# **MODE 1 FRACTURE TOUGHNESS OF NATURAL**

# FIBRE REINFORCED COMPOSITES

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## ENDORSEMENT

I, Syaiful Nizam Bin Nazri hereby declare that all corrections and comments made by the supervisor and examiner have been taken consideration and rectified accordingly

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### DECLARATION

This 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.

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Date: 21 June 2019

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#### ABSTRACT

In general, a composite is a material made from two or more constituent materials of different properties. Nowadays, composites are highly utilized due to its highperformance properties that can be tailored into specified properties such as, high strength-to-weight ratio, wear resistance, corrosion resistance, inflammable properties, and etc. But one of the drawbacks is the cost is expensive. An alternative material for composites is by using natural fibre. Natural fibre reinforced composites constitute a current area of interest in the composite research for polymer composites due to their advantages of being low weight, low cost and environmentally friendly. In this study, the Mode I fracture toughness of the natural fibre reinforced composites will be analysed and evaluated thoroughly. While common mechanical properties of composites such as tensile, compression and flexure were measured in the predecessor studies, this study will focus on the manufacturing, testing and analysis of the stress intensity factor K<sub>IC</sub> which describes the fracture toughness of the composites. A fabrication method of compression moulding was used to manufacture the specimens for the test and analysis. A Mode I fracture toughness test was carried out by referring to ASTM D5045 standards and procedures. The results from the test were tabulated and plotted into a graph for analysis. The fracture resistance is determined in term of stress intensification factor,  $K_{IC}$ . The fracture toughness of the natural fibre reinforced composites shows about the same performance of the conventional composites but with the natural fibre reinforced composites are at a much lower cost.

#### ABSTRAK

Secara umumnya, komposit adalah bahan yang terbuat daripada dua atau lebih bahanbahan konstituen yang berlainan sifatnya. Pada masa kini, komposit kerap kali digunakan kerana ciri-cirinya yang berprestasi tinggi boleh disesuaikan ke dalam sifat-sifat tertentu seperti nisbah kekuatan dan berat yang tinggi, rintangan haus, rintangan kakisan, sifat tidak boleh terbakar, dan sebagainya. Tetapi salah satu kekurangannya ialah kosnya yang mahal. Bahan alternatif untuk komposit adalah dengan menggunakan gentian semulajadi. Komposit bertetulang gentian semulajadi merupakan bidang yang menarik minat dalam penyelidikan komposit untuk komposit polimer kerana kelebihannya adalah berat jisimnya yang rendah, kos rendah dan mesra alam. Dalam kajian ini, ketahanan retakan Mode I komposit bertetulang gentian semulajadi akan dianalisis dan dinilai dengan teliti. Walaupun sifat mekanikal komposit biasa seperti tegangan, mampatan dan fleksibel diukur dalam kajian terdahulu, kajian ini akan memberi tumpuan kepada pembuatan, pengujian dan analisis faktor intensitas tegasan, K<sub>IC</sub>, yang menggambarkan ketumpatan patah komposit. Kaedah fabrikasi pengacuan mampatan digunakan untuk menghasilkan spesimen untuk ujian dan analisis. Ujian ketahanan retakan Mode I telah dijalankan dengan merujuk kepada piawaian dan prosedur ASTM D5045. Keputusan dari ujian telah ditabulasi dan dijadikan grafik untuk dianalisis. Rintangan patah ditentukan dari segi faktor intensifikasi tegasan, K<sub>IC</sub>. Kekuatan patah komposit bertetulang serat semulajadi menunjukkan prestasi yang lebih kurang sama dengan komposit konvensional tetapi dengan komposit bertetulang gentian semulajadi adalah pada kos yang lebih rendah.

