EFFECT OF BONDING AGENT ON THE PROPERTIES OF PINEAPPLE LEAF FIBRE STARCH COMPOSITE

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This report is submitted
in fulfillment of the requirement for the degree of
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DECLARATION

I declare that this project report entitled "Effect of Bonding Agent on the Properties of Pineapple Leaf Fibre Starch Composite" is the result of my own work except as cited in the references

Signature : ....
Name : Asyraf Kasyidi bin Mohd Shobi
Date : 24 May 2017

APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Structure & Materials).

Signature :. ....

Name of Supervisor : Dr Mohd Zulkefli bin Selamat

Date : 24 May 2017

DEDICATION

To my beloved family and fellow friends,

All your contributions, I will always keep in my heart.

Thanks a lot for the support and sacrificing.

ACKNOWLEDGEMENT

Alhamdulillah, thanks to Allah S.W.T, finally I have completed my thesis. First of all, I wish to express my honest appreciation to my supervisor, Dr Mohd Zulkefli bin Selamat for his valuable idea, advice, encouragement and for his guidance throughout this project. Without his continued support and interest, this thesis and research would not been the same as presented here.

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ABSTRACT

Nowadays, the usage of natural fiber to produce biodegradable composites has gained increasing interest due to their eco-friendly properties. Biodegradable composite can be defined as composite material or the combination between matrix and reinforcement used both materials from natural resources. Pineapple leaf fibre (PLF) is a waste product of pineapple cultivation. This fibre has potential as reinforcement fillers in thermosets, thermoplastics, elastomers or biodegradable matrix and exhibit excellent mechanical properties. One of biodegradable matrix is the mixture of starch (SH) and glycerol (GLY). The ratio of GLY has been vary from 10 up to 30% from total weight of mixture SH/GLY. The tensile strength of this mixture had been determine and the mixture of 70:30 (SH:GLY) shown the highest value of 0.734 MPa. Therefore, this matrix has been selected to produce composite of PLF/(SH/GLY) and the mechanical properties of this composite have been studied. The objectives in this study were to investigate the effect of fibre loading, addition of GLY as bonding agent and fibre treatment in PLF/(SH/GLY) composite by comparing the mechanical properties. The composite compositions are 50PLF/50(SH/GLY), 60PLF/40(SH/GLY) and 70PLF/30(SH/GLY) and has been fabricated through hot compression moulding in order to form composite sheet. The PLF fiber was treated used Sodium Hydroxide (NaOH) as treatment agent. The mechanical properties of the composite materials were analyzed through the tensile test (ASTM D 3039/D 3039 M-00), hardness test, density measurement and macrostructure analysis. All of the tests basically show improvement. Tensile strength shows increment of the value of 27.686MPa with the increasing of fibre loading. The same pattern also happen to the hardness and density measurement shows increment value of 55.0 in shore-D and 1.332g/cm3 of the result with the more composition of fibre in the composites. Therefore it can be concluded, the results showed 70PLF/30(SH/GLY) composite filled with treated fibre had higher in tensile strength, hardness and density.

ABSTRAK

Pada masa kini, penggunaan serat semula jadi untuk menghasilkan komposit biodegradable telah mendapat minat yang semakin meningkat kerana sifat mesra alam mereka. Komposit biodegradable boleh ditakrifkan sebagai bahan komposit atau kombinasi di antara matriks dan tetulang digunakan kedua-dua bahan-bahan dari sumber asli. Serat daun nanas (PLF) adalah produk sisa penanaman nanas. Serat ini mempunyai potensi sebagai pengisi penguat dalam termoset, termoplastik, elastomer atau matriks biodegradable dan mempamerkan sifat-sifat mekanikal yang baik. Salah satu matriks biodegradable adalah campuran kanji (SH) dan gliserol (GLY). Nisbah GLY telah berbeza-beza dari 10 sehingga 30% daripada jumlah berat campuran SH / GLY. Kekuatan tegangan campuran ini telah menentukan dan campuran 70:30(SH/GLY) yang dipaparkan nilai tertinggi 0,734 MPa. Oleh itu, matriks ini telah dipilih untuk menghasilkan komposit PLF/(SH/GLY) dan sifat-sifat mekanik komposit ini telah dikaji. Objektif dalam kajian ini adalah untuk mengkaji kesan loading serat, penambahan GLY sebagai ejen ikatan dan rawatan serat dalam PLF / (SH/GLY) komposit dengan membandingkan sifat-sifat mekanikal. Komposisi komposit adalah 50PLF/50(SH/GLY), 60PLF/40(SH/GLY) dan 70PLF/30(SH/GLY) dan telah direka melalui pengacuan mampatan panas untuk membentuk lembaran komposit. The PLF serat telah dirawat digunakan Sodium Hidroksida (NaOH) sebagai agen rawatan. Sifat-sifat mekanikal bahan komposit dianalisis melalui ujian tegangan (ASTM D 3039/D 3039 M-00), ujian kekerasan, pengukuran ketumpatan dan analisis Macrostructure. Semua ujian yang pada dasarnya menunjukkan peningkatan. kekuatan tegangan menunjukkan kenaikan daripada nilai 27.686MPa dengan meningkatkan serat loading. Corak yang sama juga berlaku kepada kekerasan dan pengukuran ketumpatan menunjukkan nilai peningkatan sebanyak 55.0 di shore-D dan 1.332g/cm^3 hasilnya dengan komposisi lebih serat dalam komposit. Oleh itu ia boleh membuat kesimpulan, keputusan menunjukkan 70PLF/30(SH/GLY) komposit penuh dengan rawatan serat mempunyai lebih tinggi dalam kekuatan tegangan, kekerasan dan kepadatan.

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LIST OF ABBREVIATIONS

PLF	Pineapple Leaf Fibre
SH	Starch
PLF/SH	Pineapple Leaf Fibre/Starch
SH/GLY	Starch/Glycerol
PLA	Polylactic Acid
NaOH	Sodium Hydroxide
HCl	Hydrochloric Acid
ASTM	American Standard Testing Method
GLY	Glycerol
PP	Polypropylene

LIST OF SYMBOLS

°C = Degree Celsius
% = Percent
MPa = MegaPascal
GPa = GigaPascal
kN = kiloNewton
mm = millimetre
g = gram
cm^3 = cubic centimetre

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Generally, bonding agents known as naturals, compounded or synthetic materials used to enhance the joining of individual members of a structure without employing mechanical jointing equipment or fasteners. These products are often used in various types of applications such as building construction, automotive, airbus and so on. For example in the application of building construction industry, the main types of bonding agents used are latex emulsions and epoxies. Furthermore, these two have their own mechanical properties and chemical properties as suitable bonding agents to be used for bonding the materials for construction process (Mailvaganam, n.d.). In biodegradable composite, regarding from that, those bonding agents are the main important elements, in order to analyze the strength of the materials involved in a certain project. Therefore, this project is focusing on the effect of bonding agents towards the composition ratio of two materials which is Pineapple Leaf Fibre (PLF) as reinforcement material and Starch (SH) as matrix material, in order to determine their mechanical properties. Bonding agent is known as additive, which is the material that can react with the composition of reinforcement and matrix, in order to increase the mechanical properties of the composites. Their presence towards the composition can affect the result on the mechanical testing whether it will show increasing or decreasing value of the composites sample. Bonding agent such as polylactic acid (PLA) and glycerol, GLY) can be available as their functions as plasticizers that can react with thermoplastic material such as starch and polypropylene (PP).

Reinforcement in a simplest word can be described as a material that will strengthen the structure. The reinforcement is made up of material or fibre. Nowadays, the natural fibres are increasingly used as reinforcement in polymer composite (Campbell, 2010). The reinforcement also can come from natural or made by human. There are other several types of reinforcement in industries such as synthetics fibre and natural fibre. The example of fibres that normally used in industries is glass fibre and carbon fibre. Glass fibre is well known as the combination made between steel sheet and zinc diecasting. For example in automotive industry, carbon fibre dominates as the main reinforcement that has low-cost composites. Furthermore, they has already reached its impressive performance level in automotive manufacturing process (Haruna et al., 2014).

There are a few examples of natural fibres such as sisal fibre, bamboo fibre, pineapple leaf fibre, hemp fibre and jute fibre. Nowadays, natural fibres such as sisal and jute fibre composite materials are being alternative natural fibres that can replace the glass and carbon fibers since the cost of glass fibre is quite expensive in market. Furthermore, the hardness and flexibility is good and compare to the glass fibre. The performance of natural fibre is improved remarkably due to the fact that the field of application is improved day by day especially in automotive industries.

Nowadays, natural fiber composites have gained increasing interest due to their eco-friendly properties. From the previous research that has been done by a researcher on natural fibre properties, the natural fibre has obtained positive results. This is due to the natural fibers such as jute, sisal, silk and coir are inexpensive, abundant and renewable, lightweight, with low density, high toughness, and biodegradable (Asim et al., 2015). Natural fibres such as jute have the potential to be used as a replacement for traditional reinforcement materials in composites for applications which require high strength to weight ratio and further weight reduction (Ashik & Sharma, 2015). Natural fibres are all kind of fibres that come from natural resources such as plant. The other word that can describe the natural fibres is a natural availability. The natural fibres that extract from the plants are actually obtained from the certain parts of the plant such as leaf. Therefore, the strength of natural fibre will increase if it undergoes chemical treatment.

1.2 PROBLEM STATEMENT

Natural fibre composites are widely used in a few industries includes an automotive industry especially as an interior part. Currently in industries, normally for the type of natural fibre material used is synthetic fibre. Thus, natural fibres like sisal, banana, jute, oil palm, kenaf and coir has been used as reinforcement in thermoplastic composite industries for applications in consumer goods, furniture, low cost housing and civil structures (Munirah, Abdul Razak, & Hassan, 2007). Since the cost is much more affordable and it is a natural availability compared to the cost of glass fibres and carbon fibres, the industries try to harness the advantages of natural fibre composite. Nowadays, the automotive field tries to diversify the natural fibre in manufacture of a car bumper from kenaf while a car seat from coconut fibre. Furthermore, most of natural fibres are renewable, cheap and recyclable. Therefore in this project, the pineapple leaf fibre (PLF) is used as a reinforce material and starch (SH) as a matrix materials to produce PLF/SH composite. Thus the composite is biodegradable composite, which is consists of those two materials in order to keep strengthening the mechanical properties. Many advantages are associated with the use of biodegradable composite, including low cost, abundance, low density, high specific properties and lack of residues upon incineration (Sahari & Sapuan, 2011). Besides, it is also a solution to maintain the sustainabale development of economical and ecological technology.

The current mechanical properties of PLF/SH composite used is might lower based on their analysis in mechanical testing. Hence, one of the alternative solution to increase the mechanical properties of PLF/SH is through added the bonding agent (1 up to 10 % from the total weight of the composite). The presence of bonding agent is to improve the interface between the matrix material and reinforcement. One of the materials can be used as bonding agent is glycerol. GLY can be mixed with SH before this mixture can be used as matrix in

PLF/(SH/GLY) composition. In order to determine the effectiveness of bonding agent several mechanical tests will be carry out such as tensile test, hardness and flexural test. Therefore, the mechanical properties of PLF/SH composite will be performed in order to investigate their actual mechanical properties. On the other hand, composite also have good potential to be used as an interior part of automotive but its potential has not been explored yet.

1.3 OBJECTIVES

There are two goals which are listed to be achieved on this project:

1. To determine the effect of GLY as binder loading on the physical and mechanical properties of SH/GLY composition.
2. To study the effect of PLF loading on the properties of PLF/(SH/GLY) composite.

1.4 SCOPE OF PROJECT

This research studies the effects of bonding agent loading on the mechanical properties of PLF/SH composite which will be carrying out. The various ratio of bonding on SH/GLY composition are being selected and determine the physical and mechanical properties. After that the composite PLF/(SH/GLY) will be fabricated through hot compression moulding with the ratio of composition in the composite is fixed at, 70:30, 60:40: 50:50. An alkaline treatment will be conducted to extract thin PLF bundles and enhance the PLF properties before the formation process of PLF/SH composite by using hot pressing machine. The mechanical properties of PLF/SH composite is need to be determined by using tensile test, hardness test, density measurement and macrostructure analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Natural fibres composites are widely used in industry in order to produce a new product since it is an environmental friendly. Nowadays, the industrial automotive is trying to use the natural fibres composites especially in the making of interior part as shown in Figure 2.1 below. This is highlighting on their advantages which are low cost and weight, also can prevent from damage towards processing equipment and great in mechanical properties (Sanjay et al., 2016). Natural fibres also can be defined as substances that come from agricultural residues. Thus, they can be used as the reinforcement with biodegradable polymer composites as a development in order to obtain useful products in industries.

Figure 2.1: Application of natural fibres in automotive industry (Farsi, 2000)

The combination of the natural fibres reinforced with biodegradable polymer composite also can provide an alternative way in order to solve the problems that associated with agriculture crop residues. All of thems are being produced everyday and they are being obtained in a large amount of quantity. Therefore, the reinforcement of natural fibres and biodegradable composite can prevent from negative effect on the environment due to the air pollution of the residues.

2.1.1 Biodegradable Composites

Biodegradable composite can be defined as composite material or the combination between matrix and reinforcement of natural fibres. These kinds of materials have their own function which keep strengthening the properties of the matrix that was used, but always providing biodegradable compatibility. It is also known as composite material made from natural fibre and polymer matrix derived from petroleum such as polylactic acid (PLA), cellulose and thermoplastic starch (Sahari & Sapuan, 2011). Thus, the matrix part of the biodegradable composite can be classified into two parts which are fully degradable and partly degradable as shown in Figure 2.2 below:

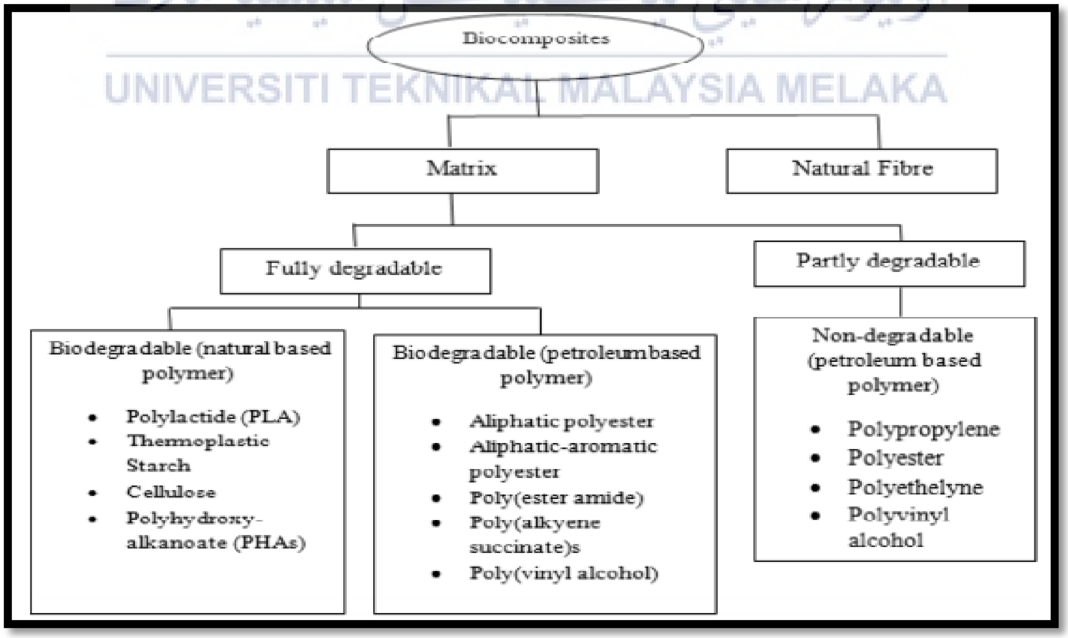


Figure 2.2: Biodegradable composite (Sahari & Sapuan, 2011)

For more specific, the combination of the composite material is being used in one of the world's major palm oil producer in Malaysia, where the natural fibre of oil palm has been successfully compounded with thermoplastics such as PLA in order to produce biodegradable composites. The forming biocomposite which is lignocellulose fibre is a renewable material and also can increased the surface roughness of the fibre by the removal of hemicellulose and surface impurities (Schaschke & Audic, 2014).

2.2 MATRIX (BINDER)

There are many types of materials used as matrix in composites. Therefore, in order to combine the matrix with the fibre, it must be in the suitable type of the matrix. This is because the combination of the fibre with the appropriate matrix will form a very good strength property. A good matrix has many advantages to the reinforcement such as can prevent the natural fibre of abrasion and defects in surface, also acting as a binder to the fibre used in composition (Verma et al., 2012).

The types of matrix that normally used in composites are metal matrix and polymer matrix. However, these two matrices have their own characteristics that could affect the performance of the composition formed. For more specific, metal matrix has good strength at higher temperatures, but high in density that can cause lower specific mechanical properties if compared with polymer matrix (Verma et al., 2012). However, the matrix used in this project is thermoplastic starch which is included in polymer matrix. The matrix will support load among the reinforcements since the reinforcements are discontinuity.

2.2.1 Starch

Starch is classified in thermoplastic type, with also included as green composite based on their compositions, preparations and properties. Because of that, the characteristics of the starch have gained many interests due to its properties as the material of environmental friendly and biodegradable renewable sources. However, the research shows that the application of starch can be further expanded, and can be developed more in order for wider usage. The starch brings many advantages especially in composites. This is because starch is naturally form in much quantities, can be renew and cheap in market (Wattanakornsiri & Tongnunui, 2014).

Before forming into the thermoplastic polymers, there are various sources of starch that has been used such as corn starch, tapioca starch, rice starch and wheat starch, which need to be bonded with addition of plasticizers in high temperature and shearing force. The purpose of this converting process is to obtain the lower friction between the molecules of the starch. The example of plasticizers that usually used is water and glycerol (Wattanakornsiri & Tongnunui, 2014).

In the chemical properties of starch, the starch contains two main elements which are amylose and amylopectin as shown in the presence of chemical structure in Figure 2.3 and Figure 2.4. These kind of elements can be hydrolyzed the compound into glucose. Initially, the starch is in crystalline form. The starch is normally need to be gelatinized which the intermolecular bonds molecules of starch is break by the water and heat, that make the hydrogen bonding sites is reduced and irreversibly dissolves the starch granules in water (Wattanakornsiri & Tongnunui, 2014).

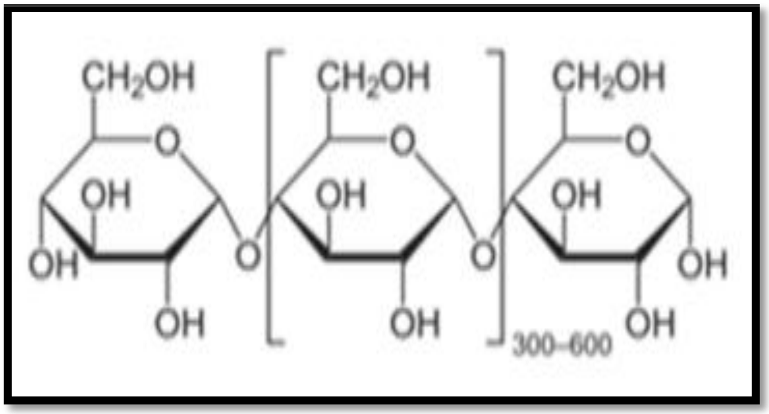


Figure 2.3: Chemical structure of amylose. (Wattanakornsiri & Tongnunui, 2014)

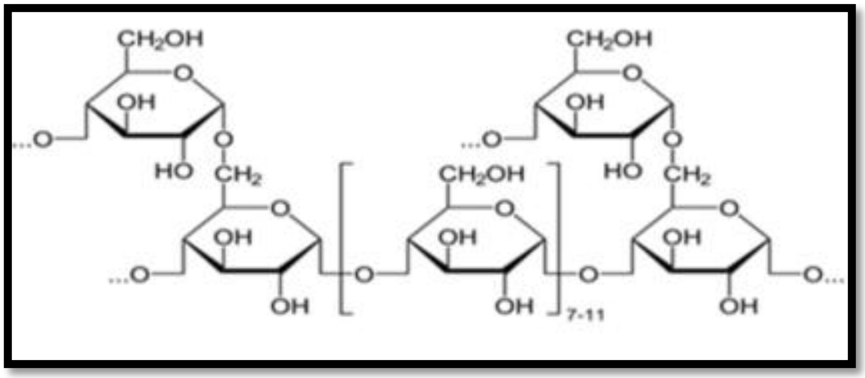


Figure 2.4: Chemical structure of amylopectin. (Wattanakornsiri & Tongnunui, 2014)

There is history about the usage of starch which it has been chosen as one of the choice in the literature in 1920s. Since the advantages of the starch have been discovered, the research towards the characteristics of the starch has shown that there are also disadvantages existed. Therefore, all of the problems can be controlled by strengthening their mechanical properties. Besides, all the chemical processes such as the addition of plasticizers or the technology that has been discovered can be used to prevent the matters that can affect the properties. The thermoplastic starch also can be considered as a new class of inexpensive and green polymers which can be returned back to its natural state without any pollution after it has been used. (Nafchi et al., 2013)

2.2.2 Epoxy

Epoxy is classified as matrix or binder. It usually used as thermoset plastic in polymer matrix composites. It has quite low shrinkage and great properties such as good chemical and environmental resistance, good adhesion to other material, and good insulating properties. Epoxy is manufactured by the combination of bisphenol and epichlorohydrin (Adalberto et al., 2014).

Besides, the epoxy resin also has its own ability which is it can be bonded or reacted with various type of substrates. The epoxy can be applied as a curing agent in cure process. It always referred as hardeners, activators and also catalyst. Normally, especially during the cured process occur, the element of amine will be used as a curing agents. This is because amine which is containing hydrogen will react with the nitrogen compound and it will make it easier to open the epoxy ring. The epoxy ring will form a covalent bonding (Singla & Chawla, 2010).

2.3 BONDING AGENT

In composites, the appearance of bonding agent is none other than to increase the mechanical properties of the composition between the particular natural fibres and matrix involved. For more specific, the properties of bamboo fibre with the addition of bonding agent can increase their properties in the aspect of tensile strength, tensile modulus, elongation at break and hardness (Ismail, 2002).

Based on the previous research, the study on the bamboo fibre composite with the addition of bonding agent has been conducted. The bonding agents used in this research are phenol formaldehyde and hexamethylenetetramine, and have obtaining the greater result in the concept of maximum and minimum elastic torque of the composition. This is strengthen the fact that the bonding agent has improved the properties of the combination between the bamboo fibre and the matrix (Rabu & Wirjosentono, 2001).

Beside that, there are some researches also have been done towards the application of advanced glycerol combine with adhesive monomer components to become as one composite resin. In order to determine its mechanical properties, the microtensile bond strength has been conducted and the presence of glycerol has shown its specification based on the result to be one of the suitable bonding agent (Dickens & Hoon, 2005).

2.4 REINFORCEMENT

Based on the intellectuals such as researchers, scientists and engineers, the utilization of natural fibres have been widely used as a composite element especially for the reinforcement in polymer. This is due to their characteristics which are low cost, fairly good mechanical properties, high specific strength, non abrasive, eco-friendly and biodegradable. Besides, the natural fibre also has been determined as alternative way in order for the replacement of conventional fibre like carbon and glass (Sanjay et al., 2016).

2.4.1 Natural Fibre

Nowadays, the application of natural fibres is quite popular and widely used in several industries in Malaysia especially in automotive industry. The research on the natural fibres has been done since the early 1900's and that means it has been used around 1000 years as reinforce material. The importance of natural fibres can be highlighted in car manufacturing sector which it concentrates on interior part and exterior part ("Natural Fiber Composites : A Review," 2010)

There are various types of natural fibres as reinforcement with the polymer composite such as jute fibre, sisal fibre, pineapple leaf fibre, bamboo fibre and banana fibre. Natural fibres also can be included as the material that comes from natural resources or in simplest word it comes from natural availability. Since it is natural availability, the price of natural fibres is cheap and easy to get, and also it is classified as renewable resources (Dhal & Mishra, 2013).

