ENHANCEMENT OF MATERIALS SURFACE PROPERTIES VIA SURFACE COATING PROCESS: POLYMER SUBSTRATE IN SILICA SAND BATH

By:

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Dissertation submitted in partial fulfilment of
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CERTIFICATION OF APPROVAL

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Approved by,

_____

(AP. Dr. Othman Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MUHAMMAD ZUL ‘AFIF BIN SARU @ SARUDDIN

ACKNOWLEDGEMENT

Given this opportunity, I would like to acknowledge those who have been helping me, either directly or indirectly, in continue working on the project – starting from preliminary research, to the development phase and until the completion phase of this Final Year Project in Universiti Teknologi PETRONAS.

First of all, I would like to express my deepest gratitude to Allah for giving me the strength and health throughout my years of study in this university, which allows me to gain enough theoretical knowledge and practical skill sets as a mechanical engineer, to be applied into this particular project or other project in the coming years.

I would like to thank AP. Dr. Othman Mamat, my supervisor for this project for the past two semesters. Dr Othman has been giving me moral and mental supports since the first day of this project begin. His excellent guidance, caring and providing me excellent atmosphere, help me a lot in conducting the project from the start to finish.

Besides that, I also like to thank my colleagues in the university for their assistants in helping me to prepare the sample and assisting me along the project period. The same gratitude goes to my beloved parents for always giving me advice and supports me with their best wishes.

ABSTRACT

In industry today, polymer is an important substance that been used since it has versatile characteristics, easy to process and low cost of production. However some mechanical properties such as strength and hardness are low. Therefore, in order to change the mechanical properties, the polymer had been modified by using several methods. One of them is thin film coating. This study of Enhancement of Materials Surface Properties via Surface Coating Process: Polymer Substrate in Silica Sand Bath aimed to introduce new concept of thin film coating using silica sand bath.

The method used in this study is by using silica sand bath to form the coating on the polymer. For this study, polypropylene was used as the sample. The polypropylene was immersed in the silica sand. The silica sand was put into the pocket of a ceramic which is brick clay then it was closed with another clay brick. The working temperature is 170°C and it took 4 hours to conduct the experiment. The results were compared by observed the microstructure of the sample using optical microscope by using Field Emission Scanning Electron Microscopy (FESEM).

Based on the results obtain, we can see the different in both testing. The optical microscope shows the shiny surface of the polypropylene which indicates the presence of silica sand. The FESEM testing indicates the image of rough surface of the sample which shows the layer of the silica sand on the surface. The XRF analysis was also used to compare the composition of the surface and the composition of the common silica sand.

As a conclusion, the objectives of this study were achieved which are to establish a “new” technique of polymer coating using silica sand bath and to characterize and analyse polymer’s surface properties after the coating process.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Polymer is the most common substance that has been used in industry today since it has low weight and it is easy to process. However, it has low strength and low surface hardness. Therefore, layers had been applied to the polymer to enhance the mechanical properties. The layer that applied to cover the surface of an object is known as coating. It is usually referred in aspect of protection. In most cases, coating improves the surface and mechanical properties such as the strength and the hardness of the substance.

For this study, Polypropylene (PP) is used as the sample. The usage of polypropylene is very wide in variety of application. It includes in automotive component, stationary, containers, laboratory equipment, textiles and event packaging. Most of the applications of PP are related to our daily life application.

Sand bath is one of the coating processes which made from a container that filled with heated silica sand. There are few studies that apply silica sand bath in coating process. However, the silica sand bath will be used in this study to coat the polymer. The surface property of the polymer is expected to improve.

1.2 Problem Statement

For normal heat treatment, alteration of microstructural and property changes was involved in the bulk of material or component which is through hardening. However, it is not needed to go through the harden parts of it because of the lack of the necessary toughness. For these applications, a small surface crack could propagate rapidly though such a part and cause total failure. For most cases, alteration of only surface properties of a part is desirable which is by case hardening.

However, case hardening usually applied to the metal. For polymer, coating of surface is widely used nowadays. Polymer coating can be applied using different techniques such as, dispersion coating, extrusion, and solution application. However, some of the techniques require high fabrication cost due to the multi-step procedure.

Therefore, this study will be focusing on the silica sand bath of the polymers. This is because the silica sand bath have a low fabrication cost. The surface of the polymer is expected to improve after the coating of the polymer take place on the surface of it.

1.3 Objective

The objectives of this study are:

a. To establish a "new" technique of polymer coating using silica sand bath.
b. The study also to characterize and analyse polymer's surface properties after the coating process.

1.4 Scope of Study

The following scope of study will be focused in order to obtain the objective. The literature review on silica coating on polymer will be the reference for this study. The literature review will determine the parameters. The important parameters will be identified. For this study, the main parameters are temperature of the experiment took place and the time for the process to occur.

In order to obtain the result for this experiment, several experiments need to be conducted. The result obtained will be compared for each of the experiment. The samples will undergo the microscopic examination. Besides that, the samples also had undergoes Field Emission Scanning Electron Microscopy (FESEM). This will enable us to compare the properties of the polymer surface before and after the sample undergoes silica sand bath. The Energy Dispersive X-ray (EDX) which gave out the composition of the sample also has been conducted.

CHAPTER 2

LITERATURE REVIEW

2.1 Surface Modification

On strategy in prolonging the lifetime of polymer, a material is used to improve their surface mechanical properties [1]. In our daily lives, polymers are widely used because of their functionality, flexibility, low cost and lightness in weight. Thus, long term performance is required. One of the methods to increase their long term performance is by improving the mechanical properties at the surface. Hard coating processes such as sputtering, vapour deposition, spraying and plating are used to improve the mechanical properties at the surface of material. However, due to multi-step procedure they possess the high fabrication cost. Therefore, they are using one-pot process which is based on preferential of segregation fed into the system in tiny amount. The surface showed prominent characteristics such as transparency, antioxidant properties and surface hardness.