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# LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CFRP	Coconut fibre reinforced polymer
PFRP	Palm fibre reinforced polymer
GFRP	Glass fibre reinforced polymer

## NOMENCLATURE

K <sub>IC</sub>	: Stress intensity factor or fracture toughness, MPa $\cdotm^{1/2}$	
G <sub>IC</sub> /G	: Energy release rate, kJ/m <sup>2</sup>	
P <sub>Q</sub>	: Maximum load, kN	
В	: Specimen thickness, cm	
W	: Specimen width, cm	
a	: Crack length, cm	
X	: a/W	
U	: Area under the curve	
arphi	: Energy calibration factor	
u <sub>Q</sub>	: Displacement at maximum load, mm	
ui	: Displacement for indentation, mm	

#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.1 General Overview

A composite also known as Fibre Reinforced Polymer (FRP) are a combination of two or more materials with different properties. Composites are made up of a polymer matrix and a reinforcement which is fibre. The fibre provide the strength and stiffness while the matrix protects and transfers the load between the fibres. Composite materials are known for their high strength and stiffness with low density or weight. A conventional composites reinforcement such as glass fibres are usually expensive. An alternative reinforcement which is natural fibres constitute an interest in the composite research due to their advantages of being low weight, environmentally friendly, and most importantly low cost. In this study, coconut and palm fibres which are easily available in Malaysia were used due to its low cost, as well as superior mechanical properties such as modulus, stiffness and flexibility compared to glass fibres (Campbell, F.C., 2010). However, the natural fibres also have it drawbacks such as poor resistance toward moisture.

Generally, a conventional material means a material that is most likely or usually used in a product. For example, most of the aircraft before the advancement of composites used the aluminium alloys for its major structural elements. However, the conventional material pose some disadvantages mainly the weight. The weight of the parts are too heavy and thus the fuel consumption is high. A composites materials are design to overcome this problem because of its superior properties such as high strength to density ratio. It also can be tailored to any specifications or requirements that is needed. An increasing concerns on environmental health has led to the development of the natural fibre reinforced composites. A lot of interests in the natural fibre reinforced composites due to its advantages of being eco-friendly and easily available in resources. It also has wide potential of applications in various industry such as, aerospace, automotive and biomedical. Natural fibres such as jute, sisal, coir, hemps, as well as banana and pineapple leaves are generally extracted from renewable sources, fully biodegradable, non-toxic, and can be easily recycled to reduce the materials' carbon footprint (Dong *et al.*, 2014). A sustainable environment could be create while reducing the cost of manufacture a product.

Fracture toughness is a macroscopic property of material that indicate the resistance of the materials to propagate a pre-existing flaw. It is a very crucial property to study as the occurrence of flaws during fabrication or manufacturing is not completely avoidable. Flaws can be existed as voids, cracks, weld defects and etc. An assumption of flaw geometry and size will be occur in some components is a common practices and approach of linear elastic fracture mechanics will be use to design critical components. Basically the approach evaluate the ability to resist fracture of a component with flaw by estimating the flaw features and sizes, the component geometry, and the loading condition.

The fracture toughness properties of fibre-polymer composites are anisotropic where the highest fracture resistance occurring with in-plane fracture that involves breakage and pull-out of the fibres. Meanwhile the lowest fracture resistance occur by interlaminar cracking (Mouritz, A.P., 2012). To improve the fracture toughness of a fibre-polymer composites, it can be done by using thermoplastic interleaving, toughening resins, through thickness reinforcement such as pinning, stitching as well as other process (Mouritz, A.P., 2012).

The mode of fracture are divided into three mode, Mode I fracture is a condition where the direction of the tensile loading is normal to the crack plane. The fracture resistance is determined in term of stress intensification factor,  $K_{IC}$ . The property of  $K_{IC}$  characterizes the resistance of a material to fracture in the presence of a sharp crack under severe tensile constraints.

#### **1.2** Motivation and Problem Statements

Since the development of composite materials mainly in aerospace industry, the interest move towards on making a better composite materials as it has superior property such as exceptional strength and stiffness to density ratio. However, there is an environmental concern on the material to produce the composite mainly the reinforcement or the fibre used. In the aerospace industry, commonly used composites are carbon fibre reinforced polymer (CFRP) and glass fibre reinforced polymer (GFRP). The material such as carbon fibre and glass fibre are difficult to obtain and expensive especially the carbon fibre. This triggered the interest onto alternative reinforcement mainly the natural fibre. Natural fibre are known for its availability, low cost and superior properties than the glass fibre but also with some drawbacks.

