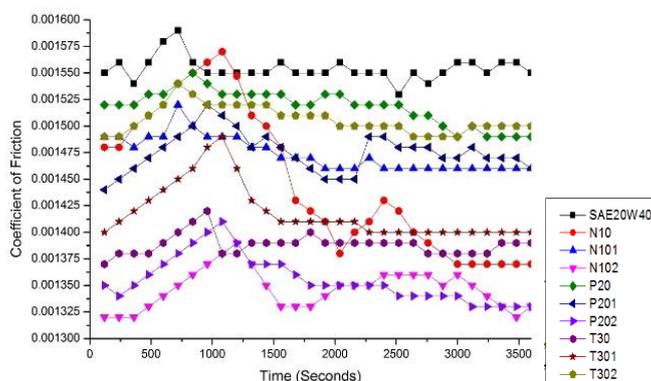


Fig. 3.8. COF comparison of all oil samples.

3.1.11. Wear scar diameter

The wear scar diameters of all the lubricant samples are shown in fig. 3.9 from observation it is evident that addition of nanoparticles has decreased the wear scar diameter which is a good outcome as expected from the literatures [33 – 35, 38]. All the biolubricant blends as well as nanoparticle added blends has shown lesser wear scar diameters. Especially in NOME samples the when compared with SAE20W40 oil's wear scar diameter (834.7 μm), the N10, N101 and N102 has wear scar diameters of 712.6 μm , 623.8 μm and 374.5 μm which are 14.6%, 25.3% and 55.1% lesser respectively. This is due to the increasing pace of density because of nanoparticle addition which increased the thickness of the wear protective film by penetration of lubricants between contact surfaces [33]. A similar trend is found in P20, P201 and P202 samples, as the nanoparticle concentration is added, the wear scar diameter decreases. In comparison with SAE oil, P20, P201 and P202 has displayed 18.2%, 28.8% and 50.5% lesser wear scar diameters at 683.2 μm , 594.3 μm and 412.9 μm . Even though POME oil has lesser density than NOME oil, it holds more percentage of unsaturated fatty acids than other two methyl esters, which is the reason for exhibiting lesser wear scar than NOME based blends [35]. In contrast the TOME blends have increasing pace of wear scar diameter still the values are lesser than the commercial lubricant SAE20W40. In comparison with SAE20W40, T30 possesses 47% (437.3 μm), T301 possesses 36% (534.1 μm) and T302 possesses 19.3% (673.9 μm) smaller wear scar diameters. It is conceivable due to the presence of more stearic acid (18.5%) than POME (6.8%) and NOME (14.4%). Saturated acids are renowned for their intermolecular interaction and the polar nature of esters, which helps to preserve the surface [35 - 37]. Furthermore, oils with a greater stearic acid content showed a better resistance to oxidation, which improves the capacity to reduce friction.

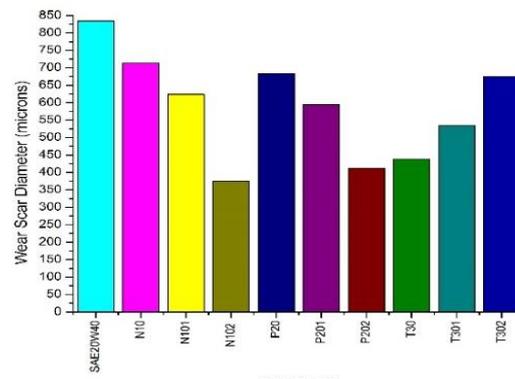


Fig. 3.9. WSD comparison of all oil samples.

3.1.12. Anti-wear and Anti friction mechanism of Al₂O₃ nanoparticles

Al₂O₃ is a hexagonal, close-packed crystal material with good hardness, heat resistance, and wear resistance. As a result, it outperforms other materials in terms of wear resistance. Because modified Al₂O₃ nanoparticles are so tiny, they may easily enter sliding contact without disrupting the hydrodynamic regime [33, 41, 42]. Since Al₂O₃ nanoparticles are generally spherical, they can function similar to ball bearings, preventing direct contact between friction pairs and converting sliding friction to rolling friction [34, 38, 41]. This helps in improving anti-wear properties under extreme pressures.

When Al₂O₃ nanoparticles are mixed with lubricating oil, they help in resisting the compressive stress by occupying the worn-out area. Furthermore, the nanoparticles are chemically adsorbed and sintered on the metal friction surface due to the elevated temperature generated by friction.

3.1.13. SEM Imaging (Wear Scar Measurement)

The SEM images of wear scar diameters are shown in figure (). It is evident that the wear pattern is having lesser grooves and the addition of Al₂O₃ nanoparticles have a beneficial effect with both NOME (N101 and N102) and POME (P201 and P202) blends, which is because of smoothing effect of the Al₂O₃ nanoparticles in the boundary lubricating regime [41]. In contrast, the nanoparticles addition presented a detrimental effect with T301 and T302 oil blends, this is attributed because of the higher density and increased viscosity values has adverse effect with nanoparticles, which lead to plowing of metals because of the higher hardness of the Al₂O₃ nanoparticles [35, 41].

