Investigation on the Mechanical Properties of Glass Fiber Reinforced Polymer (GFRP) Hybrid Composite with Eggshell Powder

by

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ENDORSEMENT

I, Lee Jie Shin hereby declare that all corrections and comments made by the supervisor and examiner have been taken consideration and rectified accordingly.

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Date:

DECLARATION

The thesis is the result of my own investigation, except where otherwise stated and has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any other degree.

(Signature of Student)

Date:

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INVESTIGATION ON THE MECHANICAL PROPERTIES OF GLASS FIBER REINFORCED POLYMER (GFRP) HYBRID COMPOSITE WITH EGGSHELL POWDER

ABSTRACT

This research explores the utilization of chicken eggshell powder, ESP as a bio-filler material in E-glass fiber reinforced epoxy. Eggshell is the domestic and industrial by-product which usually being disposed as agriculture waste. Such filler usually has low density, highly abundant, inexpensive and exhibits as a good flame-retardant material. It can be developed in an attempt to increase the strength of composites and reduced the overall cost. The aim of the research is to perform mechanical test on the GFRP hybrid composite infused with eggshell powder (ESP). The powder size used are 100 microns and 150 microns. The influence of the filler concentration on composite materials was investigated by means of their tensile and compressive strength. The collected chicken eggshells were cleaned with clear water. The eggshells with membrane removed were then dried, crushed, pulverized and sieved to obtain the powder filler. The laminates were manufactured using wet hand lay-up, incorporating with ESP filler loading of 0, 10 %, 20 %, 30 % and 40 % by weight percentage for both of the powder size. The average fiber volume was 0.34. The finding reveals that composite with smaller filler size would result in a better strength for both tension and compression. The tensile strength and compressive strength has increased by 18% and 30% respectively with the addition of 10 wt. % of 100 µm ESP. The results acquired from this research reveal the potential of eggshell wastes to be channeled towards the production of value added composite materials for sustainable development.

SIFAT-SIFAT MEKANIKAL KOMPOSIT HIBRID TETULANG GENTIAN KACA DENGAN KANDUNGAN SERBUK KULIT TELUR

ABSTRAK

Kajian ini berkisar tentang penggunaan serbuk kulit telur, ESP sebagai bahan pengisi bio dalam epoksi tetulang gentian kaca. Kulit telur yang merupakan bahan sisa pertanian adalah juga merupakan produk sampingan dalam pelbagai industri dan penggunaan domestik. Bahan pengisi ini mempunyai ketumpatan yang rendah, kuantiti yang banyak, kos berpatutan serta bertindak sebagai perencat api. Bahan ini berpotensi untuk meningkatkan kekuatan komposit, di samping mengurangkan kos menyeluruh. Kajian ini bertujuan untuk melaksanakan ujian mekanikal serbuk kulit telur, ESP terhadap komposit hibrid GFRP. Saiz serbuk yang digunakan ialah 100 micron dan 150 micron. Ketegangan dan kemampatan telah dikaji dalam penyelidikan ini. Kulit telur dikumpul dan dibersih dengan air bersih. Membran kulit telur dikeluarkan, seterusnya kulit telur dikering, dihancur, ditumbuk hingga lumat, serta diayak untuk menghasilkan serbuk kulit telur. Teknik “wet hand lay-up” digunakan untuk menghasilkan panel komposit yang melibatkan gentian kaca, epoksi dan bahan pengisi ESP yang berlainan kadar, yakni 0, 10 peratus, 20 peratus, 30 peratus dan 40 peratus. Hasil kajian menunjukkan bahawa komposit hibrid yang mengandungi kadar ESP yang rendah memaparkan keputusan tahap ketegangan dan kekuatan kemampatan yang memuaskan. Tahap ketegangan dan kekuatan kemampatan bertambah dengan 18 peratus dan 30 peratus dengan penambahan 100µm ESP sebanyak 10 wt. %. Keputusan kajian tindakan ini menunjukkan bahawa bahan sisa kulit telur berpotensi untuk disalurkan menjadi produk tambahan bahan komposit yang bernilai bagi pembangunan yang lestari.

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LIST OF ABBREVIATIONS

100-ESP10	: composite with 10% of 100μm eggshell powder
100-ESP20	: composite with 20% of 100μm eggshell powder
100-ESP30	: composite with 30% of 100μm eggshell powder
100-ESP40	: composite with 40% of 100μm eggshell powder
150-ESP10	: composite with 10% of 150μm eggshell powder
150-ESP20	: composite with 20% of 150μm eggshell powder
150-ESP30	: composite with 30% of 150μm eggshell powder
150-ESP40	: composite with 40% of 150μm eggshell powder
ASTM	: American Society for Testing and Materials
ESP	: eggshell powder
ESP0	: composite with no eggshell powder
GFRP	: glass fiber reinforced polymer
max	: maximum
min	: minimum
phr	: parts per hundred
wt	: weightage

LIST OF SYMBOL

%	: percentage
A_f	: areal weight of the fabric
M_d	: measured density
V_f	: fiber volume fraction
d_g	: density of glass
d_r	: density of resin
ρ_f	: density of fiber
σ_C	: compressive strength
σ_T	: tensile strength
E	: elastic modulus
ε	: strain
ºC	: degree Celsius
V_v	: void content, volume %
g	: glass, weight %
n	: number of fabric layers
r	: resin, weight %
t	: thickness of the laminate

CHAPTER 1 : INTRODUCTION

This introductory section provides a brief overview of the composites materials. It then goes on to describe the value of composite by introducing filler addition.

1.1 Background

Composite is a combination of two or more materials with significantly different physical or chemical properties that results in better properties than those of the composition acting individually. The constituents retain their identities; they do not dissolve or merge completely into each other although they act together. The applications have wide coverage, from a simple sporting good, to offshore piping and extensively use in boats and aircraft. The major consideration of employing composite materials in structural and transportation components is its weight savings capability.

