

# Influence of additive nano calcium carbonate (CaCO<sub>3</sub>) on SAE 10W-30 engine oil: A study on thermophysical, rheological and performance

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#### Highlights:

- Nano lubricants with calcium carbonate (CaCO<sub>3</sub>) nanoparticles significantly improved wear and friction properties.
- A 0.1 wt% concentration of CaCO<sub>3</sub> nanoparticles provided the best wear reduction.
- The nano lubricants showed better thermal conductivity, density, and viscosity.

## Abstract

Researchers have used nanomaterials as additives in base oil to improve its specifications, especially to minimize wear and friction during its applications. In this study, calcium carbonate  $(CaCO_3)$  nanoparticles were selected as an additive to serve as a protective layer between components and anti-wear properties. In this study, calcium carbonate (CaCO<sub>3</sub>) nanoparticles were selected as an additive to serve as a protective layer between components and anti-wear properties. Nano lubricant samples were prepared using mass variations of CaCO<sub>3</sub> and SAE 10W-30 base oil with concentrations of 0.05, 0.1, 0.15, and 0.2%, then homogenized. The nanolubricant samples obtained were analyzed for thermophysical, rheological properties and lubricant performance with the addition of nano CaCO<sub>3</sub> in improving the wear resistance of FC25 cast iron. The results of thermophysical and rheological properties analysis suggest that the nanolubricant has better tribological properties compared to base lubricants. The highest values of thermal conductivity, density, and viscosity (40 °C) are 0.139 W/m.K, 812.203 kg/m<sup>3</sup>, and 106 mPa.s (40 °C). Meanwhile, the highest CoF, disc mass loss, and surface roughness of nanolubricant are 0.0706, 0.0037 grams, and 0.50 µm, respectively. These results indicate that the greatest wear-reducing agent is from the nanolubricant with the addition of CaCO<sub>3</sub> nanopowder additives at 0.1 wt% concentration. These results are expected significant insights into the advancement of nano technology-based lubricants in the future.

Keywords: Nanolubricant; Calcium carbonate; Thermophysical; Rheological; Wear

# **1. Introduction**

Transportation is the main need in Indonesia as it supports daily activities. To address this demand, the automotive industry annually escalates production by 10% [1]. Consequently, lubricant has become a common need in the automotive industry since it significantly lowers friction and wear on essential components, ensuring maximum machine performance [2]. Friction and wear are the two central problems on some or all parts of the contacting surfaces, resulting in

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Universitas Muhammadiyah Magelang excessive energy loss [3]. Excellent lubrication reduces friction and energy loss by lowering the direct contact between the contacting surface [4]. In engines, lubricants function as vibration dampers, coolants, and dirt-transporting agents in internal combustion engines [5]. The improvement in the lubricant's performance and quality can be attained through the addition of additives [3].

Globally, lubricants contain less than 85% of base material in the form of petroleum [6]. The petroleum undergoes additional refining processes to yield mineral oil, which serves as the primary component for manufacturing lubricants. Generally, lubricants contain 90% of base oil, while the remaining 10% is in the form of additives [7]. The progression of lubricant involves the addition of an additive (nanoparticle) thus, its result is named nanolubricant. Nanolubricants have different physical and tribological properties that affect their performance compared to base oils [8]. Nanolubricant quality is influenced by several parameters, such as the variations in nanoparticle volume, shape and dimensions of nanoparticles, basic oil, as well concentration of nanoparticles [9]. The addition of nanoparticles accelerates the quality of basic oil, resulting in greater thermophysical properties.