Physical hardening is a reversible process that may influence the long-term performance of a material [2]. Surface modification technique can transform the polymer into high valuable product. The surface treatment such as modification technique by corona, plasmas, photons, flame and ion beams will alter the chemical and physical properties of the polymer surface. The technique used to get the surface hardening of the polymer is by using plasma polymerization [3]. Plasma polymerization is a unique technique for modifying the polymer surface by depositing a thin polymer film. This technique is a very complex process. It is an advance surface modification which can also increase the surface hardening of the polymer.

2.2 Silica Coating

2.2.1 Importance of Coating of Polymer

Polymer coating is often applied for protection purpose. Wear and scratch resistance are mainly improved by coatings. Coated polymers offer a wide field of applications. The most popular coated polymer is amorphous thermoplastic. The material with high transmission grades and has transparent coating give alternative to the glass application. However in this study, they used titanium nitrate and alumina (TiN and Al_2O_3) as their coating materials and applied to the different polymer. They are using pulse technique and High Iron Sputtering (H.I.S) ^{TM}. This technique also produced good adhesion and smooth surface were obtained for these coatings [4].

The other importance of the coating of polymer is to get smooth and uniform coat. Smaller filler (nano-sized) particles may increase polymer-filler interactions and therefore it will enhance the mechanical properties of the host polymeric coating [5]. The nano-sized particles not only give the uniform and smooth coat, it also can enhance the mechanical properties of the polymer due to its size. In previous study, dry surface treatment (DST) is one of the methods that can fulfil the criteria. At low coat weights, a crucial requirement for obtaining a uniform and smooth coating with a sufficient covering ability is an even distribution of particles of a fine particle size, since large particles require longer time to flow sufficiently and level out. The dry surface treatment employs the electrostatic deposition of dry, fine-sized particles and a thermomechanical surface treatment to fix the coating layer onto the substrate and to smoothen the coating surface [6].

2.2.2 Coating using Silica

Various type of coating is available in the industry to coat the polymer. Coating using silica is one of them. Some advantages in using the silica coating is, silica is chemically inert and optically transparent [7]. It is very important to have a coating that is chemically inert. It is to ensure that the coating will not react with other element when it is applied. Besides that, optically transparent is very important so that the coating will alter the apparent of the polymer.

In another study, in order to increase the mechanical protection such as scratch resistance and get chemical protection from the environment during processing, silicon dioxide (SiO_2) layer has been deposited at the polymer. Silicon dioxide possesses excellent physical and chemical properties, such as transparency from ultraviolet to infrared, good thermal stability, chemical inertness, wear and corrosion resistance and low gas permeation [8].

2.2.3 Experimentation Procedure of Coating using Silica

Based on the studies conducted, there are several methods used to coat the polymer using silica. Most of the methods are using nanoparticle. Some of the methods are deposited the silica in and electron cyclotron resonance (ECR) and by using hybrid coating.

In the study of using the ECR, the amorphous polyester material, AryLite^{TM} which has high glass transition temperature was used. The samples were heated at temperature below 50°C before SiO2 deposition in ECR plasma reactor. During the process, the gas flow of N2O, SiH4, and H2 gas mixture were keep constant. Deposition was performed for 13 minutes setting magnetron power to 400 W. Samples were heated at 50°C under hydrogen flow for 5 minutes before SiO2 deposition. Films were purged under nitrogen flow for 5 minutes at the end of the treatment [8].

The other study which uses the hybrid coating is a very complicated process. The modified nano-sized particle is mixed with colloidal silica; suspensions of fine amorphous, nonporous, and typically spherical silica particles in a liquid phase [9], dispersion with ethanol and propyl methacrylate, and a silence-coupling agent. It has been heated for 4 hour. After evaporation of the solvent, the coating were cured by the exposure of UV-light under nitrogen atmosphere [10]

2.2.4 Result of Coating using Silica

In the paper that using ECR method, the polymer surface has improve the adhesion properties. The increase of surface polarity enhances the adhesion of silicon dioxide deposited by ECR. The improvement of adhesion is associated with the present of SiO_2 [8]. The hard coating resulted in variation of young's modulus and hardness. Figure 1 show the result from nano-indentation test:

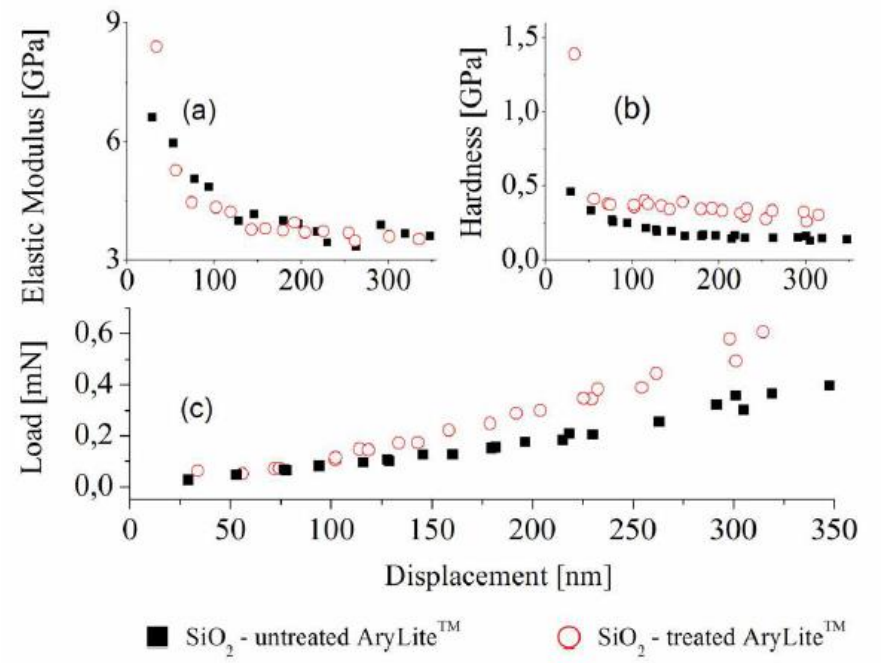


Figure 1: Young's modulus (a), hardness (b), and load vs. depth (c) of untreated and treated AryLiteTM coated with SiO2 layer [8].

