SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Degree of Bachelor Engineering in Chemical Engineering.

Signature	:	....
Name of Supervisor	:	ASSOCIATE PROFESSOR DR. MOHAMMAD DALOUR HOSSEN BEG
Date	:	....

STUDENT'S DECLARATION

I hereby declare that the work of this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature	:	....
Name	:	LEONG YI XUAN
ID Number	:	KA09049
Date	:	....

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PENGHASILAN DAN PENCIRIAN BAHAN BUMBUNG DARIPADA GENTIAN ASLI

ABSTRAK

Gentian asli diekstrakkan daripada tumbuh-tumbuhan dan mereka secara meluas digunakan sebagai tetulang komposit dalam menghasilkan bahan-bahan bumbung. Walaubagaimanapun, gentian asli menghadapi beberapa masalah seperti keboleh basahan, ketidak serasian dengan beberapa matriks polimer dan penyerapan kelembapan yang tinggi. Bahan bumbung moden juga mahal dan tidakter biodegradasi. Kos pengeluaran yang rendahdanbahan-bahan bumbung terbiodegradasikan boleh dihasilkan dengan menggunakan gentian semulajadi komposit bertetulang (NFRC). Kesandamar pengawetan ejen peratusan (RCA) dan serat jumlah lapisan NFRC pada sifat haba dan mekanikal telah dikaji. Gentian kenaf telah disediakan dalam polyester tikar berbentuk dan tak tepu telah disembuhkan dengan 10, 15 dan 20% daripada RCA (2-butanone peroxide). Kenaf-komposit poliester (KPC) telah disintesis dengan menggunakan 1 dan 2 lapisan gentian kenaf yang dicampur dengan polyester sembuh dengan menggunakan kaedah tangan meletakkan-atas. Selepasitu, specimen ujian untuk ujian tegangan, ujian hentaman Charpy dan ujian penyerapan air telah disediakan. Ujian mekanikal telah dijalankan untuk menilaisifat-sifat KPC. Tegangan dan kekuatan impak KPC telah meningkat dengan peratusan RCA dan lapisan gentian kenaf kerana kenaikan pemautkacukan antara rantaian polimer dan darjah halangan. Walaubagaimanapun, kenaikan lapisan serat meningkatkan penyerapan air komposit kerana sifat hydrophilic gentian kenaf. Ujian terma seperti analisis Termogravimetri (TGA) dan calorimeter pengimbasan kebezaan (DSC) analisis telah dijalankan untuk menilai kestabilan termakomposit. Kenaikan peratusan RCA dan lapisan gentian menurunkan suhu degradasi komposit yang menurunkan kestabilan termal komposit. Walaubagaimanapun, kenaikan peratusan RCA dan lapisan serat meningkatkan takat lebur komposit. Ia boleh membuat kesimpulan bahawa bahan-bahan bumbung yang sesuai akan dihasilkan oleh KPC selepas pengoptimuman jumlah lapisan gentian dan RCA kesan peratusan.

PRODUCTION AND CHARACTERISATION OF ROOFING MATERIALS FROM NATURAL FIBRES

ABSTRACT

Natural fibres are extracted from plants and they are widely being used as the reinforcement of composites in producing roofing materials. However, natural fibres are facing several problems such as poor wettability, incompatibility with some polymeric matrices and high moisture absorption. Modern roofing materials are also expensive and non-biodegradable. Low production cost and biodegradable roofing materials can be produced by using natural fibres reinforced composites (NFRC). The effects of resin curing agent (RCA) percentage and fibre layer amount of NFRC on thermal and mechanical properties were studied. The kenaf fibre was prepared in mat shaped and unsaturated polyester was cured with 10, 15 and 20% of RCA (2-Butanone peroxide). Kenaf-polyester composite (KPC) was synthesised by using 1 and 2 layers of kenaf fibre layer which mixed with cured polyester by using hand lay-up method. After that, test specimens for tensile test, Charpy impact test and water absorption test were prepared. Mechanical tests were carried out to evaluate the properties of KPC. The KPC tensile and impact strengths were increased with the RCA percentage and kenaf fibre layer due to the increment of cross linker between polymer chains and degree of obstruction. However, the increment of fibre layer increased the water absorption of composites due to the hydrophilic properties of kenaf fibre. Thermal tests like thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis were carried out to evaluate the thermal stability of composites. Increment of RCA percentages and fibre layers decreased the degradation temperatures of composites which decreased the thermal stability of composites. Nevertheless, increment of RCA percentage and fibre layers increased the melting points of composites. It could be concluded that roofing materials are suitable to be produced by KPC after the optimisation of fibre layer amount and RCA percentage effect.

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LIST OF ABBREVIATION

NF	Natural Fibre
NFRC	Natural Fibre Reinforced Composite
RCA	Resin Curing Agent
KF	Kenaf Fibre
KPC	Kenaf-Polyester Composite
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
T_d	Degradation Temperature
T_{max}	Temperature of Maximum Degradation
TM	Tensile Modulus
TS	Tensile Strength

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Natural fibre (NF), often referred to as vegetable fibres, are extracted from plants. They are categorised by depending on the part of the plant they are extracted from. NFs are readily available in large quantities in many countries and they represent a continuous renewable source. There are various types of NF such as sisal, kenaf, flax, jute, oil palm empty fruit bunch, coir and so on. The characteristics for those different fibres are slightly different but all of them can be chemically or mechanically processed to enhance their properties.

Nowadays, the use of NF for the reinforcement of the composites has received increasing attention. This is because NF has many significant advantages over synthetic fibres. NF can be considered as naturally occurring composites consisting mainly of cellulose fibrils (fibres) embedded in lignin matrix (resin). These cellulose fibrils are aligned along the length of the fibre, irrespective of its origin. This type of alignment provides maximum tensile and flexural strengths. Besides, these fibres are high electrical resistance and also being thermally and acoustically insulating (Satyanarayana et al., 1990). Recently, many types of natural fibres have been investigated including flax, hemp, jute straw, wood, rice husk, wheat, grass, oil palm empty fruit bunch, kenaf, sisal, coir, banana fibre and pineapple leaf fibre.

Kenaf fibres provide high stiffness and strength values. They also have higher aspect ratios making them suitable to be used as reinforcement in polymer composites (Sanadi et al. 1995). Kenaf is an herbaceous annual plant, a warm-season annual row crop and can be obtained easily in Malaysia which main plantation distributed in Kelantan and Pahang. The attractive features of kenaf fibres are the low cost, lightweight, renewability, biodegradability and high specific mechanical properties. Kenaf has a bast fibre which contains 75% cellulose and 15% lignin and offers the advantages of being biodegradable and environmentally safe (Mansur & Aziz, 1983).

Composites are materials that comprise strong load carrying material (known as reinforcement) imbedded in weaker material (known as matrix). Reinforcement provides strength and rigidity, helping to support structural load. The matrix or binder maintains the position and orientation of the reinforcement (Taj, Munawar and Khan, 2007). Natural fibres reinforced composites (NFRC) are hybrid materials made of a polymer resin which reinforced by natural fibres. It combines the high mechanical and physical performance of the fibres and the appearance, bonding and physical properties of polymers (Rijswijk, Brouwer and Beukers, 2001). The use of

NF in polymeric matrices such as polyester, polyethylene, polypropylene and epoxy resins has been explored widely. For example, jute-epoxy composites, coir-polyester composites and sisal-polyethylene composites have been commonly applied on industry.

Recently, there is increasing demand in low cost housing. On the other hand, there us the lack of suitable low cost roofing. According to Cook et al. (1978), the roof is an essential component of housing which is critical to shelter, thermal comfort and privacy. In many developing countries, roofing alone represents more than 50% of the total construction cost of a low cost house. The application of NFRC have been explored widely especially in producing roofing material due to its characteristics. Therefore, further research on low cost and biodegradable roofing material should be continued by using NFRC.

1.2 Problem Statement

There is an increasing demand for low cost roofing materials especially in developing countries due to the growth of low cost housing. On the other hand, modern roofing materials such as corrugated iron and aluminium are very expensive and non-environmentally friendly. As mentioned above, roofing is an important component for a dwelling. Therefore, NFRC by using biodegradable polymer as matrices which are cheap, environmentally friendly, biodegradable and renewable are alternative materials in producing roofing panels.

However, NFRC are facing several problems such as poor wettability, incompatibility with some polymeric matrices and high moisture absorption. Composite materials which made from unmodified natural fibres will exhibit unsatisfactory mechanical properties. Therefore, surface treatment on natural fibre can be used prior to composite fabrication. The properties can also be improved by physical treatments and chemical treatments.

1.3 Research Objectives

The objectives of this study are:

(i) To produce low cost and biodegradable roofing materials from NFRC.

(ii) To study the effect of resin curing agent (RCA) percentage on the thermal and mechanical properties of NFRC.

(iii) To investigate the effect of kenaf fibre layer amount on the thermal and mechanical properties of NFRC.

1.4 Scope of Study

This research was carried out on the preparation and characterisation of kenaf fibre reinforced polyester composites. The scopes of study are as follow:

(i) Preparation of composites with various resin curing agent percentage (10, 15 and 20%).

(ii) Preparation of composites with various amount of kenaf fibre layers (1 and 2 layers).

(iii) Characterisations of composites with various mechanical (tensile test, charpy impact test and water absorption test) and thermal (thermogravimetric analysis and differential scanning calorimetry analysis) tests.

1.5 Significance of Study

This study is able to produce biodegradable roofing material which is more environmentally friendly compared to modern roofing materials such as corrugated iron and aluminium. Besides, the low production cost will also be suitable for rural and developing country in producing roofing materials.

This study is also able to determine the effect of resin curing agent percentage and fibre layers on composites and thus, the optimum mechanical and thermal properties of kenaf fibre reinforced composites can be obtained. Therefore, it is useful not only in producing roofing materials, but also useful in other manufacturing sectors which involve NFRC as the raw materials.

