SILICONIZING OF THERMOPLASTIC VIA SILICA SAND BATH

By

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS in partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (MECHANICAL)

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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.

MUHAMMAD HAMIZAN BIN RAMLI

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ABSTRACT

Siliconizing is one of the coating processes that produce a thin film layer on the surface and improve the surface properties of the polymer. The objectives of this study are to introduce new surface coating method for thermoplastic material and to establish an optimum siliconizing parameters in producing uniform thin film coating onto thermoplastic polymers surface by using the silica sand bath. The siliconizing process was conducted by using only acrylic plate as the substrate, silica sand and brick as container. The samples were immersed in silica sand and heated in furnace at three different temperatures: 100°C, 130°C and 160°C for duration of 4 hours. In this study, the microstructure and surface properties of the samples were characterised and analysed using FESEM and EDX in order to look in the difference before and after the experiment. The evaluation focused on microstructure and analysis of the coating layer formed at the surface of the sample. After experiment, there is coating layer produced at heating temperature of 130°C and 160°C. Whereas, there were no physical changes observed on the surface of the sample at heating temperature of 100°C. Based on the result obtained, the microstructure of siliconized samples were found slightly rough compared to the original surface which is smooth and shiny. The XRF analysis of silica sand is used to compare the element composition of the surface and element composition of common silica sand. From the study that have been conducted, it was found that heating temperature of 160°C for 4 hours are suitable parameters for siliconizing of acrylic via silica sand bath.

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CHAPTER 1 INTRODUCTION

1.1 Background of Study

The application of polymer in the industry today is very widely used. This is because polymer has low weight and easy to process. However, one of the problems when using polymer is that it has low surface hardness. It is desirable to improve the hardness and scratch resistance of the thermoplastic for prolonging the life time. Therefore, layers had been applied to surface of polymer in order to enhance its hardness, scratch resistance and wear resistance. This layer is known as coating. Nowadays, there are many coating techniques used for polymer. Many researches and studies had done by researcher in order to improve the coating process of polymer for the application in the industry.

In this study, acrylic is used as the sample for siliconizing process. Acrylic can be used in applications of signs, window glazing, design, architecture, furniture, car window and boat. One of the advantages of using acrylic is that it has low density therefore it has light weight compared to glass. It also has wide variety of color and finishes. Another advantage of using acrylic is that it has excellent weather and UV resistance which make it very suitable to use for outdoor application.

1.2 Problem Statement

Nowadays, coating of polymers is normally performed by painting mainly for decoration purposes. The coatings of polymers by painting do not improve the surface properties of the polymer which are the scratch resistance and hardness. Other than painting, solgel coating also has been use in polymer coating. Solgel coating is considered the best coating process of polymer because it can improve the scratch resistance and hardness of the surface. However, this process requires high fabrication cost due to the technology involved is very expensive.

There is need to improve the surface properties of polymer by siliconizing process. Siliconizing is one of the coating processes that produce a thin film layer on the surface and improve the surface properties of the polymer. For siliconizing process, silica sand is used as the coating material for the surface of polymer. Siliconizing is considered to use for coating of polymer because there is abundant source of silica sand. This method need to be considered for further development in improving coating of polymer.

1.3 Objective

- 1. To introduce new surface coating method for thermoplastic material.
- 2. To establish an optimum siliconizing parameters in producing uniform thin film coating onto thermoplastic polymers surface by using the silica sand bath.

1.4 Scope of the Study

- 1. Understanding the mechanism of siliconizing process.
- 2. Determine the properties for the sample and coating material for siliconizing process.
- 3. Comparing the surface properties of sample before and after siliconizing process by Field Emission Scanning Electron Microscope (FESEM).

