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Experimental Evaluation of Biolubricant with Additive Nanoparticle Calcium Carbonate ($CaCO_3$) from Scallop Shell Waste as Cutting Fluids using Minimum Quantity Lubrication (MQL) in CNC Milling Process

Nano-cutting fluid sprayed using the minimum quantity lubricant (MQL) method is one example of a green manufacturing process. Meanwhile, vegetable oil is an appropriate lubricating base oil as it offers very high lubricating performance and environmental friendliness. Further, $CaCO_3$ nanoparticles are popular for their capacity to improve lubrication properties and performance. However, the optimum impact of utilizing different types of vegetable oil remains inadequately investigated. Therefore, this study aims to analyze the effect of $CaCO_3$ nanoparticles on the performance of cutting fluid, specifically on the thermophysical, rheological, and tribological properties in the CNC milling process of AISI 1045 Steel material. The nano-cutting fluid was prepared using different vegetable oils (canola, corn, soybean) added with $CaCO_3$ nanoparticles with a mass concentration of 0.15%. The results showed that the thermophysical properties, including density and viscosity, were highest when using canola oil, and the addition of $CaCO_3$ to all samples did not significantly affect thermal conductivity. Meanwhile, for the rheological properties, we observed Newtonian for all cutting fluid samples. For tribological properties, canola, and corn oil were better for obtaining a minimum Ra value, while soyabean oil was more effective in reducing cutting temperature. Based on the results of tool wear calculations, each oil presents the best performance in reducing wear, especially with the addition of $CaCO_3$. For chip formation, on average, the samples produce irregular tooth morphology with C-type, comma, and elongation shapes. Meanwhile, the resulting chip color was dark purple, which changed to dark brown and light brown, then turned silvery white due to wear and tear on the different sides of the chisel.

Keywords: *Cutting fluid, Additive $CaCO_3$, Biolubricant, CNC, MQL.*

1. INTRODUCTION

Following the rapid progression of sophisticated tech–nology, Computer Numerical Control (CNC) machines have become ubiquitous in various manufacturing pro–cesses, particularly in milling machines. These mac–hines excel at producing precise vertical or horizontal cuts on workpieces, leading to enhanced processing efficiency and impeccable surface quality. However, friction predominantly always occurs during the machining process, which causes heat and pressure in the cutting area. Therefore, lubrication is necessary to reduce heat due to friction between the cutting tool and the workpiece surface [1,2]. Specifically, lubrication during the cutting process functions to reduce heat, lower surface friction, eradicate chip dirt, and prevent corrosion on the tools [3–6]. In traditional machines, a large amount of mineral oil-based flooded cutting fluid is commonly used during the cutting process to reduce the cutting temperature. However, only a tiny amount of oil enters the grinding area to serve as the cooling and lubrication agent, obstructing effective heat transfer [7,8]. Further, the excessive use of cutting fluid also increases the machining cost [9,10] and environmental pollution, which further results in health risks [11]. Besides, its availability decreases significantly over time [12]. Consequently, a solution for those issues is required to generate a more effective machine process.

As popularly acknowledged, the commonly used cutting fluid originates from mineral oil [13], resulting in significant annual growth of mineral oil, which may lead to the disappearance of mineral oil in the future [14,15]. Additionally, 20% of mineral oil waste is al–ways disposed into the ecosystem, which pollutes the environment [16,17]. Consequently, given the progress

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in science and technology and the growing concern for environmental preservation, there is an urgent demand for research in sustainable manufacturing, which focuses on environmentally friendly practices, resource conservation, and efficient energy utilization. Vegetable oil emerges as a solution to this problem as it offers excellent properties, including non-toxic [18], greatly bio-degradable [19], abundantly available, and low cost [20,21].

A number of studies have formulated cutting fluid based on nanoparticles to enhance its heat transfer and basic lubrication performance [22]. Nano-based fluid refers to a novel liquid that emerges from the dispersion of nanoparticles, each having a size less than 100 nm, into the base cutting fluid [23]. Nano-engineering enhances the properties of the cooling agents, including their thermal, rheological, and tribological properties, mainly to form tribofilms that function to reduce wear and surface roughness [24], as well as being a rolling effect media and protective films during the friction process [25,26].

In addition, the adoption of dry cutting has become prevalent in machining operations due to its ability to reduce costs and environmental pollution. This is not solely attributed to the absence of cutting fluid in the process but because it needs no cooling medium in the cutting area. However, it generates greater friction and adhesion, resulting in higher surface roughness [27]. Simultaneously, because the cutting process generates chips that tend to accumulate on the workpiece's surface, there is heat retention in the cutting area, which further elevates the temperatures and pressures, resulting in substantial cutting tool wear [28]. Following this issue, MQL is a cutting fluid spraying technology alternative that promotes environmental friendliness [29]. It is also commonly referred to as near-dry machining, mist cooling system, and micro-lubrication through spraying cutting fluid in the cutting zone with a high-pressure air mixture for effective cooling and chip cleaning [30–32]. Additionally, MQL technology offers the advantage of precise and low liquid consumption, typically ranging from 10 to 100 mL/hour, which is significantly lower than that of Flooded cutting fluid, amounting to 1200 L/hour [33]. Besides in addition to its cost-effectiveness in liquid usage, MQL also ensures cleaner cutting zones and enhances machining efficiency [34].

To evaluate the performance of cutting fluid and vegetable oil lubrication with the addition of nanoparticles using MQL technology, many studies have applied this technology to various processes such as milling, turning, drilling, grinding, and so forth. Priarone et al. [35] investigated the effect of the MQL lubrication strategy on machining performance in milling titanium aluminide using vegetable oil, flood cutting, and dry machining. The study revealed that vegetable oil-based cutting fluids exhibit reduced flank wear, prolonged tool life, and lower average surface roughness compared to flood-cutting and dry machining. Yin et al. [36] examined the performance of four types of cutting fluid, namely dry, flooded, vegetable oil-based NMQL, and vegetable oil-based MQL and Al_2O_3 nanoparticles, in 45 steel milling machining. The study concludes that vegetable oil-based MQL and NMQL have lower force and friction coefficient values than dry and flooded cutting fluids. Afonso et al. [37] investigated the performance of cutting fluids based on vegetable, synthetic, and dry oils, suggesting that cutting fluids made from vegetable oils present excellent capacity in reducing cutting temperatures compared to synthetic and dry cutting fluids. Meanwhile, Bai et al. [38] compared the performance of cutting fluid with several types of vegetable oils, namely cottonseed, palm, castor, soybean, peanut, and synthetic, indicating that palm oil has the best performance in reducing cutting force and Ra. Yin et al. [39] and Dong et al. [40] explored the performance of cutting fluids in contrast to vegetable oil in milling Ti-6AI-4V. Their results showed that the addition of Al_2O_3 and SiO_2 nanoparticles to the base fluid results in a better lubrication and cooling effect than other cutting fluids.

For the different turning, Cetin et al. [41] employed four types of cutting fluids, including two vegetable oils (sunflower and canola), as well as two types of commercial cutting fluids (mineral and synthetic oils) to be sprayed under pressure on AISI 304L steel machining. The result showed that sunflower and canola-based cutting fluids performed better against Ra and cutting forces than other types of cutting fluids. Meanwhile, using various types of vegetable oils (coconut, sesame, and canola) mixed with MoS_2 nanoparticles and sprayed with MQL in the AISI 1040 machining process, Rapeti et al. [42] reported that coconut oil has a more remarkable ability to reduce cutting force, cutting temperatures, tool flank wear, and surface roughness. Pervaiz et al. [43] investigated the Ti6Al4V machining process with vegetable oil-based MQL conditions and compared its performance with dry cutting and flood machining. The result indicated that vegetable oil-based MQL presented lower friction. Further, Jeevan et al. [44] used vegetable oil-based cutting fluids (pongam, jatropha, neem, and mahua) and compared them to mineral oil in the AA6061 and AISI 304L lathe processes using MQL. The result confirmed that vegetable oil had better capacity in reducing tool flank wear, cutting force, and Ra due to its fatty acids that can improve lubricating properties in comparison to mineral oil.

