

**UNIVERSITY OF TURKISH AERONAUTICAL ASSOCIATION
INSTITUTE OF SCIENCE AND TECHNOLOGY**

**THE EFFECT OF RECYCLING NATURAL WASTE ON THE
MECHANICAL AND PHYSICAL PROPERTIES FOR POLYMER
MATRIX REINFORCED WITH FIBERS**



MASTER THESIS

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**MECHANICAL AND AERONAUTICAL ENGINEERING
DEPARTMENT**

MASTER THESIS PROGRAM

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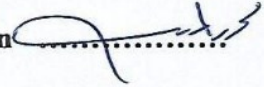
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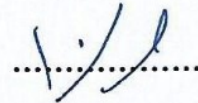
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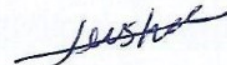
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I hereby declare that all the information in this study I presented as my Master's Thesis, called "THE EFFECT OF RECYCLING NATURAL WASTE ON THE MECHANICAL AND PHYSICAL PROPERTIES FOR POLYMER MATRIX REINFORCED WITH FIBERS" has been presented in accordance with the academic rules and ethical conduct. I also declare and certify on my honor that I have fully cited and referenced all the sources I made use of in this present study.



Rasha JWAID

21.12.2017

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LIST OF ABBREVIATIONS

FRP	:Fiber Reinforced Plastic
PMC	:Polymer Matrix Composite
RH	:Rice Husk
σ	:Tensile Strength
δ	:Deflection of the specimen
P	:Load
A	:Cross sectional Area
L	:Span length
E	:Young's modulus
UTM	:Ultimate testing machine
FS	:Flexural Strength
t	:Thickness of specimen
G	:Flexural Modulus
H	:Hardness of the composite
ρ	:Density of the composite
EB	:Flexural Modulus
V_f, V_p & V_m	:Volume Fraction of Fiber, Particle & Matrix
v_f, v_m & v_c	:Volume of Fiber, Matrix & Composite
L₀	: Original Length of Specimen
τ_{max}	:Maximum Shear Stress

ABSTRACT

THE EFFECT OF RECYCLING NATURAL WASTE ON THE MECHANICAL AND PHYSICAL PROPERTIES FOR POLYMER MATRIX REINFORCED WITH FIBERS

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M.Sc., Mechanical Engineering Department

Supervisor: Assist. Prof. Dr. Ibrahim MAHARIQ

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This work focuses on the preparation of base polymer matrix composite materials by Hand Lay-Up method, and studying the effect of selected weight fractions of adding natural powders before and after burning to epoxy resin matrix with small weight fractions glass fiber. Also, several specific tests were performed on these composites and the effects on the physical and mechanical properties were studied. The effects of the weight fraction on the mechanical properties were studied.

The maximum values for the physical properties obtained in this research were density and water absorption percentage, also thermal conductivity and thermal diffusivity were measured. The largest values of the mechanical properties obtained in this research were the modulus of elasticity, ultimate tensile strength and elongation percentage at the break for the natural composite materials. Flexural strength, flexural modulus, maximum shear stress, and Hardness shore (D) of the prepared natural composite materials increase with increasing weight fraction of filler powder, reaching a maximum value at (9% weight fraction Rice husk ash). Also the impact strength decrease with an increase in the weight fraction of the prepared system.

Keywords: Composite materials, Epoxy, Eggshells powder, Glass fibers, mechanical properties, physical properties, Rice husk powder, weight fractions.

ÖZET

DOĞAL ATIKLARIN GERİ DÖNÜŞÜMÜNÜN ELYAFLARLA GÜÇLENDİRİLMİŞ POLİMER MATRİSİN MEKANİK VE FİZİKSEL ÖZELLİKLERİNE ETKİSİ

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Tez Danışmanı: Yrd. Doç. Dr. İbrahim MAHARİQ

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Bu çalışma, El Katlama metodu ile baz polimer matris kompozit materyallerin hazırlanmasına ve düşük viskoziteli cam elyafı ile epoksi reçine matrisi yanmadan önce ve sonra doğal tozların eklenmesinin, seçilen ağırlık paylarının etkisini incelemeye odaklanmaktadır. Ayrıca, bu kompozitler üzerinde birkaç özel test yapılarak ve fiziksel ve mekanik özelliklere etkileri incelenmiştir. Ağırlık fraksiyonunun mekanik özelliklere etkileri incelendi.

Bu araştırmada elde edilen fiziksel özellikler için maksimum değerler yoğunluk ve su soğurma oranı, ayrıca ısı iletkenliği ve termal difüzyivite ölçülmüştür. Bu araştırmada elde edilen en büyük mekanik özellikler, elastikiyet modülü, nihai gerilme mukavemeti ve doğal bileşik materyallerin kopma uzama yüzdeleri idi. Hazırlanan doğal kompozit malzemelerin eğilme mukavemeti, eğilme modülü, maksimum kayma gerilmesi ve sertlik kıyası (D) dolgu tozunun ağırlık payının artması ile artmakta ve (% 9 ağırlık fraksiyonunda pirinç kabuğu külü) maksimum bir değere ulaşmaktadır. Ayrıca, darbe mukavemeti, hazırlanan sistemin ağırlık bölümünde bir artış ile azalır.

Anahtar Kelimeler: Kompozit malzemeler, epoksi, yumurta kabuğu tozu, cam lifleri, mekanik özellikler, fiziksel özellikler, pirinç kabuğu tozu, ağırlık payları.

CHAPTER ONE

INTRODUCTION

1.1 Introduction

Composite material can be distinct as a combination of two or more materials to obtain a substance that has better mechanical and physical properties of those individual components are used alone. On the other hand the metal alloys, each material retain chemical, physical and mechanical characteristics separately. The composite materials have the main advantages of stiffness and high strength intergraded with a low density material comparison to matrix materials [1]. The reinforcement materials it can usually be fibers or particles and also these composite have offers high strength with harder than matrix material. Particles of composite materials have dimensions which almost same in the directions. These particles can be platelets, spherical with irregular or regular shape. The composite material reinforced with the particles tends to be much weaker and less stiff than the composite materials reinforced with continuous fibers, but are usually the least expensive.

Typically, Composites reinforced with particulate include less strengthening because of weakness and difficulty of processing [2]. The polymer matrix composite is formed by the combination of a polymer (resin) matrix and a fibrous reinforcing phase. Polymer composites are gaining importance substitute the metals in many applications for automotive, sporting goods ,aerospace, marine, and electronics industries due to their light weight and corrosion resistant.

Polymer matrix can be classified as thermoplastics and thermoset. Thermoplastics include low density polyethylene, high density polyethylene, nylon and polypropylene while polyester and epoxy resin is an example of a thermoset [3].

Epoxy resin is a widely used thermoset material in polymeric composites. It also possesses good bond strength with the materials, good chemical resistance, and good insulation properties. Epoxy resin is also used in surface coating due to its chemical

resistance, adhesion, elasticity, and durability [4]. Because polymer composite materials reinforced with fiber have better mechanical and physical properties than conventional metallic materials there are suitable for tribological application. The different application areas are cams, gears, impellers, wheels, brakes, artificial seals, prosthetic joints, bushes, bearings [3].

Agricultural waste, which includes, for example (rice husks, wheat husks, fruit husks, hemp fiber) all these wastes and others, can be used as reinforcing materials for the preparation of polymer composite for various commercial and industrial uses. Polymers are combined with various materials to reduce costs and tailor the product to specific application [5].

In this research will be prepared composite material from epoxy (resin) with fiber glass and natural powders can be obtained from recycle of existing waste in natural. This composite polymer materials are used in different applications, such as airplanes body, pipes, car body as shown in the Figure 1.1.

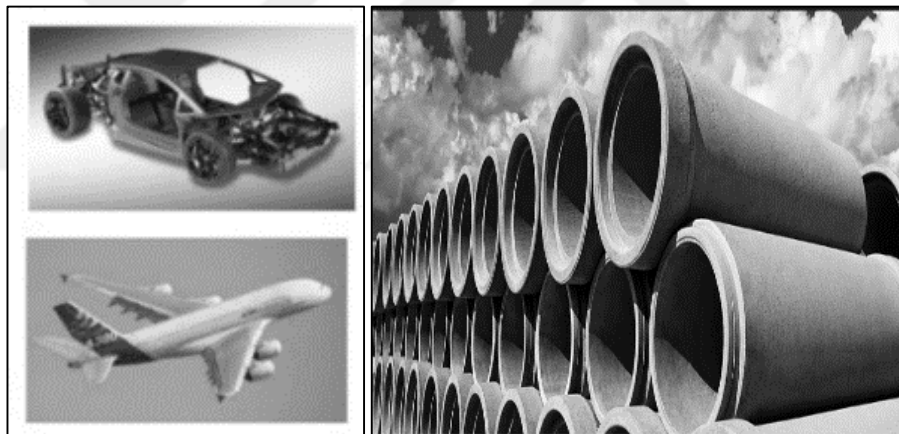


Figure 1.1: Application of polymer composite materials.

1.2 Literature Survey

1.2.1 Polymer matrix composites materials reinforced with fiber

Dhakar et. al. [6], had studied some mechanical properties of composite specimens consist of polyester as a matrix and hemp fibers as a reinforcement with the effect of moisture. Results show that the increase in fibers content lead to increase the moisture uptake of the specimens that due to the increase in the content of voids and cellulose. Also with the increase the moisture uptake the bending and tensile properties of composite specimens decreased.

Chandana [7], had studied the effects of different content of bamboo fibers reinforced in epoxy resin composites in order to investigate the thermal conductivity. The test specimens were prepared according to ASTM specifications. From results can be seen the thermal conductivity of the specimens composite materials has decreased with increases in content of bamboo fibers.

Olewi et. al., [8], had studied the effect of jute fibers on flexural properties of polyester matrix. The results illustrated that the flexural modulus, flexural strength and max. shear stress increase with increase the volume fraction of reinforcing materials.

Jagannatha [9], have studied investigation the mechanical properties of carbon and glass fibers by varying both the reinforcement in terms of weight percentage of (15%, 30%, 45% and 60%) with epoxy resin hybrid composite. Mechanical property such as micro hardness, tensile modulus, tensile strength and ductility were prepared according to ASTM. The results showed that the composite specimens reinforced by carbon fibers have higher micro hardness and ductility than other specimens.

Ozsoy et. al. [10], have studied the composite materials were manufactured by using a special designed mold. The components of the composite materials were epoxy resins such as the matrix reinforced with chopped carbon at four different weight fractions 0%, 6%, 8% and 10%. Tensile strength, bending, impact and hardness properties were measurement for composite materials. The results of the tests have shown that hardness increases with the increasing amount of carbon fiber in composites. Tensile, bending and impact performances have increased up to 8% of carbon fiber in the composite and then started to decrease.

Sanjay [11], have studied the details effect jute/E-glass fibers with epoxy resin matrix on mechanical properties. Thesis hybrid composites fabricated by hand layup method. The result shows the specimen (epoxy+jute fiber) has lower mechanical properties compared with specimen (epoxy+glass fiber). Also can be seen from result the specimen (epoxy+Jute fiber+glass fibers) has better mechanical properties than specimen only jute fibers. Also we note that the integration of jute fibers with fibers glass improves the mechanical properties and thus leads to increased use of natural materials in various industrial applications.

Ramesh [12], have studied some mechanical properties like tensile, flexural and impact strength using static test methods as per ASTM standards. The composite laminates was fabricated using hand layup method. The glass fiber and jute fibers were used as reinforcement to the polyester resin was considered as matrix material. Four

laminates were prepared with different fiber (L1: 100% jute fiber, L2: 100% glass fiber, L3: 60% jute + 40% glass fiber and L4: 60% glass fiber+40%jute fiber). The results show tensile test laminate (L1) decrease in strength when compared with (L2) also, in flexural test, composite laminate L3 & L4 improved strength than laminate L1. In the same way laminate L4 shows improved strength than laminate L3, because volume of glass fiber was higher in the laminate. The combination of these reinforcement in composite laminates improves the mechanical strength.

1.2.2 Polymer Matrix Composites Materials Reinforced with Fiber and Powder

Naga et. al. [13], have studied the thermal conductivity of hybrid composite specimens consist of polyester reinforced by glass fibers, sisal fiber and chalk powder. The results show that the thermal conductivity of polyester/ sisal fibers composite have higher thermal conductivity of polyester/ glass fibers composite. Also the results illustrated the increase in chalk powder contents lead to increase the thermal conductivity of composite.

AL_ Khazraji et. al. [14], have studied the tensile and fatigue properties of epoxy reinforced by carbon fibers, glass fiber, perlon fibers and silica particles. The results show the epoxy reinforced with carbon fibers gave the highest properties.

Vasanta et. al. [15], have studied the effect of rice husk and coir fibers with vinylester resin as the base material on the tensile strength and flexural strength. Test specimens are prepared by hand lay-up process with different weight fractions of coir fiber (0% ,5% ,10% ,15%,20%) and a small percentage of rice husks (1%,3%,5%) weight fractions are added to vinylester resin matrix . The result show the ultimate tensile strength of rice husk and coir fiber with vinyl ester resin increase at (5% wt. +15%wt.) , also from the result can be seen the ultimate flexural strength property increase at (3% wt. +17wt.) from rice husk and coir fiber with vinyl ester resin.

Rajaa et. al. [16], have studied the effect of fly ash particles in epoxy resin to produce polymer composite material. Size particles and chemical composition of fly ash with polymer improving the properties of composite materials. Different sized particles of fly ash at (10% weight fraction) used with epoxy resin to process polymer matrix composite materials. The average particle size of fly ash was obtained by the ball mill technique. The impact strength and hardness of mechanical properties have been discussed. The results indicate that the decrease in particle size of fly ash

improves the strength of polymer composite materials. The specimen (epoxy + 10% fly ash) with particle size 300nm has better impact strength and hardness than other particle size (50 μm , 480 nm and 350 nm) because of better bonding between reinforcement and matrix resin.

Singh et. al. [17], have studied the epoxy- resin reinforced with coconut shell powder at different volume fraction and evaluate some mechanical properties , density and water absorption for composites materials . The results shows the increase in CSP volume fraction the tensile strength goes on decreasing and an increase of filler volume fraction the flexural strength increases from 20% to 30%, while the flexural strength decreases when increasing filler volume fraction from 30%to 40% . Also can be seen from results the water absorption increase with volume fraction filler increasing.