CHAPTER 2

LITERATURE REVIEW

2.1 Natural Fibres

Farmers around the world produce many types of natural fibres, planting crops and rearing animals. Natural fibre is a collection of cells having long length and negligible diameter. They can be obtained as continuous filaments or discrete elongated pieces similar to thread. They can be spun or twisted into yarn such as cloth and also can be converted into nonwoven fabircs, such as paper and felt (Islam et al., 2012). Furthermore, natural fibres can be considered as naturally occurring composites consisting mainly of cellulose fibrils (fibres) embedded in lignin matrix (Satyanarayana, Sukumaran, Mukherjee, Pavithran & Pillai, 1990). Natural fibres may be from the plant's fruit (eg. cotton or coir), stems (eg. flax, jute, kenaf and hemp) or leaf (eg. sisal). They are lignocelluloses based natural plant fibres which widely be used as reinforcements in the polymer composites.

Natural fibres are natural resources which are renewable and environmentally friendly. There is a growing interest in the development of natural fibres in industry because of their advantages. According to Satyanarayana et. al. (1990), natural fibres have the following advantages: (i) they can lead to high specific strength properties although they have poor strength properties due to low density. In addition, they are high resistance to crack propagation, (ii) they are having low cost and low energy consumption compared with synthetic fibres, (iii) they are non-toxic, (iv) Most of the scientific data on the structure and properties of natural fibres are well known, so that suitable applications for them can be found. In addition, compared to synthetic fibre, natural fibre has many advantages such as recoverability, biodegradation, flammability, non-toxicity, and other excellent properties (Qin et al., 2011).

Furthermore, a lot of work has been done to take advantage of the wide range in the properties of natural fibres. Arrakhiz et al., (2012) have reported that to achieve better mechanical and thermal properties, varying the fibre content, the chemical treatment used and the choice of the polymer matrix were studied. For instance, it is found that some mechanical properties as Young's modulus increased incredibly with increasing fibre loading. It was also mentioned some examples of a variability of chemical treatments used in the literature, to improve mechanical properties of composites. Moreover, the right choice of polymer matrix also had an impact on the stress rupture of the composites (Satyanarayana et al., 1990).

2.1.1 Kenaf Fibres

Kenaf fibre is obtained from stems of plants genus Hibiscus, family of Malvaceae and the species of H. Cannibinus. It requires less water to grow because it has growing cycle of 150 to 180 days with average yield of 1700kg/ha (Thiruchitrambalam, Alavudeen, Athijayamani, Venkateshwaran, & Elaya Perumal, 2009). Traditionally, kenaf bast fibres are used and known for rope, twine, and course sacking materials. Kenaf natural fibres are biodegradable and environmentally friendly crop and have been found to be important source fibres for composites and other industrial applications. Kenaf fibres also have a potential as reinforced fibre in thermosets and thermoplastics composites (Yuhazri et.al, 2011).

According to Thiruchitrambalam et. al. (2009), kenaf fibres have many advantages as reinforcement in fibre reinforced polymer composites which are low density, low cost, non-abrasiveness during processing, high specific mechanical properties, biodegradability, good thermal properties, sustainable and renewable sources. Therefore, kenaf fibre is chosen as the reinforcing fibre for natural fibre reinforced composites.

Figure 2.1: Kenaf Fibre
(**Source**: Cheng, Kuwn, Phongsakorn, Dan & Saifudin, 2009)

2.2 Composites

Composites are materials that comprise strong load carrying material (known as reinforcement) imbedded in weaker material (known as matrix). Reinforcement provides strength and rigidity, helping to support structural load. The matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement (Taj et. al., 2007)

According to Rijswijk et. al. (2001), composites are hybrid materials made of a polymer resin reinforced by fibres, combining the high mechanical and physical performance of the fibres and the appearance, bonding and physical properties of polymers. Therefore, the combination of natural fibre and polymer resin can improve the physical and mechanical properties which are suitable for various applications.

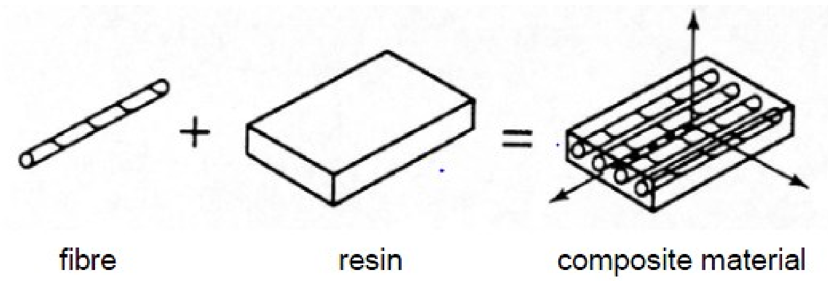


Figure 2.2: Composites Composition
(**Source**: Rijswijk et.al, 2001)

By this joining, the poor capabilities and drawbacks of the individual components disappear. For instance, composites combine a high stiffness and strength with a low weight and their corrosion resistance is often excellent. Composites have worked their way up amongst wood and metal due to their outstanding price performance ratio during a lifetime. A powerful approach in improving this ratio is to minimise the steps required from raw material to end product (Rijswijk et. al., 2001).

2.3 Natural Fibre Reinforced Composites

The combination of natural fibres as the reinforcement with the polymer resins contribute to natural fibre reinforced composites. According to Satyanarayana et. al. (1990), it is expected that when natural fibres are incorporated in low-modulus matrix such as polyester to become natural fibre reinforced composites, they would yield materials with better physical and mechanical properties which suitable for various applications.

In addition, natural fibre reinforced composites, by using biodegradable polymers as matrices, are the most environmental friendly materials, which can be composed at the end of their life cycle (Taj et. al., 2007). There are various types of natural fibre reinforced composites such as kenaf-polyester composites, sisal-polyethylene composites, coir-polyester composites, jute-epoxy composites and others. Each of them represents different type of physical and mechanical properties.

2.3.1 Kenaf-Polyester Composites

The combination of kenaf fibre as the reinforcement with the polyester as the polymer resins contribute to kenaf-polyester composites. There are many advantages of kenaf-polyester composites compared to other natural fibre reinforced composites. According to Thiruchitrambalam et al. (2009), the kenaf–polyester composites manufactured have a higher specific modulus than sisal, coir, and even E-glass thereby providing an opportunity for replacing existing materials with a higher strength, lower cost and more environmentally friendly materials. Therefore, kenaf-polyester fibre is chosen to be used in the production of roofing materials.

2.4 Methods of Improving Composites Properties

Natural fibres are hydrophilic in nature as they are derived from lignocellulose, which contain strongly polarized hydroxyl groups. Therefore, they are incompatible with hydrophobic thermoplastics such as polyester. According to John and Anandjiwala (2008), the major limitations of using natural fibres as reinforcements in polymer matrices include poor interfacial adhesion between polar-hydrophilic fibre and non-polar hydrophobic matrix, and difficulties in mixing due to poor wetting of the fibre with the matrix. This would lead to composites with weak interface.

According to Taj et. al. (2007) on the limitation of natural fibre reinforced composites; they are poor wettability, incompatibility with some polymeric matrices and high moisture absorption. Composite materials made with the use of unmodified plant fibres frequently exhibit unsatisfactory mechanical properties. Furthermore, lack of good interfacial adhesion and poor resistance to moisture absorption made the use of natural fibre reinforced composites less attractive (Joseph & Thomas, 1995).

Therefore, notable disadvantages of natural fibres are their polarity which makes it incompatible with hydrophobic matrix and their poor resistance to moisture absorption. However, these problems can be overcome by treating these fibres with suitable methods such as alkali treatment and surface modification through coupling agents.

2.4.1 Surface Modification through Silane Coupling Agent

These chemicals are hydrophilic compounds with different groups appended to silicon such that one end will interact with matrix and the other end can react with hydrophilic fibre, which act as a bridge between them (John & Anandjiwala, 2008).

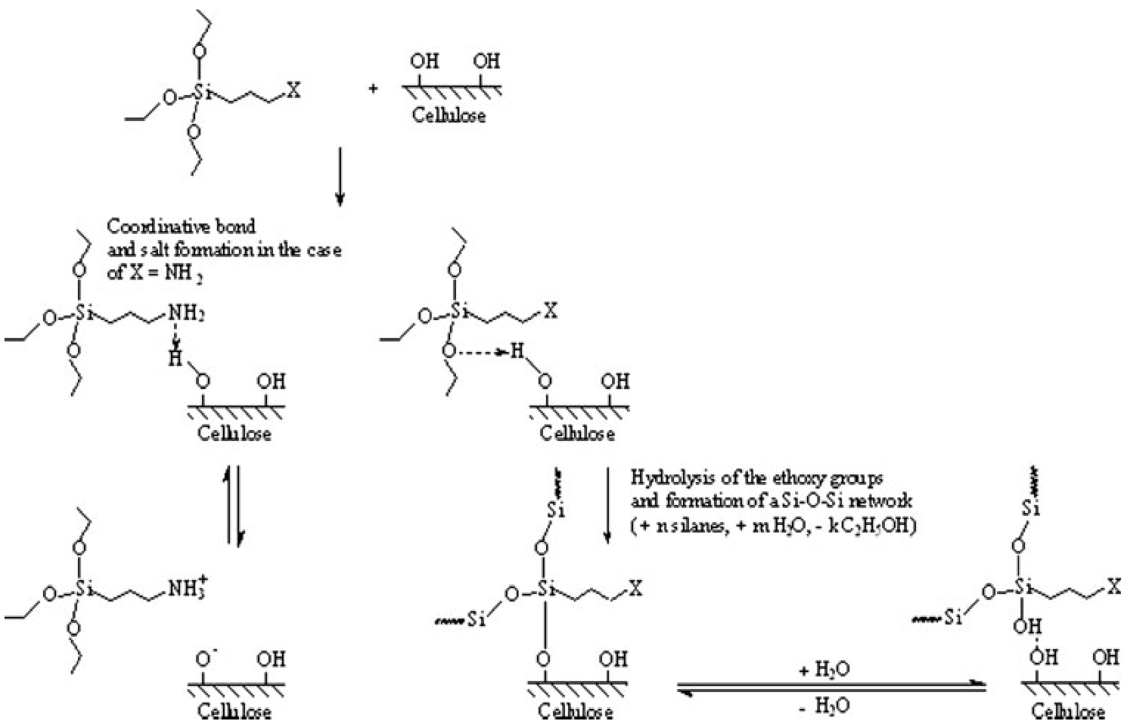


Figure 2.3: Interaction of Silanes with Cellulosic Fibres
(**Source**: John & Anandjiwala, 2008)

According to Taj et. al. (2007), silane chemical coupling presents three main advantages: (i) they are commercially available in large scale, (ii) at one end, they bear alkoxysilane groups capable of reacting with fibres OH-rich surface, and (iii) at the second end, they have a large number of functional groups which can be tailored as a function of the matrix to be used. Therefore, there will be a good compatibility between the natural fibre and the polymer matrix or even covalent bonds between them. However, the cost for this surface treatment is high.