Generally, the natural fibres are lignocellulosic in nature. Hence, the plants such as flax, cotton, hemp, jute, sisal, kenaf, pineapple as well as wood are more tend to be applied as reinforcement of composites. Since the advantages of natural fibres which are rapidly growing and partially recyclable, they become an attraction in industrial applications and fundamental research. The Figure 2.5 below shows the classification of the natural fibres:

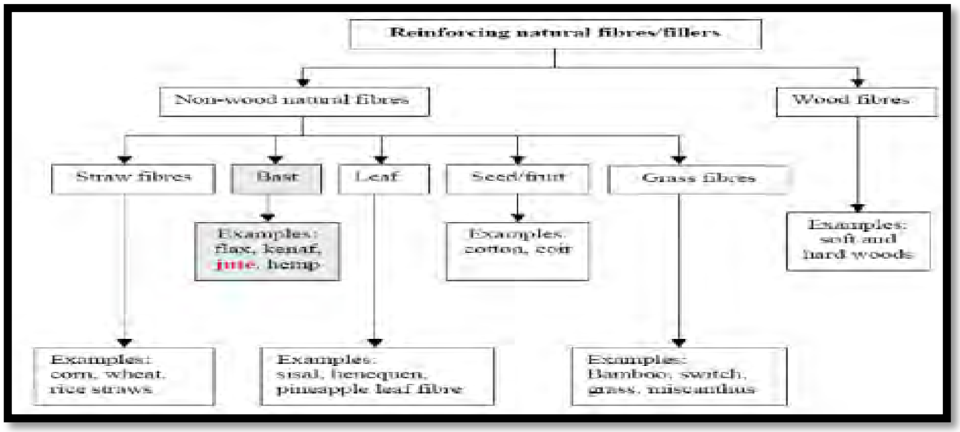


Figure 2.5: Classification of natural fibres as reinforcement.(Submitted et al., 2009)

Among all the natural fibres, the pineapple leaf fibre is one of the best fibres that usually used as reinforcement due to its greater mechanical properties. This is because pineapple leaf fibre has higher in tensile strength and contains high cellulose. In pineapple industry, basically they are only needed the pineapple fruit and leaving the leaf. Thus, the pineapple leaf will generate high volume of waste and they will be burned. Hence, it will cause pollution to environment and clearly wasting one of the best potential sources of natural fibres (Jaramillo et al., 2016).

There have been a few studies have been done by researchers. These researches have been done including with the study about pineapple leaf fibre. The fibres have obtained great results such as higher in tensile, flexural, creep in resistance and compactness. This is because the pineapple leaf fibre can be increased its properties by having chemical treatment with sodium hydroxide (NaOH) solution, and also remove the cellulose content at the surface of the pineapple leaf fibre as shown in Figure 2.6 below (Asim et al., 2015). The pineapple leaf also cannot be obtained in similar length because the leaves at the plant do not have fixed in length, as shown in Figure 2.7:

Figure 2.6: Pineapple Leaf Fibre. (Asim et al., 2015)

Figure 2.7: Pineapple Leaf. (Asim et al., 2015)

2.5 FIBRE LENGTH

According to the previous research, the pineapple leaf size that obtained from the plant is not same since there are huge and small in size. Therefore, the pineapple leaf obtained from the plant will undergo extraction process by using machine that changes the leaf into the fibre. Then the fibre will be extracted manually by hand using kitchen utensils like spoon and knife. Based on the research, the size of leaf can give effective reinforcement when it is combine with the matrix (Munirah et al., 2007).

2.6 FIBRE EXTRACTION METHOD

According to the previous studies, the PLF were extracted by two methods which are mechanical method and chemical method. Between these two methods, the chemical method is more effective and normally used in the process. This method is including the technique of immersion, which is used the step of soaking the leaves in certain times. Thus, the next process is mechanical method, which by whittling the leaves until the skin of the leaves is removed. This kind of procedure is to get rid of the cellulose content at the fibres surface. The fibres then dried by using the oven (Adam & Yahya, 2016).

2.6.1 Mechanical Extraction

The mechanical extraction is one of the methods that need to be applied to the pineapple leaf fibre. The pineapple leaf fibre has obtained in different size, which some of them are small and some are huge. The mechanical extraction brings the meaning of manually extraction by using kitchen utensils such as knife, spoon or plates. In the other words, this method is using the rebonding technique, which means it is to remove the unwanted layer at the natural fibre (Adam & Yahya, 2016) .

2.6.2 Chemical Treatment

Based on the research, the chemical treatment has the function which to increase the strength of the fibres. Chemical treatment or alkali treatment will be undergoing before the mechanical extraction were done. The solution of sodium hydroxide (NaOH) is the most common solution used as a medium to clean the natural fibre. The chemical treatment will improved and give greater result in mechanical properties. Based on the research done, the pineapple leaves is determined to be immersed in 5% of NaOH solution in about 1 hour as shown in Figure 2.8 below. After that, the natural fibre sthat have been immersed in NaOH will be neutralized in a 1% solution of hydrochloric acid (HCl). Then, the natural fibres will be put in the oven for drying method in 65°C to 70°C in the period of 3 hours (Engg & Engg, 2013).

.Figure 2.8: Sodium Hydroxide. (www.keywordsking.com)

2.7 TESTING

For the testing, the American Standard Testing Methods (ASTM) is used as the mechanical testing method for the guidance standard for the pineapple leaf fibre testing. Accroding to the previous study, there a few tests that have been done such as tensile test, hardness test, flexural test, density test and microstructure analysis.

2.7.1 Tensile test

Tensile test is used to measured the ability of the material, and in this case the ability of composite between the pineapple leaf fibre and starch will be determined by withstand forced that tend to pull and extent the material before it breaks. In the research, the tensile strength and Young’s modulus are the parameters that have to be determined after the test have done. The standard used for the tensile test is ASTM D 3039/D 3039M-00) (A N Kasim et al., 2015).

2.7.2 Hardness Test

The composition of PLF/SH also need to be determined based on their hardness. Therefore, in order to measure its hardness, the hardness test needs to be conducted toward the samples of PLF/SH. The equipment used for this hardness test is Analogue Shore Scale "D" Durometer according to the standard ASTM D1957. This is based on the hardness of the samples that were measured by the curvature occurred on samples and also the reading shown by the dial indicator (Selamat, Razi, Kasim, Dharmalingam, & Putra, 2016).

2.7.3 Density Measurement

In the research, the specific values of density which the lowest and the highest can be obtained by density measurement of the composition of PLF/SH. The density values can be measured by using Electronic Densimeter MD-300S. From the result that will be obtained, the relationship between the fibre loading and the density can be determined (Selamat et al., 2016).

2.7.4 Macrostructure Analysis

After the result from the tensile and hardness test have been obtained, the macrostrucutre analysis of PLF/SH need to be analyzed by using Dino Lite Digital Microscope. All the image appeared will be compared based on the parameters in order to choose for the best samples (Selamat et al., 2016).

CHAPTER 3

METHODOLOGY

3.1 EXPERIMENTAL OVERVIEW

Methodology is a process of the implementation of project. It is very important to describe each step to accomplish and many items to deal with. The process of the implementation of the project is shown in the flowchart in Figure 3.1. The actions that need to be carried out to achieve the objectives in this project are listed at the objectives part above. The first step is gaining all the informations about the method and process need to be gathered in order gain understanding about the project. This step highlights about the understanding through about the whole element of project, which including the finding of informations from sources such as journals, online articles, or any materials regarding the review of the project. After that, it followed by selection of material, and then proceeds with fabrication method and binder preparation. The suitable sample which is the type of PLF needs to be identified and after the selection has been done, the compression process is continued until it was repeated for unsuitable sample. Then, cutting process and testing is the last steps that need to be done for this project.

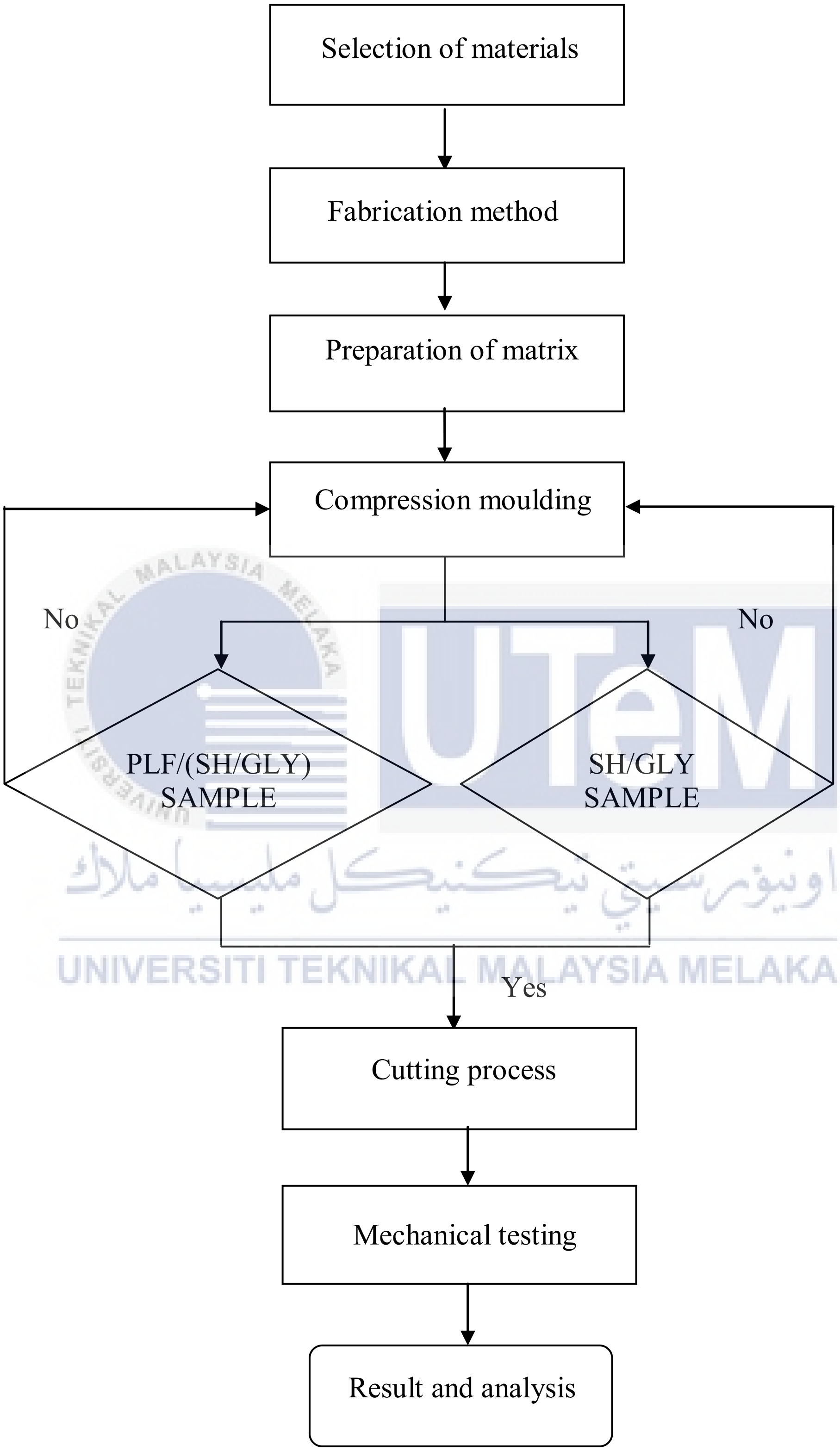


Figure 3.1: Flowchart of the methodology

3.2 SELECTION OF RAW MATERIALS

The material selection is one of the important methods which need to be alerted, due to the project that relates with the concept of composite. This is belongs to the combination of the natural fibre and the binder along with the additive of bonding agent into the composition. Generally, the selection of the material must according to a few factors such as the pineapple leaf fibre which has the strength of the plants fibre and high in cellulose content and suitable for the performance as reinforcement in polymer composites ("Effect of Pineapple Leaf Fiber Length on the Properties," 2015).

3.2.1 Pineapple Leaf Fibre

In Malaysia, there are many plants that can be made as natural fibres. Most of them have speciality to become the best natural fibres as the reinforcement with the binder for composites. Among of them, the pineapple leaf is one of the most popular based on its mechanical properties. Besides, the usage of PLF has been widely attracted to industries nowadays such as in paper and textile making. This is because of the reasonable length of PLF that can be used for the production. Despite from that, the applications of PLF have huge opportunities since it provides good advantages to be as one of the natural fibres existed in Malaysia (Takahashi, 1990).

In this project, the pineapple leaf fibre is selected because of two reasons. In Malaysia, the focus of pineapple industry is the fruits and the leaf is leaving behind, mainly because of it was composted or burned, and thus, it has wasted a good potential source of fibres. Rather than burning it and will cause negative effect of environment, it is better to extracted the fibres from the leaves obtained.

3.2.2 Starch

For the combination with the pineapple leaf fibre as reinforcement, starch has being as one of suitable matrix that can be used for the composition. This is because the starch has the characteristics of promising raw material and also become as one of the attractive sources. Based on the previous research, the combination with cellulose fibres such as PLF can improve the properties of the starch, since it is one of the thermoplastic types.

Therefore, in this project, the starch has been provided in powder form. In order to undergo the formation process with the PLF, the starch is initially heated until it melted before mixing with the PLF in the compression process (Selamat et al., 2016). Therefore, the presence of bonding agent into the formation will increase the properties and providing the best results of the composition. The following Figure 3.2 shows the image of starch in powder form:

Figure 3.2: Starch in powder form

3.2.3 Composition of SH/GLY

Before the formation process between reinforcement of PLF with matrix of SH, the properties of GLY towards the composition is initially find out. This is done by the finding of the best composition of the matrix SH with the bonding agent GLY. The process to find the composition need a repeated process until it acquires the specifications required. The specifications of the sample is high strength, non agglomerate and flexible. Therefore, the process requires several compositions between the SH and GLY in order to find the best composition among the others as shown in Figure 3.1 below.

Table 3.1: Preliminary composition of SH/GLY

Sample	SH (%)	GLY (%)
1	85	15
2	80	20
3	75	25
4	70	30
5	65	35

3.2.4 Composition of PLF/SH

In this project, the PLF is used as reinforcement while the SH is used as a binder/matrix. This composition is also added with the bonding agent of Glycerol, (GLY) in order to show the comparison of the result between the composites. Since the pineapple leave have not similar in length, the specification of the composition have been decided. The following Table 3.2 is the preliminary composition ratio of PLF/SH also with the addition of bonding agent that will be used in this project:

Table 3.2: The preliminary composition of PLF/SH with Bonding Agent

PLF (%)	SH (%)	Bonding Agent (%)
70	30	2
60	40	
50	50	
70	30	4
60	40	
50	50	
70	30	6
60	40	
50	50	

3.3 FABRICATION METHOD

The fabrication method is actually to produce the sample of the composition which includes the extraction of fibre. The extraction of fibre brings the meaning of the pieces of PLF will undergo mechanical and chemical treatment process in order to extract the fibre. The function of the treatment is to increase the fibre strength and treated the fibre surface. The following Figure 3.3 and Figure 3.4 show the methods of PLF chemical extraction by using NaOH solution before the PLF is getting involved in the mechanical extraction by using kitchen utensils:

Figure 3.3: Chemical treatment of PLF

Figure 3.4: Mechanical extraction method of PLF

3.4 BINDER PREPARATION

The binder being used is starch (SH). The SH is provided in a powder form. Then it will be mixed with the chosen bonding agent which is Glycerol (GLY), with several compositions until it melts parallel to the process of determining of the best and appropriate sample with the same composition that will be done in compression moulding with the PLF in order to choose for the final samples.

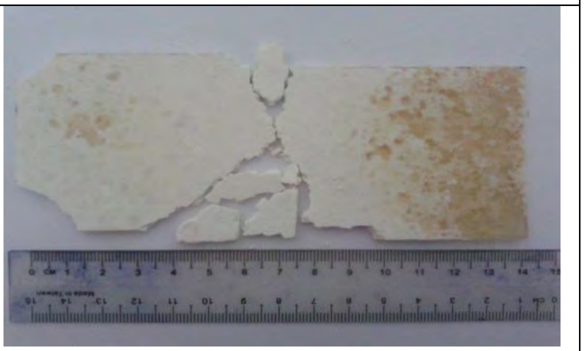
There are several compositions that have been decided in order to find the best combination of compositions between SH and GLY as shown in Table 3.3. The relationship between these two materials is being conducted in hotpress machine. The size of the mould involved for compression process is 140mm x 60mm. Since the compression process occurs in a hot temperature, the cooling process is done before SH/GLY is removed from the mould. The composition ratio of SH/GLY is being set up as in order to find out the best composition.

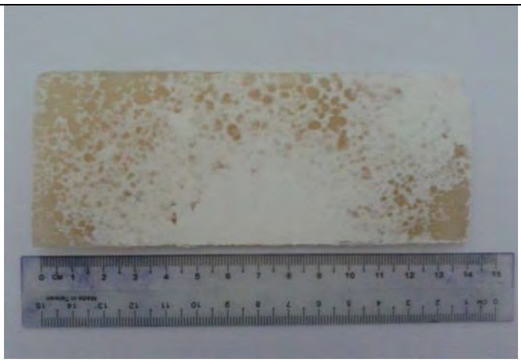
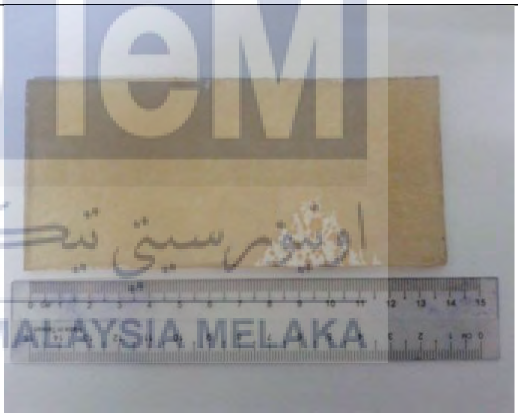
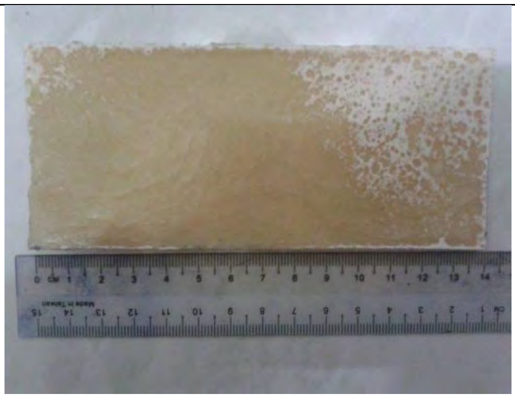
Table 3.3: Composition of SH/GLY

Sample	Composition	SH (g)	GLY(g)	Total weight (g)
1	85SH/15GLY	34.0	6.0	40.0
2	80SH/20GLY	32.0	8.0	40.0
3	75SH/25GLY	30.0	10.0	40.0
4	70SH/30GLY	28.0	12.0	40.0
5	65SH/35GLY	26.0	14.0	40.0

The probability of the combination between these two materials could obtain results in the form of high strength, shape, smooth surface, colour and flexibility. Table 3.4 below shows the samples with the composition of SH/GLY after all of them have been compressed.

Table 3.4: Samples of SH/GLY

Composition	Sample
85SH/15GLY	

80SH/20GLY	
75SH/25GLY	
70SH/30GLY	
65SH/35GLY	

3.4.1 Determine appropriate parameter of matrix SH/GLY

There are two parameters that have been set in the same composition which is 70SH/30GLY as the best composition. The best appearance among the two parameters is being chosen for the final sample. Table 3.5 below shows that the two types of parameters that have been done with the same composition and sample two has been chosen as the best and appropriate sample due to its strength, smooth surface and flexible compared to sample one.

Table 3.5: Comparison of parameters

Sample	1	2
Pressure	200 tonne	200 tonne
Temperature	165°C	165°C
Preheat time	10 minutes	15 minutes
Compressed time	15 minutes	20 minutes
Composition	70SH/30GLY	70SH/30GLY
Results		

3.5 COMPRESSION MOULDING

The sample of PLF/(SH/GLY) then is prepared by using manual mixing technique. The PLF and best composition of SH/GLY which is 70SH/30GLY powder form undergo the process of compression by using hot press machine as shown in Figure 3.5 below.

Figure 3.5: Hot Press Machine

The sample is being placed in the mould along with the arrangement of layer by layer technique (Supri & Lim, 2009). The size of the mould is 140mm x 60mm as shown in Figure 3.6 below. The temperature and time used to compress the sample, pre-heat time; pressure is already set during the compression process. Table 3.6 shows the comparison between all the compositions involved in this PLF/SH composite.

Table 3.6: Composition of PLF/(SH/GLY)

Sample	Composition	PLF (gram)	SH/GLY (gram)
1	70PLF/30(SH/GLY)	14.0	6.0
2	60PLF/40(SH/GLY)	12.0	8.0
3	50PLF/50(SH/GLY)	10.0	10.0

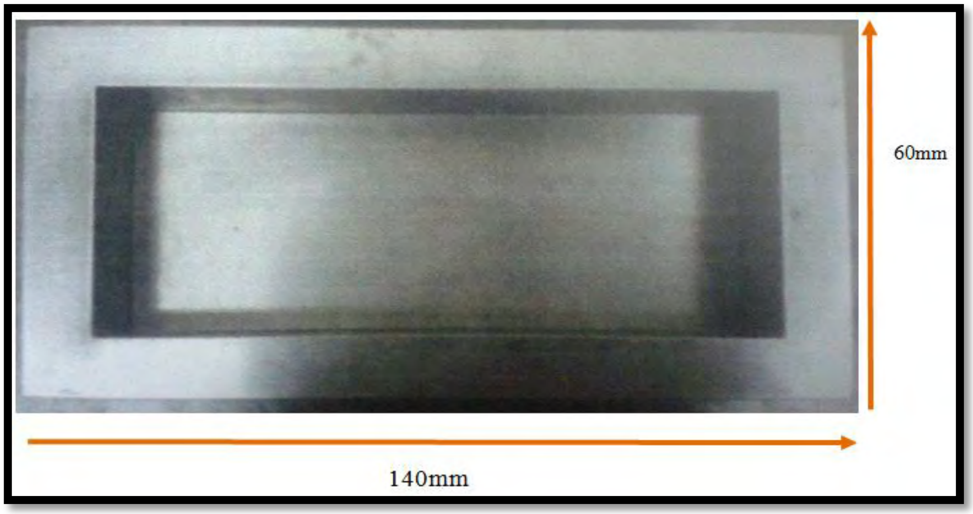


Figure 3.6: Size of mould

There are several processes need to be set at hotpress machine before the machine is operating. The temperature used to compress the sample is 165°C and the pressure set at 200 tonne. The compression processes started with pre-heat for 15 minutes and continue with compression time for about 20 minutes. Figure 3.7 shows the mould is pressed during compression process.

Figure 3.7: The mould is pressed by hotpress machine.

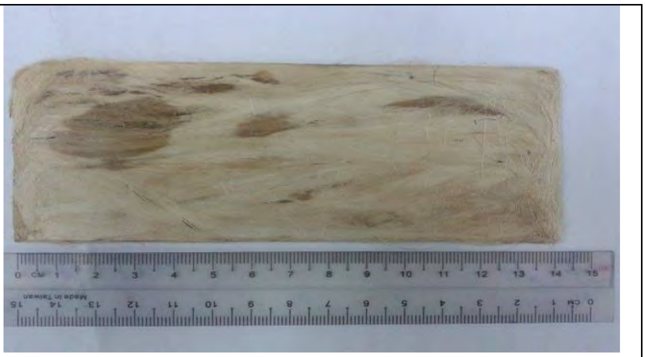
After compression process has completed, the mould is taken out from the hotpress machine and being proceeded with cooling process where it takes about 15 minutes by using low press machine as shown in Figure 3.8 below. The purpose of the cooling process is to cool down the heated mould as long as to make it easier to pull out the sample for the analysis.

Figure 3.8: The cooling process

After the sample has been removed from the mould, the observation towards the sample can be made. Therefore, all the samples for all the compositions can be compared as shown in Table 3.7 below.

Table 3.7: Samples of PLF/(SH/GLY)

Composition	Sample
70PLF/30(SH/GLY)	

60PLF/40(SH/GLY)	
50PLF/50(SH/GLY)	

3.6 CUTTING PROCESS

After the sample of PLF/(SH/GLY) having the compression method, the composites sample will be cut to the specified dimension in order for doing mechanical testing. The dimension is according to the standard required which is ASTM D 790-03 for flexural testing and ASTM D 3039/D 3039M-00 for tensile testing (Barkoula, Garkhail, & Peijs, 2010). The cutting process will be use the proxxon table saw as shown in Figure 3.9 below provided in laboratory as the equipment involved for cutting process.

Figure 3.9: Proxxon Table Saw

The dimension of sample according to the ASTM D 3039/D 3039M-00 is 13mm x 140mm. Therefore, all the samples have been cut based on the dimension given before undergo tensile test. Figure 3.10 below shows one of the PLF/(SH/GLY) sample that has been cut based on the dimension of ASTM D 3039/D 3039M-00.

Figure 3.10: Dimension of PLF/SH sample

3.7 MECHANICAL TESTING

There are four types of mechanical testing that will be undergo throughout this project which are tensile test, hardness test, density test and microstructure analysis test. These four testing have their function which is to determine and analyze the strength of the composition of the natural fibre and binder along with the bonding agent. For tensile test, it has its own standard required for doing the testing which is ASTM D 3039/ D 3039M-00. This standard highlights about the dimension of the samples required in the standard before it will be fixed into the Universal Testing Machine for tensile testing.

3.7.1 Tensile Test

Tensile test is one of the most basic in mechanical testing that performs for determining the properties of the material. The tensile test is simple, inexpensive and standardized. The strength properties of the material will be determined by pulling the material until it breaks. Thus, the behaviour of the material under the forms of loading can be analyzed.

There has tension that is applied during the tensile test. The main properties which can be obtained during tensile test are ultimate tensile stress and strain. Therefore, it is also functions to create the stress-strain curve of the material being tested (Testing, 2004). The standard of the tensile test is the American Standard Testing Machine (ASTM D 3039/ D 3039M-00) and the machine used for this tensile test operation is Universal Testing Machine Instron 8872 as shown in Figure 3.11.