In this study, nanoparticles CaCO<sub>3</sub> which originated from synthesized scallop shells in nanosized powders were chosen as an additive because they have anti-wear and friction-reducing properties that are rarely known to many researchers [10], [11] and have many polymorphic structures that can be applied to industrial products [12]. The finished calcium carbonate nanoparticles have been added as an additive to 10W-30 base oil, the choice of base oil is because the viscosity of 10W-30 is widely used as a motor vehicle lubricant in tropical countries, especially Indonesia. As a lubricant additive, calcium carbonate reduces carbon deposition carbon and sulfur, which serve as the cleaning agent in the piston rings, pistons, and cylinders. The dispersed carbonate calcium nanoparticle on the basic oil forms the protective tribofilm that coats the contacting surfaces to lower the wear and enhance the performance of the lubrication [13]. The calcium carbonates contained in the oil lubricant maintain the required total base number (TBN) in the lubricant.

However, the long-term use of nanolubricant may result in stability issues on the nanolubricant due to the agglomeration induced by the high surface activities [2]. This occurrence inhibits the nanoparticle transfer from the base oil into the contacting surface, which forms the protecting layer. Consequently, to lower the agglomeration on nanolubricant, the dissolved particle concentration can be regulated [14]. In this study, the prepared nanolubricant sample was applied to the FC25 iron cast to examine its wear level. The FC25 iron cast is frequently used on the cylinder liner [9]. A cylinder liner is a crucial component of an engine as it facilitates piston movement, resulting in reciprocating piston movement [15], minimizing material damage, lowering fuel consumption, and elongating the component life of excellent lubrication [16].

Therefore, this study aims to identify the characteristics of CaCO<sub>3</sub> nanoparticles synthesized from the scallop shell, specifically for the additive on the SAE 10W-30 lubricant. The thermophysical and rheological properties of prepared nanolubricant with variations of CaCO3 nanoparticle addition (0.05%, 0.1%, 0.15%, dan 0.2% wt) were examined. The SAE 10W-30 added with CaCO<sub>3</sub> nanoparticle variations was applied on the FC25 iron cast to identify its wear resistance using the pin-on-disc machine.

### 2. Methods

#### 2.1. Material

This study used SAE 10W-30 lubricant and calcium carbonate powder obtained from synthesized scallop shells. The details of the materials being used are summarized in Table 1. The properties of the SAE 10W-30 lubricant brand Shell Advance brand are listed in Table 2. Meanwhile, the material used for the wear and surface roughness test was the FC25 iron cast. The information related to the materials being used in this study is briefly described in Table 3.

Table 1. The material used for nanolubricant preparation

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Table 2.		Paramete	ers			Value	es				
<b>Properties of lubricant</b>		SAE viscosity	grade			10W-30					
10W-30 [17]		Density at 15 °C	C (kg/m³)			881					
	Kir	Kinematic viscosity at 40 °C (cSt)					75.1				
	Kin	Kinematic viscosity at 100 °C (cSt)					10W-30      881        75.1      11.5        6700      147        S Fe				
	Dyn	amic viscosity at	-25 °C (mPa.s)		6700						
		Viscosity in	ndex			147	,				
		•									
Table 3.	Constitution of	Composition (%)									
Chemical compositions	Conditions	С	Mn	Si	Р	S	Fe				
of grey cast iron FC25	Min	2.44	0.39	1.83	0.15	-	Delenee				
[18]	Max	3.02	0.52	2.03	0.30	0.15	Balance				

## 2.2. Nanolubricant Preparation

The nanolubricant samples were prepared using variations of CaCO<sub>3</sub> mass and SAE 10W-30 basic lubricant with concentrations of 0.05, 0.1, 0.15, and 0.2%. The use of these variations refers to the research of Jose, et al. that adding CaCO<sub>3</sub> to PAO8 oil can reduce the acidity between up to 60% [19]. so that these variations are used in this study to reduce the wear value to the maximum. The procedures for nanolubricant preparation are illustrated in Figure 1. The comprehensive information on the samples employed can be found in Table 4. Following several calculations, 200 ml of basic lubricant was prepared. The CaCO<sub>3</sub> nanoparticle powder at mass variations of 0.05, 0.1, 0.15, and 0.2% was prepared. Then, each sample was stirred using a magnetic stirrer at 1250 rpm speed for 20 minutes at room temperature. Further, a sonication process was carried out using an ultrasonic homogenizer for 30 minutes to obtain a homogeneous mixture of CaCO<sub>3</sub> and SAE 10W-30 lubricant. Homogenization aims to homogenize the particle size in order to maintain the stability of the mixture [20]. After this process, the prepared nanolubricant samples were further examined.