Deogonda [18], have studied effect (TiO_2) and (ZnS) particles on the bending properties, tensile strength and impact strength of composites composed from epoxy resin reinforced with (60% , 59% , 58% and 57% volume fraction glass fiber) and (1%,2%and 3% volume fraction TiO_2 , ZnS). The results were shown the samples (epoxy +60%, 59%,58% glass fiber + 1%, 2% , 3% ZnS) have higher value tensile strength , impact toughness and bending than samples (epoxy +60%, 59%,58 and 57% glass fiber + 1%,2%,3% TiO_2).

Tapas et. al. [19], have studied some properties of composites consist of epoxy as a matrix reinforced by Al_2O_3 particles and jute fibers with different contents .The results show the Al_2O_3 particles have greater effect on the properties of composite. Also the results show the highest strength was to composite reinforced by 30wt% of jute fibers.

Dagwal [20], have study the density ,water absorption and some mechanical properties of composite material composed from unsaturated polyester resin , 5%, 10%, 15% , 20 % weight fraction banana fiber , 5%, 10%, 15%, 20% weight fraction glass fiber and 5% weight fraction OPEFB . The natural fibers (banana fiber and OPEFB) were extracted and processed locally. From result can be seen the specimen (UP+5% banana fiber + 15% glass fiber + 5% OPEFB) has higher value density and flexural strength than other specimens, also can be seen (UP+15% banana fiber + 5% glass fiber + 5% OPEFB) has higher value water absorption and hardness than other specimens, but the specimen (UP+10% banana fiber + 10% glass fiber + 5% OPEFB) has an impact strength.

Abdul_Hussein. et. al. [21], have investigated the effect of natural powders on some properties of composite materials. The specimen composed of epoxy resin reinforced with 6% V.F glass fiber and (3%, 6% V.F) from rice husk ash, carrot, sawdust powder. The results illustrated that the sample (epoxy + 6%g.f+6% rha) has higher values , density , hardness shore D, flexural strength and maximum shear stress than samples (epoxy+6%g.f+6% carrot and sawdust) , also this sample (epoxy + 6%g.f+6% rha) has lower water absorption than other volume fraction.

Srivastava et. al. [22], have studied effect of natural fibers reinforced with polymer matrix on mechanical properties. These specimen are made from epoxy resin as matrix with 10, 20 and 30 wt % coconut shell powder have been fabricated by using hand layup technique. The tensile strength, flexural strength and hardness are study. From the results show tensile strength increases from 17 MPa of epoxy to 29, 48 and 40 MPa in case of E-10CSP, E-20CSP and E- 30CSP composites respectively. Also can be seen from the result flexural strength increases from 35 MPa in case of epoxy to 59, 78 and 67 in 10, 20 and 30 wt % composites respectively.

Kannapiran et. al. [23], have studied composite specimens consist of the treated and untreated coirs as reinforcement eggshell as filler, epoxy and polyester as resins matrix. The results illustrated that the composite material of treated coir, eggshell and polyester is the best of all other combinations. This indicates that coir and eggshell can be used for many applications. From the result can be seen that 15% of treated coir fibers and 70% of eggshell with 15% of polyester in 50mm fiber length give higher values of mechanical properties.

Suhas et. al. [24], have studied the potential of chicken eggshell as a filler material in E-glass/epoxy composites. Calcium carbonate is the major constituent in chicken eggshell and can substitute mineral calcium carbonate as fillers in composites. Chicken eggshell was treated with sodium chloride solution to remove the layer of membrane on the inner wall. Treated shells were then dried, powdered, pulverized and sieved to size of 150 μ m to get eggshell fillers. Epoxy composite panels with eleven layers of plain weave fabric were fabricated with 0%, 3% and 6% wt. egg shell fillers. Results of tests showed drop in tensile strength while young's modulus and impact properties increased with egg shell fillers.

Praveenkumar [25], have studied investigation was carried out to use jute fiber and egg shell powder in different weight percentages (0, 2, 4, 6, 8, 10 wt. %) to determine its properties. The result shows that the addition of eggshell powder in jute

epoxy increases its tensile and flexural properties but gradually decrease the impact strength, the loading of fiber increases and their strength also increases the tensile and flexural properties. Also can be seen from the results the addition of egg shell powder more than 10% it tends to brittle and reduces the strength of material. Impact strength decreases by the addition of powder due to its void content.

Reem Alaa [26], has study the effect of Al_2O_3 particles on some properties of polyester reinforced with glass and carbon fibers. The polyester resin matrix was strengthened with 3% carbon and glass fibers and 1%, 3%, 5%, 7% Al_2O_3 powders. The water absorption, hardness (shore D), impact test, and flexural strength properties are studied. The results show the specimens (UP+3% C.F+7% Al_2O_3) and (UP+3% G.F+7% Al_2O_3) had the maximum hardness (shore D) and water absorption when compared with unfilled polyester resin specimen , it can be observed that the specimens (UP+3% C.F+5% Al_2O_3) and (UP+3% G.F+5% Al_2O_3) have maximum impact strength and flexural strength compared with specimens (UP+3% C.F+7% Al_2O_3) and (UP+3% G.F+7% Al_2O_3).

Reddy et. al. [27], have studied and discuss the effect of SiO_2 , TiO_2 , glass powder on the shear strength and density of epoxy reinforced by glass fiber specimens. The volume fraction (0%, 5% , 10% and 15%) from SiO_2 , TiO_2 , glass powder material have grater effect in the production of composite specimens. From the results can be shown the value density of the specimen (epoxy + glass + 10% SiO_2) higher when compare with TiO_2 , glass powder. Composite materials reinforced with 10% of SiO_2 powder show maximum inter laminar shear strength than TiO_2 glass powder.

Abhishek [28], have studied some properties of composites reinforced with rice husk and coconut coir in an epoxy resin matrix .The rice husk and coconut coir are available in nature at very low cost they have been recycled and used in industry applications. The development of natural fiber composite materials of coconut coir and rice husk powder will reduce agricultural waste and increases the usable value. The specimens of composite materials contain (80% epoxy +5%, 10%, 15% coconut coir + 5%, 10%, 15% rice husk) untreated and treated. The hardness test, impact test and water absorption test are studied. From the results can be seen the specimen untreated (80% epoxy + 15% coconut coir+5% rice husk) higher values impact strength and hardness than other specimens untreated and treated.

Studies and research papers of the previous literature survey can be divided into two parts according to its field of interest as follows: The first part is the polymer

matrix composites materials reinforced with fibers, the second part is the polymer matrix composites materials reinforced with fibers and Powder. It can be concluded from the literature survey of available studies and research papers that is not literature available for following sentence:

- a. Utilizing powder with fine particle size (Nano powder).
- b. Utilizing different temperature and for composition (800C⁰ for 2 hours).
- c. Studying mechanical properties such as flexural modules and fracture toughness.
- d. Studying some of physical properties such as water absorption.
- e. Studying some of thermal properties such as thermal conductivity, specific heat and thermal diffusivity (by use hot disk method).

1.3 Aim of the Work

The aims of this study include the preparation different composite materials used for industrial applications. These materials are composed of a matrix of epoxy resin with glass fiber and waste materials (rice husk and eggshell). However, the work includes:-

1. Recycling of existing waste in nature and obtaining ceramic powders as support materials and study effect of weight fraction fibers and powders on mechanical and physical properties.
2. Investigation the mean particle size analyzer, X- Ray fluorescence and X-Ray diffraction.
3. Investigation of physical properties (density, water absorption and thermal properties).

Investigation of mechanical (tensile test, flexural strength, modulus of flexural modulus, max. shear stress, hardness shore D, impact and fracture toughness)

1.4 Thesis Outline

Thesis consists of five chapter:

1. The chapter one consists introduction and literature survey.
2. The chapter two contains theoretical part about composite materials. The matrix epoxy and reinforced with glass fibers and natural powder.
3. Chapter three contains details of experimental methods used in the preparation of composite materials.

4. Chapter four reports the discussion of the experimental results obtained from the preparation and tests of composite materials.
5. Chapter five shows the conclusions and recommendations arising from the experimental results.



CHAPTER TWO

COMPOSIT MATERIALS

2.1 Introduction

Composites a mixture of two or more than two materials in which one of the materials, is strengthen phase (fibers, sheets or particles) and the other is a matrix phase (polymer, metal or ceramic). The idea of linking two or more different components in a single material gives an unlimited possibility to create new engineering materials characterized by a kind of different characteristics. The composite materials are used in all fields of industry because they possess various mechanical and physical properties. Extensive use of composite materials in electrical, machine building industries, electronics, automotive, aerospace, civil engineering, leisure industry and sport [29]. Types of reinforcements and fillers added in matrix are so many that it is no way to enumerate all. The main forms of filler particles and reinforcement are fibers, powders of different shapes, fabrics and mats [29]. Almost all characteristics can be changed by merging the filler particles or reinforcement into the matrix. The main classes of characteristics influenced by reinforcements and fillers are mechanical properties: stiffness strength, wear resistance, hardness, thermal, electrical and optical characteristics. There are modern classifications of composites into carbon-carbon matrix composite CCC, intermetallic matrix composite IMC, and hybrid composite [30]. Composite materials has advantages includes inherent damping, lower manufacturing costs because of the lower part count , good strength , stiffness and no corrosion . But the disadvantages of composite are low toughness, environmental degradation of the matrix, difficulty with analysis and possible weakness of transverse properties [31]:

From Figure 2.1, it can be noticed that composites are classified according to the geometry of reinforcement into three main sections: particulate reinforced, fiber reinforced and structural reinforced composite materials [3].

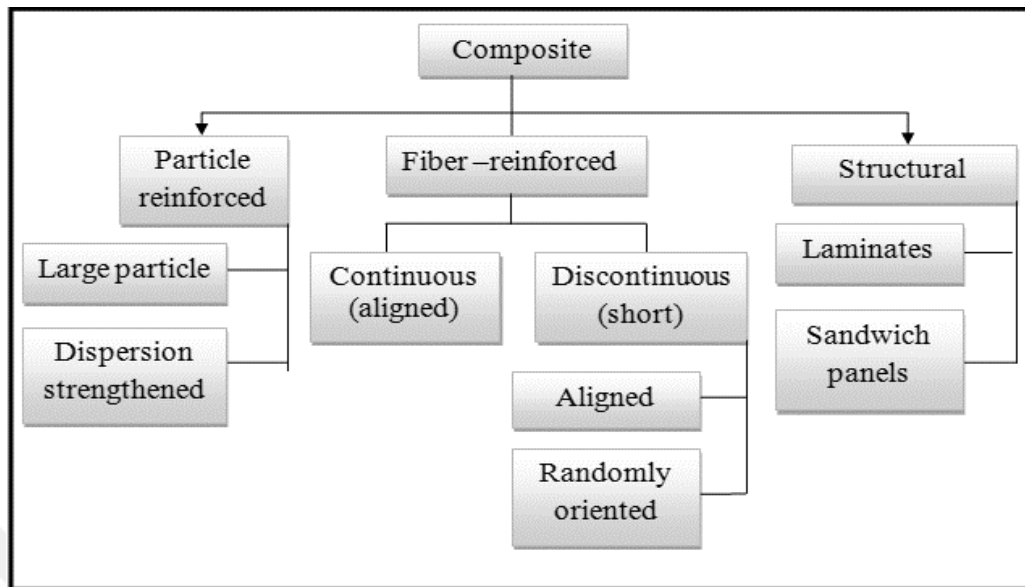


Figure 2.1: Classification scheme for the various composite types [3].

2.2 Fiber Reinforced Composite

The main purpose of the fiber in a composite are carry the load applied to composite. For this reason, fibers materials which have high tensile strength and a high elastic modulus. Ceramics are frequently used for the fiber reinforced in composite material [32].

2.3 Structural Composite materials

A structural composite usually consists of homogenous composite materials, which depend on the characteristic of the component materials and the engineering design of the various structural elements. Of the most common structural composites in use are laminar and sandwich panels only a relatively superficial examination is made here for this type of composite [3].

2.4 Particle Reinforced Composite

In engineering applications polymers reinforced with particles are widely used. These particle work to control most physical properties such as density and thermal properties. Although material scientists have studied filled polymer systems for many years, surprisingly little basic understanding exists regarding how the size, shape, distribution, surface chemical nature, and concentration of the tiller particles affect the

mechanical and rheological behavior of polymers during the curing process [33]. Particle reinforced composite can be classified into two type groups, based on particle size, as indicated below [34 & 35]:-

- Larger particles.
- Dispersion hardening composite.

2.4.1 Larger Particle Composite materials

The large particles composite is stiffer and harder than the matrix. These strengthen particles tend to restrain movement of the matrix media. The improvement of mechanical behavior or the degree of reinforcement depends on the strong bonding at the particle - matrix interface. Furthermore moreover, the volume fraction of the two constituents effect on the behavior of mechanical properties which are enhanced by increasing particulate content [3, 36 & 37].

2.4.2 Dispersion Hardening Composite

The matrix – particle interactions that lead to strengthening occur at the atomic or molecular level. Mechanism of reinforcement is similar to that for precipitation hardening [30&38]. So the plastic deformation is restricted such that tensile strength and yield as well as the hardness improve. The reinforcement is retained at high temperatures and for long time because the particles are chosen to be non- reactive with the matrix media.

2.5 Factors Affecting the Properties of Particulates Filled Composite Materials

1. **Volume Fraction**: There is a critical volume fraction at which the properties of a filled polymer is marked by change, where critical volume concentration (CVC) is a stage, where the binder is just enough to fill all the voids between particles [39, 40 & 41]. The critical volume fraction depends on filler particles and the nature of the polymer [39].
2. **Particles Size**: It has been found that change in particles size at constant volume fraction has clear and important influence on elasticity modulus of composite materials. In general, fine particles show better mechanical properties than large ones, as an example; there is reduction in tensile strength when there is increase in particle size at constant volume fraction

as a result of increase in stress concentration factor that in turn leads to enlarge crack volume [42 & 43].