For the grinding and drilling, Wang et al. [45] investigated the lubrication properties using MQL in the grinding process with various types of vegetable oils (soybean, peanut, corn, rapeseed, palm, castor, and sunflower) and reported that vegetable oil carries better ability as the cooling medium compared to flood grinding machining. Zhang et al. [46] assessed the performance of MoS_2 nanoparticles added to various vegetable oils (soybean, palm, and rapeseed oil) in comparison to liquid paraffin in the grinding process using MQL technology. The study suggested that palm oil presents the advantages of reducing the coefficient of friction, specific energy, and surface roughness. Pal et al. [47], using vegetable oil added with different nanoparticles (Al_2O_3, MoS_2, SiO_2, CuO, and Graphene) in MQL bottom drilling compared to flooded and dry machining, reported that the MQL strategy with Al_2O_3 mixture had the best drilling performance compared to others, especially on the thrust, torque, Ra, drill tip temperature, tool wear by forming a good tribofilm. Virdi et al. [48] explored the machining process of MQL drilling using vegetable oil with various concen–trations of CuO nanoparticles and compared the results with flooded and dry cutting. The study suggested that the cutting fluid improved machining performance, specifically in the surface roughness and energy con–sumption, because the polishing and rolling effects on the tool work interface were observed from the sample with 0.5% CuO nanoparticles compared to the flooded and dry cutting.

Referring to a large number of studies on the performance of vegetable oil-based cutting fluids, vege–table oil has been proven to be compatible as a base oil in MQL technology with various machining applica–tions. Further, the addition of nanoparticles also produ–ces better cooling and lubrication effects. However, in general, there have not been many studies that inves–tigate the performance of various vegetable oils. Hence, further research and development are required to exp–lore the performance of vegetable oils, especially the ones with diverse types, compositions, and molecular structures, to yield distinct lubrication and cooling effects. To identify the vegetable oil with the best cut–ting fluid performance in the machining process, we used three different types of vegetable oil (namely canola, corn, and soybean) as base oils in the AISI 1045 steel milling process. We specifically assess the R_a, tool wear, and wear debris formed during the milling pro–cess. In addition, the use of $CaCO_3$ nanoparticles as an additive in cutting fluid is still limited and there are still many who have not researched it, so in this study using $CaCO_3$ made from scallop shell waste to be mixed into vegetable oil to realize green machining at a lower cost in the future.

In addition, we also used calcium carbonate ($CaCO_3$) nanoparticles added to the vegetable oil. $CaCO_3$ was obtained from scallop shell waste (Amu–sium pleuronectes) which had been synthesized into nanoparticle powder. The $CaCO_3$ made from scallop shells was selected as we aim to reduce the waste, which contains 98% $CaCO_3$, is abundantly available in nature, and is more economical. To find the level of novelty of this research, we have analyzed previous studies that used the same additive material in their research, namely $CaCO_3$ nanoparticles as a comparison and bridge of originality of this research. Additionally, the $CaCO_3$ lubricant additive offers a number of advantages, including excellent anti-wear properties, reduced friction, load-carrying capacity, and extreme pressure resulting in good tribology. [49]. $CaCO_3$ also presents excellent chemical stability, enhances the anti-wear and friction properties, great mechanical properties and anti-wear [50–53].

However, in previous studies, the use of $CaCO_3$ additives was still limited to the exploration of varia–tions in concentration in water and mineral oil media, and most of the test applications were only in the pin-on-disk test, no one had analyzed their performance in machining process applications, especially using the MQL technology spraying method which is starting to develop a lot today, Therefore, this research tries to explore experimentally by varying the use of various types of vegetable oils as the base fluid with the addition of synthesized $CaCO_3$ nanoparticles derived from scallop shells as nano-cutting fluid additives.

The performance of $CaCO_3$ will be evaluated expe–rimentally to analyze its effect on the thermophysical, rheological, and tribological properties of various types of vegetable oils as base oils for nano-cutting fluids. The practical application of nano-cutting fluid in this research is as a cooling medium in the CNC milling machining process to improve cutting performance effi–ciency, reduce friction, and cutting tool wear in cutting AISI 1045 steel using the MQL method.

2. EXPERIMENTAL PROCEDURES

2.1 Materials

For preparing the cutting fluid samples, we used canola oil from the Duogo brand, corn oil brand Mazola, and soybean oil used from the Sania brand as base oils. The properties of the vegetable oil are shown in Table 1. The $CaCO_3$ material added to the base oil was synthesized from scallop shell waste. The Properties of $CaCO_3$ are summarized in Table 2.

Table 1. Properties of Vegetable Oile

Properties	Lubricant Type		
	Canola	Corn	Soyabean
Color	Light yellow	Pale yellow	Pale yellow
Density (g/cm^3)	0.916	0.921	0.919
Flash Point (^{o}C)	325	325	329
Kinematic Viscosity, 40 ^{o}C (mm^2/s)	35.6	32.3	29.5
Acid Value (mg KOH/g)	0.48	0.31	0.20
Refraction Index (n D^{40})	Max 0.3	Max 0.3	Max 0.3

Table 2. Properties of $CaCO_3$ from Synthesized Scallop Shell

Properties	$CaCO_3$
Color	White
Melting Point (^{o}C)	825
Density (g/cm^3)	2.80
Thermal Conductivity (W/mK)	2.19
Spec. Surface Area (m^2/g)	60-80
Specific Heat Capacity (MJ/m^{3}K)	1.077

Additionally, the material for MQL cutting fluid in the milling process was AISI 1045 steel with a workpiece size of 50mm x 50mm x 20mm (L x W x H). Its chemical composition and mechanical properties are presented in Tables 3 and 4. Endmill high-speed steel (HSS) brand SOLID with a diameter 8 mm and four flutes, and its specifications are shown in Table 5.

Table 3. Chemical Composition of AISI 1045 Steel

Element	Component (%)
Carbon	0.045
Manganese	0.69-0.83
Silicon	0.19-0.29
Phosphorus	0.008-0.039
Sulfur	0.015-0.02
Iron	Balance

Table 4. Mechanical Properties of AISI 1045

Mechanical Properties	AISI 1045
Ultimate Tensile Strength (MPa)	569
Yield Strength (MPa)	343
Elongation (%)	20
Modulus of Elasticity (GPa)	205
Machinability (%)	55
Shear Modulus (GPa)	80

Table 5. Specification of Endmill HSS

Specification	HSS
Material	High-Speed Steel
Brand	SOLID
Diameter (mm)	8
Length (mm)	70
Number of Flutes	4 flute

2.2 Cutting Fluids Preparation

The cutting fluid samples were prepared using the two-step method through a series of stages consisting of a stirring process and a sonication process as described in previous literature [54–56]. In the first step, we determined the concentration of the $CaCO_3$ nanopar–ticle. In this study, we used 0.15% of the $CaCO_3$ mass fraction dispersed into base oil. The cutting fluid sample design experiment in the study is shown in Table 6.