A fiber is the primary load-carrying element of the composite material. Composite material is particularly strong and stiff in the longitudinal direction. Most of the fibers are brittle material with high specific strength and specific stiffness. However, the fibers have little or no transverse strength by themselves. The fiber volume fraction is a key factor to dictate the quality of the fiber reinforced plastic. Generally, the most common synthesis fibers for composite materials are carbon, glass, aramid, boron and ceramics.

Matrix is a bonding material in composites. The common matrix materials are thermosets, thermoplastics, metals and ceramics. Its main function is to hold fibers together, distributing loads between fibers while protecting fibers from the environment. Polymer matrix is usually less strong if compared to fibers, but it is tough. Hence, the fiber reinforced plastic would combine the good properties for both matrix and fiber, thereby producing an advance material with extraordinary strength to weight ratio.

Hybrid composite is defined as traditional composite material incorporated with two or more reinforcement with several phases (Bouhfid et al., 2018). The newly added reinforcement can be in any forms such as flakes and powder which are typically known as filler. The third constituent can be inorganic or organic, continuous or discontinuous at macroscopic, microscopic or nanometer level. The hybrid composites which combine the advantages of all the reinforcements, can be designed based on the necessity of applications and design requirement with desired properties such as lightweight, wear resistance (Saritha et al., 2018).

Investigating hybrid composites is a continuing concern in order to develop a material with high compatibility with respect to its specific application. In this paper, hybrid composite with eggshell powder is studied by way of investigating the tensile strength and compressive strength. Specifically, the effect of eggshell powder as the filler is ascertained based upon the filler size and filler loading.

The reason for choosing eggshells as the filler in this research is that it was realized that the disposal of a substantial amount of eggshells could create pollution. Kang et al. (2010) pointed out that chicken eggshell has been listed worldwide as one of the most overwhelms environmental problems. However, this problem could not be solved by reducing the consumption of eggs. Likewise, it is impossible to stop human from consuming eggs across the world. Therefore, this kind of waste will be generated repeatedly without exceptions and reversal. In short, food waste treatment or some attempts must be developed to minimize its pollution impact on the environment.

Figure 1.1: Eggshell waste.

1.2 Problem Statement

Several million tons of eggshells are being generated as bio-waste across the world every day (Wu et al., 2016). The consumption of egg is increasing due to the growth of population worldwide. The disposal of eggshell waste rises with egg consumption. However, eggshells have the potential to become a reusable material in order to highlight the sustainability features on the materials. Eggshells are abundant, non-costly, and low density.

Composites are made up of resin and fiber as the reinforcement. The combination of the constituents would enhance its physical and chemical properties especially its specific strength. In modern engineering, the design flexibility in composite materials allows their use in automobiles, space, aeronautical, sports good related application and industries (Nayak et al., 2015). This is due to their advantages such as lightweight, corrosion resistance, high fatigue strength and tailorable. However, the current composite materials' constituents are not sustainable. Adding natural fillers into composites might cut the overall cost by reducing the requirement of resin (Hassan and Aigbodion, 2015). Therefore, this project is to discover the latent mechanical behavior of eggshell powder as a bio-filler in composite materials without compromising its primitive properties.

The literature reviews which had been performed shows that there are few gaps which is further conversed. The previous studies were mostly about inserting the eggshells flakes or powder in thermoplastics, whereas the investigation with the thermoset polymers is limited. Meanwhile, the investigation with filler-filled epoxy is relatively less. Thus, it is interesting to investigate the influence of eggshell powder filler content in glass fiber reinforced epoxy based on its mechanical properties.

Moreover, the specimens that investigated by the previous researchers were basically neat resin with the eggshell fillers without employing fiber. There is a possibility that the fine eggshell powder would fuse into the space between the fiber bundle and hence affect the properties of the composite materials. The gaps between this project and previous researches are also including the processing way of the eggshells, the fabrication method of the specimens, the particle sizes, as well as the ASTM standards used for the mechanical tests. There were some researchers used chemicals such as sodium chloride and sodium hydroxide to process the eggshells. However, for the case of this final year project, chemical was not used because it might affect the properties of the eggshells alone which required further study. In addition, usage of chemical in processing eggshell may increase the manufacturing cost when it goes into the production phase.

1.3 Objectives

The objectives of this research are:

- To fabricate the glass fiber reinforced polymer (GFRP) hybrid composite with 100 microns and 150 microns of chicken eggshell powder at different filler loading by using wet hand lay-up.
- To perform tensile test and compression test on the hybrid composites.
- To study the tension and compression properties of the hybrid composites with different particle size and at different filler loading.

CHAPTER 2 : LITERATURE REVIEW

The purpose of this chapter is to review the literature on this topic. It begins by the basic ideas of the composites materials regarding its base material, reinforcement, fiber volume fraction and void content. The next part would move to the composite incorporating with natural filler.

2.1 Fiber Volume Fraction

Fiber volume fraction is the volume percentage of fiber that consists in composite materials. It would influence the strength and stiffness of the material (MÅNson et al., 2000). There are many ways to determine the fiber volume fraction such as Rules of Mixtures, acid digestion, and ignition. Despite having these methods, the fiber volume fraction of the samples in this research is calculated by using the below equation (Raheem, 2002). This is because this parameter is not a major concern in this study, and a constant number of fabric layer would be used to manufacture all the specimens.

$$V_f = \frac{n \times A_f}{\rho_f \times t} \qquad \text{Equation 2.1}$$

2.2 Void Content

Void is a very common detrimental defect in composite materials. In fact, it is an important aspect that can bring about negative impacts on the composites structure. Properties such as tensile strength, compressive strength, shear strength and fatigue life may be affected by voids. There is a consensus among researches that the presence of voids would affect the mechanical properties of the composites (Almeida and dos Santos Nogueira Neto, 1994, Yang et al., 2018). Furthermore, it was reported that the moisture absorption would be influenced by the void content (Costa et al., 2006).

