CHAPTER 1

INTRODUCTION

1.1 BACKGROUND STUDY

Resin infusion process has been found to be very versatile for making large composite structures. In the process, a vacuum bag is laid over the laminate, and a vacuum is pulled. Resin enters the laminate through feeder pipes and a runner system, and impregnates the glass, aramide or carbon fibers. As a result, high quality composite laminate, with high fiber/resin ratio, and perfect adhesion to core materials is introduced [1]. There are some advantages by using this technique, which are:

- high quality laminate (low void content, high fiber to resin ratio)
- user friendly
- large objects can be infused with a minimum of workforce
- environmentally friendly (reduction of VOC, when using polyester)
- repeatable results
- weight reduction of the part (especially sailing boat hulls, multihull and fast powerboats can benefit)

Basically, this process is a manufacturing technique suitable for large load-carrying composite and sandwich structures, such as marine vessels, cooling trailers, etc. As these structures are becoming more optimized, in order to reduce weight and material cost, the requirements on mechanical properties are increasing in terms of performance and consistency. One aspect, which may have a strong influence on mechanical properties, is void content [2,3,4,5]. During injection, voids may form due to:

i. local variations in reinforcement permeability at the resin flow front,
ii. leakage,
iii. boiling of volatile components in the resin or,
iv. gas dissolved in the resin coming out of solution.

After injection, voids can also be formed as a result of shrinkage of the resin. The formation of voids can be controlled by using manufacturing parameters such as vacuum pressure, cure temperature, autoclave pressure, and resin viscosity [2]. One of the applications that are related to this process is wind turbine blade manufacturing. Basically, the most dominant processes in making the blade are infusion, vacuum-assisted resin transfer molding (VARTM) and hand layup of prepreg in open molds. Apart from that, some form of resin infusion process is preferable instead of using hand layup, in order to improve the fiber-to-resin ratio of the fiberglass blades. In this project, resin with different viscosity is used as a reinforced composite. Void content of the samples will be measured to see how the effect of different viscosity resins to the void content.

1.2 PROBLEM STATEMENT

Resin infusion process can produce high quality laminate which is low void content and high fiber to resin ratio [1]. Meanwhile, during the resin impregnation process, the vacuum causes the nucleation and formation of air bubbles from gasses dissolved in the resin. As a result, there will be formation of void in the final product. To overcome this problem, voids can be reduced by controlling manufacturing parameters such as vacuum pressure, cure temperature, autoclave pressure, and resin viscosity [2]. The presence of voids in the final product is an unavoidable fact. They are formed due to the variations in permeability on a filament and filament bundle scale, outgassing of dissolved gas in the resin, evaporation of volatile components in the resin, shrinkage of the resin and leakage in connections and mold. The inclusion of voids in the final part will have a detrimental impact on the mechanical properties of the composites even at a very low volume fraction [2]. So, it is important to know how voids are distributed due to the resin infusion process and the viscosity of the resin.

1.3 OBJECTIVES AND SCOPE OF THE STUDY

1.3.1 Objectives

The objectives of the project are:

1. To investigate the effect of different viscosity resin to the void content.
2. To produce a mapping of void content.

1.3.2 Scope of the study

The scope of the study covered the effect of manufacturing parameter which is resin viscosity, to the formation of void of the polymer composite. In this project, a plate sample was fabricated using the resin infusion process, while the resin viscosity will be varied using a certain solvent. Besides that, we wanted to know how voids were distributed at different location of the sample by producing mapping of void content at different location of the samples.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 COMPOSITE MATERIALS

Generally, a composite is considered to be any multiphase material that exhibits a significant proportion of the properties of both constituent phases such that a better combination of properties is realized. Besides multiphase metal alloys, ceramics and polymers, there are also a number of composites that occur in nature such as wood consist of strong and flexible cellulose fibers surrounded and held together by a stiffer material called lignin [6].

In the present context, a composite is referred to a multiphase material that is artificially made, as opposed to one that occurs of forms naturally. This means that most metallic alloys and ceramics do not fit this statement because of natural phenomena. In order to design a composite, various metals, ceramics, and polymers are combined to produce a new material that is better in term of mechanical characteristics such as toughness, stiffness, and strength. Basically composite materials are consists of two phases; matrix phase and dispersed phase.

Figure 1 below shows the classification of composite materials [6].

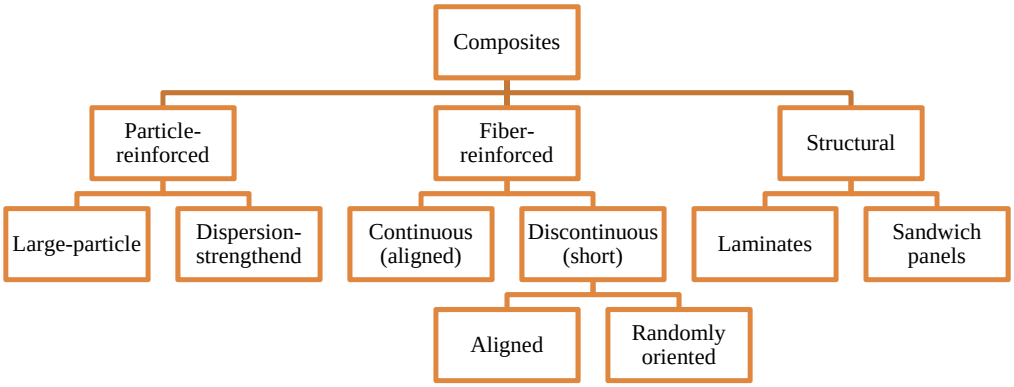


Figure 1: Classification of composite materials

The focus in this project is the fiber reinforced composites using the polymer resin as the matrix and fiber as the reinforcement medium. For example, a glass fiber-reinforced polymer is a composite consisting of glass fiber, contained within a polymer matrix. There are many reinforcement types such as glass, aramid and carbon type [7]. Glass is popular as a fiber reinforcement material because:

1. Easily drawn into high-strength fibers from the molten state.
2. Readily available and may be fabricated into a glass-reinforced plastic economically using a wide variety of composite-manufacturing techniques.
3. Relatively strong, and produces a composite having a very high specific strength when embedded in a plastic matrix.
4. Possesses a chemical inertness that renders the composite when coupled with the various plastics (useful in a variety of corrosive environment).

For the polymer matrix, the most commonly used are polyesters and vinyl esters because of least expensive. Meanwhile, the epoxies are more expensive but are utilized for aerospace applications because they have better mechanical properties and resistance to moisture than the polyesters and vinyl resins.

2.2 VACUUM INFUSION PROCESS

For processing of the fiber-reinforced composite, resin infusion or vacuum infusion process is chosen. The vacuum infusion is actually the popular techniques in manufacturing these composites. There are many terms in vacuum infusion processes such as VARTM-Vacuum Assisted Resin Transfer Moulding, VARIM-Vacuum Assisted Resin Infusion Moulding, SCRIMP™-Seemann Composites Resin Infusion Moulding Process, VBRTM-Vacuum Bag Resin Transfer Moulding, VARI-Vacuum Assisted Resin Infusion process [7] and so on. All of these methods involve with the same principle of process which based on the impregnation of a dry reinforcement by liquid thermoset resin driven under vacuum.