Table 6. Experimental Design

Exp. No	Cutting Fluid	Nanoparticles concentration (wt%)	Lubricating Condition
1.	Without cutting fluid	-	Dry condition
2.	Pure canola oil	0	Pure oil MQL
3.	Pure corn oil	0	Pure oil MQL
4.	Pure soyabean oil	0	Pure oil MQL
5.	Canola + $CaCO_3$	0.15	Nano-cutting fluid MQL
6.	Corn + $CaCO_3$	0.15	Nano-cutting fluid MQL
7.	Soybean + $CaCO_3$	0.15	Nano-cutting fluid MQL

Secondly, we weighted 0.15% of CaCO3 nano–particle powder using a digital scale. Third, the weig–hted nanoparticle powder was mixed into the base oil used. Fourth, the mixtures were stirred using a magnetic stirrer for 20 minutes at 1250 rpm. Fifth, after the stirring process, the samples were sonicated using an ultrasonic homogenizer for 30 minutes to generate an excellent dispersion level for each cutting fluid sample. Then, the cutting fluid sample was ready to be tested and applied to the CNC milling machining process for optimal performance analysis. Fig 1. illustrates the cutting fluid preparation process.

Fig 1. Process of Sample Nano-Cutting Fluid Preparation using a Two-Step Method

2.3 Characterization Techniques

In the first step, the $CaCO_3$ nanoparticles were characterized. This characterization involved SEM ana–lysis using FEI Japan (Inspect-S50), XRD using Pan Analytical (Expert Pro), and FTIR using Shimadzu (Irprestige 21). *Secondly*, the density of the cutting fluid sample was analyzed using the results of the sample weighing with a digital scale, while the volume value was obtained by measuring the base oil dose using a measuring cup. *Thirdly,* thermal conductivity tests were carried out to determine the conduction heat transfer in the cutting fluid using a KD2 pro thermal properties analyzer. *Fourth,* dynamic viscosity tests were con–ducted to determine the level of viscosity or the resis–tance value of a given flow and to calculate the rheo–logical value of the cutting fluid using the NDJ-8S Viscometer.

The rheological analysis was carried out by calculating the value of the shear rate and shear stress from the obtained viscosity results. The shear stress value was obtained from the multiplication of the dynamic viscosity with the shear rate, while the shear rate was garnered from the following equation 1:

$$\gamma = \frac{2\omega R_c^2 R_b^2}{x^2\left(R_c^2 - R_b^2\right)} \quad (1)$$

In which γ is the shear rate (/s); ω is shaft angular speed (*spindle*) (rad/sec); R_c vessel radius (cm); R_b represents the shaft radius (*spindle*) (cm); x is the radius shear rate is being calculated (cm).

After the milling process, several other charac–terizations were carried out, including the surface rou–ghness test using Mitutoyo SJ-301, cutting temperature using an IR thermometer, endmill edge wear using macro photos, and chip morphology using SEM. Fig. 2 shows the scheme of tests performed in this research.

2.4 Experimental Setup CNC Milling Machining

The CNC milling process was examined to identify the performance of cutting fluid samples from different types of vegetable oils (canola, corn, and soybean oil) added with $CaCO_3$. The machining process was comp–leted using milling parameters which are controlled according to criteria presented in Table 7 and Fig. 3.

Table 7. Milling Parameters

Milling Parameter	Parameter Setting
Milling way	Face Milling
Workpiece	AISI 1045
Tool Type	Endmill cutter with 4 flute
Tool diameter (mm)	8
Spindle Speed (rpm)	3000
Feed Rate (mm/min)	0.12
Dept of Cut (mm)	1.5
Cutting Speed (m/min)	110
Cooling Type	MQL
MQL Nozzle Distance (mm)	20
MQL Nozzle Angle (°)	45
MQL Pressure (Bar)	4

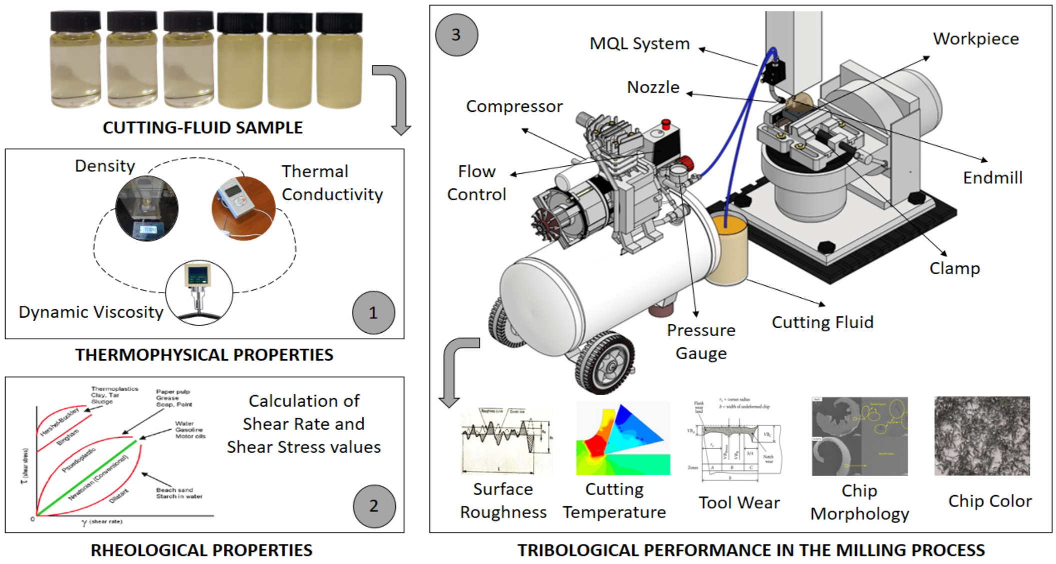

Fig 2. Sample Characterization Scheme for Cutting Fluid

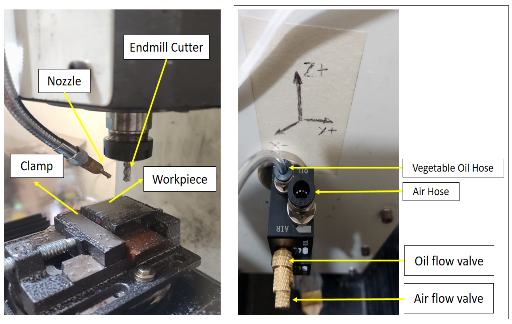

Fig 3. Experimental Equipment

3. RESULT AND DISCUSSION

3.1 Characterization of $CaCO_3$ Nanoparticles

3.1.1 SEM of $CaCO_3$ Nanoparticles

SEM analysis was carried out on $CaCO_3$ nanoparticle additives to investigate and identify their morphological structure. The results of the SEM analysis are the images with various magnifications. SEM test results on $CaCO_3$ with magnifications of 50,000 and 100,000 are shown in Fig. 4.

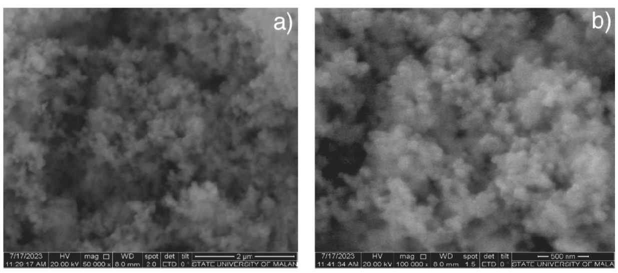

Fig 4. $CaCO_3$ Nanoparticles Morphology in (a) 50.000X, (b) 100.000X Magnification

The results suggested that the calcium carbonate synthesized from scallop shell waste has a spherical shape and is not uniform in size [57–59]. Besides, the test results also indicate the presence of agglomeration caused by several factors, such as sintering temperature, sintering time, uneven crushing, and the duration of the crushing process [58,60,61]. To overcome the agglomeration of $CaCO_3$ nanoparticles being added to base oil, special treatment using a magnetic stirrer and sonication is required to produce a stable nanolubricant.

