Investigate the Dispersion and Suspension of Zinc and Silver Nano additives in Nano composites

by

Nursyazana binti Mohd Noor

Dissertation submitted in partial fulfilment of

the requirement for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

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Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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BACHELOR OF ENGINEERING (Hons)
(MECHANICAL ENGINEERING)

Approved by,

_____

(Assoc. Prof Dr. Hussain Hammud Ja'afer Al Kayiem)

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 acknowledgments, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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NURSYAZANA BINTI MOHD NOOR

ABSTARCT

Phase Change Material has a great potential for use in conjunction with thermal energy storage application, as storage medium. Many previous studies have been undertaken to investigate the feasibility of storing solar-thermal energy using Phase Change Materials and thus uses the energy generated to heat water, air, and oil for domestic and industrial purposes, during night. However, in the solar collector integrated with thermal energy storage, the storage unit is essential at times when only a very small amount of solar radiation or no radiation is received. Thus, the storage of thermal energy as latent heat of fusion has attractive features over the sensible heat as it has a high storage density as well as isothermal nature of storage process at melting temperature. Hence, the phase change from solid to liquid or vice-versa is preferable. Practically, Phase Change Material such as paraffin wax is very ideal and it is widely in usage. The only setback in the paraffin wax is the low thermal conductivity. To enhance the thermal properties, mainly the thermal conductivity of the Phase Change Material, Nano additives are one of the suggested approaches. This paper focus on the dispersion and suspension of Metal-based, Zn and Ag, nanoparticles in the paraffin wax to form Nano-PCM composition. Eight samples will be prepared consisting of different percentage of Nano additives in additional to sample for pure paraffin wax. The Nano particles dispersion will be investigated at middle level of the samples. The produced Nano composites are demonstrating better storing capability. Various researches have been conducted to explore the enhancement in the thermal properties of the combination of Nano additives into the Phase Change Material.

ACKNOWLEDGEMENTS

Alhamdulillah, after almost a year working on this project, at last the project is managed to be completed successfully. Hence, I aspire to take the opportunity to thank and acknowledge all the people involved from the beginning till the end of this project.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1 Background

Solar energy is an environmental friendly energy and the most abundant energy resources in the world. It has been utilized in many applications as well as supplement for numerous of energy requirements. In addition, the solar energy also can be used in the form of heat where for example, it has been used in solar water heating system to heat up water to be used by the consumers. This has been the most cost-effective way to heat up water for water heating system. Hence, to increase the efficiency of the solar water heater system, efficient thermal energy storage is vital. So, to have a system that could promise advantages like high heat storage capacity, small unit size and isothermal behaviour, the latent heat thermal energy storage system using Phase Change Material (PCM) could make the cut. Unfortunately, the system has a poor heat transfer rates during heat storage and recovery processes. Numerous studies have been taken and found that by producing a Nano-PCM composition, the problem with the latent heat thermal energy storage can be overcome. In addition, this paper also keen to investigate the settlement of the Nano additives throughout the mixture after many thermal cycles. We will observe the pattern of the settlement of both of the nanoparticles all over the PCM, paraffin wax.

1.2 Problem Statement

The PCM that is being used in this project is paraffin wax. The paraffin wax acts as the storage medium in the thermal energy storage. However, unfortunately, the paraffin wax has a low thermal conductivity. It is so sensitive to heat and temperature. So, to overcome this problem, Nano additives are proposed as one of a technique to improvise the thermal properties of the PCM.

1.2 Objective

The objective of the study is:

1. To investigate the dispersion of Metal-based nanoparticles in the PCM Nano composites.
2. To investigate the effect of the thermal cycles on the settlement of the Nano additives throughout the mixture of the Nano composite.

1.4 Scope of Study

Nano-PCM composition will be formed using an electrically ultrasonication device to improve the thermal conductivity of the PCM. The PCM used in this study is paraffin wax while Nano additives used are Zinc nanoparticles and Silver nanoparticles. This particular ultrasonication device is able to heat and vibrate simultaneously. Hence, the temperature of the mixture can be maintained at 70^{o}C while it is been mix well. All the samples had been ultrasonicate for nine hours in order to make sure that the Nano composites are mix well.

Then, the samples preparation process will proceed with thermal cycle process. During this process, all the samples will go through the melting and solidifying phases. The thermal cycles are being repeated for 200 times before the analysing phase takes place.

After completes with the preparation for all the samples, the samples now are ready to be further analyse. The analysis step is very crucial to enable the observation process of the pattern of the settlement of the mixture. The characterization techniques will be using the Field Emission Scanning Electron Microscopy (FESEM) will identify the shape as well as the size of the nanoparticles within the Nano composite. Next, the characterization techniques mention above will be repeated three times.

CHAPTER 2

LITERATURE REVIEW

2.1 Solar Water Heater

Solar water heater is a device which mainly used for heating the water. In is operated by sun, hence it is called as solar water heater. There are various industries, which require water temperatures of more than 50 degree or up to 70 degree Celcius. Solar water heater system possess many advantages includes it does not require any cost of electricity.it has also very low running cost as well as maintenance cost [12].

According to Tsakalakos 2010 with reference from Fang et al. 1970; Ghosh and Feng 1973; Lyons and Newman 1971; Mukherjee 1970; Reucroft et al. 1968, 1969, the first organic solar cells were fabricated in the late 1960s and early 1970s and often consisted of a single organic layer (e.g., tetracene) sandwiched between a low-work-function metal layer (aluminium) and a high-work-function metal (gold). However, the efficiencies of the solar cells were extremely low which are down to ~10^{-5}% according to Ghosh and Feng 1973. The researchers from different groups however still continue on searching a wide selection of materials, but unfortunately the efficiency is still low which is below 1% (Chamberlain 1983).

2.1.1 Active Solar Water Heater System

Active solar system comprises of storage device, an assembly of collectors, and working fluid which converts solar energy into thermal energy. An additional energy is used to solar input in order to accomplish the thermal energy transferred [13]. In addition, to circulate water or other heat-transfer fluids through the solar collectors, electric pumps, valves and controllers are usually used in the systems. They are usually more expensive

than passive systems but are also more efficient [14]. Active systems are usually easier to retrofit than passive systems because their storage tanks do not need to be installed above or close to the collectors.

2.1.2 Passive Solar Water Heater System

Passive solar system depends on heat driven convection to circulate water or working fluid throughout the system [12]. Thus, it does not need pumps to operate. The system naturally controls the circulation flow rate in phase with the radiation level. The warmer the water, the less dense it will be. The less dense water will rises to the top of system, displacing the colder water to the lowest point. Passive solar water heaters also known as thermosyphon systems are more reliable, easier to maintain, and possibly longer lasting compared to active solar water systems [15]. They can be built with inherent freeze resistance so they can be used in areas that are subject to extended periods of freezing temperatures.

Passive solar system can be cheaper compared to active solar system. However, it can also be less efficient [14].

2.1.3 Solar Water Heating System with Latent Heat Storage Materials

The efficiency of solar water heater as well as the outlet water temperature during the evening hours will increases along with the increases in the thermal conductivity of the solid-liquid phases of the materials.

Built in storage type water heater containing a layer of PCM filled capsules at the bottom as Figure 1 [25]

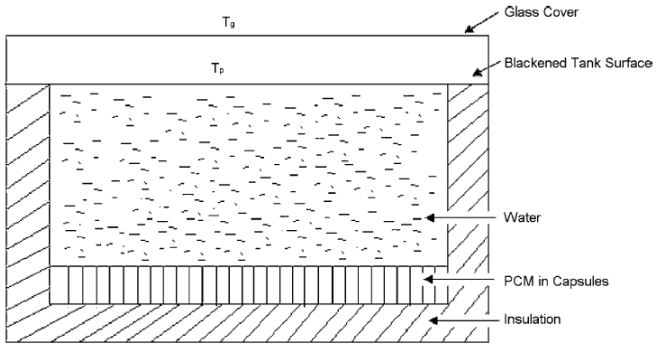


FIGURE 1: Solar Water Heater [25]

The water inside the tank will gets heated up during the sunshine hours. Hence, in turn transfers heat to the PCM below it. The PCM will then collects energy in form of latent heat before it melts. Without the presence of sunshine, the hot water will get cold, which then gains energy from the PCM [25]. The phase changing from liquid to solid of the PCM will releases energy.