3. **Modulus and Strength of Particles:** Particle phase has effects on rising or reducing elastic modulus of composite materials and that depends on elastic module of particles, whether it is higher or lower than that of the matrix. It has been observed, when using particles with higher elastic modulus, there will be an increasing in elastic modulus of resultant composite materials, reverse is right, as an example; when a weak particle is used that leads to decrease in cast toughness [44].
4. **Anisotropy:** Usually particulate composites consist of isometric particles. Even when all positions in space are identical with respect to a composition, directions in space may differ when spherical particles align in layers or linear aggregates, or when elongated particles are oriented in one direction [45].
5. **Particles Geometry:** Fillers are affected in stiffening of polymer. The degree of stiffening is a direct function of surface area of the particle .This area can be determined by particle shape, particle size, porosity, crevices and cracks in the fillers [39 & 40].
6. **Interface:** The interface is the zone of the link between the matrix media with reinforced material, formed during the operation of manufacture of composite material [46]. Mechanism to transfer the force from the matrix of the reinforced material is a basis to strengthen the matrix material with materials possessing high resistance, depends on the bonding strength between these materials, and otherwise it will be difficult to transfer force, and thus, act in reinforced materials as gaps within the matrix [47 & 48]. In addition, the behavior of the interface affects the material failure and required work in producing cracks and ruptures [49]. The properties of the interface and how they behave depends mainly on the ability of matrix material to wetting of the strengthening material (where matrix is liquid during manufacture of material composite) and this property is called (wettability) which can be defined as the extent to which the liquid has diffusion on the surface of a solid. In addition to the capability of moistening, interface depends on the type of bonds between two materials which is located between them. The two most important

types of bonds in PMCs are mechanical bonding and chemical bonding [47 & 50].

7. **Mechanical Bonding**: This type of connection is possible through the shape of both materials, in which one of them may contain holes or cracks that are penetrated or overlapped by other materials. Figure 2.2a shows the mechanical bonding mechanism between the fillers and the base material [51 & 52].
8. **Chemical Bonding**: It is considered as one of the main types of bonding. For the purpose of obtaining, an interface which has strength close to strength of composite materials, resort to this type of interconnection. The chemical interconnection may be achieved across the molecular forces that are resulting from the influence of materials molecule one on the other hand. The efficiency of this type of bonding count on the type of chemical interconnection that gets between molecules as in Figure 2.2 b. In this type of bonding, linking may be in the form of covalent, ionic or metallic bond, and the more wettability there is, the more is the efficiency of this type of bonding [42 &52].

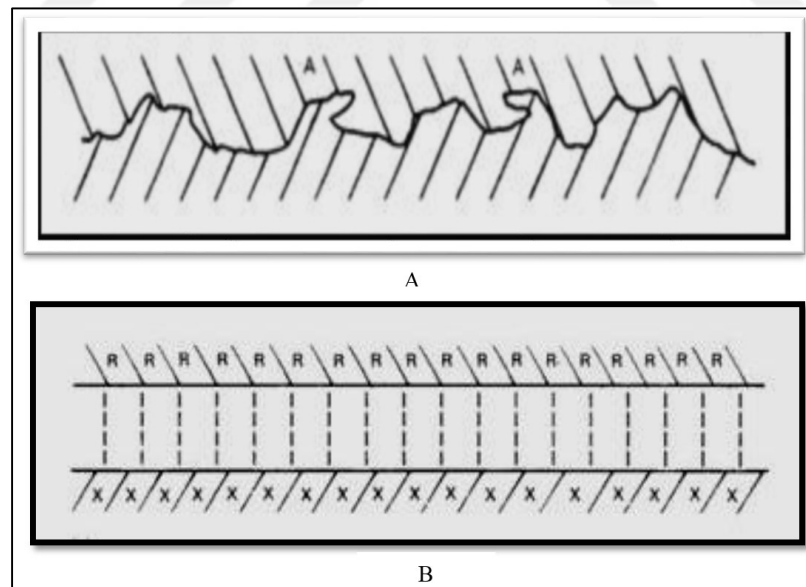


Figure 2.2: (a) Mechanical bonding, (b) Chemical bonding R and X represent compatible chemical group [42].

Consequently, it can be concluded that the interface is an important element and has effects on the behavior of the composite material. Binding strength in this region must be considered, the most important way in which binding occurs is [51& 52].

- Property and ability of wet.
- Inter- diffusion.
- Electrostatic attraction.
- Chemical connection and mechanical adhesion.

2.6 Matrix Material

In fibrous composites the matrix phase may be a polymer, metal, or ceramic. The matrix phase materials in case of fiber-reinforced composites serves the following functions [53]:-

1. Matrix binds the fibers together.
2. Works to protect individual fibers from surface damage caused by chemical reaction with the environment or mechanical abrasion.
3. It separates the fibers.

The thermosetting polymer resins are the most common material used as a matrix material and are the least expensive. The type of resins selected depending on the application of the fiber reinforced polymer.

Epoxy resin provides excellent resistance to acid and water environments and is used in wastewater treatment plants, general outdoor applications and shoreline applications [54]. Matrix has properties includes low density and strength, bonding the fibers together, carry load to the fiber, be thermally and chemically compatible with fiber, Protect fibers surfaces from damage.

2.6.1 Epoxy Resin

Epoxy resins represent the highest performance matrix between matrices (vinyl ester resin, polyester resin). The epoxy resin is typically used in high performance applications, for example sports and aerospace applications compared to vinyl ester resin and unsaturated polyester resin. There is a wide range of modifications that can be made of epoxy resin to suit a particular application more than in unsaturated polyester resin. The advantages of epoxy resin include has the strength when exposing it to a continuous load compared to the vinyl ester and polyester resin, low shrinkage processing, low creep, resistance to chemical and good adhesion properties with other substrates . But the epoxy resin has many disadvantages include expensive compared with other resins leading to a product that has a high material cost, corrosive handling,

complex manufacturing processing of the composite leads to an expensive product, health problem with allergies and critical mixing [55]. The type of hardener has a major effect on the characteristics and applications of epoxies resin [56].

2.7 Fibers Materials

Materials in the form of fibers are stiffer and stronger than those used in bulk form. There is a possibility of defects in bulk materials that effect on strength while internal defects are mostly absent in the case of fibers. Moreover the fiber has strong molecular or crystallographic aligned in the shape of very small crystals. Fiber is the most important component of fiber reinforced composite materials. It also occupies the largest volume fraction of the composite. Properties of fiber are [57]:-

1. High modulus of elasticity and high ultimate strength.
2. Low variation of strength between individual fibers.
3. Uniform fiber cross section.
4. Stability and retention of strength during fabrication.

2.7.1 Glass Fibers

Glass fiber is the used reinforcement for rubber and plastic. It is the predominant material in industries such as boat construction and corrosion equipment, medical, industrial equipment, recreational, and automotive [57& 58].

The main advantage and disadvantages of the glass fiber

Advantages [59]

1. Glass fiber has low cost.
2. It is high tensile strength.
3. It is excellent insulating properties and good corrosion resistance.
4. Glass fibers are susceptible to sustained loads, as they cannot withstand loads for long periods.
5. Light weight.

Disadvantages [59]

1. Low stiffness.
2. Short fatigue life.
3. High temperature sensitivity.

2.8 Natural Materials

2.8.1 Rice Hush (RH) and Rice Husk Ash (RHA)

Rice husk is an agrarian waste available in rice-producing country. Removal during the refining of rice, these husks have no commercial benefit [60]. Because rice husks contain cellulose and a low percentage of sugar content, it cannot be used as feed for cattle. Rice husks include 20% of non-crystalline silica, carbon and 80% of volatile organic. The climatic conditions and the nature of the fertilizer used affects the chemical composition of rice husk ash [61]. The researchers found that rice husk is an excellent source of high temperature amorphous silica [62&63]. Based on this, silica extracted from rice husk is used in different industries such as toothpaste, cosmetics, rubber industry as an arming agent, insulating materials, portland cement and pottery ware [60].

2.8.1.1. Applications of Rice Husk (RH)

The use of rice husk in different chemical and physical applications depends on silica content and ash content. Rice husk can be used in power plants, insulating fire brick, production of (xylitol, furfural, ethanol, acetic acid, lingo sulphonic acids) , formation activated carbon, polishing agent and industrialization building material [64, 65, 66 &67].

2.8.1.2 Applications of (RHA)

RHA was used in different industrial applications, such as processing of steel, refractory industry, cement, source of SiO₂ and construction industry [68&69].

2.8.1.3 Characteristics of RH and RHA [70]

1. Rice husk is resistant to moisture and is also difficult to burn except with air.
2. Good insulating material.
3. RH decomposes slowly because it contains a high silica.
4. RH contains an average high thermal value of 3410 kcal / kg, thus a good and renewable energy source.

5. Rice husks are easily burned, but turning them into ashes is difficult, and this occurs because of the low melting point of the ash of rice.

2.8.1.4 Properties of RH and RHA

The properties of RH and RHA are given in tables (2.1) and (2.2).

Table 2.1: Characteristic of Rice Husk.

Physical state	Solid and not hazardous
Manifestation	Powder
Mean particle size	53.7 μm
Color	Earthy color
Scent	Scentless

Table 2.2: Characteristic of rice husk ash.

Physical state	Solid and not hazardous
Manifestation	Powder
Scent	Scentless
Color	Black
Density of rice husk ash	0.462 g/cm^3

2.8.2 Eggshells

Eggshell is a food waste and thus a problem in the environment and at the same time possesses good mechanical properties which give reason to consider it as a modifier for epoxy [71]. The composite material strengthened with egg shell powder to give off a high modulus, strength and other mechanical embedded or is linked with matrix. Usually the eggshells containing calcium carbonate with polymeric materials give composite materials are carrying the load. Thus, although fibers provide strengthened for the matrix material, the final as well as serving a number of advantageous functions an eggshell reinforced polymer composite material [72]. Epoxy resin with eggshell powder can be used in different engineering applications, thus allowing their use in automobiles, space, aeronautics, sports goods related

applications and industries [22]. The properties of eggshell powder before and after burning as show in the tables (2.3) and (2.4).

Table 2.3: Characteristic of rice husk.

Physical state	Solid and not hazardous
Manifestation	Powder
Mean particle size	19.4 μm
Color	White color
Scent	Scentless

Table 2.4: Characteristic of rice husk ash.

Physical state	Solid and not hazardous
Manifestation	Powder
Mean particle Size	16 μm
Color	Gray color
Scent	Scentless

2.9 Rule of Mixture

The essential idea of the simplest rule of the mixture is that the evaluation of some of the microscopic physical quantity describing a complex solid as a whole uses a total value equal to the sum of the appropriate quantities of discrete parts of the solids multiplied by the volume fractions of the solid parts. The density of the composite material can be calculated by the following rule [34, 73 & 74].

$$\rho_c = V_f \cdot \rho_f + V_m \cdot \rho_m \quad (2.1)$$

Where;

ρ_c ; Density of the composite material.

ρ_m, ρ_f ; Density of matrix and fiber.

V_m, V_f ; Volume fraction of matrix and fiber.

1. Volume fraction is defined as

$$V_f = \frac{v_p}{v_c} * 100\% \quad (2.2)$$

$$V_m = \frac{v_m}{v_c} * 100\% \quad (2.3)$$

Where;

V_m, V_p ; Volume fraction of matrix and reinforcement.

v_p, v_m, v_c ; Volume of reinforcement, matrix and composite respectively.

2. Weight fraction is defined as

$$V_p = \frac{\rho_c}{\rho_p} W_p \quad (2.4)$$

$$V_m = \frac{\rho_c}{\rho_m} W_m \quad (2.5)$$

Where;

ρ_c, ρ_p, ρ_m ; Density of composite, reinforcement and matrix respectively.

W_p, W_m ; Weight fraction of reinforcement and matrix respectively.

$$W_p = \frac{w_p}{w_c} * 100\% \quad \dots\dots\dots (2.6)$$

$$W_m = \frac{w_m}{w_c} * 100\% \quad W_m = \frac{w_m}{w_c} * 100\% \quad (2.7)$$

Where;

w_p, w_c, w_m ; Weight of reinforcement, composite and matrix respectively.

We should notice the following:

$$W_p + W_m = 1 \quad (2.8)$$

$$V_p + V_m = 1 \quad (2.9)$$

2.10 Physical Properties

2.10.1 Measured Density

The measured density (ρ_t) is calculated from the method of immersion in water (Archimedes base) using the following relationship [75].

$$\rho_t = \frac{W_d}{W_s - W_n} * D \quad (2.10)$$

Where:

ρ_t : Measured density (g/cm^3).

D: Density of distilled water (1 g/cm^3).

W_d : Dry weight of the specimen (g).

W_n : Weight of the specimen when submerged in water (g).

W_s : Weight of the sample after saturation in water for 24 hour (g).

2.10.2 Water Absorption Test

Polymer composite materials are usually exposed to liquids. It is important that this liquid does not negatively affect the performance and lifetime of the product. It is a diffusion controlled process which causes volume change for polymer component. Therefore the liquid effect depends basically on the immersion time, temperature, and polymer thickness in addition to the type of liquid and polymer. Conditioned specimens are then immersed in distilled water 2 days at room temperature [76& 6]. By inundation of the sample in water, the water absorption change in mass can be calculated depending on the equation [77& 78]:-

$$M (\%) = \frac{(m_t - m_o)}{m_o} \times 100 \quad (2.11)$$

Where:

M (%): Water absorption percentage.

m_o ; Mass of samples before immersion (g).

m_t ; Mass of samples after immersion for 2 days (g).

2.10.3 Thermal Properties Test

The thermal properties determine the rate of temperature change in material during fabrication and use. They are fundamental to measurement the (resistance to thermal stress, operating temperature and temperature gradient) [79].

2.10.3.1 Thermal Conductivity

Thermal conductivity is the property of a material which relates to its ability to conduct heat. The coefficient thermal conductivity of can be calculated according to (Fourier's Law). Thermal conductivity of polymer between (0.17-0.25 W/m.K), therefore most of polymer using as insulated material for this reason [3].

2.10.3.2 Specific Heat

Specific heat is the amount of thermal energy required to raise one kg of material one degree. This property depends on the type of material and density of materials. When increasing porosity lead to increasing the specific heat. This property represents very important in cooling and heating operation [79].

2.10.3.3 Thermal Diffusivity

Thermal diffusivity is one of most important thermal properties which are defined as the ratio of thermal conductivity of material to the heat storage capacity. This depends upon the density of materials, when increasing density lead to decreasing the diffusivity and sometime represented insulator materials because of good heat storage. Thermal diffusivity can be calculated by the following formula [79]:

$$DT = \frac{K}{\rho C_p} \quad (2.12)$$

2.11 Mechanical Properties

2.11.1 Tensile Test

Tensile test is widely used to provide the designer with information about material strength and maximum elongation and others.

The tensile stress: Is the point at which the sample elongation is increased without a corresponding increase in force at yield point. This force can be calculated depending on the equation.

$$\sigma = P/A \quad (2.13)$$

where:

σ : Stress for tasted (MPa).

P: applied force (N).

A: original cross sectional area of the original specimen (m²).