## 2.3. Material Characterization

The characteristics of CaCO<sub>3</sub> nanoparticles originating from scallop shell waste were analyzed using X-ray Diffraction (XRD) type E'xpret Pro, specifically to identify its crystal structure, crystallite size, and phase. Besides, the samples were also characterized using Scanning Electron Microscopy (SEM) type Inspect-S50 for the identification of the CaCO<sub>3</sub> nanoparticle's morphology. Another characterization using Fourier Transform Infra-Red (FTIR) type Irprestige 21 was also performed to identify the compounds, functional groups, and impurities within the CaCO<sub>3</sub> nanoparticle powder from the scallop shell waste.

### 2.4. Thermophysical Properties Test

The thermophysical test was conducted to determine the thermal conductivity of the samples using the thermal properties analyzer KD2 Pro illustrated in Figure 2a. In specific, this test was carried out to identify the heat transfer value through conduction. The viscosity of the nanolubricant was tested using the NDJ-8S Viscometer illustrated in Figure 2b. Meanwhile, the den-



sity was tested using an analytical digital balance by dividing the mass by its volume to determine the density of the nanolubricant. Further, sedimentation was after assessed the nanolubricant solution was set aside for 30 days. The sedimentation results were photographed to examine the precipitation the in solution.

## 2.5. Rheology Characterization

The rheological properties are employed to ascertain the flow of the nanolubricant, investigating the correlation between shear rate and stress. The shear rate and shear stress analysis required the viscosity results. in rheology testing of lubricants conducted at temperatures of 40 °C and 100 °C [21]. The shear rate was calculated using Eq. (1) [22], while the shear stress calculation was performed using Eq. (1).

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

in which  $\gamma$  means the shear rate (1/s),  $\omega$  is the angular velocity (rad/sec), Rc is the contained radius (cm), Rb represents the rotor radius (cm), X represents the radius at which the shear rate was computed (cm). Meanwhile, to calculate the shear stress value, we can use Eq. (2) [22].

$$\tau = \mu \times \gamma \tag{2}$$

where  $\tau$  is the shear stress (mPa.s),  $\mu$  is the dynamic viscosity (kg/m.s),  $\gamma$  is the shear rate (1/s).

### 2.6. Experimental Setup

Table 5.	Category	Materials	Hardness (HV)
Specification materials	Pin Stainless Steel		265.1
of pin on disc test	Disc	Grey Cast Iron FC25	194.7
Table 6.	Parameters		Value
rameters of pin on disc	Force (N)		20
testing [23]	Sliding Speed (m/s)		0.1
	Sliding Distance (m)		1000
	Track Diame	30	
	Temperat	23	
	Pin Diamet	6	
	Disc Diame	60	



The performance of the nanolubricant with CaCO3 nanopowder additive was analyzed using a wear test through a pin on disc tribometer from Ducom Instruments. The materials used in this wear test are shown in Table 5 obtained from the test results in the mechanical engineering laboratory. The use of stainless-steel material for the pin was assumed to be the material for the piston rings. Meanwhile, the use of FC25 gray cast iron material was assumed to be in accordance with the material in the cylinder liner on the engine.

The wear test was conducted following the ASTM G99 standards, and referring to the research of Kurre, et al. with parameters summarized in Table 6. Meanwhile, the pin on disc test is illustrated in Figure 3. Besides, the surface structure of the obtained pin-on-disc test was observed through the photo taken from a microscope.