1.5 Feasibility of the Project within the Scope and Time Frame

Fourteen weeks have been allocated to FYP 1 for the preparation of the first phase of this research. Project work and preparation will begin on week 10 after the proposal is approved. However, research will be continuously done throughout the 14 weeks. As for the experimental activity which will be done in May until September, proper planning should be done which involves lab and equipment booking. The final week of phase one of this project was allocated in order to generate good insights of the experimental activities. Referring to the Gantt chart planned, 14 weeks is highly feasible for the first phase of the project.

For the second phase of this project, it will be begun on the May which involves the experimental and testing of the sample. The author aims to complete all the experimental and testing activities by week 7, and complete analyzing the result by week 10. The last four weeks of FYP 2 will involve viva presentation and report preparation of the project.

CHAPTER 2 LITERATURE REVIEW

2.1 Surface Modification

In order to extending the lifetime of the polymer, coating is one of the techniques that been used improve the surface properties. Nowadays, polymers are widely used in the manufacturing process because of their functionality, flexibility, low cost and lightness in weight. Therefore, coating of the polymers is very important to ensure the long term performance. Some of the coating process that have been used in industry today are sputtering, vapour deposition, spraying and plating.

2.2 Silica Sand

Silica is minerals that found in the earth crust that composed of silicon and oxygen to form chemical composition of SiO_2 [1]. Silica is one of the most abundant mineral in the earth crust. Usually silica is found in crystalline state and rarely found in amorphous state.

Silica sand or known as industrial sand is a type of sand that has high level of silica at least 95% in its composition. The other minor composition in the silica sand is the metallic elements such as CaCo₃, MgO, CaO, Al₂O₃ and Fe₂O₃. Therefore, silica sand is suitable to use in the industry because of its composition. Some applications of silica sand in the industry are glass making, metal casting, chemical product, paints and coatings, filtration in water production and building products.

2.3 Silicone Hard Coating

Silicone (polysiloxane) hard coatings can provide high abrasion resistance and inertness to hostile environment. They consist of several monomers and other ingredients. The formulations of the coating are varies. Silicone coatings are solventborne coatings and some of the possible solvents are alcohol and glycol ethers. The application of silicone coatings are usually on nonmetallic surfaces, especially plastics. Silicone coatings also can improve the material surface optical properties as they have excellent light transmission. Silicone hard coatings do not have automatic adhesion to many plastic but polycarbonate and acrylic have tape adhesion without need to surface treatment, etch or primer coat.

The coatings cure thermally at baking schedules that vary with heat stability of the substrate [2]. Since there are many types of silicon coatings, this most data given will have large variances. Silicone coatings are solvent borne coatings. This means that if they are placed in heat immediately after coating, the higher area of coating strata will cure first, leaving solvent entrapped in the film and non-uniform film coverage will produce on the surface [3]. This will cause such possible problems as lower abrasion resistance, weaker adherence to the surface substrate, chemical resistance and loss of some durability.

2.4 Solgel Coatings

Solgel processing technology is used for thin films and coatings. Solgel process has become an alternative to chemical vapor deposition, sputtering and plasma spray. The solgel processes involving a solution or sol that undergoes a solgel transition. For solgess process, first step is choosing the right reagent which is a metal or inorganic compound such as tetraethylorthosilicate (TEOS). The other ingredients are alcohol and water. Ethanol uses as solvent for TEOS and water. The reaction of TEOS with ethanol and water will lead to hydrolyzation and polymerization. For coating process, the solution is undergoing solgel transition while the coating is on the substrate [4].

2.5 Experimentation of Coating Using Silica

From the study that has been conducted, one of the methods of coating using silica is hybrid coating. The preparation of silica-based hybrid coating by the sol-gel process requires careful control of the hydrolysis and condensation of silicon alkoxide. In this process, some of the parameters which are organic:silicon alkoxide ratio, water and solvent amount need to be controlled in order to regulate the interactions between the organic and inorganic component to produce a fine coating on the surface. The hybrid coating sol is prepared by mixing Diethylenetriamine (DETA), tetramethyl orthosilicate (TMOS), 2-propanol and acetic acid [3]. The formulation of all hybrid coating is given in Table 2.1. Then, the PC substrate is dip-coated in hybrid coating sols and cured at 135°C for 4 hour. The results of this experiment are shown in Table 2.2.

