http://jurnal.unmer.ac.id/index.php/jtmt
TRANSMISI
ISSN (print): 9-772580-228020
ISSN (online): 2580-2283

# Investigation of Thermophysical and Rheological Properties of Scallop Shell Powder/SAE 5w-30 Nanolubricant

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**ARTICLE INFORMATION**

Received: 19 February 2023
Revised: 1 March 2023
Accepted: 7 March 2023
Published: 15 March 2023

**ABSTRACT**

Nanolubricant is a type of nanofluid containing base-fluid lubricant (water or oil) and nanoparticles. This study aims to analyze the thermophysical and rheological properties of lubricants with the addition of nanoparticles. For the nanoparticles, we used Calcium Carbonate (CaCO3) made from scallop shell waste. The base lubricant was SAE 5W-30 synthetic oil with relatively good performance. Synthesis of Calcium Carbonate (CaCO3) into SAE 5W-30 lubricant was carried out using a two-step method. The volume fraction variation of Calcium Carbonate (CaCO3) was 0.05, 0.10, and 0.15%. Furthermore, the nano lubricant was tested for its thermophysical properties, which included thermal conductivity, specific heat, density, viscosity, and sedimentation. After that, the rheology of the nano lubricant was investigated from the viscosity data by calculating the shear rate and shear stress.

*Keywords: Oil 5W-30, Calcium Carbonate, Nanolubricant, Thermophysical Properties, Rheology.*

**ABSTRAK**

Nanolubricant merupakan jenis nano fluida yang mengandung pelumas base-fluid (air atau minyak) dan nanopartikel. Penelitian ini bertujuan untuk menganalisis sifat termofisik dan reologi pelumas dengan penambahan nanopartikel. Nanopartikel yang digunakan merupakan Kalsium Karbonat (CaCO3) yang terbuat dari limbah cangkang kerang simping. Pelumas dasar merupakan oli sintetik SAE 5W-30 yang memiliki performa cukup baik. Sintesis Kalsium Karbonat (CaCO3) ke dalam pelumas SAE 5W-30 menggunakan two-step methode. Variasi penambahan fraksi volume Kalsium Karbonat (CaCO3) sebesar 0,05%, 0,10%, 0,15%. Selanjutnya nanolubricant di uji sifat termofisiknya yang meliputi konduktivitas termal, kalor spesifik, densitas, viskositas, dan sedimentasi. Setelah itu reologi dari nanolubricant dapat diketahui dari data viskositas dengan perhitungan yang meliputi shear rate dan shear stress.

DOI: 10.26905/jtmt.v19i1.9638

Kata Kunci: Oli 5W-30, Kalsium Karbonat, Nanolubricant, Sifat Termofisik, Reologi.

## 1. Introduction

Nanolubricant is a type of nanofluid containing fluid-base lubricant and nanoparticles that can enhance lubrication. Lubrication on the machine increases its usability. In contrast, less accurate lubricant selection may damage and lower the machine's performance. Recently available studies reported that lubrication with nanoparticle additives could improve the thermophysical properties of the basic lubricant. The basic lubricant added with nanoparticle additive is commonly known as nano lubricant. The addition of additive nanoparticles reduces wear and tear caused by friction while also improving the lubricant's tribological and rheological properties [1]. Further, this addition of material also enhances the lubricant features and optimizes its thermophysical properties through the formation of tribofilm [2]. Thermophysical property is an essential factor in lubrication as it determines the engine heat transfer performance. The thermophysical properties involve density, viscosity, and thermal conductivity [3]. Besides, the nanoparticle also improves the connection between lubricant stress and strain, which correlates with its rheological characteristics [4].