Numerous intensive studies have been done to evaluate the properties of the natural fibre reinforced polymer. Mainly research on the mechanical properties of the natural fibre reinforced composites have been done thoroughly and it has been proven its superior properties. While the mechanical properties have been studied, the macroscopic property cannot be neglected. Same as the conventional composites, natural fibre reinforced composites also exposed to the occurrence of flaws. This flaws will become some of the factor in failure of the composites. So, it is crucial to study the ability of the natural fibre reinforced composites to resist the propagation of a pre-existing flaws or cracks.

## **1.3** Objectives of Research

The studies of this research were based on the following objectives:

- (i) Fabricate specimen of bio-composite using natural fibre for testing purpose.
- (ii) Perform fracture toughness test onto the specimens.
- Study and analyse the stress intensity factor K<sub>IC</sub> which describes the fracture toughness of natural fibre reinforced composites.
- (iv) To compare the fracture toughness between nature fibre and glass fibre composites.

#### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Composite materials

Generally, composite also known as Fibre Reinforced Polymer (FRP) is a combination of two or more materials which resulted in superior properties than the individual material components. Each component materials do not completely disappeared or lose their properties, they combine and contribute their useful properties to improve the final product (CompositesLab, 2019). Many interests have been towards to composites materials due to its advantages of being high strength and stiffness will maintaining a low density compared to bulk materials. This allows for a significant weight reduction in the finished parts.

Composites are made of a polymer matrix that is reinforced with a fibre whether man-made or natural fibre. Commonly used man-made composite materials especially in aerospace industry are carbon fibre reinforced polymer (CFRP) and glass fibre reinforced polymer (GFRP) which are consist of carbon and glass fibre respectively. The fibres are strong and stiff but brittle, while the polymer matrix are tough but lack strength and stiffness. The combination makes a composite with most or all of the benefits with few or none weaknesses of the individual components.

Furthermore, composites materials can be formed into more complex shapes or parts rather than their metallic counterparts. This is advantages as it reduces the number of parts for a given component, while also reduces the amount needed for joints and fasteners. In addition, the joint and fastener are the weak point of a component as it is the region where stress concentrated. Therefore, fewer joints and fasteners means less chance of failure of the component.

#### 2.2 Fibres or Reinforcement

As a composites, the fibres or reinforcement provides strength and stiffness to the composites (CompositesLab, 2019). Fibres can be oriented to provide properties according to the requirement in the direction of the loads. This is useful as it allows for optimized performance of a product which translates to reduction of weight and cost savings. The fibres can come in many forms to suit wide applications and requirements. Figure 2.1 shows examples and types of fibres forms. The forms include chopped strands, milled fibre, continuous and thermoformable mat. A continuous fibres have long aspect ratios with specific preferred orientation while a discontinuous fibres have short aspect ratio with random orientations. The continuous fibres with preferred orientation are anisotropic where the properties vary depending on the direction of the load with respect to the orientation for each layer to form a laminate. Mostly in aerospace industry, this approach is favourable where the layers are oriented differently in a specific sequence to meet the required properties of the laminate to be able to withstand the loads which it will be subjected.



Figure 2.1: types of fibres forms (Fei, L.Y., 2017)

### 2.3 Natural fibres

Mostly the natural fibres are mad of cellulose or plant which can be obtained from nearly every part of the plant such as root, leaf, stem and bark of a different tree species. They are also known as photosynthetic fibres, lignin and cellulose containing fibre. Examples of natural fibres include feather, wool, hair and silk fibres. It also can be extracted from a leaf which are fibrous, liable, strong and green. Table 2.1 shows the examples of fibres extraction from various plants (Fei, L.Y., 2017).

Root	Stem	Branch	Leaf	Fruit/Seed
Khus	Bamboo Kora grass Jute Hemp Water hyacinth Banana Kauna reed Cane palm Moonj grass Sarkanda Wagoo reed Sikki grass Cannabis/ pulla Wicker Bhindi Nettle Flax Arhar/Pigeon pea	Willow	Palmyra Palm date Palm coconut Arecanut palm <i>Sisal</i> Banana Pineapple Screw pine	Cotton Coir Arecanut

Table 2.1: Fibres extraction from various plants (Fei, L.Y., 2017)

In the recent years, the interest of composites area has been towards to the application of the natural fibres as the reinforcements for the polymer matrix. The low cost and superior properties make the natural fibres more favourable than the manmade fibres. It has a good potential as reinforcement in thermoset and thermoplastic polymers due to its low density and high specific properties. Natural fibres have the properties, composition, structures and features that are suitable to be used as reinforcements or fillers in polymer composites (Salit, Mohd Sapuan., 2016).