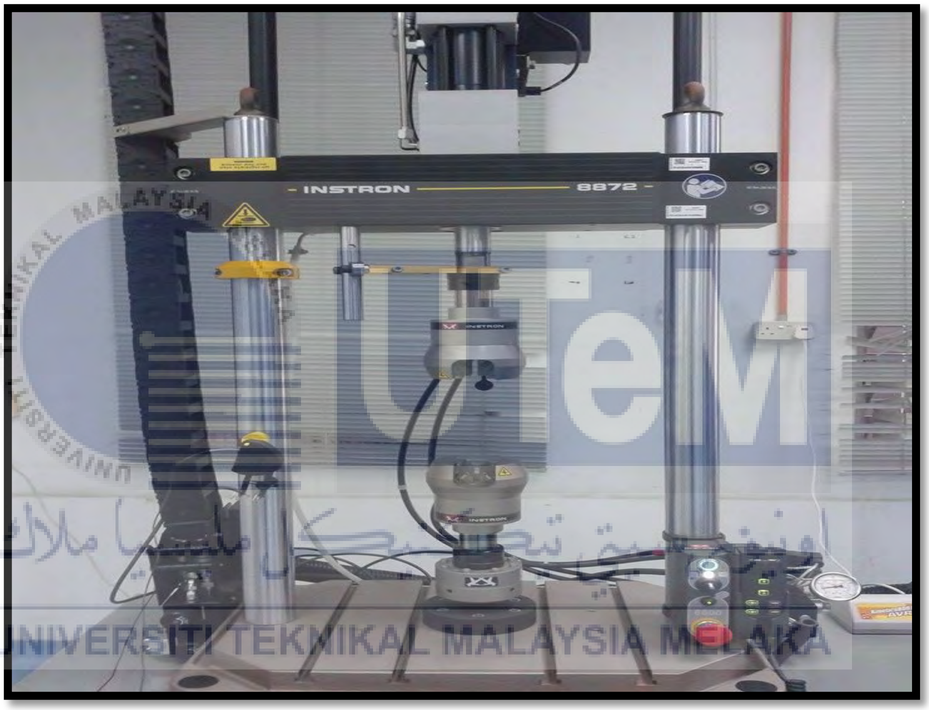


Figure 3.11: Universal Testing Machine Instron 8872.

In order to conduct the tensile test, the sample of PLF/(SH/GLY) is being cut by using proxxon table saw before it is undergo tensile test with the length of the sample is 140mm and the width is 13mm. This dimension is according to the ASTM D 3039/ D 3039M-00, and the span length is marked on the sample as shown in Figure 3.12 before it is mounted on the machine as shown in Figure 3.13.

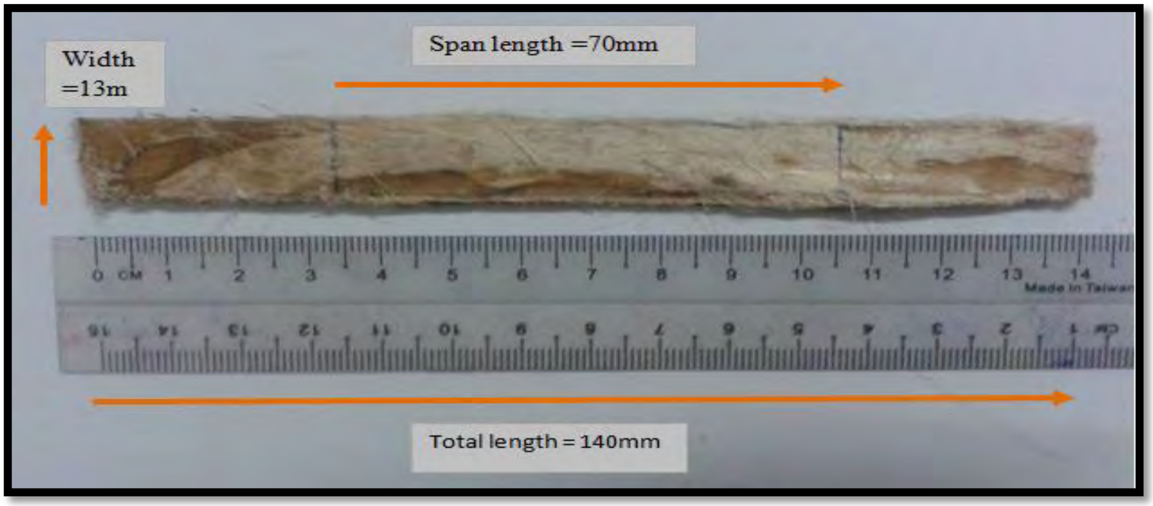


Figure 3.12: Dimension of sample for tensile test

Figure 3.13: The sample is mounted on the machine

Lastly, all the parameters and conditions involved such as total length, span length, width and thickness is set on the software provided. The speed used is being set before the test is conducted which the speed used is 2.2mm/minute. The tensile test operation is started until fracture occurs at the sample and the result of stress-strain curve is being printed as shown in Figure 3.14.

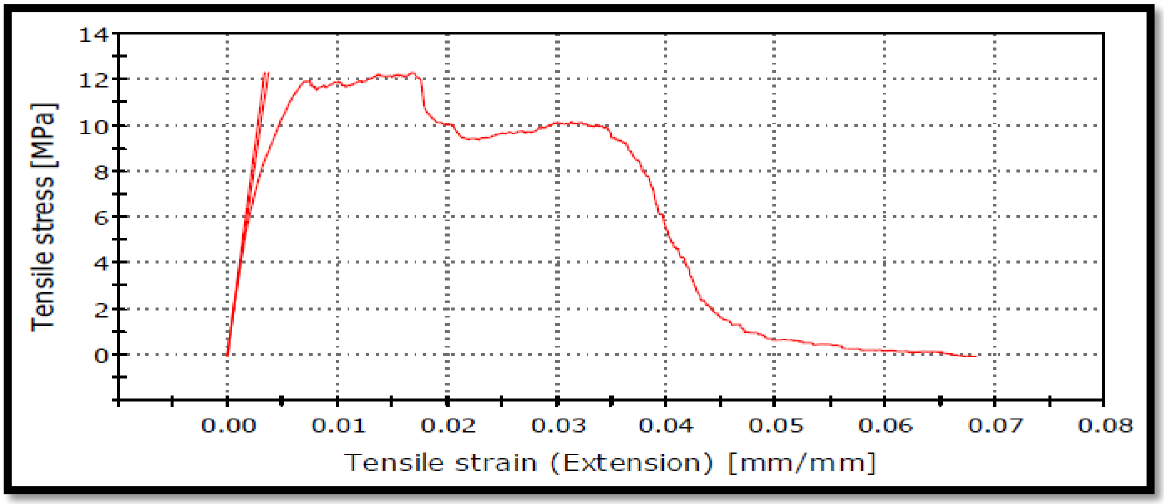


Figure 3.14: Stress-strain curve

3.7.2 Hardness Test

Hardness is conducted to test the hardness composition of PLF/(SH/GLY). There are five factors that can be used to determine the correct hardness test which is material used, approximate hardness, the shape, heat treatment and also the production requirement. For measuring the hardness of the sample, the hardness device which is shore hardness tester is pressed firmly at a constant speed vertically to the sample at a flat surface and the reading of the hardness will be read manually (Zhao, Allanson, & Ren, 2015).

In simplest word, hardness test has the function to determine the properties of hardness between PLF and SH/GLY. The Analogue Shore Scale is used to run the hardness test. Figure 3.15 shows the Analogue Shore Scale.

Figure 3.15: The Analogue Shore Scale

There are several steps in order to conduct this hardness test. Firstly, the sample is placed on a flat surface. Then, the indenter is pressed into the sample and the reading is taken by referring the red scale on the Analogue Shore Scale. Figure 3.16 below shows the method on measuring the hardness of the sample. All the steps are repeated with other sample of different compositions of the PLF/(SH/GLY) composite materials.

Figure 3.16: Measurement of the sample

3.7.3 Density Test

For this section, the density test is being conducted in order to determine the specific gravity or density of PLF/(SH/GLY) composite materials. This density test is done by using Electronic Densimeter as shown in Figure 3.17. There are a few steps to conduct this test. Firstly, the sample is being cut into smaller pieces and then being placed on the container in order to find the mass value of the sample.

Next, the sample is placed into the water and the specific gravity of the sample and also the volume is automatically being measured by the densimeter. Then, the reading is being displayed and all the steps are repeated with other samples. The average value then is being calculated.

Figure 3.17: Electronic Densimeter

3.7.4 Macrostructure Analysis

The objective of macrostructure analysis on the composite sample is to study the behaviour inside of the microstructure of the PLF/(SH/GLY) composite. There are a few elements that can be made from this analysis such as surface morphology of the material that includes crack, porosity and the voids of the material.

In order to run the microstructure analysis, the Dino Lite Digital Microscope is used as the device to determine the behaviour inside of the composite material. Figure 3.18 below shows the Dino Lite Digital Microscope.

Figure 3.18: Dino Lite Digital Microscope

There are a few steps that need to be taken before the analysis can be started. The first step is the digital microscope need to be connected with the computer. Next, the sample that wanted to be analyzed is being put under the lens of the microscope. Then the images of the sample can be controlled by adjusting the magnification of the lens. Therefore, the analysis can be finished with the systematic capture by the computer and all the steps are repeated for the other compositions.

CHAPTER 4

DATA AND RESULTS

This chapter shows all the experimental data and results for all the mechanical testings that have been conducted through all the composite samples. The all four mechanical testing involved are tensile test (ASTM D 3039/D 3039 M-00), hardness test, density test and also microstructure analysis.

4.1 TENSILE TEST

The tensile test is used to determine the mechanical properties or behaviour of the composite sample that has been subjected under the form of loading. Therefore, the Universal Testing Machine Instron 8872 is used in order to do the test operation. The standard test method that has been used is based on Tensile Properties of Polymer of Composite Material (ASTM D 3039/D 3039 M-00) and it has been run with constant speed (2mm/minute). The samples used consist of the composition of 50PLF/50(SH/GLY), 60PLF/40(SH/GLY), 70PLF/30(SH/GLY) and also one sample of 70SH/30GLY, and then the data collected are shown in graph of tensile stress (MPa) versus fibre loading (%).

4.1.1 PLF(SH/GLY) and SH/GLY Tensile Test Result

Figure 4.1 shows the graph of tensile stress (MPa) versus composition (%). The data of all samples are shown in Figure 4.1 with the composition of 50PLF/50(SH/GLY), 60PLF/40(SH/GLY), and 70PLF/30(SH/GLY). Based on the graph obtained, it shows the overall increasing value from 50PLF/50SH to 70PLF/SH but it also shows a slightly decreases in tensile stress value at 60PLF/40(SH/GLY) composition. For the matrix material composition which is 70SH/30GLY, it shows the lowest value because of the properties of the material which is elastic and does not require much load and easy to break the sample.

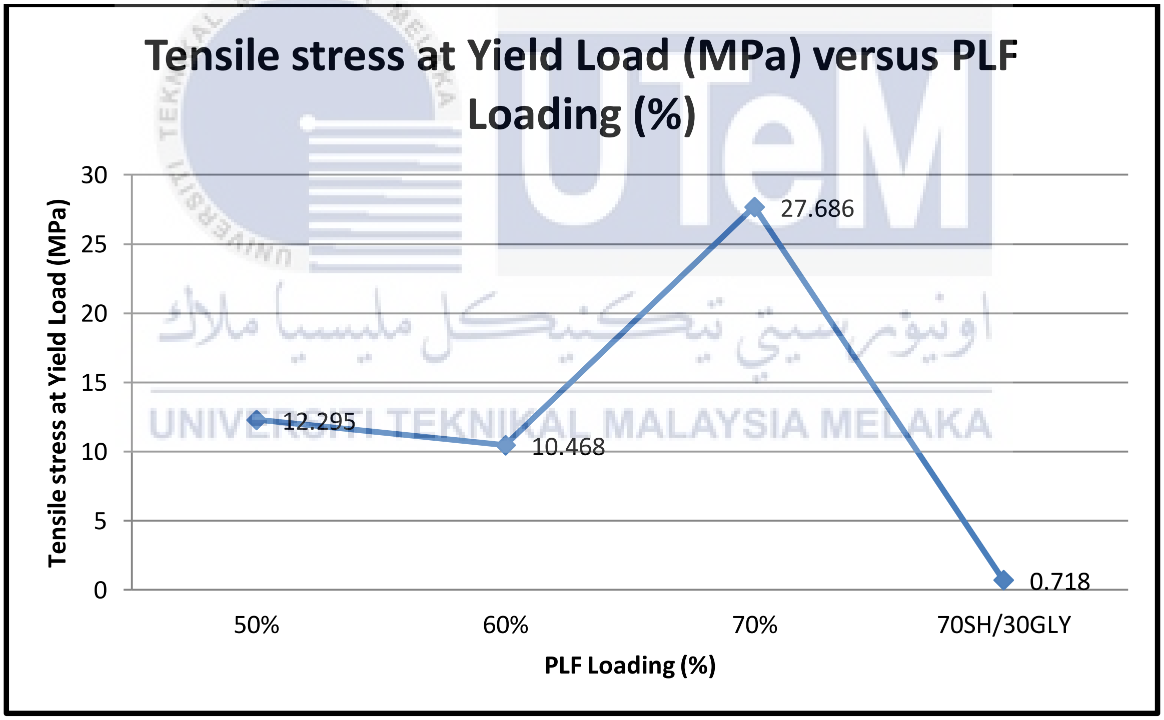


Figure 4.1: Tensile Stress (MPa) versus PLF Loading (%)

All the data that includes tensile stress (σ), Young Modulus (E) and strain (ε) for the composition 50PLF/50(SH/GLY), 60PLF/40(SH/GLY), and 70PLF/30(SH/GLY) have been calculated as shown in Table 4.1 respectively. According to the Table 4.1, the lowest value of

Young Modulus between reinforcement and matrix composite is 0.37 which at the composition of 60PLF/40(SH/GLY) while the highest value is 1.06 at the composition of 70PLF/30(SH/GLY). Besides, the matrix composition 70SH/30GLY shows the value of 0.02 for the Young Modulus.

Table 4.1: Tensile test data

No	PLF Loading (%)	Tensile stress, σ (MPa)	Young Modulus, E (GPa)	Strain, ε (%)
1	50.0	12.295	1.02	0.012
2	60.0	10.468	0.37	0.028
3	70.0	27.686	1.06	0.026
4	70SH/30GLY	0.718	0.02	0.033

4.2 HARDNESS TEST

For this section, the hardness of the composite sample is measured on the composition of reinforcement and matrix of the material. The Analgue Shore Scale type-D is used in order to test the hardness each of the PLF/(SH/GLY) and 70SH/30GLY samples. The repeated measurement is taken and the average value of the hardness is calculated.

4.2.1 PLF/(SH/GLY) and SH/GLY Hardness Test Result

All the data have been collected and being analyzed. Thus, the graph of hardness versus composition is being constructed for all the compositions as shown in Figure 4.2. Besides, Table 4.2 shows the tabulation of data to show that all the data have been obtained during the test.

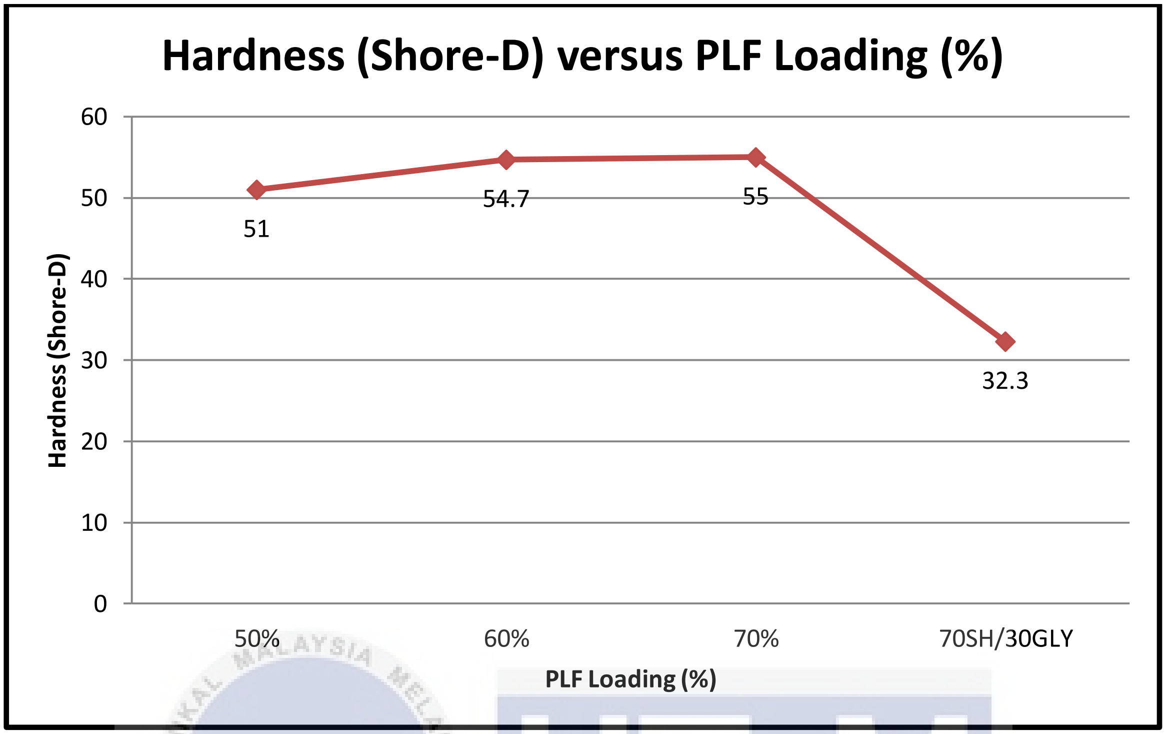


Figure 4.2: Hardness (Shore-D) versus PLF Loading (%)

Table 4.2: Hardness test data

PLF Loading (%)	Hardness (Shore-D)			Average
50.0	51.0	52.0	50.0	51.0
60.0	50.0	56.0	58.0	54.7
70.0	57.0	55.0	53.0	55.0
70SH/30GLY	33.0	33.0	31.0	32.3

Based on the Table 4.2, the hardness test results show that the composition of PLF/(SH/GLY) composite material is increasing from 50PLF/50(SH/GLY) to 70PLF/30(SH/GLY). In overall in the Figure 4.2, there are increases in graph pattern which is directly proportional with the increasing of PLF to the samples. The matrix material of 70SH/30GLY shows the lower value of hardness due to its elastic properties of the material.

4.3 DENSITY TEST

The Electronic Densimeter is used in order to conduct the testing and the reading is recorded in Table 4.3 below. Therefore, the graph of density of the samples versus composition is constructed as shown in Figure 4.3 below.

4.3.1 PLF/(SH/GLY) and SH/GLY Density Test Result

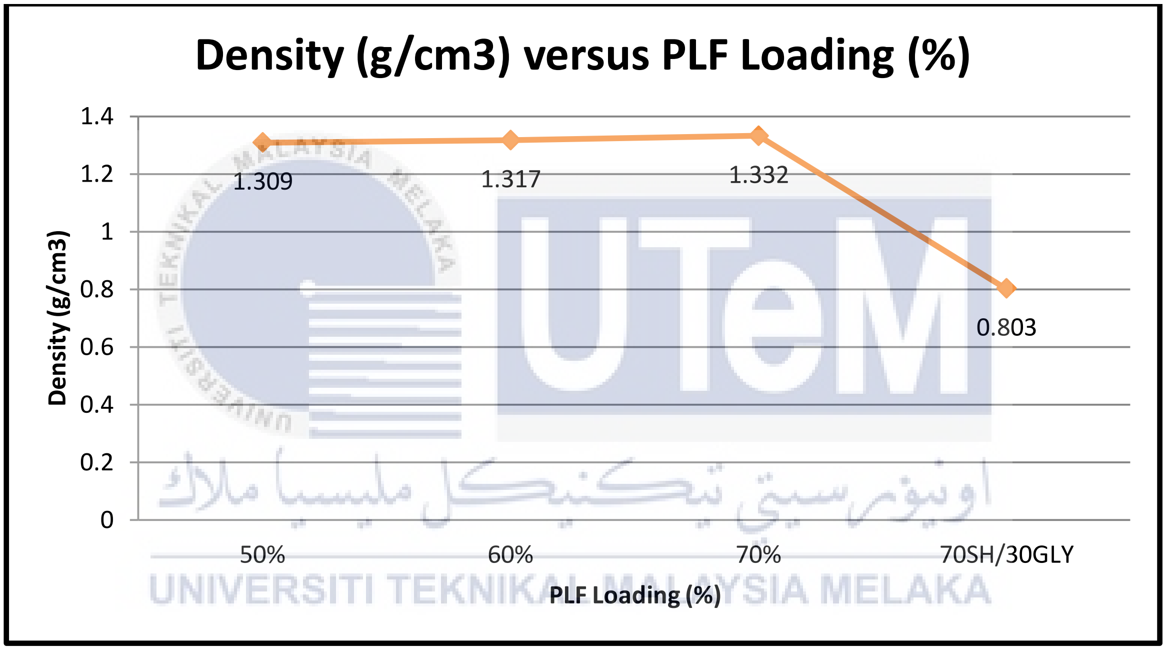


Figure 4.3: Density (g/cm^3) versus PLF Loading (%)

Table 4.3: Data of density test

PLF Loading (%)	Density (g/cm^3)			Average
50.0	1.330	1.300	1.298	1.309
60.0	1.380	1.260	1.310	1.317
70.0	1.304	1.351	1.342	1.332
70SH/30GLY	0.659	0.863	0.887	0.803

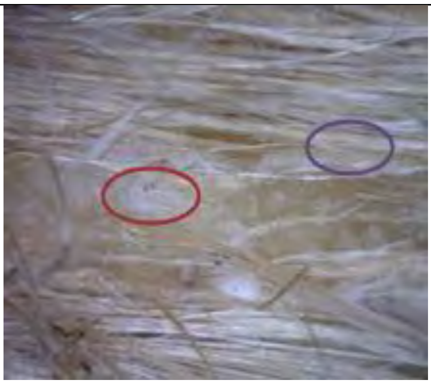
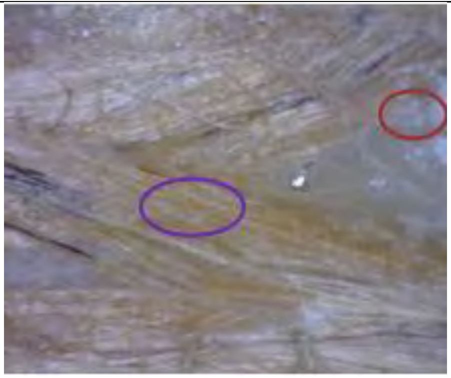
Based on the Figure 4.3, the graph shows an increasing pattern from 50PLF/50(SH/GLY) until 70PLF/30(SH/GLY). The highest value of density is shown at 70PLF/30(SH/GLY) composition with the value of 1.332 g/cm^3. Therefore, it can be summarized as the increasing the composition of PLF is making the value of density getting higher. Thus, the value of density of 70SH/30GLY remains lowest with the value of 0.803 g/cm^3.

4.4 MACROSTRUCTURE ANALYSIS

In this section, macrostructure analysis is being conducted in order to view the macrostructure of each sample. The results shown are the structure of the surface morphology of the sample if there have any attendance of void, crack, and impurities based on its composition. Table 4.4 below shows the view of data for the macrostructure analysis.

4.4.1 PLF/(SH/GLY) Result

Table 4.4: Macrostructure analysis view

Composition	Macrostructure View	
	200mm magnification	150mm magnification
50PLF/50(SH/GLY)		

60PLF/40(SH/GLY)		
70PLF/30(SH/GLY)		

◯ = PLF

◯ = SH/GLY

Based on the all data in the Table 4.4, the behaviour of PLF and SH/GLY can be observed clearly by using Dino Lite Digital Microscope. The behaviour between these two materials seems to be accumulated among the PLF with the higher composition percentage of the SH/GLY. The figure also shows some SH/GLY is spotted to accumulate at the certain spot only. Thus, this kind of analysis may be affecting the result on the mechanical properties like tensile test.

CHAPTER 5

DISCUSSION AND ANALYSIS

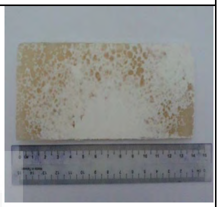
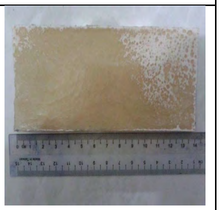
In this chapter, all the results obtained from all the mechanical testing to the samples is shown and discussed. The behaviour and properties of the PLF and SH which determined from tensile test (ASTM D 3039/D 3039 M-00), hardness test, density measurement and macrostructure analysis also being analyzed.

5.1 PARAMETER FINDINGS

For the parameter findings, it is all about finding the parameters composition of SH with the bonding agent of glycerol, (GLY). For this finding, the trial and error method is used in order to find the best composition of SH/GLY. Therefore, in order to find the appropriate parameter for the matrix, it is obviously a crucial step as it is the main matrix component before the sample of the best composition can be done for the best matrix composition.