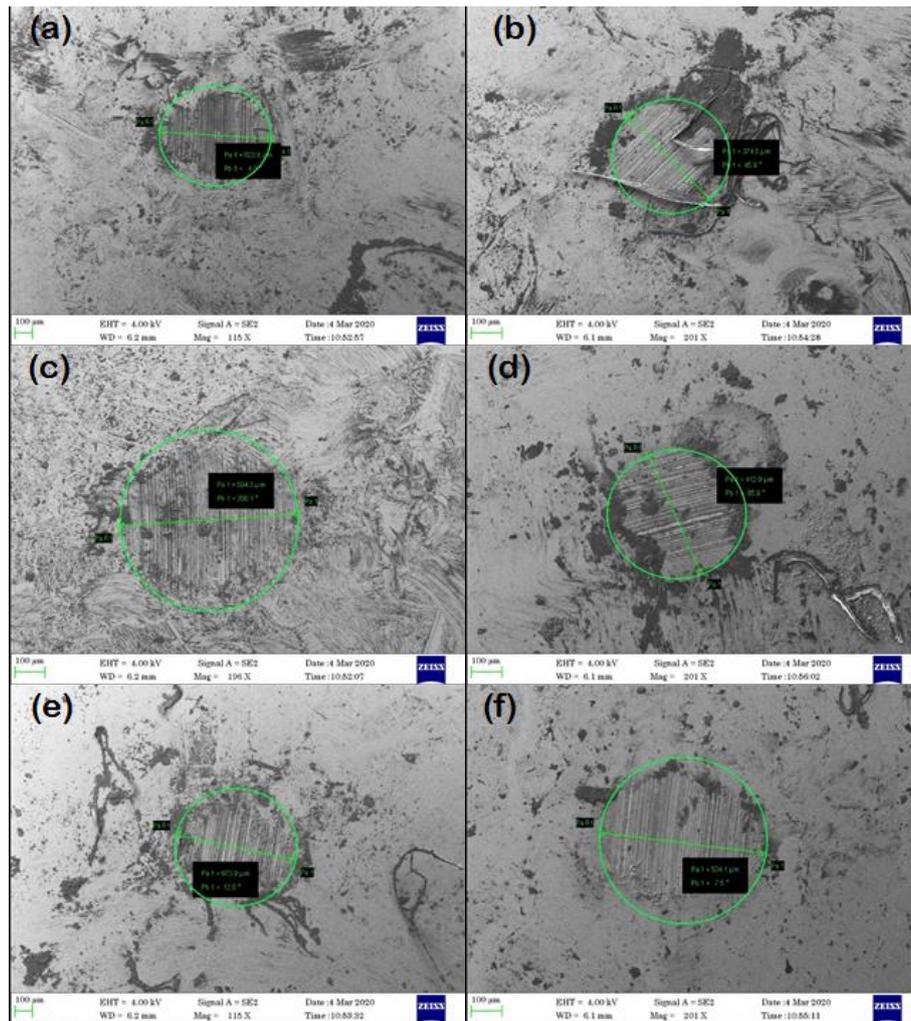


Fig. 3.10. Comparison of Wear scar diameters (a) N101, (b) N102, (c) P201, (d) P202, (e) T301, (f) T302.

4. Conclusion

The different oil blends based on vegetable oil methyl esters with 1% and 2% Al_2O_3 nanoparticles were studied based on its physiochemical properties and tribological properties. The following observations were made up of the results:

The addition of nano Al_2O_3 particles has showcased beneficial effects in physiochemical properties viz. density, viscosity and viscosity index in NOME and POME based oil blends which strongly recommends the use of appropriate quantity nano Al_2O_3 with these blends since the values are superior than the commercial lubricant SAE20W40.

In flash point, the addition of nano Al_2O_3 has made positive changes to the property, which is highly appreciable. Especially the T302 (1% less) showcased almost on par value with SAE20W40.

In friction characteristics, the blends N102 (13.3% lesser friction) and P202 (12.8% lesser friction) which has highest percentage of nano Al_2O_3 (2% wt.) have outperformed all the other blends by a fair margin. In addition, the other blends maintained better frictional resistance (about 3.1 to 13.3 percentage), demonstrating that bio-nano additions may be employed in high-friction applications.

A similar behavior was observed in wear scar analysis also. As the percentage of nano Al_2O_3 increases, the wear scar diameter reduced correspondingly. Specifically, N102 and P202 showcased smallest wear scar diameters at 374.5 (55.1% smaller) and 412.9 (50.5% smaller) in

comparison with SAE20W40 oil. Other blends also reduced the WSD about 15 - 50% which shows an optimum Bio-nano additive combo with commercial lubricant will help in reduction of wear in contacting surfaces.

Throughout the study, the behavior of Tamanu oil Methyl ester-based blends has adverse effects (except density and flash point) with the nanoparticle addition due to the non-compatible properties of both, which suggests to avoid the combination in any of the anti-wear and antifriction applications.

Overall, the results show that the friction and wear characteristics of four ball wear test were strongly depend on the concentration of nano Al₂O₃ particles.

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