2.2 Phase Change Material

Phase Change Material (PCM) are materials that under phase cycle. They melt and solidify at different temperature. One of most important criteria during the selection of the material is the conformable melting point and the high latent heat of fusion. The selection of the substances used is largely depends upon the temperature level of the application [17].

According to Sharma [18], the PCM to be used in the design of any thermal storage systems should pass desirable thermophysical, kinetics and chemical properties which are given in Table 1.

TABLE 1: Main Desirable Properties of Phase Change Materials [19]

Thermal properties	Suitable phase-transition temperature, High latent heat of transition, High thermal conductivity in both liquid and solid phases, Good heat transfer
Physical properties	Favorable phase equilibrium, High density, Small volume change, Low vapor pressure
Kinetic properties	No supercooling, Sufficient crystallization rate
Chemical properties	Long-term chemical stability, Compatibility with materials of construction, No toxicity, No fire hazard
Economic Properties	Abundant, Available, Cost effective

2.2.1 Paraffin wax

The paraffin wax as phase change material (PCM) is widely investigated as thermal energy storage material. Al-Kayiem [1] has reported the application of PCM in the thermal energy storage (TES) to utilize waste heat, space heating and cooling etc. have been increasing extensively in recent years.

The paraffins are waxes at room-temperature. These are hydrocarbons. As the number of C-atoms increases, the melting point will also increase. The normal paraffin of type C_nH_{2n+2} are a family of saturated hydrocarbons with very similar properties. However, the paraffins between C_5 and C_{15} are liquids while the rest are waxy solids. Paraffin wax is the most commonly used commercial organic heat storage PCM [20]. Paraffin waxes are cheap and have moderate thermal energy storage density but low thermal conductivity and thus require large surface area [16].

2.3 Nanoparticles

Nanoparticles have great potential to more effectively improve the thermal transport properties of HTFs than micrometer and millimeter sized particles. This is mainly due to the tininess of nanoparticles or other nanostructures, which is not only improve the stability and the applicability of liquid suspension, but also increases the specific surface are (SSA) [26].

Han et al. 2011 and Shi et al. 2013 have made some improvements by introducing metal and non-metal nanoparticles into the PCM and thus producing a mixture.

According to Wang et al. 1999, it is very likely that the motion of nanoparticles in the fluid will also enhance heat transfer. Shaikh et al. 2008 reported that latent energy storage capacity of carbon nanoparticles doped phase change materials (PCMs). In addition, Yu et al. 2009, has proven that the thermal conductivity of ZnO-EG nanofluids increases nonlinearly with an increase in volume fraction of the nanoparticles.

Thus, with this property of PCM, it can be used in many ways such as thermal energy storage where heat can be stored and be used later in future when it is needed. Besides, it is also very useful in providing thermal barriers or insulation, for example, in temperature controlled transport.

2.4 Nano composites

A Nano composite is a composite material in which at least one of the phase domains has at least one dimension of the order of nanometers.

However, Nano composites also refers to a multi-phase material where at least one phase has dimensions of the order of nanometers (e.g. nanoparticles) and one phase is a continuous phase (bulk phase e.g. polymer matrix) [27].

A composite material is a combined material between two or more components, purposely to achieve specific characteristics or properties that were not there before. The matrix which is the continuous phase, generally control the properties of the composites.

CHAPTER 3

METHODOLOGY

At preparing stage of mixture Nano additives and paraffin wax, thorough procedures must be followed as the process is the most crucial part of the experiment. The materials and equipment must be handled with an appropriate method hence that no error occurred.

3.1 Experimental Procedure

So, to begin with the experiment, the solid paraffin wax needs to be cut into small pieces and be ready to be measured into required quantity.

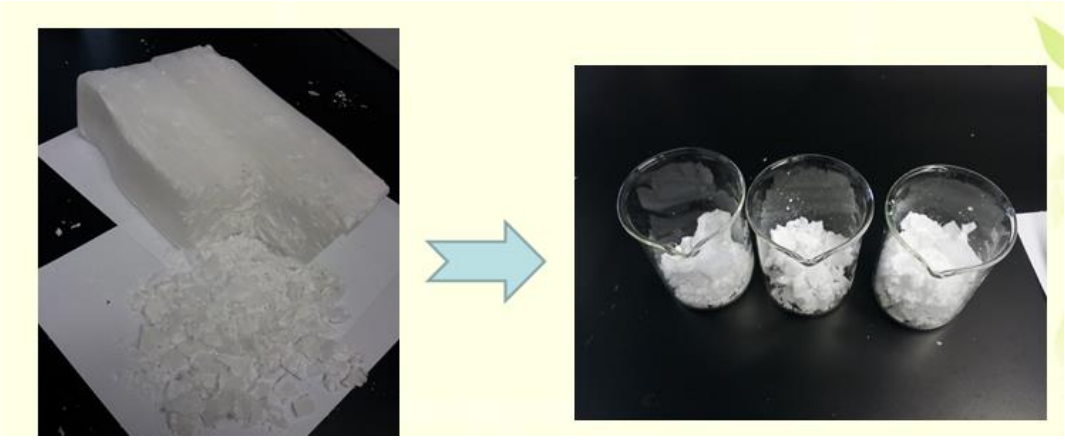


FIGURE 2: Paraffin Wax Chopped into Small Pieces

Then, we need to heat up the paraffin wax using hot plate equipment. Then, pour 150ml of liquid paraffin wax into 250ml of beaker. We need to maintain the temperature of the paraffin wax at 70 ˚C on the hot plate.

FIGURE 3: Liquid Paraffin Melted on the Hot Plate

Next, measure the weight of nanoparticle additive that want to be used of 150ml or 1.5g.

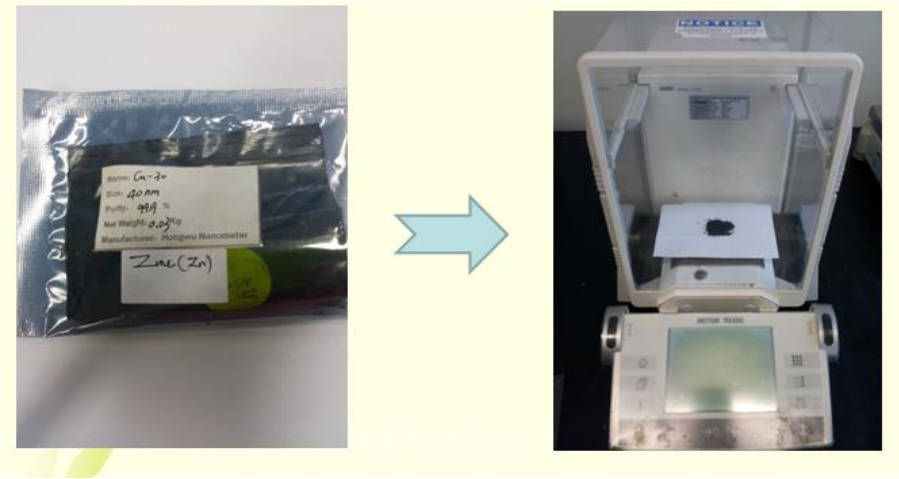


FIGURE 4: Nanoparticle Measured on the Scale

Fill the nanoparticle additive into the beaker. Make sure the temperature of the paraffin liquid is still maintained at 70 ˚C.