By the way, the results of SH/GLY obtained from this part are not accurate as the reasons can be done in a various ways. The matrix content, heating time for compress the materials and size of the mould can be the problems or reasons why this kind of study seems could not to be achieved. The temperature of 165^{o}C is chosen for compressing the material. The best composition for matrix material is observed based on its higher strength, non-agglumerate surface and flexible. Table 5.1 shows the result obtained from this experiment.

Table 5.1: Parameters findings of SH/GLY

Sample	Composition	Temperature (^{o}C)	Pressure (tonnue)	Preheat Time (mins)	Compress Time (mins)	Sample
1	85SH/15GLY	165	0	10	10	
2	80SH/20GLY	165	0	10	10	
3	75SH/25GLY	165	200	10	20	
4	70SH/30GLY	165	200	15	20	
5	65SH/35GLY	165	200	15	20	

The temperature is set to be 165^{o}C because of the melting temperature for the starch to be form with the glycerol which contains high amylose content inside its properties. Therefore, the higher temperature will give harm for the starch to be melted and it also can affect the other result of mechanical testing.

Based on the Table 5.1, it also shows the increasing of time for pre-heat and compress gives greater result for the matrix sample. The best composition sample among the rest is 70SH/30GLY for have the requirement of most smooth surface, non agglomerate, more high strength and flexible. This is because of longer time taken for the materials form to be heated; it will affect more content of the matrix composition. For this case, the phenomenon of heat transfer is need to be considered. The heat being transferred from the hotpress machine to the mould requires sufficient of time to form thermal equilibrium. Otherwise, the result obtained for the sample composition is unsuccessful.

5.2 EFFECT OF PLF COMPOSITION AND PLF LOADING ON TENSILE

The tensile properties of the composites material are shown in the previous Table 4.1. The results show based on the composition from 50PLF/50(SH/GLY), 60PLF/40(SH/GLY) and 70PLF/30(SH/GLY). The results obtained finding different composition with a different size of particulate matrix is providing a different result significantly.

Figure 5.1 shows the result of comparison for all the samples involved in the tensile testing. The highest value is 27.686 MPa at composition 70PLF/30(SH/GLY) while the lowest value is 10.468 MPa at the composition 60PLF/40(SH/GLY). Meanwhile, the composition of 50PLF/50(SH/GLY) shows high value compared to 60PLF/40(SH/GLY) which is 12.295. However, generally it can be summarized that the tensile stress are gradually increased as the composition of the PLF content is increased.

The tensile stress is higher at 70% of PLF loading as compared to the other two fibre loadings, which is the highest value of 27.686 MPa. This is because more fibre loading or content enhances the strength of the composites. Hence, the function of matrix is to transfer the forces to the fibres. If the fibre loading increased, it can withstand higher forces as compared to lower fibre contents.

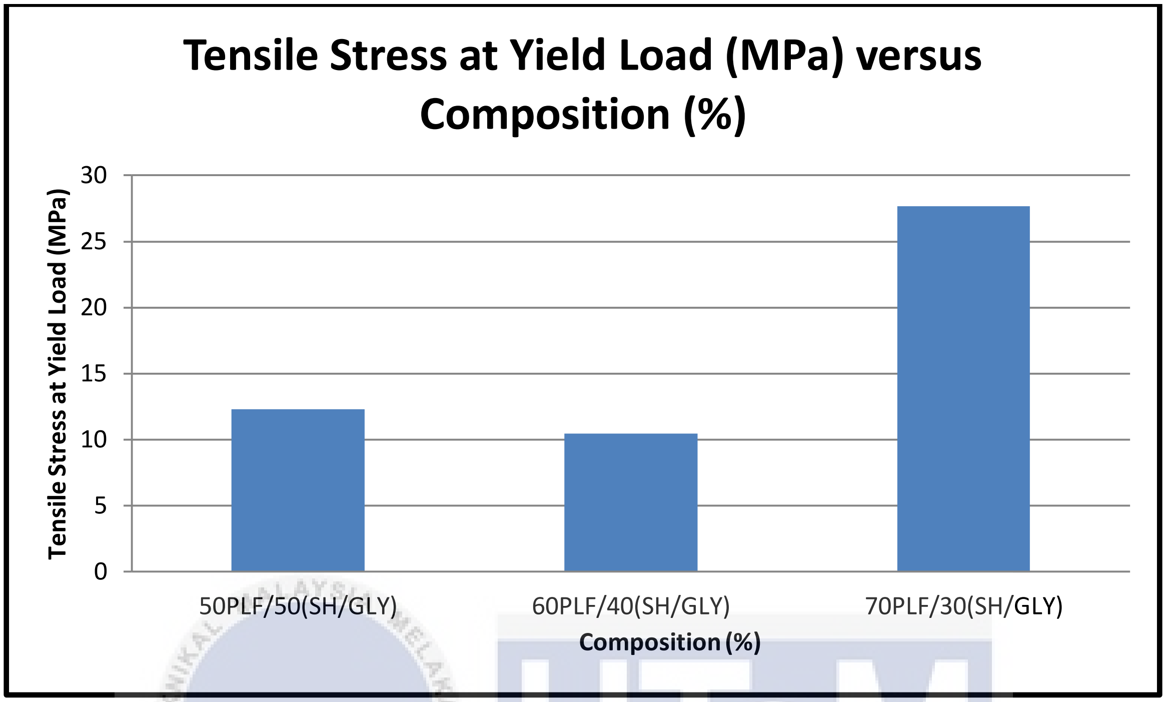


Figure 5.1: Tensile Stress (MPa) versus composition (%)

Beside that, the composition of matrix in the composite material is being analyzed whether it could affect the result on the mechanical properties or not. The content of matrix should be in finest condition in order to give higher value in the mechanical properties of the sample. The matrix has the ability to hold the fibre content firmly as it is used to react and melt very well as it being pressed during compression process (Ayu Natasya Kasim et al., 2015).

Besides, the orientation of fibre is should be in the transverse or vertical direction in order to get better results. Figure 5.2 below shows the view of the position of PLF along with the content of matrix for the composition 70PLF/30(SH/GLY) which is to hold the fibre firmly in order to get better result. Thus, it will make the mechanical properties went stronger.

Figure 5.2: Macrostructure view of 70% fibre loading

5.3 EFFECT OF PLF COMPOSITION AND PLF LOADING ON HARDNESS

Figure 5.3 below shows the results of hardness (Shore-D) with the various percentages of compositions of PLF and SH/GLY content composites material. The results in the Figure 5.3 shows that the hardness value is increase with the increment of the fibre loading from 50% to 70% and particulate size of the matrix material for all the samples composition. The average value is taken in order to get more accurate results.

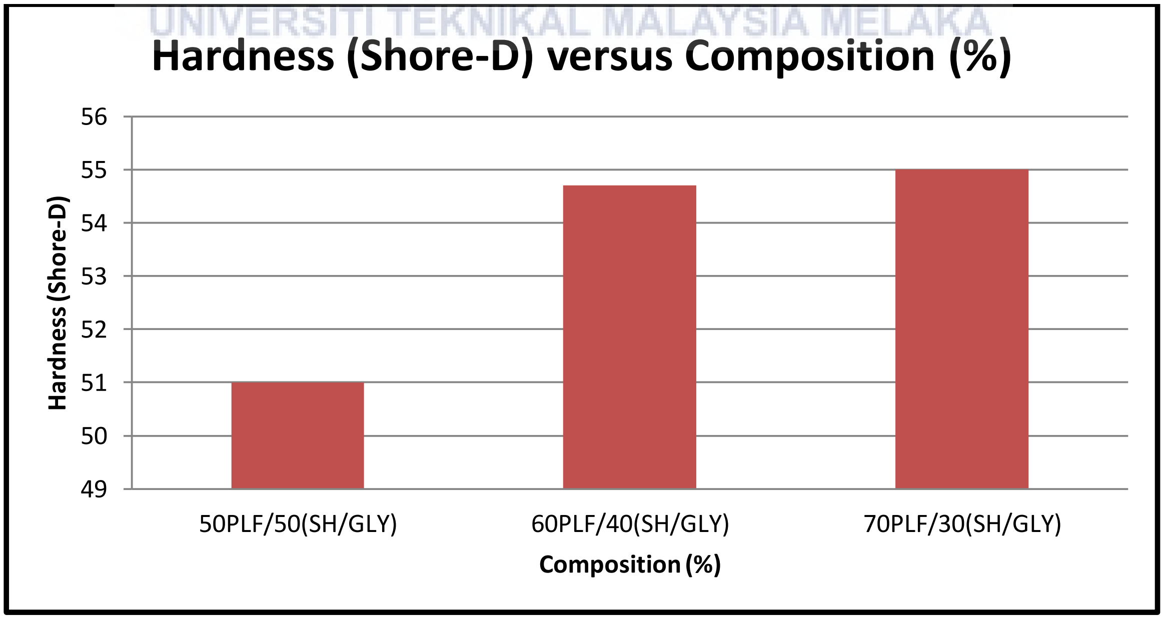


Figure 5.3: Hardness (Shore-D) versus composition (%)

Based on the graph, the highest value of hardness is 55.0 at the composition of 70PLF/30(SH/GLY) while the lowest value is 51.0 at composition 50PLF/50(SH/GLY). Since the reinforcement material is in the random discontinuous orientation, it makes an uneven dispersion of matrix. In addition, the higher value of matrix content will makes it harder for the PLF to mix well with the SH/GLY content. Therefore, it makes the sample with the larger content of PLF the highest value of hardness compared to the lower amount of PLF composition for the composite material.

For composition 60PLF/40(SH/GLY). It shows a very small difference of hardness with the value of 54.7 compared to the 70% fibre loading with the value of 55.0. For fibre loading, it can be seen obviously that, the highest content of fibre loading which is 70% has shown the highest value of Shore-D hardness. Besides, the results may be due to the unsmooth surface or the matrix and the fibre are not properly mixed during the compression process and make the matrix does not melt properly. Hence, the sample cannot withstand the wear resistance of the composites. This is the reason why the experimental to be repeated for a several times in order to get better results.

5.4 EFFECT OF PLF COMPOSITION AND PLF LOADING ON DENSITY

For the density measurement, all the data are collected and being tabulated based on their composition for all the samples. The compositions are 50PLF/50(SH/GLY), 60PLF/40(SH/GLY) and 70PLF/30(SH/GLY). Figure 5.4 shows the graph of the density with the comparison between all the compositions involved. Overall result for the composites materials do not show much a large distinct.

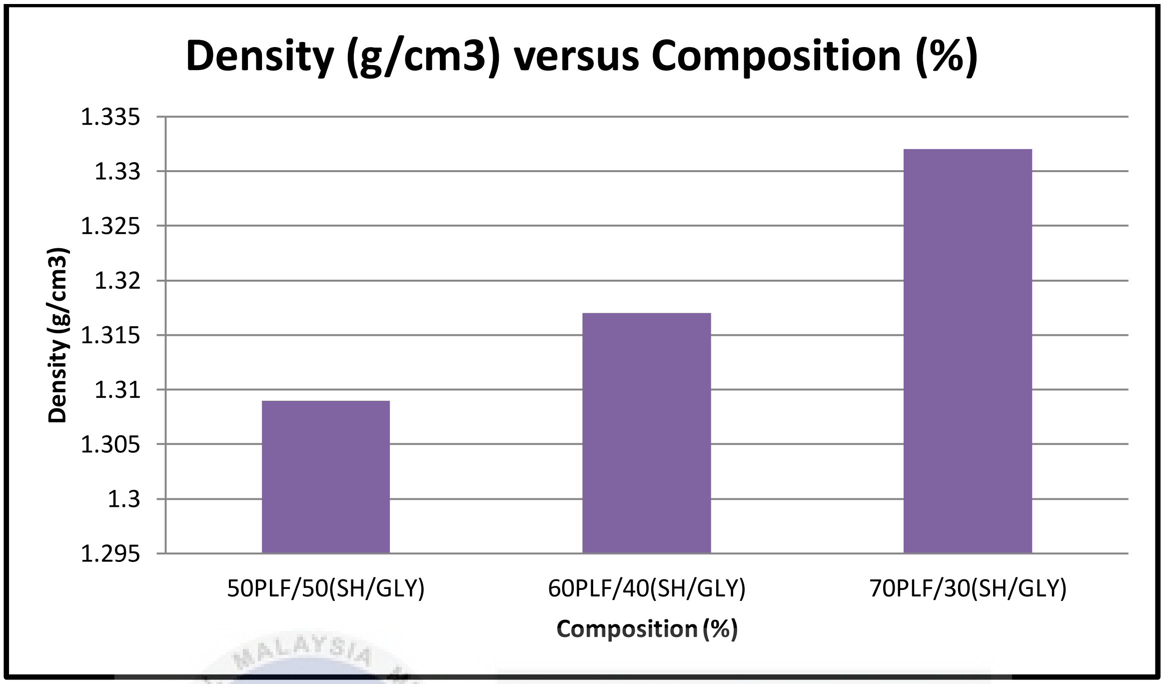


Figure 5.4: Density (g/cm^3) versus composition

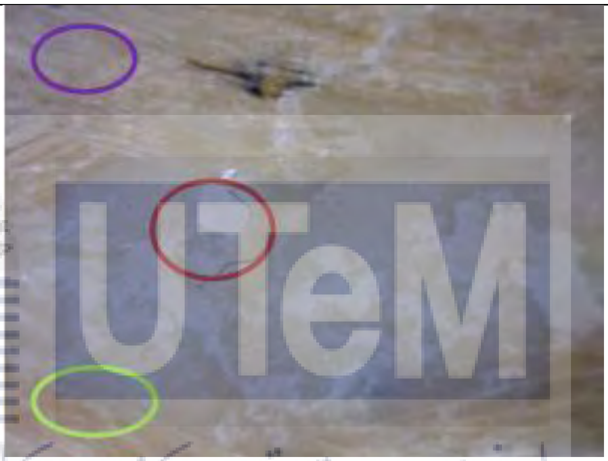
Based on the Figure 5.4, the lowest density value is in 50PLF/50(SH/GLY) with the value of $1.309 g/cm^3$ while the highest value is in 70PLF/30(SH/GLY) with $1.332 g/cm^3$. However, the increment between 50% fibre loading to the 70% fibre loading showing small difference density value between them. Therefore, it shows that the increasing in fibre loading will increase the density measurement of the samples. The increasing of fibre loading is directly proportional to the value of density measurement.

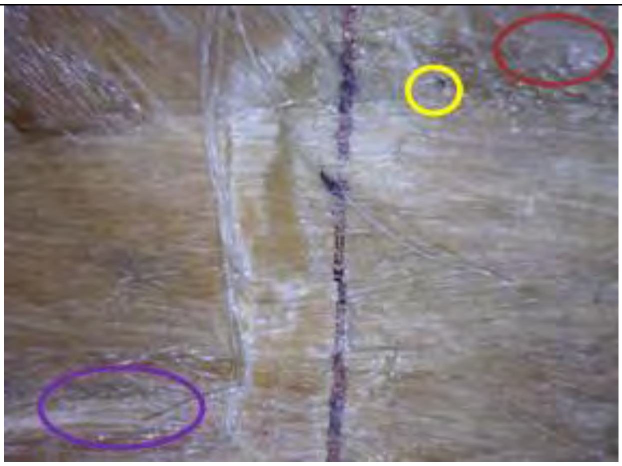
For this case, the highest density is found at the composition 70PLF/30(SH/GLY) because of the ability of the starch to fully melt as it is in less composition. This make the SH/GLY is fully covered with the presence of PLF, and hence make it less water absorption through the samples (Selamat et al., 2016). Therefore, it can be concluded that the content of fibre and the loading of fibre can affect the density of the composites materials. In order to get accurate results, the measurement is repeated with three different specimens for each samples and the average data is recorded.

5.5 MACROSTRUCTURE ANALYSIS

The macrostructure analysis for all the composition based on the percentage of fibre loading and also the matrix materials is being tabulated as shown in Table 5.2. There are a few elements which is surface morphology that can be considered in this analysis such as PLF content, SH content, voids and impurities.

Table 5.2: Macrostructure Analysis

Composition	Macrostructure View
50PLF/50(SH/GLY)	
60PLF/40(SH/GLY)	

70PLF/30(SH/GLY)	

🟣 = PLF mix well with SH/GLY

🔴 = SH/GLY accumulates at certain point only

🟢 = void

🟡 = impurities

Based on Table 5.2 above, the samples with higher composition of SH/GLY tend to have more voids compared to the sample which has less content of SH/GLY. This is because from the higher level of matrix make the material hardly to be mix properly with PLF and the percentage of SH/GLY to be accumulated at the certain point on the sample is high. Therefore, the result shows that the 70PLF/30(SH/GLY) composition previews a greater surface of composite materials with no voids at all.

Besides, the surface of the 50% and 60% fibre loading seems to be agglomerate at the certain part. This is because the PLF do not have a good interfacial reaction when the SH is decreases. Therefore, this could affect the mechanical properties of the sample especially for tensile testing result.

5.6 COMPARISON EFFECT OF PLF COMPOSITION AND PLF LOADING ON DENSITY AND HARDNESS

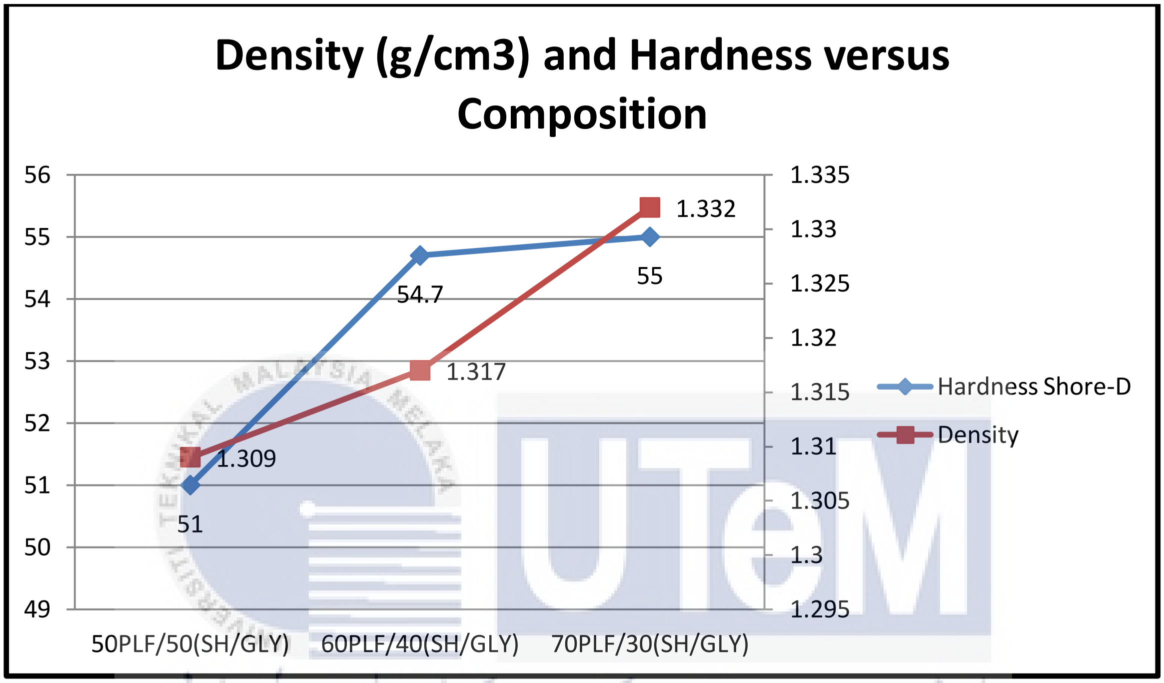


Figure 5.5: Comparison graph between density and hardness

Figure 5.5 shows the comparison of graph between density test and hardness test of all PLF/(SH/GLY) samples. The highest values for both testings are at the composition of 70PLF/30(SH/GLY) where the values are 1.332 g/cm^3 and 55.0 respectively. At the 70PLF/30(SH/GLY) composition, the interaction between PLF and SH/GLY shows that the content of the PLF is more than SH/GLY that leads to the uneven dispersion of SH/GLY towards the PLF content. Besides, more content of PLF will produce less water absorption through the samples. Therefore, the more fibre loading will give high value of hardness and density.

The lowest values for both testing are at 50PLF/50SH composition with the values of 1.309 g/cm^3 and 51.0 respectively. The addition of SH with bonding agent GLY makes the matrix material hardness value is at 32.3 as shown in Figure 4.2 previously. This is proving that the hardness properties of the matrix material are lower. Therefore, that is possible reason why the formation of fibre with high matrix content as shown in Figure 5.5 gives a lower result as shown at 50PLF/50(SH/GLY) and 60PLF/40(SH/GLY) compositions compared to 70PLF/30(SH/GLY). The more increase PLF content is giving higher values for both density and hardness value and surely it can affect the mechanical properties of the samples.

CHAPTER 6

CONCLUSION AND RECOMMENDATIONS

6.1 CONCLUSION

This project is about studying on the green composites which have many advantages for many applications especially in automotive industry in order to make an interior part of automobile. This research also exposed on how to produce natural composites which is from the combination of natural fibre and natural polymer. Therefore, in this study the pineapple leaf fibre is used as the reinforcement material while starch with the addition of glycerol as bonding agent, is used as the matrix material.

Based on the result and discussions, the matrix or binder preparation for this project has been determined. The presence of GLY has shows its effect towards the SH content in order to determine their mechanical properties with the PLF loading. The tensile, hardness and density test show that the sample increases value at the composition of 70PLF/30(SH/GLY) which means that the more fibre loading will give more impact towards the mechanical properties of the samples. From the results obtained in this research, the mechanical properties have showed that these biodegradable materials are useful in the production of composite material.

The preliminary study which is the matrix preparation that is to find the best composition with the trial and error method has been determined even though the result obtained is not too accurate. From the result obtained, the mechanical properties of the matrix material has prove the glycerol properties as plasticizers that could react with starch properties in order to affect the mechanical properties of the PLF.

Besides, the ability of the matrix to be mixed properly and easier to be melt in the fabrication process with the fibre is need to be considered. Thus, this also gives a crucial hint that the matrix could give a good potential in producing a higher performance of the composite material of PLF/SH. Therefore, based on overall on these findings, it can be concluded that the fibre loading will affect the mechanical properties of the composites material.

6.2 RECOMMENDATIONS

There are a few recommendations that can be suggested for further research in this study. The recommendations are intended in order to improve the mechanical properties of PLF/SH composite. The recommendations are listed as below:

1. Fibre length

The PLF can be included in one of the finest fibre that can be used. Therefore in this project, the long fibre is used as the reinforcement in order to determine its mechanical properties. However, it is quite hard to obtain a proper length in order to put inside the mould for compression process. In addition, the orientation of PLF also could be one of the factors that can affect the mechanical properties of the PLF loading. Therefore, a suggestion has been made as the short length with specific 3mm, 4mm and 5mm to be used in order to determine the mechanial properties of composite sample.

2. Compositions of materials

Since this research only used three compositions which are 50PLF/50SH, 60PLF/40SH and 70PLF/30SH, there has a suggestion to add more type of compositions like 40PLF/60SH and 30PLF/70SH in order to find its mechanical properties. This is because this time with the matrix of SH content which is higher than fibre, it could give a wide range of result, observation, assumption and analysis that can be made.

3. Cutting samples

Sometimes when handling the cutting process to the sample by using the proxxon table saw, the rotating saw blade that cut the sample can leads the PLF compressed turns into hairy, and thus can make the PLF stuck and fall inside the hole between rotating saw blade and the possibly can cause injured. The suggestion besides the usage of proxxon table saw is by using cutter or scissors in order to cut the sample for ASTM standard.