Recently, the use of lubricants has accelerated significantly, primarily in the industry and automotive sector. According to the data from the Central Bureau of Statistics, the number of motor vehicles in Indonesia continuously increases. The increase was observed from 105,303,318 units in 2015 to 133,617,012 in 2019 [5]. This increase in motor vehicles is recorded along with the higher production of motor vehicles and spare parts. Consequently, this increasing consumption demands faster and easier production to accelerate the economic cycle. The efficiency and increasing performance in the industrial sector is inseparable from the instrument's condition and usage. The frequently used tools frequently experience instability, along with wear and tear. This wear and tear are induced by inaccurate lubricant viscosity and lubricant quality [6].

The addition of nanoparticles into the lubricant will result in four enhanced performances which directly contribute to the lubricant quality, such as rolling of nanospheres, formation of tribofilm as a result of the tribo-chemical reaction, mending effect, and polishing [7]. Besides, the addition of nanoparticles into the lubricant also offers a number of advantages, such as better stability observed during the suspension in an oil base than macro or microparticles; nanoparticles can reach the contact surface faster and form a protecting layer against wear and tear; nanoparticle requires no induction period to reach the expected tribology properties as nanoparticle is effective at the surrounding temperature, unlike the microparticle; and nanoparticle present better thermophysical properties than microparticle [8].

From those superiorities, this study aims to analyze the thermophysical properties of the lubricant added with calcium carbonate (CaCO3) as the additive material expected to enhance the lubricant performance [9]. The thermophysical analysis contained analysis of the thermal conductivity, density, viscosity, as well as sedimentation and rheology, which included shear rate and shear stress of the lubricant.

## 2. Methodology of Research

### *2.1. Materials*

In this study, we used calcium carbonate (CaCO3) originating from scallop shell waste to reduce the organic waste. Scallops are abundantly available in Indonesia due to their lengthy coastline and scallops ability to survive in tropical areas. For the basic lubricant, we used SAE 5W-30, which is commonly used in various motor vehicles. The SAE 5W-30 lubricant has been reported to have relatively good performance [10].

**Table 1.** Sspecification of SAE 5W-30 Lubricant

| Analysis | Standard | Value | Unit |
|---|---|---|---|
| *Grade* oli | - | SAE 5W-30 | - |
| *Pour Point*, , | ASTM D97 | -48 | °C |
| *Density* @ 15°C, , | ASTM D4052 | 848 | kg/m$^3$ |
| *Kinematic viscosity at* 40°C, | ASTM D445 | 73,95 | mm$^2$/s |
| *Kinematic viscosity at* 100°C | ASTM D445 | 12,02 | mm$^2$/s |
| *Flash Point, Cleveland Open-Cup* | ASTM D92 | 220 | °C |

### *2.2. Characterization of Material*

Material characterization was conducted to identify the structure of scallop shell waste powder which was used as the additive nano lubricant material. To investigate the surface morphology of the material, we carried out a Scanning Elektron Microscopy (SEM) test [11]. We used FEI Inspect-S50 for the SEM test with 13-100.000 magnification. Meanwhile, X-Ray Diffraction (XRD) is a non-destructive test commonly used to characterize microcrystalline material [12]. This technique is commonly used for phase identification, quantitative analysis, and investigation of defects in a material. For the XRD analysis, we used Pan Analytical E'xpert with ceramic Cu X-Ray Tube (2 kW). Further, the functional group was identified using Fourier Transform Infrared (FTIR) [13]. The infrared spectrum is a method for identifying the molecular structure through atom vibration and rotation [14]. We used Shumadzu Irprestige 21 for the FTIR analysis with a single beam optical system.

*2.3. Nanolubricant Preparation*

Nanolubricant was prepared using the two-step method due to its simple preparation procedures and low cost. First, the nanoparticle and base oil were stirred using Thermo Scientific Cimarec+™ Stirring Hot Plates Series Magnetic Stirrer at 1250 rpm speed for 20 minutes. Then, we carried out homogenization using Ultrasonic Homogenizer at 30Khz frequency for 30 minutes. We prepared three samples with a variation in the volume fraction of 0.05%, 0.10%, and 0.15%. For the base oil, we used 100 m of SAE 5W-30 lubricant.