3.1.2 XRD of CaCO3 Nanoparticles

In this study, XRD analysis was performed to characterize the properties of $CaCO_3$ nanoparticles synthesized from scallop waste. This analysis included phase, crystal structure, and crystal size identification. The XRD results produce a graph with peaks to be further analyzed. Fig. 5 shows the XRD results for the $CaCO_3$ nanoparticle.

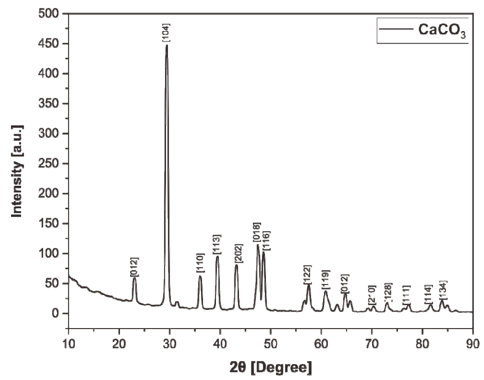

Fig 5. XRD Graph of $CaCO_3$ Nanoparticles

Based on the XRD results presented in Figure 6, $CaCO_3$ has a single phase with calcite crystal symmetry indicated by the highest peak of 1194.07 at 2θ = 29.4554°. Meanwhile, its shape is a trigonal crystal (hexagonal axes) [57]. The Miller index for $CaCO_3$ synthesized from scallop's shell at 29.45, 39.45, 43.21, 47.55, and 48.57 angles are (104), (113), (202), (018), (116). The crystallite size of $CaCO_3$ synthesized from scallop shell waste was calculated using the Scherrer Formula [61,62].

$$d = \frac{K \cdot \lambda}{\beta \cos \theta} \tag{2}$$

From the estimation results, we found a 52.18 nm crystallite size of the $CaCO_3$ nanoparticle synthesized from a scallop shell.

3.1.3 FTIR of CaCO3 Nanoparticles

FTIR was carried oy to identify the functional groups of $CaCO_3$ powder synthesized from the scallop shell waste. The obtained FTIR for $CaCO_3$ testing is shown in Fig. 6.

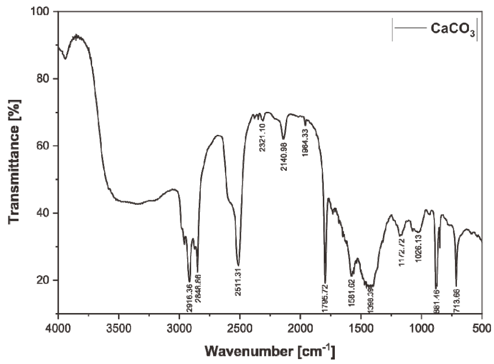

Fig 6. FTIR Graph of $CaCO_3$ Nanoparticles

The results showed sharp peaks of 2916.36 and 2848.86 $cm^{-1,}$ which indicate C-H bending vibrations. These vibrations emerge due to the presence of an organic layer of amino acids in the scallop shell [63]. The absorption peak identified at 2511.31 cm^{-1} is a C-H stretching vibration, which indicates the growth of different $CaCO_3$ bonds [64]. Meanwhile, the peak at 1398.39 cm^{-1} shows the characteristics of $CaCO_3$ from scallop shells [65]. The absorption peaks for pure cal–cium carbonate are at 881.46 cm^{-1} and 713.66 cm^{-1} peaks, indicating the characteristics of calcium carbo–nate with the type of calcite [65].

3.2 Thermophysical Properties

Thermophysical properties are the physical properties that correlate with heat transfer. These thermophysical properties are essential in evaluating, designing, and modeling heat transfer processes from a fluid, especially for the cutting fluid, whose main function is as a cooling agent. The thermophysical properties include density, thermal conductivity, and viscosity.

3.2.1 Density

In a fluid, density represents the amount of a substance contained in one unit of volume. Measurement of the density in the cutting fluid presents a pivotal role in assessing the performance of the cutting fluid during its application, specifically in the machining process. Fig. 7. presents the results of the density test of cutting fluid samples made from vegetable oil mixed with $CaCO_3$ nanoparticles.

As illustrated in Fig. 7, each sample added with a variation of $CaCO_3$ nanoparticles has different density values statistically. The highest density value of 864 kg/m^3 has been observed at 0.20% mass fraction. Meanwhile, the lowest density value was obtained for corn oil without nanoparticle addition, which was 827 kg/m^3. Therefore, these results signify that the higher the mass fraction of nanoparticles addition to the base oil increases the density. In this density test, $CaCO_3$ nanoparticles have a role in changing fluid properties, where the addition of $CaCO_3$ nanoparticles to the base fluid can increase the density of the cutting fluid sample, it is because the CaCO3 nanoparticles mixed can increase the amount of substances contained in a unit volume of base fluid [66].

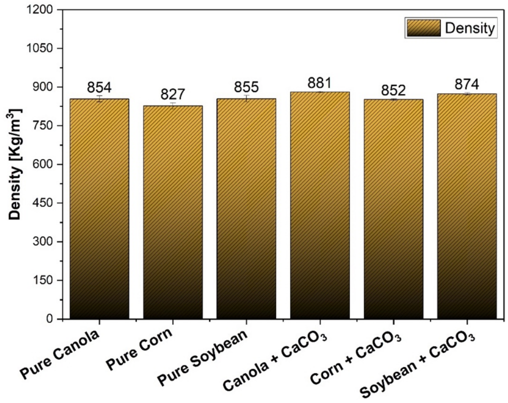

Fig 7. Density of Cutting Fluid Samples

These results are in accordance with previous research reporting increase in density occurs following the increase in nanoparticle concentration [67]. This increase is due to the increasing number of nanoparticles which accelerates the mass in the cutting fluid samples, resulting in greater density [68–70]. Nanofluid presents a higher density value than the base fluid because of the higher density of the nanoparticles contained in vegetable oil. In addition, the density of the cutting fluid affects the performance of the cutting fluid, with higher density of the cutting fluid increases the hydrostatic pressure generated by the cutting fluid. Further, this hydrostatic pressure speeds up the machining process by strengthening the flow of cutting fluid in the cutting zone.

3.2.2 Thermal Conductivity

Thermal conductivity shows the heat transfer capacity of a fluid. It serves as the central parameter of the thermophysical properties of a cutting fluid due to its main function as the cooling agent. Fig. 8. presents the results of the thermal conductivity test on the cutting fluid made from corn oil with $CaCO_3$ mass fraction variation.

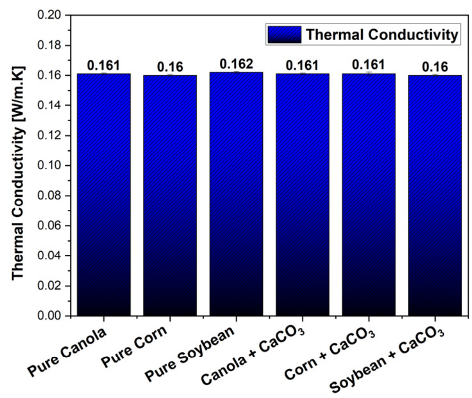

Fig 8. Thermal Conductivity of Cutting Fluid Sample

Fig. 8 shows the thermal conductivity of cutting fluid samples from various types of vegetable oil added with $CaCO_3$ nanoparticles. The obtained results indicate that the thermal conductivity of each sample experiences no significant increase or decrease. These results explain that the addition of $CaCO_3$ nanoparticles to the base fluid of vegetable oil does not significantly increase its thermal conductivity. These results are different from a study reporting that thermal conductivity increases due to the higher volume fraction variations [71].

