



Synthesis, characterization and tribological performance enhancement of Karanja Neem blends with hBN as nanoparticles

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KEYWORD	ABSTRACT
Neem hBN Non-edible vegetable oil Karanja	<p>Metal cutting fluids (MCFs) have been widely used in machining processes due to their superior lubricating properties. Mineral-based MCFs are the most commonly utilised type in the machining industry. However, high consumption of mineral-based MCFs led to environmental and health-related hazards. Thus, the effectiveness of non-vegetable oils (NEVOs) blends with nanoparticles as an alternative to mineral-based MCFs investigated. The NEVOs chosen in this research are karanja and neem. The karanja and neem oil was blended at three different ratios (25:75, 50:50, 75:25) with addition of different hexagonal boron nitride (hBN) concentrations (0.05 wt.% and 0.5 wt.%) and a constant concentration of sodium dodecyl sulfate (SDS) (0.025 wt.%). The developed nanolubricants were evaluated in terms of kinematic viscosity, viscosity index (VI), wettability, coefficient of friction (COF) and wear scar diameter (WSD). Based on the results, karanja neem blends with 0.5 wt.% hBN outperformed the one with 0.05 wt.% hBN by providing higher VI and excellent wettability. The base oils observed the lowest COF due to their high oleic acid percentage. The karanja neem blends with 0.5 wt.% hBN again provide the smallest WSD compared to others. Overall, karanja neem blends with 0.5 wt.% demonstrated promising solutions to mineralbased MCFs in machining.</p>

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1.0 INTRODUCTION

In the manufacturing industry, most of the products require some metal removal operation at some point. Machining parameters hold significant importance in machining. The significant heat generation in the machining zone during dry machining limits the maximum limit of the machining parameters. The hardest parts of the metal removal process include controlling and removing heat, which affects the cutting tools' sharpness and hardness and causes premature breaking. In order to lower the temperature in the machining zone, metal cutting fluids (MCFs) would be beneficial. The primary purpose of MCFs is to remove chips from the machining zone and to cool and lubricate the cutting tool-workpiece interfaces. (Singh et al., 2017).

However, using MCFs excessively also endangers people and pollutes the environment. This is because mineral-based MCFs are commonly utilised in metal-cutting operations. The application of mineral-based MCFs may cause skin irritation, respiratory illness, and cancer to the labourers. The mineral-based MCF effluents also reported poor biodegradability and the discharge of the waste fluid is not allowed without proper treatment (Wickramasinghe et al., 2017). To achieve sustainability, manufacturers must implement new clean materials and technology for the formulation, application methods, and disposal of MWFs. Furthermore, the development of such green MWFs for cutting-edge technology would protect labourers' health and safety, as well as the well-being of the environment (Talib & Rahim, 2018).

Generally, the MCFs should possess excellent lubrication, high viscosity index (VI), non-corrosive, higher flash point, lower pour point and high chemical stability. Lawal et al.,(2012) claimed that the structure of triglycerides in vegetable oils provides favourable lubricating properties which vegetable oils minimise friction at the work-tool contact by forming a stronger lubricant coating between metallic surfaces due to their long polar fatty acid chains. Vegetable oil's saturated fatty acids allow for a more effective layer of lubrication at the work-tool contact than mineral oils (Guo et al., 2017). Moreover, vegetable oil-based MCFs can be degraded into environmentally benign chemicals through biological or chemical processes. The broken-down bio-based substances can be disposed of in natural resources without affecting their current properties, ensuring environmental sustainability which also has lesser toxicity than mineral oil-based MCFs (Wickramasinghe et al., 2020).

Although the usage of vegetable oil as MCFs could improve the lubrication performance in both pure and soluble states, they are subjected to auto-oxidation and drop in properties at higher loads and temperatures. Chanes de Souza et al., (2019) explained that there was a drop in cooling and lubricating performance caused by oxidation which a higher temperature was exhibited by the tribological mechanism despite the reduction in wear area. Therefore, Katna et al., (2020) highlighted that this constraint of vegetable-based MCFs could be improved through several ways of modifications including blending different vegetable oils and nanoparticle additions. This is because of the differences between oils in terms of their chemical and physical characteristics. One unaltered vegetable oil may have poor physicochemical qualities and weak oxidative stability. Thus, a new form of oil with improved functional features is produced by combining many different vegetable oils, each with its special capabilities. When blending various vegetable oils, the triglyceride structure is also changed, which affects the oil's viscosity, density, and oxidative stability.