Paramete

Figure 3.

disc test

Schematic of pin on

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## **3.** Results and Discussion

### 3.1. Additive Characterization

Scanning Electron Microscopy (SEM) was used to examine the morphology of CaCO<sub>3</sub> powder from the synthesized scallop shell's waste. The SEM results are illustrated in Figure 4, suggesting the spherical shape and uniform size of the calcium carbonate synthesized from the scallop's shell [24]–[26]. The results also indicate the presence of agglomeration caused by numerous factors, such as sintering temperature, sintering time, uneven demolition, and the period of demolition [26]. For resolving the agglomeration on the calcium carbonate added into the basic lubricant, further processes using the magnetic stirrer and sonication are required to produce a more stable nanolubricant [27].



Figure 4. Scanning electron microscopy of calcium carbonate additive, (a) Magnification 50.000x, (b) Magnification 100.000x

X-ray diffraction (XRD) characterization was performed to identify the properties of  $CaCO_3$  material synthesized from the scallop shell waste, including its phase, crystal structure, and crystallite size [28]. The crystallite size of  $CaCO_3$  material synthesized from the scallop shell waste was calculated using the Scherrer formula [29], with Eq. (3).

$$d = \frac{K\lambda}{\beta\cos\theta}$$
(3)

Where, d: Crystallite diameter; K: 0.9;  $\lambda$ : Wavelength = 1.5406 Å. and  $\beta$  : Full-Width Half Maximum (FWHM).



The crystallite size of CaCO<sub>3</sub> powder is 52.18 nm. Additionally, according to the XRD results illustrated in Figure 5, the scallop shell's powder sample has a single phase with calcite symmetry crystals indicated from the highest peak of 1194.07 at  $2\theta$ =29.4554° and trigonal crystal (hexagonal axes) [24].

Through the FTIR analysis, the functional group of calcium carbonate was examined. The FTIR results for CaCO<sub>3</sub> are presented in Figure 6, showing a sharp peak at 2916.36 cm<sup>-1</sup> and 2848.86 cm<sup>-1</sup>, indicating C-H bending vibration due to the

Figure 5. XRD analysis of calcium carbonate



organic layer from the amino acid from the scallop shell [30]. Besides, the absorption peak at 2511.31 cm<sup>-1</sup> shows the C-H stretching vibration, suggesting the existence of different calcium carbonate bonds [31], [32]. Meanwhile, the peak at 1398.39 cm-1 represents the characteristics of calcium carbonate of scallop shell [33]. The absorption peak of pure calcium carbonate was detected at 881.46 cm<sup>-1</sup> and 713.66 cm<sup>-1</sup>, suggesting its calcite characteristic [33], [34].

### 3.2. Thermophysical Properties of Nanolubricant

#### 3.2.1. Thermal Conductivity of Nanolubricant

Thermal conductivity represents the heat transfer ability of a material. The thermal conductivity of nano-sized particles presents a significant influence on the nanolubricant conductivity [35]. Figure 7 presents the thermal conductivity of the base oil and the nanolubricant with the addition of a CaCO<sub>3</sub> nanoparticle. The obtained data suggested 0.137 W/m.K and 0.139 W/m.K thermal conductivity of the base oil and the lubricant added with the CaCO<sub>3</sub>, respectively.



This increase in thermal conductivity is caused by the addition of inorganic nanomaterial [36], [37]. Further, the different addition of CaCO<sub>3</sub> nanoparticles shows no significantly different increase in thermal conductivity due to a number including of factors, the nonhomogeneous particle size [38], change of temperature [39], particle size of CaCO<sub>3</sub> synthesized from the shell waste [40], and nanoparticle conductivity [41]. In addition, the addition of nanoparticles will produce brownian motion that can increase the heat transfer rate of the nanolubricant [42].

