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The Effect of Sintering Temperature on Hydrophobic Characteristic of Silica Manganese /Polystyrene (SiMn/PS) Nanocomposite

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ABSTRACT

Currently a lot of research has been done on hydrophobic layers, but in its application the layer is easily damaged and is not corrosion resistant. Therefore, this research intends to decide the effect of variations in sintering temperature on the hydrophobic characteristic of SiMn/PS nanocomposite layers using a sintering temperature of 60ºC, 100ºC, 140ºC, 180ºC and 200ºC for 1 hour using a furnace. This research was conducted in the Material Physics Laboratory of the Faculty of Mathematics and Natural Sciences and the Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences, Padang State University.This research uses HEM-3D (High Energy Milling Ellipse-3D Mention), XRD (X-Ray Difraction) and SEM (Scanning Electron Microscope) tools. The precursor was made by giving 0.5 grams of polystyrene, 0.2 grams of silica powder and 0.2 grams of manganese powder. Coating is done by spin coating method. The results of this research from the variation of the sintering temperature showed that the SiMn/PS nanocomposite layer was hydrophobic based on the contact angle test. The highest contact angle is at a temperature of 60^{0}C.

Keywords : Hydrophobic, Contact Angle, Durability, Nanocomposite, Polystyrene, Silica Oxide (Sio2), Manganese (Mn).

I. INTRODUCTION

With the development of this era, there have been many research to create a hydrophobic surface. However, in use, the layer is easily damaged and easily corroded due to contact with other objects. Therefore, the development of a strong and durable hydrophobic layer and corrosion resistance with an easy and efficient method is very necessary.

Hydrophobic can be defined as water-repellent properties, has a large contact angle of 90^{0}C and an unpleasant surface. A surface can be said to be hydrophobic if it has certain characteristics. Surface characteristics that are hydrophobic include having anti-wet properties, always looking clean, having a large contact angle of 90^{0}C [1]. The hydrophobicity of a surface can be dictated by estimating the contact angel shaped between the water and the sample surface [2]. Surfaces that are hydrophobic have a contact angle between 90°- 150°, while a surface that has a contact angle of >150° is called superhydrophobic [3].

The substrates used are those that have not corrosion characteristic such as manganese and strong characteristic such as silica which are made into nanocomposites. Nanocomposite is a combination of two or more materials consisting of a matrix and a reinforcement measuring 1.0 x 10^{-9} m. Silica is a metal oxide compound that is widely found in nature, but its presence is not free but is bound to other compounds both physically and chemically [4]. The three predominant crystalline phases of silica are quartz, tridymite, and crystobalite [5]. The course of action of particles in shapeless silica happens haphazardly with a low level of regularity. Therefore, it is necessary to carry out a silica sand purification process to remove impurities.

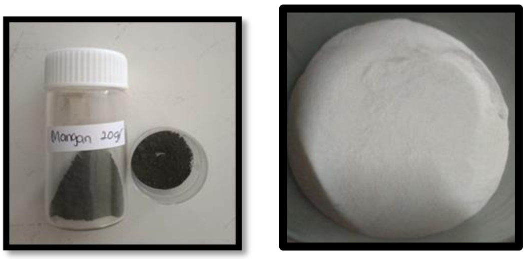
Manganese is one of the most abundant elements found in the earth's crust which has gray black color.

For the manufacture of the coating required a polymer, the polymer used is polystyrene. This polystyrene is resistant to acids, bases and other rusting agents [6]. Many methods are used for this purpose, such as electrochemical reactions,phase separation, sin coating, dip coating, sol-gel, coprecipitation and fillingparticles [7]. The method used to create layers SiMn/PS nanocomposite is spin-coating. Relative spin-coating methodeasy to use, homogeneous coating, and not very expensive.

Temperature sintering is a heating process at a high temperature in any case, beneath the dissolving point until a change in the microstructure occurs [8]. Giving this temperature variation to see a change in the contact angle [9] and the morphology of the layers [10]. There is treatment heat causes bonding between powder particles and increases thestrength of the resulting product [11].