Edible and non-edible vegetable oils (NEVOs) can be distinguished from one another. According to research by Bhuiya et al., (2014), there has been a notable increase in concern around the use of edible vegetable oils. This issue results from the continuous discussion about the tension between industrial applications and food production, which could lead to starvation,

especially in developing countries. Additionally, the use of edible vegetable oil leads to social and environmental problems, such as the requirement for additional agricultural land and the uprooting of rural communities from their marginal holdings. Since edible vegetable oils are sorely needed as a food source, it is imperative to apply NEVOs in the machining industry. Furthermore, NEVOs are becoming more and more popular for industrial use since they are far less expensive than edible vegetable oil crops and may be cultivated in locations that are not good for food crops.

Base oil has an important and large impact on the lubrication mechanism. A weight fraction of nanoparticles is added to the basic oil to enhance its lubricating qualities. Nanoparticles play a significant role in lubricant performance optimisation by exerting a significant influence on features of wear, corrosion resistance, oxidation, and friction (Shahnazar et al., 2016). Furthermore, it was noted that the use of nanoparticles enhanced the lubricant's tribological characteristics. According to Thampi et al., (2021), there are four nanoparticles' mechanisms as follows: (a) the rolling effect, (b) the polishing, (c) the mending effect and (d) the formation of protective layer by nanoparticles between the friction pairs.

Talib et al., (2017) stated that the application of green nanoparticles, such as hexagonal boron nitride (hBN) which is in the form of lamellar powder from layered crystal formations results in minimal friction. This was because hBN forms weak Van der Waals bonds between individual layers while still forming strong covalent bonds inside each layer. The lamellar powder clung to the contact surface, which formed a protective boundary lubrication coating that minimised contact surfaces and wear. It tended to arrange its layers parallel to the direction of motion, consequently minimising friction. The hBN particles, on the other hand, produced exceptionally stable chemicals that are safe to handle, non-toxic, and have no constraints during the application. These elements demonstrated an effective lubricant additive property through their anti-wear capabilities.

In this paper, karanja and neem blends will be developed at three different ratios before the addition of two different concentrations of hBN nanoparticles. Then, the developed nanolubricants will be analysed in terms of the kinematic viscosity, VI, wettability (contact angle and surface tension) and tribological behaviours (coefficient of friction and wear scar diameter).

2.0 EXPERIMENTAL PROCEDURE

2.1 Preparation of Nanolubricant

Figure 1 shows the flow of the base oils blending process. Initially, the Karanja and Neem oil were weighed and blended at three different ratios of 25:75, 50:50 and 75:25. The blending was done using a mechanical stirrer at 200 rpm for 30 minutes. Then, the oil blends were dispersed with two different concentrations of hBN nanoparticles (0.05 wt.% and 0.5 wt.%) and a 0.025 wt.% concentration of sodium dodecyl sulfate (SDS). The weight percentage of the nanoparticle and surfactant was estimated as per Equation 1. The mixture was then stirred on a hot plate using a magnetic stirrer at a temperature of 100°C and 500 rpm before being sonicated using Branson digital sonifier as shown in Figure 2. The sonication process was done for three hours with one-hour time intervals at an amplitude of 25%. Table 1 shows the list of MCFs that will be analysed in this study.

$$\text{Weight percentage concentration (wt. \%)} = \frac{\text{Weight of solute (g)}}{\text{Weight of solution (g)}} \times 100 \quad (1)$$