*2.4. Analysis of Thermophysical Property*

Thermophysical property is a central factor as it correlates with the engine heat transfer efficiency. This property covers density, thermal conductivity, specific heat capacity, and a viscosity [3]. Fluid density is the mass per volume unit symbolized by *ρ* (*rho*). Meanwhile, density is frequently used in characterizing fluid mass systems [15]. The density was measured using Optima Scale *OPD-E 4-Decimal High Precision Analytical Balance*. Then, the results were calculated using Formula 1.

$$\rho = \frac{m}{v} \quad (1)$$

In which ρ represent the fluid density ($kg/m^3$), m is the mass (kg), and V is the volume ($m^3$).

Thermal conductivity is a material property that represents its ability to transfer heat. Fourier Law was used to investigate the material's conductivity. The higher heat transfer commonly occurs in materials with a greater thermal conductivity than materials with low thermal conductivity [16]. The thermal conductivity measurement was completed using KD2 Pro Thermal Properties Analyzer with accuracy ranging from 5-10% resistivity.

Specific heat capacity is a thermodynamic feature of a material that represents the required amount of heat per mass unit for enhancing the temperature by one degree Celsius. Similar to measuring the thermal conductivity, we used KD2 Pro Thermal Properties Analyzer to measure the specific heat capacity. Then, it was followed by calculation using Formula 2.

$$C_{p,nf} = \frac{(1-\emptyset)(\rho.C_p)_f+\emptyset(\rho.C_p)_{np}}{\rho_{nf}} \quad (2)$$

Where $C_{p,nf}$ represent the nanofluid's specific heat capacity (J/Kg.K), $\emptyset$ is the nanoparticle's volume fraction, $\rho_f$ is the basic fluid's density ($gr/cm^3$), $C_{p,f}$ is the basic fluid's specific heat capacity (J/Kg.K), $\rho_{np}$ is the nanoparticle density ($gr/cm^3$), and $C_{p,np}$ is the nanoparticle's specific heat capacity (J/Kg.K)

Viscosity is a physical measurement of fluid toward the flow. It is divided into two, namely dynamic and kinematics viscosity. The dynamic viscosity is also known as the viscosity coefficient, which relates to the ratio between shear, stress, and the shear rate which is measured in Poise (P) or *Centipoise* (cP) unit. Meanwhile, the kinematics viscosity is a calculation of liquid density as the result of dynamic viscosity, measured in (kg/m.s) or *Centistokes* (cSt) unit [15]. The dynamic viscosity was measured using an NDJ-8S viscometer. The rotor used in the measurement was rotor no 1, with a variation of speed, from 6, 12, 30, and 60 rpm. The viscosity test was completed at different temperatures, ranging from 30°C to 100°C.

*2.5. Analysis of Rheological Property*

Rheology is a branch of science describing the deformation and flow of a material. It aims to identify the correlation between the stress and strain for the specimen (non-Newtonian) incapable of being discussed by Newton and Hooke laws. A number of materials present non-Newtonian characteristics, such as lubricants, the flow of paint, adhesive, and so forth [17]. Rheology discusses the connection between strain and stress in a material, which also closely correlates with viscosity.

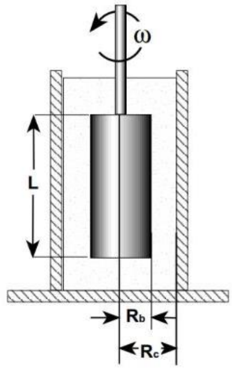


**Figure 1.** *Spindles and Vessels for Rheological Measurements*

The shear rate represents the speed at which the fluid shears or works during the flow. The shear rate was determined based on the geometry and rate of flow. The shear rate was calculated using Formula 3.

$$\gamma = \frac{2\omega R_c{}^2 R_b{}^2}{R_b{}^2(R_c{}^2 - R_b{}^2)} \quad (3)$$

in which γ is the *shear rate* (/s), ω is the angular velocity of the shaft (*spindle*) (rad/sec), $R_c$ is the vessel radius (cm), and $R_b$ is the axis radius (*spindle*) (cm).