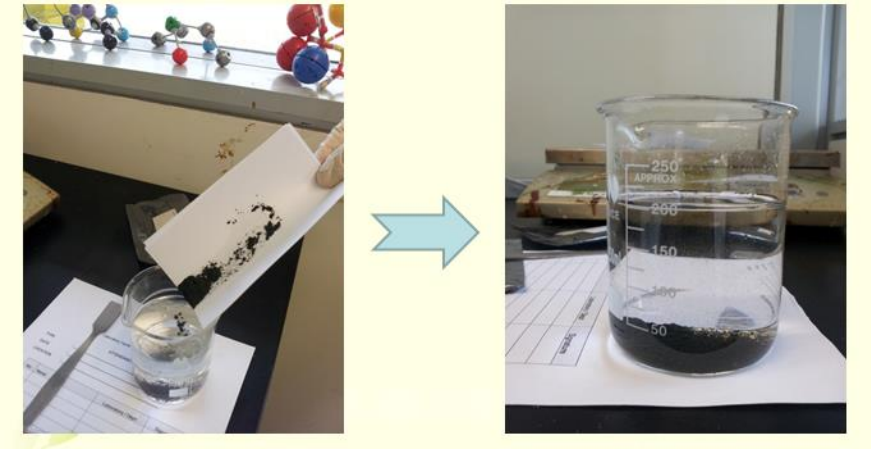


FIGURE 5: Nanoparticle Added to Liquid Paraffin

Continue on, the sonication process will starts right away by switching on sonicate mode on the ultrasonication device. The sonication process will need to be repeated for at least nine hours in order for the nanoparticles additive being distributed evenly throughout the paraffin wax.

FIGURE 6: Sonication Process

The preparing procedures above will be repeated for each type of nanoparticle additive. Complete all eight samples of nanoparticle additives. Else, switch off the ultrasonication device and put aside the heated beakers to allow them to cool down completely.

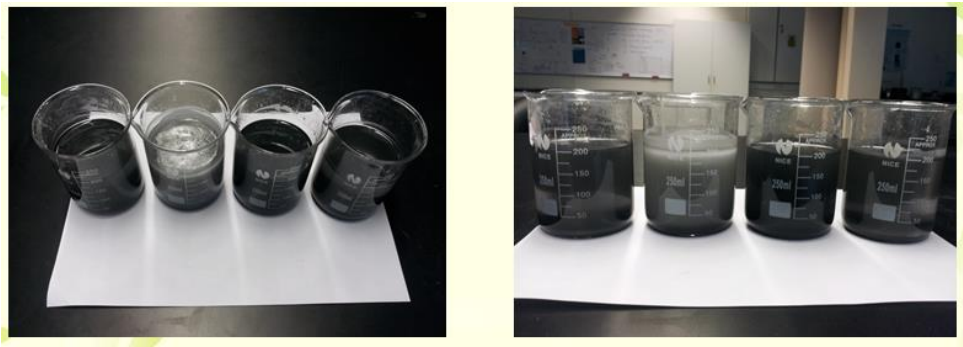


FIGURE 7: Cooling Down Process

After the preparing stage, the next step is the thermal cycle process. All the samples prepared earlier will be undergoing a heat thermal cycle for 200 cycles. A heat thermal cycle consists of melting and solidifying the Nano composite completely.

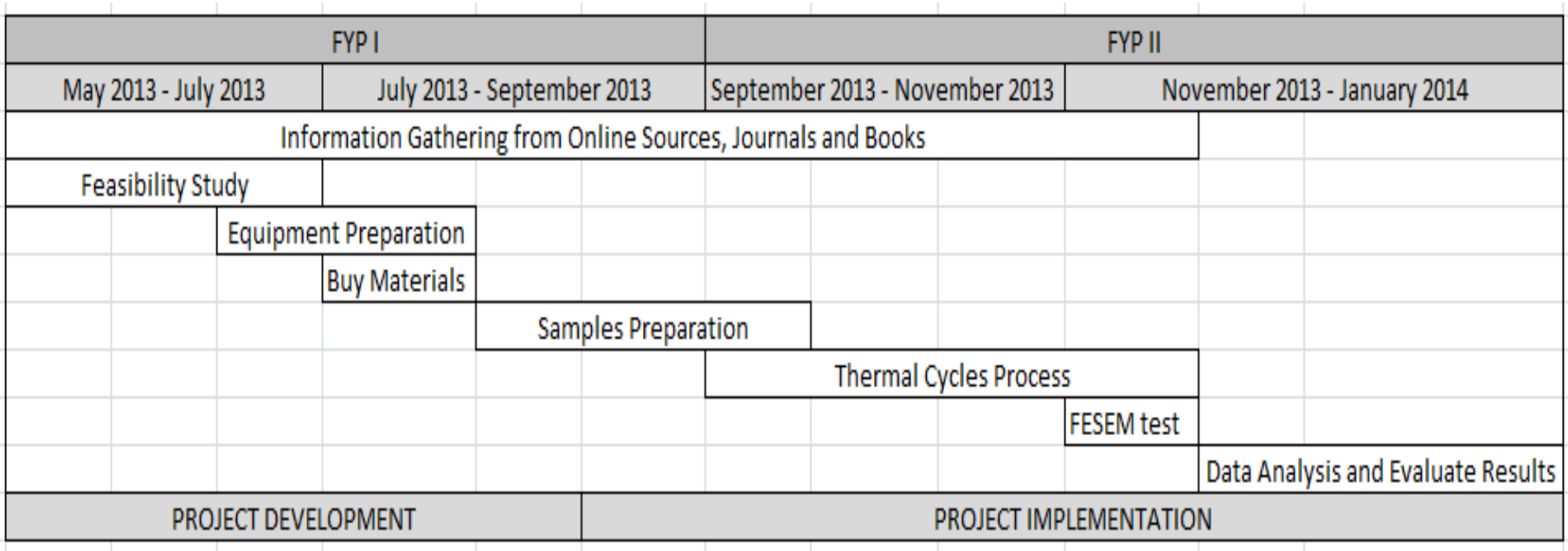
FIGURE 8: 200 Thermal Cycles

Last but not least, the samples we prepared earlier will be used for a characterize test to identify their properties. The characterize tests that are going to be taken is Field Emission Scanning Electron Microscopy (FESEM) experiment.

3.2 Gantt chart and Key Milestones

All activities that involves in the completion of the project has been put in an appropriate schedule known as Gantt chart. The schedule is very important as it will keep the project stay on track without any single activities being delayed as well as left out. Hence, the experiment for the project can be done within the time frame. The Gantt chart for the project is as shown in Table 2.

Table 2: Project Gantt chart



3.3 Procedures Identification

The strategy of completing this study is based on the workflow illustrated below:

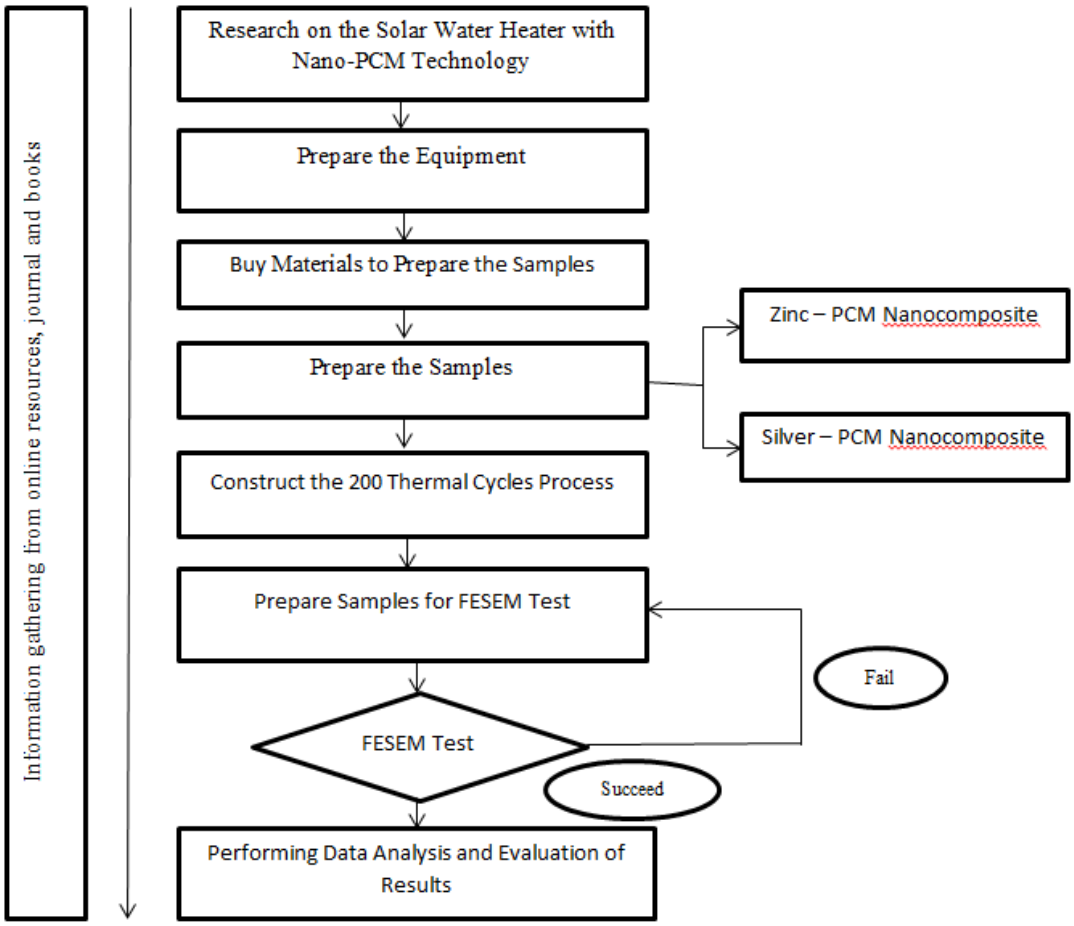


FIGURE 9: Methodology Process Flow Chart

3.4 Tools / Equipment

Ultrasonication Device

Ultrasonication device is used for dispersing nanoparticles within Nano composites. Ultrasonication device is an advanced mixing technology providing higher shear and stirring energy without scale-up limitations. The ultrasonic cavitation induces intense micro mixing and dissipates high power locally. Nano additive is mixed with paraffin wax in ultrasonic frequency generated from the ultrasonic stirrer of 5-10 MHz. The stirrer is going to run for nine hours for stable suspension of nanoparticles with no precipitation [24]. The procedures of sonication are attached with diaghram shows in Figure 10.

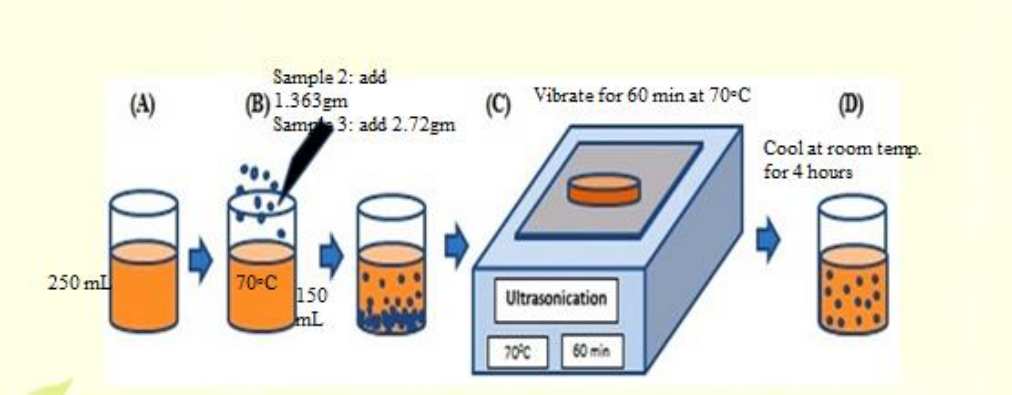


FIGURE 10: Sonication Processes for Nano – Wax Sample

Field Emission Scanning Electron Microscope (FESEM)

In order to identify the shape and size of nanoparticles that are being suspended and dispersed throughout the Nano composites, Transmission Electron Microscopy is used. The device used to accomplish the analysis is known as Field Emission Scanning Electron Microscope (FESEM). FESEM is microscope that works with electrons instead of light. This method not only enhancing resolutions but also able to obtain more information from the sample. In order to accomplish it, the electron microscopes use a beam of highly energetic electrons to analyse objects on a very fine scale [23].

Electrons are enlightened from a field emission source and accelerated in a high electrical field gradient. Primary electrons within the high vacuum column are focused and deflected by electronics lenses to generate a narrow scan beam that bombards the object. Thus, secondary electrons are emitted from each spot on the object. An electronic signal produced from the secondary electrons will be detected by a detector. This signal is thus amplified and transformed to a video scan-image that can be seen on a monitor or to a digital image that can be saved and processed further [24].

3.5 Moulding Preparation

Before the prepared samples could be sent for FESEM testing, they need to be size down accordingly in order to fit into the FESEM device. Hence, moulds have been created to shape up the Nano-PCM composite samples according to the standard requirement. Here are the steps to prepare the samples.

Step 1: Cut the polystyrene mould according to the size and shape required.

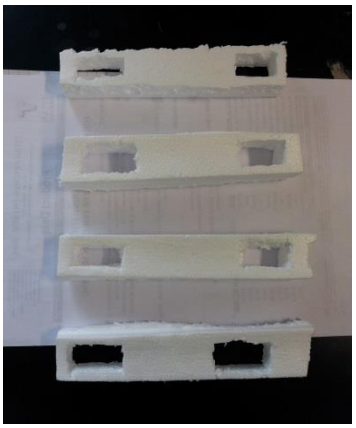


FIGURE 11: Moulds for the Samples

Step 2: Solidify and sonicate the samples for 5 minutes. Then, pour the samples in excess into the mould in order to get a smooth and flat surface.

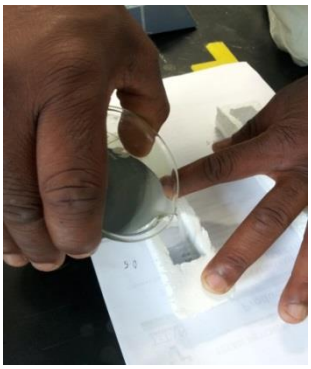


FIGURE 12: Liquid Nano composite Poured into the Mould

Step 3: Let the samples cool down and solidify completely.

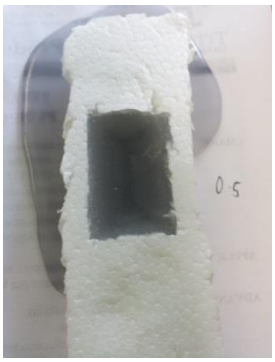


FIGURE 13: The Sample Solidify

Step 4: Remove the solidified samples from the moulds.

FIGURE 14: Solidified Sample

Step 5: Cut the centre part of the samples for FESEM test.

FIGURE 15: Sample for FESEM Test

CHAPTER 4

RESULT AND DISCUSSION

This chapter presents the dispersion of both Silver and Zinc nanoparticles in the paraffin wax using the FESEM test (EDX and Mapping). The samples were mould into required size before they can be tested. There were slight differences in weight for all the samples after the 200 cycles of thermal cycle. Hence, it is considered negligible.

The suspensions of the Nano composites are not well distributed as most of the nanoparticles are accumulated at the bottom. It is because the nanoparticles are heavy and thus they sink at the bottom of the samples.