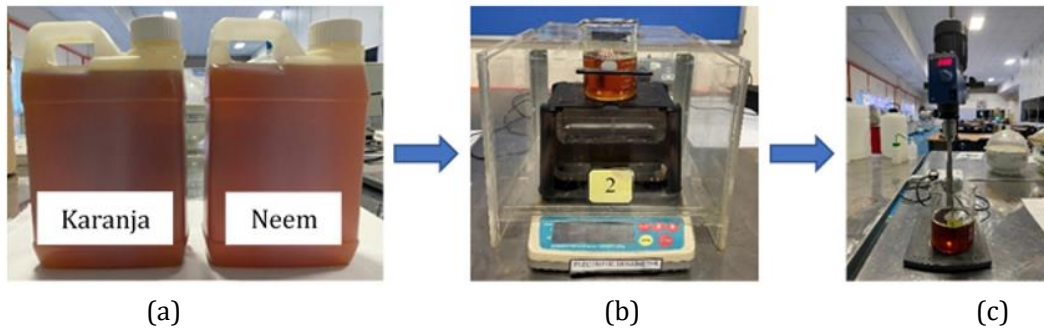


Figure 1: Blending flow; (a) Base oil, (b) weighting base and (c) blending.

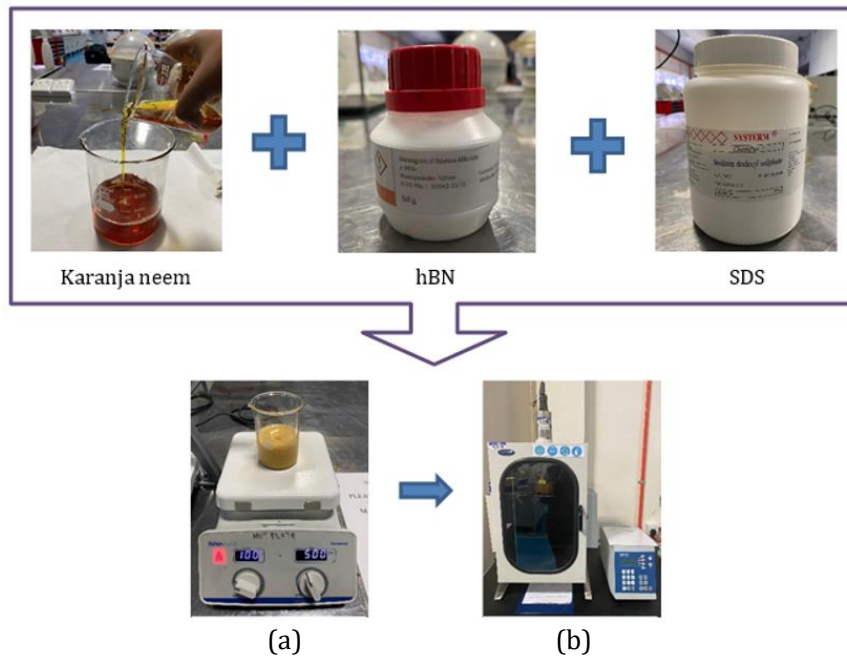


Figure 2: Development of nanolubricant; (a) stirring and (b) sonication.

2.2 Kinematic Viscosity and Viscosity index (VI)

The kinematic viscosity and VI were used to determine the rheological parameters. An Anton Parr V3000 viscometer was used to measure the kinematic viscosity at 40 and 100°C according to ASTM D445. Meanwhile, the rate at which the lubricant's viscosity changes as a result of temperature changes is measured by the viscosity index (VI) according to ASTM 2270.

2.3 Wettability

The LAUDA Surface Analyzer LSA 100, equipped with software, was used to measure the contact angle and surface tension. The MCF samples were dropped on the workpiece surface using

a syringe. Then, the contact angle was determined by measuring the tangent line interface between the MCF droplet and the workpiece surface (Stainless Steel 316L), as illustrated in Figure 3. The contact angle and droplet images were measured, and the surface tension was determined using the software. The experiment was carried out at ambient temperature as per (Prajitno et al., 2016).

Table 1: List of MCF samples.