Figure 2 below shows the sequence of the events that comprises vacuum infusion and figure 3 shows the final arrangement of the process [8].

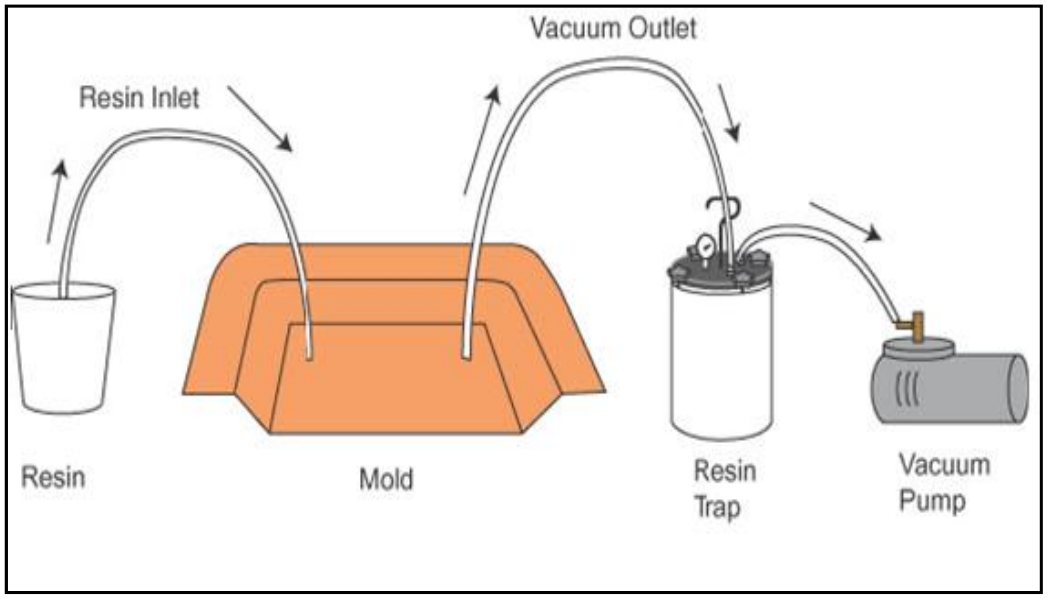


Figure 2: Sequence of the vacuum infusion

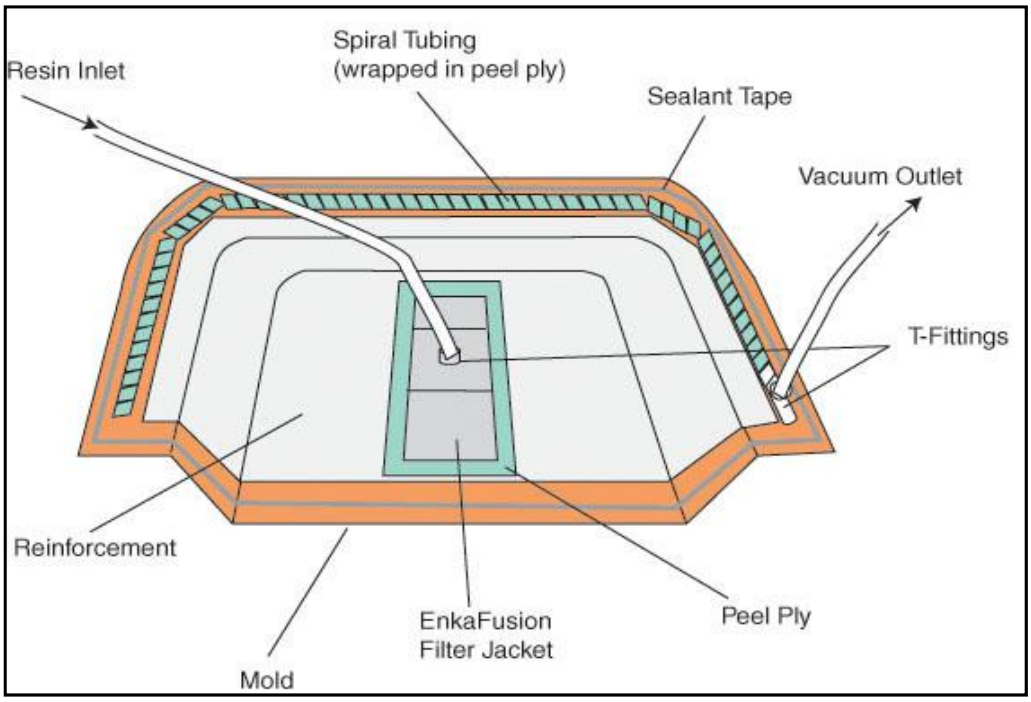


Figure 3: Final arrangement of the process

2.3 VOID CONTENT

Voids are one of the most common types of manufacturing process induced defects in composite materials that have detrimental effect on the materials properties. Void, generally air bubbles entrapped in the matrix during the composite fabrication, have a pervasive effect on composite properties even at low volume percent: ultimate mechanical properties, especially interlaminar shear strength, are markedly reduced, and resistance to environmental degradation also suffers [9]. Void content measures the voids in reinforced polymers and composites. Information on void content is useful because high void contents can significantly reduce the composites' strength. Monitoring void contents can also act as a measure of the consistency of the composites' manufacturing process [10]. Measurement of void content can be done by two processes which are acid digestion method and loss on ignition method.

In acid digestion method, a sample of at least 0.5 g per replicate is required and it takes several hours to perform [11]. According to the standard ASTM D3171, method I, the specimens are dried and cooled. The density of the test specimens is determined using ASTM D792. The specimens are then placed in an appropriate dissolution medium and heated/refluxed until all of the organic material is dissolved. The resulting solution is then filtered and the amount of remaining fiber is determined. From the specimen density, constituent densities, and the fiber content, the reinforcement content, matrix content, and void content can then be determined [12].

Apart from that, the ignition method used for glass fiber composites is quicker than the digestion method. In this method, the densities of the resin, the reinforcement and the composites are measured separately. Then, the resin content is measured and a theoretical composite density is calculated. This is compared to the measured composite density. The different in densities indicates the void content. A good composite may have 1% void or less, while a poorly made composite can have a much higher void content [13]. The standard used for this method is ASTM D2734.

2.4 LITERATURE REVIEWS

Some manufacturing parameters can control the formation of voids such as vacuum pressure, cure temperature, autoclave pressure and resin viscosity [2]. For this project, resin viscosity has been chosen to be a controlled parameter in order to analyze the differences in void content for each sample. Many approaches [2,3,4,5] have been reported to establish relationships between the void content and material properties such as strength.

Md. Afendi [3] had established the relationship between void and mechanical properties which is tensile strength. During his research, he tried to eliminate the micro-bubbles from the resin prior to resin infusion process. There were two types of resin used in the research; untreated resin and treated resin by capillary separation method and vacuum degassed.