4.1 Electron Dispersion X-Ray (EDX) Results

The polystyrene mould that was prepared previously using the same method was cut at the middle. Each cut was mapped using FESEM device to image the surface morphology and detect the suspension and dispersion nature of both Zinc and Silver nanoparticles in paraffin wax.

i) Zn-PCM Nano composites

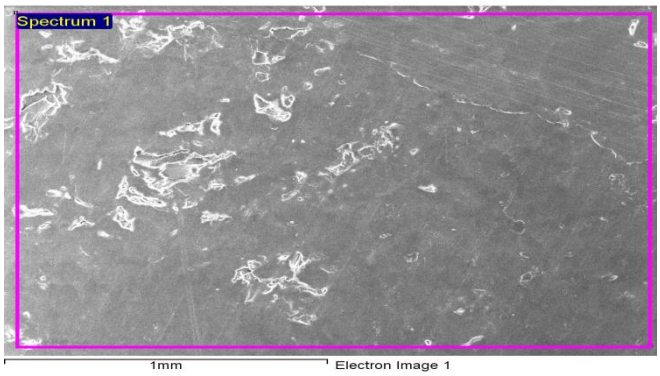


FIGURE 16: S1 – Paraffin Wax + 0.5% Nano Zn Powder Microscope Scanning

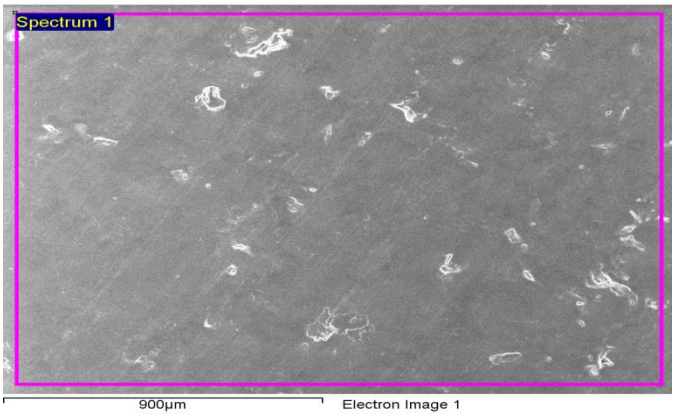


FIGURE 17: S2 – Paraffin Wax + 1.0% Nano Zn Powder Microscope Scanning

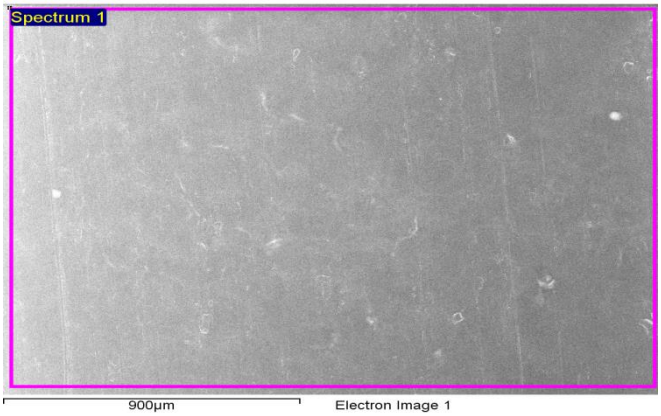


FIGURE 18: S3 – Paraffin Wax + 1.5% Nano Zn Powder Microscope Scanning

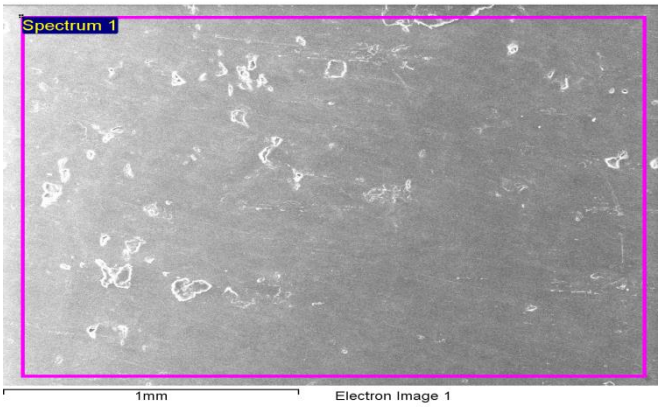


FIGURE 19: S4 – Paraffin Wax + 2.0% Nano Zn Powder Microscope Scanning

ii) Ag-PCM Nano composites

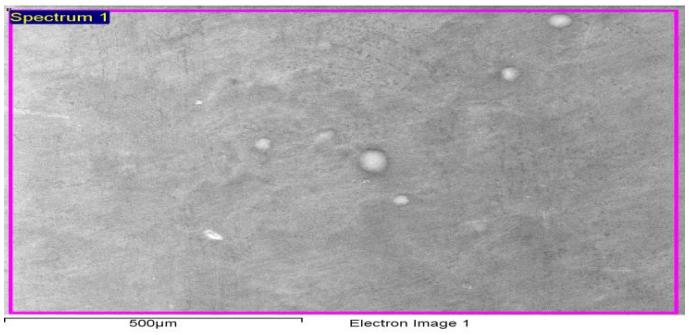


FIGURE 20: S5 – Paraffin Wax + 0.5% Nano Ag Powder Microscope Scanning

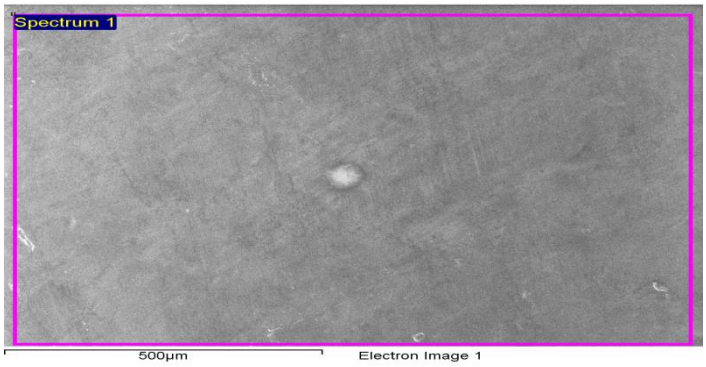


FIGURE 21:S6 - Paraffin Wax + 1.0% Nano Ag Powder Microscope Scanning

FIGURE 22: S7 - Paraffin Wax + 1.5% Nano Ag Powder Microscope Scanning

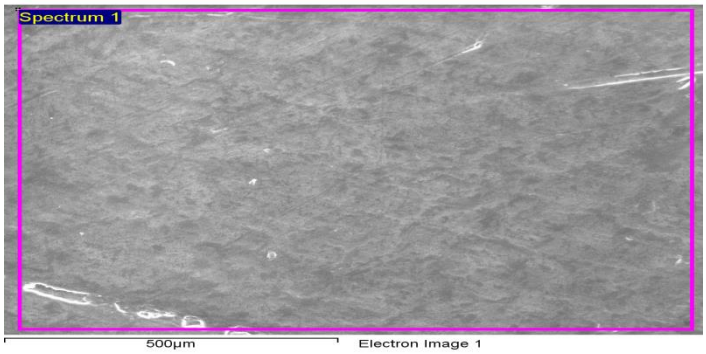


FIGURE 23: S8 – Paraffin Wax + 2.0% Nano Ag Powder Microscope Scanning

FESEM device is capable to map the element on the surface of the paraffin wax mixed with Zn and Ag nanoparticles. Though from the image mapping it is not able to show the Zn and Ag nanoparticles but it can scan to detect the existence of those elements in the samples. For example in Figure 24, it shows that both Carbon (C) and Zn elements are presence while in Figure 25 shows both Carbon (C) and Ag elements. Carbon (C) shows paraffin wax whereas Zn and Ag show the nanoparticles that are found in both types of nanoparticles samples.