The strain which is used in such stress-strain curve is a linear strain and can be expressed as:

$$\epsilon = \frac{\Delta L}{L_0} \quad (2.14)$$

Where:

ϵ : strain.

$$\Delta L = L - L_0$$

Where:-

L: the final length (m).

L₀:the original length (m).

From the tensile curve, the following can be calculated ultimate tensile strength, young modulus and Elongation percentage at break [80].

2.11.2 Flexural Test

The flexural strength represents the strength when breaking a beam section when tested. Flexural strength can be determined by “3- Point Test” or “4- Point Test” [81]. Three point flexural test as shown in Figure 2.4 below [3&82].

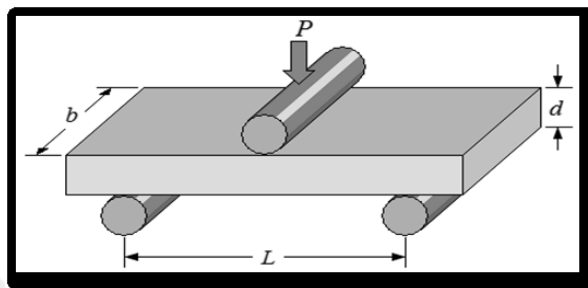


Figure 2.3: The 3- point flexural test [3&82].

The flexural strength and maximum shear stress can be calculated by the formula equation [81,82 & 83].

$$F. S = (3 pL) / [2bd] \quad (2.15)$$

Where :

- F.S: Flexural strength (MPa).
- P: Force at fracture (N).
- L: Length of the sample (mm).
- b:Thikness of the sample (mm).
- d:Width of the sample (mm).

$$\tau = 3P / (4bd) \quad (2.16)$$

Where

- τ : Maximum shear stress (MPa)
- P: Force at fracture (N).
- b:Thikness of the sample (mm).
- d:Width of the sample (mm).

Also from the flexural strength can be calculated the flexural modulus of elasticity, from it drawing (load-deflection) curve by following equation: [84].

$$EB = (PL^3) / (4 b d^3 \delta) = (m L^3) / 4bd^3 \quad (2.17)$$

Where:

EB: Flexural modulus (MPa).

δ : is the deflection of the beam (mm).

P: Load applied (N).

L: Span length (mm).

b: Width of sample (mm).

d: Depth (Thickness) of sample (mm).

m: Slope of the tangent in the load- deflection curve (N/mm).

2.11.3 Impact Strength Test

The impact properties of a material represent its capacity to absorb and dissipate energies used to measure the strength of material under impact or shock loading [85 & 86]. Izod and Charpy tests are ordinarily conducted to assess impact strength of falling weight impact test (ISO - 180) [87]. Polymers may exhibit brittle fracture or ductile under impact force depend on the mode of loading, of size specimen, temperature and strain rate. Both semicrystalline and amorphous polymers are brittle in impact test used to measure the strength of materials under high rate of loading (sudden loading). The material may show different behavior from load to another. For example, the material may show ductile behavior in tensile test or bending test, while the same material will show brittle behavior in the impact test. Impact strength can be calculated by the formula equation [85 & 86]:-

$$G_c = \frac{U_c}{A} \quad (2.18)$$

Where;

G_c ; toughness of material (J/m^2).

U_c ; impact energy (J).

A ; area of specimen cross- sectional (m^2).

The fracture toughness can be defined as the ability of a material containing a crack, to resist fracture and can be calculated by the formula equation [86]:-

$$K_{Ic} = \sqrt{G_c * Eb} \quad (2.19)$$

Where;

K_{Ic} ; fracture toughness of material / ($MPa.m^{1/2}$).

G_c ; impact strength of material / (J/m^2).

E_b : Flexural Modulus of Material / (MPa).

2.11.4 Hardness Test

Hardness of material expressing its resistance to scratching, cutting, wear, indentation, penetration and machinability. Material hardness generally depends on the type of interatomic or intermolecular bonds, surface condition, temperature, and others. Hardness increases with decreasing particles size. Hardness covers [31]:-

1. Elasticity, plasticity.
2. Strength and strain.
3. Brittleness/ ductility and toughness.

Hardness shore is measured with a device known as the Durometer scale and therefore also known as the "Durometer hardness". The hardness shore is determined by the penetration of the Durometer foot into the specimen. Due to elastic rubber and plastic indentation may change over time. So the indentation time is sometimes reported along with the hardness number [84].

CHAPTER THREE

EXPERIMENTAL WORK

3.1 Introduction

The chapter shows the materials and the equipment used in this work, Figure 3.1 includes three major points:

The definition of the raw materials used in the preparation of composites. The reference of how to prepare moulds for preparing composites, and how to prepare specimens for each test and methods of testing as well as specimen pictures before and after the examination and diagrams of the geometric shapes required of these samples and for each test according to international standard. The tests that are conducted on the prepared samples.

3.2 Materials Used

The materials, basic used in the preparation of research samples are epoxy resin, woven E-glass fibers materials and reinforcements, which are waste materials like (rice husk, rice husk ash, eggshell before and after burning).

3.2.1 Epoxy Resin (ER)

Epoxy resin (EUXIT 50 KI) base was used as the matrix. It was provided from (Al-Rakaez Building Materials in Amman) made in Egypt Arabic, Thermoset polymer which is thermally hardened. Epoxy resin excellent bond strength, low viscosity, low creep, low shrink and exhibit good chemical resistance .Table 3.1 shows the characteristics of epoxy used in the research by the manufacturer specifications [88]. Figure 3.2 shows epoxy resin and hardener (EUXIT 50 KI) used in the research.

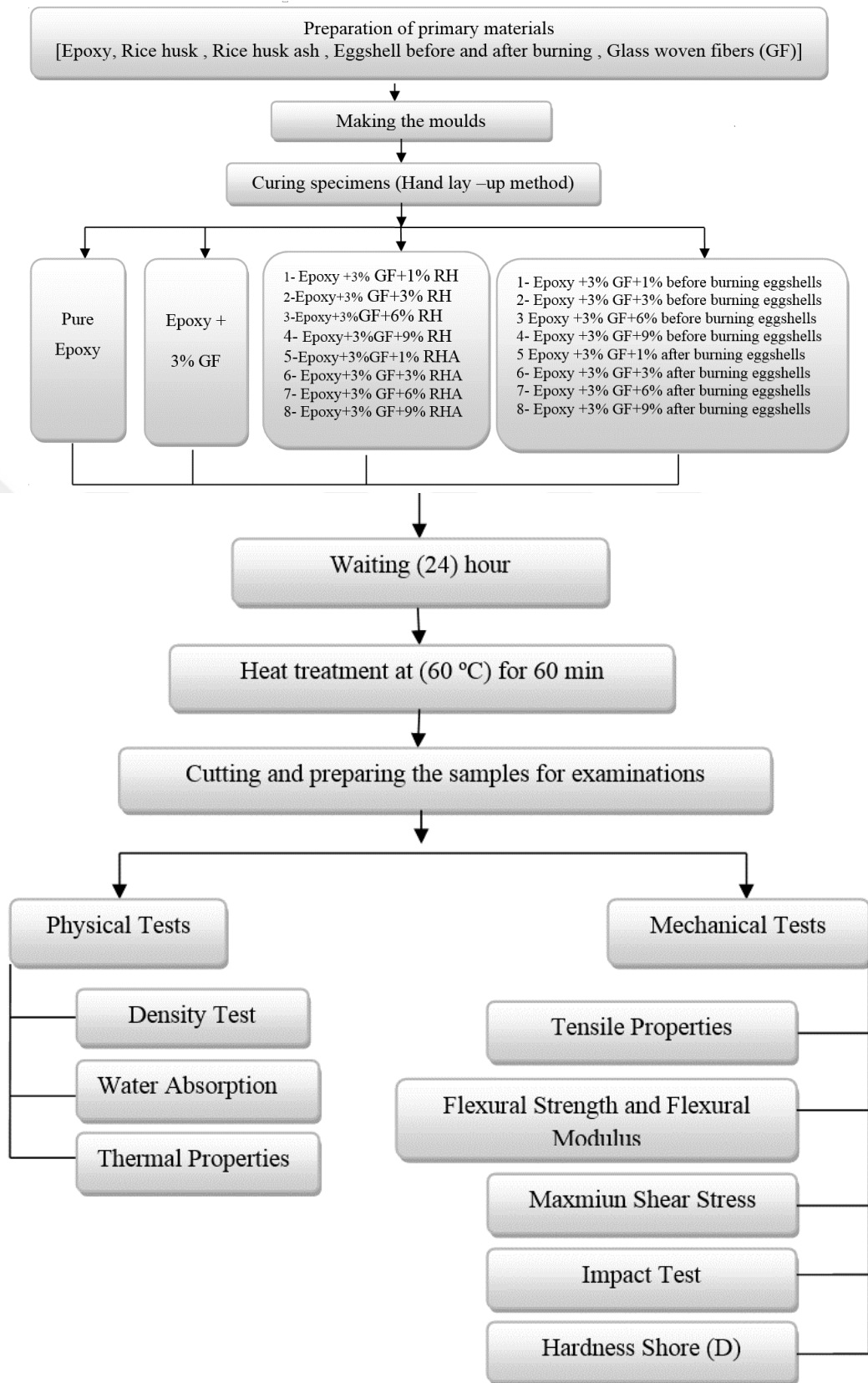


Figure 3.1: Flow chart of the experimental work.

Table 3.1: Characteristics of epoxy used in the research [88].

Modulus of elasticity N/mm ²	Bending N/mm ²	Viscosity at 20 C°cps	Application temperature (C°)	Volume shrinkag e (%)	Linear shrinkag e (%)	Density gm/cm ³
2800	45	300	5	3.5	0.3	1.05



Figure 3.2: (a) The cans of epoxy resin (EUXIT 50 KI) and hardener, (b) The sample of epoxy resin and hardener.

3.2.2 Glass Fiber

Glass fiber (E type) is the most widespread material used for commercial fiber reinforced polymer composite. These materials make up more than 90% of the fiber used in the reinforced plastics industry due to their low cost and relatively good strength to weight properties. The type of glass fiber is used in this work as reinforced materials (woven E- glass fiber) from the Tenax company, England made are used as reinforcement as shown in Figure 3.3. Table 3.2 shows mechanical properties of E-glass fiber [89&90]. The main advantage of E- glass fiber are low cost, high production rates, relatively low density, relatively insensitive to moisture , chemical-resistant , able to maintain the properties of strength on a wide range of conditions and non-flammable and heat-resistant [91].

Table 3.2: The mechanical properties of the glass fibers used in the research [89&90].

Compressive strength	1080 MPa
Tensile strength	3445 MPa
Thermal expansion	5.4 $\mu\text{m}/(\text{m}\cdot\text{C}^\circ)$
Young modulus	72.5 Gpa
Percentage elongation	4.3
Poisson 's ratio	0.22
Softening	846 C°
Density	2.58 gm/cm^3

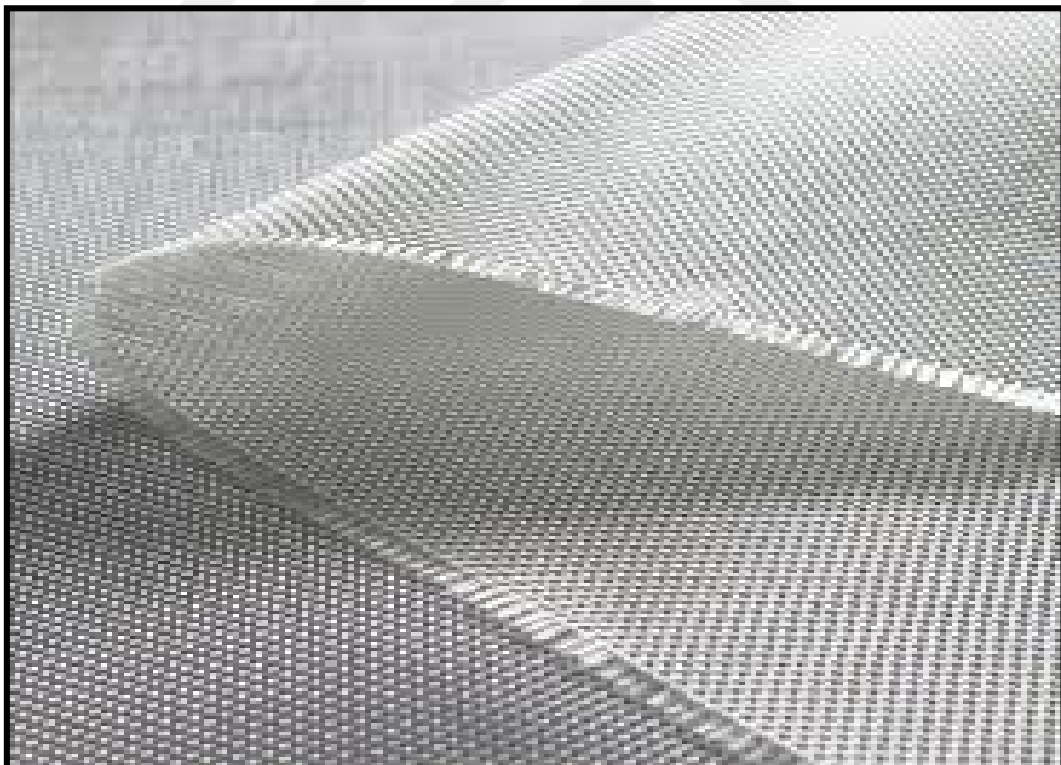


Figure 3.3: Woven E- glass fiber used in the research.