2.4.2 Alkaline Treatment

Alkaline treatment leads to the increase in the amount of amorphous cellulose at the expense of crystalline cellulose. The important modification occurring here is the removal of hydrogen bonding in the network structure. In this structure, the OH-groups of the cellulose are converted into ONa-groups, expanding the dimensions of molecules. Subsequent rinsing with water will remove the linked Na-ions and convert the cellulose to a new crystalline structure (John & Anandjiwala, 2008).

Besides that, according to Taj et. al. (2007), the effect of chemical treatment of natural fibres with sodium hydroxide has been reported for coir, kenaf and sisal fibres. This modification results in an increase in adhesive bonding and thus improves ultimate tensile strength up to 30%. Joseph et. al. (1996) have also investigated the influence of chemical treatment with sodium hydroxide on the properties of kenaf-polyester composites. The observed enhancement in properties of the composites and attributed this to the strong bonding between kenaf and polyester matrix. Therefore, due to the advantages and the lower cost of alkaline treatment, it is chosen for the chemical treatment for kenaf fibre.

2.5 Production Methods

In order to produce roofing material from kenaf-polyester composites, there are many methods can be used such as hand lay-up method, extrusion moulding, injection moulding and compression moulding.

2.5.1 Hand Lay-up Method

The fibres which usually in mats shaped are cut and placed in a mould as shown in Figure 2.4. The resin is applied by rollers. One option is to cure while using a vacuum bag, then it's called vacuum bagging. By applying vacuum, excess air is removed and the atmospheric pressure exerts pressure to compact the composite. The advantages are the high flexibility and the simplicity of the process and the cheap tooling (Rijswijk et.al, 2001).

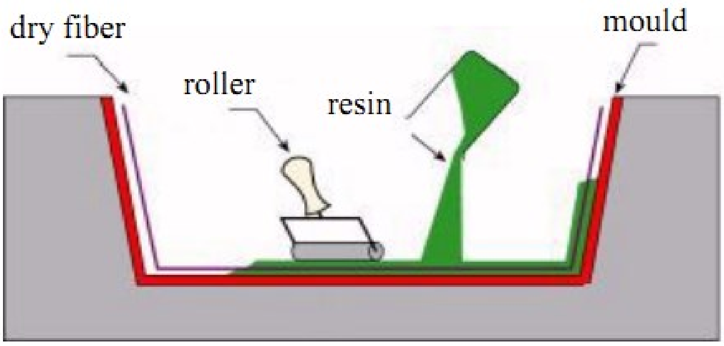


Figure 2.4: Hand Lay-up Method
(**Source**: Rijswijk et.al, 2001)

2.5.2 Extrusion Moulding

Extrusion moulding is a process of producing a continuous work piece by forcing molten plastic through a shaped die. As the hot material exits the die, the material is carried along a conveyor, cooled and after that cut to the desired length. In extrusion moulding, thermoplastic materials are fed from a hopper into the heated barrel of an extruder. A rotating helical screw inside the barrel pushes the plastic through the barrel toward the die located at the end of the machine (Santos et. al., 2007).

Extrusion moulding of plastics is used to make any long shape that has a constant cross section. Pipes, gutters, window sections and decorative trims can all be made using the process. Thermoplastics such as PVC (polyvinylchloride), LDPE (low-density polyethylene, or polythene), HDPE (high density polyethylene) and PP (polypropylene) can all be extruded. Thermoplastics are pliable when they are heated but form a rigid shape once cooled.

However, according to Takashima et. al. (2003) on the disadvantages of extrusion moulding, when the hot plastic exits the extruder, it frequently expands. The expansion of the plastic at this stage of the process is called die swell. Predicting the exact degree of expansion remains problematic as it arises from different factors in the process. Due to unpredictable expansion, we must often accept significant levels of deviation from the product dimensions or tolerance.

2.5.3 Compression Moulding

Compression moulding is a method of moulding in which the moulding material is generally preheated and then placed in an opened and heated mould cavity. The mould is closed with a top force or plug member, pressure is applied to force the material into contact with all mould areas, and heat and pressure are maintained until the moulding material has cured. The process employs thermosetting resins in a partially cured stage, either in the form of granules, putty-like masses, or preforms (Parasnis et. al., 2008).

Compression moulding is a high-volume and high-pressure method that which is suitable for moulding complex and high-strength fibreglass reinforcements. According to Orgéas et. al., (2008) on the advantages of compression moulding, unlike other composites processing systems, the compression moulding press is capable of reproducing fibre-reinforced plastic parts in significant volumes, with the accuracy, repeatability and high speed. Besides that, compression moulding also produces fewer knit lines and less fibre-length degradation than injection moulding.

Furthermore, as the inherent cost and performance advantages of this process, the resins in the compression moulders' gamut can be gradually expanded, from the early thermoset resins (phenolic, epoxy, melamine and urea) to unsaturated polyester (the ideal material for glass fibre-reinforced composite parts). With the development of polyester and polypropylene, compression moulding process has been modified to accommodate these low-cost commodity thermoplastics as well (Kim et. al., 2010).

CHAPTER 3

METHODOLOGY

3.1 Research Design

This study was to investigate the effect of resin curing agent (RCA) percentage and kenaf fibre layer amount on the thermal and mechanical properties of kenaf fibre reinforced with polyester composite (KPC). Firstly, kenaf fibre layers were prepared. Polyester was also prepared with different percentage of RCA. After that, hand lay-up method was used to produce KPC by kenaf fibre layers and cured polyester. The next step was composites characterisation. For mechanical properties, tensile test, charpy impact test and water absorption test were conducted while for thermal properties; thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis were accessed. The overall research flow chart was shown in Figure 3.1.

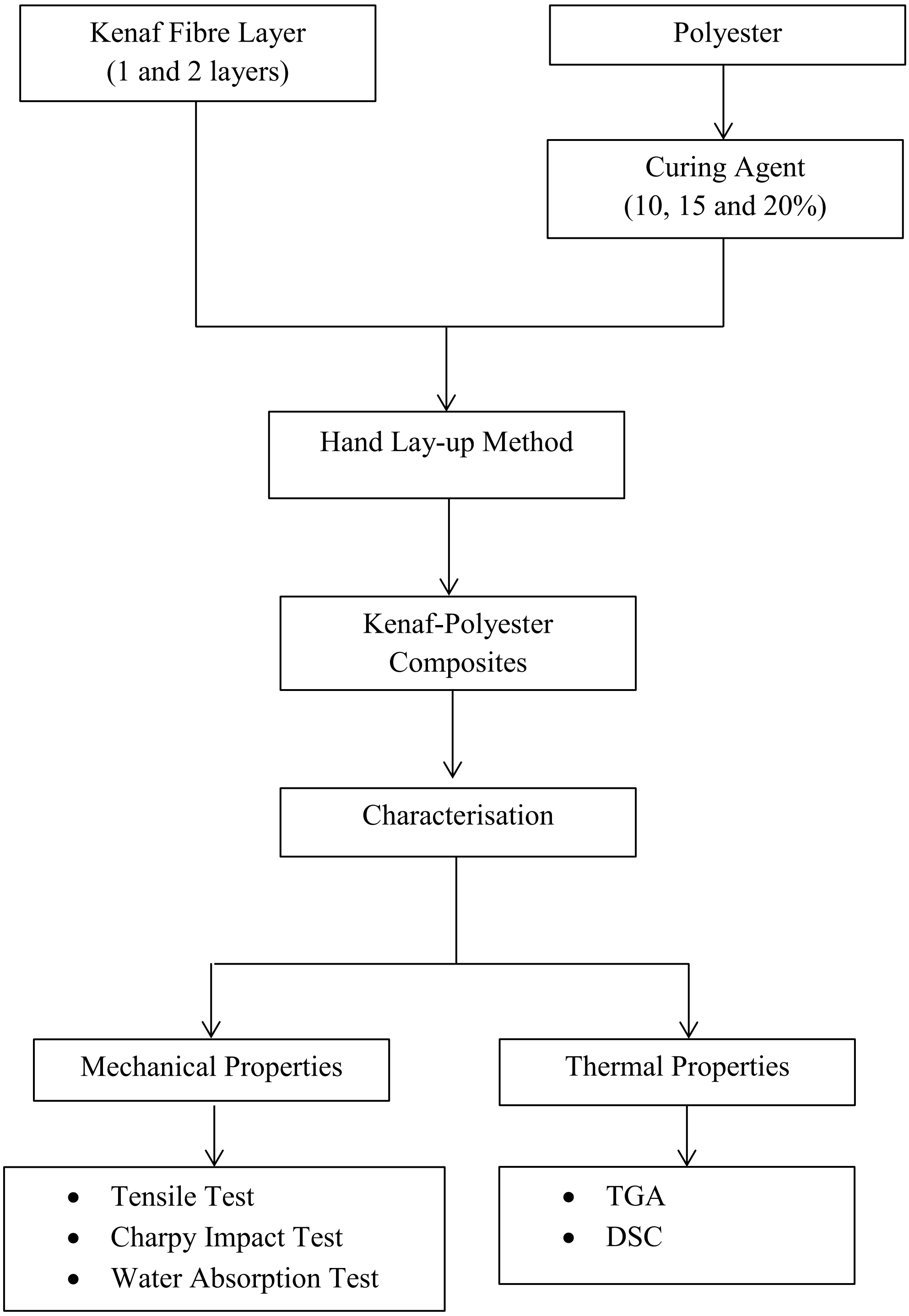


Figure 3.1: Flow Chart of Research Procedures

3.2 Materials

In order to produce kenaf-polyester composites, kenaf fibre and polyester were the main raw materials. Kenaf fibre was supplied by National Tobacco Authority (Lembaga Tembakau Negara) Terengganu. The kenaf fibre was supplied in the mat form as shown in Figure 3.2. For the polyester, it was supplied by Efficient Growth Sdn. Bhd., Selangor, Malaysia. The supplied polyester is in commercial grade. For the resin curing agent, 2-butanone peroxide was chosen to cure the polyester. It was supplied from local enterprise.