#### 2.3.1 Advantages of natural fibres

Natural fibres have some advantages compared to other conventional reinforcement (Salit, Mohd Sapuan., 2016). The advantages are mainly it low in cost and renewable, abundant and has continuous supply of raw materials. It also could become potential source of income for agricultural community. The natural fibre is non-toxic and environmentally friendly which free from health hazard and fully biodegradable. Then it also reduce dermal and respiratory irritation (Salit, Mohd Sapuan., 2016).

#### 2.3.2 Disadvantages of natural fibres

However, they also imposed some drawbacks which are it has low resistance to moisture and poor fire resistance. The fibre also degrade after being stored for a long time. During processing, it tends to form aggregates and has restricted maximum processing temperature. Then, it has bad compatibility with hydrophobic polymer matrix. (Salit, Mohd Sapuan., 2016).

#### 2.4 **Polymer matrix**

In composites, the matrix provides protection to the fibres from environmental and external damage, also transfers the load between the fibres. Polymer matrix composites are favourable due to their advantages of being low cost and easy fabrication method. Reinforcement of polymers by strong fibrous network allows fabrication of PMCs is characterized such as high coefficient of thermal expansion, good fatigue and impact resistance, high specific strength and stiffness, also good abrasion and corrosions resistance (Jose *et al.*, 2012).

Different properties of various polymers will determine the application which it will suitable. The polymer matrix can be classified to 3 types which is thermoset, thermoplastic and rubber. A thermoset polymers are used to make most composites nowadays. Through a process called cross-linking or polymerization, the polymers are converted from a liquid to solid. The thermoset polymers are cured by using a catalyst, heat or combination of both. Once curing has been achieved, they cannot be converted back to its original liquid form. Common thermosets are epoxy, polyester, vinyl ester and polyurethane. Meanwhile, a thermoplastic polymer does not cross-linked which resulted in it can be melted, formed, re-melted and re-formed. They are known for their ability to be able to shape and mould while in a heated semi-fluid state and become rigid when cooled. Thermoplastics cam be characterized by materials such as polyethylene, polystyrene, polycarbonate and ABS.

#### 2.5 Fabrication methods

#### 2.5.1 Compression moulding

Compression moulding is a method of high volume and high pressure which is suitable for moulding complex parts on a rapid cycle of time. It can be divided into several types defined by the type of material moulded (CompositesLab, 2019):

- Sheet moulding compound (SMC)
- Bulk moulding compound (BMC)
- Thick moulding compound (TMC)
- Wet lay-up compression moulding

A compression moulding tooling consists of a metal mould which are mounted in a hydraulic press. It enables for part design to be flexible and features such as ribs, inserts, attachments and bosses. Good surface finish can be obtained and thus contribute to lower cost of part finishing.

The reinforcement and matrix are placed in the metallic mould. The mould is then mounted onto a hydraulic or mechanical moulding press machine. The two halves of mould are closed and pressure is applied for a certain period of time. Curing of the composites can be done at room temperature or at some elevated temperature. After the composites have been cured, the mould is opened and the finished part is removed.



Figure 2.2: Compression moulding set-up (CompositesLab, 2019)

#### 2.5.2 Vacuum bagging

Vacuum bagging able to improve the mechanical properties of an open-mould laminates. The process of vacuum bagging can produce laminates which at a uniform degree of consolidation while at the same time, removing entrapped air, thus reducing the finished void content. It uses a flexible and transparent film to fully enclose and compact the wet laminate by reducing the pressure inside the vacuum bag, which resulted in external atmospheric pressure exerts force on the bag. The force exerted onto the laminates removes the entrapped air, excess resin and compacts the laminate which results in higher percentage of fibre reinforcement.