As for the hybrid coating, hardness and elastic tests were done to test the influence of coating thickness on the mechanical properties. The good agreement of the various curves based on the test and data obtained shows the reliability of load and depth sensing indentation for the tested coating with a different thickness [10]. Thus the mechanical properties were found that it is not dependence on the thickness of the coating.

CHAPTER 3

METHODOLOGY / PROJECT WORK

3.1 Research Methodology

To conduct the study on Enhancement of Material Surface Properties via Surface Coating Process: Polymers Substrate in Silica Sand Bath, the understanding of the basic concept of the project is crucial. It is important to understand the rationale behind the study and identify the problem. In this study, the main objective is to introduce new method to improve the surface properties of the surface of the polymer by using silica sand bath. In polymer coating, the use of silica sand bath is a new technique. Therefore the study of the polymer coating using silica sand bath had been widen and fixed into the other study which will be discussed in the next step.

The next step is to carry out the literature review. Surface modification and silica coating are the properties that related to this study. Thus, we can study other people work on the related field and take it as our reference in conducting this project. The most important things in doing literature review is to obtain the parameters that are needed to conduct our experiment. Besides that, it is very important to ensure the proposed methodology fits the literature review. Figure 2 shows the overall flow of the study.

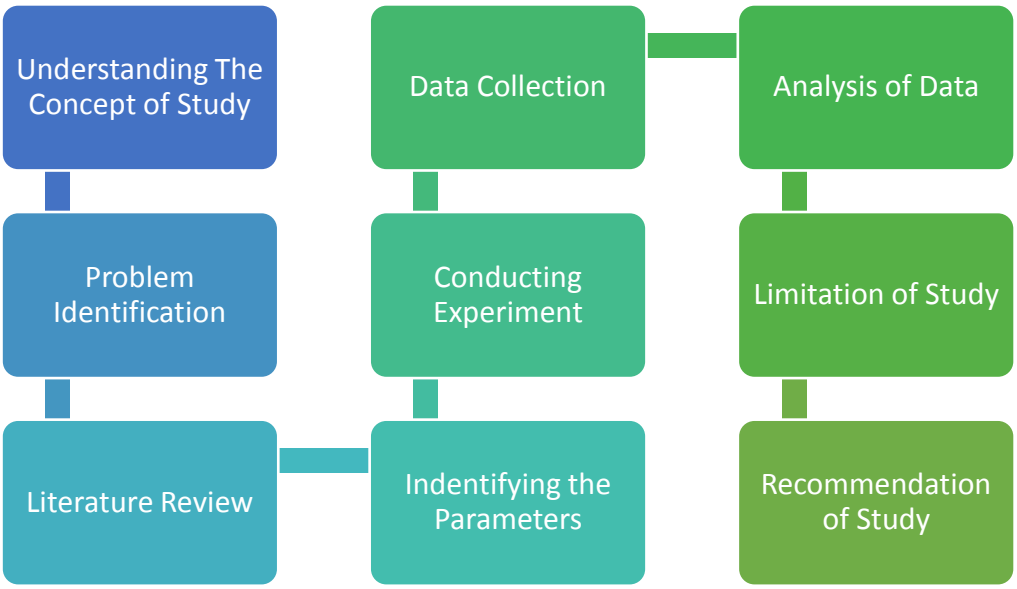


Figure 2: Overall flow of the study on Enhancement of Material Surface Properties via Surface Coating Process: Polymer Substrate in Silica Sand Bath.

3.2 Experiment Methodology

The experiment was conducted after all the parameters had been figured out. For this case, the two main parameters that will be controlled are temperature of the oven and time taken to heat the specimen. The temperature estimated for the process to occur is at 170°C. The experiment then was conducted at a period of 4 hours. The silica sand with the size of 212µm was used as the coating mechanism. The procedure for the experiment is as below:

1. The ceramic container with a pocket as per Figure 3 was filled with silica sand with the size of 212 µm until the silica sand cover half of the hole.
2. The specimen which is polypropylene with sizing of 30mm x 25mm x 4mm was placed into the ceramic pocket. It is then was covered with the silica sand and the pocket was closed with ceramic cover.
3. Set the temperature at 170°C then pre heat the oven for 10 minutes.
4. Put the ceramic container into the oven. Leave the specimen in the oven for 4 hours.

Before the experiment is conducted, the specimen was weighted and the surface physical properties of the sample were observed. The surface was observed by microscope and by using Field Emission Scanning Electron Microscope (FESEM). The data obtained such as picture of the structure of the specimen and the Energy Dispersive X-ray (EDX) result from the test will be compared later. The analysis of the results will be conducted to verify the objective of the experiment.