Two samples were made and void content analysis had been done to those samples followed by the tensile test. Figure 4 below shows the stress-strain curve for sample A and B.

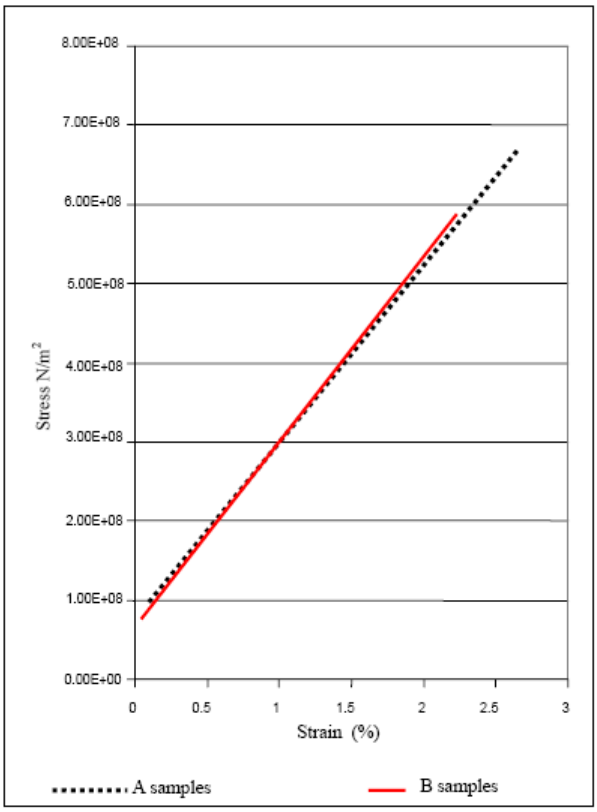


Figure 4: The stress-strain curve for sample A and B

Besides that, Hansong H. et al [2] also had done some researches relating the void with the elastic properties. Their objective was to examine the effects of void microstructures on the elastic response of composite laminates. During the study, they used two-phase approaches which were finite element analysis approach and analytical approach with voids in unidirectional polymer matrix composites produced by the autoclave technique.

Meanwhile, C. Santulli et al [14] had tried to measure the void content by using image analysis from optical micrographs instead of acid digestion method and loss ignition method. In their study, they used twill weave fabric commingled E-glass and polypropylene as the material for the sample.

From the microscopy observations, they had distinguished four types of voids which were defined as microvoids, intratow voidage, coplanar voids and extensive voids. Figures 5 to 8 below show the four types of voids.

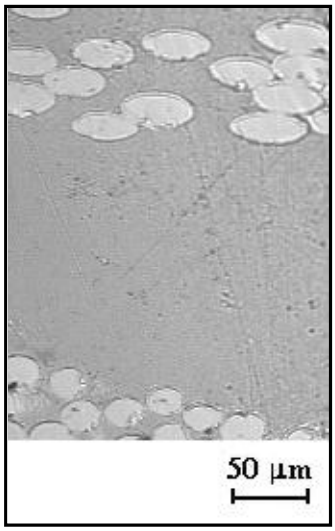


Figure 5: Microvoids in the matrix

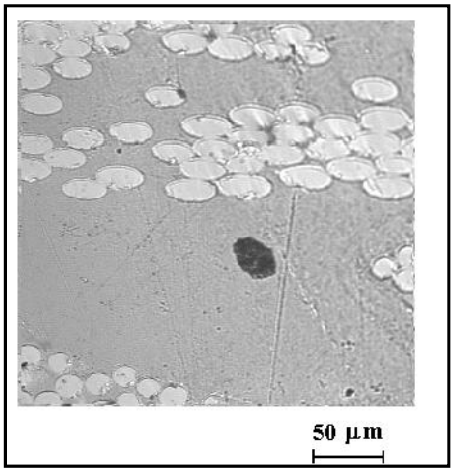


Figure 6: Coplanar void

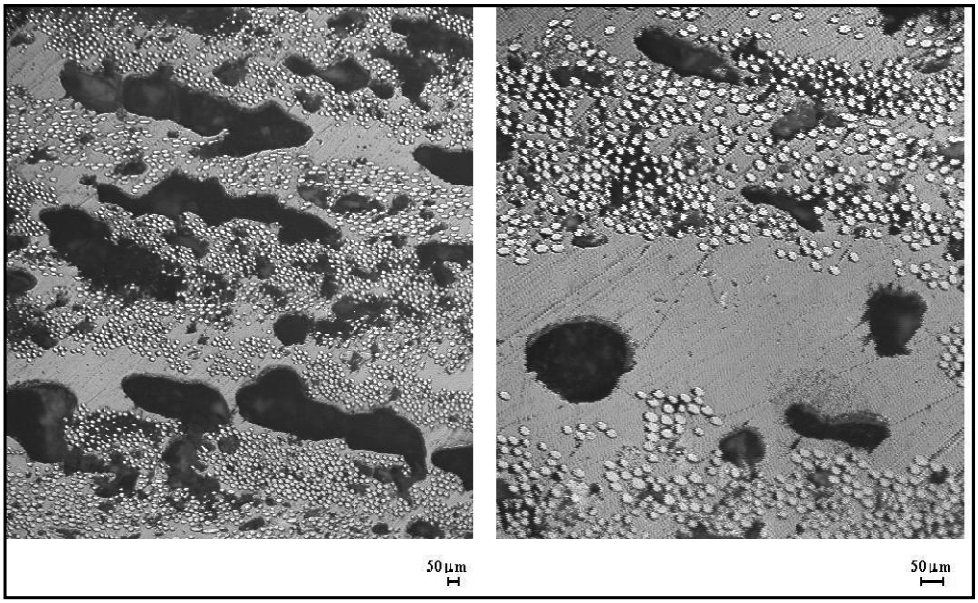


Figure 7: Extensive voidage

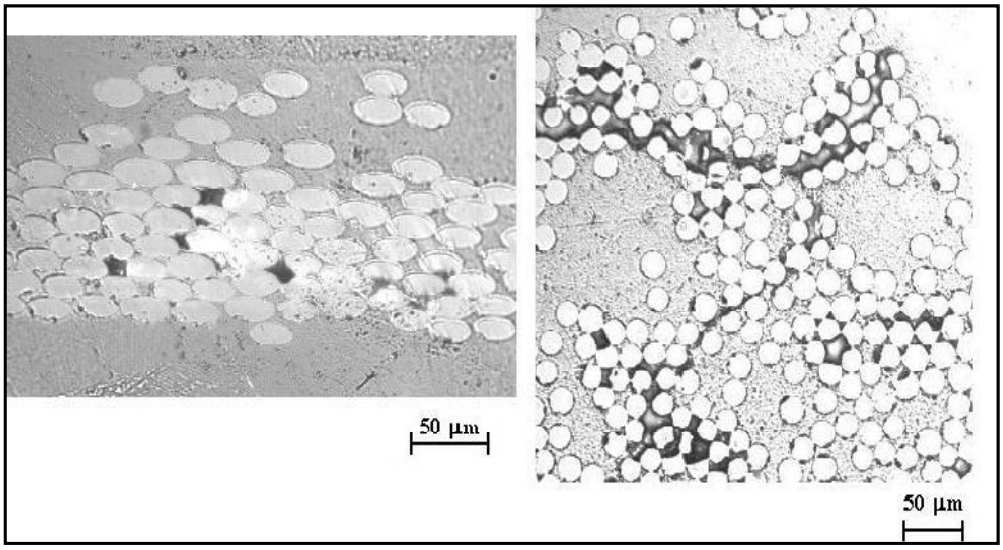


Figure 8: Intratow voidage

CHAPTER 3

METHODOLOGY

An experiment had been carried out to prepare the sample for the void content analysis. In this experiment, the fiber was reinforced with matrix material using vacuum infusion process.