We identified different densities from each sample of nanolubricant. The increase in density is directly proportional composition the higher of to nanoparticles [43]. Figure 8 also presents the results of the density test from the basic lubricant and nanolubricant with variations of CaCO<sub>3</sub> nanoparticles. The highest density of 823.888 kg/m<sup>3</sup> is observed from the nanolubricant with a 0.2% addition of CaCO<sub>3</sub>. In contrast, the lowest density of 812.203 kg/m<sup>3</sup> is from the basic oil. The nanoparticle addition on base oil is directly proportional to the higher density [44], [45].

conductivity of nanolubricant







Figure 9 shows the results of viscosity examination from base lubricant and nanolubricant with variation addition of CaCO<sub>3</sub>. Figure 9 shows the highest viscosity from the nanolubricant with a 0.2% addition of CaCO<sub>3</sub> in comparison to the base oil. At 40 °C and 100 °C temperatures, the sample has 106 and 36.5 mPa.s, density. This finding signifies that high temperature correlates with low viscosity, which heads closer to the base oil [46] due to the lower Van Der Waals force among the molecules [47]. This viscosity value further impacts the rheology and nanolubricant features [48].

#### 3.2.4. Sedimentation of Nanolubricant

The nanolubricant stability was observed using the sedimentation method. The nano lubricant's stability highly relies on the features of suspended particles and base oil [49]. Figure 10 suggests the results of observation on the base oils and lubricants sedimentation at observation periods of 0, 10, 20, and 30 days. The results obtained showed that samples (b-e) had sedimentation on 10 days, as shown by the white sediment on the base oil. Further, the same results were also identified on the 20 and 30 days of sedimentation. Several factors that affect the nano lubricants' stability include the nanoparticles concentration, use of surfactant, types of nanolubricants can be caused by gravity when nanoparticles are mixed in the base oil. Safril, et.al. also showed that the first day there was no sedimentation and after 30 days sedimentation occurred in a limited manner [51].



Figure 10. Visual observation of nanolubricant sedimentation

## 3.3. Rheological Behavior of Nanolubricant

The rheological property on nanolubricant represents the correlation between shear rate and shear stress. The calculation of rheology properties relies heavily on viscosity. The obtained rheology properties are presented in Figure 11, showing the comparison between the shear rate and shear stress on the base lubricant and nanolubricant added with CaCO<sub>3</sub> nanoparticle. At 40 °C and 100 °C temperature, a linear correlation between the shear rate and shear stress on SAE 10W-

30 and nanolubricant is observed. Thus, the SAE 10W-30 and nanolubricant with  $CaCO_3$  nanoparticles have Newtonian flow [52]. Further, at 30 °C, the nanolubricant with nanoparticles experiences slight curve transformation, which is caused by the different structure of the base lubricant due to its broken molecule, resulting in the nanoparticle as the interface correlating the layers of oil [53].



## 3.4. Evaluation Performance of Nanolubricant

#### 3.4.1. Wear and Friction

Figure 12 presents the effect of CaCO<sub>3</sub> lubricant addition on the coefficient of friction (COF) and mass loss. The results of the COF test are shown in Figure 12a, signifying the maximum results



of 0.117 obtained from nanolubricant with 0.2 wt% of CaCO<sub>3</sub> that enhance the COF of the base lubricant. Meanwhile, the lowest COF (0.0706) was garnered from the nanolubricant with the addition of 0.1 wt% CaCO<sub>3</sub>. The decrease in the COF value is due to the higher viscosity of the nanolubricant, along with the addition of nanoparticles that forms a thin layer of tribofilm [54]. The decrease of COF is only found on the sample with 0.1 wt% of nanoparticle since the nanoparticle fills the gap between the surface maximumly, lowering the friction [55]. Figure 12b shows the mass loss from the disc after the pin-on-disc test. These results indicate that the highest mass loss (0.011 gram) is in the base lubricant. While the lowest mass loss occurs in nanolubricant (0.0037 gram) is found on the nanolubricant with the addition of 0.1 wt%. In summary, the mass loss relies on the COF, with the lower the COF reduces the mass loss value and vice versa. The lower coefficient of friction is caused by the addition of nanoparticles to the base lubricant [56].