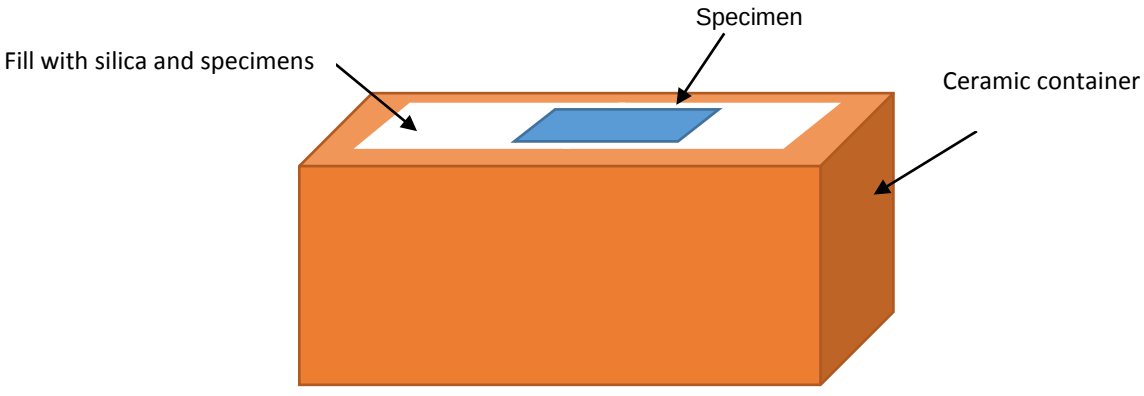


Figure 3: Sketch of ceramic container and substance inside it

Graphical procedure before the heating process.

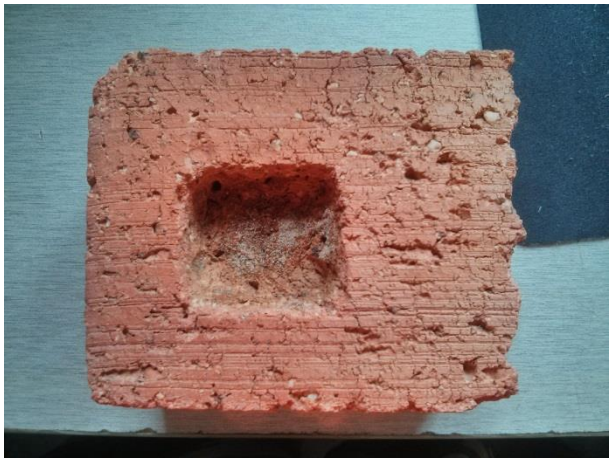


Figure 4: Empty ceramic container with a pocket

Figure 5: Half of the ceramic container filled with silica sand

Figure 6 : The polypropylene is placed at the centre of the pocket

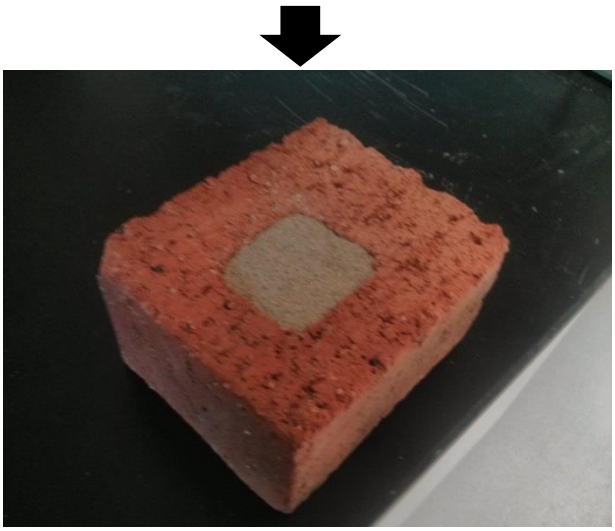


Figure 7 : Fill the rest of the pocket with the silica sand

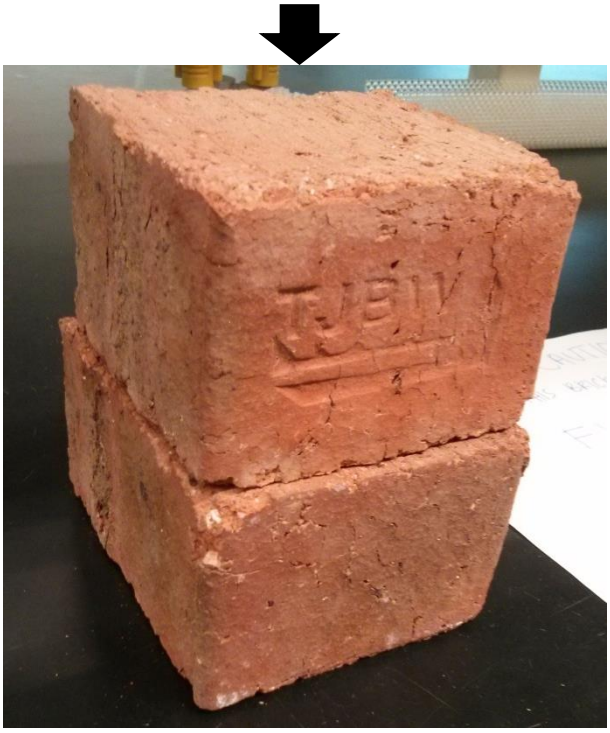


Figure 8 : Closed the pocket with another block of ceramic

3.3 Materials and Tool/Equipment

3.3.1 Materials

- Polypropylene
- Clay brick
- Tronoh Silica Sand. Table 1 below shows the composition of the silica sand.