3.3 Preparations of Natural Materials From Waste

3.3.1 Preparations of Rice Husk

Figure 3.4 shows the steps for the preparation rice husk

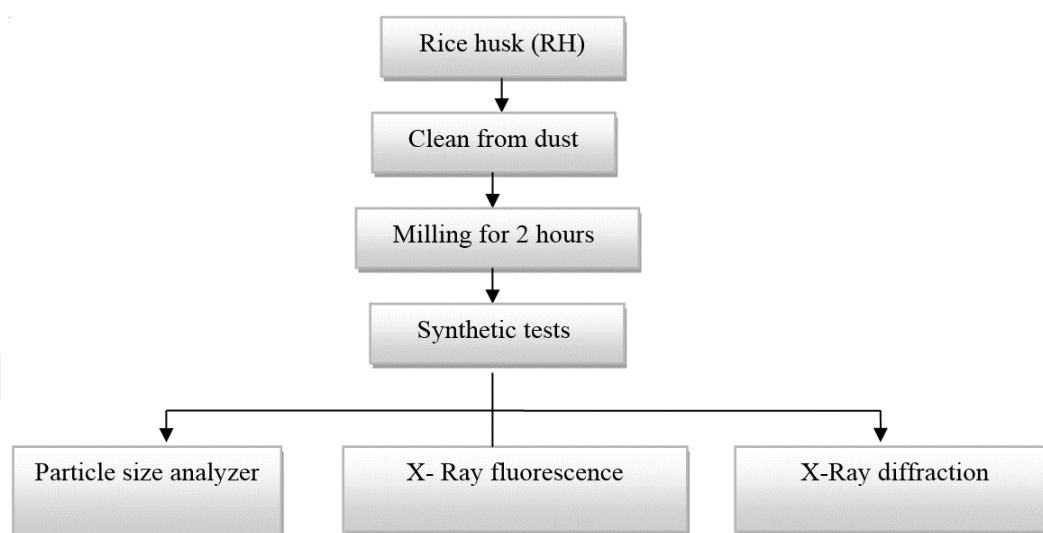


Figure 3.4: Preparation rice husk material.

The rice husk is an agricultural byproduct material. It constitutes for about 20% of the weight of rice. It contains for about 25–30% lignin, 38-50% cellulose, and 15–20% of silica and other element of oxides. Figure 3.5 shows the waste of rice husk around a factory that might contain many impurities.



Figure 3.5: Woven E- glass fiber used in the research.

Figure 3.6 (a-b) shows the shape of a mill used to grind the rice husks for two hours and shape of rice husk after milling. The specifications of the mill (High speed multi-functional crusher , Model 200 , Made in china, Voltage 220 v/60 HZ , Rotating Speed 25000 r/min , Working time 5 min , Powder – factor 1000w , Grinding degree

50-30 μm , interval time 10 min) .The mill is located in powder technology Laboratory, Materials Engineering Department, University of Technology.

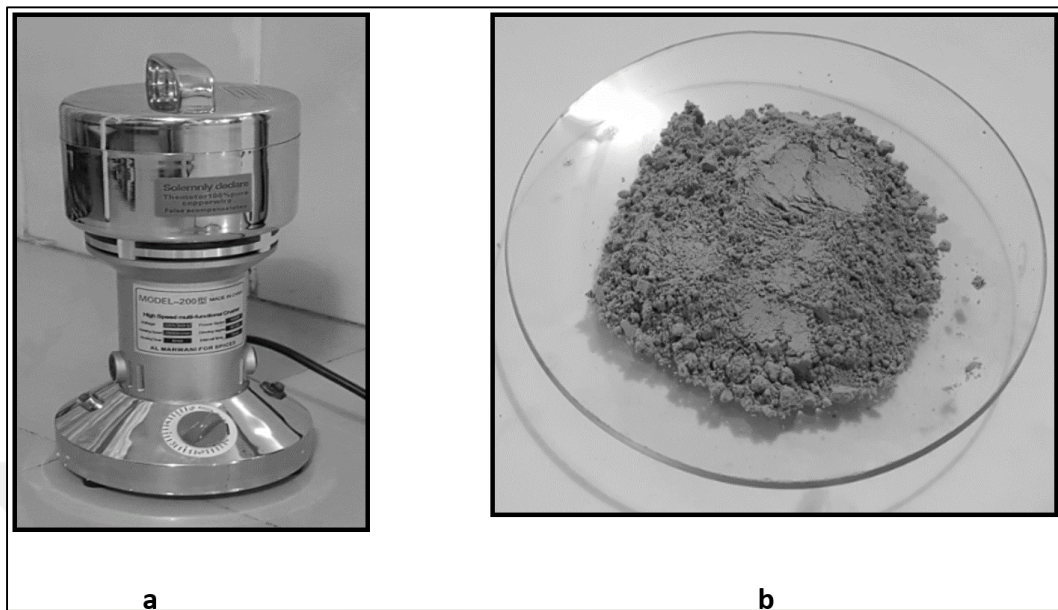


Figure 3.6: (a) Shape mill used to grind the rice husks, (b) Shape the rice husk after milling.

3.3.2 Preparations Rice Husk Ash

Figure 3.7 shows steps to prepare the rice husk ash.

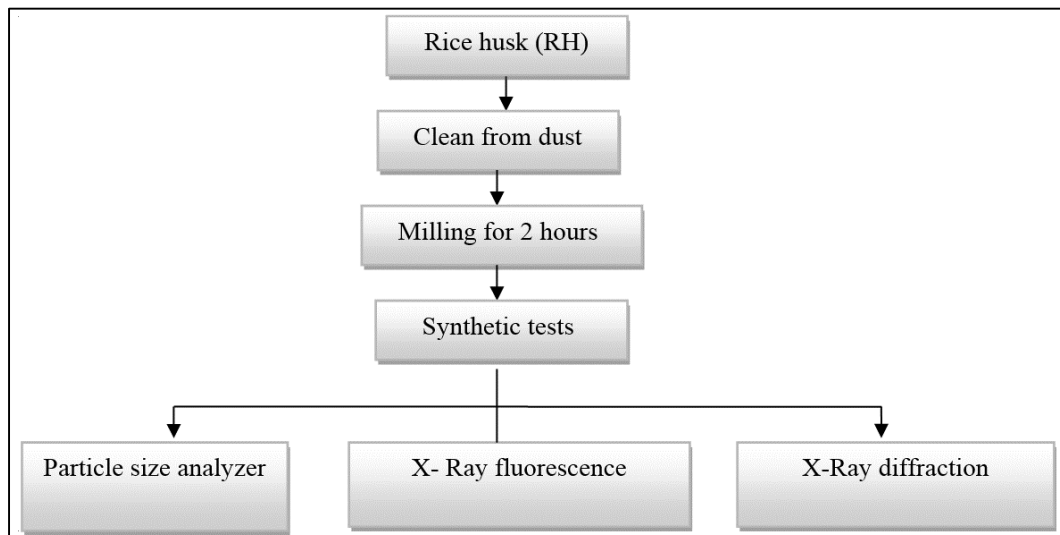


Figure 3.7: Preparation rice husk ash material.

Rice husk converts to rice-husk ash when burnt. When rice husks are burned, cellulose and lignin are removed, leaving silica ash behind. The temperature and burning environment control the quality of rice husk ash, particle size and specific area

dependent on the burning condition [92&93]. For the purpose of producing the best pozzolanas, the temperature of the burning of the crusts must be controlled at 800C° and guarantee that the creation of carbon is kept to a minimum by supplying an adequate quantity of air. In burning temperatures 800C° an ash rich in amorphous silica is formed which is very reactive. Temperatures above 800C° silica crystalline products, which is much less reactive. The presence of large amounts of carbon in the ash will negatively affect the strength. Carbon content should be limited to 10% ash maximum. There are many designs of small simple burners, usually made of mud bricks fire, which are able to burn ash at 800C° and without excessive amounts of carbon [94]. Figure (3.8) shows the furnace used for combustion (the melting furnace with 1200 degree chamber size: 250×250×300, heated by resistance wire and max temperature 1200 C°) the furnace is located in (Casting) Laboratory, Materials Engineering Department, University of Technology. Figure 3.9 shows rice husk after combustion.



Figure 3.8: Furnace used for combustion rice husk.



Figure 3.9: Rice husk after combustion.

The third step in the processing is grind the RHA into a fine powder, and the mills are usually used for this purpose. Crystalline ash is harder and require more milling in order to achieve the required fineness [94]. Figure 3.10 shows rice husk ash after milling for 2 hours.



Figure 3.10: Rice husk ash after milling 2 hours.

3.3.3 Preparations of Eggshells

3.3.3.1 Preparation eggshells powder before combustion

Eggs were bought locally from the market. The first step eggshells are washed and dried to remove all foreign matter such as egg yolk or albumin, second step milling eggshell for 2 hours. So the steps to prepare the eggshell powder can be seen in Figure 3.11, also Figure 3.12 indicates the images preparations of egg shells before and after milling.

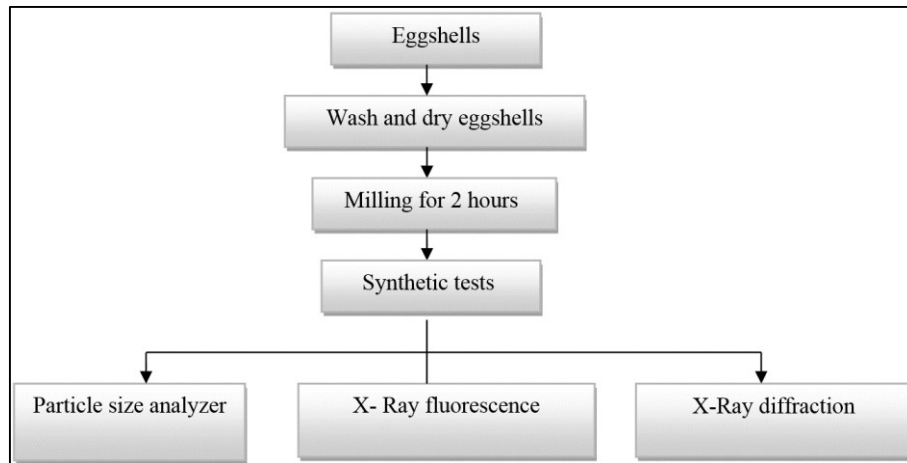


Figure 3.11: Preparation material eggshells powder.

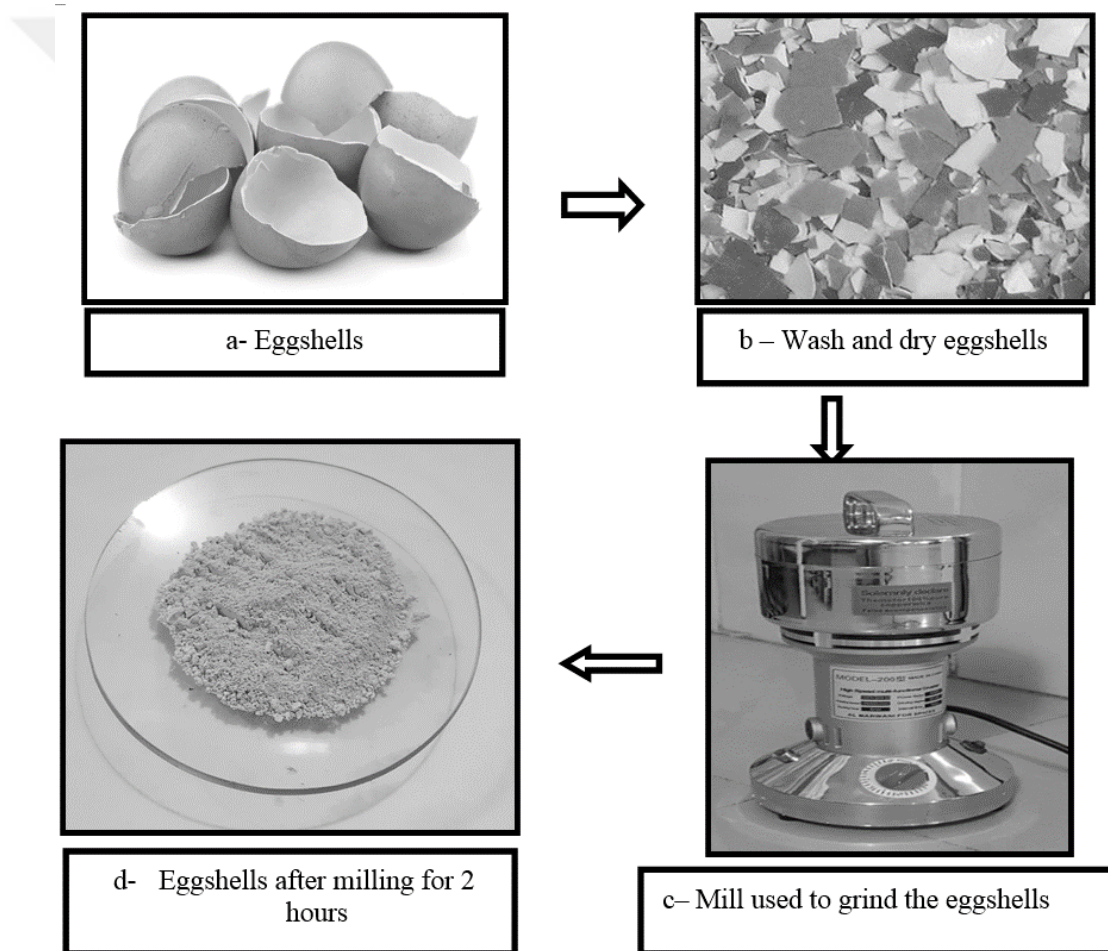


Figure 3.12: Images preparations of egg shells before and after milling (a) Eggshells, (b) Wash and dry eggshells (c) Mill used to grind the eggshells, (d) Eggshells after milling.

4.3.3.2 Preparation eggshells powder after combustion

Figure 3.13 steps for preparing the eggshells powder after combustion. The step of burning eggshells is very important and its purpose to improve the physical and

mechanical properties. As the temperature of burning eggshells increase the proportion of calcium oxide increase .The Figure 3.14 indicates the images preparations egg shells powder after combustion.

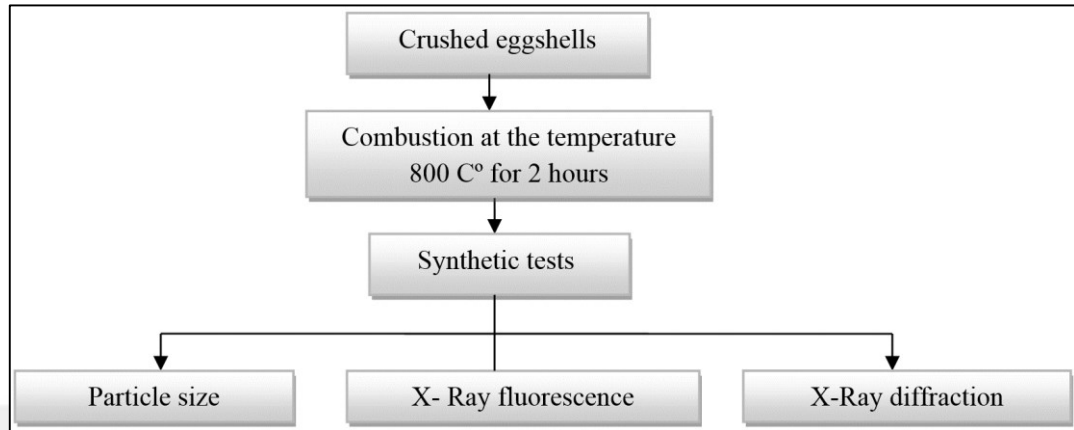


Figure 3.13: Steps for preparing the eggshells powder after combustion.