Thermal conductivity is influenced by several parameters, including nanoparticle concentration, nanoparticle shape, nanoparticle thermal conductivity, base oil type, nanoparticle size, temperature, and the preparation technique [72,73]. Insignificant thermal conductivity obtained in this study may be induced by the nanoparticle concentration, which affects the Brownian motion force, which shows the random movement of the particles in the solution. Ideally, the addition of $CaCO_3$ nanoparticles should increase the thermal conductivity, but the excessive addition of nanoparticles exacerbates the Brownian force of the motion within the nanoparticles, whereas too minimum addition of nanoparticles produces no significant effects on the fluid's thermal conductivity. Therefore, the concentration of nanoparticles added to the base fluid must be thoroughly considered because it carries a significant effect on the thermal conductivity of the nanofluid.

3.2.3 Viscosity

Viscosity is the resistance to a fluid flow or can be referred to as a measure of the fluid's thickness. The analysis of the dynamic viscosity of the cutting fluid is highly crucial because it carries an important role in the nano lubricant's thermal properties. Besides, lubrication with appropriate thickness will form a strong film layer in the bearing gap during the machining process, which minimizes friction and wear.

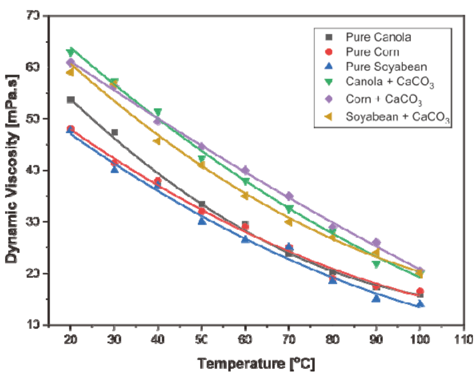

Fig 9. Dynamic Viscosity of Cutting Fluid Sample

Fig 9. shows the results of the dynamic viscosity test for each sample with various types of vegetable oil added with 0.15% $CaCO_3$ nanoparticles. The results indicate differences in dynamic viscosity from each sample, with the highest dynamic viscosity obtained for canola oil, followed by corn oil and soybean oil. Besides, the highest dynamic viscosity for each variation of vegetable oil is obtained due to the addition of 0.15% mass fraction of $CaCO_3$ nanoparticles.

The results also suggest a particular phenomenon that the cutting fluid sample has higher viscosity after the addition of nanoparticles. These results are in accordance with previous studies describing that the higher density of nanoparticles than lubricant increases its viscosity [74]. Additionally, these phenomena are also caused by the Van Der Walls force, which accelerates the viscosity of the lubricant [75,76]. A decrease in viscosity value on the cutting fluid sample has also been identified following increasing temperature. These results are in accordance with studies that explain that the viscosity decreases along with increasing temperature due to the weakened intermolecular interactions in the fluid [77].

The application of heat to the lubricant induces molecular energy gain, leading to continuous molecular motion, resulting in weakened intermolecular forces and a decrease in viscosity. This refers to various references that viscosity can be affected by several aspects, including the concentration of nanoparticles, size, and shape of nanoparticles, temperature, and shear rate [78]. The dynamic viscosity analysis on the cutting fluid samples is crucial as it presents a significant role in the nano lubricant's thermal properties. Additionally, appropriate lubrication with suitable viscosity will generate a strong film layer within the bearing clearance during the machining process, minimizing friction and wear.

3.3 Rheological Properties

The results of the comparison between the shear stress and the shear rate of each cutting fluid sample with various types of vegetable oil added with $CaCO_3$ nanoparticles at 40°C and 100°C are illustrated in Fig. 10 a) and b).

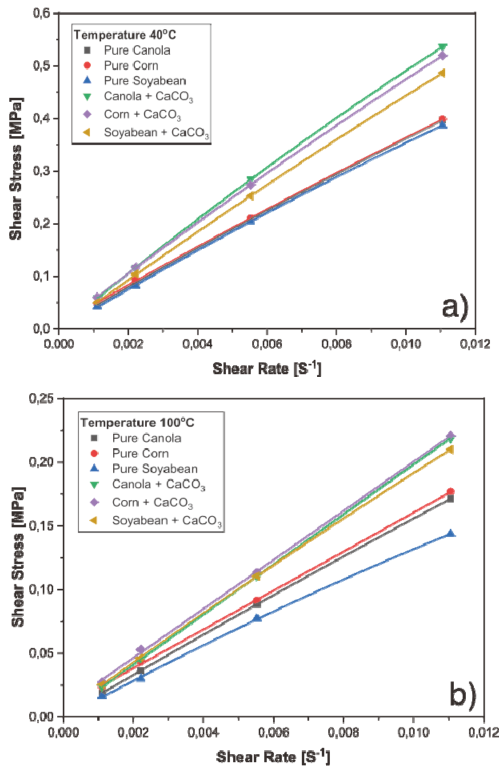

Fig 10. Rheological properties of Cutting Fluid Sample at a) 40°C and b) 100 °C

The results suggested a linear relationship between shear stress and shear rate from all cutting fluid samples. Therefore, the vegetable oil mixed with $CaCO_3$ nanoparticles presents Newtonian flow behavior [79]. Newtonian flow is vital for the thermal application of nanofluid, such as convective heat transfer.

In addition, the base oil of the cutting fluid sample has a lower shear rate value compared to the vegetable oil sample added with $CaCO_3$ nanoparticles. The highest shear stress value at 40°C is obtained from canola oil, while at 100°C, the highest shear stress value is from corn oil. Fig. 10 and Fig. 11 also illustrate the decrease of shear stress following the increase of temperature from 40°C to 100°C, showing a decline in Van Der Waals for–ces at high temperatures [80]. The nanoclusters derived from nanoparticle incorporation impede the relative movement of oil layers, exerting a more significant influence on viscosity at lower temperatures. This occurs due to an increase in temperature, facilitating particles to overcome the attractive Van der Waals forces [75].

3.4 Cutting Fluid Performance on CNC Milling Machining

3.4.1 Surface Roughness

Figure 11 shows the results of the workpiece Surface Rou–ghness (R_a) from the CNC Milling machining process.

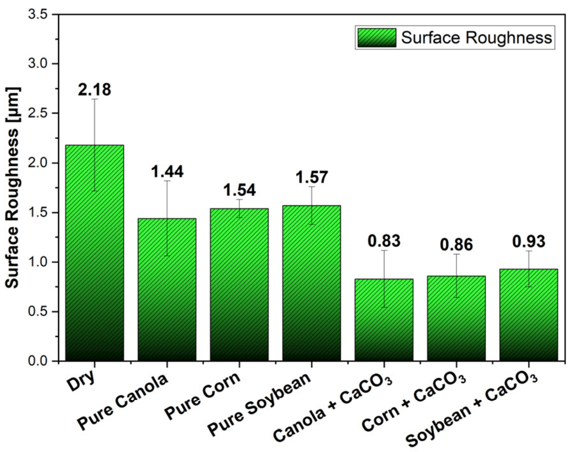

Fig 11. Surface Roughness Graph from Different Vegetable Oil and 0,15% $CaCO_3$

The results indicate different R_a performances from each sample, with the lowest R_a value (of 0.83 µm) found in the canola oil, followed by the corn oil (0.86 µm) and soybean oil (0.93 µm). In contrast, the highest Ra value is observed in dry use (2.18 µm). Further, the addition of $CaCO_3$ nanoparticles in vegetable oil for each sample also results in excellent performance, where the addition of 0.15% $CaCO_3$ significantly lowers the R_a value. These results are in accordance with the research reporting the superior performance of canola and corn oils in reducing surface roughness compared to soybean oil [81].