3.1 MATERIALS AND EQUIPMENTS

Table 1 below shows the equipments that were used in this project.

Table 1: Materials and equipments

MATERIALS/EQUIPMENTS	DETAILS
Polymer resin	- The resin used during the experiment was epoxy.
Reinforcement	- Woven fabric, E-glass fiber
Resin infusion kit	- Vacuum pump - Resin storage and resin trap - Plastic bag - Sealant tape - Peel ply - Net - Aluminum plate - Degassing chamber
Electrical balance	- To know how much resin will be used to infuse the glass fiber
Acetone	- To vary the viscosity of the resin
Muffle furnace	- To burn the specimens in loss ignition test.

Figures 9 to 12 below show some of the equipments used in this project.

Figure 9: Vacuum pump

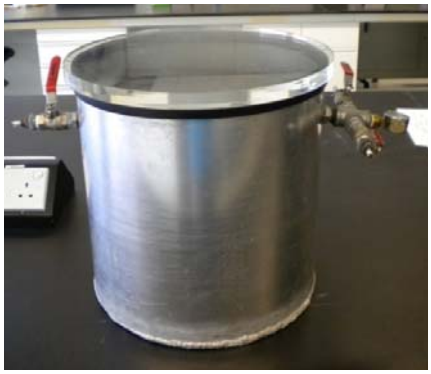


Figure 10: Degassing chamber

Figure 11: Muffle furnace

Figure 12: Electrical balance

In this project, epoxy was used as a polymer resin and acetone as the solvent to vary the viscosity. Table 2 shows the compositions of the mixture.

Table 2: Composition of the resin mixture

Epoxy (%)	Acetone (%)
95	**5**
90	**10**
85	**15**
80	**20**

Some other calculations were made in order to prepare the resin used for the infusion process. The calculations involved were:

Thickness for 10 layers of glass fiber = 2.0175 mm

Volume for 10 layers of glass fiber = 300 mm x 200 mm x 2.0175 mm

$= 121.05\ cm^3$

Since the volume fraction of fiber and matrix is 50%, volume for the matrix is also 121.05 cm^3. Using the density of the resin, we can find the weight of the resin that will be used

Weight of resin = Density x Volume

$= 1.24\ g/cm^3 \times 121.05\ cm^3$

$= 150.10\ g$

Considering some factors that will increase the resin consumption, we increased the weight of resin by 30%.

3.2 EXPERIMENTAL

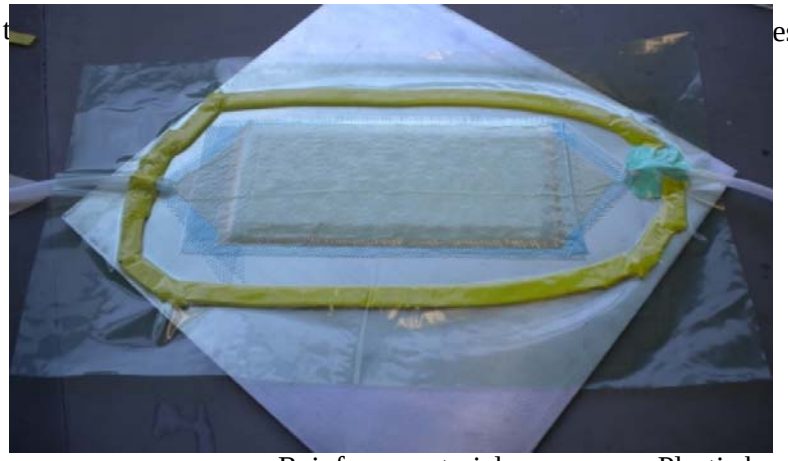
3.2.1 Sample Fabrication

1. Resin was prepared by mixing with the hardener using the ratio given by the supplier. Then, 5% of acetone was added to the mixture.
2. The resins were degassed in order to remove the bubbles inside.
3. Meanwhile, the glass fiber was cut into a rectangular shape with the dimension of 200 mm x 300 mm.

4. Then, the glass fibers were placed, together with peel ply, and net in an open, plane mould and a plastic vacuum bag is placed on the top of the mould. The one-sided mould is connected with a resin source and a vacuum pump.
5. The vacuum pump was turned on and the air was evacuated from the sealed plastic bag.
6. The mixture with hardener from the resin source was drawn into the component by vacuum.
7. The resin was infused into the fibers until complete impregnation.
8. Curing and de-moulding steps followed the impregnation to finish the product.
9. Steps 3 to 8 were repeated using different concentration of acetone in the resins.

Figures 13, 14 and 15 below show the setup for the process.

Connected t esin



Sealant Reinforce material Plastic bag

Figure 13: Resin infusion setup at the mold

Figure 14: Overall resin infusion setup

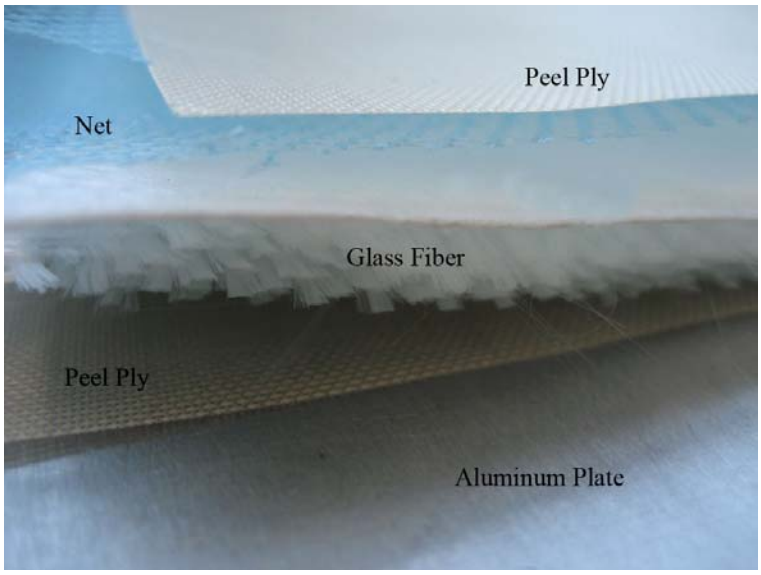


Figure 15: Arrangement of the materials involve in resin infusion process

The function for each material is:

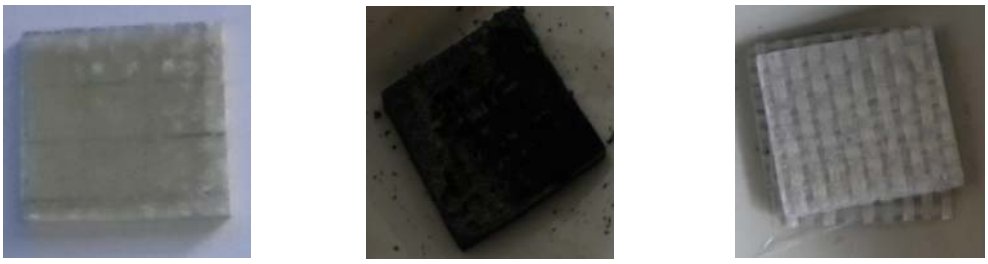
- ❖ Peel ply - allows resin to pass through it but will not stick to the resin once it is cured.
- ❖ Net - create a path for resin to flow.
- ❖ Aluminum plate - as a mold.

3.2.2 Loss Ignition Test

This test was carried out according to the test standard ASTM D 2584.

1. The sample from the fabrication part was cut into 3 specimens with the dimension of 25 mm x 25 mm. The weight (m_1) and density of every specimen is measured.
2. A crucible is heated at 500-600^{o}C for 10 minutes.
3. The crucible is cooled and weighted (m_2).
4. The specimen is placed inside the crucible and is heated using a Bunsen until only carbon material remains.
5. Then, the crucible is heated inside a furnace at 565^{o}C for 40 minutes.
6. The crucible is cooled and weighted (m_3).

Figure 16 below shows the specimen that had been tested using loss ignition process.



(a) (b) (c)

Figure 16: Sequence of specimen tested in loss ignition process. (a) Before burning; (b) After burning using Bunsen; (c) Complete burning process in a furnace

3.3 DATA ANALYSIS

Data analysis is referred to the void content analysis of each sample. Void content analysis is performed according to the standard test method ASTM D 2734. The steps involve in this method is:

1. The densities of the composite, resin and glass fiber are measured.
2. The resin content of composite is measured using the value gained from the loss ignition test and the theoretical composite density is calculated.
3. The void content is measured from the difference between the theoretical and measured density of composite.

Below are the equations involved in the void content calculation [13,16]:

(i) Calculation for resin and fiber content:

$$R_{wt} = \frac{(m_1 + m_2) - m_3}{m_1} \times 100$$

$$F_{wt} = 100 - R_{wt}$$

where; R_{wt} = weight percent of resin, %w

F_{wt} = weight percent of fiber, %w

m_1 = weight of specimen, g

m_2 = weight of crucible, g

m_3 = weight of crucible + residue, g

(ii) Calculation for void content:

$$T_d = \frac{100}{\frac{R_{wt}}{D} + \frac{F_{wt}}{d}} \qquad V = \frac{T_d - M_d}{T_d}$$

T_d = theoretical composite density V = Void content (volume %)

M_d = measured composite density R_{wt} = Resin weight %

F_{wt} = Fiber weight % D = Density of resin

d = Density of fiber

CHAPTER 4

RESULTS AND DISCUSSIONS

In this project, four types of samples were prepared using the vacuum infusion process with different resin viscosity. Each sample was weighed and the density was measured. Table 3 below shows the weight and density of each sample.

Table 3: Measured weight and density for each sample

RESIN MIXTURE	SAMPLE TAG	WEIGHT (g)	DENSITY (g/cm^3)
Epoxy + 5% Acetone	A1	2.123	1.674
	A2	2.067	1.696
	A3	2.439	1.661
Epoxy + 10% Acetone	B1	1.949	1.692
	B2	2.015	1.694
	B3	1.967	1.696
Epoxy + 15% Acetone	C1	2.361	1.645
	C2	2.370	1.648
	C3	2.202	1.699
Epoxy + 20% Acetone	D1	2.053	1.683
	D2	1.996	1.723
	D3	2.100	1.710

The measured densities were used in order to calculate the void content by comparing the measured density and theoretical density. The samples were then tested in the furnace according to standard test method ASTM D 2584. Table 4 shows the result from the lost ignition test.

Table 4: Lost ignition test result

Resin Mixture	m_1 (g)	m_2 (g)	m_3 (g)	F_{wt} (%w)	R_{wt} (%w)	T_d (g/cm^3)	M_d (g/cm^3)	Void (%)
Epoxy + 5% Acetone	2.210	123.315	124.662	60.926	39.074	1.802	1.677	6.934
Epoxy + 10% Acetone	1.977	122.746	123.973	62.027	37.973	1.817	1.694	6.742
Epoxy + 15% Acetone	2.311	122.737	124.142	60.838	39.162	1.801	1.664	7.598
Epoxy + 20% Acetone	2.050	123.309	124.636	64.743	35.257	1.855	1.705	8.052

Figure 17 and 18 show comparison between measured and theoretical density, and void content, respectively.