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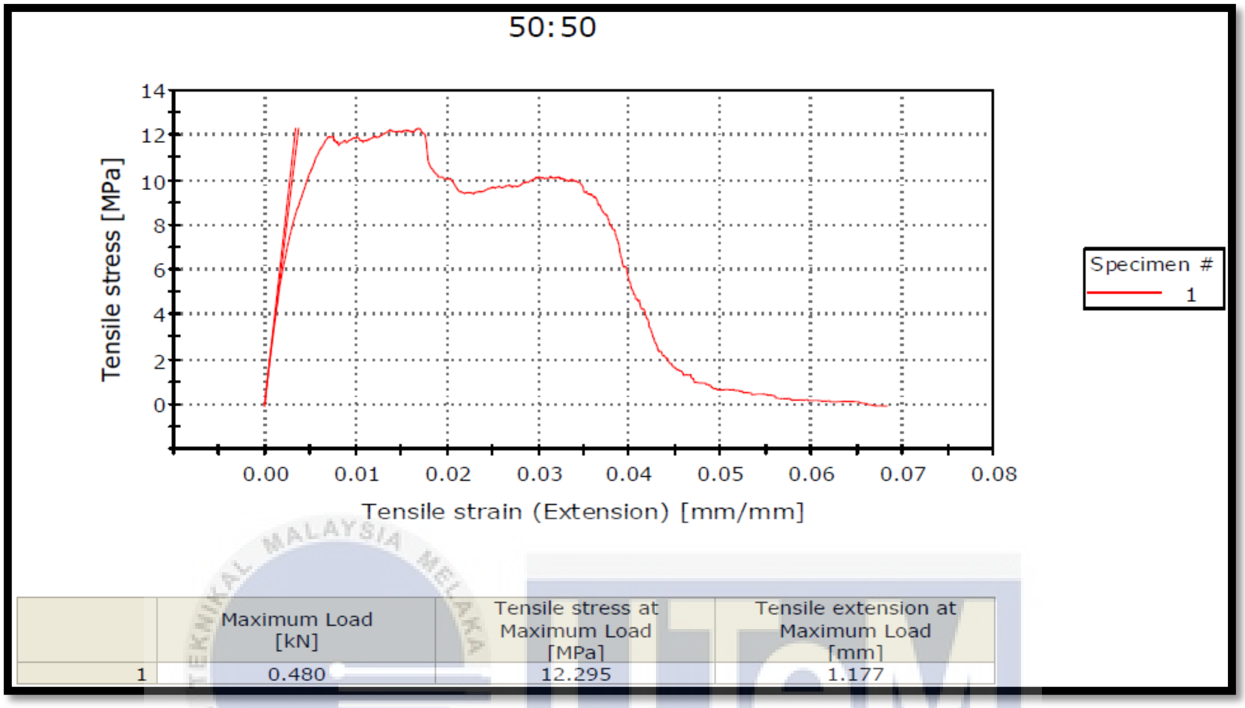
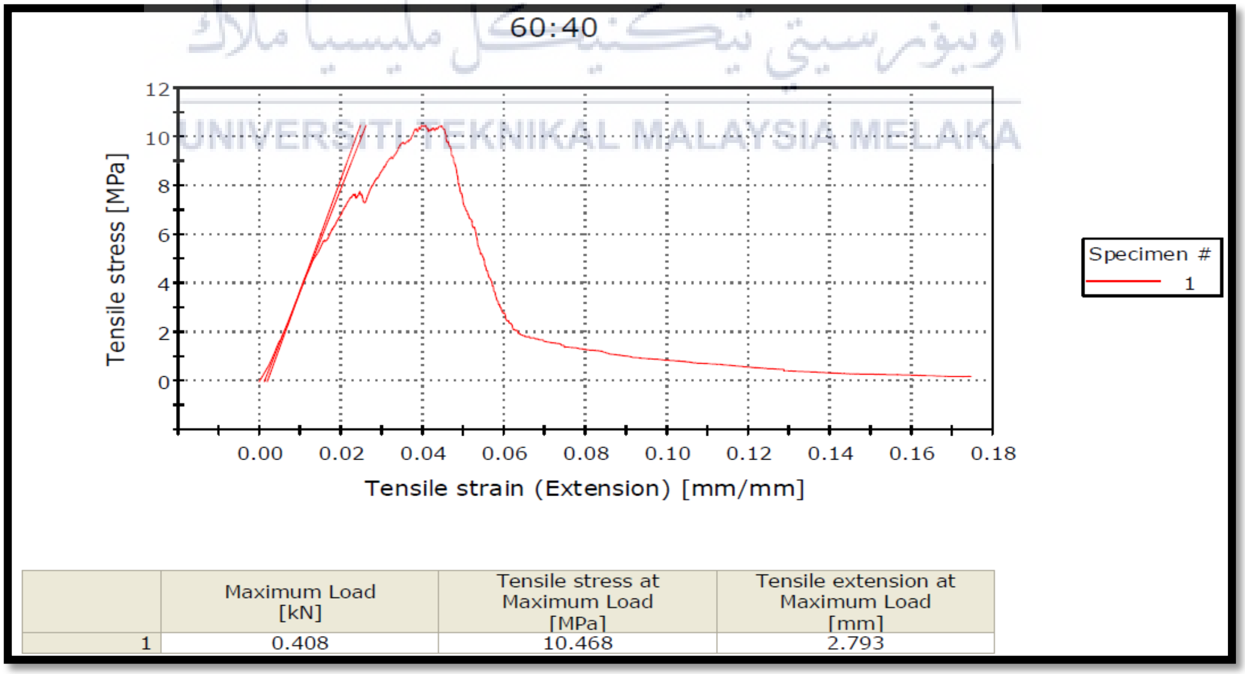
APPENDIX

APPENDIX



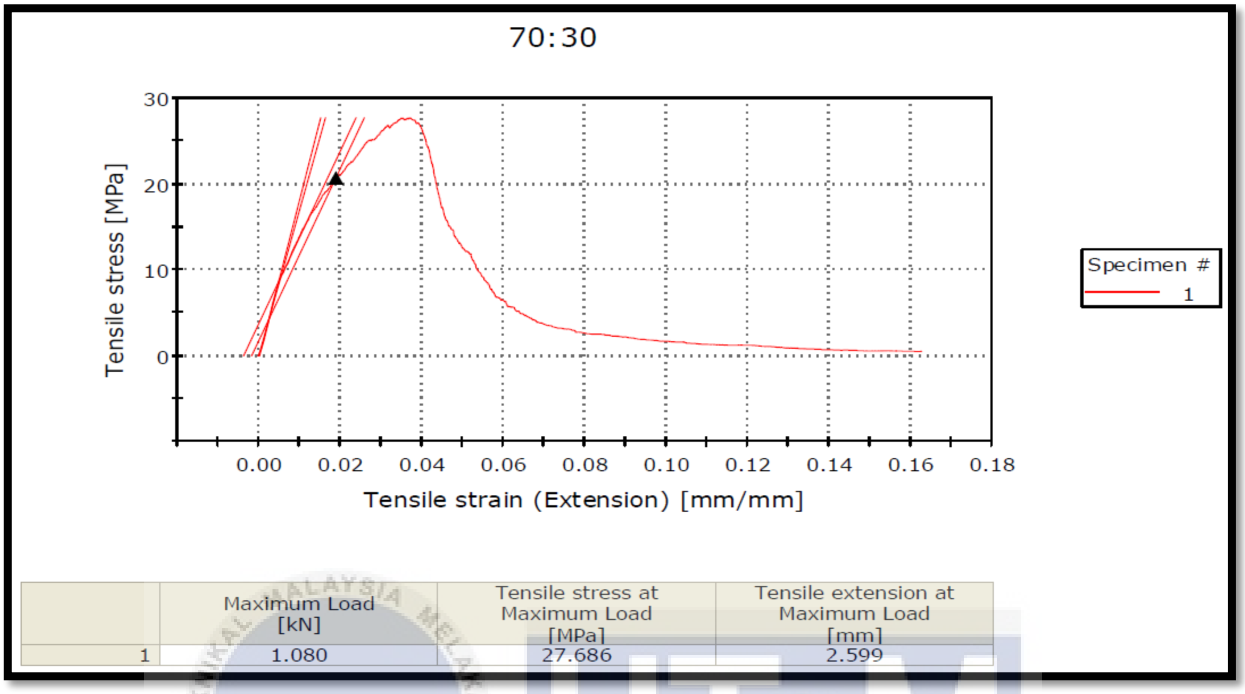
	Maximum Load [kN]	Tensile stress at Maximum Load [MPa]	Tensile extension at Maximum Load [mm]
1	0.480	12.295	1.177

Tensile Test on one sample of 50PLF/50SH



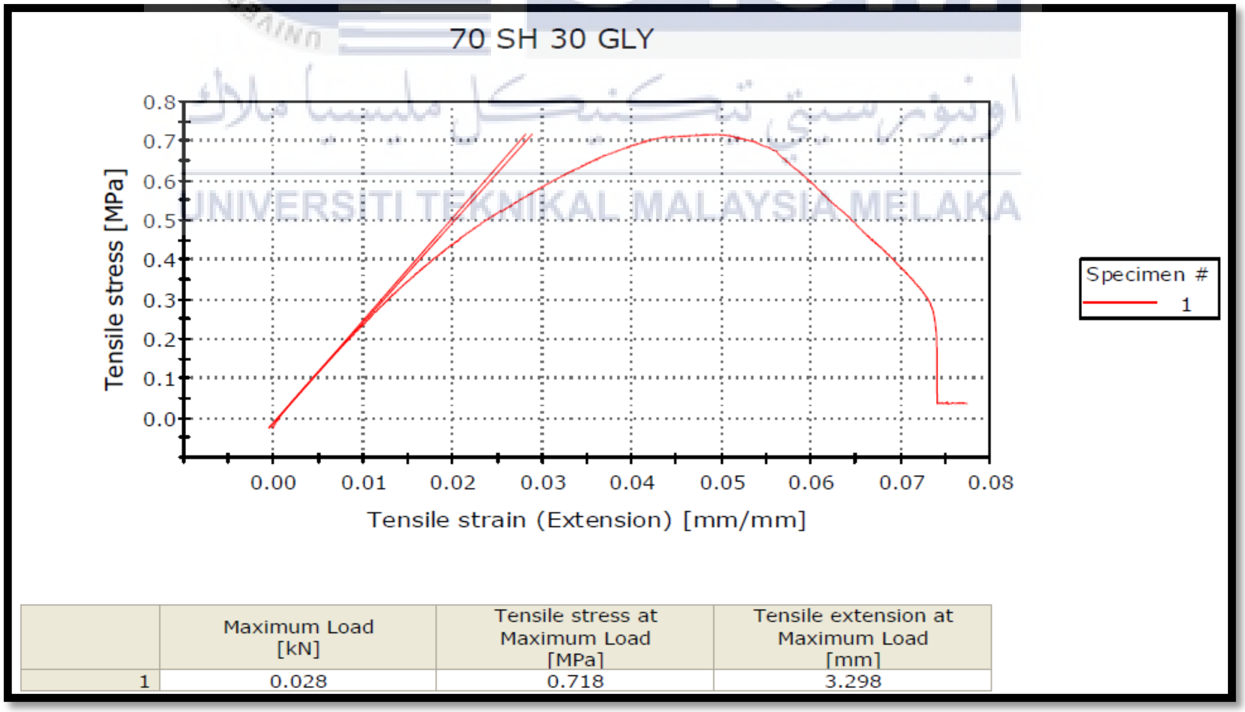
	Maximum Load [kN]	Tensile stress at Maximum Load [MPa]	Tensile extension at Maximum Load [mm]
1	0.408	10.468	2.793

Tensile Test on one sample of 60PLF/40SH



	Maximum Load [kN]	Tensile stress at Maximum Load [MPa]	Tensile extension at Maximum Load [mm]
1	1.080	27.686	2.599

Tensile Test on sample of 70PLF/30SH



	Maximum Load [kN]	Tensile stress at Maximum Load [MPa]	Tensile extension at Maximum Load [mm]
1	0.028	0.718	3.298

Tensile Test on one sample of 70SH/30GLY

GANTT CHART PSM I

NO.	TASK	WEEK													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	CHOOSE PSM TOPIC	■	■					░							
2	STUDY ABOUT NATURAL FIBRE & COMPOSITE			■	■	■	■	░							
3	PREPARATION ON PROGRESS REPORT					■	■	░	■	■	■	■	■	■	
4	CONSTRUCT & PREPARE SUBMISSION FINAL DRAFT OF THE PROJECT					■	■	░	■	■	■	■	■	■	
5	REPORT SUBMISSION							░			■	■	■		
6	PREPARATION FOR PSM 1 SEMINAR							░						■	■

GANTT CHART PSM II

NO.	TASK	WEEK													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	BRIEFING FOR PSM 2	■	■					░							
2	PREPARING FOR THE RAW MATERIALS		■	■	■	■	■	░							
3	FINDING PARAMETERS FOR SAMPLE BEST MATRIX COMPOSITIONS					■	■	░	■	■	■	■	■	■	
4	PREPARATION OFMIXING FIBRE WITH MATRIX FOR TESTING					■	■	░	■	■	■	■	■	■	
5	PREPARE FOR MORE SAMPLE IF THEY NOT IN SATISFACTORY CONDITION							░		■	■	■			
6	CONDUCT MECHANICAL TESTING							░					■	■	
7	REPORT WRITING AND PREPARE FOR PSM 2 SEMINAR													■	■

Designation: D 3039/D 3039M – 00$^{\epsilon 1}$

Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials[1]

This standard is issued under the fixed designation D 3039/D 3039M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ϵ^1 NOTE—Eq 5 was revised editorially in December 2002.

1. Scope

1.1 This test method determines the in-plane tensile properties of polymer matrix composite materials reinforced by high-modulus fibers. The composite material forms are limited to continuous fiber or discontinuous fiber-reinforced composites in which the laminate is balanced and symmetric with respect to the test direction.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[2]

D 883 Terminology Relating to Plastics[2]

D 2584 Test Method for Ignition Loss of Cured Reinforced Resins[3]

D 2734 Test Method for Void Content of Reinforced Plastics[3]

D 3171 Test Methods for Constituent Content of Composites Materials[4]

D 3878 Terminology for Composite Materials[4]

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[4]

E 4 Practices for Force Verification of Testing Machines[5]

E 6 Terminology Relating to Methods of Mechanical Testing[5]

E 83 Practice for Verification and Classification of Extensometers[5]

E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus[5]

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[6]

E 132 Test Method for Poisson's Ratio at Room Temperature[5]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[6]

E 251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages[5]

E 456 Terminology Relating to Quality and Statistics[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading[5]

E 1237 Guide for Installing Bonded Resistance Strain Gages[5]

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. Terminology E 6 defines terms relating to mechanical testing. Terminology E 456 and Practice E 177 define terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other standards.

3.2 *Definitions of Terms Specific to This Standard:*

NOTE—If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental

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[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

Current edition approved April 10, 2000. Published July 2000. Originally published as D 3039 – 71T. Last previous edition D 3039 – 95a.

[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 08.02.

[4] *Annual Book of ASTM Standards*, Vol 15.03.

[5] *Annual Book of ASTM Standards*, Vol 03.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

dimensions, shown within square brackets: [*M*] for mass, [*L*] for length, [*T*] for time, [Θ] for thermodynamic temperature, and [*nd*] for nondimensional quantities. Use of these symbols is restricted to analytical dimensions when used with square brackets, as the symbols may have other definitions when used without the brackets.

3.2.1 *nominal value*, *n*—a value, existing in name only, assigned to a measurable property for the purpose of convenient designation. Tolerances may be applied to a nominal value to define an acceptable range for the property.

3.2.2 *transition region*, *n*—a strain region of a stress-strain or strain-strain curve over which a significant change in the slope of the curve occurs within a small strain range.

3.2.3 *transition strain*, $\epsilon^{transition}$ *[nd]*, *n*—the strain value at the mid range of the transition region between the two essentially linear portions of a bilinear stress-strain or strain-strain curve.

3.2.3.1 *Discussion*—Many filamentary composite materials show essentially bilinear behavior during loading, such as seen in plots of either longitudinal stress versus longitudinal strain or transverse strain versus long longitudinal strain. There are varying physical reasons for the existence of a transition region. Common examples include: matrix cracking under tensile loading and ply delamination.

3.3 *Symbols:*

3.3.1 *A*—minimum cross-sectional area of a coupon.

3.3.2 B_y—percent bending for a uniaxial coupon of rectangular cross section about *y* axis of the specimen (about the narrow direction).

3.3.3 B_z—percent bending for a uniaxial coupon of rectangular cross section about *z* axis of the specimen (about the wide direction).

3.3.4 *CV*—coefficient of variation statistic of a sample population for a given property (in percent).

3.3.5 *E*—modulus of elasticity in the test direction.

3.3.6 F^{tu}—ultimate tensile strength in the test direction.

3.3.7 F^{su}—ultimate shear strength in the test direction.

3.3.8 *h*—coupon thickness.

3.3.9 L_g—extensometer gage length.

3.3.10 L_{min}—minimum required bonded tab length.

3.3.11 *n*—number of coupons per sample population.

3.3.12 *P*—load carried by test coupon.

3.3.13 P^f—load carried by test coupon at failure.

3.3.14 P^{max}—maximum load carried by test coupon before failure.

3.3.15 s_{n-1}—standard deviation statistic of a sample population for a given property.

3.3.16 *w*—coupon width.

3.3.17 x_i—test result for an individual coupon from the sample population for a given property.

3.3.18 $\bar{x}$—mean or average (estimate of mean) of a sample population for a given property.

3.3.19 δ—extensional displacement.

3.3.20 ϵ—general symbol for strain, whether normal strain or shear strain.

3.3.21 ϵ—indicated normal strain from strain transducer or extensometer.

3.3.22 σ—normal stress.

3.3.23 ν—Poisson's ratio.

4. Summary of Test Method

4.1 A thin flat strip of material having a constant rectangular cross section is mounted in the grips of a mechanical testing machine and monotonically loaded in tension while recording load. The ultimate strength of the material can be determined from the maximum load carried before failure. If the coupon strain is monitored with strain or displacement transducers then the stress-strain response of the material can be determined, from which the ultimate tensile strain, tensile modulus of elasticity, Poisson's ratio, and transition strain can be derived.

5. Significance and Use

5.1 This test method is designed to produce tensile property data for material specifications, research and development, quality assurance, and structural design and analysis. Factors that influence the tensile response and should therefore be reported include the following: material, methods of material preparation and lay-up, specimen stacking sequence, specimen preparation, specimen conditioning, environment of testing, specimen alignment and gripping, speed of testing, time at temperature, void content, and volume percent reinforcement. Properties, in the test direction, which may be obtained from this test method include the following:

5.1.1 Ultimate tensile strength,

5.1.2 Ultimate tensile strain,

5.1.3 Tensile chord modulus of elasticity,

5.1.4 Poisson's ratio, and

5.1.5 Transition strain.

6. Interferences

6.1 *Material and Specimen Preparation*—Poor material fabrication practices, lack of control of fiber alignment, and damage induced by improper coupon machining are known causes of high material data scatter in composites.

6.2 *Gripping*—A high percentage of grip-induced failures, especially when combined with high material data scatter, is an indicator of specimen gripping problems. Specimen gripping methods are discussed further in 7.2.4, 8.2, and 11.5.

6.3 *System Alignment*—Excessive bending will cause premature failure, as well as highly inaccurate modulus of elasticity determination. Every effort should be made to eliminate excess bending from the test system. Bending may occur as a result of misaligned grips or from specimens themselves if improperly installed in the grips or out-of-tolerance caused by poor specimen preparation. If there is any doubt as to the alignment inherent in a given test machine, then the alignment should be checked as discussed in 7.2.5.

6.4 *Edge Effects in Angle Ply Laminates*—Premature failure and lower stiffnesses are observed as a result of edge softening in laminates containing off-axis plies. Because of this, the strength and modulus for angle ply laminates can be drastically underestimated. For quasi-isotropic laminates containing significant 0° plies, the effect is not as significant.

7. Apparatus

micrometer with a 4- to 5-mm [0.16-
meter double-ball interface shall be

used to measure the thickness of the specimen. A micrometer with a flat anvil interface shall be used to measure the width of the specimen. The accuracy of the instruments shall be suitable for reading to within 1 % of the sample width and thickness. For typical specimen geometries, an instrument with an accuracy of ±2.5 μm [±0.0001 in.] is adequate for thickness measurement, while an instrument with an accuracy of ±25 μm [±0.001 in.] is adequate for width measurement.

7.2 *Testing Machine*—The testing machine shall be in conformance with Practices E 4 and shall satisfy the following requirements:

7.2.1 *Testing Machine Heads*—The testing machine shall have both an essentially stationary head and a movable head.

7.2.2 *Drive Mechanism*—The testing machine drive mechanism shall be capable of imparting to the movable head a controlled velocity with respect to the stationary head. The velocity of the movable head shall be capable of being regulated as specified in 11.3.

7.2.3 *Load Indicator*—The testing machine load-sensing device shall be capable of indicating the total load being carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the load with an accuracy over the load range(s) of interest of within ±1 % of the indicated value. The load range(s) of interest may be fairly low for modulus evaluation, much higher for strength evaluation, or both, as required.

NOTE 1—Obtaining precision load data over a large range of interest in the same test, such as when both elastic modulus and ultimate load are being determined, place extreme requirements on the load cell and its calibration. For some equipment, a special calibration may be required. For some combinations of material and load cell, simultaneous precision measurement of both elastic modulus and ultimate strength may not be possible and measurement of modulus and strength may have to be performed in separate tests using a different load cell range for each test.

7.2.4 *Grips*—Each head of the testing machine shall carry one grip for holding the test specimen so that the direction of load applied to the specimen is coincident with the longitudinal axis of the specimen. The grips shall apply sufficient lateral pressure to prevent slippage between the grip face and the coupon. If tabs are used the grips should be long enough that they overhang the beveled portion of the tab by approximately 10 to 15 mm [0.5 in.]. It is highly desirable to use grips that are rotationally self-aligning to minimize bending stresses in the coupon.

NOTE 2—Grip surfaces that are lightly serrated, approximately 1 serration/mm [25 serrations/in.], have been found satisfactory for use in wedge-action grips when kept clean and sharp; coarse serrations may produce grip-induced failures in untabbed coupons. Smooth gripping surfaces have been used successfully with either hydraulic grips or an emery cloth interface, or both.

7.2.5 *System Alignment*—Poor system alignment can be a major contributor to premature failure, to elastic property data scatter, or both. Practice E 1012 describes bending evaluation guidelines and describes potential sources of misalignment during tensile testing. In addition to Practice E 1012, the degree of bending in a tensile system can also be evaluated using the following related procedure. Specimen bending is considered separately in 11.6.1.

7.2.5.1 A rectangular alignment coupon, preferably similar in size and stiffness to the test specimen of interest, is instrumented with a minimum of three longitudinal strain gages of similar type, two on the front face across the width and one on the back face of the specimen, as shown in Fig. 1. Any difference in indicated strain between these gages during loading provides a measure of the amount of bending in the thickness plane (B_y) and width plane (B_z) of the coupon. The strain gage location should normally be located in the middle of the coupon gage section (if modulus determination is a concern), near a grip (if premature grip failures are a problem), or any combination of these areas.

7.2.5.2 When evaluating system alignment, it is advisable to perform the alignment check with the same coupon inserted in each of the four possible installation permutations (described relative to the initial position): initial (top-front facing observer), rotated back to front only (top back facing observer), rotated end for end only (bottom front facing observer), and rotated both front to back and end to end (bottom back facing observer). These four data sets provide an indication of whether the bending is due to the system itself or to tolerance in the alignment check coupon or gaging.

7.2.5.3 The zero strain point may be taken either before gripping or after gripping. The strain response of the alignment coupon is subsequently monitored during the gripping process, the tensile loading process, or both. Eq 1-3 use these indicated strains to calculate the ratio of the percentage of bending strain to average extensional strain for each bending plane of the alignment coupon and the total percent bending, B_{total}. Plotting percent bending versus axial average strain is useful in understanding trends in the bending behavior of the system.

7.2.5.4 Problems with failures during gripping would be reason to examine bending strains during the gripping process in the location near the grip. Concern over modulus data scatter would be reason to evaluate bending strains over the modulus evaluation load range for the typical transducer location. Excessive failures near the grips would be reason to evaluate bending strains near the grip at high loading levels. While the

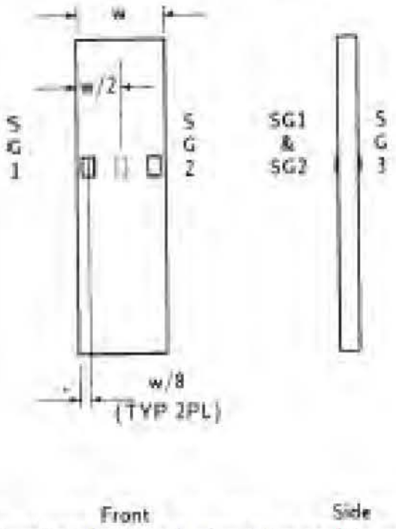

FIG. 1 Gage Locations for System Alignment Check Coupon

maximum advisable amount of system misalignment is material and location dependent, good testing practice is generally able to limit percent bending to a range of 3 to 5 % at moderate strain levels (>1000 με). A system showing excessive bending for the given application should be readjusted or modified.

$$B_y = \frac{\epsilon_{ave} - \epsilon_3}{\epsilon_{ave}} \times 100 \qquad (1)$$

$$B_z = \frac{4/3\,(\epsilon_2 - \epsilon_1)}{\epsilon_{ave}} \times 100 \qquad (2)$$

where:

B_y = percent bending about system y axis (about the narrow plane), as calculated by Eq 1, %;

B_z = percent bending about system z axis (about the wide plane), as calculated by Eq 2, %;

ϵ_1, ϵ_2, *and* ϵ_3 = indicated longitudinal strains displayed by Gages 1, 2, and 3, respectively, of Fig. 1, με; and

ϵ_{ave} = $(|\epsilon_1| + |\epsilon_2|)/2 + |\epsilon_3|/2$.

The total bending component is:

$$B_{total} = |B_y| + |B_z| \qquad (3)$$

7.3 *Strain-Indicating Device*—Load-strain data, if required, shall be determined by means of either a strain transducer or an extensometer. Attachment of the strain-indicating device to the coupon shall not cause damage to the specimen surface. If Poisson's ratio is to be determined, the specimen shall be instrumented to measure strain in both longitudinal and lateral directions. If the modulus of elasticity is to be determined, the longitudinal strain should be simultaneously measured on opposite faces of the specimen to allow for a correction as a result of any bending of the specimen (see 11.6 for further guidance).