Sample name	Base oil	Ratio	hBN (wt%)	SDS (wt%)
-	Solcut	-	-	-
-	Karanja	-	-	-
-	Neem	-	-	-
KN 1 (a)	Karanja Neem	25:75	0.05	
KN 1(b)	Karanja Neem	25:75	0.5	
KN 2 (a)	Karanja Neem	50:50	0.05	
KN 2(b)	Karanja Neem	50:50	0.5	0.025
KN 3 (a)	Karanja Neem	75:25	0.05	
KN 3(b)	Karanja Neem	75:25	0.5	

2.4 Four-Ball Test

The tribology behaviour of the MCF samples was analysed by conducting four-ball tests. The device used for this test was DUCOM FBT-3 the testing was conducted as per ASTM D4172-B. The testing involved the use of four steel balls made of AISI 52100 with a diameter of 12.7 mm and hardness ranging from 64 to 66 HRC. One of the steel balls was securely tightened to the spindle, while the other three stationary balls were assembled in the test cup. For each test, 10 ml of the MCF sample was poured into the test cup. A force of $392\text{N} \pm 2\text{N}$ was applied to press the top ball and it was then rotated at a speed of 600rpm for 60 minutes ± 1 minutes. The temperature of the MCF samples was maintained at $75^\circ\text{C} \pm 2^\circ\text{C}$. Figure 4 presents the schematic diagram of the four-ball test.

After the testing, the steel balls were removed from the test cup and cleaned with acetone to remove the oil stains. The coefficient of friction (COF) was determined using the Winducom software and the average COF was calculated from a constant region of 2500 seconds until the end of sliding time. Meanwhile, the wear scar diameter (WSD) of the three stationary balls was estimated with the application of the image acquisition system.

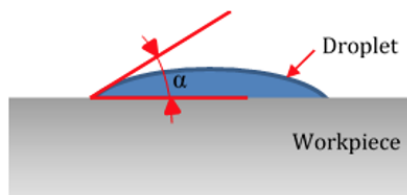


Figure 3: Contact angle measurement.

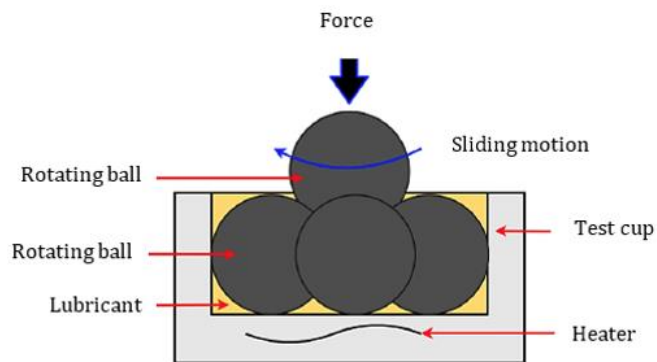


Figure 4: Schematic diagram of four-ball test.

3.0 RESULTS AND DISCUSSION

3.1 Kinematic Viscosity and Viscosity Index (VI)

Amiril et al., (2017) found that oil viscosity affects the creation of a thin lubrication layer at metal contacts, reducing friction and wear during machining operation. Figure 5 presents the kinematic viscosity and VI of all the samples. Based on the results, the viscosity ranged between 14.9 mm²/s to 48.02 mm²/s at 40°C and 3.25 mm²/s to 14.96 mm²/s at 100°C. Solcut recorded the lowest kinematic viscosity at both 40°C (14.96 mm²/s) and 100°C (3.25 mm²/s). The VI calculated for Solcut was 71.5, which is the lowest among the tested samples. According to the lubricant standard of ISO VG 46, the VI of a good lubricant should be more than 90 which Solcut failed to reach the supposed value of VI.

Meanwhile, other tested samples had successfully recorded VI values of more than 90. The VI of the karanja neem blends containing a higher concentration of hBN (0.5 wt.%) recorded a higher VI compared to a lower hBN concentration (0.05 wt.%) for all blend ratios. This indicates that the karanja neem blends with 0.5 wt.% hBN delivers a more stable lubrication film compared to 0.05 wt.%. The improvement of the VI was significantly influenced by the low thermal expansion of the hBN nanoparticles (Talib et al., 2019). As per the study by Farhanah & Syahrullail, (2016), the addition of nanoparticles resulted in a more stable film with temperature changes, leading to a higher VI value. Furthermore, with a higher value of viscosity index, oil viscosity decreases slowly and maintains in a fluid form as the temperature increases.