Shear stress represents the multiplication results between the shear stress and the dynamic viscosity obtained using Formula 3. The shear stress was calculated using Formula 4.

$$\tau = \gamma\mu \quad (4)$$

in which τ is the shear stress (mPa.s), γ is the shear rate (/s), and μ is the dynamic viscosity (kg/m.s).

## 3. Result and Discussion

### *3.1. Scanning Electron Microscope (SEM) Analysis*

The SEM analysis was completed on calcium carbonate ($CaCO_3$) material to investigate and identify its morphological structure. The results of SEM analysis were in the form of pictures with various multiplication, which were analyzed further. The results of the SEM test on calcium carbonate ($CaCO_3$) with 50.000 times magnification are shown in Figure 2.

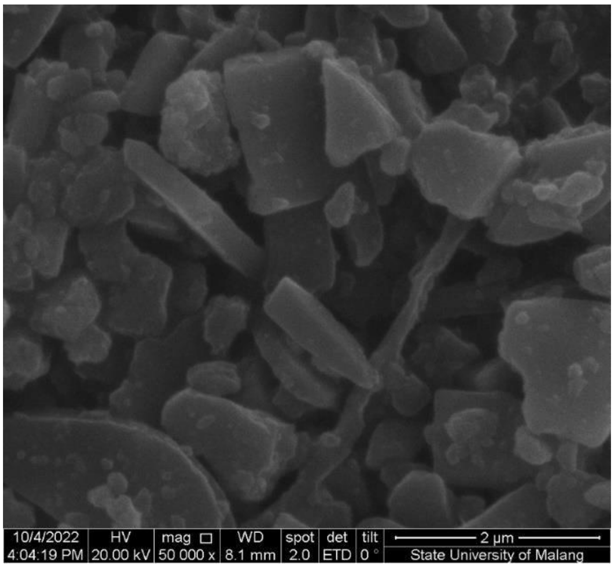


**Figure 2.** SEM Results on Calcium Carbonate (CaCO3) Nanoparticle with 50,000 Times Magnification

Figure 2 presents the SEM results on calcium carbonate ($CaCO_3$) with 50.000 magnification, showing the irregular surface morphology of the particle [18]. Besides, the particle also experienced agglomeration due to the sintering temperature, sintering time, uneven crushing, and duration of crushing [19]. The measurement of the particle was completed using ImageJ software with particle measurement results of 636, 652, 778, and 825 nm in random order. The particles with those sizes are classified as microparticles since, according to Bayda et al. (2020), nanoparticles' size ranges between 1-100 nm.

### *3.2. X-Ray Diffraction (XRD) Analysis*

The XRD test on calcium carbonate ($CaCO_3$) was conducted to identify its phase, grain shape, and crystallinity. The XRD test resulted in a graphic of peaks which were further analyzed. The result of the XRD test is illustrated in Figure 3.