7.3.1 *Bonded Resistance Strain Gage Selection*—Strain gage selection is a compromise based on the type of material. An active gage length of 6 mm [0.25 in.] is recommended for most materials. Active gage lengths should not be less than 3 mm [0.125 in.].[7] Gage calibration certification shall comply with Test Methods E 251. When testing woven fabric laminates, gage selection should consider the use of an active gage length that is at least as great as the characteristic repeating unit of the weave. Some guidelines on the use of strain gages on composites follow. A general reference on the subject is Tuttle and Brinson.[8]

7.3.1.1 Surface preparation of fiber-reinforced composites in accordance with Practice E 1237 can penetrate the matrix material and cause damage to the reinforcing fibers resulting in improper coupon failures. Reinforcing fibers should not be exposed or damaged during the surface preparation process. The strain gage manufacturer should be consulted regarding surface preparation guidelines and recommended bonding agents for composites pending the development of a set of standard practices for strain gage installation surface preparation of fiber-reinforced composite materials.

7.3.1.2 Consideration should be given to the selection of gages having larger resistances to reduce heating effects on low-conductivity materials. Resistances of 350 Ω or higher are preferred. Additional consideration should be given to the use of the minimum possible gage excitation voltage consistent with the desired accuracy (1 to 2 V is recommended) to reduce further the power consumed by the gage. Heating of the coupon by the gage may affect the performance of the material directly, or it may affect the indicated strain as a result of a difference between the gage temperature compensation factor and the coefficient of thermal expansion of the coupon material.

7.3.1.3 Consideration of some form of temperature compensation is recommended, even when testing at standard laboratory atmosphere. Temperature compensation is required when testing in nonambient temperature environments.

7.3.1.4 Consideration should be given to the transverse sensitivity of the selected strain gage. The strain gage manufacturer should be consulted for recommendations on transverse sensitivity corrections and effects on composites. This is particularly important for a transversely mounted gage used to determine Poisson's ratio, as discussed in Note 11.

7.3.2 *Extensometers*—For most purposes, the extensometer gage length should be in the range of 10 to 50 mm [0.5 to 2.0 in.]. Extensometers shall satisfy, at a minimum, Practice E 83, Class B-1 requirements for the strain range of interest and shall be calibrated over that strain range in accordance with Practice E 83. For extremely stiff materials, or for measurement of transverse strains, the fixed error allowed by Class B-1 extensometers may be significant, in which case Class A extensometers should be considered. The extensometer shall be essentially free of inertia lag at the specified speed of testing, and the weight of the extensometer should not induce bending strains greater than those allowed in 6.3.

NOTE 3—It is generally less difficult to perform strain calibration on extensometers of longer gage length as less precision in displacement is required of the extensometer calibration device.

7.4 *Conditioning Chamber*—When conditioning materials at nonlaboratory environments, a temperature/vaporlevel-controlled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to within ±3°C [±5°F] and the required relative vapor level to within ±3 %. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

7.5 *Environmental Test Chamber*—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the gage section of the test specimen at the required test environment during the mechanical test.

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[7] A typical gage would have a 0.25-in. active gage length, 350-Ω resistance, a strain rating of 3 % or better, and the appropriate environmental resistance and thermal coefficient.

[8] Tuttle, M. E. and Brinson, H. F., "Resistance-Foil Strain-Gage Technology as Applied to Composite Materials," *Experimental Mechanics*, Vol 24, No. 1, March 1984; pp. 54–65; errata noted in Vol 26, No. 2, June 1986, pp. 153–154.

8. Sampling and Test Specimens

8.1 *Sampling*—Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E 122 should be consulted. Report the method of sampling.

NOTE 4—If specimens are to undergo environmental conditioning to equilibrium, and are of such type or geometry that the weight change of the material cannot be properly measured by weighing the specimen itself (such as a tabbed mechanical coupon), then use another traveler coupon of the same nominal thickness and appropriate size (but without tabs) to determine when equilibrium has been reached for the specimens being conditioned.

8.2 *Geometry*—Design of mechanical test coupons, especially those using end tabs, remains to a large extent an art rather than a science, with no industry consensus on how to approach the engineering of the gripping interface. Each major composite testing laboratory has developed gripping methods for the specific material systems and environments commonly encountered within that laboratory. Comparison of these methods shows them to differ widely, making it extremely difficult to recommend a universally useful approach or set of approaches. Because of this difficulty, definition of the geometry of the test coupon is broken down into the following three levels, which are discussed further in each appropriate section:

Purpose	Degree of Geometry Definition
8.2.1 *General Requirements*	Mandatory Shape and Tolerances
8.2.2 *Specific Recommendations*	Nonmandatory Suggested Dimensions
8.2.3 *Detailed Examples*	Nonmandatory Typical Practices

8.2.1 *General Requirements:*

8.2.1.1 *Shape, Dimensions, and Tolerances*—The complete list of requirements for specimen shape, dimensions, and tolerances is shown in Table 1.

8.2.1.2 *Use of Tabs*—Tabs are not required. The key factor in the selection of specimen tolerances and gripping methods is the successful introduction of load into the specimen and the prevention of premature failure as a result of a significant discontinuity. Therefore, determine the need to use tabs, and specification of the major tab design parameters, by the end result: acceptable failure mode and location. If acceptable failure modes occur with reasonable frequency, then there is no reason to change a given gripping method (see 11.10).

TABLE 1 Tensile Specimen Geometry Requirements

Parameter	Requirement
Coupon Requirements:	
shape	constant rectangular cross-section
minimum length	gripping + 2 times width + gage length
specimen width	as needed^A
specimen width tolerance	±1 % of width
specimen thickness	as needed
specimen thickness tolerance	±4 % of thickness
specimen flatness	flat with light finger pressure
Tab Requirements (if used):	
tab material	as needed
fiber orientation (composite tabs)	as needed
tab thickness	as needed
tab thickness variation between tabs	±1 % tab thickness
tab bevel angle	5 to 90°, inclusive
tab step at bevel to specimen	feathered without damaging specimen

^{A} See 8.2.2 or Table 2 for recommendations.

8.2.2 *Specific Recommendations:*

8.2.2.1 *Width, Thickness, and Length*—Select the specimen width and thickness to promote failure in the gage section and assure that the specimen contains a sufficient number of fibers in the cross section to be statistically representative of the bulk material. The specimen length should normally be substantially longer than the minimum requirement to minimize bending stresses caused by minor grip eccentricities. Keep the gage section as far from the grips as reasonably possible and provide a significant amount of material under stress and therefore produce a more statistically significant result. The minimum requirements for specimen design shown in Table 1 are by themselves insufficient to create a properly dimensioned and toleranced coupon drawing. Therefore, recommendations on other important dimensions are provided for typical material configurations in Table 2. These geometries have been found by a number of testing laboratories to produce acceptable failure modes on a wide variety of material systems, but use of them does not guarantee success for every existing or future material system.

8.2.2.2 *Gripping/Use of Tabs*—There are many material configurations, such as multidirectional laminates, fabric-based materials, or randomly reinforced sheet-molding compounds, which can be successfully tested without tabs. However, tabs are strongly recommended when testing unidirectional materials (or strongly unidirectionally dominated laminates) to failure in the fiber direction. Tabs may also be required when testing unidirectional materials in the matrix direction to prevent gripping damage.

8.2.2.3 *Tab Geometry*—Recommendations on important dimensions are provided for typical material configurations in Table 2. These dimensions have been found by a number of testing laboratories to produce acceptable failure modes on a wide variety of material systems, but use of them does not guarantee success for every existing or future material system. The selection of a tab configuration that can successfully produce a gage section tensile failure is dependent upon the coupon material, coupon ply orientation, and the type of grips being used. When pressure-operated nonwedge grips are used with care, squared-off 90° tabs have been used successfully. Wedge-operated grips have been used most successfully with tabs having low bevel angles (7 to 10°) and a feathered smooth transition into the coupon. For alignment purposes, it is essential that the tabs be of matched thickness.

8.2.2.4 *Friction Tabs*—Tabs need not always be bonded to the material under test to be effective in introducing the load into the specimen. Friction tabs, essentially nonbonded tabs held in place by the pressure of the grip, and often used with emery cloth or some other light abrasive between the tab and the coupon, have been successfully used in some applications. In specific cases, lightly serrated wedge grips (see Note 2) have been successfully used with only emery cloth as the interface between the grip and the coupon. However, the abrasive used

TABLE 2 Tensile Specimen Geometry Recommendations[A]

Fiber Orientation	Width, mm [in.]	Overall Length, mm [in.]	Thickness, mm [in.]	Tab Length, mm [in.]	Tab Thickness, mm [in.]	Tab Bevel Angle,°
0° unidirectional	15 [0.5]	250 [10.0]	1.0 [0.040]	56 [2.25]	1.5 [0.062]	7 or 90
90° unidirectional	25 [1.0]	175 [7.0]	2.0 [0.080]	25 [1.0]	1.5 [0.062]	90
balanced and symmetric	25 [1.0]	250 [10.0]	2.5 [0.100]	emery cloth	—	—
random-discontinuous	25 [1.0]	250 [10.0]	2.5 [0.100]	emery cloth	—	—

[A] Dimensions in this table and the tolerances of Fig. 2 or Fig. 3 are recommendations only and may be varied so long as the requirements of Table 1 are met.

must be able to withstand significant compressive loads. Some types of emery cloth have been found ineffective in this application because of disintegration of the abrasive.[9]

8.2.2.5 *Tab Material*—The most consistently used bonded tab material has been continuous E-glass fiber-reinforced polymer matrix materials (woven or unwoven) in a [0/90]ns laminate configuration. The tab material is commonly applied at 45° to the loading direction to provide a soft interface. Other configurations that have reportedly been successfully used have incorporated steel tabs or tabs made of the same material as is being tested.

8.2.2.6 *Bonded Tab Length*—When using bonded tabs, estimate the minimum suggested tab length for bonded tabs by the following simple equation. As this equation does not account for the peaking stresses that are known to exist at the ends of bonded joints. The tab length calculated by this equation should normally be increased by some factor to reduce the chances of joint failure:

$$L_{min} = F^{tu}h/2F^{su} \tag{4}$$

where:

L_{min} = minimum required bonded tab length, mm [in.];
F^{tu} = ultimate tensile strength of coupon material, MPa [psi];
h = coupon thickness, mm [in.]; and
F^{su} = ultimate shear strength of adhesive, coupon material, or tab material (whichever is lowest), MPa [psi].

8.2.2.7 *Bonded Tab Adhesive*—Any high-elongation (tough) adhesive system that meets the environmental requirements may be used when bonding tabs to the material under test. A uniform bondline of minimum thickness is desirable to reduce undesirable stresses in the assembly.

8.2.3 *Detailed Examples*—The minimum requirements for specimen design discussed in 8.2.1 are by themselves insufficient to create a properly dimensioned and toleranced coupon drawing. Dimensionally toleranced specimen drawings for both tabbed and untabbed forms are shown as examples in Fig. 2 (SI) and Fig. 3 (inch-pound). The tolerances on these drawings are fixed, but satisfy the requirements of Table 1 for all of the recommended configurations of Table 2. For a specific configuration, the tolerances on Fig. 2 and Fig. 3 might be able to be relaxed.

8.3 *Specimen Preparation:*

8.3.1 *Panel Fabrication*—Control of fiber alignment is critical. Improper fiber alignment will reduce the measured properties. Erratic fiber alignment will also increase the coefficient of variation. The specimen preparation method shall be reported.

8.3.2 *Machining Methods*—Specimen preparation is extremely important for this specimen. Mold the specimens individually to avoid edge and cutting effects or cut from them plates. If they are cut from plates, take precautions to avoid notches, undercuts, rough or uneven surfaces, or delaminations caused by inappropriate machining methods. Obtain final dimensions by water-lubricated precision sawing, milling, or grinding. The use of diamond tooling has been found to be extremely effective for many material systems. Edges should be flat and parallel within the specified tolerances.

8.3.3 *Labeling*—Label the coupons so that they will be distinct from each other and traceable back to the raw material and in a manner that will both be unaffected by the test and not influence the test.

9. Calibration

9.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

10. Conditioning

10.1 *Standard Conditioning Procedure*—Unless a different environment is specified as part of the experiment, condition the test specimens in accordance with Procedure C of Test Method D 5229/D 5229M and store and test at standard laboratory atmosphere (23 ± 3°C [73 ± 5°F] and 50 ± 10 % relative humidity).

11. Procedure

11.1 *Parameters To Be Specified Before Test:*

11.1.1 The tension specimen sampling method, coupon type and geometry, and conditioning travelers (if required).

11.1.2 The tensile properties and data reporting format desired.

NOTE 5—Determine specific material property, accuracy, and data reporting requirements before test for proper selection of instrumentation and data-recording equipment. Estimate operating stress and strain levels to aid in transducer selection, calibration of equipment, and determination of equipment settings.

11.1.3 The environmental conditioning test parameters.

11.1.4 If performed, the sampling method, coupon geometry, and test parameters used to determine density and reinforcement volume.

11.2 *General Instructions:*

11.2.1 Report any deviations from this test method, whether intentional or inadvertent.

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[9] E-Z Flex Metalite K224 cloth, Grit 120-J, available from Norton Company, Troy, NY 12181, has been found satisfactory in this application. Other equivalent types of emery cloth should also be suitable.

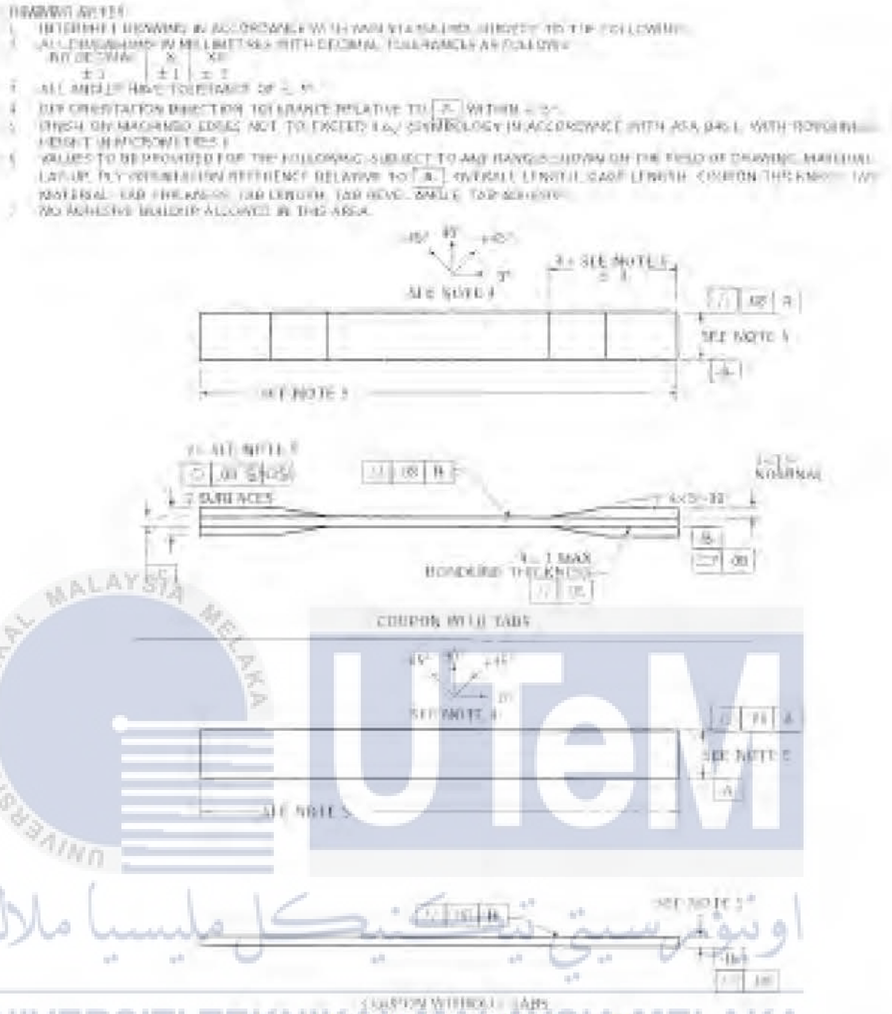


FIG. 2 Tension Test Specimen Drawing (SI)

11.2.2 If specific gravity, density, reinforcement volume, or void volume are to be reported, then obtain these samples from the same panels being tension tested. Specific gravity and density may be evaluated by means of Test Methods D 792. Volume percent of the constituents may be evaluated by one of the matrix digestion procedures of Test Method D 3171, or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method D 2584. The void content equations of Test Methods D 2734 are applicable to both Test Method D 2584 and the matrix digestion procedures.

11.2.3 Following final specimen machining and any conditioning, but before the tension testing, determine the specimen area as $A = w \times h$, at three places in the gage section, and report the area as the average of these three determinations to the accuracy in 7.1. Record the average area in units of mm^2 (in.^{2}).

11.3 *Speed of Testing*—Set the speed of testing to effect a nearly constant strain rate in the gage section. If strain control is not available on the testing machine, this may be approximated by repeated monitoring and adjusting of the rate of load application to maintain a nearly constant strain rate, as measured by strain transducer response versus time. The strain rate should be selected so as to produce failure within 1 to 10 min. If the ultimate strain of the material cannot be reasonably estimated, initial trials should be conducted using standard speeds until the ultimate strain of the material and the compliance of the system are known, and the strain rate can be adjusted. The suggested standard speeds are:

11.3.1 *Strain-Controlled Tests*—A standard strain rate of 0.01 min^{-1}.

11.3.2 *Constant Head-Speed Tests*—A standard head displacement rate of 2 mm/min [0.05 in./min].

NOTE 6—Use of a fixed head speed in testing machine systems with a high compliance may result in a strain rate that is much lower than required. Use of wedge grips can cause extreme compliance in the system, especially when using compliant tab materials. In some such cases, actual strain rates 10 to 50 times lower than estimated by head speeds have been observed.

11.4 *Test Environment*—Condition the specimen to the desired moisture profile and, if possible, test under the same conditioning fluid exposure level. However, cases such as

DRAWING NOTES:

1. INTERPRET DRAWING IN ACCORDANCE WITH ANSI Y14.5M-1982, SUBJECT TO THE FOLLOWING:
2. ALL DIMENSIONS IN INCHES WITH DECIMAL TOLERANCES AS FOLLOWS:

.X	.XX	.XXX
±.1	±.03	±.01

3. ALL ANGLES HAVE TOLERANCE OF ±.5°.
4. PLY ORIENTATION DIRECTION TOLERANCE RELATIVE TO -A- WITHIN ±.5°.
5. FINISH ON MACHINED EDGES NOT TO EXCEED 64√ (SYMBOLOGY IN ACCORDANCE WITH ASA B46.1, WITH ROUGHNESS HEIGHT IN MICROINCHES.)
6. VALUES TO BE PROVIDED FOR THE FOLLOWING, SUBJECT TO ANY RANGES SHOWN ON THE FIELD OF DRAWING: MATERIAL, LAY-UP, PLY ORIENTATION REFERENCE RELATIVE TO -A-, OVERALL LENGTH, GAGE LENGTH, COUPON THICKNESS, TAB MATERIAL, TAB THICKNESS, TAB LENGTH, TAB BEVEL ANGLE, TAB ADHESIVE.
7. NO ADHESIVE BUILDUP ALLOWED IN THIS AREA.

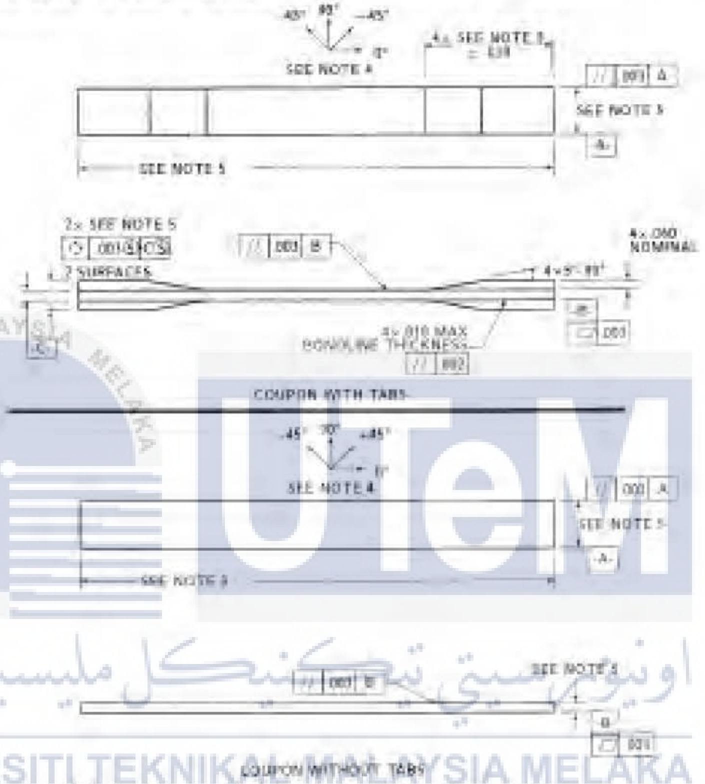


FIG. 3 Tension Test Specimen Drawing (inch-pound)

elevated temperature testing of a moist specimen place unrealistic requirements on the capabilities of common testing machine environmental chambers. In such cases, the mechanical test environment may need to be modified, for example, by testing at elevated temperature with no fluid exposure control, but with a specified limit on time to failure from withdrawal from the conditioning chamber. Modifications to the test environment shall be recorded.

11.4.1 Store the specimen in the conditioned environment until test time, if the testing area environment is different than the conditioning environment.

11.5 *Specimen Insertion*—Place the specimen in the grips of the testing machine, taking care to align the long axis of the gripped specimen with the test direction. Tighten the grips, recording the pressure used on pressure controllable (hydraulic or pneumatic) grips.

NOTE 7—The ends of the grip jaws on wedge-type grips should be even with each other following insertion to avoid inducing a bending moment that results in premature failure of the specimen at the grip. When using untabbed specimens, a folded strip of medium grade (80 to 150 grit) emery cloth between the specimen faces and the grip jaws (grit-side toward specimen) provides a nonslip grip on the specimen without jaw serration damage to the surface of the specimen. When using tabbed specimens, insert the coupon so that the grip jaws extend approximately 10 to 15 mm [0.5 in.] past the beginning of the tapered portion of the tab. Coupons having tabs that extend beyond the grips are prone to failure at the tab ends because of excessive interlaminar stresses.

11.6 *Transducer Installation*—If strain response is to be determined attach the strain-indication transducer(s) to the specimen, symmetrically about the mid-span, mid-width location. Attach the strain-recording instrumentation to the transducers on the specimen.

11.6.1 When determining modulus of elasticity, it is recommended that at least one specimen per like sample be evaluated with back-to-back axial transducers to evaluate the percent bending, using Eq 5, at the average axial strain checkpoint value (the mid range of the appropriate chord modulus strain range) shown in Table 3. A single transducer can be used if the

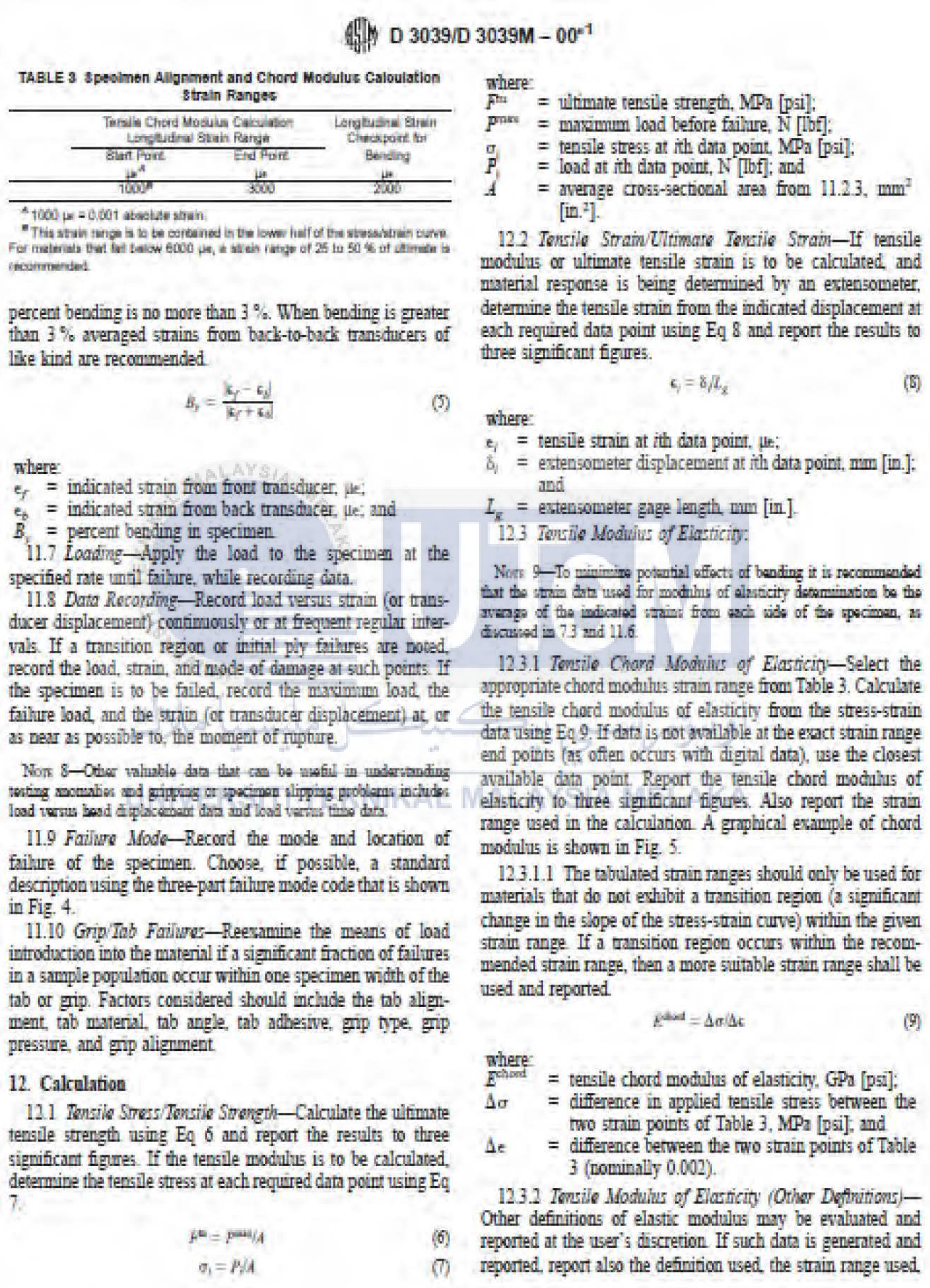


TABLE 3 Specimen Alignment and Chord Modulus Calculation Strain Ranges

Tensile Chord Modulus Calculation Longitudinal Strain Range		Longitudinal Strain Checkpoint for Bending
Start Point µε[A]	End Point µε	µε
1000[B]	3000	2000

[A] 1000 µε = 0.001 absolute strain.