The lower R_a caused by the increase of nanoparticle concentration added into the cutting fluid is also affected by the rolling and sliding properties of the small nanoparticles, which aids its proper entrance into the cutting zone, forming a film layer to reduce contact between the tool and the workpiece [82,83].

Based on the results obtained, the role of $CaCO_3$ nanoparticles in this study has been analyzed and proven to reduce friction by decreasing the R_a value when the base oil is added with $CaCO_3$ nanoparticles. This can happen because it is related to the mechanism involved that the small nano-sized $CaCO_3$ particles have a spherical shape as shown in Fig. 4. and its good properties to form a mechanical shield will roll between the friction surfaces of the cutting tool and the workpiece and will play the role of repair effect and sanding effect to reduce the friction [84–86]. The mechanism is described in Fig. 12.

These findings are further reinforced by the imple–mentation of MQL during the liquid spraying process, wherein nanoparticles combined with base oil are atomized through a high-pressure nozzle, facilitating their incorporation into the interface between the cutting tool and workpiece. By filling the cavity of the interface between the cutting tool and the workpiece, the nano-sized particles can form a protective film with a repairing effect, while some particles can show a rolling effect for polishing, resulting in a smoother workpiece.

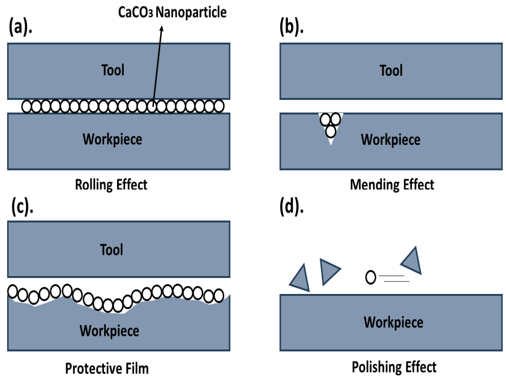

Fig 12. Lubrication mechanisms of nanoparticles effect, a). The rolling effect, b). Mending effect, c). Protective film, d). Polishing effect

3.4.2 Cutting Temperature

Fig 13. shows the results of the cutting temperature from each type of vegetable oil used as a cutting fluid in the CNC milling machining process. The measurement of cutting temperature was carried out five times, and the average was estimated, while each type of sample was measured three times to obtain accurate results.

As illustrated in Fig. 13, each vegetable oil presents a different performance in lowering the cutting tem–perature, with the highest cutting temperature obtained from dry conditions. Meanwhile, the utilization of soy–bean oil demonstrates superiority in temperature reduc–tion when compared to canola and corn oils. Besides, the presence of $CaCO_3$ nanoparticles in the base oil presents great performance against cutting temperature. These results are in accordance with research reporting that nanoparticles can form thin films on the friction between the cutting tool and the workpiece, along with the chips, providing a ball-bearing effect that can reduce the friction coefficient [87].

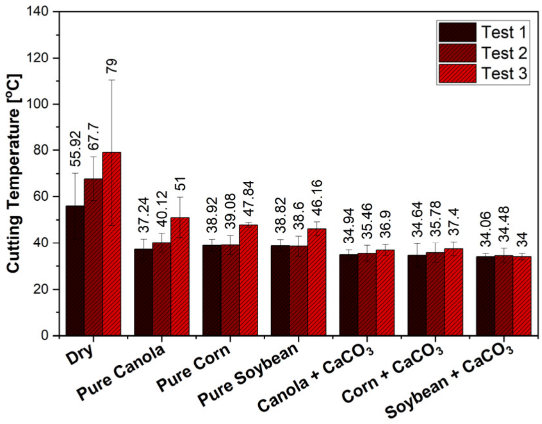

Fig 13. Cutting Fluid Sample Performance in Lowering Cutting Temperature

The decrease in cutting temperature has also been confirmed by the experimental results, which show the maximum recorded cutting temperature of 79.00°C for dry, 51.00°C for cutting fluid without nanoparticles, and 34.00°C for cutting fluid with the addition of $CaCO_3$ nanoparticles. This outcome may be attributed to the application of MQL, which efficiently removes hot chips from the cutting contact area by directly spraying cutting fluid with high-pressure air, leading to rapid dissipation of the generated heat. This strategy can effectively slow down the temperature increase, reducing tool wear and surface roughness [88].

3.4.3 Tool Wear Measurement

Tool wear is one of the most significant and necessary parameters for process planning and lifetime usage-related machining process costs [89]. In this study, we compared the endmill wear growth of the dry machine, refined vegetable oil, and nano-cutting fluid. Fig. 14 presents the results of three endmill wear tests which are selected to represent the three test iterations that have been performed.

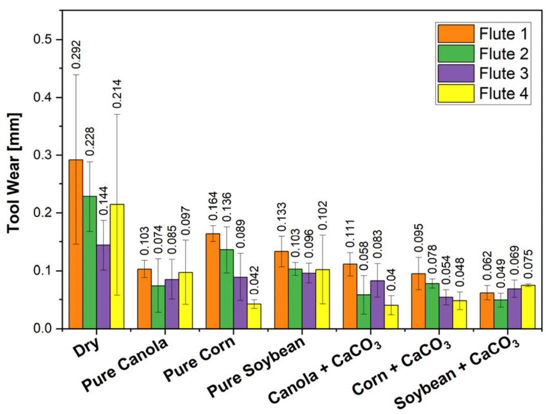

Fig 14. Endmill Tool Wear Measurement

The analysis results suggested that the highest endmill wear growth is observed during dry specimen machining. Meanwhile, MQL machining with pure vegetable oil coolant results in lower growth of the endmill than the wear dry specimen machining experiments. Besides, the use of nano-cutting fluid in the machining process generates the lowest reduction in tool wear.

The MQL machining using a nano-cutting fluid with several vegetable oils presents a very close and almost the same average tool wear pattern. The significant difference in average tool wear between the two machining conditions of pure vegetable oil and nano-cutting fluid will occur in a lengthy machining process [90]. These results also signify the great advantage of $CaCO_3$ addition into the cutting fluid in extending tool life. The superiorities of nano-cutting fluid are observable from its ability to maintain very thin film layers. Thus, when the endmill and specimen come into contact, it results in reduced wear.