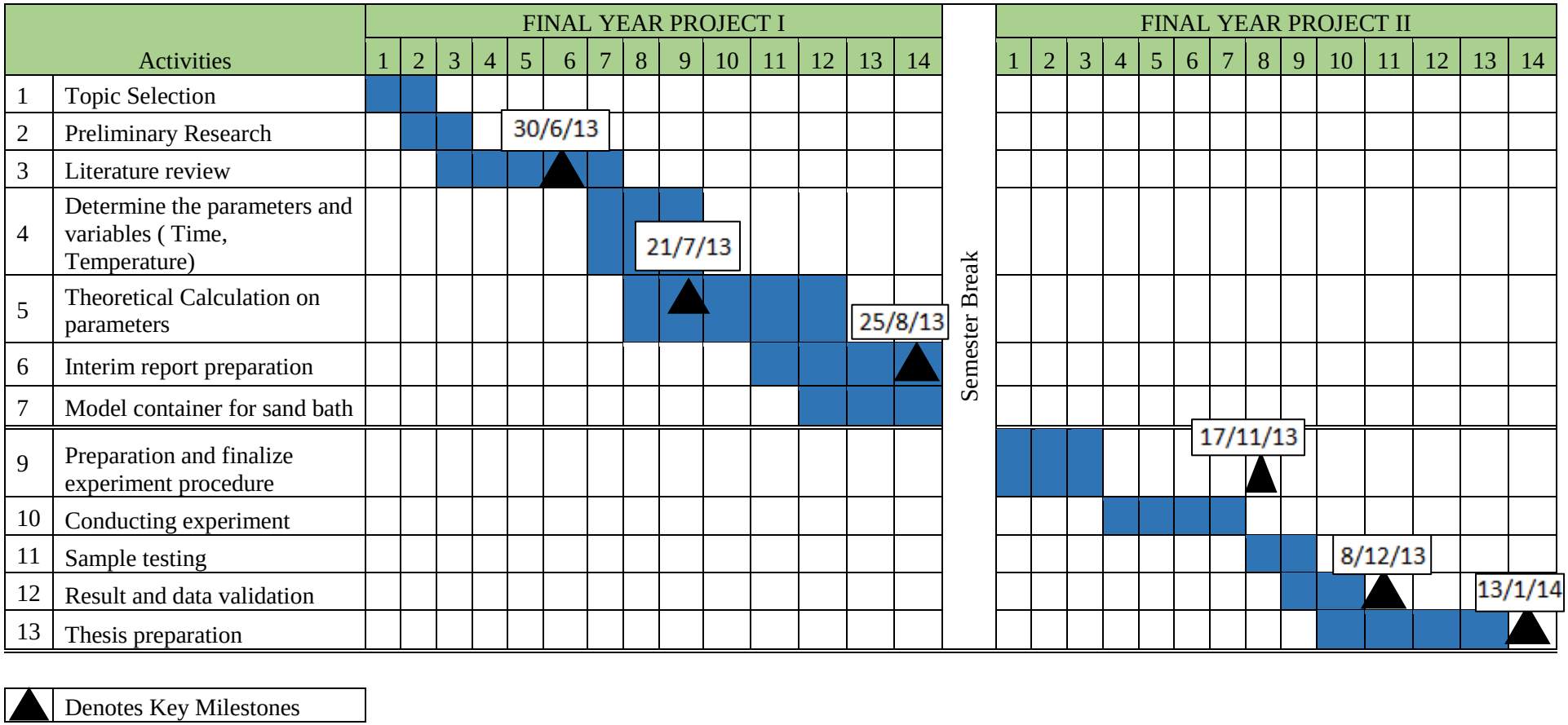
Table 1: XRF's analysis results on chemical composition of silica sand quote from "Characterization and Properties of Iron/Silica-Sand-Nanoparticle Composites" [12]

Al_2O_3 (wt.%)	SiO_2 (wt.%)	P_2O_5 (wt.%)	K_2O (wt.%)	CaO (wt.%)	TiO_2 (wt.%)	Fe_2O_3 (wt.%)
2.99	95.22	0.77	0.095	0.139	0.16	0.121

3.3.2 Tools and Equipment

- Furnace / oven
- Field Emission Scanning Electron Microscopy (FESEM)

3.3 Gantt Chart



CHAPTER 4

RESULTS AND DISCUSSIONS

In order to conduct the experiments, the time taken to reach the process temperature needs to be obtained. The equation for the calculation is as below:

$$E_{in} = mc\Delta T_{Brick} + mc\Delta T_{Silica\ Sand} + mc\Delta T_{Polypropylene}$$

Data provided:

Mass of Brick : 2.39kg

Mass of Silica Sand : 0.025kg

Mass of Polypropylene : 0.0025kg

Energy rate of furnace: 1.8 kJ/s

Required Temperature : 170°C

Initial Temperature : 23°C

Specific Heat Capacity of :

- Brick: 0.79kJ/kg.K
- Silica Sand : 0.83kJ/kg.K
- Polypropylene : 1.8kJ/kg.K

E_{in} = (2.39kg) (0.79kJ/kg.K)(170-23)°C + (0.025kg)(0.83kJ/kg.K)(170-23)°C +

(0.0025kg)(1.8kJ/kg.K)(170-23)°C

= 277.55kJ + 3.050 kJ + 0.662kJ

= <u>281.262kJ</u>

Therefore, the time needed for this furnace to supply 281.262kJ of heat to increase the temperature of brick, silica and polypropylene from 23°C to 170 °C is determined from

$$\Delta t = \frac{Total\ energy\ transfered}{Rate\ of\ energy\ transfer} = \frac{E}{\dot{E}} = \frac{281.262kJ}{1.8kJ/s} = 156.257s = 2.61\ minutes$$

∴ The time needed is about <u>2 minutes and 37 seconds</u>.

Therefore, the experiment period of 4 hours is valid since it only takes almost 3 minutes for the polypropylene to obtain process temperature before siliconizing process occurs.

In this section of study, it will discuss on the result from the experiment conducted. It is divided into 4 parts which are:

I. The effect on the sample at different temperature

II. The microstructure of the sample using optical microscope

III. FESEM testing and analysis

IV. EDX testing and analysis

4.1 The effects of the samples at different temperature.

The melting temperature of the polypropylene is between 165°C to 175°C. However, in order to see the effect of polypropylene in the silica sand bath above and below the melting temperature, few experiments were conducted. The figures below show the results from the experiment.