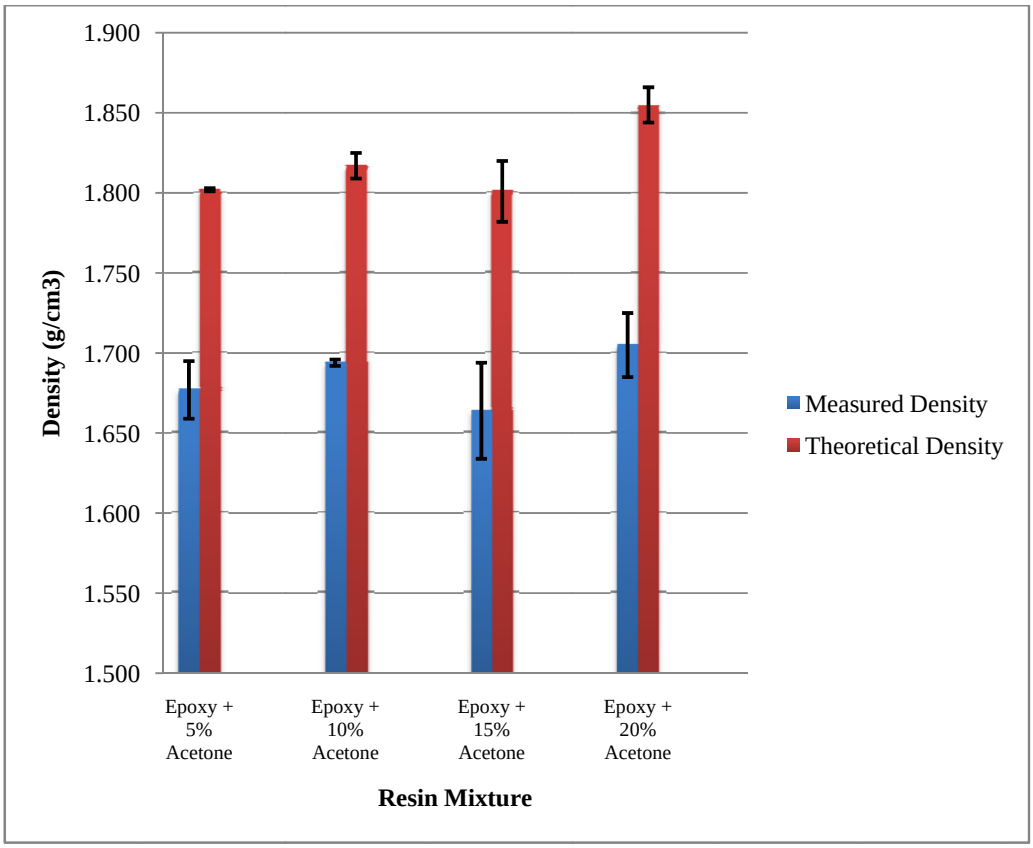


Figure 17: Comparison between measured and theoretical density

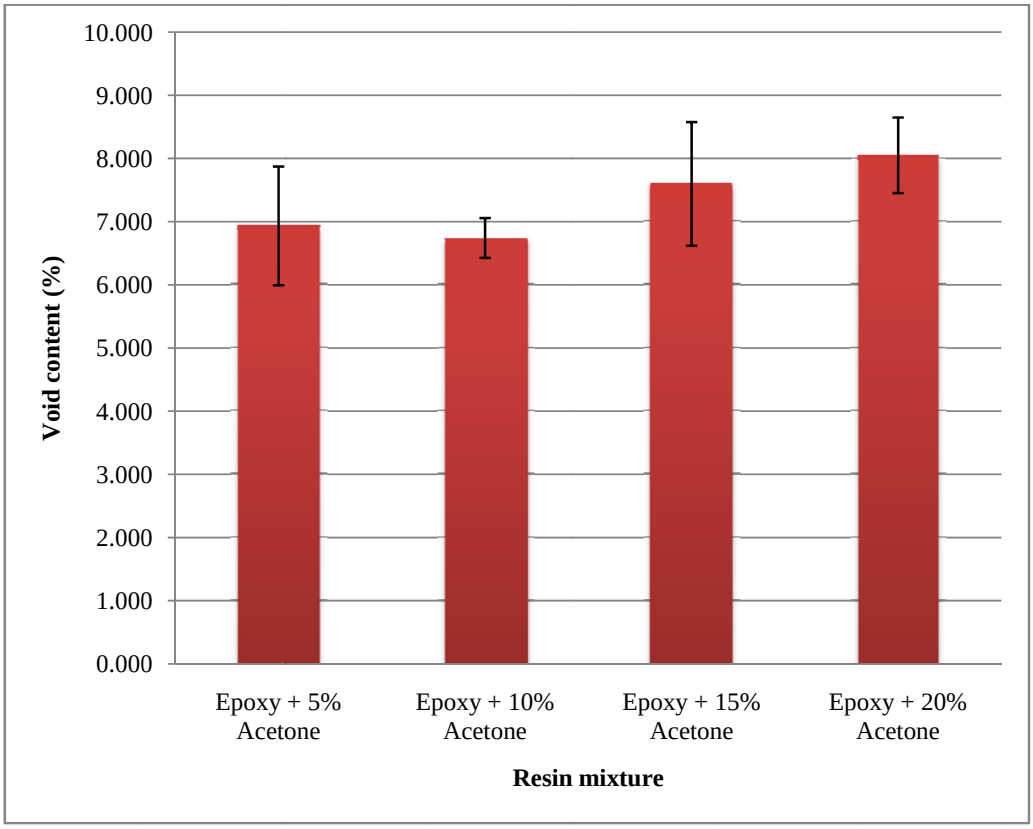


Figure 18: Void content

Theoretically, using a low viscosity of resin mixture can reduce the void content [17]. Acetone, in the experiment, was used as a solvent to dilute the resin mixture as it decreases the resin viscosity. This mean that epoxy with 20% addition of acetone has a lowest viscosity value. From the data analysis, void content vary for different resin viscosity used and epoxy with 10% acetone mixture had a lowest average value of void compared to other mixtures. Meanwhile, resin mixture with 20% of acetone had higher void content and this was because the viscosity mixture was too low and this might increasing the possibility of air bubble entrapment. When the viscosity was too high, the air bubble was difficult to get out from the mixture during the degassing process and as a result, high void content was produced in the composite.

In the loss ignition test, three specimens were taken at different location from each sample in order to achieve the second objective of the project which is to produce mapping of void content. The location of each specimen was referred as distance from the resin inlet. Table 5 below shows the location of the each specimen.

Table 5: Location of each specimen

Resin Mixture	Sample Tag	Distance from Inlet (cm)	Void Content (%)
Epoxy + 5% Acetone	A1	8.4	7.123
	A2	9.2	5.913
	A3	15.9	7.765
Epoxy + 10% Acetone	B1	7.7	6.470
	B2	15.8	7.087
	B3	17.0	6.669
Epoxy + 15% Acetone	C1	7.7	8.577
	C2	16.0	7.597
	C3	27.3	6.620
Epoxy + 20% Acetone	D1	8.6	8.740
	D2	17.1	7.663
	D3	28.9	7.752

Based on the data gained from the analysis, a mapping of void content was produced for each sample. Figures 19 to 23 show the mapping for each sample.