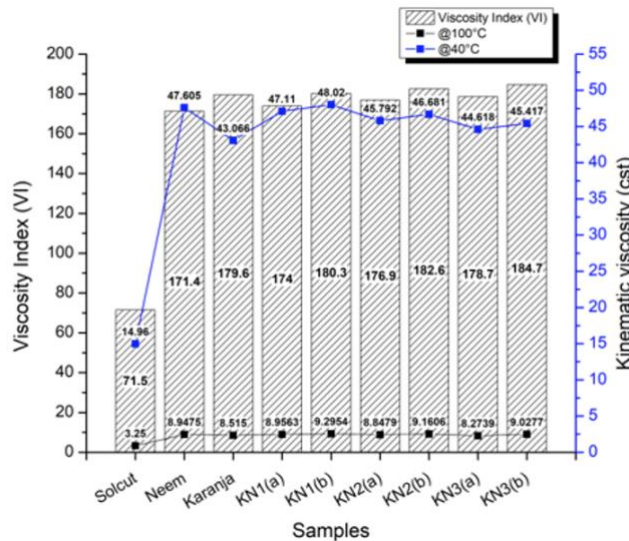


Figure 5: Kinematic viscosity and VI of MCF samples.

3.2 Wettability

The wettability refers to a lubricant’s ability to disperse, penetrate, and coat a surface. Spreading the lubricant over a surface reduces the contact angle and increases wettability. Measuring contact angles is crucial for determining fluid wettability. Şirin et al., (2023) claimed that the contact angle of a lubricant is generally related to its viscosity. Moreover, the author highlighted that lubricants should have low contact angle values to penetrate better on the

surfaces between parts which also improves the lubrication by reducing friction. Figure 6 shows the contact angles of the tested samples.

The results present that the industrial MCF, Solcut observed the highest contact angle of 51.2°, followed by the base oils of Neem (50.1°) and Karanja (49.8°) respectively. On the other hand, all the karanja neem blends recorded a lower contact angle compared to Solcut, Neem, and Karanja which there are large differences can be observed. This proves that the blending of two different base oils could improve the wettability of a lubricant. Moreover, it could be noticed that the contact angle value decreases with the increase of hBN concentrations. This corresponds to the VI values in which karanja neem blends with 0.5 wt.% perform better than karanja neem blends with 0.05 wt.%. The recorded contact angle values are in line with the research by Nguyen et al., (2012), in which a higher concentration of hBN (0.5 wt.%) provides a lower contact angle. The lowest contact angle was for the KN3(b) with a contact angle value of 18.2. Smaller contact angles of KN3(b) exhibit an increase in lubrication effect and the ability to enhance the machining performance (Sirin et al., 2021).

According to a study by Tanvir & Qiao, (2012) and Zhang et al., (2015), there is an interaction between the contact angle and surface tension. A significant factor influencing a lubricant's wettability is surface tension. The literature claimed that a drop in the surface tension led to a smaller contact angle between the lubricant and the surface, which increased the lubricant's ability to spread over a larger area. Figure 7 illustrates the surface tension for the MCF samples. The highest surface tension was recorded by Solcut with a value of 74.304 mN/mm. Meanwhile, Neem, Karanja and karanja neem blends at all ratios observed lower surface tension values compared to Solcut.

Again, the addition of a higher concentration of hBN decreases the surface tension of the samples. The surface tension also shows a similar trend as the contact angle value. The drop-in surface tension is due to Brownian motion and nanoparticle adsorption at interfaces (Estellé et al., 2018). The lowest surface tension was recorded by KN3(b) with a value of 33.62 mN/mm. The effectiveness of higher hBN concentration dispersion in karanja neem blend corresponding to the finding by Huminic et al, (2017) highlighted that dispersion of 0.5 wt.% of nanoparticles decreases the surface tension in comparison to the base oil.