II. METHOD

This research is an experimental research type. This study examines the effect of variations in the sintering temperature of SiMn/PS nanocomposite on the hydrophobic characteristic. This research was directed at the Material Physics and Biophysics Laboratory of the Faculty of Mathematics and Natural Sciences and the Chemistry Laboratory of the State University of Padang. The materials used are nanoparticles of silica, manganese, polystyrene (PS), tetrahydrofuran (THF) and aquadest. The tools used are measuring cups, beakers,magnetic stirrer, glass (size 0.5 cm x 0.5 cm and 1 cm x 1cm), camera, XRD, FTIR, HEM, furnace, oven and spin coating. The forms of silica and manganese powders are shown in Figure 1.



(a) (b)

Fig. 1. (a) Manganese Powder and (b) Silica

The manganese powder used is black. This powder was milled using HEM-E 3D for 16 hours. And the silica powder used is white which has been previously purified. This silica powder was milled using HEM-E 3D for 5 hours. silica that has been ground using HEM-E 3D and then purified by the coprecipitation method. The coprecipitation method is carried out by dissolving silica into a strong base solution (NaOH), resulting in a sodium silicate solution then titrated using 2M HCl and the purification results obtained amorphous silica. To get the crystal structure from amorphous to polymorphic, calcination treatment was given.

The next step is the manufacture of a hydrophobic layer of Silica Manganese/Polystyrene (SiMn/PS) nanocomposite with 0.1 g polystyrene (PS) dissolved in 10 mL of tetrahydrofuran (THF) stirred with a magnetic stirrer until homogeneous at 50ºC. Furthermore, 0.2 grams of silica nanoparticles and 0.2 grams of manganese were mixed with polystyrene (PS) solution. The mixture was then stirred for 60 minutes with a magnetic stirrer until homogeneous.

The Thin Layer Growth Stage Using the Spin Coating Method is carried out by placing the glass substrate to be coated into the spin coater. Then drip a homogeneous Silica Manganese/Polystyrene (SiMn/PS) composite solution on a glass substrate using a dropper and set a time of 60 seconds and a rotational speed of 500 rpm. Pressing the star button to start playback and pressing the off button to stop playback according to the preset time.Sillica Manganese/Polystyrene (SiMn/PS) nanocomposite layers that have been formed are then in a furnace with temperature variations of 60°C, 100°C, 140°C, 180°C and 200°C for 60 minutes. Heating the sample is done by entering the sample into the furnace and the sintering temperature is set for 1 hour. After 1 hour the sample will be removed from the furnace and cooled to room temperature.

Furthermore, the dried samples were tested for contact angles. The contact angle measurement is done by dripping water on the surface of the sample with the image taken with a DSLR camera. In this method the contact angle is determined using ImageJ Software to avoid parallax errors during measurement. ImageJ Software display as Figure 2.

Fig. 2. ImageJ Software Display

To measure the contact angle, select the image to be measured in the file tool, after the image appears then select the angle tool and then draw a straight line between the surface and the water droplet and draw the line up until it forms an angle between the sample surface and the droplet.

Fig. 3. Measuring Contact Angle

To find out the contact angle, select "Analyze" then select "Maesure" and the results of the contact angle will be displayed. After making repeated measurements and then looking for the average.

The contact angle analysis obtained from the measurement results of the SilicaManganese/Polystyrene (SiMn/PS) nanocomposite layer can be calculated by the following formula:

$$Contact\ angle = \frac{right\ contact\ angle + left\ contact\ angle}{2} \quad (1)$$

Then the resulting data is described in the form of a graph. The effect of temperature on the contact angle can be determined by plotting the data on the X and Y coordinates using the Microsoft Excel program.

III. RESULTS AND DISCUSSION

Contact angle testing was carried out with temperature variations of 60°C, 100°C, 140°C, 180°C and 200°C. It is important to measure the contact angle to find out which layer at what temperature is more hydrophobic. The hydrophobic surface has a large contact angle of 90^0 [12]. In accordance with the conclusion of Bhusan's research which concluded that the hydrophobic nature is influenced by the roughness factor and the resulting contact angle. The magnitude of the resulting contact angle at temperatures of 60°C, 100°C, 140°C, 180°C and 200°C can be seen in Figure 4-8.