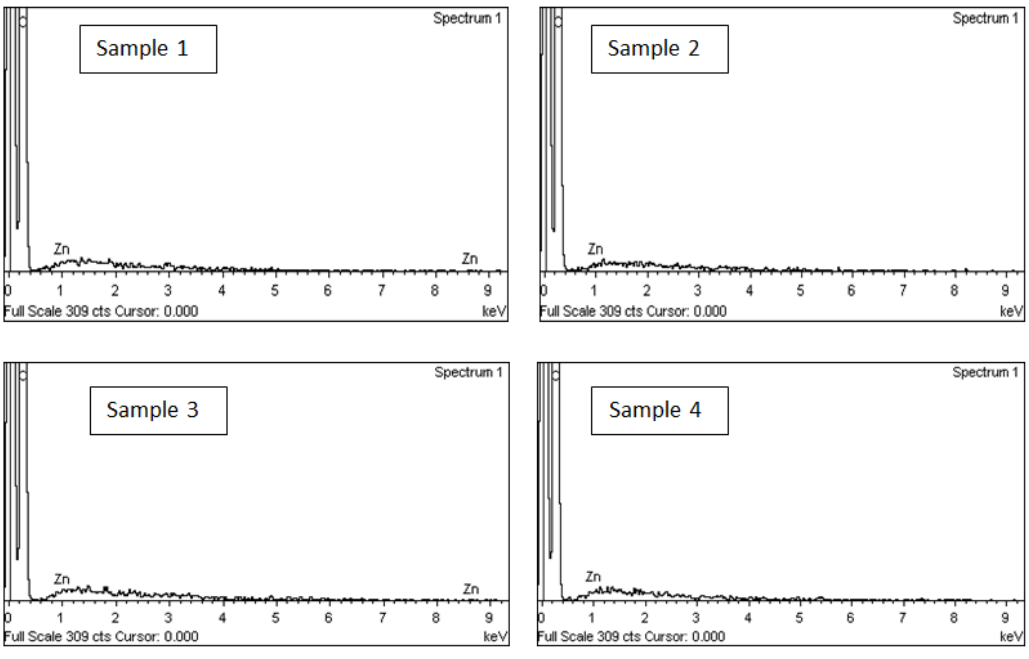


FIGURE 24: Content of Nano Zn in Nano-PCM composite

Figure 24: The graphs show the composition of the Zinc nanoparticles which are represented by Zn and paraffin wax which is represented by C. From all the samples, 0.5% of Zinc Nano composites sample is the best as it has a clearer peak compared to other Nano composites samples. This shows that the nanoparticles are well distributed throughout the Nano composites. It also has a better suspension compared to other samples.

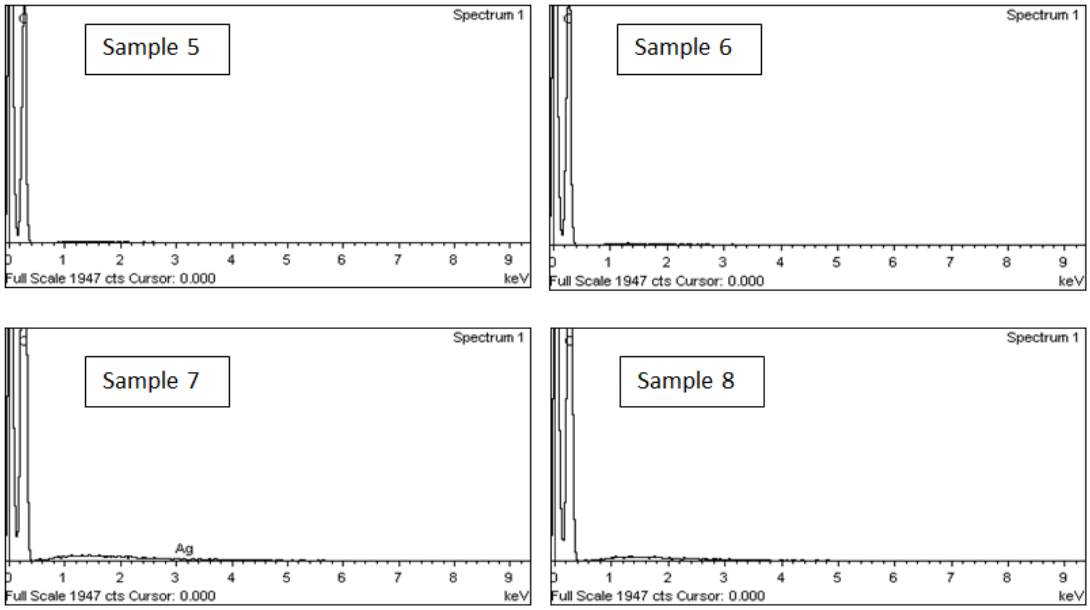


FIGURE 25: Content of Nano Ag in Nano-PCM composite

Figure 25: The graphs show the composition of the Silver nanoparticles which are represented by Ag and paraffin wax which is represented by C. From the results, 0.5% Ag and 1.5% Ag samples have an existent of Ag nanoparticles while 1.0% Ag and 2.0% Ag do not have an existent of Ag nanoparticles. The best peak can be seen in 0.5% Ag sample as it has a clearer peak compared to other samples.

The percentages of Zn and Ag suspended in paraffin wax for all the samples are recorded in Table 3 and Table 4 respectively.

TABLE 3: Zn Nanoparticles Percentages in Nano-PCM composites

No.	Materials	Elements	% Weight	% Atomic
1.	Paraffin Wax + 0.5% Zn Nanoparticles	C Zn	99.50 0.50	99.91 0.09
Total			100	100
2.	Paraffin Wax + 1.0% Zn Nanoparticles	C Zn	99.95 0.05	99.99 0.01
Total			100	100
3.	Paraffin Wax + 1.5% Zn Nanoparticles	C Cu	99.72 0.28	99.95 0.05
Total			100	100
4	Paraffin Wax + 2.0% Zn Nanoparticles	C Zn	99.94 0.06	99.99 0.01
Total			100	100

TABLE 4: Ag Nanoparticles Percentages in Nano-PCM composites

No.	Materials	Elements	% Weight	% Atomic
1.	Paraffin Wax + 0.5% Ag Nanoparticles	C Ag	99.89 0.11	99.99 0.01
Total			100	100
2.	Paraffin Wax + 1.0% Ag Nanoparticles	C Ag	100 -	100 -
Total			100	100
3.	Paraffin Wax + 1.5% Ag Nanoparticles	C Ag	99.90 0.10	99.99 0.01
Total			100	100
4	Paraffin Wax + 2.0% Ag Nanoparticles	C Ag	100 -	100 -
Total			100	100

4.2 Mapping Results

Detection of Nanoparticles and Carbon (C) atom distribution and surface texture were carried out using FESEM device. The texture of both types of Nanoparticles which are Nano Zn and Nano Ag are shown below.