Figure 12. Nanolubricant test result on: (a) Coefficient of friction; (b) Mass loss

#### 3.4.2. Surface Roughness

**Figure 13** presents the results of the surface roughness test from the FC25 iron cast after the pinon-disc test on the wear track area. The results showed that the highest surface roughness (1.42  $\mu$ m) is observed on the base oil sample. Meanwhile, the lowest roughness occurs in nanolubricant sample with the addition of 0.1 wt% CaCO<sub>3</sub>. The nanoparticles' addition into base oil can reduce friction effectively, in comparison to the use of base oil without addition of the nanoparticles [57].



Figure 13. Surface roughness for all nanolubricant samples However, the excessive addition of nanoparticles exceeding the maximum limit also decreases the wear rate caused by the formation of aggregate [58].

## 3.5. Analysis of Worn Surface

#### 3.5.1. Macro Structure

The worn disc surface was analyzed through macro photos using an optical microscope to identify its type of wear after the pin ondisc test. Figure 14a shows the morphology of the disc surface from

wear testing in dry conditions with delamination wear and parallel grooves in the direction of friction [59]. Figure 14b-Figure 14f shows the surface morphology with base lubricants and nanolubricant with the addition of CaCO<sub>3</sub> nanoparticle mass variations. The results suggest there are precise and similar grooves in the direction of friction, thus indicating wear of the abrasive and adhesive [54]. Additionally, the obtained surface morphology from basic lubricants and nanolubricant is smoother compared to dry conditions without lubricants. This occurrence is caused by the smoothing phenomenon [60].



Figure 14. Macrostructure of grey cast iron FC25: (a) Dry condition; (b) Base oil A0; (c) Nanolubricant A005; (d) Nanolubricant A010; (e) Nanolubricant A015; (f) Nanolubricant A020



## 3.5.2. Morphology of Grey Cast Iron FC25

In addition to the macrostructure test, the worn surface of the disc was also analyzed through the microstructure using scanning electron microscopy (SEM). This analysis aims to determine the surface morphology of the disc for more apparent results at a certain magnification. The test results are shown in **Table 7** showing that in dry conditions without lubrication, the wear on discs exhibits significant delamination wear, which steadily increases throughout the test duration [61]. The use of SAE 10W-30 base oil reduces wear on the material as there is less direct rubbing of surfaces [62]. Meanwhile, the addition of CaCO<sub>3</sub> additive at a concentration of 0.05 wt% and 0.1 wt% in base oil has better wear reduction results compared to base oil. Meanwhile, the addition of CaCO<sub>3</sub> additive at 0.15 wt% and 0.2 wt% concentration exhibit higher wear result compared to the base lubricant.

## **4.** Conclusion

According to the analysis of the performance of nanolubricant with CaCO<sub>3</sub> nanoparticle addition at mass variations of 0.05, 0.15, and 0.2 wt% to enhance wear resistance in FC25 cast iron material, several conclusions have been made. The surface morphology of CaCO<sub>3</sub> powder has a round shape and CaCO<sub>3</sub> particles have a size of 52.18 nm. The addition of CaCO<sub>3</sub> nanoparticles to the base lubricant can increase the wear resistance value as evidenced by the test results. The addition of CaCO<sub>3</sub> nanoparticles in base oils can have a good impact on thermophysical properties as evidenced by the test results of thermal conductivity, density, and viscosity which are increasing. The addition of CaCO<sub>3</sub> to the base oil also results in a linear flow between the shear rate and shear stress, corresponding with the Newtonian flow. In the nanolubricant performance test, the best COF, mass loss, and surface roughness values of 0.0706, 0.0037 gram, and 0.05  $\mu$ m are observed from samples with a concentration of 0.1 wt%, respectively. Therefore, the best wear reduction is from nanolubricant with the addition of nano CaCO<sub>3</sub> powder additives at 0.1 wt% concentration. It proves that the use of CaCO<sub>3</sub> nanoparticles has a good impact on the performance of the nano lubricant.

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## **Authors' Declaration**

**Authors' contributions and responsibilities** - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

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