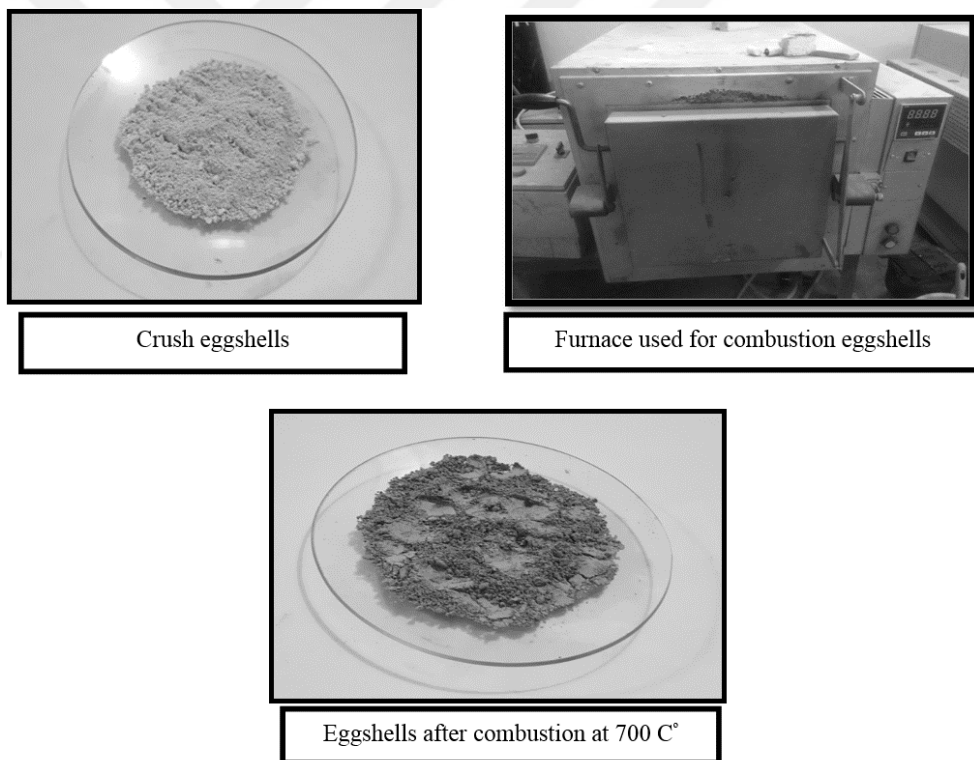


Figure 3.14: Images preparations eggshells powder after combustion.

3.4 Particle Size Analyzer

3.4.1 Particle Size Analyzer Rice Husk Powder

Distribute the particle size of the rice husk powder as shown in Figure 3.15. From figure can be see the minimum particle size (0.5 μm) and the maximum particle size

(315 μm), while the average particle size of the rice husk powder equal (53.7 μm). Figure 3.16 shows the particle size analyzer device (MALVERN INSTRUMENTS LIMITED , Made in U.K , Model ADA2000 , Serial No: MAL 1099136 , Dimensions (320mm W , 600mm D, 260mm H), Weight 21kg, Power 100W) examination has been conducted at the Ministry of Industry & Minerals / Corporation for Research and Industrial Development / Ibn Sina Center for Researches.

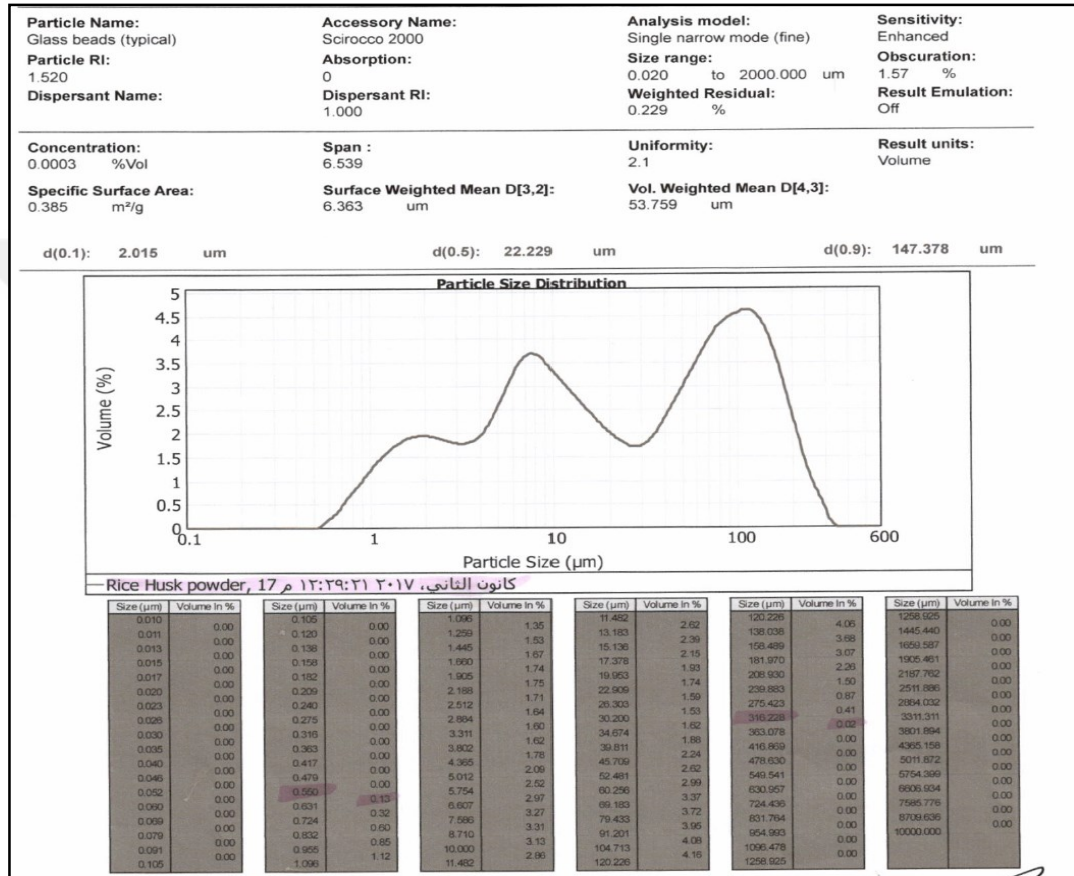


Figure 3.15: Particle size analyzer of the rice husk powder.



Figure 3.16: Device of particle size analyzer device.

3.4.2 Particle Size Analyzer of the Rice Husk Ash Powder after Combustion

Distribute the particle size of rice husk ash powder as shown in Figure 3.17. From figure can be seen the minimum particle size (0.5 μm) and the maximum particle size (22 μm), while the average particle size of the rice husk ash powder equal (10.4 μm).

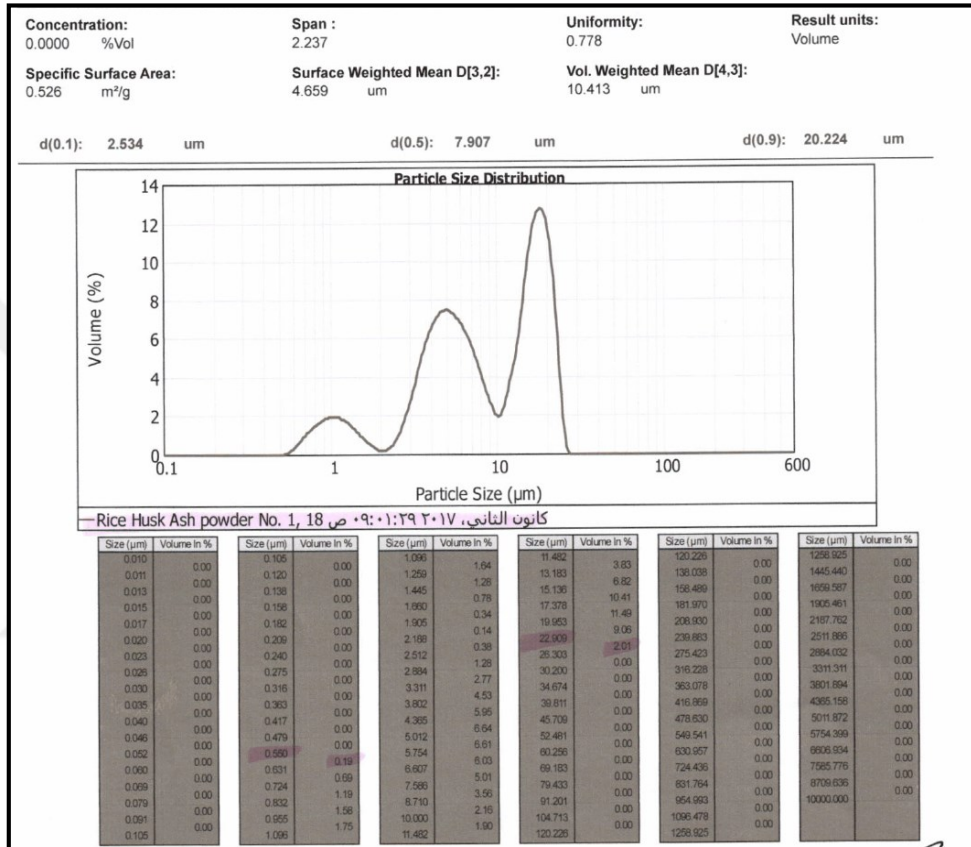


Figure 3.17: The particle size analyzer of the rice husk ash powder.

3.4.3 Particle Size Analyzer of Eggshells Powder before Combustion

Distribute the particle size of the eggshell powder before combustion, as shown in Figure 3.18. From figure can be seen as the minimum particle size (0.2 μm) and the maximum particle size (138 μm), while the average particle size of eggshells powder equal (19.4 μm).

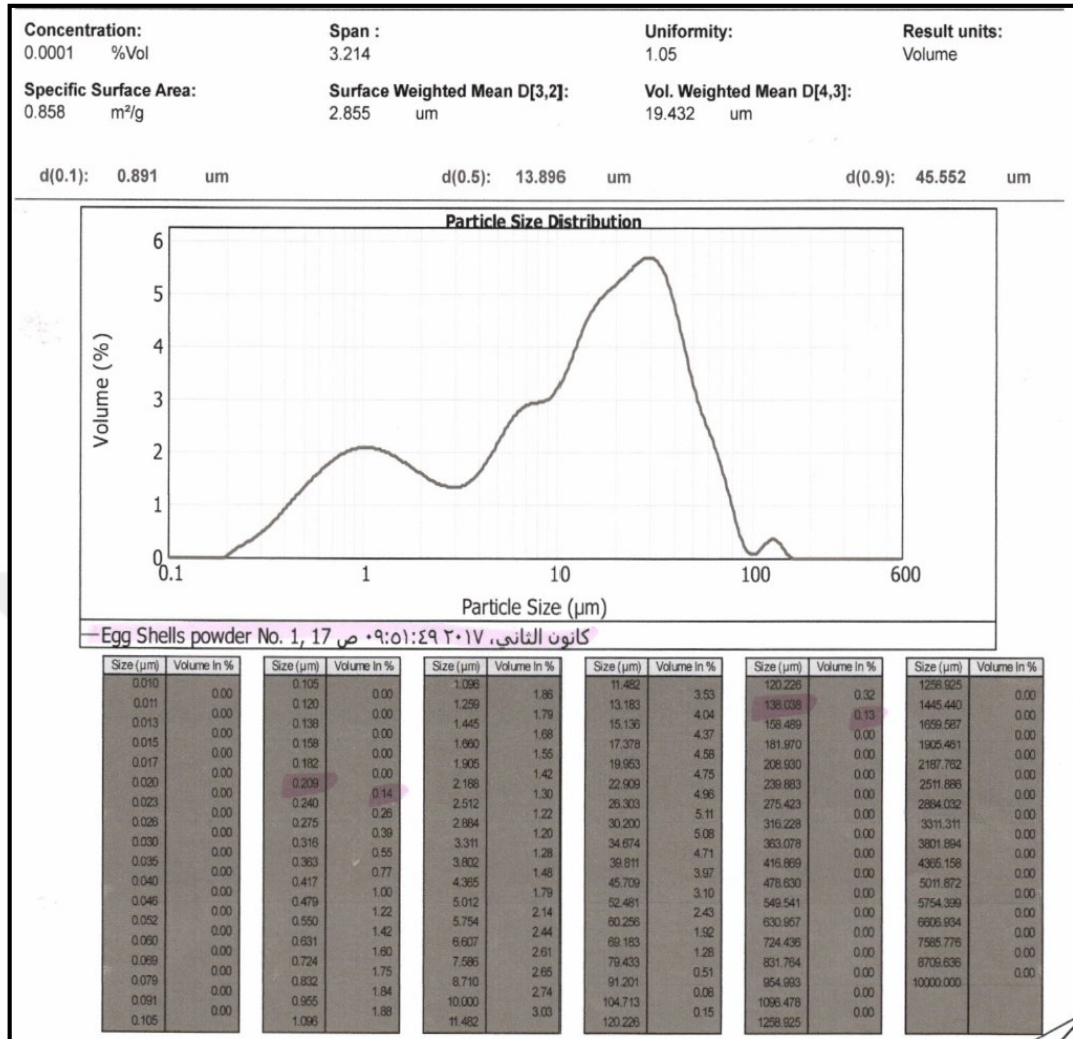


Figure 3.18: Particle size analyzer of (ES) powder before combustion.

3.4.4 Particle Size Analyzer of Eggshells Powder after Combustion

The particle size distribution of eggshell powder after combustion, as shown in Figure 3.19. From the figure can be seen the minimum particle size (0.1 µm) and the maximum particle size (120 µm), while the average particle size of eggshells powder equal (16 µm).

These all powders are expected to mixed homogeneously with the epoxy resin and glass fiber, providing reinforcement centers in the epoxy resin matrix at macro scale dimension.