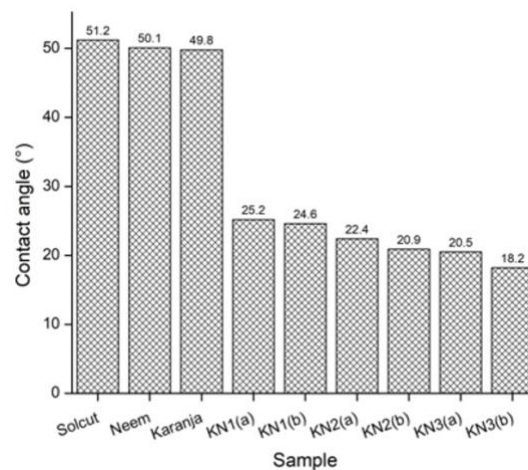


Figure 6: Contact angle of MCF samples.

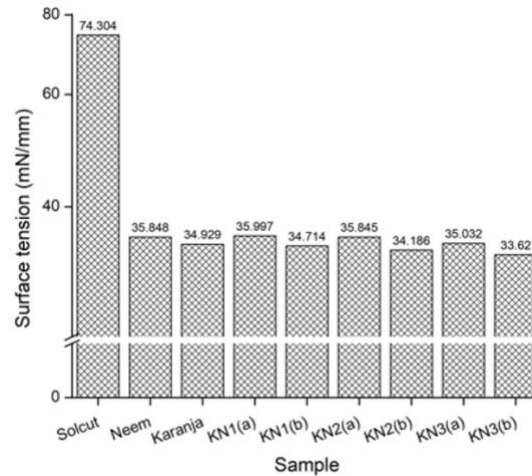


Figure 7: Surface tension of MCF samples.

3.3 Coefficient of Friction

Figure 8 shows the COF of the MCF samples against the sliding time. It can be observed that the COF of the MCF samples were unstable at the beginning. However, the COF stables towards the end of the testing. The average COF of the MCF samples were calculated at the constant region/steady state of the 2500s to the end of the sliding time. Figure 9 presents the calculated average of the MCF samples. According to the average COF, Solcut recorded the highest COF with an average of 0.0558. Meanwhile, Neem recorded the lowest average COF of 0.0459 followed by Karanja with an average value of 0.0469. This is because of the triglyceride chain found in vegetable oils (Neem and Karanja) is the reason for their low friction properties (Sajeeb & Rajendrakumar, 2019). According to the study by Pillay et al., (2017), pure vegetable oils produce lower COF due to the high scar depth which led to the trapping of lubricant inside the scar grooves, acting as an oil reservoir on the mating surface.

The karanja neem blends at all ratios provide lower COF in comparison to the industrial lubricant, Solcut. The addition of hBN to the karanja neem blends reduces the COF value. This is because The thin lubricating coating created by the hBN particles changed the type of friction from sliding to rolling (Talib et al., 2017). This also could be supported by the research by (Reeves et al., (2013) that the hBN particles in canola oil provide lubricating components by aligning in the same direction as the relative motion of a solid body and sliding over one another easily. Among the karanja neem blends, it can be observed that the COF decreases with an increase in hBN concentration. KN1(b), KN2(b) and KN3(b) recorded lower COF values compared to KN1(a), KN2(a) and KN3(a). Moreover, KN2(b) recorded the lowest COF among the karanja neem blends with an average COF of 0.0521. The high concentration of 0.5 wt.% hBN improved the lubricity while minimizing the friction at the sliding interfaces. Moreover, a thin lubrication film was developed by the hBN nanoparticles by filling the valley at sliding interfaces which helped in the reduction of friction. According to Sani et al., (2024), a lubricant's viscosity index determines its ability to sustain the tribo-film at different temperatures which the existing ball bearing effect changed to a sliding effect at the contact surfaces of the steel balls. This works as a barrier across the metal surfaces, preventing them from clashing, and contributing to the reduced COF value of KN2(b).

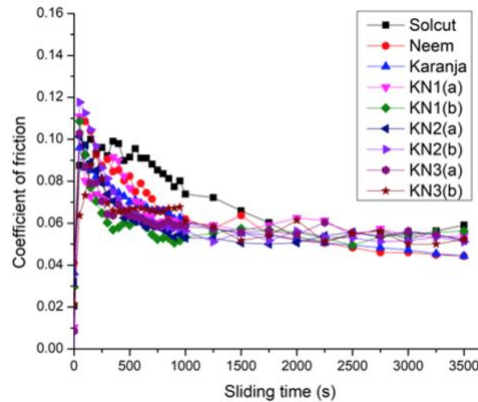


Figure 8: Coefficient of friction versus sliding time.