For this purpose, the analysis of void content must be included in this research. As refer to ASTM D2734, the equation used in estimating the void content is as follow (ASTM, 2009) :

$$V = 100 - M_d\left(\frac{r}{d_r} + \frac{g}{d_g}\right) \quad \text{Equation 2.2}$$

Where

V	void content, volume %
M_d	measured density
r	resin, weight %
g	glass, weight %
d_r	density of resin
d_g	density of glass

There are several assumptions has been made with respect to Equation 2.2:

- The theoretical density of composite is of no interest.
- The density of the eggshell powder is neglected.
- The weight of ESP is not included in the void content estimation.
- The major concern is given to the reduction in the resin used.

2.3 Fillers in Composite

Due to economic and environmental apprehensions, natural fillers are usually employed by researchers to replace the synthesis fiber or as an extra reinforcement in composite materials. A considerable amount of literature has been published regarding natural fillers topic.

Krishnamurthi et al. (2003) studied the effect of wood flour fillers density and mechanical properties of polyurethane foams. The wood waste was obtained from the furniture industry. The size and concentration of the wood filler were varied and various mechanical properties have been studied. The investigation reveals that for the filler content ranging from 0 to 20 %, the compressive strength has decreased up to 3 times for particle size greater than 212 microns. Besides, the relative compressive strength has declined up to 10 times when the filler concentration is further increased to 80%.

In addition, natural fillers such as coconut shell, rice husk and teakwood were used by Sajith et al. (2016) in examining the viability of using lignocellulose fillers in conventional composites. These fillers are low cost and possess the potential to be alternative reinforcement for the expensive and non-renewable fillers. The study concludes that the deformability of the composites is affected by the filler size, mass percentage and filler type. Specifically, the filler type provides the most significant effect on the composite samples.

N.S.Mohan et al. (2016) has carried out an investigation on the hybrid composite by adding the coconut fiber in glass fiber-reinforced epoxy. From the result, the optimum tensile characteristic and flexural strength was provided by the sample with 6 % treated coir fiber while the best impact strength was obtained from the 4 % treated coir fiber specimens. However, this study found that strength decreases when the percentage of alkali treatment of the natural filler increases. The finding suggests that the degradation of natural fibers is triggered by the high percentage of chemical treatment.

A study by Hiremath et al. (2017) involved chicken feather in manufacturing the hybrid composites. The research suggested that it is possible to engage chicken feathers in designing lightweight composites, while this might be a remedy to reduce the disposal of the feathers. The experiment was carried out by using glass fiber and the specimens were prepared by hand lay-up. According to the paper, the hybrid composites with 10 wt. % of chicken feathers indicated the best tensile strength, flexural strength and impact strength.

2.4 Chicken Eggshell

Egg is rich in nutrients especially protein that can be transformed into a variety of mouthwatering food. Undeniably, the consumption of egg comes along with a lot of benefits especially for human's body development. In addition, eggshell is a byproduct from aviculture industries and considered as a bio-waste from hatcheries, fast-food industries, restaurants and domestic use. The growing population worldwide and the spring up of food industries would definitely increase the consumption of egg. Thus, generating considerable disposal of eggshells and indeed exacerbate land pollution. However, these eggshell wastes can be transformed into value-added product by means of sustainability due to their ample mineral contents within the eggshells. Its potential to become a filler material in the world of composites cannot be underestimated.

There is 95 % of calcium carbonate and 5 % of inorganic content exists in chicken eggshell (Hassen et al., 2015, Nayak et al., 2015). Its bulk quantity in calcium carbonate provides the opportunity for eggshell to become a latent replacement for commercial and synthetic calcium carbonate in industry. Many experiments have been carried out to study the replacement in an attempt to reduce the material cost.

Chicken eggshell contains 2300.33 mg/L calcium, 850.00 mg/L magnesium, 33.83 mg/L sodium, 17.06 mg/L potassium, 1.4 mg/L iron, 0.99 mg/L zinc and 0.063 mg/L copper (Ajala et al., 2018). The authors determined the chemical compositions of the chicken eggshell by using the atomic absorption spectrophotometer and flame photometer. The study outlined that there was 71.8 % of calcium in eggshell.

Research by Ashok et al. (2014) also pointed out that there is 90.5 % calcium carbonate, 6.8 % calcium hydroxide and 0.7 % calcium oxide, while the remaining compositions are made up of sulfated polysaccharides, type-X collagen. The large portion of calcium carbonate has made the eggshell a potential biocompatible material (Ganesan et al., 2018). Moreover, it was reported that the eggshell alone exhibits good properties, both mechanical and physical, such as tensile strength, compressive strength and low water absorption (Supri et al., 2010).

Anand Parkash and Singh (2017) conducted experiment on the behavior of concrete containing egg shell powder as cement replacing material. This paper presented an investigation on the influence of powdered eggshell on various properties such as compressive strength, split tensile strength, flexural strength and workability. These experiments were conducted to analyze the potential of eggshell powder as an alternative material to replace cement in a partially amount. In this research work, the eggshell powder used was 0 %, 6 %,12 %, 18 % and 24 %. As a results, the concrete sample with 12 % eggshell powder has obtained the optimum compressive strength and flexural strength. While, the maximum split tensile strength was obtained at 6 % replacement of eggshell powder.