Sol Formulation	DETA (wt%)	TMOS (wt%)	2-Propanol (wt%)	H_20 (wt%)
R1	30	30	30	10
R2	30	30	20	20
R3	30	30	10	30
R4	15	15	35	35
R5	40	10	25	25
R6	10	40	25	25

Table 2.1. Formulation of organic-inorganic hybrid coating sol. [3]

Table 2.2. Average thickness and Vickers hardness values of coating product. [3]

Sol Formulation	Thickness (µm)	Vickers hardness (HV1)
R1	46	39 ± 12
R2	50	71 ± 25
R3	-	70 ± 20
R4	3	45 ± 2
R5	20	32 ± 10
R6	-	47 ± 10

CHAPTER 3 METHODOLOGY

3.1 Research Methodology

To conduct the study siliconizing of thermoplastic via silica sand bath, it is very important to understand the mechanism of siliconizing. It is also very important to understand the foundation of the study and problem identification. The main objective of this study is to introduce a new surface hardening process by using silica sand as the coating.

Many research works had been performed in order to have better understanding of siliconizing process. The understanding of siliconizing process is very crucial for conducting this study. This can be done by carry out literature review. The study on the experiments by other researchers had been conducted in order to understanding the basic concept of siliconizing.

After the parameters of the experiment have been figured out, the experiment is conducted. After that, the thermoplastic specimen is put in a ceramic container with silica sand. This container is placed inside a furnace for designated time and temperature. Later, the thermoplastic specimens underwent microscopic examinations and FESEM testing to get the result after the experiment. Both results before and after the experiment were compared and analysed. The final step was identification of the limits in the study and recommendations for further research. Figure 3.1 shows the flow chart of the research methodology of the study.



Figure 3.1. Research Methodology of the Study

3.2 Materials and Tools/Equipment

3.2.1 Materials

The materials used for conducting the study are:

- Acrylic plate (3x3x0.3 cm)
- Silica Sand
- Container: Ceramic brick

3.2.2 Tools and Equipment

Table 3.1 shows the tools and equipment used in order to conducting the study.

Tools / Equipment	Function				
Furnace / oven	To heat the acrylic with silica sand in				
	ceramic container.				
FESEM Machine	To analyze the surface microstructure of				
	the sample before and after siliconizing.				
Drilling Machine	To make pockets on the ceramic brick to				
	place silica sand and acrylic plate.				

Table 3.1. List of tools and equipment used

3.3 Project Activities

3.3.1 Sample Preparation

The samples used as the substrate for coating is acrylic plates. The plates were cut by dimension of 30mm x 30mm x 3mm as shown in Figure 3.2. Next, the specimen is weighed to in order to compare the weight of the sample after siliconizing process. Before placing the sample in the brick container, the surface of the sample must be cleaned in order to remove the impurities that can affect the siliconizing process.



Figure 3.2. Acrylic plate (3x3x0.3 cm)

As for the silica sand, the sand is filtered to fine size sand (Figure 3.3). The size of silica sand is very crucial to ensure the coating will produce on the surface of the specimen. The small size silica sand will be easier to attach on the surface of the specimen when it is heated in the furnace. Therefore, the sand is sieved by using sieve shaker (Figure 3.4) to obtain small size of silica sand which is about 200µm.



Figure 3.3. Sieved silica sand



Figure 3.4. Sieve shaker

As for the container to place the silica sand and acrylic plate in the furnace, a ceramic brick was used. Ceramic brick is considered to use as the container because it has high melting point therefore it can withstand high temperature during heating process. Apart from that, it is a good insulator where heat trapped inside will not easily escape. Two pockets have been milled on the ceramic brick by drilling machine as shown in Figure 3.5. This pockets are used to put silica sand and acrylic plate together in the furnace. Figure 3.6 shows the ceramic brick container with two pockets.