The hydrophobic nature of a surface can be seen from the water contact angle test parameters. After obtaining the contact angle value of the water that was dripped on the SiMn/PS nanocomposite layer, the contact angle was observed on the heat-treated layer. The temperature variation of 60°C, 100°C, 140°C, 180°C and 200°C for 60 minutes each showed a large change in the contact angle.

The size of the contact angle on the sample tends to decrease with each increase in heating temperature. At 60°C the contact angle formed is 104.7°. At a temperature of 100°C, 140°C and 180°C the contact angle changes, only by 3°, which is 95.497^o, 92.523^o and $90{,}228^o$. When the coating is heated at a temperature of 200°C, the change in contact angle reaches 82.522^o.

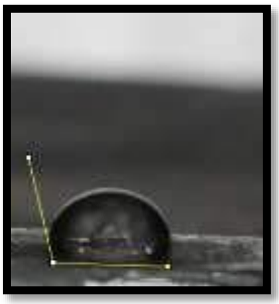


Fig. 4. Contact Angle from a Temperature of 60ºC

Based on Figure 4, it can be seen that the measurement of the contact angle at a temperature of 60ºC which was measured using ImageJ software. The results of the measurement of the contact angle at a temperature of 60ºC can be seen in Table 1.

Table 1. Results of contact angel from a temperature of 60ºC

Contact Angel		
Measurement Results		**The Calculation Results**
θ Right	θ Left	θ
104,986º	105,457º	105,221º
104,484º	104,339º	104,411º
105,287º	105,097º	105,197º
104,706º	104,925º	104,817º
104,321º	103,389º	103,855º
Average		104,7º

Contact angle measurements were performed using ImageJ software. Measurements are carried out repeatedly in order to get a more accurate value and avoid parallax errors on the sample. At a temperature of 60ºC obtained a high contact angle of 104.7º. means that at a temperature of 60ºC the layer is hydrophobic.

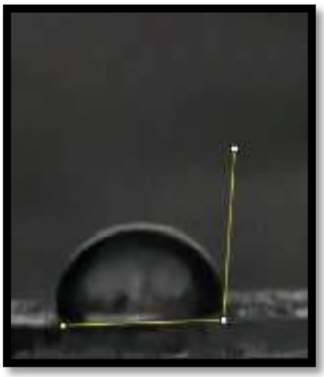


Fig. 5. Contact Angle from a Temperature of 100ºC

Based on Figure 5, it can be seen that the measurement of the contact angle at a temperature of 100ºC which was measured using ImageJ software. The results of the measurement of the contact angle at a temperature of 100ºC can be seen in Table 2.

Table 2. Results of contact angel from a temperature of 100^oC

Contact Angel		
Measurement Results		**The Calculation Results**
θ Right	θ Left	θ
95,229^o	94,251^o	95.74^o
95,805^o	95,744^o	95,774^o
96,881^o	95,756^o	96,318^o
95,036^o	95,541^o	95,288^o
95,200^o	95,537^o	95,368^o
Average		95,497^o

Contact angle measurements were performed using ImageJ software. Measurements are carried out repeatedly in order to get a more accurate value and avoid parallax errors on the sample. At a temperature of 100^oC obtained a high contact angle of 95.497^o. means that at a temperature of 100^oC the layer is hydrophobic.

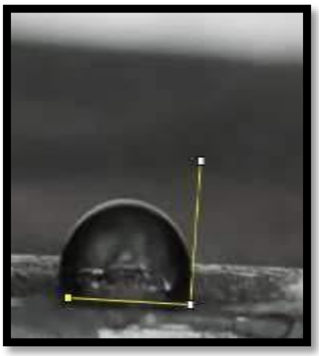


Fig. 6. Contact Angle from a Temperature of 140^oC

Based on Figure 6, it can be seen that the measurement of the contact angle at a temperature of 140^oC which was measured using ImageJ software. The results of the measurement of the contact angle at a temperature of 140^oC can be seen in Table 3.