Figure 3.2: Kenaf Fibre Mat

3.3 Experiment Procedure

3.3.1 Preparation of the Composites (Hand Lay-Up Method)

First of all, the kenaf fibre mat was cut into 26mm x 26mm of size by using scissors. Then about 300ml of unsaturated polyester was pour into a 1000 ml beaker.

The polyester was then cured by resin curing agent (RCA). After that, acetone was used to clean the mould surface (mild steel 30mm x 30mm). Then, cured polyester was applied onto the mould. The mould was shown in Figure 3.3. Following by that, kenaf fibre was aligned manually on the cured polyester and then, cured polyester was applied on the kenaf fibre layer by using a brush (similar to painting technique). After that, the wet kenaf fibre was rolled with pressing force by using an aluminium roller to make them spread evenly. Lastly, the sample was left to cure overnight and the cured sample was peel off from the mould by using a wedge. The procedure was repeated by using different amount of kenaf fibre layers (1 and 2) and different RCA percentage (10, 15 and 20%). One of the cured samples (20% with 1 fibre layer) was shown in Figure 3.4 and the parameter of each sample was presented in Table 3.1.

Figure 3.3: Hand Lay-up Method Mould

Figure 3.4: KPC with 20% RCA and 1 Fibre Layer

Table 3.1: Parameters of Kenaf Fibre Layers and Rein Curing Agent Percentage

Code	Curing Agent Percentage (%)	No. of Kenaf Fibre Layer
10% + 2 layers	10	2
15% + 1 layer	15	1
15% + 2 layers	15	2
20% + 1 layer	20	1
20% + 2 layers	20	2

3.4 Testing and Characterisation

3.4.1 Mechanical Properties

3.4.1.1 Tensile Test

The tensile test was carried out using Universal Testing Machine AG-1 (Shidmadzu, Japan) as shown in Figure 3.5. The tensile tests were carried out according to ASTM D 638 – 01 and the composite specimen for tensile testing was shown in Figure 3.6. The machine was used as cross-head speed of 10mm/minute. The testing samples were positioned vertically in the grips of the testing machine. Then, the grips were tightened evenly and firmly to prevent any slippage with gauge length kept at 65mm.

Figure 3.5: Universal Testing Machine AG-1 (Tensile Test)

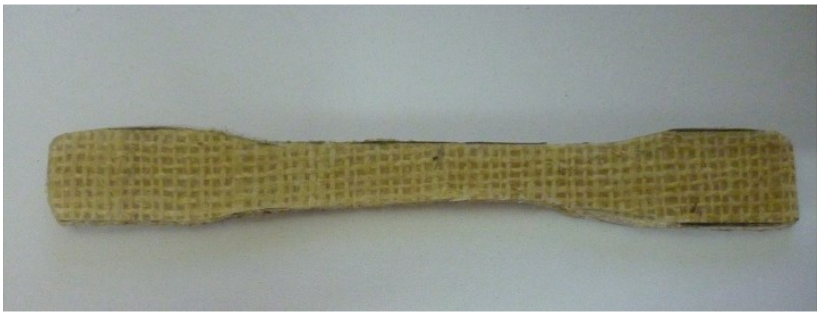


Figure 3.6: Composite Specimen for Tensile Testing

3.4.1.2 Charpy Impact Test

The charpy impact tests were performed using impact pendulum tester (model- ZWICK/ROELL) at 90^{o} swing angle with a hamme of load 1J as shown in Figure 3.7. The charpy impact tests were carried out according to ASTM D256 standard with rectangular specimen test bar with a fixed dimension (55 x 3.3 x 10mm). Davenport notch cutting apparatus were used to notch the specimen and the notch depth was fixed at 2mm with angle of $45^{o}C$ as shown in Figure 3.8.

Figure 3.7: Impact Pendulum Tester (Model- ZWICK/ROELL)

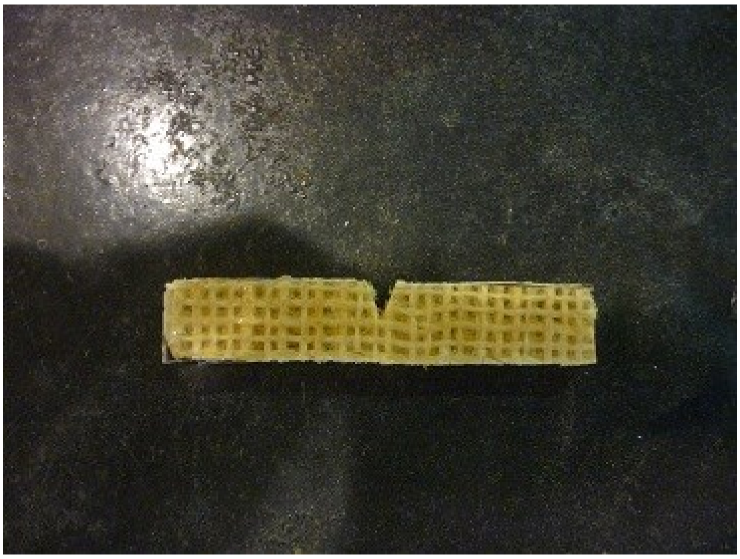


Figure 3.8: Notched Specimen for Charpy Impact Test

3.4.1.3 Water Absorption Test

Samples with dimension 50 x 10 x 3 mm were cut from tensile specimens and used to examine the water absorption behaviour after drying at room temperature for more than one month. Initial weight for each sample was measured. The samples were immersed in distilled water (27^oC) for 8 days and taken out of water at each 24 hours to measure the weight. The percentage of water absorption was calculated by the expression as shown below:

$$H = \frac{P - Po}{Po}\ x\ 100\% \qquad \text{(Eq. 3.1)}$$

Where P is the weight of specimen after immersed in water and P_O is the initial dried weight of the specimen.

3.4.2 Thermal Properties

3.4.2.1 Thermogravimetric Analysis (TGA)

TGA measurements were performed using thermogravimetric analyser (TA instrument, TGA Q500) as shown in Figure 3.9. Each specimen was measured about 5mg and analysed at scanning temperature range of 25-600^{o}C with the heating rate of 20^{o}C/min by using Ramp heating method. Degradation temperature values were evaluated as temperatures corresponding to the maximum rate of the weight loss.

Figure 3.9: Thermogravimetric Analyser (TA Instrument, TGA Q500)

3.4.2.2 Differential Scanning Calorimetry Analysis (DSC Analysis)

DSC analysis was carried out by using TA instrument, Q-1000 (Figure 3.10) with heat/cool/heat method. The heating range was 40-400^{o}C. The heating rate was 10^{o}C and the cooling rate was 5^{o}C. The samples were prepared in approximately 4-5mg and aluminium lid and pan as shown in Figure 3.11 were used in the test. The melting points, enthalpy and percentage of cystallinity of each sample were calculated by using TA instrument software.

Figure 3.10: TA instrument, Q-1000 (DSC Analysis)

Figure 3.11: Aluminium Lid and Pan with Samples Inside

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

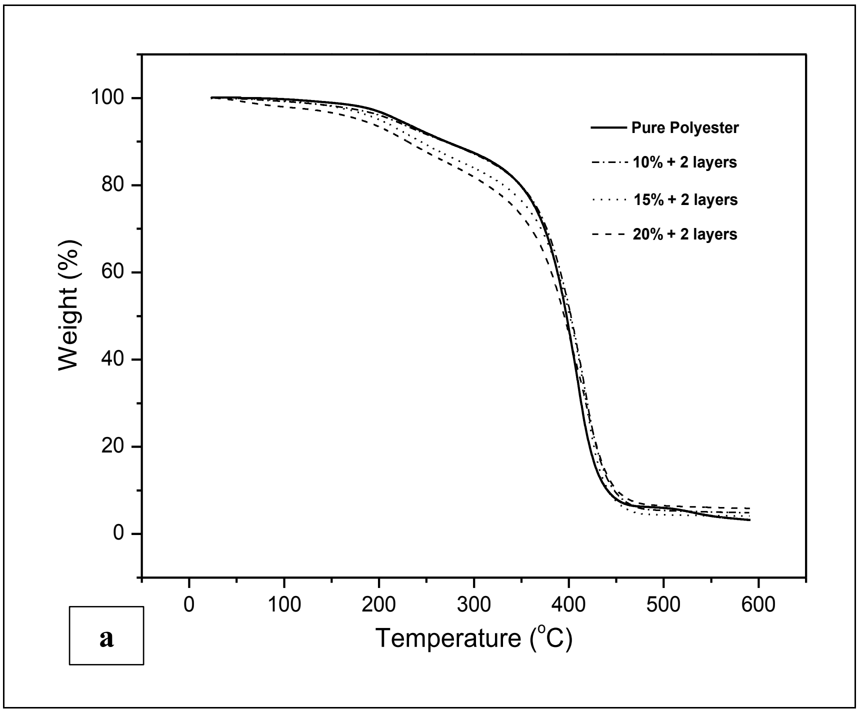
Overall experimental results and data analysis were discussed in this chapter. The details on the effect of resin curing agent (RCA) percentage and the fibre layer amount on the composites properties were studied. From this research, characterisations of composites on the mechanical and thermal properties have been done. For mechanical properties, tensile test, charpy impact test and water absorption test have been conducted while for thermal properties; TGA and DSC have been conducted. Further explanation was provided in the following sections.

4.2 Effect of Resin Curing Agent Percentage on the Composites Properties

4.2.1 Thermal Properties

4.2.1.1 Thermogravimetric Analysis (TGA)

The thermal degradation of composites with different RCA percentage has been investigated in terms of weight loss by TGA as shown in Figure 4.1.