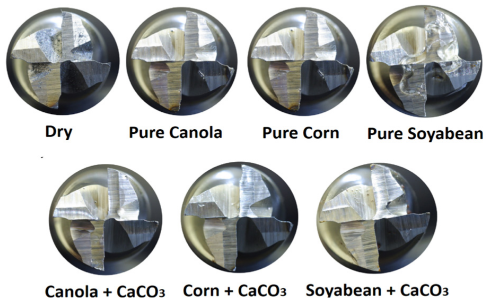

Fig 15. Endmill Tool Wear Macro Photo

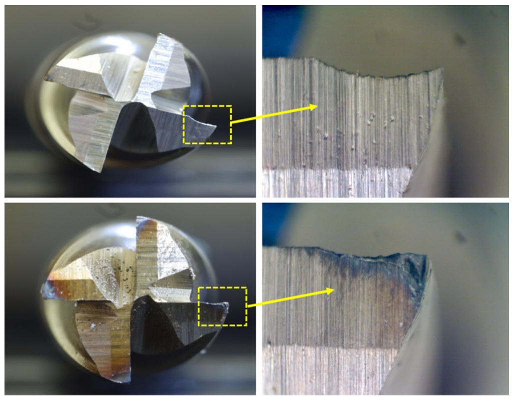

Fig 16. Examples of Endmill with Before and After Wear Incidents

The machining process that has been carried out on AISI 1045 steel (as a specimen) and an 8mm diameter HSS endmill (as a cutting tool) results in an endmill VBB (Average Width Of Flank Wear) wear calculation. According to ISO3685, its standard wear limit is 0.3 mm. Fig 15. shows the highest endmill wear from the dry machining process (0.246mm), followed by the canola oil (0.152mm), corn oil (0.188mm), and soya oil (146mm). Meanwhile, the nano-cutting fluid from canola oil has a wear value of 0.112mm, followed by corn oil (0.124mm) and soybean oil (0.77mm). Further, our analysis results also showed that nano-cutting fluid accelerates the service life of the endmill. Hence, the incorporation of $CaCO_3$ nanoparticles into a novel nano-cutting fluid formulation shows promising potential in extending the endmill's lifespan while minimizing friction and wear. In this case, the additive added to the base oil forms a tribofilm through a tribochemical reaction on the surface which reduces friction and wear as it maintains the tribofilm layer [91]. Fig 16. Illustrates the detail of endmill wear before and after the wear on the flank areas.

Based on the tool wear calculation results obtained, in the CNC Milling machining process with Dry and Pure conditions, vegetable oil tends to have higher wear and can be significantly reduced with the addition of $CaCO_3$ nanoparticles. This phenomenon can be obse–rved in Fig. 17.

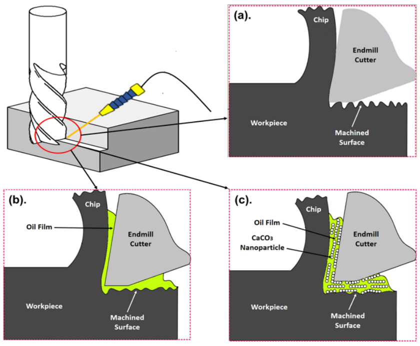

Fig 17. Milling schematic under the sample cutting fluid, (a). Dry Condition, (b). MQL + Pure Vegetable Oil Condition, (c). MQL + Vegetable oil with CaCO3 Nanoparticle

That in dry conditions the workpiece and endmill will rub directly without any cooling medium, so the heat generated will be very high and with it the tool wear becomes high, while the use of pure vegetable oil as cutting fluid can reduce wear due to the lubricating oil layer formed in the cutting zone to dissipate heat effectively and can reduce the intensity of friction that occurs, while in the condition of cutting fluid mixed with $CaCO_3$ nanoparticles obtained the minimum wear value among other conditions. This can occur because in the lubricating oil layer formed in the cutting zone there are small particles of $CaCO_3$ which show excellent anti-friction and load-bearing between the lubricating layers, in addition, $CaCO_3$ nanoparticles can play a role in forming a protective film that rolls between the friction that occurs to reduce friction and increase convective heat transfer, and with that, the heat formed will be able to be disposed of optimally and tool wear will decrease as heat decreases [90,91].

3.4.4 Morphology of Chip Surface

Fig 18. shows normal, generalized, and enlarged SEM results for the collected chips after machining trials using dry media and MQL. The chip variations are obtained from the dry process, as well as the machining using pure canola oil, canola oil + 0.15% $CaCO_3$, pure corn oil, corn oil + 0.15% $CaCO_3$, pure soybean oil, and soybean oil + 0.15% $CaCO_3$. The comparison of the different lubrication methods suggests significantly different chip surfaces on the above areas (dry media).

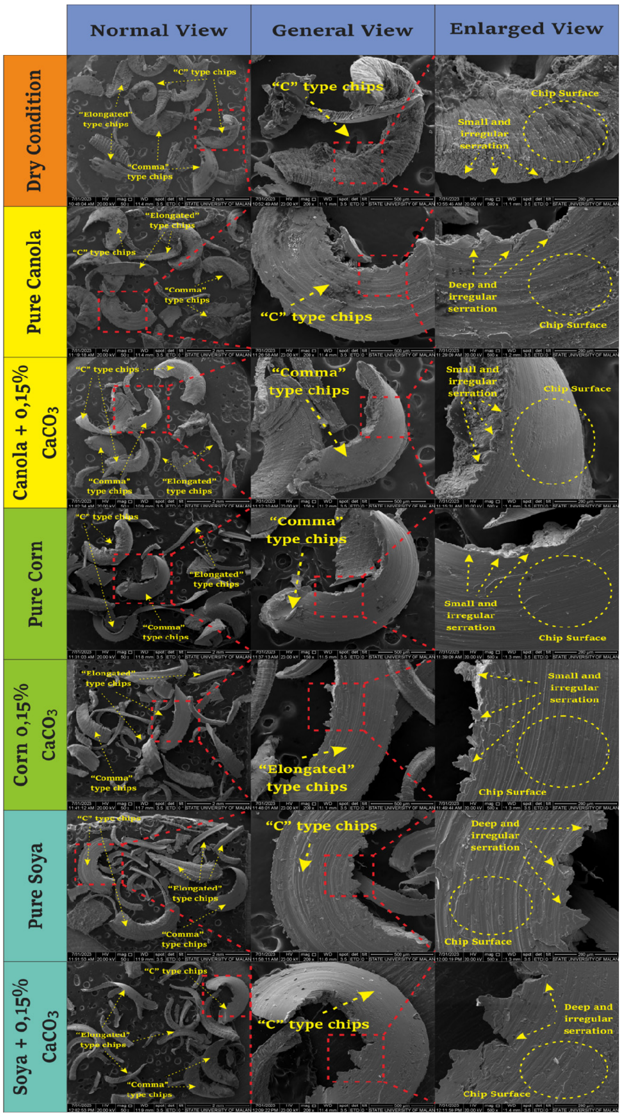

Fig 18. Chip Morphology in CNC Milling Process

Fig 19. Chip Color in CNC Milling Process

Among various types of chip types, type C and type comma, with elongation shape, are the most common forms of chip. Further, the change in the chip's con–dition arises from one surface of the chip infiltrating the other surface, leading to the fragmentation of particles into slender or curved shapes, resulting in debris formation [93].

In addition, the chip surface on dry media appears to be rougher than from other media. In contrast, the MQL presents the opposite results. Thus, the MQL method greatly influences the chip surface structure. Mean–while, in terms of chip serration formation, dry media, and vegetable oil lubrication media present similar results of irregular serrations. The difference lies in pure oil and oil addition of nanoparticles. The results sug–gested that vegetable oil causes deep serrations, while the oils with nanoparticles generate small serrations. These results are consistent with studies reporting that MQL is more effective in reducing dentition, improving surface quality, and reducing tool wear [94,95].

Fig. 18. also shows that the chips formed have a surface morphology with fewer scratches and a smoot–her surface as $CaCO_3$ nanoparticles are added. This phenomenon reveals that the presence of $CaCO_3$ nano–particles added to the vegetable oil base fluid can form a good protective layer when the endmill scratches to form chips. The $CaCO_3$ nanoparticles act as a protective barrier to hold the chip against the high friction and shear forces at the interface of the workpiece and cutting tool, dramatically reducing the rubbing contact area and the resulting chip has a smooth surface. In contrast to dry conditions and in the absence of nano–particles in the lubrication observed to have a coarser chip morphology. When chips are formed in the absence of a protective layer of nanoparticles, it results in higher contact stress and shear stress, consequently, linear scratches are formed along the chip exit direction [96]. In the absence of the protective layer of nanoparticles, the back of the chip has many solid lines caused by high plastic shear deformation.