Table 3. Results of contact angel from a temperature of 140^oC

Contact Angel		
Measurement Results		**The Calculation Results**
θ Right	θ Left	θ
92,245^o	93,072^o	92,658^o
92,726^o	92,513^o	92,637^o
92,984^o	92,441^o	92,712^o
92.821^o	92,122^o	92,472^o
92,153^o	92,214^o	92,183^o
Average		92,523^o

Contact angle measurements were performed using ImageJ software. Measurements are carried out repeatedly in order to get a more accurate value and avoid parallax errors on the sample. At a temperature of 140^oC obtained a high contact angle of 92.523^o. means that at a temperature of 140^oC the layer is hydrophobic.

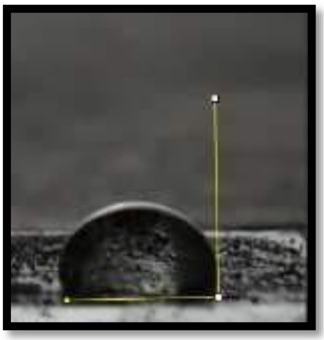


Fig. 7. Contact Angle from a Temperature of 180ºC

Based on Figure 7, it can be seen that the measurement of the contact angle at a temperature of 180ºC which was measured using ImageJ software. The results of the measurement of the contact angle at a temperature of 180ºC can be seen in Table 4.

Table 4. Results of contact angel from a temperature of 180ºC

Contact Angel		
Measurement Results		**The Calculation Results**
θ Right	θ Left	θ
90,300º	90,515º	90,407º
90,000º	90,175º	90,087º
90,384º	90,000º	90,192º
90,212º	90,395º	90.303º
90,124º	90,184º	90,154º
Average		90,228º

Contact angle measurements were performed using ImageJ software. Measurements are carried out repeatedly in order to get a more accurate value and avoid parallax errors on the sample. At a temperature of 180ºC obtained a high contact angle of 90.228º. means that at a temperature of 180ºC the layer is hydrophobic.

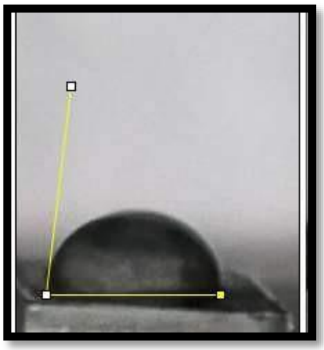


Fig. 8. Contact Angle from a Temperature of 200ºC

Based on Figure 8, it can be seen that the measurement of the contact angle at a temperature of 200ºC which was measured using ImageJ software. The results of the measurement of the contact angle at a temperature of 200ºC can be seen in Table 5.

Table 5. Results of contact angel from a temperature of 200°C

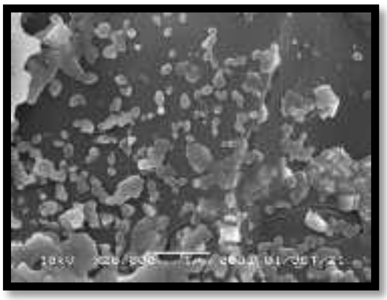
Contact Angle		
Measurement Results		The Calculation Results
θ Right	θ Left	θ
82,593°	83,202 °	82,897°
82,638°	82,110 °	82.37°
82,143°	83,018 °	82,580°
82,544°	82,532 °	82,538°
82,314°	82,143 °	82,228°
Average		82,522°

Contact angle measurements were performed using ImageJ software. Measurements are carried out repeatedly in order to get a more accurate value and avoid parallax errors on the sample. At a temperature of 200°C obtained a high contact angle of 82.522°. means that at a temperature of 200°C the layer is hydrophilic.