Commonly to be used with vacuum bagging are wet-lay laminates and prepreg advanced composites. In a wet lay-up bagging process, the reinforcement is saturated by using hand lay-up which then the vacuum bag is placed onto the mould and be used to compact the laminates and remove air voids. Meanwhile for the pre-impregnated advanced composites moulding, the prepreg material is laid up onto the mould which then the vacuum bag is placed. The mould is heated or placed in an autoclave that applies both external pressure and heat which adding the force of the atmospheric pressure. In aircraft and military products, the prepreg vacuum bag autoclave method is favourable to create advanced composites parts.



Figure 2.3: Vacuum bagging method (CompositesLab, 2019)

# **2.6** Fracture toughness of natural fibres/castor oil polyurethane composites (Silva *et al.*, 2006)

The main interest in studying the natural fibres reinforced composites are due to the fact that both fibres and matrix are derived from renewable resources. A polyurethane resins are interesting due to its structural versatility and compatibility to vegetable fibres in relation to other resins. This is due to the possible reaction of hydroxyl groups of the fibres and the isocyanate groups of the polyurethane (Silva *et al.*, 2006).

In these studies, the standard procedures for testing the fracture toughness of a composites can be followed to ASTM D5045. The test is developed for homogeneous materials while the composites naturally are heterogeneous which means the mechanical

properties are orthotropic, strongly dependent on direction. The conventional fracture mechanics could be applied but have to consider its limitations.

Commonly, the composites are manufactured with high elastic module fibres and brittle matrices. For these materials, an evaluation for fracture toughness can be made through Linear Elastic Fracture Mechanics concepts. The ability to resist fracture can be determined in terms of stress intensification factor, K<sub>IC</sub>, and energy release rate, G. The fracture toughness result for the natural fibre composites tested at 0.5 and 1.0 mm/min are shown in Figure 2.4. The area of interest in the result is the fracture toughness of the coconut short fibre composite. It can been observed that from a resin rich composites, the crack growth rate is locally accelerated as a result of the relatively low tenacity of the polymeric phase. Meanwhile, in the fibre rich composites, the crack is deflected and pinned by the reinforced obstacles so that its velocity tends to slow down, resulting in a tortuous crack path (Silva *et al.*, 2006).

The main energy absorption mechanism in composites are, crack deflection (tilting or twisting motion around the fibre), debonding between the fibre and matrix, pull-out (extraction of fibre from the matrix), and fibre bridging mechanism. Generally, numerous mechanism resulting in fracture toughness and it is difficult to determine which mechanism is the dominant mechanism (Silva *et al.*, 2006).



Figure 2.4: Fracture toughness for all short fiber and fabric composites (Silva et al.,

2006)

# 2.7 Effect of Interface Control on Mode I Interlaminar Fracture Toughness of Woven Carbon/Carbon Composite Laminates (Hojo *et al.*, 2002)

The aim of this research is to study the effect of fibre and matrix microstructure on the mode I interlaminar fracture toughness of composite materials by changing the heat treatment temperature (HTT) and using a coat of bismaleimide-triazine co-polymer (BT-resin) on the carbon fibre surface (Hojo *et al.*, 2002). For mode I interlaminar fracture toughness, the tests were based on JIS K 7086 standards and procedures.



Figure 2.5: Initial fracture toughness



Figure 2.6: Propagation values of fracture toughness



Figure 2.7: Electron micrographs for mode I fracture toughness (Hojo et al., 2002)

It can be observed that the laminates with HTT 1600°C for initial fracture toughness was unaffected to the weakening interface by BT-resin coating. Meanwhile it was increase during crack propagation by the coating. Next for laminates with HTT 2500°C, the initial fracture toughness and crack propagation decreased with BT-resin coating. A large bridged fibre at the crack wake was occurred for laminates with HTT of 1600°C. Meanwhile for laminates with HTT of 2500°C, the fracture surface was smooth

without bridging. The mesoscopic difference was responsible for the different sensitivity of interlaminar fracture toughness (Hojo *et al.*, 2002).

#### 2.8 Characterization

#### **2.8.1** Fracture toughness test – ASTM D5045 (ASTM International, 2013)

ASTM D5045 is used to measure plane-strain fracture toughness and strain energy release rate. The test are designed to characterize the toughness in terms of critical-stress-intensity factor,  $K_{IC}$ , and the critical energy release rate,  $G_{IC}$ , at fracture initiation. This test characterizes the ability of a material to resist fracture in a neutral environment in the presence of a sharp crack under severe tensile constraint. The property of stress intensification factor,  $K_{IC}$ , is believed to be the lower limiting value of fracture toughness (ASTM International, 2013). It is crucial to meet all of the criteria of the test as there is no assurance the validity of the value of  $K_{IC}$ .