2.5 Eggshell Powder as Filler in Composites

In recent years, a considerable amount of literature has been published on the use of eggshell flakes or powder as a filler in composites. Numerous studies have revealed that thermoplastic is the most common type of resin employed for the researches. In between, most of the authors (Toro et al., 2007, Shuhadah and Ghani, 2009, Hassan et al., 2012, Iyer and Torkelson, 2014, Hassen et al., 2015, Hiremath et al., 2018, Saisamorn Lumlong et al., 2018) used thermoplastics such as polyester, natural rubber, polypropylene and polyethylene as the base material. However, only a small number of participants have conducted the experiment by using thermoset: epoxy as the matrix (Nayak et al., 2015, S.Kenganal et al., 2017, Panchal et al., 2017, Petrasek and Müller, 2017). Different authors have investigated the effect of eggshell filler on the properties of composites in a variety of ways. Given all that has been mentioned so far, the methodology carried out by each of the researchers was different in terms of eggshell processing, type of equipment, standards referred, mechanical tests performed.

The relationship between the addition of eggshell in metal matrix composites and the mechanical properties has been examined by Chaithanyasai et al. (2014). In the study, 5 %, 10 % and 15 % of white eggshell powder were added into Aluminum 6061. The analysis reported the decrement in the density and electrical conductivity and an increment in the Brinell hardness when the filler loading increased.

In an investigation into the effect of eggshell powder on the flexural strength and hardness of composites, Hiremath et al. (2018) impregnated 5 % wt and 10 % wt of ESP in polyester resin and 50 % of glass fiber. As a result, the sample with 5 % of ESP recorded the highest flexural strength and maximum deflection. The increment in flexural strength was about 40%. However, the hardness reduces when the filler was added.

Hassan et al. (2012) investigated the tensile, compressive, flexural, impact, density and hardness properties of the uncarbonized and carbonized eggshell particulates in polyester composite. The polyester used was thermosetting-type, and no fiber was engaged in fabrication. The main concern will be drawn on the addition of uncarbonized eggshell in the polymer. According to the paper, the ultimate tensile strength increases by 54.1 % with the addition of 40 % eggshell filler. For the compressive strength, it recorded a 15.1 % increase from 0 % to 50 % eggshell-added composites. There was a significant increase in hardness.

In the studies involved natural rubber, Saisamorn Lumlong et al. (2018) demonstrated the use of 0, 10phr, 20phr, 30phr, 40phr of eggshell in his research. An increase in the tensile strength and hardness was observed, but the highest value was obtained from the composite with 20phr eggshell. Conversely, Intharapat et al. (2012) found a steadily decrease in tensile strength, tear strength, elongation at break, and a rise in hardness when the filler's loading level was increasing.

There has been little quantitative analysis of the tensile and compression properties by applying eggshell powder in thermoset polymer. Starting from this point in the current section, the limited researches related to the topic by using epoxy are going to be reviewed.

One study by S.Kenganal et al. (2017) investigated the composites samples with 0 %, 10 % and 20 % weight fraction of eggshell based on the properties of flexural strength, Vickers hardness and water absorption. A remarkable procedure of the study is that a type of chemical, sodium hydroxide was used for eggshell cleaning purpose. There is the possibility that the chemical used would affect the overall properties of the samples. S.Kenganal et al. (2017) reported a fluctuating trend in the flexural strength. It has decreased at 10 % of filler loading and increased when the weightage of the filler was 20 %. On the other hand, the hardness and the water absorption of the samples increased with filler loading. Furthermore, Panchal et al. (2017) also found that the absorption of the composites increases with filler loading. Both research never involved fiber in their respective study.

Previously, Nayak et al. (2015) has studied the use of 150 microns eggshell fillers in E-glass/epoxy composites. Sodium chloride solution was used as the treatment to remove the membrane, not to mention that the process was carried out at 70 ºC. It uncovers a significant decrease in the tensile strength when the filler content is increasing. Similarly, Shuhadah and Ghani (2009) who used polyethylene as the matrix and sodium hydroxide for eggshell cleaning purpose also found that the tensile strength decreased with the addition of eggshell particulate.

In addition, the analysis conducted by Petrasek and Müller (2017) has introduced the mechanical properties of composites by involving 0 to 40 % of 100 microns eggshell powder, with 10 % weightage as the interval. The highest tensile strength was held by the composite sample with 10 % filler loading; the increment in the strength was about 20 %. Next, there was a decrease in the elongation at break when the filler loading increased.

CHAPTER 3 : METHODOLOGY

This chapter describes and discusses the methods used in this investigation. The first section provides information about the materials used. The second part moves on to describe in greater detail preparation of eggshell powder, following by the specimens' fabrication stage and mechanical tests performed. Figure 3.1 reveals the flow chart of this project.