i) Zn-PCM Nano composites

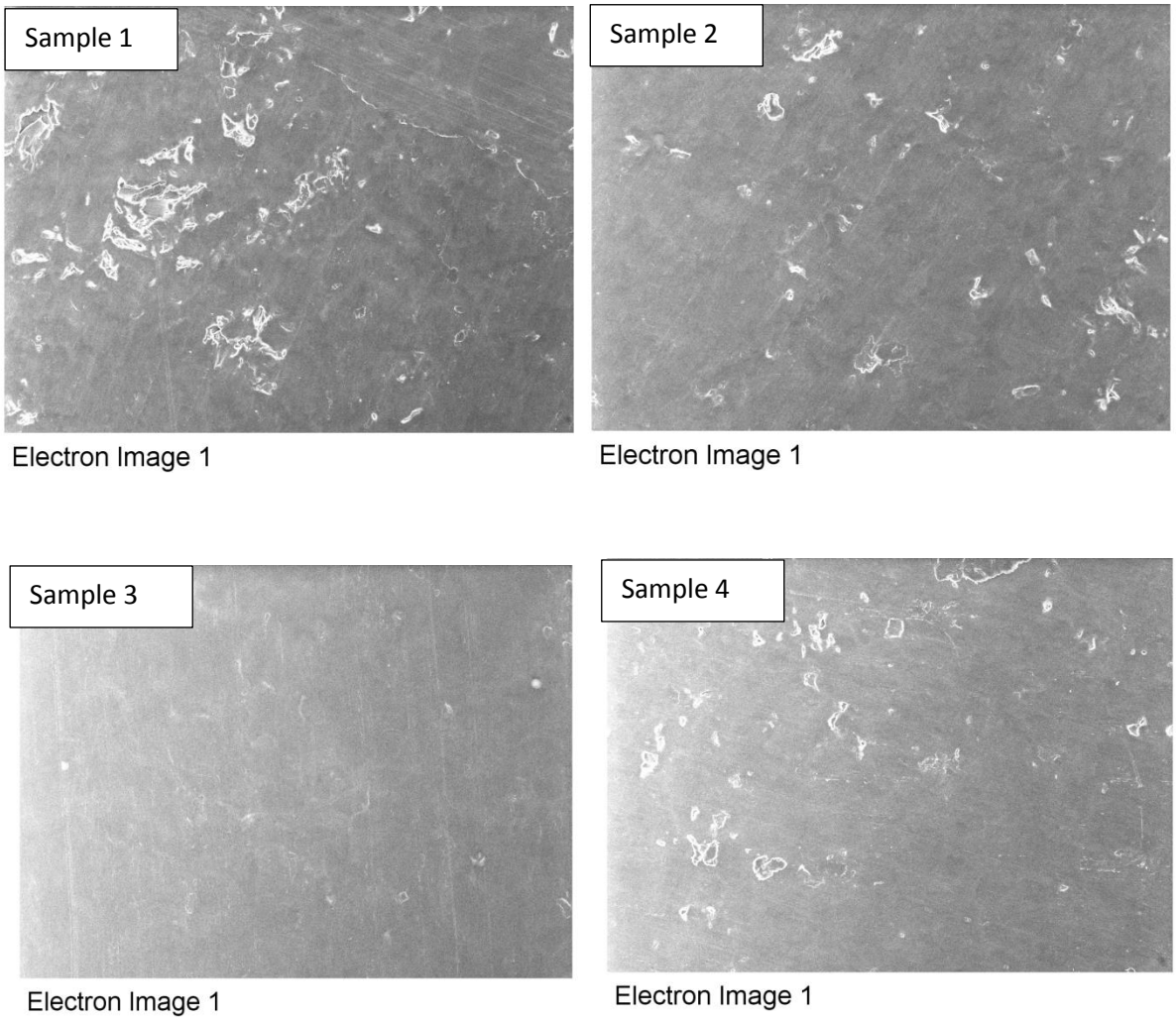


FIGURE 26: Texture mapping of the Zn-PCM Nano composites Samples

ii) Ag-PCM Nano composites

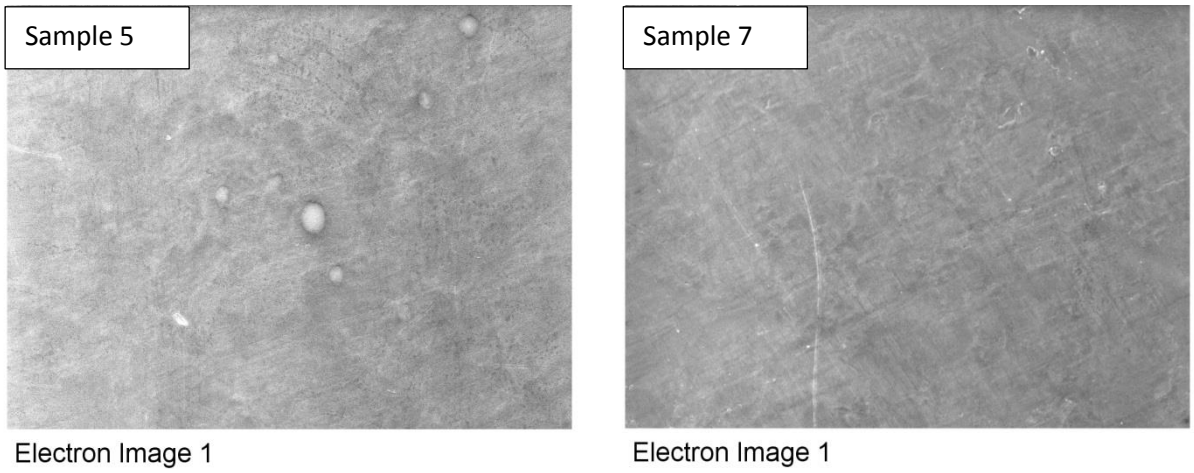


FIGURE 27: Texture mapping of Ag-PCM Nano composites Samples

The distribution of Carbon atoms of paraffin wax are in Figure 28 and Figure 29. Bright red dot colour showed the carbon atom.

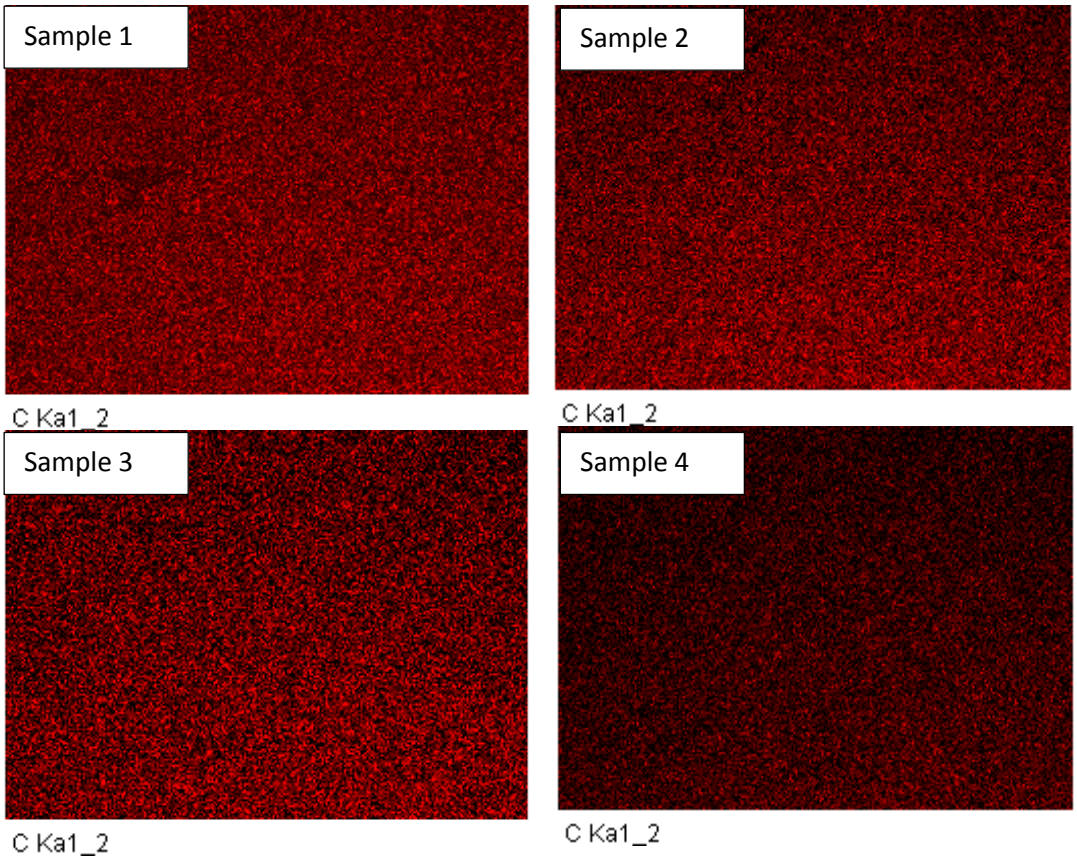


FIGURE 28: Carbon Atom Distribution of the Nano Zn Samples

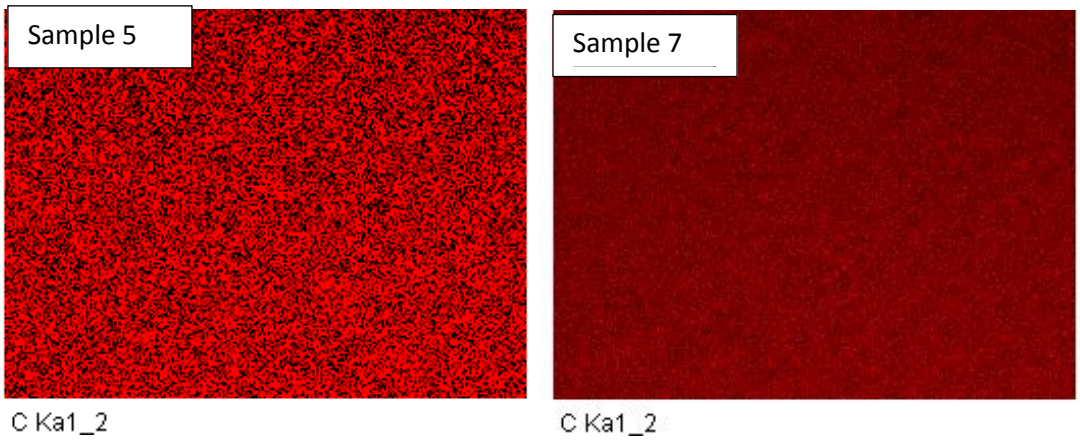


FIGURE 29: Carbon Atom Distribution of the Nano Ag Samples

Thus, agglomerations found on surface of samples are shown in Figure 30 and Figure 31. Bright green dots colours show both Nano Zn and Nano Ag.