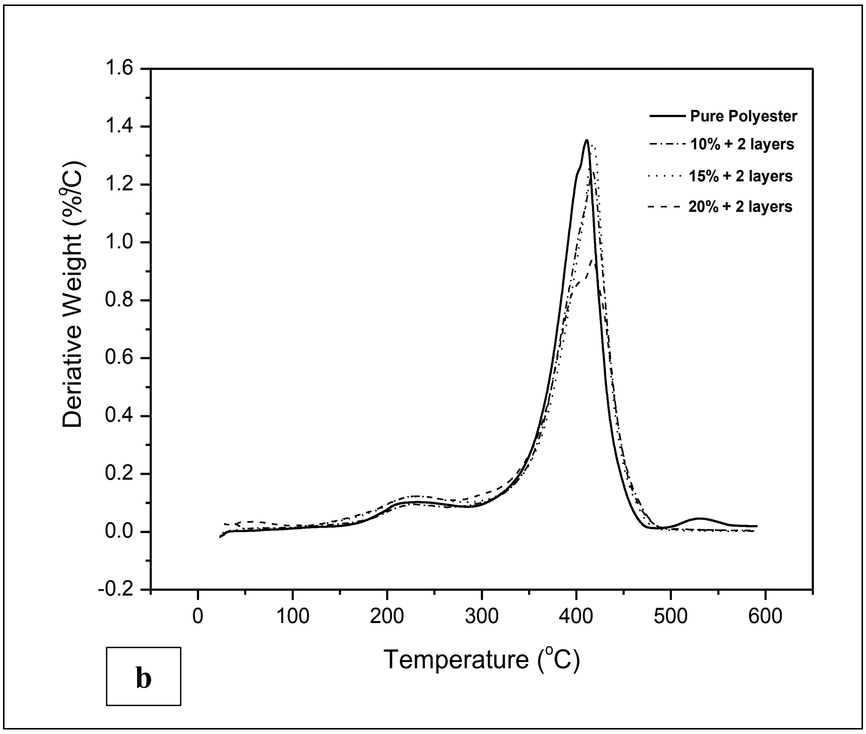


Figure 4.1: Thermogravimetric Curves of Pure Polyester and 2 Fibre Layers Composites with Various RCA Percentages (a) and the Derivative Curves of the Same Samples (b).

The range of degradation temperature (T_d) and the temperature of maximum degradation (T_{max}) of samples were tabulated in Table 4.1.

Table 4.1: The Values of T_d and T_{max} for Various Samples, Observed by Figure 4.1.

Samples	T_d Range(°C)	T_{max} (°C)
Pure Polyester	150-450	415
10% + 2 Layers	130-460	430
15% + 2 Layers	128-460	425
20% + 2 Layers	65-460	445

From the thermogravimetric curves, Figure 4.1(a), it was observed that the onset temperature of thermal degradation for pure polyester, 2 fibre layers composites with 10%, 15% and 20% RCA were 200, 180, 175 and 150°C, respectively. The derivative curves for various samples were shown in Figure 4.1(b).

It indicated the maximum rate of weight lost for pure polyester, 2 fibre layers composites with 10%, 15% and 20% RCA at 415, 430, 425 and 445^{o}C, respectively.

In the comparison of pure polyester to the composites, the degradation temperature of pure polyester was higher than composites. The presence of kenaf fibre decreases the T_d due to the nature of kenaf fibre (Qin, et al., 2011). Hence, pure polyester showed the higher thermal stability than composites.

On the other hand, by comparing the composites with different RCA percentage, the thermal stability for the 20% RCA composites was the lowest as the initial weight lost temperature (65^{o}C) was the lowest. It was followed by the 15% RCA composite (128^{o}C) and 10% RCA composite (130^{o}C). The composite which cured with higher RCA was less stable, while the composite with lower RCA percentage was more stable. This could be explained that, the composite with higher RCA percentage presented more cross linker between the chains of polymer. This promoted more interaction between the degradation processes of the 2 components (fibre and resin). In addition, the containing amount of impurities might initiate more active sites and accelerated the beginning of thermal degradation (Wielage et al., 1999). Hence, the degradation of one component might be accelerating the degradation of other component. Therefore, the TGA results confirmed the higher RCA percentage decreased the thermal stability of composites.

4.2.1.2 Differential Scanning Calorimetry Analysis (DSC Analysis)

The behaviour of pure polyester and the composites with different RCA percentage upon heating/cooling within the range of 40°C to 400°C was observed by using TA instrument software and data were tabulated in Table 4.2. Figure 4.2 shows the pure polyester properties (melt peak temperature, enthalpy of fusion and crystallinity) which obtained from TA instrument software.

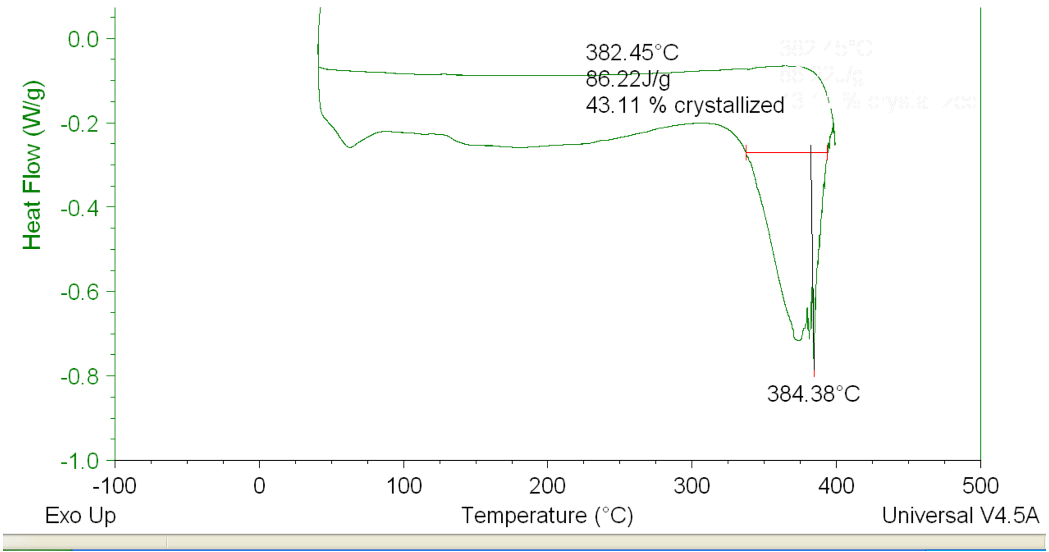


Figure 4.2: Various Thermal Properties of Pure Polyester

Table 4.2: The Values of Various Thermal Properties for Various Samples.

Samples	Melting Point (°C)	Melt Peak Temperature(°C)	Enthalpy (J/g)	Crystallinity (%)
Pure Polyester	340	384.38	86.22	43.11
10% + 2 Layers	343	385.33	87.12	43.56
15% + 2 Layers	349	387.09	88.76	44.38
20% + 2 Layers	352	387.6	85.92	42.96

The results showed that the melting points of pure polyester, 2 fibre layers composites with 10%, 15% and 20% RCA were 340, 343, 349 and 352°C, respectively while the melt peak temperatures for the same samples were 384.38, 385.33, 387.09 and 387.6^{o}C, respectively. It showed that the melting points would be increased when the RCA percentage was increased. This result suggested that, increased of the cross linker between the polymer chains could slightly increase the melting point of composites. It also showed that for the cooling cycle in Figure 4.2, there was no any heat flow peak on the cycle. This could be explained that the polyester is thermosetting resins which contain cross-link structures.

For the enthalpy of fusion of pure polyester, 2 fibre layers composites with 10%, 15% and 20% RCA, the results showed 86.22, 87.12, 88.76 and 85.92 j/g, respectively while for the crystallinity of the same samples; it showed 43.11, 43.56, 44.38 and 42.96%, respectively. The enthalpy of fusion and crystallinity of those samples are more or less the same. This could be explained that the increment of RCA percentage did not show the obvious effect on these thermal properties on composites.

4.2.2 Mechanical Properties

4.2.2.1 Tensile Test

The results of tensile strength (TS) and tensile modulus (TM) of composites with different RCA percentage were obtained and the data was tabulated in Figure 4.3 and Table 4.3.

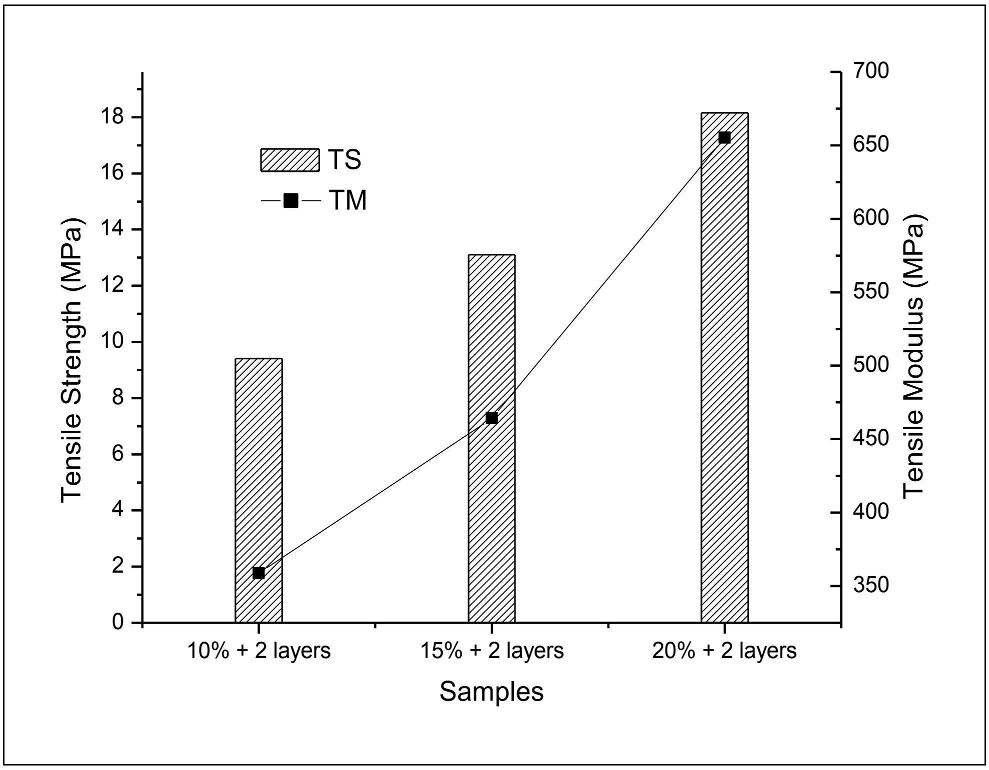


Figure 4.3: TS and TM of 2 Layers Composites with Various RCA Percentages

Table 4.3: TS and TM Data of 2 Layers Composites with Various RCA Percentages

Samples	Tensile Strength (MPa)	Tensile Modulus (MPa)
10% + 2 Layers	9.41278	358.771
15% + 2 Layers	13.1024	464.234
20% + 2 Layers	18.1614	655.089