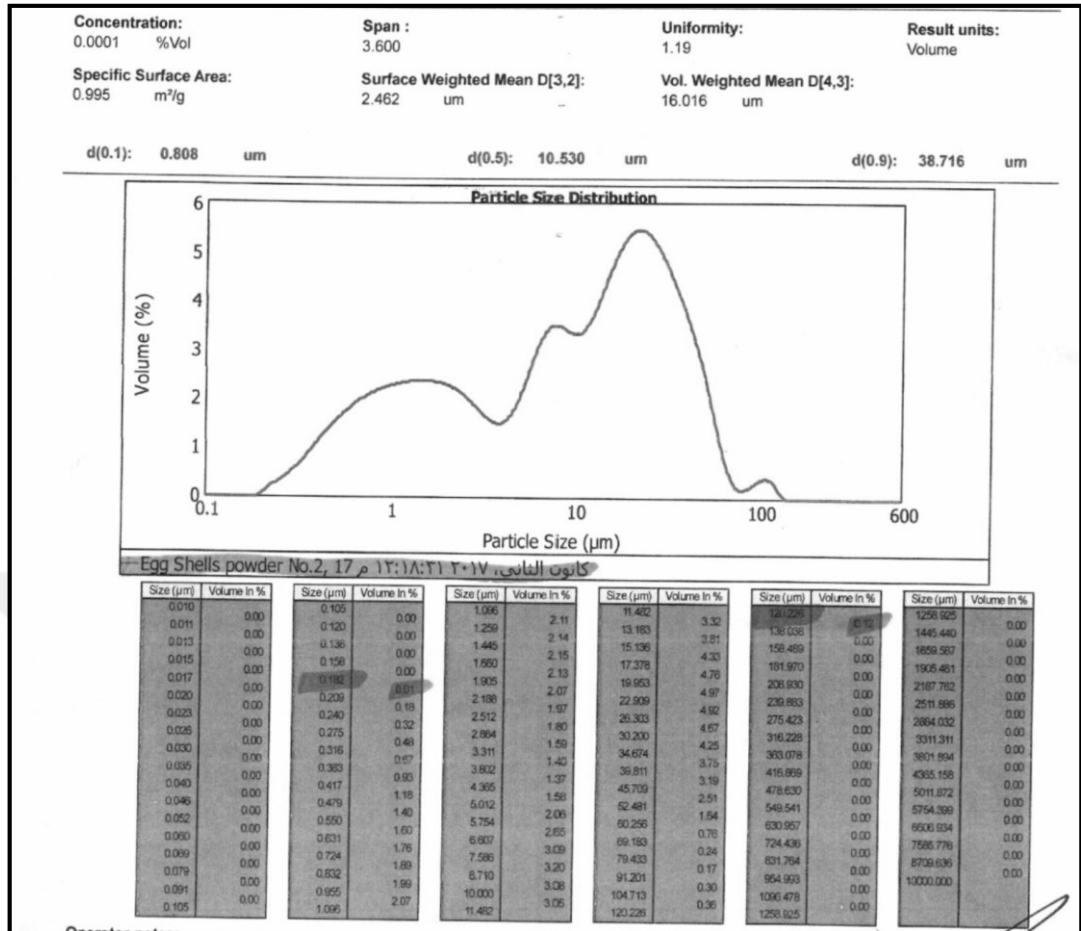


Figure 3.19: Particle size analyzer of (ES) powder after combustion.

3.5 X- Ray Fluorescence

3.5.1 X- Ray Fluorescence of RH Powder

The chemical composition of the rice husk by x-ray fluorescence is given in the Table 3.3 and Figure 3.20 shows the x-ray fluorescence device (EAIE) dispersive X-Ray fluorescence spectrometers, speed of Measurement from 10 to 300 second , specification of 60 Kn / 300 W ,Resolution of 123 Ev to 134 Ev , X- Ray source of 50 Kn / 50 W ,Long Battery life 10-12 hours, South Korea , Model: XEPOS, Type 78004814) examination was conducted at Ministry of Science and Technology / Materials Research Department .

Table 3.3: Chemical composition of rice husk by x-ray fluorescence.

Chemical Composition	Ratio %
Cellulose	35-50
Lignin	25-30
Silica	15-20
K ₂ O	0.52
CaO	0.47
Fe ₂ O ₃	0.30
Na ₂ O	0.12
MgO	0.23
P ₂ O ₃	0.02
Cr ₂ O ₅	0.005
MnO	0.08
Moisture	1-2

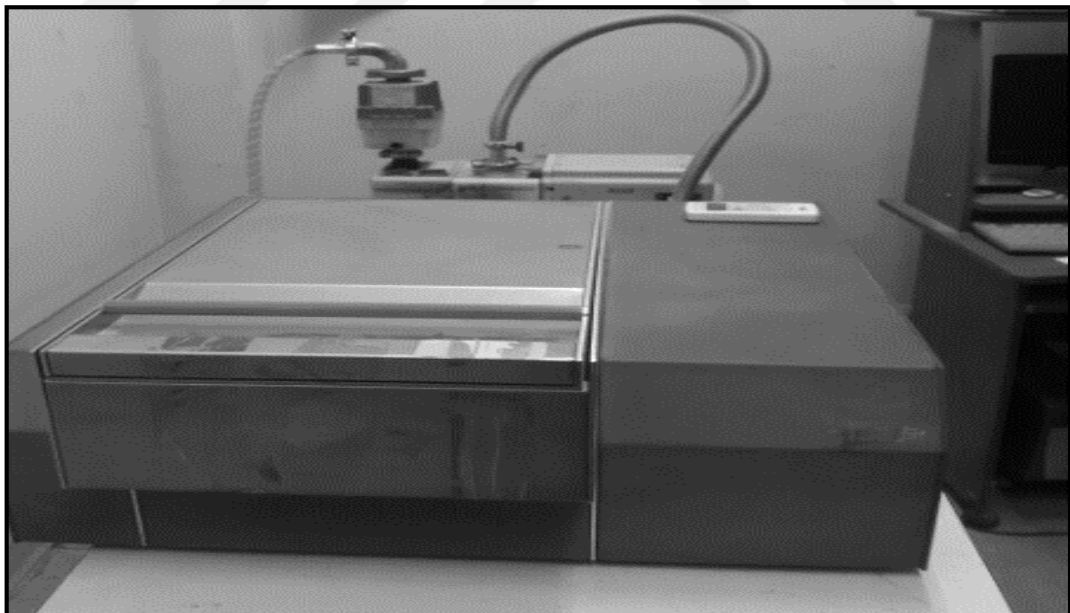


Figure 3.20: X-Ray Fluorescence Device.

3.5.2 X- Ray Fluorescence of RHA Powder after Combustion

Chemical composition of the rice husk ash powder after combustion by x-ray fluorescence is given in the table (3.4).

Table 3.4: Chemical composition of the rice husk ash powder after combustion by x-ray fluorescence.

Chemical Composition	Ratio %
Silica	87.9
Al ₂ O ₃	0.74
TiO ₂	0.20
K ₂ O	2.37
CaO	1.2
Fe ₂ O ₃	0.89
Na ₂ O	0.28
MgO	0.42
P ₂ O ₃	3.8
Cr ₂ O ₅	0.014
MnO	0.312
ZnO	0.11
ZrO ₂	0.02
Cr ₂ O ₃	0.014
Loss on Ignition	1.25

3.5.3 X- Ray Fluorescence of Eggshells Powder before Combustion

The chemical composition of eggshells powder before combustion by x-ray fluorescence is given in the Table 3.5.

Table 3.5: The Chemical composition of eggshells powder before combustion by x-ray fluorescence

Chemical Composition	Ratio %
CaO	52.30
SiO ₂	0.013
Al ₂ O ₃	0.018
MgO	0.048
Fe ₂ O ₃	0.05
Na ₂ O	0.24
P ₂ O ₃	0.30
SrO	0.19
NiO	0.0013
SO ₃	0.60
Cl	0.022
K ₂ O	0.09
MnO	0.029
TiO ₂	0.0025

3.5.4 X- Ray Fluorescence of Eggshells Powder after Combustion

The chemical composition of eggshells powder before combustion by x-ray fluorescence is given in the Table 3.6.

Table 3.6: The chemical composition of eggshells powder after combustion by x-ray fluorescence.

Chemical Composition	Ratio %
CaO	85
SiO ₂	0.035
Al ₂ O ₃	0.025
MgO	0.057
Fe ₂ O ₃	0.08
Na ₂ O	0.30
P ₂ O ₃	0.35
SrO	0.26
NiO	0.0020
SO ₃	0.69
Cl	0.031
K ₂ O	0.014
MnO	0.032
TiO ₂	0.0033
Loss on Ignition	12.89

3.6 X- Ray Diffraction

3.6.1 X- Ray Diffraction of RH Powder

XRD analysis of the rice husk powder was recorded from 10° to 90° range of 2 θ . The XRD pattern of the rice husk shown in Figure 3.21 indicates that, silica in the rice husk exists in the amorphous form only. Figure 3.22 shows the x-ray diffraction device (XRD-6000 Shimadzu Japan , frequency 220 v/50 HZ, powder 3 KW, current 80 MA, Volts 60KN, $n\lambda$ 2 dsin θ) ,the examination was conducted at the Ministry of Science and Technology / Department of Materials Research / Center for Advanced Materials.

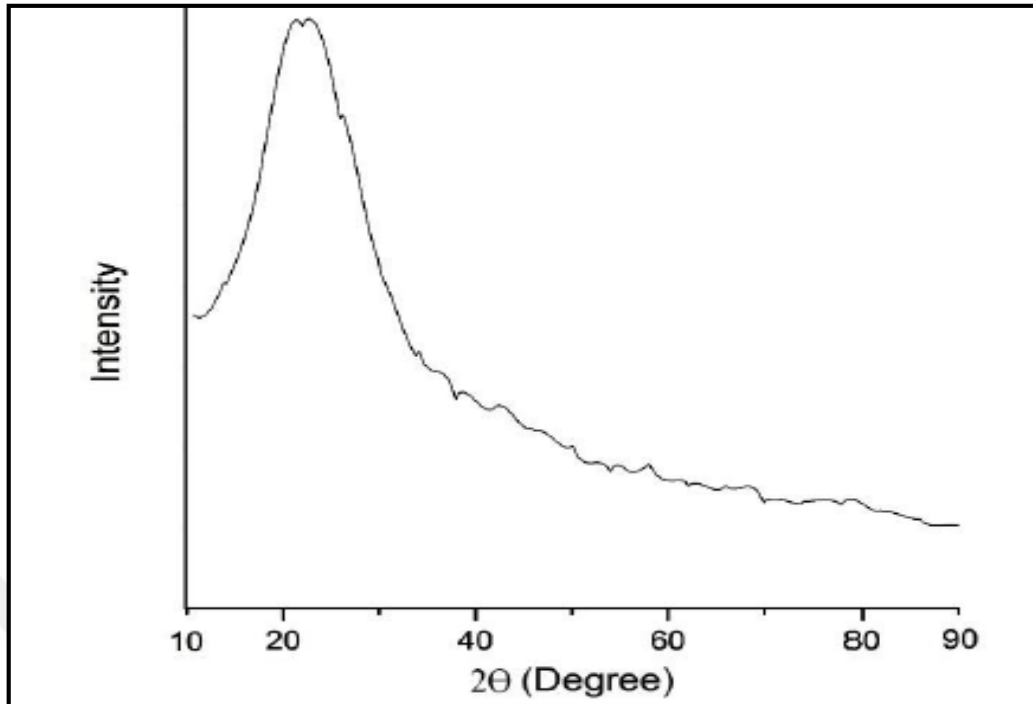


Figure 3.21: The X-ray diffraction of rice husk powder.



Figure 3.22: X-ray diffraction device.

3.6.2 XRD of RHA Powder after Combustion:

From Figure 3.23 can be seen sharp XRD peaks of RHA at 2θ was recorded (20.9° and 26.65°) indicate the presence of silica in crystalline form. Tridymite appeared at 20.966° and cristobalite appeared at 26.651° .

These XRD peaks indicate that the rice husk ash has mixed phases of both forms of crystalline silica. At calcination temperatures above 900C° the SiO₂ in rice husk ash consists of some triadite and cystopalite phases due to the solubility of silica ash particle surfaces and particle bonding together [95]. It was observed that at a temperature of about 1000 C° the rice husk convert to ash with the crystalline silica, 83% of rice husk convert to crystalline silica at temperature 1350C°

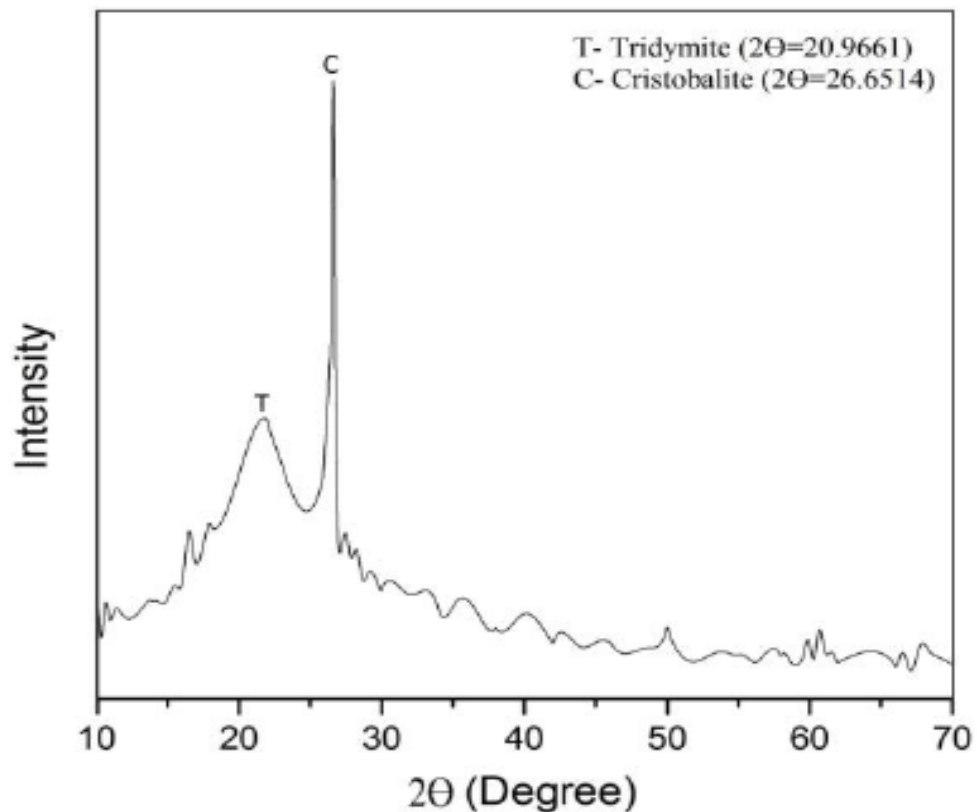


Figure 3.23: The X-ray diffraction rice husk ash powder after combustion.