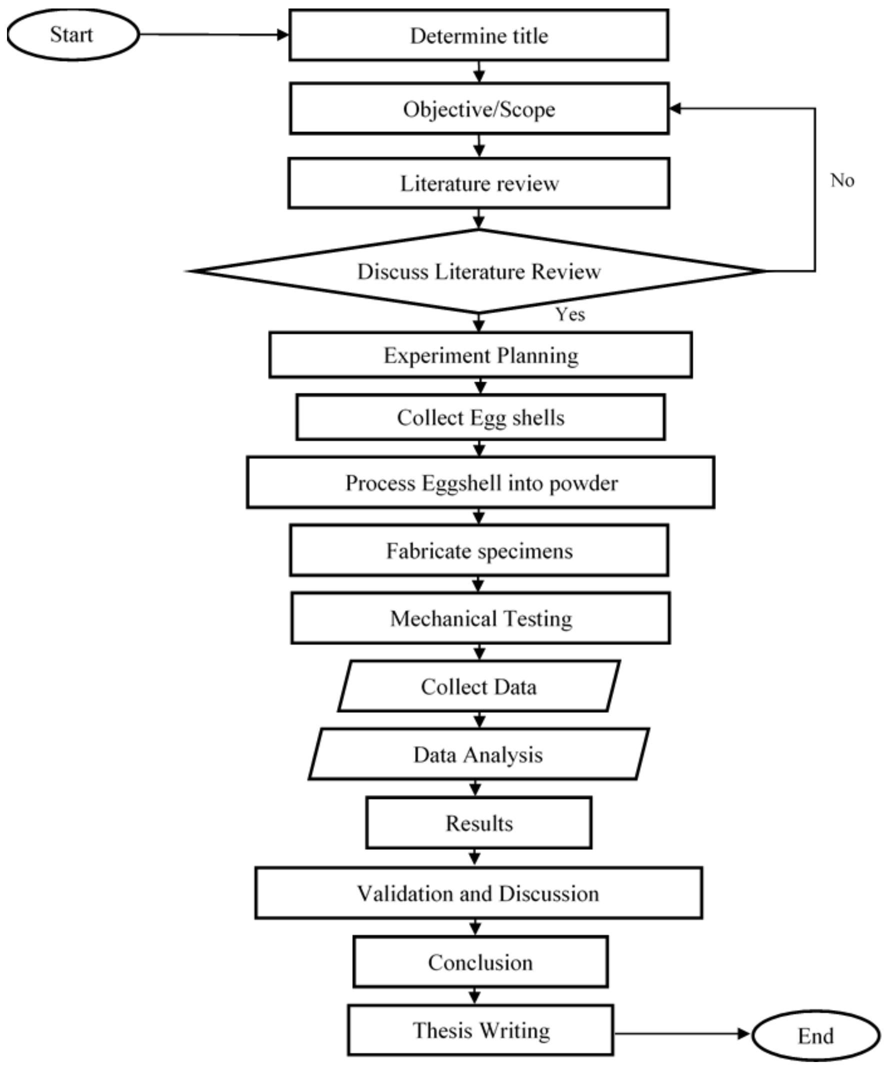


Figure 3.1: Project flow chart.

3.1 Materials

The general purpose epoxy, namely EpoxyAmite^{TM} 100 as pictured in Figure 3.2 together with the 102 MEDIUM hardener was used as the matrix material for the composite fabrication. Table 3.1 depicts the overview of the matrix's properties extracted from the datasheet provided by the manufacturer (SMOOTH-ON, 1895). For the reinforcement, four plies of 800gsm E-glass woven roving fabric as illustrated in Figure 3.3 were employed, and the number of ply of the fiber was kept constant for all the samples. The eggshell powder was added into the resin by weight percentage

Figure 3.2: EpoxyAmite^{TM} 100.

Table 3.1: Handling properties and physical properties of EpoxyAmite™ 100 Epoxy Laminating System with 102 Medium Hardener.

	102 Medium Hardener
Handling Properties	
Mix ratio by volume	3A : 1B
Mix ratio by weight	100A : 29B
Mixed viscosity-CPS ASTM D2393	650
Specific gravity- Mixed; g/cc (ASTM D1475)	1.11
Spec. volume- Mixed; cu.in/lb (ASTM D792)	25
Pot life - minutes (ASTM D2471)	22
Thin film working time- minutes	90
Cure time- hours	10 -15
Colour- Mixed	Clear yellow
Physical Properties	
Shore D Hardness (ASTM D2240)*	80
Ultimate tensile (ASTM D638*)	8180
Ultimate tensile (ASTM D638*)**	26800
Tensile Modulus (ASTM D638*)	450000
Tensile Elongation (ASTM D638*)	3.15
Flexural Strength- psi (ASTM D790*)	12220
Flexural Strength- psi (ASTM D790*)**	28500
Flexural Modulus- psi (ASTM D790*)	423000
Compressive strength- psi (ASTM D695*)	10970
Compressive modulus- psi (ASTM D695*)	122000
Heat Deflection Temp (ASTM D648*)	120 ºF / 49 ºC

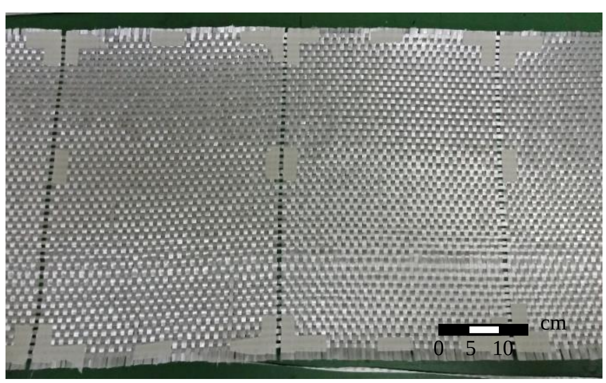


Figure 3.3: 800gsm E-glass Woven Roving Fiber.

3.2 Preparation of Eggshell Powder

The unwanted chicken eggshells were collected from the café in Engineering Campus Universiti Sains Malaysia until the desired amount was obtained.

3.2.1 Processing of Chicken Eggshell

The eggshells were washed thoroughly with cleaned water manually to remove the egg pulp residue and any dirt on the eggshell wall. At the same time, the membranes were removed easily with the help of water. To remove the moisture of the wetted eggshells, they were then dried under the sunlight for 24 hours. Next, they were gathered to be smashed into small flakes. The crushed eggshells were ready to go for the next procedure. The process is illustrated in Figure 3.4.