From the results obtained, it showed that the tensile strength of 2 fibre layers composites with 10%, 15% and 20% RCA were 9.41278, 13.1024 and 18.1614 MPa while for the tensile modulus; it showed 358.771, 464.234 and 655.089 MPa. The results showed that, by increasing the RCA percentage of composites, the tensile strength and tensile modulus would be increased. It indicated that increased of the RCA percentage would increase the maximum capacity of tensile load of composite and also the elasticity of composites. The results suggested that the increment of cross linker between polymer chains could enhance the stiffness of composites. It might exhibit greater interaction between the fibre and matrix which resulting in a good dispersion in the composite.

4.2.2.2 Charpy Impact Test

The Charpy impact test results for composites with different RCA percentage were shown in Figure 4.4 and Table 4.4.

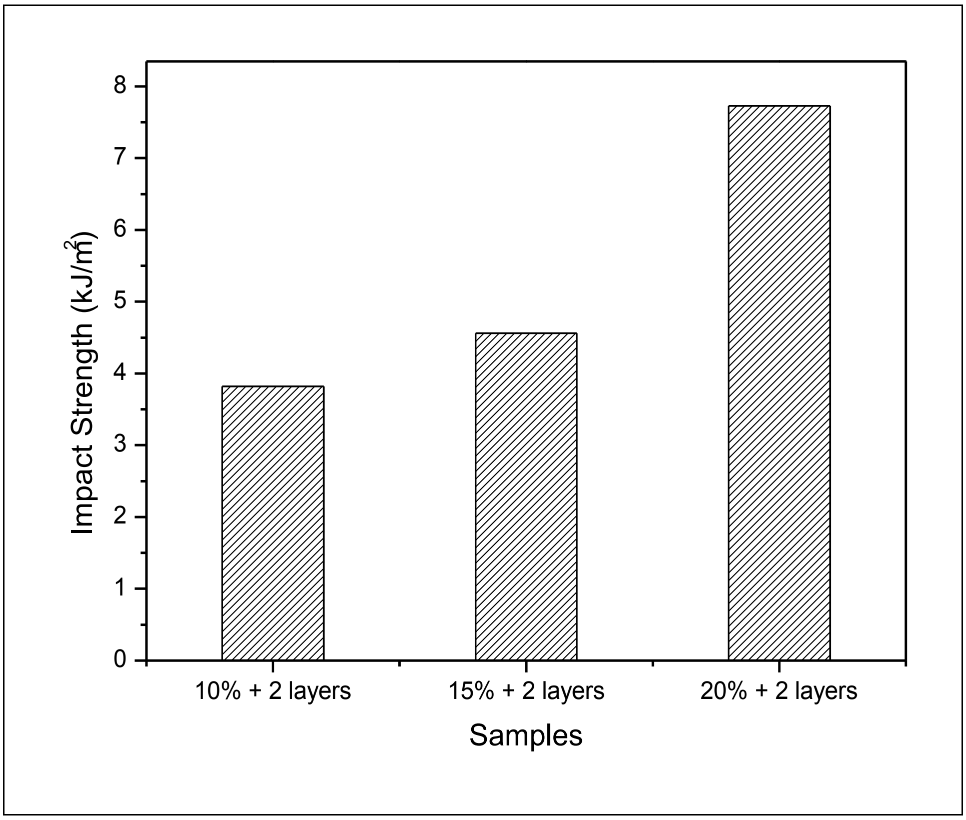


Figure 4.4: Impact Strength of 2 Layers Composites with Various RCA Percentages

Table 4.4: Impact Strength Data of 2 Layers Composites with Various RCA Percentages

Samples	Impact Strength (kJ/m^2)
10% + 2 Layers	3.82
15% + 2 Layers	4.56
20% + 2 Layers	7.73

The results showed that the impact strength of 2 fibre layers composites with 10%, 15% and 20% RCA were 3.82, 4.56 and 7.73kJ/m^2. The impact strength of composites increased with the increment of RCA percentage. It indicated that the composite energy absorption increased with RCA percentage. Generally, brittle materials absorb very low energy prior to fracture whereas tough materials absorb huge energy (Islam et al., 2012). This result suggested that the increment of cross linker between polymer chains could enhance the capability of composites in absorbing energy. Hence, the Charpy impact test results showed that the increment of RCA percentage in composites could be the cause of the improvement of the impact strength of composite materials.

4.2.2.3 Water Absorption Test

The water absorption of composites with different RCA percentage was shown in Figure 4.5.

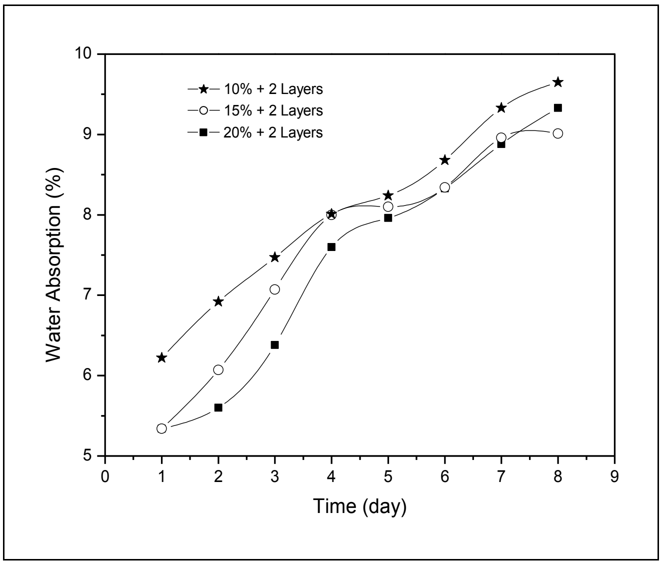


Figure 4.5: Water Absorption of 2 Layers Composites with Various RCA Percentages

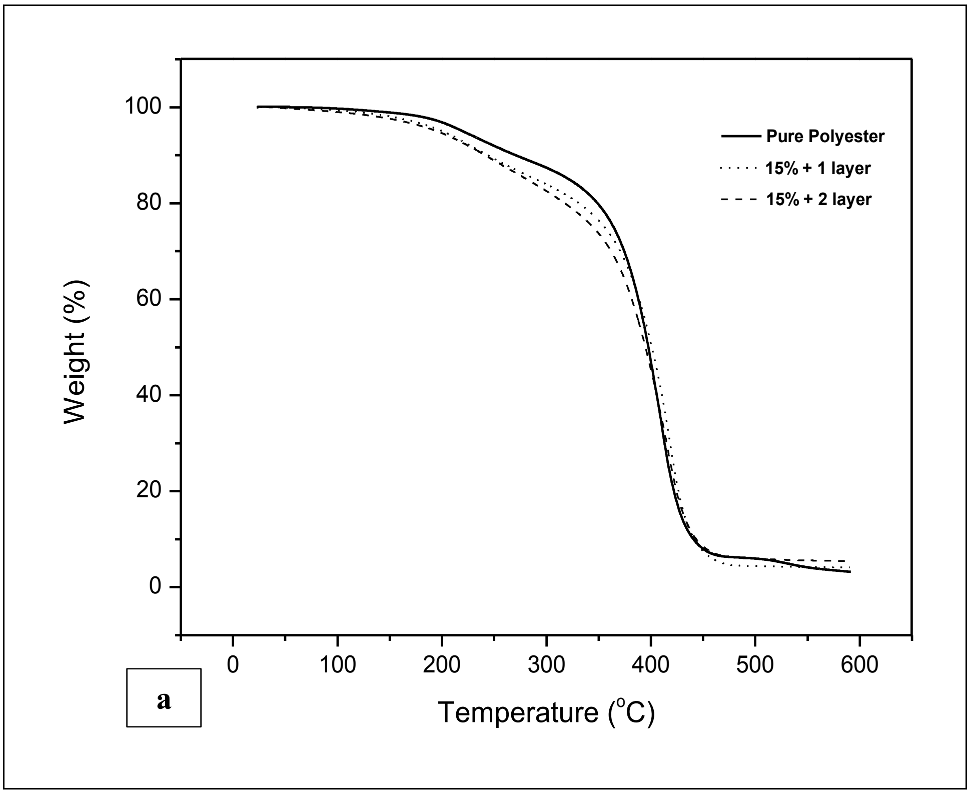
The results showed that the water absorption for the composites with same fibre layers were more or less the same regardless the difference of RCA percentages. However, it was slightly decreased when increasing the RCA percentage. This might be caused by the cross linker between the polymer chains of polyester. The higher the cross linker amount, the interfacial adhesion would be better. Better interfacial adhesion decreased the width of the interface area between the fibres and the composite which decreased the water absorption through this area into inner parts of the material (Toro et al., 2005). Dobreva et al. (2010) also reported that a strong fibre/matrix interfacial adhesion could help to diminish the water penetration, reducing the hygroscopicity and consequently which avoiding the worsening of mechanical performances of composites.

4.3 Effect of Kenaf Fibre Layers Amount on the Composites Properties

4.3.1 Thermal Properties

4.3.1.1 Thermogravimetric Analysis (TGA)

The thermal degradation of composites with different layer amount of kenaf fibre has been investigated in terms of weight loss by TGA as shown in Figure 4.6 and Figure 4.7.