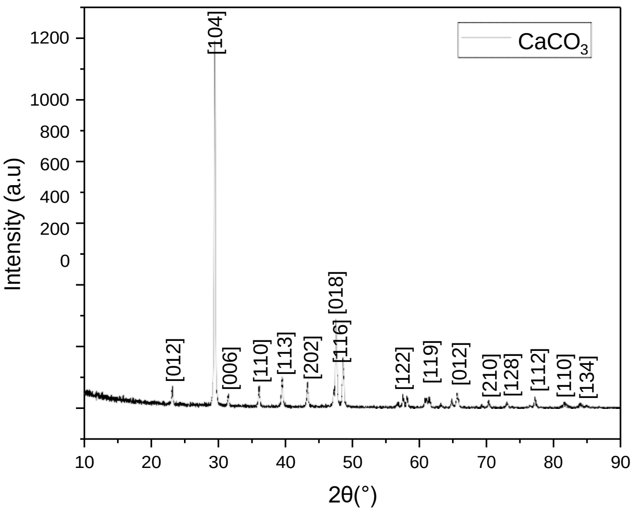


**Figure 3.** Graphic of XRD Test on $CaCO_3$

Figure 3 presents the XRD test results on $CaCO_3$ with peaks ranging between 10° - 90°. As shown in Figure 3, $CaCO_3$ has a single phase with calcite symmetry crystals, illustrated by the highest peak intensity of 1180,82 at two θ =29,51° [21]. The crystal of calcium carbonate ($CaCO_3$) from the scallop shell was analyzed using MTCH software. The analysis results showed trigonal (hexagonal axed) of $CaCO_3$. This finding is linear with a previous study conducted by Ningrum et al. (2022) reporting the trigonal crystal shape of calcium carbonate ($CaCO_3$).

The crystallite size was estimated using the Scherrer formula presented in the following formula [23].

$$D = \frac{K\,\lambda}{\beta \cos \theta}$$

Where D represents the crystal diameter (nm), K is the factor of crystal shape (0.9), λ is the wavelength of the x-rays (0,15406), β is the FWHM (Full Width at Half Maximum) values (rad), and θ is the diff (°). From the estimation using Scherrer Formula, we obtained crystallite size at 2θ =29.51° peak of 39.65 nm.

### *3.3. Fourier Transform Infrared Spectroscopy (FTIR) analysis*

The FTIR test on calcium carbonate (CaCO3) was conducted to identify its element and functional groups. The FTIR results were in the form of the graphic at 4000-500 wavelength. The results of the FTIR test are illustrated in Figure 4.

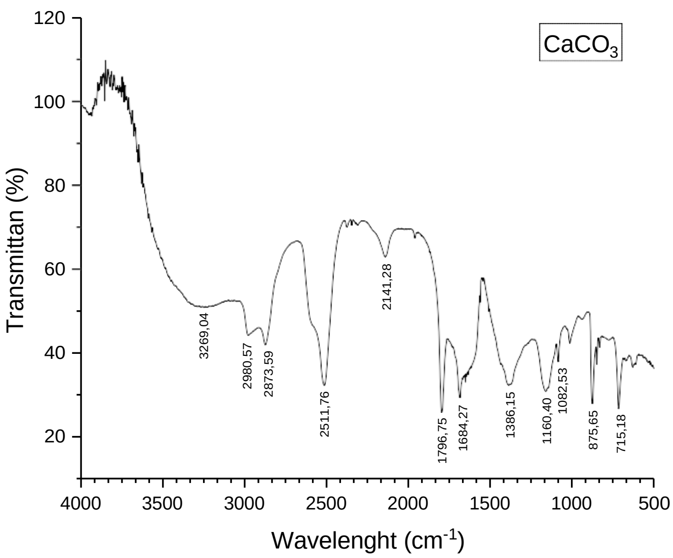


**Figure 4.** Analysis Results of FTIR Test on $CaCO_3$

## 4. Conclusion

Based on the results, it can be concluded that the study of the material properties of calcium carbonate (CaCO3) and nano lubricant SAE 5W-30 by adding volume fluctuations of calcium carbonate (CaCO3) fraction 0.05%, 0, 10%, 0.15% thermophysical and rheological properties at several locations as follows.