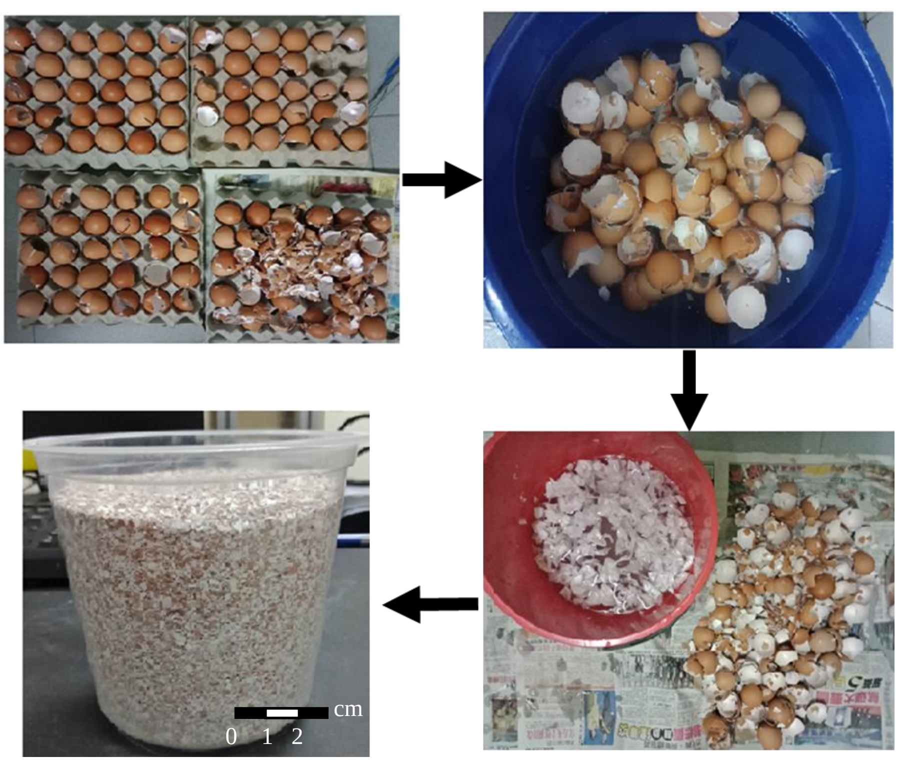


Figure 3.4: Eggshell processing.

3.2.2 Ball-milling

Ball-milling is an effective method to grind coarse material into fine and uniform size by utilizing the principle of centrifugal force. This grinding method was employed by Tsai et al. (2008), Chaithanyasai et al. (2014), Baláž et al. (2014), Nayak et al. (2015), Wu et al. (2016) and Ganesan et al. (2018) in pulverizing the eggshell particle in their respective study. For this research, the ball-milling machine used is portable and was built by Ir. Dr. Mohd Zulkifly Abdullah for his research. The rotational speed was unknown. In order to determine the speed of the portable ball mill machine, a tachometer and reflector were used to measure the rotation speed of the shaft that would drive the vessel.

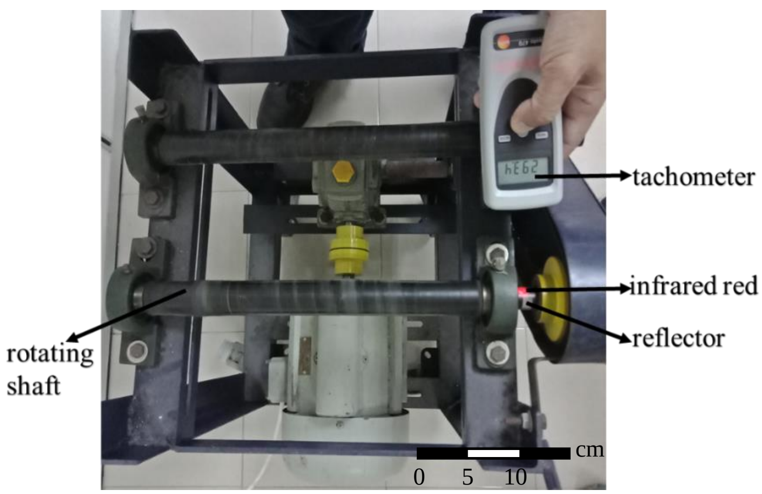


Figure 3.5: Measuring the speed of the ball mill machine.

The speed of the machine was 294.2 revolution per minute and data is tabulated in Table 3.2.

Table 3.2: Speed measured for the ball mill machine.

No.	1	2	3	4	5	Average
Speed (RPM)	293.8	295.0	293.2	294.3	294.7	294.2

Next, the eggshell flakes were feed into the ball-milling machine to be ground and pulverized for 4 hours. 20 steel balls with diameter 31.7 mm and another 20 balls with diameter 19 mm were used for grinding the eggshells. The larger balls were used to break down the coarse feed particles while the smaller balls could break down them to finer particulates by filling the void spaces between the balls.

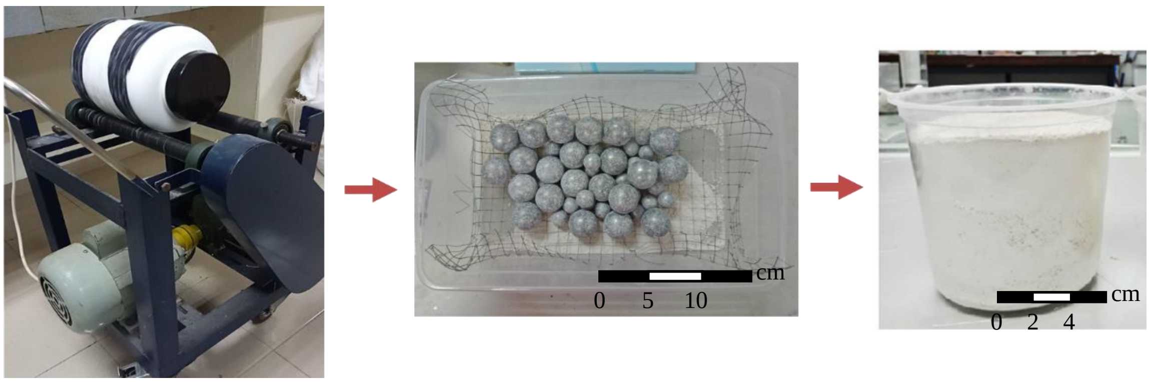


Figure 3.6: Grinding eggshell.