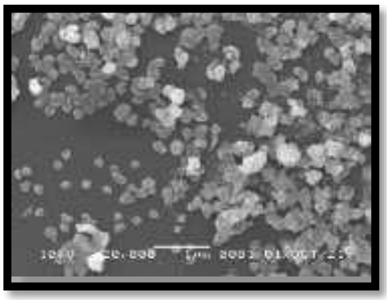
This happens because of the high level of roughness. However, at a temperature of 200°C, the surface becomes hydrophilic due to the melting of the PS matrix which causes the SiMn nanoparticles to rise to the surface and the volume of air trapped in the nanostructure decreases which causes a decrease in the contact angle. Surface physical properties will affect the hydrophobic properties of the material, materials coated with nano-sized materials can be hydrophobic and even superhydrophobic, this is because nano-sized materials have a larger surface area than bulk-sized materials [13].

From the results of measurements and analysis of the contact angle of the sintering temperature, the highest contact angle was obtained at a temperature of 60°C. This indicates that the highest contact angle is not obtained at high or low temperatures, but at the optimum temperature. At high temperatures, the contact angle obtained on the surface of the coating is not maximal or very small so that at high temperatures it is hydrophilic.

Temperature variations on the morphology of the hydrophobic layer greatly affect the particle size distribution, where the smaller the particle size distribution, the larger the roughness will be. However, with a large particle size distribution it will reduce the specific surface area. This slight roughness will result in a large interaction with the water. So the surface will tend to be wet because the water is not in a state of equilibrium [14].



(a)



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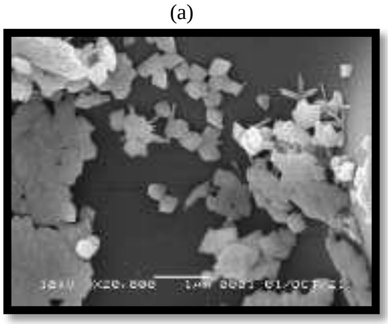

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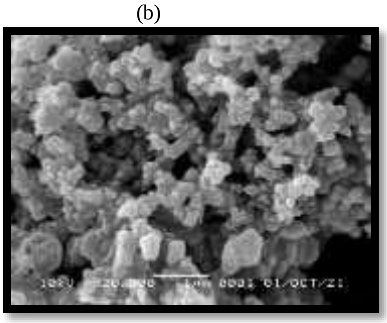

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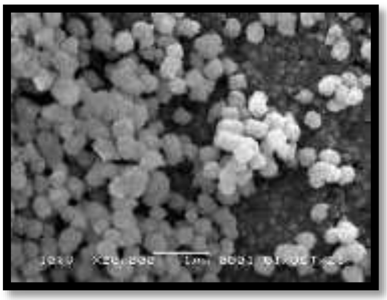

(e)
Fig. 9. Differences in the morphology of the SiMn/PS nanocomposite at each variation of the heating temperature with a magnification of 20000X. (a) Temperature 60°C (b) Temperature 100°C (c) Temperature 140°C (d) Temperature 180°C and (e) Temperature 200°C

The morphology of the SEM test results on the surface with calcined manganese fillers 60°C, 100°C, 140°C, 180°C, and 200°C. Where at that temperature the average particle size distribution is 51,425 nm, 55,495 nm, 57,217 nm, 62,343 nm, and 63,016 nm. The existence of temperature variations in this study can be seen from the effect of particle size distribution produced, namely the higher the temperature, the greater the particle size. Small particle size distribution will have a large effect on roughness. So that there will be less solid-liquid interaction and greater liquid-air interaction. This fits the Cassie model. In this study, it is known that the layer with silica manganese calcined at 60°C has the highest water contact angle of 104.7°. There are many bumps that give each particle a high roughness. These protrusions are agglomerated nano-order SiMn particles. The structure of the SiMn/PS nanocomposite layer affects the level of hydrophobicity. Because when water is dropped onto the surface of the substrate, air is trapped in the middle of the roughness.

IV. CONCLUSION

There is an effect of sintering temperature on the durability of the contact angle of the hydrophobic layer of SiMn/PS nanocomposite. At temperature 60^{o}C is a temperature that has better hydrophobic properties than other temperatures which is characterized by a high contact angle of 104.7^o this is because the contact angle value is influenced by the roughness hierarchy formed, has a nano particle size of 51.425 nm. , the crystal size is also nano at 24,261 nm and the functional group looks complete.