Between all samples, the lubrication using canola oil and corn oil causes small serration, while soybean oil causes deep serration. Yuan et al. [93] described that the formation of wear particles or debris may be caused by various factors, including rubbing, fatigue, cutting, and severe adhesion between the workpieces. The different wear levels and chip shapes signify that determining the features of similar particles produced by machines, services, and conditions is challenging [93].

3.4.5 Chip Color

Fig. 19. presents a photographic macrograph of chips collected at various stages of tool wear using a CNC Milling machine. The obtained chip color is dark pur–plish, which changes to dark brown and light brown, then to silvery white due to wear and tear on the different sides of the chisel. The results suggested that the chips obtained from lubricants with the addition of nanoparticles are much brighter than pure lubricants and dry media. The results also show that the changes in the chip color and shape depend on the cutting conditions [97].

The chip formation process is very important for changing phenomena such as force and temperature during the cutting process. Chip color can be used as a medium to observe the frictional properties that occur between the workpiece and the cutting tool [84]. The chip color results correlate with the chip morphology results in dry and pure vegetable oil conditions, where the chips produced appear darker in color compared to the cutting fluid added with $CaCO_3$ nanoparticles, it indicates that the machining conditions using cutting fluid with the addition of $CaCO_3$ nanoparticles are more effective in reducing the cutting temperature, so that it will have a positive impact on the contact area to reduce friction, due to the role of $CaCO_3$ nanoparticles that can roll to form a rolling effect in the cutting zone. When forming chips in which there is heat due to friction, it can be effectively discharged through a thin film that can be formed through CaCO3 nanoparticles, resulting in a brighter chip color [98].

In this study, the correlation between chip surface chromaticity and tool wear has been investigated by analyzing the color of the chip, as suggested by a study that formulates a system for assessing and predicting tool wear by observing chip color [98]. Currently, the prediction of cutting tool life is measured and predicted indirectly using vibration and current. In this research, chip color change is used to predict tool wear.

4. CONCLUSION

The morphology of $CaCO_3$ from the scallop shell has a spherical form and irregular size, with agglomeration due to the synthesis process. The $CaCO_3$ particles have a single-phase calcite and trigonal (hexagonal axes) crystal form with a size of 52.18 nm. The $CaCO_3$ functional group is located at the absorption peaks 1398.39, 881.46, and 713.66 cm^{-1}.

Based on the results of thermophysical property analysis on the cutting fluid samples, the highest density values were obtained from canola, soybean, and corn oils. Further, the density increases following the addition of nanoparticles. The addition of $CaCO_3$ additive to base oil results in poor stability which affects the non-significant increase of thermal conductivity. The highest dynamic viscosity value is obtained from canola oil, while the lowest is from soybean oil. In addition, the samples' viscosity increases following the addition of nanoparticles, while the dynamic viscosity value decreases as the temperature increases.

Meanwhile, the rheological properties analysis showed Newtonian flow from all cutting fluid samples. Besides, the base oil used as the cutting fluid sample has a lower shear rate compared to the vegetable oil sample mixed with $CaCO_3$ nanoparticles.

For tribological properties, canola, and corn oil present a better performance in obtaining the minimum R_a value, but soybean oil is more effective in reducing cutting temperature. Based on the results of tool wear calculations, each sample has the best performance in reducing wear, with $CaCO_3$ nanoparticles addition presenting the best performance in reducing wear. For chip formation, on average, the samples produce irregular serration morphology with C-type, comma, and elongation shapes. Meanwhile, the obtained chip color is dark purplish, which changes to dark brown and light brown, then silvery white due to wear and tear on the different sides of the chisel.

The results of this study show that the performance of $CaCO_3$ nanoparticles in nano-cutting fluid can obtain quite optimal results for the performance of CNC milling machining processes in reducing friction and wear. This research is expected to make a significant contribution to helping the manufacturing industry develop more effective cooling solutions in the future. These results can also help in creating nano-cutting fluid with better performance and lower production cost and in the future can realize green manufacturing that is more environmentally friendly.

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NOMENCLATURE

R_c	vessel radius (cm)
R_b	shaft radius (spindle) (cm)
x	radius shear rate is being calculated (cm)
K	constant (0.9)
d	crystallite size (nm)
R_a	surface roughness

Greek symbols

γ	Shear rate (/s)
ω	Shaft angular speed (*spindle*) (rad/sec)
λ	X-Ray wavelength (1.5406 Å)
β	Full-width half maximum (2θ)
θ	Bragg's angle (^{o})

ACRONYMS AND ABBREVIATIONS

$CaCO_3$	Calcium carbonate
Al_2O_3	Aluminum oxide
SiO_2	Silicon dioxide
MoS_2	Molybdenum disulfide
CuO	Copper oxide
MQL	Minimum Quantity Lubrication
NMQL	Nanofluid Minimum Quantity Lubrication
CNC	Computer Numerically Control
HSS	High-speed steel
XRD	X-ray diffraction
FTIR	Fourier transform infrared
SEM	Scanning Electron Microscopy
VBB	Average Width of Flank Wear

ЕКСПЕРИМЕНТАЛНА ПРОЦЕНА БИОМАЗИВА СА ДОДАТКОМ НАНОЧЕСТИЦА КАЛЦИЈУМ КАРБОНАТА ($CACO_3$) ИЗ ОТПАДА ЉУСКЕ КАПИЦЕ КАО ТЕЧНОСТИ ЗА СЕЧЕЊЕ КОРИШЋЕЊЕМ МИНИМАЛНЕ КОЛИЧИНЕ ПОДМАЗИВАЊА (МКЛ) У CNC ПРОЦЕСУ ГЛОДАЊА

Течност за нано резање прскана методом минималне количине мазива (МКЛ) је један пример зеленог производног процеса. У међувремену, биљно уље је одговарајуће базно уље за подмазивање јер нуди веома високе перформансе подмазивања и еколошку прихватљивост. Даље, наночестице $CaCO_3$ су популарне због свог капацитета да побољшају својства подмазивања и перформансе. Међутим, оптималан утицај употребе различитих врста биљног уља остаје неадекватно истражен. Стога, ова студија има за циљ да анализира ефекат наночестица $CaCO_3$ на перформансе течности за сечење, посебно на термофизичка, реолошка и триболошка својства у CNC процесу глодања материјала AISI 1045 челика. Течност за нанорезивање је припремљена коришћењем различитих биљних уља (канола, кукурузно, сојино) са додатком наночестица ЦаЦО3 масене концентрације од 0,15%. Резултати су показали да су термофизичка својства, укључујући густину и вискозност, највећа када се користи уље каноле, а додавање $CaCO_3$ свим узорцима није значајно утицало на топлотну проводљивост. У међувремену, за реолошка својства, посматрали смо Њутнов за све узорке течности за сечење. Што се тиче триболошких својстава, уље каноле и кукуруза су били бољи за добијање минималне вредности Ра, док је сојино уље било ефикасније у смањењу температуре резања. На основу резултата прорачуна хабања алата, свако уље представља најбоље перформансе у смањењу хабања, посебно уз додатак $CaCO_3$. За формирање струготине, у просеку, узорци производе неправилну морфологију зуба са Ц-типом, зарезом и обликом издужења. У међувремену, резултујућа боја струготине је била тамнољубичаста, која се променила у тамно браон и светло браон, а затим је постала сребрно бела због хабања на различитим странама длета.