3.2.3 Determination of Eggshell Powder Size

The sieve shaker was then used to sieve the eggshell powder to obtain the desired particle size. The material and the mesh type was stainless steel and woven sieve respectively. Before the sieving process started, all the equipment was cleaned by using the air gun. This was to remove any residue that would blend with the eggshell powder. The sieve stacking sequence was started with the largest mesh size of 150µm placed at the top, followed with a subsequent sieve of 100 µm and a sieve pan at the bottom. The setup of the sieve shaker is shown in Figure 3.7 (a). Nayak et al. (2015) and S.Kenganal et al. (2017) grinded the eggshell powder to 150 µm while 100 µm of the filler was used by Petrasek and Müller (2017) in their respective study.

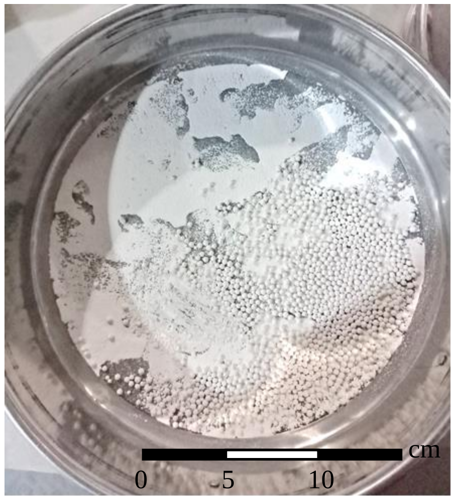
After setting up the equipment, 60 g of eggshell powder was placed at the top sieve and it was sieved for 15 minutes. The remains were then collected. To achieve the consistency of the powder size, the frequency, timing and the amount of the powder that underwent for each set of sieving process must be fixed. The processes were repeated until all the powder had filtered.

(a) (b) (c)

Figure 3.7: (a) sieving; (b) baking; (c) storing in desiccator.

3.2.4 Baking of Eggshell Powder

A very fine powder would absorb moisture from the air very easily. In other words, they are hygroscopic. As pictured in Figure 3.8 (a), the sieved powder formed into granules due to its ability of moisture absorption. Therefore, the powder was oven-baked at 80 ºC to remove its moisture content as shown in Figure 3.7 (b). It took four hours to bake in order to achieve the constant weight of the dried eggshell powder. Following this, the eggshell filler was stored in a desiccator that filled with silica gel desiccants to prevent its exposure to the air. Figure 3.7 (c) portrays the desiccator while Figure 3.8 shows the form of eggshell powder before and after bake. The powder was very fine after dried.



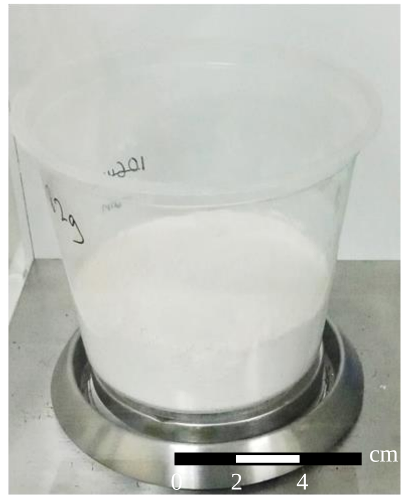
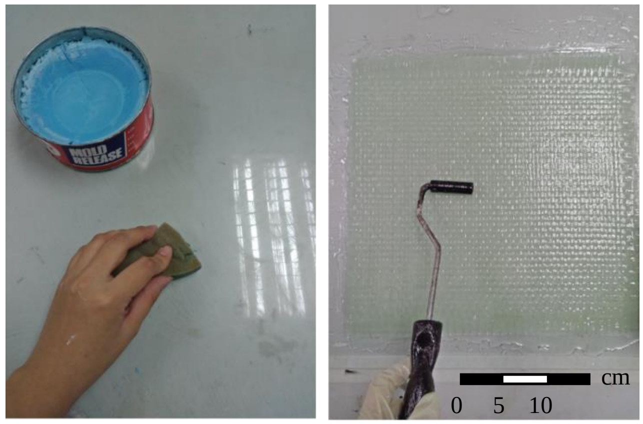


Figure 3.8: (a) before bake; (b) after bake.

3.3 Specimen Fabrication

4 pieces of woven glass fiber fabrics were cut out and their weight was recorded. Wax was applied to the working table to ease the demoulding process. Next, the resin was prepared by combining the epoxy and hardener. The ratio of epoxy to hardener was 100 : 29 as prescribed in Table 3.1. For the hybrid composites that contained filler, the eggshell powder was substituted in the matrix by weight percentage of 10 %, 20 %, 30 % and 40 %. The mixture was gently mixed well for 5 minutes. Wet hand layup technique as shown in Figure 3.9 (b) was used to manufacture the specimens. This fabrication method was also used by a number of researchers in their investigation (S.Kenganal et al., 2017, Nayak et al., 2015, Hiremath et al., 2018). The mixed resin was then poured onto the working table evenly. After that, the fiber fabric and the resin were applied alternately. It was made sure that every portion of the fiber got wetted throughout the process. On the completion of the lay-up process, the composites were left to cure at room temperature for 24 hours. The same

procedures were applied to fabricate the samples with 150 µm and 100 µm eggshell powder with various filler loadings.