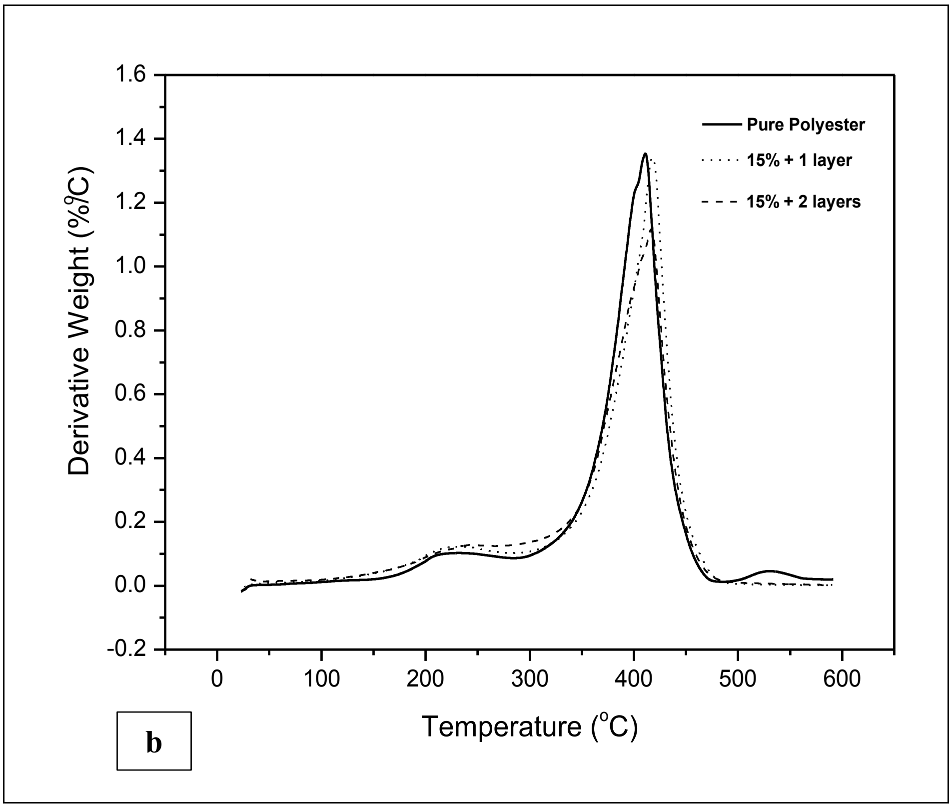
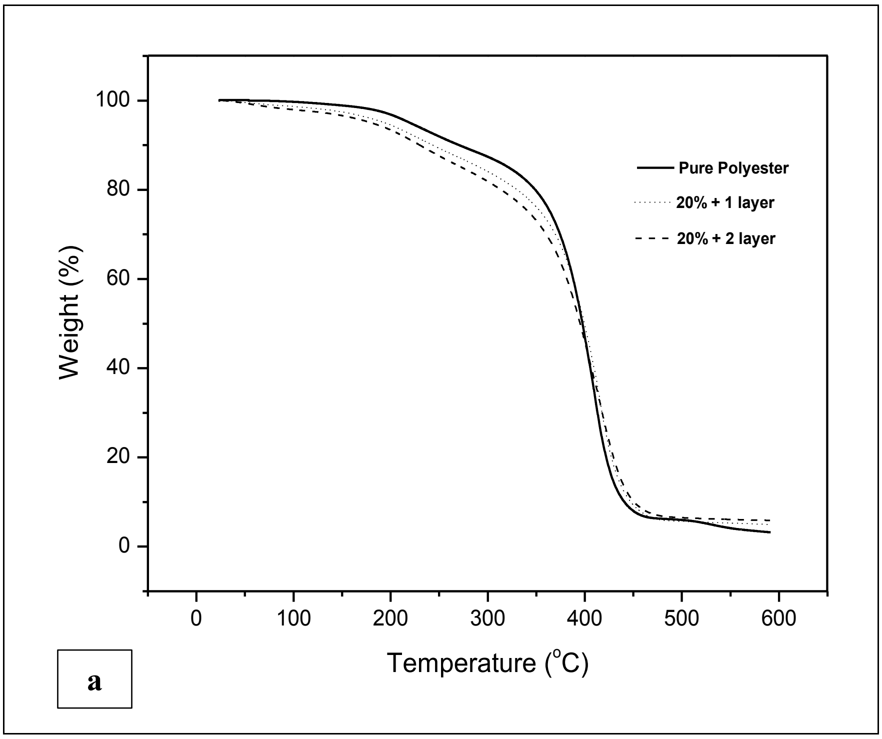


Figure 4.6: Thermogravimetric Curves of Pure Polyester and 15 % RCA Composites with Different Kenaf Fibre Layer (a), and the Derivative Curves of the Same Samples (b).



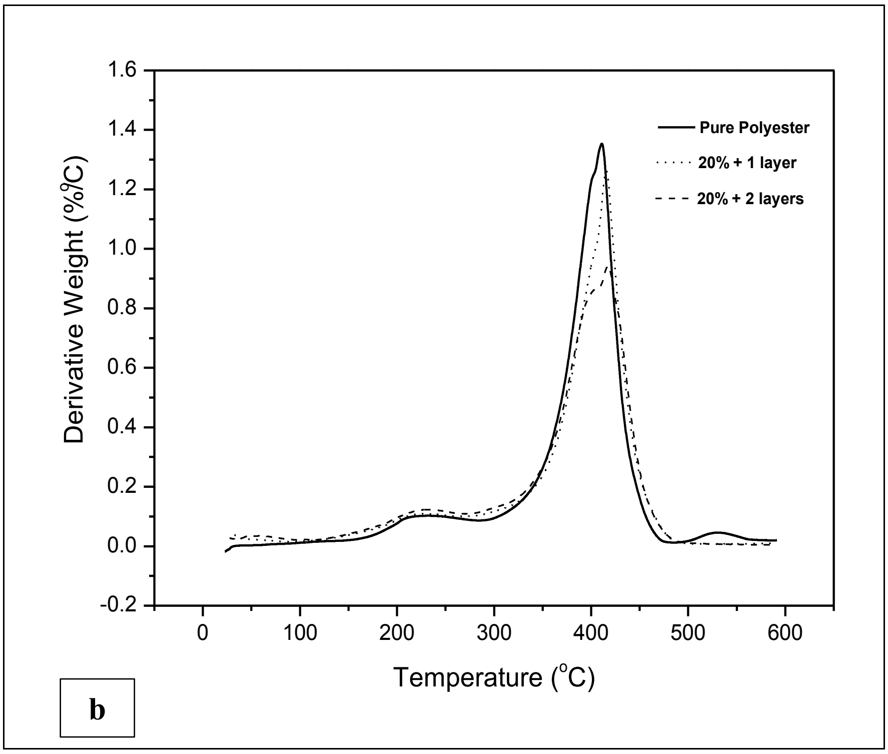


Figure 4.7: Thermogravimetric Curves of Pure Polyester and 20 % RCA Composites with Different Kenaf Fibre Layer (a), and the Derivative Curves of the Same Samples (b).

The range of degradation temperature (T_d) and the temperature of maximum degradation (T_{max}) of samples were tabulated in Table 4.5.

Table 4.5: The Values of T_d and T_{max} for Various Samples, Observed by Figure 4.6 and Figure 4.7.

Samples	T_d Range(^oC)	T_{max} (^oC)
Pure Polyester	150-450	415
15% + 1 Layer	130-460	423
15% + 2 Layers	128-460	425
20% + 1 Layer	100-470	430
20% + 2 Layers	65-460	445

From the thermogravimetric curves, Figure 4.6(a) and Figure 4.7(a), it was observed that the onset temperature of thermal degradation for pure polyester, 15% RCA with 1 and 2 layers, 20% RCA with 1 and 2 layers, were 200, 172, 175, 155 and 150^oC, respectively. The derivative curves for various samples were shown in Figure 4.6(b) and Figure 4.7(b). It indicated the maximum rate of weight lost for pure polyester, 15% RCA with 1 and 2 layers, 20% RCA with 1 and 2 layers at 415, 423, 425, 430 and 445^oC, respectively.

By comparing the composites with different amount of kenaf fibre layers, the degradation temperatures of 2 fibre layers are higher than 1 fibre layer. It also shown that the initial weight lost temperatures for 2 fibre layers (128^oC and 65^oC) were lower than 1 fibre layer (130^oC and 100^oC). Thus, it indicated that addition of fibre layers decrease the overall thermal stability of composites (Arrakhiz et al., 2012). Therefore, the TGA results confirmed the higher amount of fibre layers decreased the thermal stability of composites.

4.3.1.2 Differential Scanning Calorimetry Analysis (DSC Analysis)

The behaviour of pure polyester and the composites with different layer amount of kenaf fibre upon heating/cooling within the range of 40°C to 400°C was observed by using DSC analysis and data were tabulated in Table 4.6.

Table 4.6: The Values of Melting Points and Melt Peak Temperatures for Various Samples.

Samples	Melting Point (°C)	Melt Peak Temperature(°C)	Enthalpy (J/g)	Crystallinity (%)
Pure Polyester	340	384.38	86.22	43.11
15% + 1 Layer	347	386.91	85.36	42.68
15% + 2 Layers	349	387.09	88.76	44.38
20% + 1 Layer	350	387.47	81.42	40.71
20% + 2 Layers	352	387.6	85.92	42.96

The results showed that the melting points of pure polyester, 15% RCA with 1 and 2 layers, 20% RCA with 1 and 2 layers, were 340, 347, 349, 350 and 352°C, respectively while the melt peak temperatures for the same samples were 384.38, 386.91 387.09, 387.47 and 387.6°C, respectively. It showed that the melting points for different amount of fibre layer were more or less the same as the addition of fibre did not affect much on the melting point of composites.

For the enthalpy of fusion of pure polyester, 15% RCA with 1 and 2 layers, 20% RCA with 1 and 2 layers, the results showed 86.22, 85.36, 88.76, 81.42 and 85.92 j/g, respectively while for the crystallinity of the same samples; it showed 43.11, 42.68, 44.38, 40.71 and 42.96%, respectively. From the results, we could see that by increasing the fibre layer of composites, the crystallinity of composites also increased. This result suggested that incorporated fibres might act as nucleating agent, which ultimately increased the crystallinity of composites (Islam et al., 2012).