[B] This strain range is to be contained in the lower half of the stress/strain curve. For materials that fail below 6000 µε, a strain range of 25 to 50 % of ultimate is recommended.

percent bending is no more than 3 %. When bending is greater than 3 % averaged strains from back-to-back transducers of like kind are recommended.

$$B_y = \frac{|\epsilon_f - \epsilon_b|}{|\epsilon_f + \epsilon_b|} \quad (5)$$

where:

e_f = indicated strain from front transducer, µε;

e_b = indicated strain from back transducer, µε; and

B_y = percent bending in specimen.

11.7 *Loading*—Apply the load to the specimen at the specified rate until failure, while recording data.

11.8 *Data Recording*—Record load versus strain (or transducer displacement) continuously or at frequent regular intervals. If a transition region or initial ply failures are noted, record the load, strain, and mode of damage at such points. If the specimen is to be failed, record the maximum load, the failure load, and the strain (or transducer displacement) at, or as near as possible to, the moment of rupture.

NOTE 8—Other valuable data that can be useful in understanding testing anomalies and gripping or specimen slipping problems includes load versus head displacement data and load versus time data.

11.9 *Failure Mode*—Record the mode and location of failure of the specimen. Choose, if possible, a standard description using the three-part failure mode code that is shown in Fig. 4.

11.10 *Grip/Tab Failures*—Reexamine the means of load introduction into the material if a significant fraction of failures in a sample population occur within one specimen width of the tab or grip. Factors considered should include the tab alignment, tab material, tab angle, tab adhesive, grip type, grip pressure, and grip alignment.

12. Calculation

12.1 *Tensile Stress/Tensile Strength*—Calculate the ultimate tensile strength using Eq 6 and report the results to three significant figures. If the tensile modulus is to be calculated, determine the tensile stress at each required data point using Eq 7.

$$F^{tu} = P^{max}/A \quad (6)$$

$$\sigma_i = P_i/A \quad (7)$$

where:

F^{tu} = ultimate tensile strength, MPa [psi];

P^{max} = maximum load before failure, N [lbf];

σ_i = tensile stress at *i*th data point, MPa [psi];

P_i = load at *i*th data point, N [lbf]; and

A = average cross-sectional area from 11.2.3, mm^2 [in.^{2}].

12.2 *Tensile Strain/Ultimate Tensile Strain*—If tensile modulus or ultimate tensile strain is to be calculated, and material response is being determined by an extensometer, determine the tensile strain from the indicated displacement at each required data point using Eq 8 and report the results to three significant figures.

$$\epsilon_i = \delta_i/L_g \quad (8)$$

where:

ϵ_i = tensile strain at *i*th data point, µε;

δ_i = extensometer displacement at *i*th data point, mm [in.]; and

L_g = extensometer gage length, mm [in.].

12.3 *Tensile Modulus of Elasticity*:

NOTE 9—To minimize potential effects of bending it is recommended that the strain data used for modulus of elasticity determination be the average of the indicated strains from each side of the specimen, as discussed in 7.3 and 11.6.

12.3.1 *Tensile Chord Modulus of Elasticity*—Select the appropriate chord modulus strain range from Table 3. Calculate the tensile chord modulus of elasticity from the stress-strain data using Eq 9. If data is not available at the exact strain range end points (as often occurs with digital data), use the closest available data point. Report the tensile chord modulus of elasticity to three significant figures. Also report the strain range used in the calculation. A graphical example of chord modulus is shown in Fig. 5.

12.3.1.1 The tabulated strain ranges should only be used for materials that do not exhibit a transition region (a significant change in the slope of the stress-strain curve) within the given strain range. If a transition region occurs within the recommended strain range, then a more suitable strain range shall be used and reported.

$$E^{chord} = \Delta\sigma/\Delta\epsilon \quad (9)$$

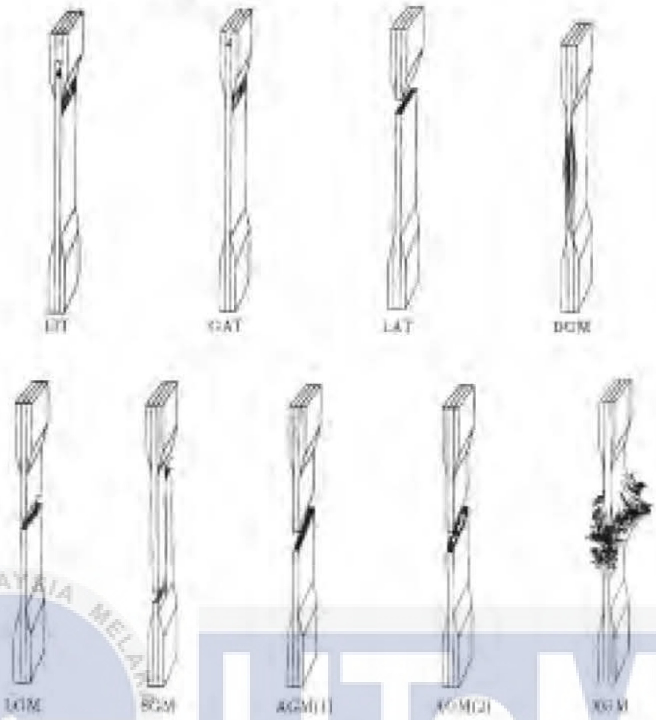
where:

E^{chord} = tensile chord modulus of elasticity, GPa [psi];

$\Delta\sigma$ = difference in applied tensile stress between the two strain points of Table 3, MPa [psi]; and

$\Delta\epsilon$ = difference between the two strain points of Table 3 (nominally 0.002).

12.3.2 *Tensile Modulus of Elasticity (Other Definitions)*—Other definitions of elastic modulus may be evaluated and reported at the user's discretion. If such data is generated and reported, report also the definition used, the strain range used,



First Character

Failure Type	Code
Angled	A
edge Delamination	D
Grip/tab	G
Lateral	L
Multi-mode	M(xyz)
long. Splitting	S
eXplosive	X
Other	O

Second Character

Failure Area	Code
Inside grip/tab	I
At grip/tab	A
<1W from grip/tab	W
Gage	G
Multiple areas	M
Various	V
Unknown	U

Third Character

Failure Location	Code
Bottom	B
Top	T
Left	L
Right	R
Middle	M
Various	V
Unknown	U

FIG. 4 Tensile Test Failure Codes/Typical Modes

and the results to three significant figures. Test Method E 111 provides additional guidance in the determination of modulus of elasticity.

NOTE 10—An example of another modulus definition is the secondary chord modulus of elasticity for materials that exhibit essentially bilinear stress-strain behavior. An example of secondary chord modulus is shown in Fig. 5.

12.4 *Poisson's Ratio:*

NOTE 11—If bonded resistance strain gages are being used, the error produced by the transverse sensitivity effect on the transverse gage will generally be much larger for composites than for metals. An accurate measurement of Poisson's ratio requires correction for this effect. The strain gage manufacturer should be contacted for information on the use of correction factors for transverse sensitivity.

12.4.1 *Poisson's Ratio By Chord Method*—Select the appropriate chord modulus longitudinal strain range from Table 3. Determine (by plotting or otherwise) the transverse strain (measured perpendicular to the applied load), ϵ_t, at each of the two longitudinal strains (measured parallel to the applied load), ϵ_l, strain range end points. If data is not available at the exact strain range end points (as often occurs with digital data), use

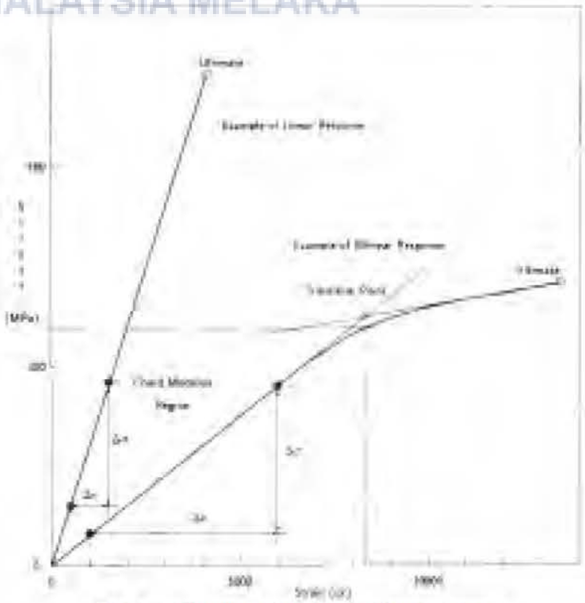


FIG. 5 Typical Tensile Stress-Strain Curves

the closest available data point. Calculate Poisson's ratio by Eq 10 and report to three significant figures. Also report the strain range used.

$$\nu = -\Delta\epsilon_t / \Delta\epsilon_l \qquad (10)$$

where:

ν = Poisson's ratio;

$\Delta\epsilon_t$ = difference in lateral strain between the two longitudinal strain points of Table 3, $\mu\epsilon$; and

$\Delta\epsilon_l$ = difference between the two longitudinal strain points of Table 3 (nominally either 0.001, 0.002, or 0.005).

12.4.2 *Tensile Poisson's Ratio (Other Definitions)*—Other definitions of Poisson's ratio may be evaluated and reported at the user's direction. If such data is generated and reported, report also the definition used, the strain range used, and the results to three significant figures. Test Method E 132 provides additional guidance in the determination of Poisson's ratio.

12.5 *Transition Strain*—Where applicable, determine the transition strain from either the bilinear longitudinal stress versus longitudinal strain curve or the bilinear transverse strain versus longitudinal strain curve. Create a best linear fit or chord line for each of the two linear regions and extend the lines until they intersect. Determine to three significant digits the longitudinal strain that corresponds to the intersection point and record this value as the transition strain. Report also the method of linear fit (if used) and the strain ranges over which the linear fit or chord lines were determined. A graphical example of transition strain is shown in Fig. 5.

12.6 *Statistics*—For each series of tests calculate the average value, standard deviation and coefficient of variation (in percent) for each property determined:

$$\bar{x} = \left(\sum_{i=1}^{n} x_i\right)/n \qquad (11)$$

$$s_{n-1} = \sqrt{\left(\sum_{i=1}^{n} x_i^2 - n\bar{x}^2\right)/(n-1)} \qquad (12)$$

$$CV = 100 \times s_{n-1}/\bar{x} \qquad (13)$$

where:

$\bar{x}$ = sample mean (average);

s_{n-1} = sample standard deviation;

CV = sample coefficient of variation, in percent;

n = number of specimens; and

x_i = measured or derived property.

13. Report

13.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable (reporting of items beyond the control of a given testing laboratory, such as might occur with material details or panel fabrication parameters, shall be the responsibility of the requestor):

13.1.1 The revision level or date of issue of this test method.

13.1.2 The date(s) and location(s) of the test.

13.1.3 The name(s) of the test operator(s).

13.1.4 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.

13.1.5 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch number, source (if not from manufacturer), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, prepreg matrix content, and prepreg volatiles content.

13.1.6 Description of the fabrication steps used to prepare the laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used.

13.1.7 Ply orientation stacking sequence of the laminate.

13.1.8 If requested, report density, volume percent reinforcement, and void content test methods, specimen sampling method and geometries, test parameters, and test results.

13.1.9 Average ply thickness of the material.

13.1.10 Results of any nondestructive evaluation tests.

13.1.11 Method of preparing the test specimen, including specimen labeling scheme and method, specimen geometry, sampling method, coupon cutting method, identification of tab geometry, tab material, and tab adhesive used.

13.1.12 Calibration dates and methods for all measurement and test equipment.

13.1.13 Type of test machine, grips, jaws, grip pressure, alignment results, and data acquisition sampling rate and equipment type.

13.1.14 Results of system alignment evaluations, if any such were done.

13.1.15 Dimensions of each test specimen.

13.1.16 Conditioning parameters and results, use of travelers and traveler geometry, and the procedure used if other than that specified in the test method.

13.1.17 Relative humidity and temperature of the testing laboratory.

13.1.18 Environment of the test machine environmental chamber (if used) and soak time at environment.

13.1.19 Number of specimens tested.

13.1.20 Speed of testing.

13.1.21 Transducer placement on the specimen and transducer type for each transducer used.

13.1.22 If strain gages were used, the type, resistance, size, gage factor, temperature compensation method, transverse sensitivity, lead-wire resistance, and any correction factors used.

13.1.23 Stress-strain curves and tabulated data of stress versus strain for each specimen.

13.1.24 Percent bending results for each specimen so evaluated.

13.1.25 Individual strengths and average value, standard deviation, and coefficient of variation (in percent) for the population. Note if the failure load was less than the maximum load before failure.

13.1.26 Individual strains at failure and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.27 Strain range used for chord modulus and Poisson's ratio determination.

13.1.28 If another definition of modulus of elasticity is used in addition to chord modulus, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

13.1.29 Individual values of modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.30 If another definition of Poisson's ratio is used in addition to the chordwise definition, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

13.1.31 Individual values of Poisson's ratio, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.32 If transition strain is determined, the method of linear fit (if used) and the strain ranges over which the linear fit or chord lines were determined.

13.1.33 Individual values of transition strain (if applicable), and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.34 Failure mode and location of failure for each specimen.

14. Precision and Bias

14.1 *Precision:*

14.1.1 The precision and bias of tension test strength and modulus measurements depend on strict adherence to the Test Method D 3039/D 3039M and are influenced by mechanical and material factors, specimen preparation, and measurement errors.

14.1.2 Mechanical factors that can affect the test results include: the physical characteristics of the testing machine (stiffness, damping, and mass), accuracy of loading and displacement/strain measurement, speed of loading, alignment of test specimen with applied load, parallelism of the grips, grip pressure, and type of load control (displacement, strain, or load).

14.1.3 Material factors that can affect test results include: material quality and representativeness, sampling scheme, and specimen preparation (dimensional accuracy, tab material, tab taper, tab adhesive, and so forth).

14.1.4 The mean tensile strength for a strain rate sensitive, glass/epoxy tape composite testing in the fiber direction was found to increase by approximately two standard deviations with decreasing time to failure tested at the limits of the recommended time to failure prescribed in Test Method D 3039/D 3039M. This result suggest that caution must be used when comparing test data obtained for strain rate sensitive composite materials tested in accordance with this standard.

14.1.5 Measurement errors arise from the use of specialized measuring instruments such as load cells, extensometers and strain gages, micrometers, data acquisition devices, and so forth.

14.1.6 Data obtained from specimens that fracture outside the gage are should be used with caution as this data may not be representative of the material. Failure in the grip region indicates the stress concentration at the tab is greater than the natural strength variation of the material in the gage section. A tapered tab, bonded with a ductile low-modulus adhesive has a relatively low-stress concentration and should result in the lowest frequency of grip failures. Low-strength bias increases with the frequency of grip failures by an amount proportional to the stress concentration at the tab.

14.1.7 An interlaboratory test program was conducted where an average of five specimens each, of six different materials and lay-up configurations, were tested by nine different laboratories.[10] Table 4 presents the precision statistics generated from this study as defined in Practice E 691 for tensile strength, modulus, and failure strain. All data except that for Material B (90° lay-up) was normalized with respect to an average thickness. The materials listed in Table 15 are defined as:

A	IM-6/3501–6 uni-tape (0)n
B	IM-6/3501–6 uni-tape (90)n
C	IM-6/3501–6 uni-tape (90/0)n
F	Glass/epoxy fabric (7781 glass/Ciba R 7376 Epoxy)-warp aligned
G	Carbon/epoxy fabric (66108 carbon/Ciba R 6376

TABLE 4 Precision Statistics

Material	$\bar{x}$	$s_{\bar{x}}$	S_r	S_R	$S_r/\bar{x}$, %	$S_R/\bar{x}$, %
			Strength, ksi			
A	342.69	8.49	10.68	12.78	3.12	3.73
B	8.52	0.52	0.85	0.92	9.94	10.84
C	156.37	3.84	10.85	10.85	6.94	6.94
F	66.18	3.20	1.52	3.48	2.30	5.26
G	121.62	1.59	3.92	3.92	3.23	3.23
			Modulus, Msi			
A	23.57	0.65	0.63	0.86	2.69	3.66
B	1.30	0.05	0.04	0.06	3.12	4.57
C	12.38	0.28	0.37	0.44	2.98	3.54
F	3.95	0.08	0.04	0.09	1.01	2.28
G	9.47	0.16	0.12	0.20	1.29	2.06
			Failure Strain, %			
A	1.38	0.06	0.07	0.08	4.95	6.15
B	0.68	0.04	0.08	0.09	12.47	13.02
C	1.22	0.03	0.06	0.06	5.25	5.27
F	2.04	0.15	0.07	0.16	3.19	8.03
G	1.27	0.03	0.05	0.05	3.83	4.13

14.1.8 The averages of the coefficients of variation are in Table 5. The values of S_r/X and S_R/X represent the repeatability and the reproducibility coefficients of variation, respectively. These averages permit a relative comparison of the repeatability (within laboratory precision) and reproducibility (between laboratory precision) of the tension test parameters. Overall, this indicates that the failure strain measurements exhibit the least repeatability and reproducibility of all the parameters measured while modulus was found to provide the highest repeatability and reproducibility of the parameters measured.

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[10] International Harmonization of Composite Materials—Phase 1: Harmonization of ASTM D 3039/D 3039M and ISO 527–5, Final Report, ASTM Institute for Standards Research, April 1997.

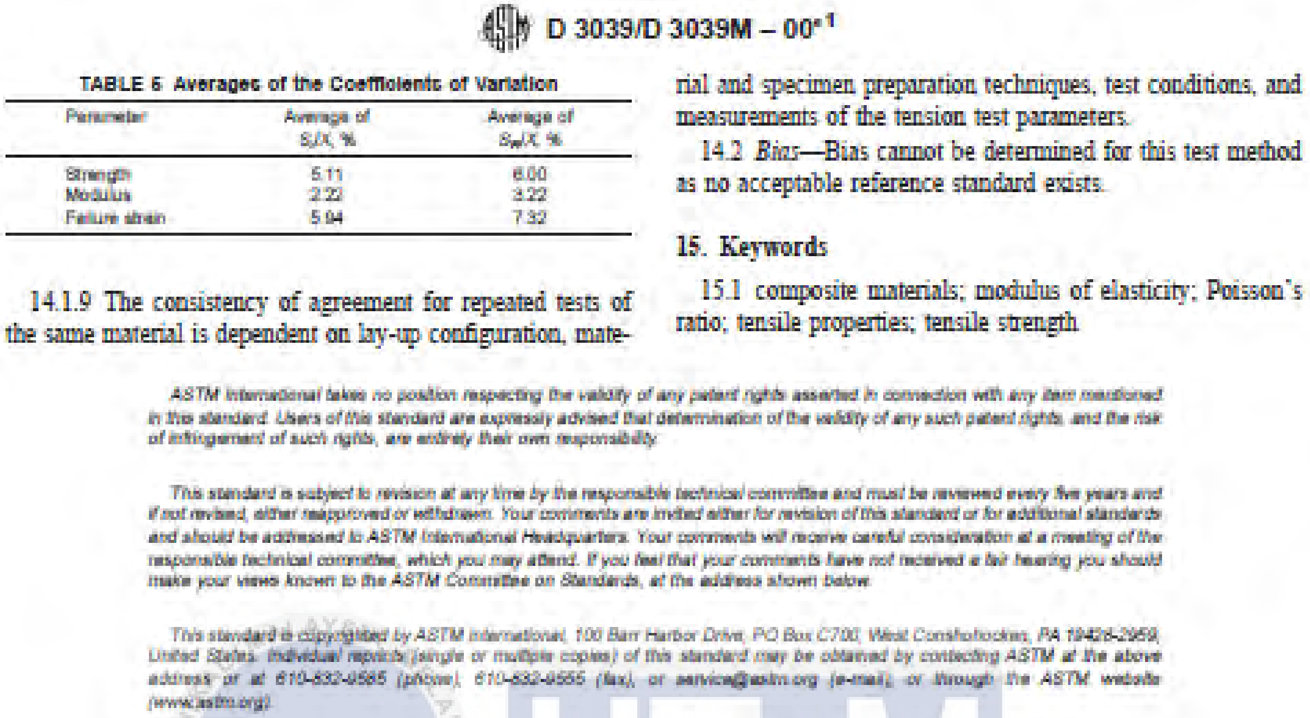

TABLE 6 Averages of the Coefficients of Variation

Parameter	Average of $S_r/\bar{X}$, %	Average of $S_R/\bar{X}$, %
Strength	5.11	6.00
Modulus	2.22	3.22
Failure strain	5.94	7.32

14.1.9 The consistency of agreement for repeated tests of the same material is dependent on lay-up configuration, material and specimen preparation techniques, test conditions, and measurements of the tension test parameters.

14.2 *Bias*—Bias cannot be determined for this test method as no acceptable reference standard exists.

15. Keywords

15.1 composite materials; modulus of elasticity; Poisson's ratio; tensile properties; tensile strength

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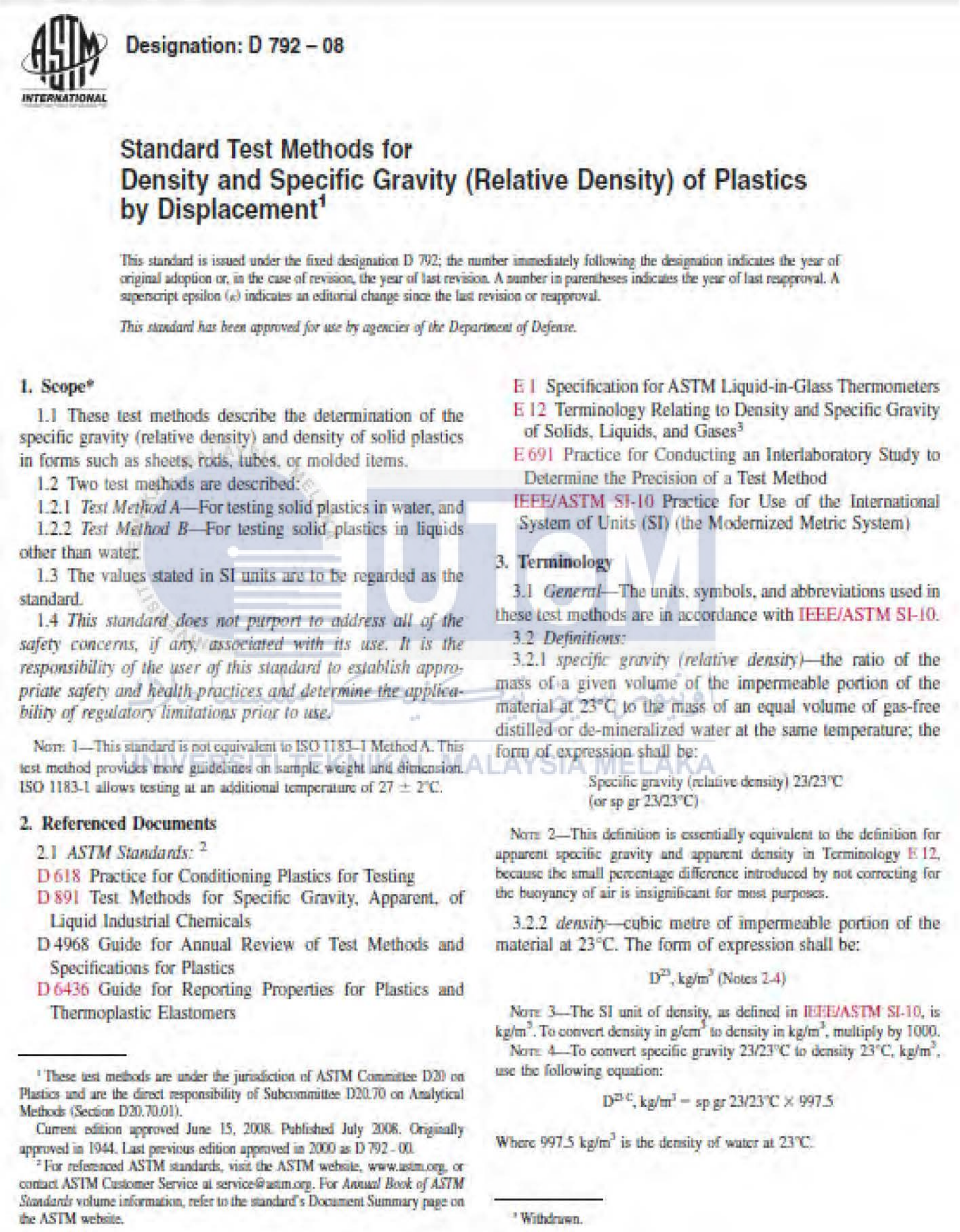

Designation: D 792 – 08

Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[1]

This standard is issued under the fixed designation D 792; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 These test methods describe the determination of the specific gravity (relative density) and density of solid plastics in forms such as sheets, rods, tubes, or molded items.