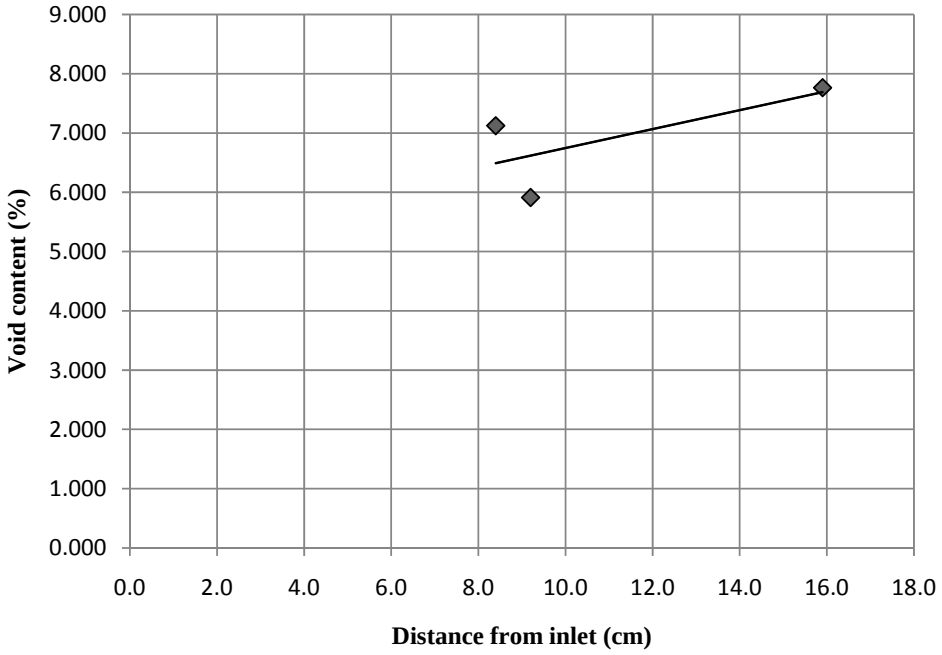


Figure 19: Mapping for epoxy + 5% acetone mixture

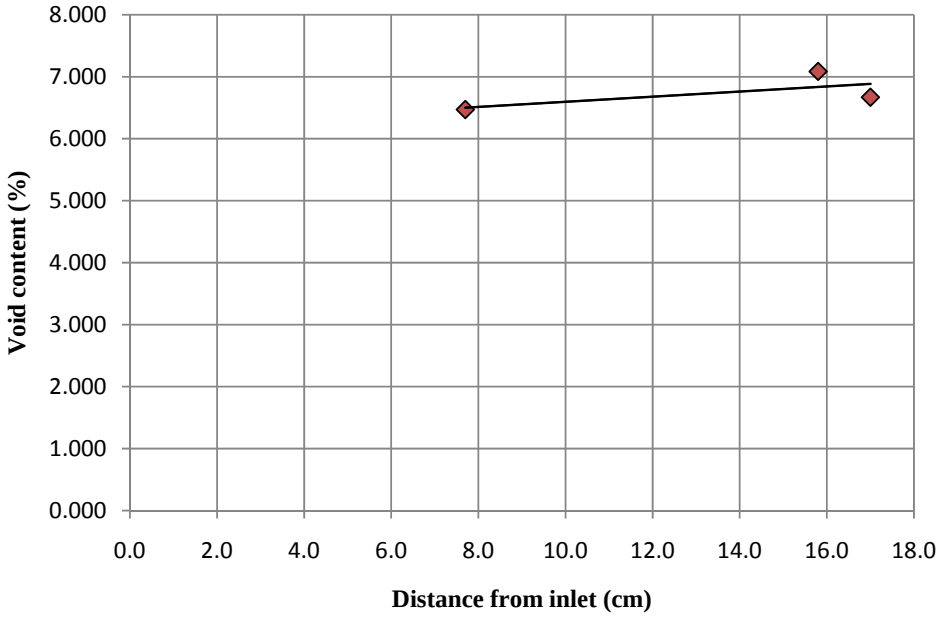


Figure 20: Mapping for epoxy + 10% acetone mixture

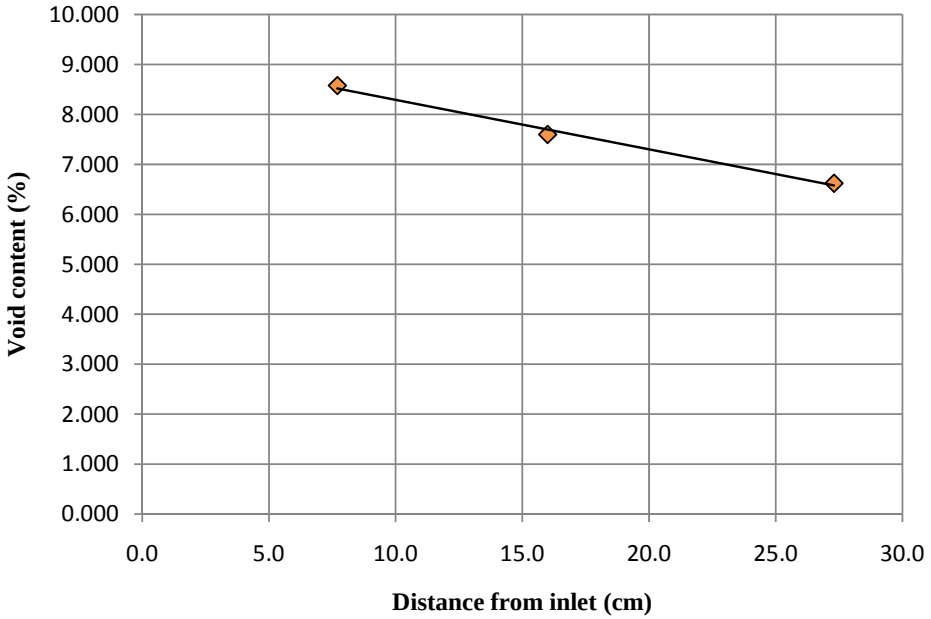


Figure 21: Mapping for epoxy + 15% acetone mixture

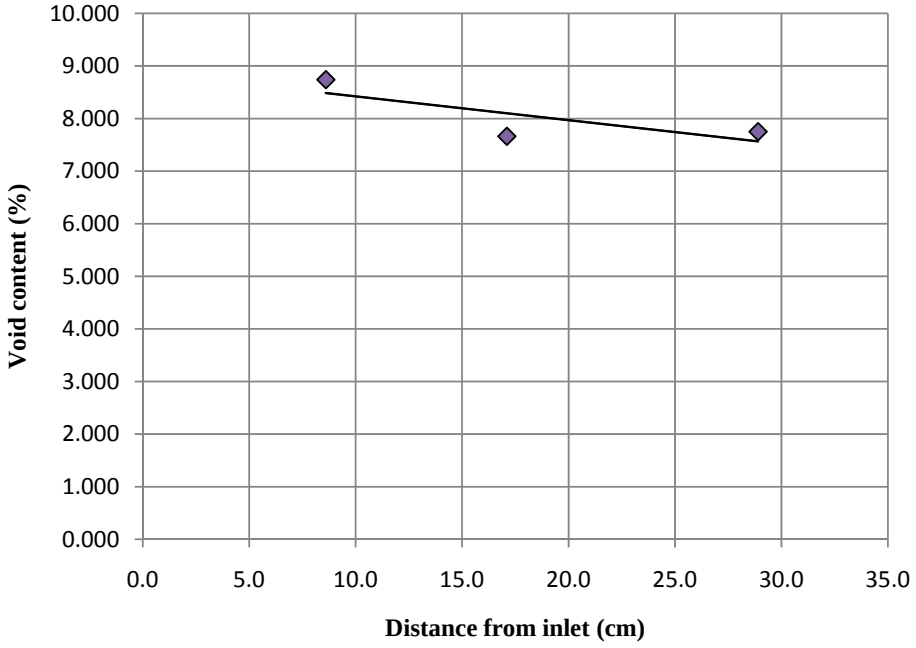


Figure 22: Mapping for epoxy + 20% acetone mixture

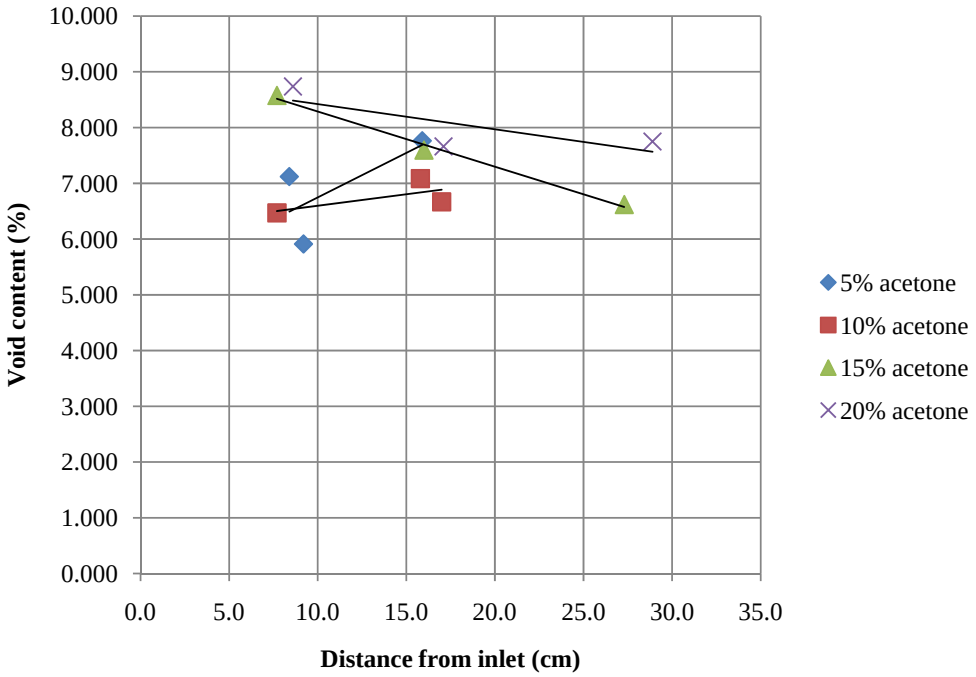


Figure 23: Mapping for all specimens

The reason of this mapping was to know the distribution of void in the composite. During the resin infusion process, the connection between the reinforcement to the resin and vacuum pump is a crucial part. If the connection is not done properly, vacuum condition cannot be achieved and this will cause the formation of void in the final product. In the experiment, there might be some leakage at connection. As a result, void tended to gather near the resin inlet. For the first two mixtures, 5% and 10%, void content increased as the distance from inlet increases but for the other mixtures, void decreased. The differences in the results might be caused by the properties of acetone that gave additional void in the final product and good mapping could not be achieved. So, the distribution of void could not be concluded whether increasing or decreasing if the distance from inlet increases. Besides that, in order to produce a good mapping, several specimens must be tested but in this project, only three specimens from each sample were tested. This is because of some limitations had occurred during the execution of the project such as limited of samples and equipments.

Apart from that, some assumptions had been made such as in the loss ignition test. If only the glass fabric is used as the reinforcement of an organic resin that is completely decomposed to volatile materials under the conditions of the test (loss ignition) and the small amount of volatiles that may present is ignored, the ignition loss can be considered to be the resin content of the sample [14]. Meanwhile, the density of the resin in the composite was assumed to be the same with the bulk resin density. Even there were some conditions that change the composite resin density such as differences in curing, heat and pressure, and molecular forces from the reinforcement surface [9], this assumption was unavoidable.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

Information on void content is useful because the presence of voids can significantly degrade the material properties even at low volume fraction. Monitoring void contents can act as a measure of the consistency of the composites' manufacturing process.

From the experiment and analysis done, the objectives of this project have been achieved successfully. By controlling one of the manufacturing parameter which is resin viscosity, the formation of void in the final product can be reduced. In this project, we tried to vary the viscosity of the resin by using acetone as a solvent and studied the differences on the void content for each sample. The result showed that the epoxy mixture with 10% addition of acetone produced lowest void compared to other mixtures.