Figure 3.5. Milling process of the pockets



Figure 3.6. Ceramic brick container

3.3.2 Siliconizing Process (Heating)

In order to perform the siliconizing experiment, the brick pockets is filled up with sieved sand and the sample is immersed in the middle of the sand lump. The furnace used is Carbolite CWF 1300 Electric Furnace (Figure 3.7). The furnace is set to have working temperature of 100°C for 4 hours. The heating process is repeated with temperature of 130°C and 160°C.

3.3.3 Data Analysis of the Siliconized Sample

After the siliconizing, the weight of the sample is measured. The sample undergoes FESEM testing for further analysis on microstructure and its chemical composition by using Phenom Pro X FESEM Machine (Figure 3.8). All the data results can be referred in next chapter of Results and Discussion.



Figure 3.7. Carbolite CWF 1300 Electric Furnace



Figure 3.8. Phenom Pro X FESEM Machine

3.4 Experimental Methodology

Before conducting the experiment, the relevant parameters should be finalized. For this experiment, the parameters needed to be controlled are the temperature of the furnace and time taken to heat the specimen with silica sand. For the temperature of the furnace, the temperature must be below the melting point of the acrylic plate which is 160°C. Therefore the temperature of the furnace is set at three different values which are 100°C, 130°C and 160°C. The heating process of specimen with silica sand is done at a period of 4 hours. The procedure of experiment is shown as below:

- 1. The ceramic container with pockets is filled up with silica sand until it covers half of the pocket.
- 2. The specimen which is the acrylic plate with size of 30 mm x 30 mm x 3 mm was placed into the ceramic pocket.
- 3. Then, silica sand is poured into the pocket until it fully covers the pockets and specimen.
- 4. The furnace temperature was set to 100°C and preheats the oven for 10 minutes.
- 5. The ceramic container which contains silica sand and specimen was placed in the furnace.
- 6. Leave the ceramic container in the furnace for 4 hours.
- 7. The experiment is repeated with different furnace temperature of 130°C and 160°C.

3.5 Gantt Chart

No.	Activity/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic														
2	Literature Review and Analysis														
3	Preparation of Extended Proposal Draft														
4	Submission of Extended Proposal						Δ								
5	Proposal Defense														
6	Theoretical Calculation on Parameters														
9	Submission of Draft of Interim Report														
10	Submission of Interim Report														Δ



Process

No.	Activity/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Preparation of Experiment Procedure														
2	Experiment Conducting														
3	Submission of Progress Report														
4	Analysis of Experiment Result														
5	Pre-SEDEX														
6	Submission of Draft Report												Δ		
9	Submission of Dissertation (Soft bound)														
10	Submission of Technical Paper														
11	Oral Presentation														Δ

Milestone

Process

CHAPTER 4 RESULTS AND DISCUSSION

This section is further divided into four categories, which are:

- The samples before and after the siliconizing.
- Comparison of the weight of the sample.
- Surface morphology evolution of siliconized sample.
- EDX testing and analysis.

4.1 The Samples Before and After the Siliconizing

Before the experiment was conducted, the surface thermoplastic samples were ensure to be clean and free from impurities as this impurities can affect the adherent of silica sand on the surface. Figure 4.1 shows sample before the siliconizing process takes place. The average weights of the samples were 2.72 g. Figure 4.2, 4.3 and 4.4 show the result obtained from the experiments.

As shown in Figure 4.2, the sample is heated at a temperature of 100°C for duration of 4 hours. No apparent changes were observed on the surface of the sample. The properties of the sample remained the same although it had been underwear heating process in the silica sand bath. No silica sand particles adhered on the surface of acrylic plate. From the observation, it can be deduced that heating temperature of 100°C is not favorable for siliconizing of acrylic polymer.