The test involves either tension or three point bending with a notched specimen that has been pre-cracked. The value for stress intensification factor,  $K_{IC}$ , can be calculated from the load by a set of equation that has been established from the elastic stress analysis. The validity of the value  $K_{IC}$  can be determined by the establishment of a sharp-crack conditions at the tip of the crack to give linear elastic behaviour. Method to determine the energy release rate can be determined from the integration of the load versus load-point displacement graph, while adding a correction for indentation.



Figure 2.8: Specimen configurations for testing (ASTM International, 2013)

#### **CHAPTER 3**

#### METHODOLOGY

#### 3.1 Materials

For the composites, the reinforcement or fibres used are natural fibres which are coconut and palm fibres. The density of the coconut fibre is  $1.2 \ g/cm^3$ , while the density for palm fibre is  $1.01 \ g/cm^3$ . The natural fibres obtained has already in the form of sheets or mats which make it easier for the fabrication of the composites. The glass fibres used for this study was the chopped strand mat (CSM) with a density of  $450 \ g/m^2$ . Chopped strand mat was selected due to its properties of being randomly distributed which is similar to the natural fibres. The resin used was the polyester resin as the quantity is abundant and easy to obtain. The polyester resin was mixed with hardener at a ratio of 1:100 to obtain the matrix for the composites.



Figure 3.1: Coconut fibre in mat form

Materials	Images
Coconut fibres	4 cm
Palm fibres	4 cm
Chopped strand mat glass fibres	10 cm
Polyester resin	8 cm

Table 3.1: List of materials



Table 3.2: List of apparatus

Apparatus	Images
Aluminium base	8 cm





## **3.2 Fabrication process**

## 3.2.1 Manufacture of the mould frame

The mould frame consists of four aluminium bar as the mould and an aluminium plate as its base. The fabrication of the mould frame begins by cutting the aluminium bar

to its desired length so that the inner length of the mould would be 16cm x 16cm. The aluminium bars are then welded together to form a square shape mould.

Then the mould is glued onto the aluminium base with a hot glue gun at the entire mould so it does not move around. To prevent from the matrix leaked into the gap between the mould and the base, silicon sealant is used at the around of the inner part of the mould to cover any gap between the mould and the base.

#### 3.2.2 Preparation of the fibres

For the natural fibres, it came in the form of mat or sheets which really help in making the fabrication of the composites much easier. The dimensions set for the specimens are 16cm x 16cm, which follows the moulding. The fibres were cut into its dimensions, while the quantity or layers depends on the mass of the fibre required. The natural fibres in sheets form are non-compact materials which full of voids of air. This causes the fibres to become thick even for just a layer of fibres. This could cause trouble as the specimens also need to achieve specific thickness which is less than the thickness of overall layers of fibres. To overcome this, the sheet of fibres were rolled into a roller machine to compress the fibre into becoming a compact material and thus reduce the thickness of the fibre.



Figure 3.2: Palm fibre before rolling



Figure 3.3: Palm fibre after rolling

#### 3.2.3 Fabrication of the composites

The method used to fabricate the composites is the compression moulding. It is favourable as it is easy to manufacture and produce a good surface finishes composite. Before using the compression moulding, calculations have to be made to determine the amount of fibres and matrix required for specific dimensions or volume of specimens.

Calculations for the coconut fibre is as follows:

The required dimensions of composites  $=160mm \times 160mm \times 12mm$ 

=307200 mm<sup>3</sup>

The volume of fibre	=40% of the total volume
	=40% × 307200
	=122880 mm <sup>3</sup>

Mass of fibre needed

=Density of fibre  $\times$  volume of fibre

 $=1.2 \times 122.88$ 

=147.5g

Calculations for the palm fibre is as follows:

The required dimensions of composites	=307200 mm <sup>3</sup>
The volume of fibre	=122880 mm <sup>3</sup>
Mass of fibre needed	=Density of fibre × volume of fibre
	=1.01 × 122.88
	=124.1g