Figure 9: Sample at temperature 150°C

Figure 10: Sample at temperature 170°C

Figure 11: Sample at temperature 200°C

As stated above, the melting temperature of polypropylene is between 165°C to 175°C. Therefore, the experiments were conducted to observe the effect on the polypropylene in silica sand bath for temperature below melting temperature, at melting temperature, and above melting temperature of polypropylene.

At 150°C, as shown in Figure 9, there is nothing changes to the surface, colour and size of the polypropylene. The properties of the sample remain the same although it had been undergoes heating process in the silica sand bath. There is no coating of silica sand found at this sample.

For Figure 10, the temperature used is at 170°C which is at the working temperature of the polypropylene. The sample is almost coated with the silica sand. The layer produced is irregular shape, porous and brittle. The shape of the sample also shrink compared to the original shape of the sample. This is the best sample for the silica sand coating for this study.

The last one is the sample at 200°C which is shown in Figure 11. The colour of the sample becomes brownish. The shape is irregular and it shrinks compared to the original sample. A part from that, the silica sand is attached to the sample. Although silica sand is found attached to the sample, however, the changes of the shapes and the colour is slightly run off from the objective of this study which is to enhance the surface of the polymer instead of to change it.

The weight comparison of the sample before and after siliconizing process is measured in order to measure the weight of the coating layer produced. The analytical balance is used to measure the weight. The values are summarised in the Table 2 below:

Table 2: Table of sample with different temperature and weight of the sample

Samples at temperature (°C)	**Weight (g)**
Before siliconizing	2.573
150	2.573
170	8.925
200	1.796

4.2 The microstructure of the sample using optical microscope

As discussed in the previous section, the best sample for this study is the sample that undergoes the experiment at heating temperature of 170°C. Therefore, the cross section of the sample was observed under the optical microscope.

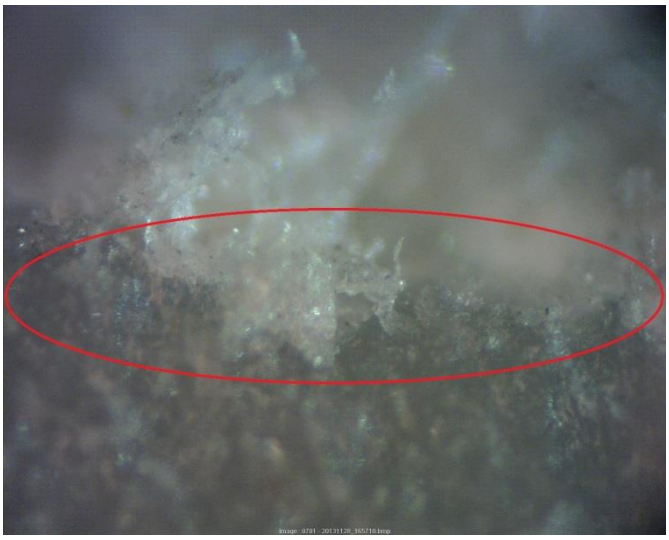


Figure 12: Upper side of coating layer

Figure 13: Lower side of coating layer

Figure 12 and Figure 13 shows the upper and lower side of layer at the cross sectional area of the sample. The oval red line in the picture indicates the layer of silica at the polypropylene surface. The bond between the silica and the polypropylene had form the layer.

For Figure 14 and Figure 15 below, it shows the comparison of the surface of the polypropylene before and after the siliconizing process.

Figure 14: Before siliconizing

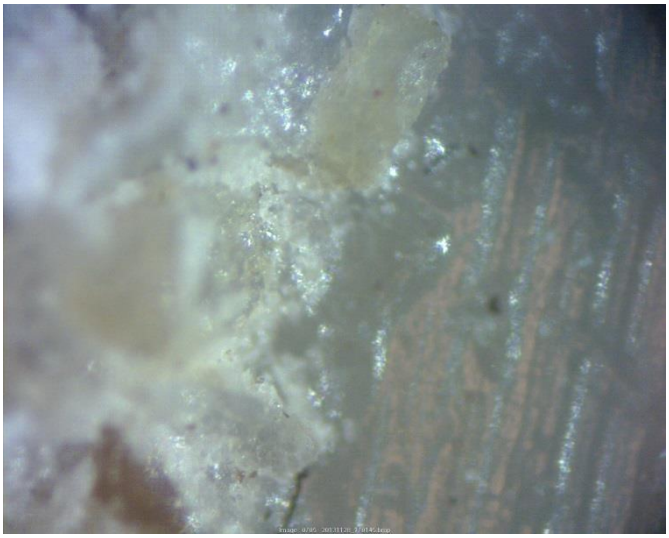


Figure 15: After siliconizing

Based on the figures, there is shiny part on the surface of the polypropylene in Figure 15 compare to the Figure 14. It is believed that the shiny surface of the polymer is due to the presence of silica. The optical microscope only gives the magnified image of the layer. Therefore the further analysis using Field Emission Scanning Electron Microscopy (FESEM) will be discussed in the next section.

4.3 FESEM testing and analysis

Field Emission Scanning Electron Microscopy (FESEM) was selected as the testing method because it produces clearer image with the resolution higher than the conventional Scanning Electron Microscopy (SEM). For this study, the polypropylene samples had undergone FESEM testing before and after the siliconizing process occur. The Figure 16 and Figure 17 are the images of polypropylene without coating at magnification 100x and 500x.