1.2 Two test methods are described:

1.2.1 *Test Method A*—For testing solid plastics in water, and

1.2.2 *Test Method B*—For testing solid plastics in liquids other than water.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—This standard is not equivalent to ISO 1183-1 Method A. This test method provides more guidelines on sample weight and dimension. ISO 1183-1 allows testing at an additional temperature of 27 ± 2°C.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 618 Practice for Conditioning Plastics for Testing

D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D 4968 Guide for Annual Review of Test Methods and Specifications for Plastics

D 6436 Guide for Reporting Properties for Plastics and Thermoplastic Elastomers

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI-10 Practice for Use of the International System of Units (SI) (the Modernized Metric System)

3. Terminology

3.1 *General*—The units, symbols, and abbreviations used in these test methods are in accordance with IEEE/ASTM SI-10.

3.2 *Definitions:*

3.2.1 *specific gravity (relative density)*—the ratio of the mass of a given volume of the impermeable portion of the material at 23°C to the mass of an equal volume of gas-free distilled or de-mineralized water at the same temperature; the form of expression shall be:

Specific gravity (relative density) 23/23°C
(or sp gr 23/23°C)

NOTE 2—This definition is essentially equivalent to the definition for apparent specific gravity and apparent density in Terminology E 12, because the small percentage difference introduced by not correcting for the buoyancy of air is insignificant for most purposes.

3.2.2 *density*—cubic metre of impermeable portion of the material at 23°C. The form of expression shall be:

$$D^{23}, \text{kg/m}^3 \text{ (Notes 2-4)}$$

NOTE 3—The SI unit of density, as defined in IEEE/ASTM SI-10, is kg/m^3. To convert density in g/cm^3 to density in kg/m^3, multiply by 1000.

NOTE 4—To convert specific gravity 23/23°C to density 23°C, kg/m^3, use the following equation:

$$D^{23C}, \text{kg/m}^3 = \text{sp gr } 23/23°\text{C} \times 997.5$$

Where 997.5 kg/m^3 is the density of water at 23°C.

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[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

Current edition approved June 15, 2008. Published July 2008. Originally approved in 1944. Last previous edition approved in 2000 as D 792 - 00.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

4. Summary of Test Method

4.1 Determine the mass of a specimen of the solid plastic in air. It is then immersed in a liquid, its apparent mass upon immersion is determined, and its specific gravity (relative density) calculated.

5. Significance and Use

5.1 The specific gravity or density of a solid is a property that is conveniently measured to identify a material, to follow physical changes in a sample, to indicate degree of uniformity among different sampling units or specimens, or to indicate the average density of a large item.

5.2 Changes in density of a single material are due to localized differences in crystallinity, loss of plasticizer, absorption of solvent, or to other causes. It is possible that portions of a sample differ in density because of their differences in crystallinity, thermal history, porosity, and composition (types or proportions of resin, plasticizer, pigment, or filler).

5.3 Density is useful for calculating strength-weight and cost-weight ratios.

6. Sampling

6.1 The sampling units used for the determination of specific gravity (relative density) shall be representative of the quantity of product for which the data are required.

6.1.1 If it is known or suspected that the sample consists of two or more layers or sections having different specific gravities, either complete finished parts or complete cross sections of the parts or shapes shall be used as the specimens, or separate specimens shall be taken and tested from each layer. The specific gravity (relative density) of the total part shall not be obtained by adding the specific gravity of the layers, unless relative percentages of the layers are taken into account.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2°C and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, unless otherwise specified by the contract or relevant material specifications. In cases of disagreement, the tolerances shall be ±1°C and ±2 % relative humidity.

7.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C and 50 ± 5 % relative humidity, unless otherwise specified in this specification or by the contract or relevant material specification. In cases of disagreement, the tolerances shall be ±1°C and ±2 % relative humidity.

TEST METHOD A FOR TESTING SOLID PLASTICS IN WATER (SPECIMENS 1 TO 50 g)

8. Scope

8.1 This test method involves weighing a one-piece specimen of 1 to 50 g in water, using a sinker with plastics that are lighter than water. This test method is suitable for plastics that are wet by, but otherwise not affected by water.

9. Apparatus

9.1 *Analytical Balance*—A balance with a precision of 0.1 mg or better is required for materials having densities less than 1.00 g/cm^3 and sample weights less than 10 grams. For all other materials and sample weights, a balance with precision of 1 mg or better is acceptable (see Note 5). The balance shall be equipped with a stationary support for the immersion vessel above the balance pan ("pan straddle").

NOTE 5—The balance shall provide the precision that all materials tested have three significant figures on density. In case that materials with different densities are tested on one single balance, use the balance that provides at least three significant figures for all materials concerned.

NOTE 6—To assure that the balance meets the performance requirements, check on zero point and sensitivity frequently and perform periodic calibration.

9.2 *Sample Holder*, corrosion-resistant (for example, wire, gemholder, etc.).

9.3 *Sinker*—A sinker for use with specimens of plastics that have specific gravities less than 1.00. The sinker shall: (*1*) be corrosion-resistant; (*2*) have a specific gravity of not less than 7.0; (*3*) have smooth surfaces and a regular shape; and (*4*) be slightly heavier than necessary to sink the specimen. The sinker shall have an opening to facilitate attachment to the specimen and sample holder.

9.4 *Immersion Vessel*—A beaker or other wide-mouthed vessel for holding the water and immersed specimen.

9.5 *Thermometer*—A thermometer readable to 0.1°C or better.

10. Materials

10.1 *Water*—The water shall be substantially air-free and distilled or de-mineralized water.

NOTE 7—Air in water can be removed by boiling and cooling the water, or by shaking the water under vacuum in a heavy-walled vacuum flask. (**Warning**—Use gloves and shielding.) If the water does not wet the specimen, add a few drops of a wetting agent into the water. If this solution does not wet the specimen, Method B shall be used.

11. Test Specimen

11.1 The test specimen shall be a single piece of material with a size and shape suitable for the testing apparatus, provided that its volume shall be not less than 1 cm^3 and its surface and edges shall be made smooth. The thickness of the specimen shall be at least 1 mm for each 1 g of weight. A specimen weighing 1 to 5 g was found to be convenient, but specimens up to approximately 50 g are also acceptable (see Note 8). Care shall be taken in cutting specimens to avoid changes in density resulting from compressive stresses or frictional heating.

NOTE 8—Specifications for certain plastics require a particular method of specimen preparation and should be consulted if applicable.

11.2 The specimen shall be free from oil, grease, and other foreign matter.

12. Procedure

12.1 Measure and record the water temperature.

12.2 Weigh the specimen in air. Weigh to the nearest 0.1 mg for specimens of mass 1 to 10 g and density less than 1.00 g/cm^3. Weigh to the nearest 1 mg for other specimens.

12.3 If necessary, attach to the balance a piece of fine wire sufficiently long to reach from the hook above the pan to the support for the immersion vessel. In this case attach the specimen to the wire such that it is suspended about 25 mm above the vessel support.

NOTE 9—If a wire is used, weigh the specimen in air after hanging from the wire. In this case, record the mass of the specimen, a = (mass of specimen + wire, in air) – (mass of wire in air).

12.4 Mount the immersion vessel on the support, and completely immerse the suspended specimen (and sinkers, if used) in water (see 10.1) at a temperature of 23 ± 2°C. The vessel must not touch sample holder or specimen. Remove any bubbles adhering to the specimen, sample holder, or sinker, by rubbing them with a wire. Pay particular attention to holes in the specimen and sinker. If the bubbles are not removed by this method or if bubbles are continuously formed (as from dissolved gases), the use of vacuum is recommended (see Note 10). Determine the mass of the suspended specimen to the required precision (see 12.2) (see Note 11). Record this apparent mass as b (the mass of the specimen, sinker, if used, and the partially immersed wire in liquid). Unless otherwise specified, weigh rapidly in order to minimize absorption of water by the specimen.

NOTE 10—Some specimens may contain absorbed or dissolved gases, or irregularities which tend to trap air bubbles; any of these may affect the density values obtained. In such cases, the immersed specimen may be subjected to vacuum in a separate vessel until evolution of bubbles has substantially ceased before weighing (see Test Method B). It must also be demonstrated that the use of this technique leads to results of the required degree of precision.

NOTE 11—It may be necessary to change the sensitivity adjustment of the balance to overcome the damping effect of the immersed specimen.

12.5 Weigh the sample holder (and sinker, if used) in water with immersion to the same depth as used in the previous step (Notes 12 and 13). Record this weight as w (mass of the sample holder in liquid).

NOTE 12—If a wire is used, it is convenient to mark the level of immersion by means of a shallow notch filed in the wire. The finer the wire, the greater the tolerance is permitted in adjusting the level of immersion between weighings. With wire Awg No. 36 or finer, disregard its degrees of immersion and, if no sinker is used, use the mass of the wire in air as w.

NOTE 13—If the wire is used and is left attached to the balance arm during a series of determinations, determine the mass a with the aid of a tare on the other arm of the balance or as in Note 9. In such cases, care must be taken that the change of mass of the wire (for example, from visible water) between readings does not exceed the desired precision.

12.6 Repeat the procedure for the required number of specimens. Two specimens per sample are recommended. Determine acceptability of number of replicate test specimens by comparing results with precision data given in Tables 1 and 2. Use additional specimens if desired.

13. Calculation

13.1 Calculate the specific gravity of the plastic as follows:

$$\text{sp gr } 23/23°\text{C} = a/(a + w - b)$$

where:

a = apparent mass of specimen, without wire or sinker, in air,

b = apparent mass of specimen (and of sinker, if used) completely immersed and of the wire partially immersed in liquid, and

w = apparent mass of totally immersed sinker (if used) and of partially immersed wire.

13.2 Calculate the density of the plastic as follows:

$$D^{23C}, \text{kg/m}^3 = \text{sp gr } 23/23°\text{C} \times 997.5$$

13.3 If the temperature of the water is different than 23°C, use the density of water listed in Table 3 directly, or use the following equations to calculate the density of water at testing temperature:

$$M = \Delta D/\Delta t \tag{1}$$

$$D(\text{conversion to } 23°\text{C}), \text{kg/m}^3 = \text{sp gr } t_a/t_w \times [997.5 + (t_w - 23) \times M] \tag{2}$$

and

$$\text{sp gr } 23/23 = D\ (\text{conversion to } 23°\text{C})/997.5 \tag{3}$$

where:

M = slope,

ΔD = difference between the lowest and highest temperature tolerance for the standard density of water (D @ 21°C – D @ 25°C),

Δt = difference between the highest and lowest temperature tolerance recommended, (21°C–25°C),

t_a = temperature of air, and

t_w = temperature of water.

14. Report

14.1 Report the following information:

TABLE 1 Test Method A Specific Gravity Tested in Water

Material	Mean	S_r[A]	S_R[B]	r[C]	R[D]
Polypropylene	0.9007	0.00196	0.00297	0.00555	0.00841
Cellulose Acetate Butyrate	1.1973	0.00232	0.00304	0.00657	0.00860
Polyphenylene Sulfide	1.1708	0.00540	0.00738	0.01528	0.02089
Thermoset	1.3136	0.00271	0.00313	0.00767	0.02171
Polyvinyl Chloride	1.3396	0.00243	0.00615	0.00688	0.01947

[A] S_r = within laboratory standard deviation for the individual material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories:

$$S_r = [[(s_1)^2 + (s_2)^2 \ldots + (s_n)^2]/n]^{1/2}$$

[B] S_R = between-laboratories reproducibility, expressed as standard deviation: $S_R = [S_r^2 + S_L^2]^{1/2}$ where S_L is the standard deviation of laboratory means.

[C] r = within-laboratory critical interval between two test results = 2.8 × S_r.

[D] R = between-laboratories critical interval between two test results = 2.8 × S_R.

TABLE 2 Test Method B Specific Gravity Tested in Liquids Other Than Water

Material	Mean	S_r[A]	S_R[B]	r[C]	R[D]
Polypropylene	0.9023	0.00139	0.00239	0.00393	0.00669
LDPE	0.9215	0.00109	0.00195	0.00308	0.00546
HDPE	0.9678	0.00126	0.00189	0.00356	0.00529
Thermoset	1.3130	0.00160	0.00217	0.00453	0.00608

[A] S_r = within laboratory standard deviation for the individual material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories:

$$S_r = [[(s_1)^2 + (s_2)^2 \ldots + (s_n)^2]/n]^{1/2}$$

[B] S_R = between-laboratories reproducibility, expressed as standard deviation: $S_R = [S_r^2 + S_L^2]^{1/2}$ where S_L is the standard deviation of laboratory means.

[C] r = within-laboratory critical interval between two test results = 2.8 × S_r.

[D] R = between-laboratories critical interval between two test results = 2.8 × S_R.

TABLE 3 Standard Density of Water[A]

°C	ρ/kg m^{-3}									
	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
21	997.9948	9731	9513	9294	9073	8852	8630	8406	8182	7957
22	997.7730	7503	7275	7045	6815	6584	6351	6118	5883	5648
23	997.5412	5174	4936	4697	4456	4215	3973	3730	3485	3240
24	997.2994	2747	2499	2250	2000	1749	1497	1244	0990	0735
25	997.0480	0223	9965[B]	9707[B]	9447[B]	9186[B]	8925[B]	8663[B]	8399[B]	8135[B]

[A] Obtained from *CRC Handbook of Chemistry and Physics*, 78th edition, 1997-1998.

[B] The leading figure decreases by 1.

14.1.1 Complete identification of the material or product tested, including method of specimen preparation and conditioning,

14.1.2 Average specific gravity (relative density) for all specimens from a sampling unit corrected to 23.0°C (Table 3) are reported as sp gr 23/23°C = ___, or average density reported as D^{23C} = ___ kg/m^3,

NOTE 14—Reporting density in g/cm^3 is also acceptable provided that it is agreed upon by the users.

14.1.3 A measure of the degree of variation of specific gravity or density within the sampling unit such as the standard deviation and number of determinations on a homogeneous material or the averages plus these measures of dispersion on different layers or areas of a nonhomogeneous product,

14.1.4 Report the temperature of the water.

14.1.5 Report the density and specific gravity with three significant figures.

14.1.6 Any evidence of porosity of the material or specimen,

14.1.7 The method of test (that is, Method A of Test Method D 792), and

14.1.8 Date of test.

15. Precision and Bias

15.1 See Section 23.

TEST METHOD B FOR TESTING SOLID PLASTICS IN LIQUIDS OTHER THAN WATER (SPECIMENS 1 TO 50 g)

16. Scope

16.1 Test Method B uses a liquid other than water for testing one-piece specimens, 1 to 50 g, of plastics that are affected by water or are lighter than water.

17. Apparatus

17.1 The apparatus shall include the balance, wire, and immersion vessel of Section 8, and, optionally, the following:

17.2 *Pycnometer with Thermometer*—A 25-mL specific gravity bottle with thermometer, or

17.3 *Pycnometer*—A pycnometer of the Weld type, preferably with a capacity of about 25 mL and an external cap over the stopper.

17.4 *Thermometer*—A thermometer having ten divisions per degree Celsius over a temperature range of not less than 5°C or 10°F above and below the standard temperature, and having an ice point for calibration. A thermometer short enough to be handled inside the balance case will be found convenient. ASTM Thermometer 23C (see Specification E 1) and Anschütz-type thermometers have been found satisfactory for this purpose.

17.5 *Constant-Temperature Bath*—An appropriate constant-temperature bath adjusted to maintain a temperature of 23 ± 0.1°C.

18. Materials

18.1 *Immersion Liquid*—The liquid used shall not dissolve, swell, or otherwise affect the specimen, but shall wet it and shall have a specific gravity less than that of the specimen. In addition, the immersion liquid shall be non-hygroscopic, has a low vapor pressure, a low viscosity, and a high flash point, and shall leave little or no waxy or tarry residue on evaporation. A narrow cut distilled from kerosine meets these requirements for many plastics. The specific gravity 23/23°C of the immersion liquid shall be determined shortly before and after each use in this method to a precision of at least 0.1 % relative, unless it has been established experimentally in the particular application that a lesser frequency of determination also provides the desired precision.

NOTE 15—For the determination of the specific gravity of the liquid, the use of a standard plummet of known volume or of Method A, C, or D of Test Methods D 891, using the modifications required to give specific gravity 23/23°C instead of specific gravity 60/60°F, is recommended. One suggested procedure is the following:

If a constant-temperature water bath is not available, determine the mass of the clean, dry pycnometer with thermometer to the nearest 0.1 mg on an analytical balance. Fill the pycnometer with water (10.1) cooler than 23°C. Insert the thermometer-stopper, causing excess water to be expelled through the side arm. Permit the filled bottle to warm in air until the thermometer reads 23.0°C. Remove the drop of water at the tip of the side arm with a bit of filter paper, taking care not to draw any liquid from within the capillary, place the cap over the side arm, wipe the outside carefully, and determine the mass of the filled bottle again to the nearest 0.2 mg. Empty the pycnometer, dry, and fill with immersion liquid. Determine the mass with the liquid in the same manner as was done with the water. Calculate the specific gravity 23/23°C of the liquid, d, as follows:

$$d = (b - e)/(w - e)$$

where:

e = apparent mass of empty pycnometer,
w = apparent mass of pycnometer filled with water at 23.0°C, and
b = apparent mass of pycnometer filled with liquid at 23.0°C.

If a constant-temperature water bath is available, a pycnometer without a thermometer may be used (compare 30.2).

NOTE 16—One standard object which has been found satisfactory for this purpose is the Reimann Thermometer Plummet. These are normally supplied calibrated for measurements at temperatures other than 23/23°C, so that recalibration is not necessary for the purposes of these methods.

19. Test Specimen

19.1 See Section 11.

20. Procedure

20.1 The procedure shall be similar to Section 12, except for the choice of immersion liquid, and the temperature during the immersed weighing (12.3) shall be 23 ± 0.5°C.

21. Calculation

21.1 The calculations shall be similar to Section 13, except that d, the specific gravity 23/23°C of the liquid, shall be placed in the numerator: (see 13.1)

$$Sp\,gr\,23/23°C = (a \times d)/(a + w - b)$$

22. Report

22.1 See Section 14.

23. Precision and Bias

23.1 Tables 1 and 2 are based on an interlaboratory study[4] conducted in 1985 in accordance with Practice E 691, involving 5 materials tested with Test Method A by six laboratories or four materials tested with Test Method B by six laboratories. Each test result was based on two individual determinations and each laboratory obtained four test results for each material. (**Warning—**The explanations of r and R are only intended to present a meaningful way of considering the approximate precision of these test methods. The data of Tables 1 and 2 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to the materials and laboratory (or between specific laboratories). The principles of 23.2-23.2.3 would then be valid for such data.)

23.2 *Concept of* r *and* R *in Tables 1 and 2*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from 4 test results for each material, then:

23.2.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the r value for that material. The concept r is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

23.2.2 *Reproducibility*—Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the R value for that material. The concept R is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

23.2.3 Any judgment in accordance with 23.2.1 or 23.2.2 would have an approximate 95 % (0.95) probability of being correct.

23.3 There are no recognized standards by which to estimate bias of this test method.

24. Keywords

24.1 density; relative density; specific gravity

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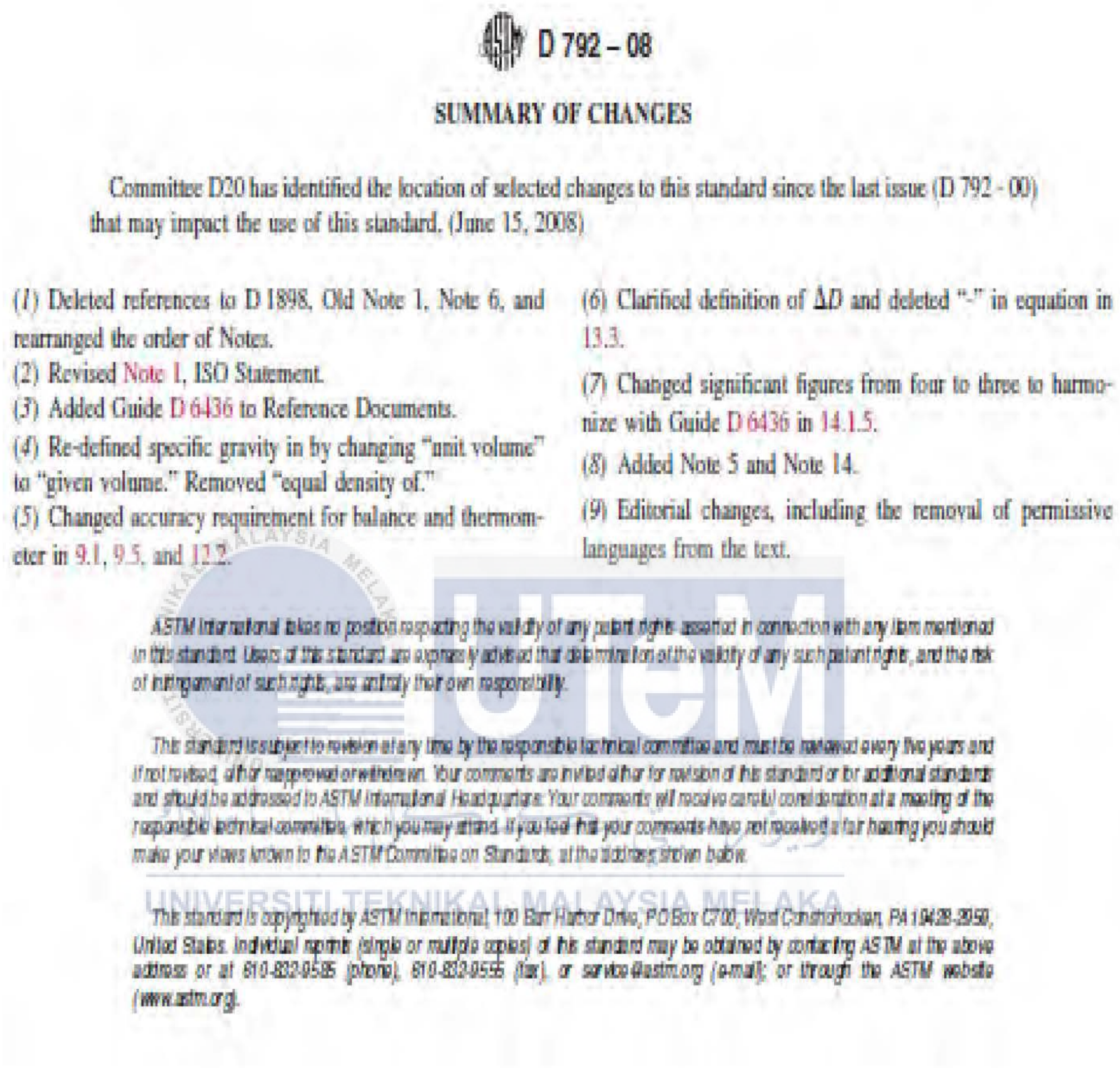
[4] Supporting data are available from ASTM Headquarters. Request RR:D20-1133.



SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D 792 - 00) that may impact the use of this standard. (June 15, 2008)

(*1*) Deleted references to D 1898, Old Note 1, Note 6, and rearranged the order of Notes.
(2) Revised Note 1, ISO Statement.
(*3*) Added Guide D 6436 to Reference Documents.
(*4*) Re-defined specific gravity in by changing "unit volume" to "given volume." Removed "equal density of."
(*5*) Changed accuracy requirement for balance and thermometer in 9.1, 9.5, and 12.2.
(6) Clarified definition of ΔD and deleted "-" in equation in 13.3.
(*7*) Changed significant figures from four to three to harmonize with Guide D 6436 in 14.1.5.
(*8*) Added Note 5 and Note 14.
(9) Editorial changes, including the removal of permissive languages from the text.

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