(a) (b)

Figure 3.9: (a) Waxing the working table; (b) Wet hand lay-up.

After the specimens had cured, they were cut to the desired dimensions as refer to the ASTM standards for mechanical tests. Einhell wetted cutter machine as pictured in Figure 3.10 was employed during the specimen cutting process. After all, the specimens in Figure 3.11 were completed and ready for testing. It was observed that the higher the filler content, the darker the colour of the samples.

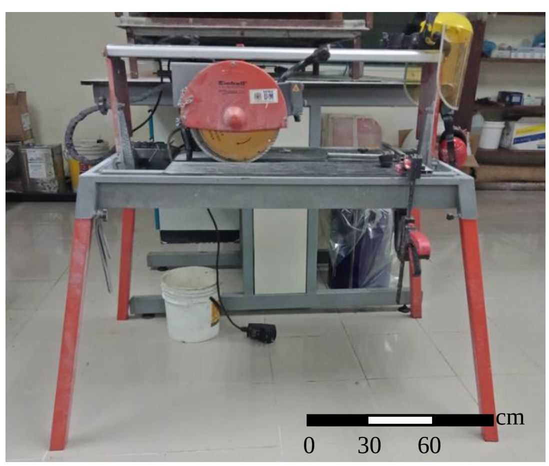


Figure 3.10: Einhell wetted cutter machine.

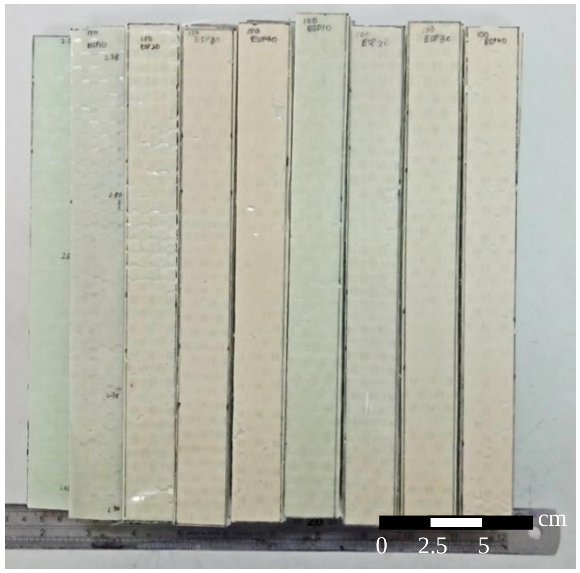


Figure 3.11: Finished samples.

Seven to ten specimens were prepared for each type of samples. In this dissertation, the naming of the types of sample was abbreviated as the figure below for the ease of data analysis.

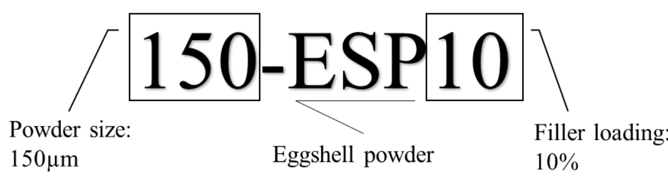


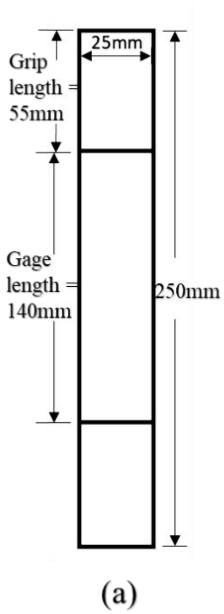
Figure 3.12: Abbreviation of sample's name.

3.4 Mechanical tests

The universal testing machine: Instron 3367 was used to test the tensile strength and compressive strength of all the samples. The properties that can be obtained including the ultimate strength, maximum elongation, failure load and tensile strain. The load cell for the machine was 30 kN.

3.4.1 Tensile Test

The test was conducted in accordance with ASTM D3039. Therefore, the specimen had a constant rectangular cross-section. As illustrated in Figure 3.13 (a), the width and length of the specimen was 25 mm × 250 mm (ASTM, 2014). In addition, the crosshead speed was set to 2 mm/min. For the tensile test, both ends of the specimen were mounted in the grips of the machine. Emery cloth was inserted for clamping to increase the friction between the clamped object and prevent slipping as well. The extensometer with a gauge length of 25 mm was then placed at the center of the specimen. The function of the extensometer was to detect the elongation of the specimen during the test. Once the test was started, the load was applied accordingly by the machine until the specimen failed.



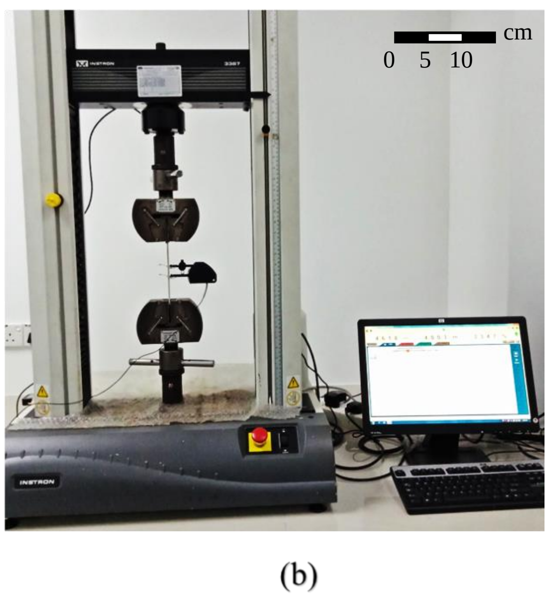


Figure 3.13: (a) Dimensions of specimen; (b) Setup for tensile test.