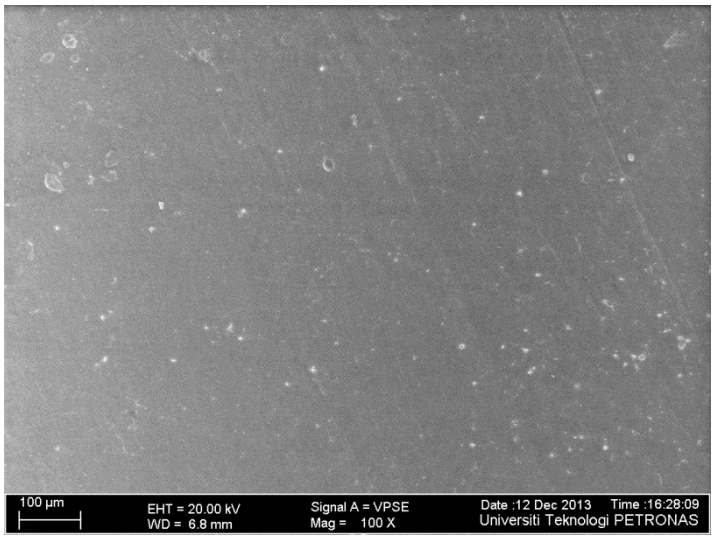


Figure 16: Morphology of polypropylene surface before coating. Magnification: 100x

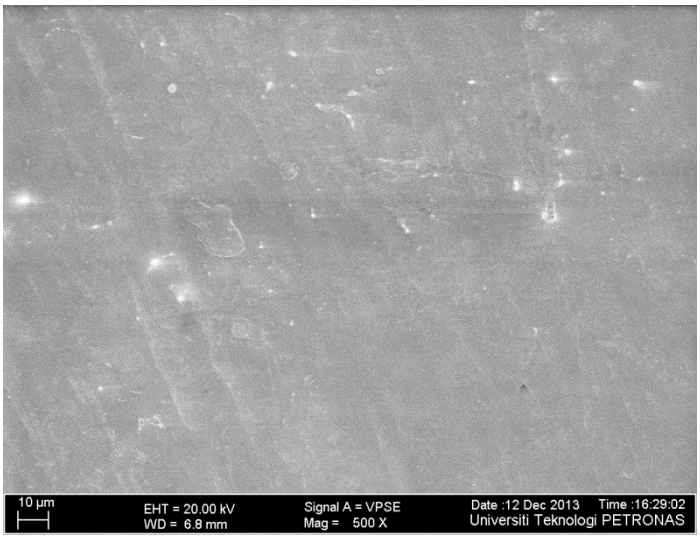


Figure 17: Morphology of polypropylene surface before coating. Magnification: 500x

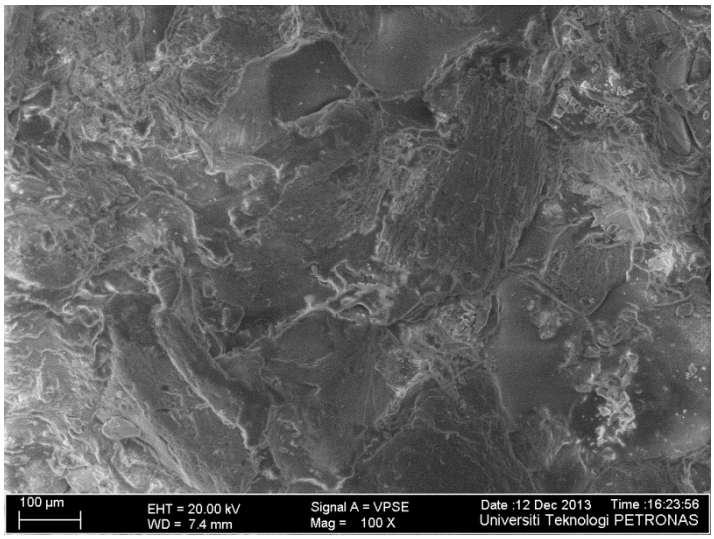


Figure 18: Morphology of polypropylene surface after coating. Magnification: 100x

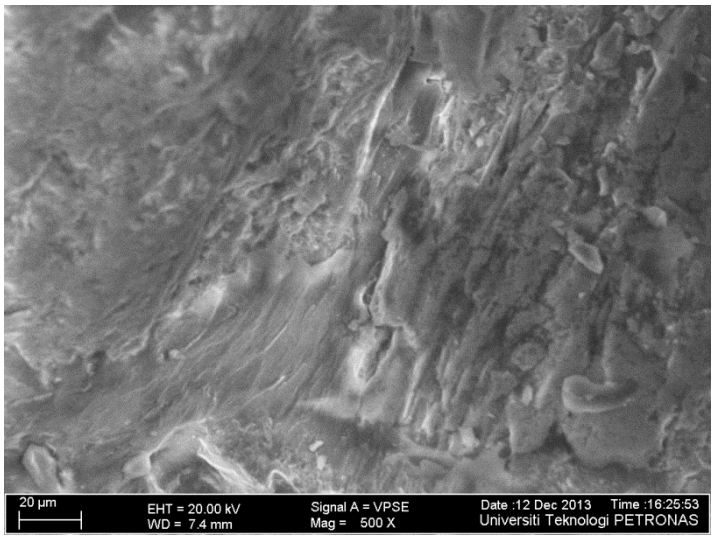


Figure 19: Morphology of polypropylene surface after coating. Magnification: 500x

Figure 18 and Figure 19 shown are the image of the polypropylene with the coating at magnification of 100x and 500x. Comparing the image before and after the surface coating, there are really huge different on both image. The image in Figure 16 and Figure 17 show a clearer and smooth surface of the sample. While in Figure 18 and Figure 19 show a slightly rough surface of the sample. Therefore, in order to get the smoother finishing after the siliconizing, the smaller size of silica should be used.