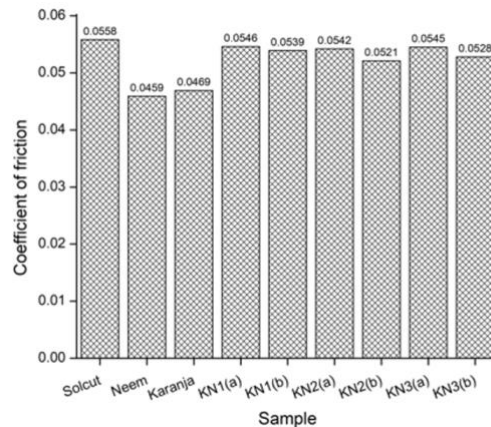


Figure 9: Average coefficient of MCF samples.

3.4 Wear Scar Diameter

Figure 10 shows the results on average WSD of the MCF samples. WSD refers to damage caused by material removal from both sliding and solid surfaces. The highest WSD was recorded by Solcut with an average of 1009 μm followed by Neem and Karanja with an average WSD of 859.6 μm and 833.7 μm respectively. Although vegetable oil has a low coefficient of friction, the wear rate is significant due to chemical action on the surface by the fatty acids inside the oil. During sliding, the soapy metallic film wears away and makes non-reactive detergent, which increases the wear (Habibullah et al., 2015).

In correlation to the COF value, a decrease in WSD can be observed with an increase in hBN concentration in the karanja neem blends. 0.5 wt.% hBN in the karanja neem blends further decrease the WSD compared to 0.05 wt.%. This is because, a minimum hBN concentration (0.05 wt.%) offered insufficient protection between the friction surfaces, increasing the exposed area, resulting in a rise in COF and a greater WSD. KN2(b) observed the lowest average WSD with a value of 711.3 μm . The lower value of WSD indicates the hBN nanoparticles developed a ball-

bearing effect by converting the sliding friction between the frictional pairs to rolling friction, resulting in a decrease in contact area (Abdullah et al., 2013, Guglea et al., 2020). Moreover, Sabri et al., (2023) also explained that the presence of nanoparticles reduces wear by generating a stronger protective transfer layer by filling in the gaps between the contact surfaces.

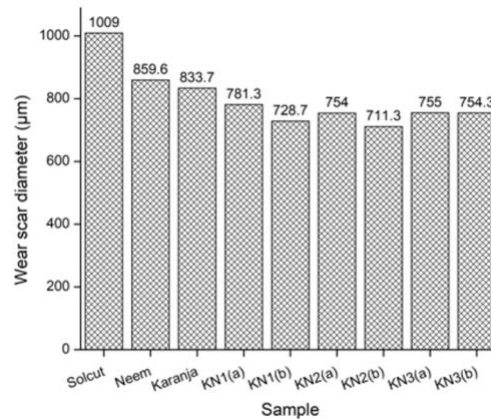


Figure 10: Average WSD of the samples.

CONCLUSIONS

Based on the experiment and analytical results, the following conclusions can be drawn:

- The karanja neem blends observed an increasing viscosity index with increasing concentration of hBN at all blend ratios. The highest VI recorded by KN3(b) with a value of 184.7
- In terms of wettability, the karanja neem blends overall perform better compared to the Solcut by providing a lower contact angle and surface tension, especially the one with a higher concentration of hBN nanoparticles. KN3(b) delivered the lowest contact angle (18.2°) and surface tension (33.62 mN/mm).
- Neem and Karanja recorded the lowest average COF due to reduced friction by lubrication boundary by the fatty acids. However, in contrast, the karanja neem blends at all ratios provide a better WSD, especially Karanja Neem with 0.5 wt% hBN which the lowest WSD was recorded by KN2(b) with an average of 711.3 µm.

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