Surface morphology of shellfish powder (CaCO3) showed amorphous crystal precipitates and irregular particle shapes with particle sizes of 636 nm, 652 nm, 778 nm and 825 nm. CaCO3 particles also experience agglomeration due to unequal sintering temperature, sintering time and destruction. CaCO3 particles have one-phase calcite crystal symmetry and are trigonal crystal (hexagonal axis) with a crystal size of 39.65 nm with a peak of 20 = 29.51°. It is known that the functional groups of shellfish powder (CaCO3) are located at the peaks at 1386.15, 875.65 and 715.18 cm-1. The addition of calcium carbonate (CaCO3) to nano-lubricant based on SAE 5W-30 with volume fraction variations of 0.05%, 0.10%, 0.15% affected the value of thermophysical properties. Nano-lubricant based on SAE 5W-30 with volume fraction fluctuations of 0.05%, 0.10%, 0.15% has poor stability because CaCO3 particles and SAE 5W-30 lubricant do not bond properly. This affects the value of thermal conductivity which does not show a significant increase. The density value of the nano-lubricant increases with increasing volume fraction compared to SAE 5W-30 base oil. The highest density value was achieved at a volume fraction of 0.15% with 775.250 kg/m3, while the lowest density value was achieved at a volume fraction of 0.05% with 767.505 kg/m3. In addition, the viscosity value increased with increasing volume of the fraction and decreased with increasing temperature. The highest viscosity values were obtained at a volume fraction of 0.15% and 105.8 mPa.s at a spindle speed of 30 rpm and a temperature of 30 °C. while the lowest viscosity value was obtained at 0.05% volume fraction of 23 mPa.s at a spindle speed of 6 rpm and a temperature of 100 °C. The addition of CaCO3 to SAE 5W-30 base oil shows a linear flow relationship between shear rate and shear stress, so that it can be said to be Newtonian flow. At 40°C and 100°C the value of the shear stress increases. The increase was caused by an increase in the volume of the base lubricant fraction. The shear stress value of the nanolubricant at 40°C is lower than the shear stress value at 100°C because the van der Waals forces decrease at high temperatures. Van der Waals forces increase the viscosity value of nano-lubricants while at high temperatures can help the particles overcome the tensile forces caused by these forces.

### 5. Acknowledgments

The authors would like to thank Universitas Negeri Malang for Hibah Publikasi Skripsi (Internal UM) on 2023 for PP.

## References

[1] A. Beel, M. Gottschalk, A. Huetten, K. Toensing, and D. Anselmetti, “Tribological Performance of TiO2-Nanostructured Particles as Oil-Lubricant Additives for Different Iron-Carbon Alloys,” *Mater. Today Proc.*, vol. 4, pp. S75–S80, 2017, doi:

10.1016/j.matpr.2017.09.170.

[2] J. Zhao, Y. Huang, Y. He, and Y. Shi, “Nanolubricant additives: A review,” *friction*, vol. 9, no. 5, pp. 891–917, 2021, doi: 10.1007/s40544-020-0450-8.

[3] A. Kotia, P. Rajkhowa, G. S. Rao, and S. K. Ghosh, “Thermophysical and tribological properties of nanolubricants: A review,” *Heat Mass Transf. und Stoffuebertragung*, vol. 54, no. 11, pp. 3493–3508, 2018, doi: 10.1007/s00231-018-2351-1.

[4] J. R. Stokes and Y. Xu, *Rheology of Food Materials: Impact on and Relevance in Food Processing*. Elsevier, 2019.

[5] S. P. Dewi, R. Alsakinah, S. A. Sara, and D. H. Amrina, “Pajak Lingkungan Sebagai Upaya Pengendalian Pencemaran Udara Dari Gas Buang Kendaraan Bermotor Di Indonesia,” *J. Ilm. Ekon. dan Pajak*, vol. 2, no. 1, pp. 7–13, 2022, [Online]. Available: https://ojs-ejak.id/index.php/Ejak.

[6] S. K. Choudhury and M. Muaz, *Natural Oils as Green Lubricants in Machining Processes*. Elsevier Ltd., 2020.

[7] W. Dai, B. Kheireddin, H. Gao, and H. Liang, “Roles of nanoparticles in oil lubrication,” *Tribol. Int.*, vol. 102, pp. 88–98, 2016, doi: 10.1016/j.triboint.2016.05.020.

[8] M. A. Bin Saufi and H. Bin Mamat, “A Review on Thermophysical Properties for Heat Transfer Enhancement of Carbon-Based Nanolubricant,” *Adv. Eng. Mater.*, vol. 23, no. 10, pp. 1–20, 2021, doi: 10.1002/adem.202100403.