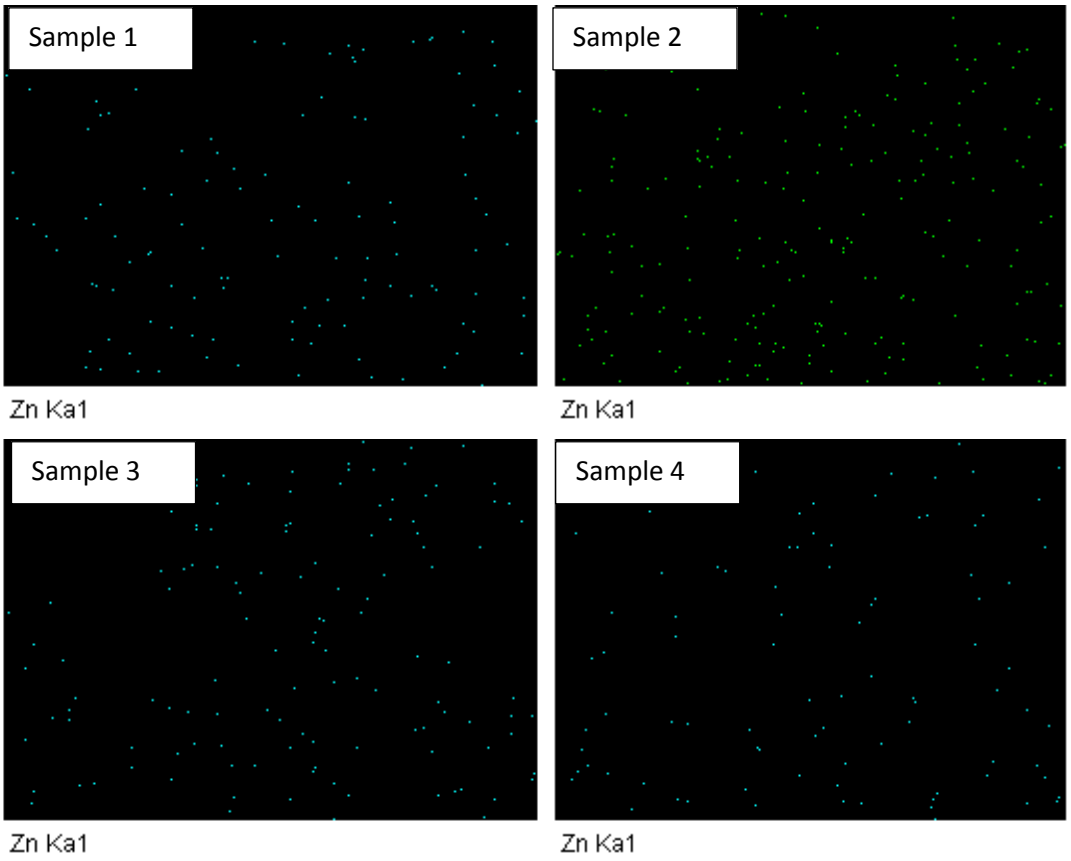


FIGURE 30: Nano Zn Distribution Mapping of the Samples

Figure 30: Figures show the distribution of the Zinc nanoparticles in the samples. The green dots represent zinc nanoparticles. As shown, the nanoparticles are well distributed throughout the samples though their existences are minor. Hence, the distribution of nanoparticles is not depending on its concentration.

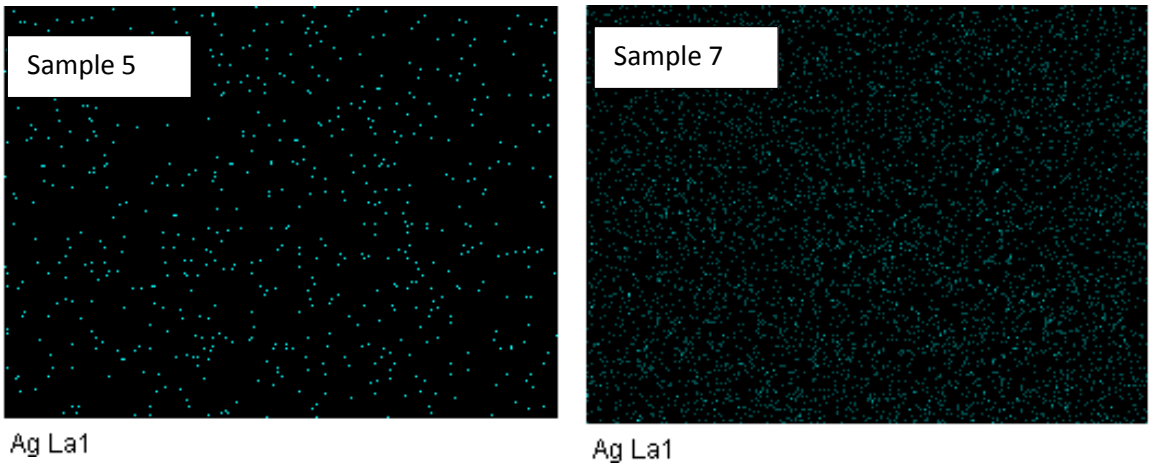


FIGURE 31: Nano Ag Distribution Mapping of the Samples

Figure 31: Figures show the distribution of the Silver nanoparticles in the samples. The samples are 0.5% Ag and 1.5% Ag. There are no existence of Silver nanoparticles in the samples of 1.0% Ag and 2.0% Ag. It happens because the samples are not well mixed. Hence, they are not well suspended. To have a good suspension, a sample must be even, stable and durable.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Referring to the objectives, the dispersion of the nanoparticles throughout the Nano composite is not well dispersed. It is may be caused by the mixing procedure during samples preparation processes. The experimental error might occur during those steps.

Both types of Nano composites, Silver-PCM and Zinc-PCM, have been possessed thermal cycles of 200 times before they are acceptable to be tested using the FESEM test. The thermal cycle process may contribute to the changing in the thermal properties of the Nano composites.

The required samples for FESEM test have been prepared by taking a small part, specifically at the middle part, from both types of Nano composites. The required samples are then moulded before been tested.

The part of the samples at which the samples are taken to prepare the mould to run the test might not contain the nanoparticles. As the nanoparticles are not well dispersed, the probability of the samples to have a little existence of nanoparticles for a particular part of the samples is high.

Most of the nanoparticles are suspended at the bottom part of the samples. The nanoparticles are heavy, so they are most likely to sink at the bottom. To have a good suspension, the nanoparticles must be even, stable and durable.

5.2 Recommendation

It is recommended that during samples preparation, the nanoparticles were dispersed in liquid paraffin without any surfactant added. This step may increase the dispersion of the nanoparticles throughout the paraffin wax.

In addition, a step may be added into the procedure by degassed the samples in a vacuum oven for two hours at 105 ^{o}C after which the nanoparticles was dispersed in the liquid paraffin and accomplished by shear mixing (magnetic stirrer) for 25 minutes at constant 80 ^{o}C. The samples should be degassed to remove unwanted air or water from the samples. Hence, pure Nano composites could be produced.

Besides, each of the samples should be tested at three different layers. There are for example top layer, middle layer and bottom layer. This approach will allow us to observe the suspension of the nanoparticles more clearly. Hence, we can make a conclusion where is the most part of the sample that the nanoparticles are more likely to suspense. Further steps could be taken then to ensure that the nanoparticles are finely suspended as well as well dispersed throughout the samples.

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