Scanning Electron Microscope (SEM) results at a temperature of 60^{o}C show that many pores are formed and the pore walls form a hierarchical structure. Because of these characteristics, a temperature of 60^{o}C can form the largest contact angle of all temperature variations. The phenomenon experienced by at a temperature of 60°C is similar to the Cassie-Baxter theory, when water is dropped on the surface it will hit particles measuring 51.42 nm so that air is trapped below which causes the largest contact angle formed.

The increase in sintering temperature causes a change in the durability of the hydrophobic properties of the coating where the contact angle decay constant increases and the layer is hydrophilic with a contact angle of <90^o. This is because heat treatment makes the particle size bigger, due to the particle agglomeration process.

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REFERENCES

[1] L. Feng *et al.*, “Super-hydrophobic surfaces: From natural to artificial,” *Adv. Mater.*, vol. 14, no. 24, pp. 1857–1860, 2002, doi: 10.1002/adma.200290020.

[2] H. J. Ensikat, P. Ditsche-Kuru, C. Neinhuis, and W. Barthlott, “Superhydrophobicity in perfection: The outstanding properties of the lotus leaf,” *Beilstein J. Nanotechnol.*, vol. 2, no. 1, pp. 152–161, 2011, doi: 10.3762/bjnano.2.19.

[3] B. Karthick and R. Maheshwari, “Lotus-inspired nanotechnology applications,” *Resonance*, vol. 13, no. 12, pp. 1141–1145, 2008, doi: 10.1007/s12045-008-0113-y.

[4] Hildayati, “Sintesi dan Karakterisasi Bahan Komposit Karet Alam Silika,” *ITS Surabaya*, 2009.

[5] Szostak R, “Mollecular Sieves: Principles of Synthesis and Identification.,” *Springer*, 1998.

[6] Ratnawulan, “Fabrication of superhydrophobic CuO/polystyrene nanocomposite coating with variation concentration,” *J. Phys.*, vol. 1185, 2019.

[7] T. Setiawati, A. I.S., S. G.S, and W. A.A, “Sintesis Lapisan Tipis TiO2 Dan Analisis Sifat Fotokatalisnya,” *J. Sains Mater. Indones.*, no. Vi, pp. 141–146, 2006.

[8] A. and R. Putri, “ANALISIS TEORISTIK NANOPARTIKEL ZIRKONIUM DIOKSIDA (ZrO2),” *Pillar Phys.*, vol. 12, no. 1, pp. 70–76, 2019.

[9] M. Rohmah, R., & Zainuri, “Pengaruh Variasi Temperatur Kalsinasi SiO2 terhadap Sifat Kebasahan pada Permukaan.,” *J. Sains dan Seni ITS*, vol. 5, no. 2, 2016.

[10] M. Irawati, A.F, Zainuri, “Pengaruh Temperature Perlakuan Panas Pada Lapisan Hydrophobic Komposit PDMS/SiO2 Dengan Fasa Silika Kristobalit.,” *J. sains dan Seni ITS*, vol. 5, 2016.

[11] D. Darvina, Y., & Desnita, “Pengaruh Suhu Sintering Terhadap Sifat Penyerapan Gelombang Mikro dari Nanokomposit Cofe2o4/PVDF0yang di Preparasi Dengan Metode Sol-Gel,” *PILLAR Phys.*, vol. 12, no. 12, 2019.

[12] D. Bhusan, Bharat, “Micro-, nano- and hierarchica structures for superhydrophobicity, self-cleaning and low adhesion.,” *J. Phil. Trans*, 2009.

[13] M. Abdullah, “Sintesis Nano material,” *J. Nanosains dan Teknol.*, vol. 1, no. 2, 2008.

[14] R. Rohmah and M. Zainuri, “Pengaruh Variasi Temperatur Kalsinasi SiO2 terhadap Sifat Kebasahan pada Permukaan,” *J. Sains dan Seni ITS*, vol. 5, no. 2, pp. 3–6, 2016.