[9] S. B. Prakash, “A Study on CaCO 3 -Water Nano Fluid Properties as a Heat Transfer Fluid,” vol. 6, no. 1, pp. 26–31, 2016, doi: 10.17605/OSF.IO/PQD2C.

[10] U. . Evans, E. . Okore, and E. Edem, “Thermo-physical properties of selected oil samples as predictor of marine engines life span,” *IOSR J. Appl. Phys.*, vol. 5, no. 2, pp. 01–06, 2013, doi: 10.9790/4861-0520106.

[11] A. Srivastava *et al.*, *Characterizations of nanoscale two-dimensional materials and heterostructures*. INC, 2020.

[12] D. Louër, “Powder X-ray diffraction, applications,” *Encycl. Spectrosc. Spectrom.*, pp. 723–731, 2016, doi: 10.1016/B978-0-12-803224-4.00257-0.

[13] J. S. Gaffney, N. A. Marley, and D. E. Jones, “FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY,” *Fourier Transform Infrared Spectra*, 2012, doi: 10.1016/c2009-0-22072-1.

[14] X. Hou, S. Lv, Z. Chen, and F. Xiao, “Applications of Fourier transform infrared spectroscopy technologies on asphalt materials,” *Meas. J. Int. Meas. Confed.*, vol. 121, no. March, pp. 304–316, 2018, doi: 10.1016/j.measurement.2018.03.001.

[15] Munson, Young, and Okiishi’s, *Fundamentals of Fluid Mechanics*. 2016.

[16] F. Jabbari, A. Rajabpour, and S. Saedodin, “Thermal conductivity and viscosity of nanofluids: A review of recent molecular dynamics studies,” *Chem. Eng. Sci.*, vol. 174, pp. 67–81, 2017, doi: 10.1016/j.ces.2017.08.034.

[17] C. Riedel, A. Alegria, J. Colmenero, and P. Tordjem, *Polymer Rheology by Dielectric Spectroscopy*. 2012.

[18] N. S. W. Supriyanto, Sukarni, P. Puspitasari, and A. A. Permanasari, “Synthesis and characterization of CaO/CaCO3 from quail eggshell waste by solid state reaction process,” *AIP Conf. Proc.*, vol. 2120, no. July 2019, 2019, doi: 10.1063/1.5115670.

[19] P. Puspitasari, D. M. Utomo, H. F. N. Zhorifah, A. A. Permanasari, and R. W. Gayatri, “Physicochemical determination of calcium carbonate (CaCO3) from chicken eggshell,” *Key Eng. Mater.*, vol. 840 KEM, no. April, pp. 478–483, 2020, doi: 10.4028/www.scientific.net/kem.840.478.

[20] S. Bayda, M. Adeel, T. Tuccinardi, M. Cordani, and F. Rizzolio, “The history of nanoscience and nanotechnology: From chemical-physical applications to nanomedicine,” *Molecules*, vol. 25, no. 1, pp. 1–15, 2020, doi: 10.3390/molecules25010112.

[21] A. Ghadami Jadval Ghadam and M. Idrees, “Characterization of CaCO3 nanoparticles synthesized by reverse microemulsion technique in different concentrations of surfactants,” *Iran. J. Chem. Chem. Eng.*, vol. 32, no. 3, pp. 27–35, 2013.

[22] R. A. Ningrum, S. Humaidi, S. Sihotang, D. Bonardo, and Estananto, “Synthesis and material characterization of calcium carbonate (CaCO3) from the waste of chicken eggshells,” *J. Phys. Conf. Ser.*, vol. 2193, no. 1, 2022, doi: 10.1088/1742-6596/2193/1/012009.

[23] P. Puspitasari *et al.*, “ Synthesis and characterization of CaCo 3 /CaO from

Achatina fulica in various sintering time ," *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 1034, no. 1, p. 012093, 2021, doi: 10.1088/1757-899x/1034/1/012093.