As shown in Figure 4.3, the sample is heated with temperature of 130°C for the duration of 4 hours. Physical changes were observed on the surface of the sample. There is

slightly change in the weight of the sample which is 3.196 g. The silica sand particles were seen on the surface of acrylic. The coating produced on the surface is uneven. It was also observed that some part of the surface was not covered by silica sand particles. The silica sand particles also were weakly adhered to the surface and it can be removed easily from the surface.

As shown in Figure 4.4, the sample is heated at temperature of 160°C for duration of 4 hours. Some morphology changes were observed on the surface of the sample. The weight of the sample had been increased to 3.918 g. The silica sand particles were seen on the surface of acrylic. The coating formed on the surface are almost even. Almost all part of the sample surface are covered by silica sand particles. The adhesion of silica particles on the surface is stronger than the previous sample. However, there is slightly change in the shape of the sample. The shape of the sample becomes irregular comparing to its original shape before siliconizing process. This may due to the heating temperature of furnace as it is approaching the melting temperature of acrylic which is between 160°C to 165°C.

From the observations that have been made on three different heating temperature, the suitable and most likely the optimum temperature for siliconizing of acrylic polymer is 160°C. As temperature of 160°C is approaching the melting point of sample, the further increase in the heating temperature can affect the shape of the sample. The heating duration of 4 hours also found to be suitable for this process.





Figure 4.1. Sample before siliconizing

Figure 4.2. Sample after siliconizing at 100°C



Figure 4.3. Sample after siliconizing at 130°C



Figure 4.4. Sample after siliconizing at 160°C

4.2 Comparison of the Weight of the Samples

The weight of the sample before and after the siliconizing process is measured in order to measure the weight of the amount of adhered silica sand particles on the surface. The weight is measured by using analytical balance. The values are summarized in the Table 4.1.

Table 4.1. Weight comparison of sample at different heating temperature

Sample at	Sample at Weight of Sample After			
Temperature (°C)	Siliconizing (g)	Layer Produced (g)		
100	2.720	0		
130	3.196	0.476		
160	3.918	1.198		

4.3 Surface Morphology Evolution of Siliconized Sample

Field Emission Scanning Electron Microscopy (FESEM) was selected as testing to analyze the surface morphology of the sample before and after siliconizing process. FESEM testing was selected because it can produces clearer image with higher resolution compare to the conventional Scanning Electron Microscopy (SEM). In this testing, the surface features, size and shape of features and crystalline structure of the sample are observed and analyzed.

For the surface of the sample without the coating layer (as shown in Figure 4.1), the microstructure of the surface features with magnifications of 500x and 1000x are shown in Figure 4.5 and Figure 4.6 respectively. From the image produced, it was found that surface is almost smooth and clean.



Figure 4.5. Morphology of acrylic surface before siliconizing process. Magnification: 500x.



Figure 4.6. Morphology of acrylic surface before siliconizing process. Magnification: 1000x.

For the surface of the sample with the coating layer at heating temperature of $160^{\circ}C$ (as shown in Figure 4.4), the microstructure of the surface features with magnifications of 500x and 1000x are shown in Figure 4.7 and Figure 4.8 respectively. By comparing the morphology of the sample surface before siliconizing, there are a huge different at both images. From the image produced, the surface of the sample is found slightly rough and there is silica sand particles attached to the surface. However, there are some regions on the surface that are not attached by the silica sand. The size of the silica sand coating on the surface also measured (as shown in Figure 4.9). The size of the silica sand is about 150 μ m.



Figure 4.7. Morphology of acrylic surface after siliconizing process at 160°C. Magnification: 500x.



Figure 4.8. Morphology of acrylic surface after siliconizing process at 160°C. Magnification: 1000x.



Figure 4.9. Size of silica sand

4.4 EDX Testing and Analysis.

In order to know about elemental information and composition of the sample surface, Energy Dispersive x-ray spectroscopy (EDX) which is coupled with FESEM is used. Figure 4.10 shows the surface points where the EDX is performed on sample without coating layer.



Figure 4.10. Spectrum 1 and 2 shows the surface points of the sample where the EDX is performed.