5.2 RECOMMENDATIONS

Some recommendations can be made in order to improve the results such as:

1. Using other solvent to replace the acetone used in this project because acetone is easy to volatile and this can produce some bubbles inside the mixture during the infusion process.
2. In order to produce a good mapping, several specimens must be tested but in this project, only three specimens from each sample were tested. By testing more specimens, we can monitor the distribution of void in the composites.

REFERENCES

1. Retrieved on 2 August 2009 <http://www.resininfusion.info/>.
2. Hansong Huang, and Ramesh Talreja, 2005, "Effects of void geometry on elastic properties of unidirectional fiber reinforced composites", *Composites Science and Technology*, 65: 1964-1981.
3. Md Afendi M Yusuf, 2007, "The Effect of Micro-Bubbles Elimination Prior Resin Infusion Process", *Jurnal Mekanikal*, No. 24: 32-39.
4. S. N. Aqida, M. I. Ghazali, and J. Hashim, 2004, "Effects of Porosity on Mechanical Properties of Metal Matrix Composite: An Overview", *Jurnal Teknologi*, 40(A): 17-32.
5. Ling Liu, Bo-Ming Zhang, Dian-Fu Wang, and Zhan-Jun Wu, 2006, "Effects Of Cure Cycles on Void Content and Mechanical Properties of Composite Laminates", *Composite Structures*, 73: 303-309.
6. William D. Callister, Jr., 2007, *Materials Science and Engineering: An Introduction*, John Wiley & Sons, Inc.
7. A. Goren, and C. Atas, 2008, "Manufacturing of polymer matrix composites using vacuum assisted resin infusion moulding", *International Scientific Journal*, vol. 34: 117-120.
8. Vacuum Infusion, *The Equipment and Process of Resin Infusion.*
9. E. Cilley, D. Roylance, and N. Schneider, 1974, "Methods for Fiber and Void Measurement in Graphite/Epoxy Composites", *Composite Materials: Testing and Design (Third Conference), ASTM STP 546*, American Society for Testing and Materials: 273-249.
10. Retrieved on 2 August 2009 <http://www.ptli.com/testlopedia/tests/Void_content-D2734.asp>

11. R.Y. Yee, and T.S. Stephens, 1996, “A TGA Technique for Determining Graphite Fiber Content in Epoxy Composites”, *Thermochimica Acta 272:* 191-199.
12. Retrieved on 2 August 2009 <http://www.ptli.com/testlopedia/tests/Composite-Content-D3171.asp>
13. Annual Book of ASTM Standards, *Standard Test Methods for Void Content in Reinforced Plastics*, Vol. 08.02.
14. Santulli C, Garcia Gil R, Long AC, Clifford MJ, 2002, “Void content measurements in thermoplastic composite materials through image analysis from optical micrographs”, *Science and Engineering of Composite Materials*, 10 (2): 77-90
15. P. Pachpinyo, P. Lertprasertpong, S. Chuayjuljit, R. Sirisook, and V. Pimpan, 2006, “Preliminary Study on Preparation of Unsaturated Polyester Resin/Natural Rubber Latex Blends in the Presence of Dispersion Aids”, *Journal of Applied Polymer Science*, vol. 101: 4238-4241.
16. Annual Book of ASTM Standards, *Standard Test Methods for Ignition Loss of Cured Reinforced Resins*, Vol. 08.02.
17. J. Muric-Nesic, P. Compston, N. Noble, Z.H. Stachurski, 2009, “Effect of low frequency vibrations on void content in composite materials”, *Composites: Part A*, 40: 548–551.

APPENDICES