4.3.2 Mechanical Properties

4.3.2.1 Tensile Test

The results of tensile strength (TS) and tensile modulus (TM) of composites with different RCA percentage were obtained and tabulated in Figure 4.8 and Table 4.7.

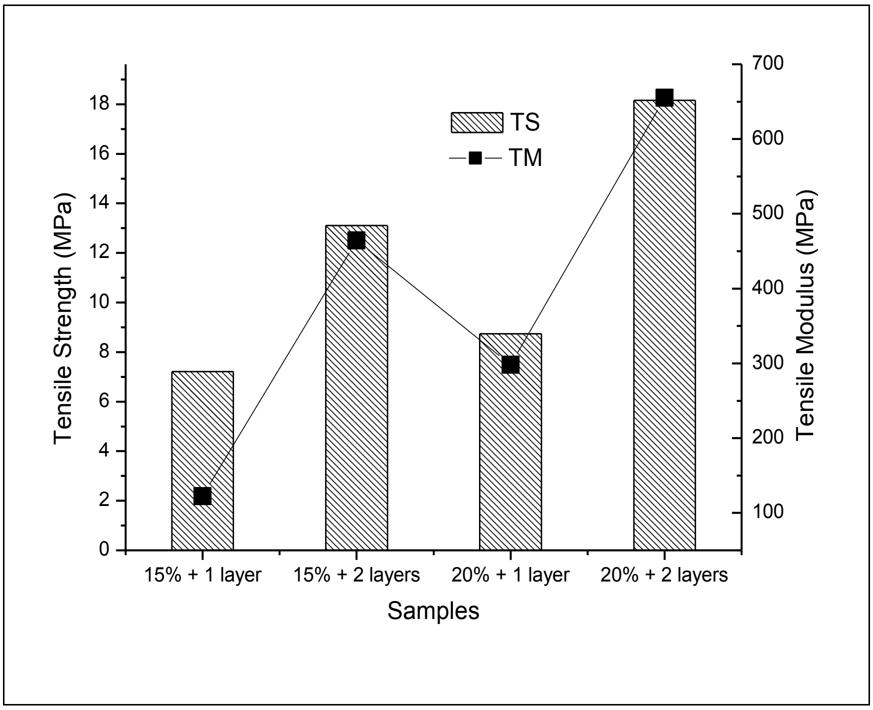


Figure 4.8: TS and TM of 15 and 20 % RCA Composites with Different Kenaf Fibre Layer

Table 4.7: TS and TM Data of 15 and 20 % RCA Composites with Different Kenaf Fibre Layer

Samples	Tensile Strength (MPa)	Tensile Modulus (MPa)
15% + 1 Layer	7.2142	122.42
15% + 2 Layers	13.1024	464.234
20% + 1 Layer	8.7364	298.172
20% + 2 Layers	18.1614	655.089

From the results obtained, it showed that the tensile strength of 2 fibre layers composites with 10%, 15% and 20% RCA were 7.2142, 13.1024 and 8.7364 and 18.1614 MPa while for the tensile modulus; it showed 122.42, 464.234, 298.172 and 655.089 MPa. The results showed that, by increasing the kenaf fibre layer of composites, the tensile strength and tensile modulus would be increased. The significant enhancement in tensile strength and modulus was obtained with the increasing of fibre layer. Arrakhiz et al., (2012) reported that fibre mainly played an interesting role to enhance the stiffness of the composites by supporting the applied load. This could be explained that, by increasing the fibre layer, the degree of obstruction increased which consequently increased the stiffness of composites (Rahman et al., 2008).

4.3.2.2 Charpy Impact Test

The Charpy impact test results for composites with different layer amount of kenaf fibre were shown in Figure 4.9 and Table 4.8.

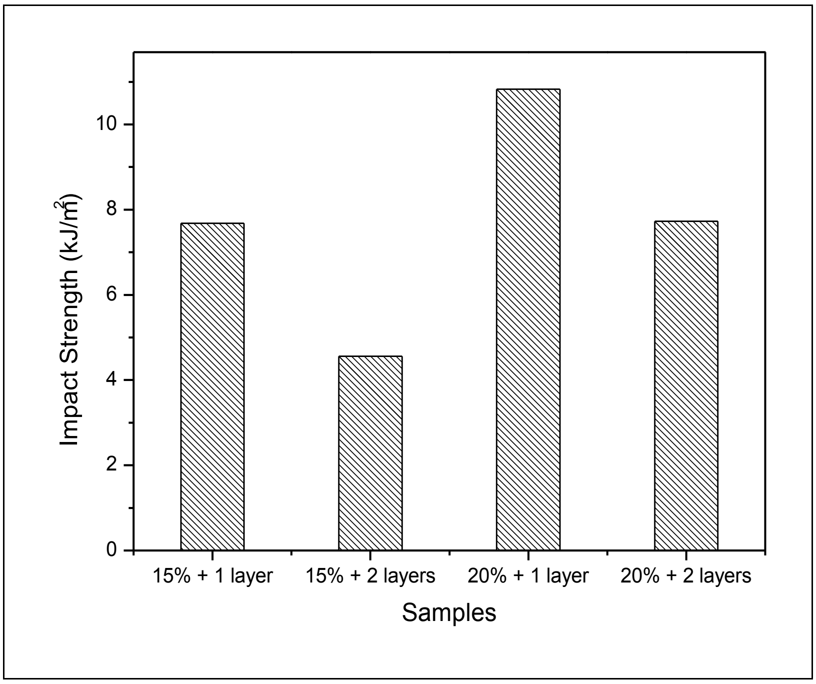


Figure 4.9: Impact Strength of 15 and 20 % RCA Composites with Different Kenaf Fibre Layer

Table 4.8: Impact Strength Data of 2 Layers Composites with Various RCA Percentages

Samples	Impact Strength (kJ/m^2)
15% + 1 Layer	7.68
15% + 2 Layers	4.56
20% + 1 Layer	10.83
20% + 2 Layers	7.73

The results showed that the impact strength of composites with 15% RCA with 1 and 2 layers, 20% RCA with 1 and 2 layers, were 7.68, 4.56, 10.83 and 7.73kJ/m^2, respectively. The impact strength of composites decreased with the increment of fibre layer. It has been reported that high fibre content increased the probability of fibre agglomeration which resulted in region of stress concentration which required less energy for crack propagation (Rahman et al., 2008). In addition, it was also reported that the impact strength decreased with fibre loading due to the weak interfacial bonding between fibre and matrix which caused micro crack to occur at the point of impact (Islam et al., 2012). Thus, the Charpy impact test results showed that the increment of fibre layers could decrease the impact strength of composites.

4.3.2.3 Water Absorption Test

The water absorption of composites with different kenaf fibre layers was show in Figure 4.10.

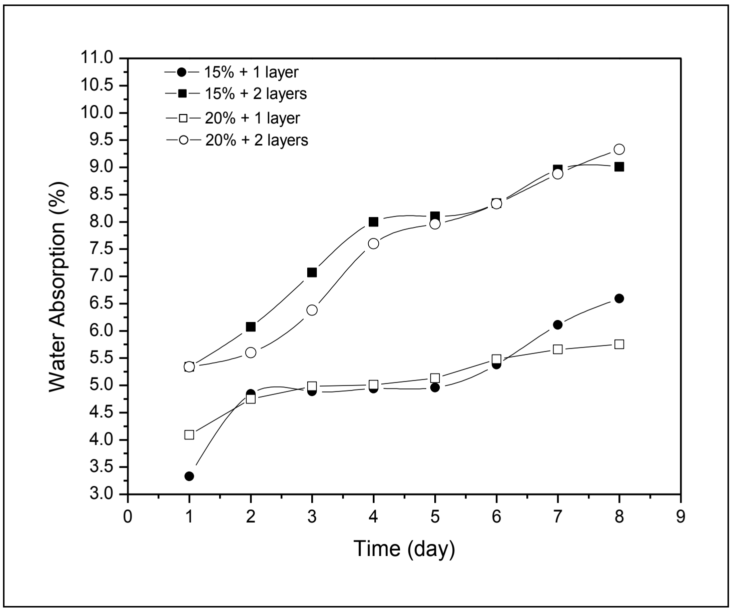


Figure 4.10: Water Absorption of 15 and 20 % RCA Composites with Different Kenaf Fibre Layer

From the result, it showed that by increasing the fibre layer, the water absorption would be obviously increased. These results were in good accordance with the published data which showed that the lignocellulosic polar fibres displayed a higher tendency to absorb water (Qin et al., 2011). Cellulose molecule in fibre contained hydroxyl group which is polar in nature and absorbs moisture from atmosphere (Rahman et al. 2008). Thus, the water absorption would be increased with fibre layer amount.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In conclusion, the mechanical and thermal properties on natural fibre reinforced composites are depending on the resin curing agent percentages and fibre layer amount. Several tests have been done to examine the mechanical and thermal properties of composites. For the mechanical properties, The KPC tensile and impact strengths were increased with the RCA percentage and kenaf fibre layer due to the increment of cross linker between polymer chains and degree of obstruction. On the other hand, the increment of fibre layer increased the water absorption of composites due to the hydrophilic properties of kenaf fibre. For thermal properties, increment of RCA percentages and fibre layers decreased the degradation temperatures of composites which decreased the thermal stability of composites. However, increment of RCA percentage and fibre layers increased the melting points of composites.

5.2 Recommendation

For this experiment, untreated kenaf fibre was used in producing composites. Instead of kenaf fibre, glass fibre can be used as different fibre properties can be evaluated. Besides, treated fibre with alkaline, coupling agent and others method can also be considered.

Besides, some others mechanical test like flexural test can be conducted so that the modulus of elasticity of composites can be calculated. For physical test, density analysis also can be conducted. Thus, the influence of the overall quality of composites can be measured.

Furthermore, optimisation on the effect of RCA percentage and fibre layer amount on composites properties should be continued. The optimum parameters to produce optimum properties of composites can be chosen to produce roofing panels by using compression moulding.

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