Figure 4.11. EDX result for Spectrum 1

Table 4.1. Element present in Spectrum 1 with their respective weight%

Element	Element Name	Weight %
С	Carbon	47.0
0	Oxygen	53.0



Figure 4.12. EDX result for Spectrum 2

Table 4.2. Element present in Spectrum 2 with their respective weight%

Element	Element Name	Weight %
С	Carbon	48.4
0	Oxygen	51.6

From the EDX result at spectrum 1 and 2 as shown in Figure 4.11 and 4.12 respectively, the elements present on the surface of sample before siliconizing are mainly Carbon (C) and Oxygen (O). This shows that the basic composition of acrylic is C and O. The weight% of C and O at both Spectrums are shown in Table 4.1 and Table 4.2.

EDX testing also performed on the siliconized sample in order to identify elemental information and composition of the sample surface. The result obtained from this testing will be compared to the previous result. Figure 4.13 shows the surface points where EDX is performed on the siliconized sample at 160°C.



Figure 4.13. Spectrum 1 and 2 shows the surface points of the siliconized sample where the EDX is performed.



Figure 4.14. EDX result for Spectrum 1

Element	Element Name	Weight %
0	Oxygen	65.9
Br	Bromine	17.6
Si	Silicon	12.6
Fe	Iron	2.1
Na	Sodium	1.7

Table 4.3. Element present in Spectrum 1 with their respective weight%



Figure 4.15. EDX result for Spectrum 2

Element	Element Name	Weight %	
Si	Silicon	19.9	
0	Oxygen	70.5	
С	Carbon	5.7	
Al	Aluminium	3.8	

Table 4.4. Element present in Spectrum 2 with their respective weight%

From the EDX result at spectrum 1 and 2 as show in Figure 4.14 and 4.15, the elements present on the surface of sample after siliconizing are Carbon (C), Oxygen (O), Silicon (Si), Ferum (Fe), Bromine (Br), Sodium (Na) and Aluminum (Al). This shows that there are changes in the composition on the sample surface. By comparing with Table 4.5, the composition of element present is consistent with composition of silica sand. Therefore it is proven that silica sand grains diffuse and form coating layer on the surface.

			-			
Al2O3	SiO2	P2O5	K2O	CaO	TiO2	Fe2O3
(wt.%)						
2.99	95.22	0.77	0.095	0.139	0.16	0.121

Table 4.5. XRF composition of silica sand [8]

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

As the conclusion, siliconizing process is a simple process for coating the material. The application of silica sand as the coating material is suitable for coating because there is abundant source of silica sand. From the study that has been conducted, it was shown that the surface properties can be improved by coating using silica. It was shown that the temperature and time have an influential effect on the siliconizing process of acrylic specimen. The time of heating must be long enough for the coating to form on the surface of the sample. The temperature also must be controlled in order to prevent the sample from melting due to high temperature.

The outcomes of this study showed that there was coating layer produced after the experiment at heating temperature of 130°C and 160°C. The layer produced at 160°C was found to be almost even and more strong adhered to the surface compare to layer produced at 130°C. Almost all region of the sample surface are coated by silica sand. The study also reveals that heating temperature of 160°C for 4 hours are optimal parameters for siliconizing of acrylic via silica sand bath.

5.2 Recommendations

Simulation study should be performed in order to determine rate of heat transfer and the temperature inside the ceramic bricks can be done for more thorough analysis. It is also recommended that during the siliconizing, apply higher pressure when heating so that the silica will diffuse into the sample and binded instead of become outer case of the sample. Longer siliconizing time also can be used in order to increase the coating layer thickness. Apart from that, it is recommended to add in inert additives and catalyst in order to prevent sticking of grain to the sample, accelerate the process and also for dense and pore-free coating. Hardness and scratch test should be performed in order to evaluate the mechanical properties of siliconized. This is possible once the coating layer produced is dense, pore-free and has even surface.

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