4.4 EDX testing and analysis

In order to know the element and chemical composition of the sample surface before and after coating, Energy Dispersive X-ray (EDX) is used. The result shown for the EDX testing is usually in the form of graph and composition table.

Before Coating

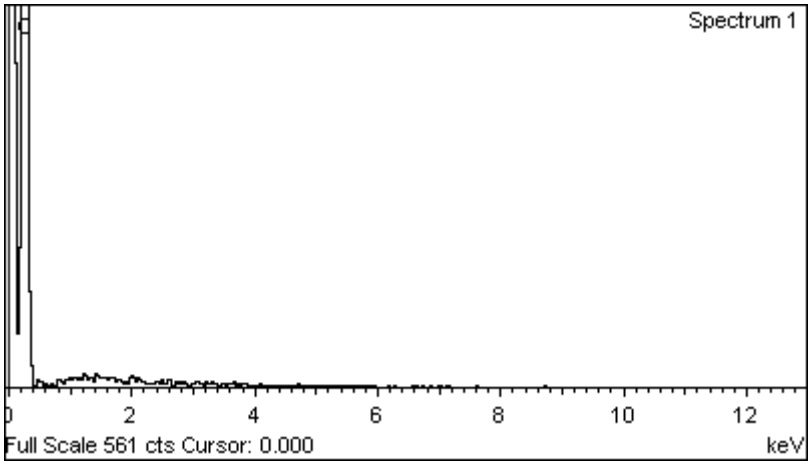


Figure 20: EDX result before coating

Table 3: Chemical composition of the sample before coating

Element	**Weight %**	**Atomic %**
C K	100.00	100.00
Total	100.00	

Figure 20 EDX line profile is higher at the Carbon (C). In Table 3 also shows the composition of carbon detected at the sample's surface before the experiment is 100%. This is because the basic composition of polypropylene is from carbon. Thus, this result will be the benchmark to compare the composition of the sample's surface after siliconizing process occurs.

After coating

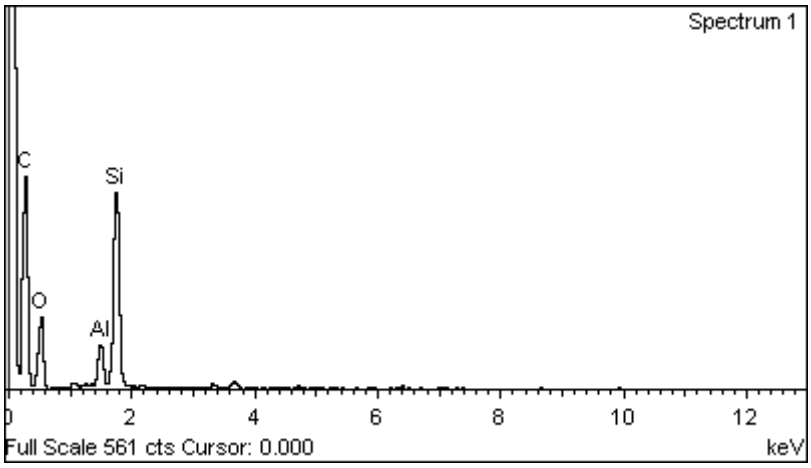


Figure 21: EDX result after coating

Table 4: Chemical composition of the sample after coating

Element	**Weight %**	**Atomic %**
C K	63.08	71.95
O K	27.18	23.27
Al K	1.56	0.79
Si K	8.18	3.99

For Figure 21, the EDX line profile shows several line which are Carbon (C), Oxygen (O), Aluminium (Al) and Silicon (Si). For Table 4, it also shows the same result. The composition of the results present is consistent with the composition of the silica sand shown in the Table 5 below. Therefore, compare with the qualitative of the both composition, it is proven that the sand grain made the bonding and forms the coating layer with the polypropylene.

Table 5: XRF's analysis results on chemical composition of silica sand quote from "Characterization and Properties of Iron/Silica-Sand-Nanoparticle Composites" [12]

Al_2O_3 (wt.%)	SiO_2 (wt.%)	P_2O_5 (wt.%)	K_2O (wt.%)	CaO (wt.%)	TiO_2 (wt.%)	Fe_2O_3 (wt.%)
2.99	95.22	0.77	0.095	0.139	0.16	0.121

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

As a conclusion, the objectives of this study were achieved. The first and the main objective which is to establish a "new" technique of polymer coating using silica bath was done by the methodology stated in this report. However, there are limitations that need to be figure out in the other study. Not only that, this study also able to characterize and analyse polymer's surface properties after the coating process. The result from the optical microscopic and FESEM testing shows the characteristic of the surface coating of the polypropylene sample. The rough surface of the sample obtained from the FESEM shows that more study need to be done.

5.2 Recommendations

A part from that, since this is the early study for the enhancement of polymer surface using silica sand bath, some mechanical properties cannot be tested. Although the most important parameters had been obtained which are the working temperature which is 170°C and the time taken for the experiment to take place which is 4 hours, there are many more parameters need to be discover. Thus, further research need to be done. The recommendation of the study and improvement is needed to improvise this study.

From the FESEM image obtained, it shows the rough surface area of the coating obtained due to big size of silica sand used which is 212μm. Thus, some improvement can be done by using the silica sand which is smaller than the size that has been used in this study. Not only that, the coating of the polymer is also brittle. Therefore, the mechanical testing such as scratch test cannot be done for this study due to that factor. In order to get better bonding between the silica sand and the polymer, the smaller size of silica sand should also be used.

The experiment also took a long time for the siliconizing process to occur. It is proposed for the silica sand to undergo acid treatment first as it will act as catalyst. Therefore the time taken for the experiment to take place will be cut off.

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