3.6.2 X- Ray Diffraction of Eggshells Powder before Combustion

Figure 3.24 shown the X-ray diffraction analysis of eggshells powder before combustion at 2θ was recorded from 20° to 80° range. Of the shape can be observed at (29°,30°,47°,49°) found CaO exists in the crystalline form .

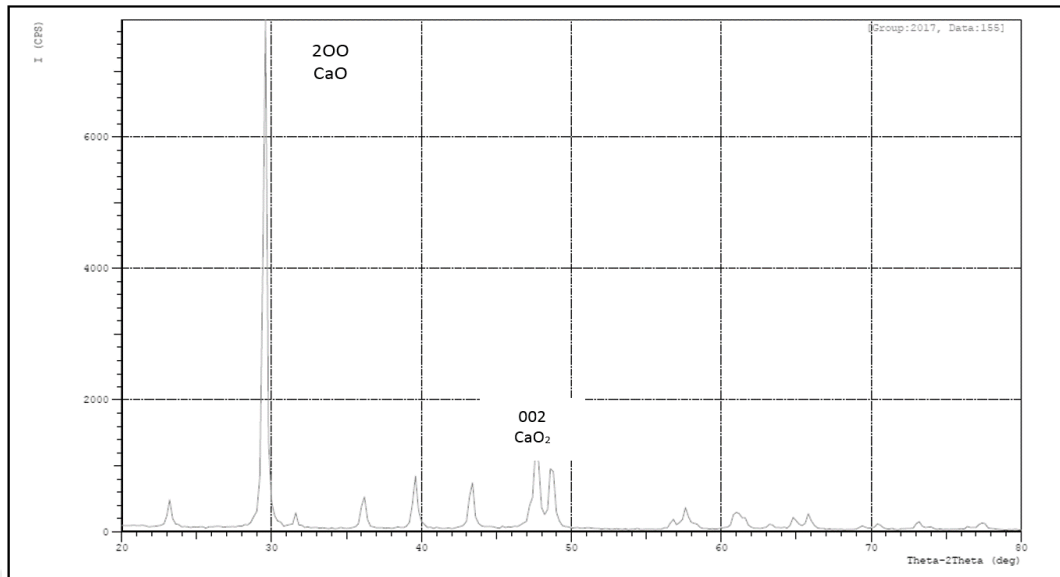


Figure 3.24: X-ray diffraction of eggshells powder before combustion.

3.6.2 X- Ray Diffraction of Eggshells Powder after Combustion

Figure 3.25 shows the X-ray diffraction analysis of eggshells powder after combustion at 2θ was recorded from 20° to 80° range. Of the shape can be observed at ($29^\circ, 30^\circ, 40^\circ, 47^\circ, 49^\circ$) found CaO exists in the crystalline form more than before combustion.

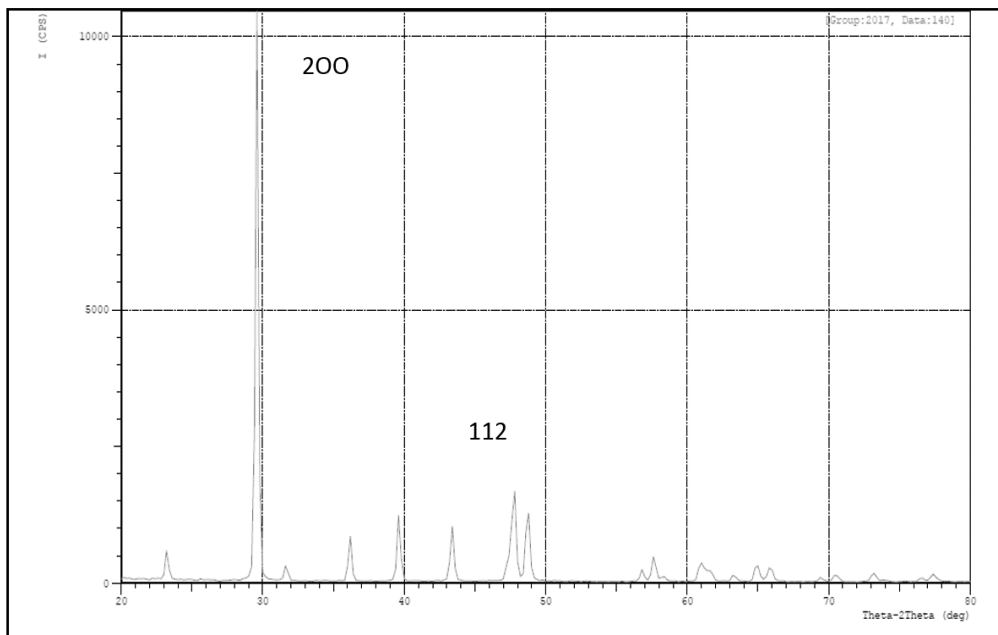


Figure 3.25: X-ray diffraction of eggshells powder after combustion.

3.7 Mould Preparation

The molds for the preparation of the samples were made of glass by dimension (130×130×5 mm) .The internal face of the mould has been enveloped with a layer of nylon (nylon thermal paper) made from poly vinyl alcohol (PVA) instead Vaseline to ensure no-adhesion of the resin with the mould. Figure 3.26 shows the shape of the prepared mould.

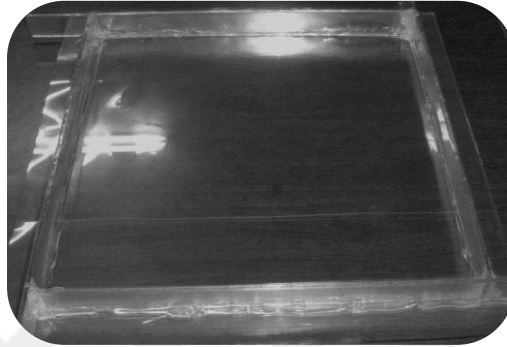


Figure 3.26: The shape of the prepared mould.

3.8 Preparation of Samples

The methods of preparing the composite materials are many and each of them has its advantages and disadvantages. The Hand lay-Up Molding was used in the preparation of the samples in this research, it is simple to use and can make different shapes and sizes of composite materials .The volume and weight fraction of the components is calculated depend on the following relations [96].

$$v_f = \frac{m_f}{\rho_f} \quad (3-1)$$

$$V_f = \frac{v_f}{v_c} \quad (3-2)$$

$$v_m = \frac{m_m}{\rho_m} \quad (3-3)$$

$$V_m = \frac{v_m}{v_c} \quad (3-4)$$

Where:

m_f, m_m : Mass of fiber and matrix materials, respectively. (gm)

v_c, v_m, v_f : Volume of (composite, matrix and fiber) materials respectively, (cm³)

ρ_f, ρ_m : Density of fiber and matrix materials respectively, (gm / cm³).

V_f, V_m : Volume fraction of fibers and matrix materials ,respectively.

Composites are prepared according to the following steps:

1. Preparation of glass fibers woven of dimensions (130 × 130) mm according to the dimensions of the mould. The used weight fractions are (3%).
2. Weighing the reinforcing natural powder to specify a weight fraction of (1%, 3%, 6% and 9%).
3. Weighing the epoxy resin depending on the weight fraction of reinforcement materials (fiber and natural powder), while taking into consideration the weight of hardener.
4. Mixing the epoxy resin with the hardener continuously and slowly by using a glass rod to avoid bubbles, the mixing is carried out at room temperature.
5. Add the powder gradually to the mixture and wait for 10-15 minutes to obtain a homogeneous mixture. A rise in the temperature of the mixture will result as an indication to the beginning of the interaction process. It is very important that the mixture must have a good viscosity for the purpose of protecting the particles from precipitation which may result in the heterogeneity of the mixture that leads to the agglomeration after hardening.
6. Pouring the mixture into the mould, then putting the glass fiber mat into the mould and continuing of mixture pouring until it covers the entire mat.
7. Pressing the mixture with an appropriate load.
8. Leave the sample in the mold for 24 hours at room temperature, for the purpose of completing the sample hardening.
9. The samples extracted from the mold are treated with heat in the oven at (60c°) for a period of (60) minutes [97]. This process is very important for the purpose of obtaining the best cross linking between polymeric chains, and to remove the stresses generated from the preparation process and complete the full hardening of the samples. Figure 3.27 shows some of the prepared samples.

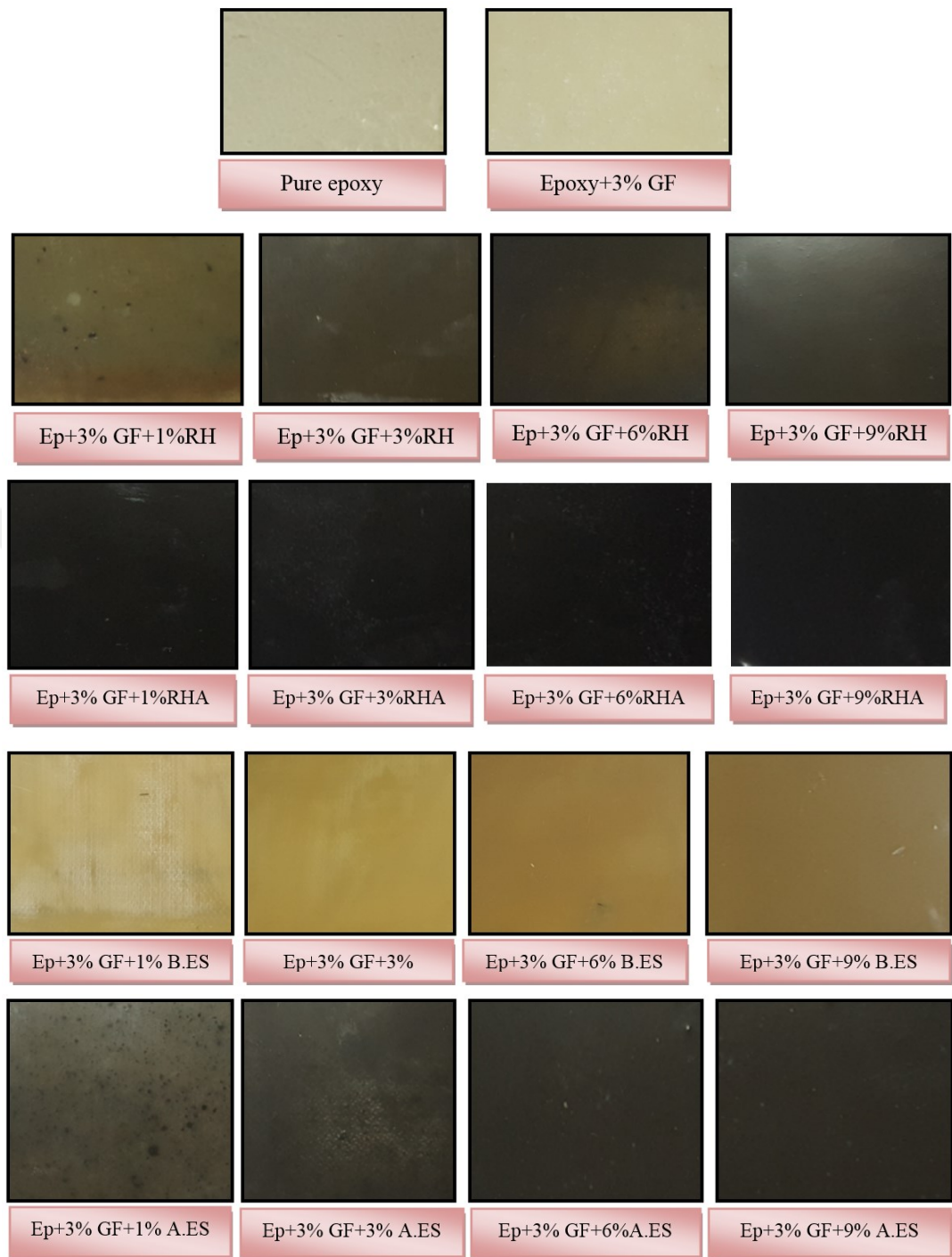


Figure 3.27: Composite samples prepared by this research.

3.9 Cutting and Preparing the Samples for Examinations

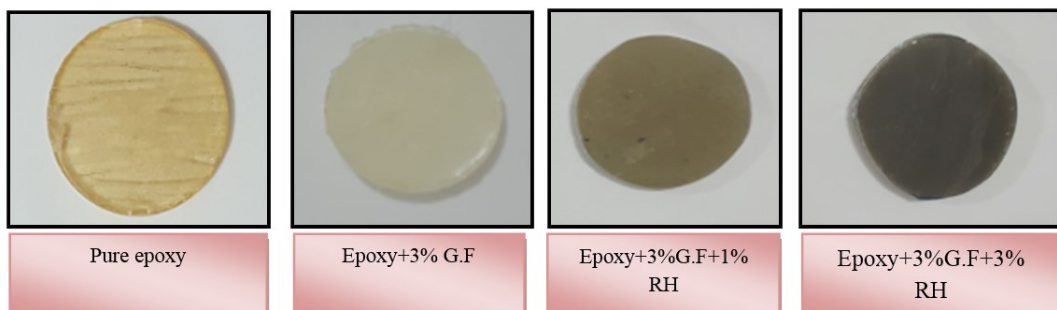
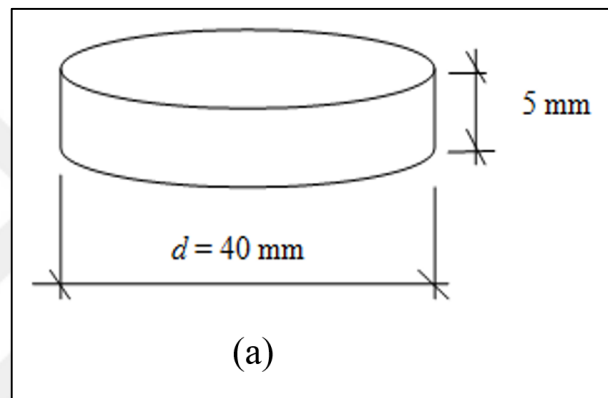
After obtaining the samples of (pure epoxy, epoxy +3% glass fiber and RH , RHA , B.ES , A.ES) natural composite materials that are prepared in the form of plates with dimensions (130*130*5) mm, they have been cut and machined to obtain the samples according to the international standards for each test. The cutting and

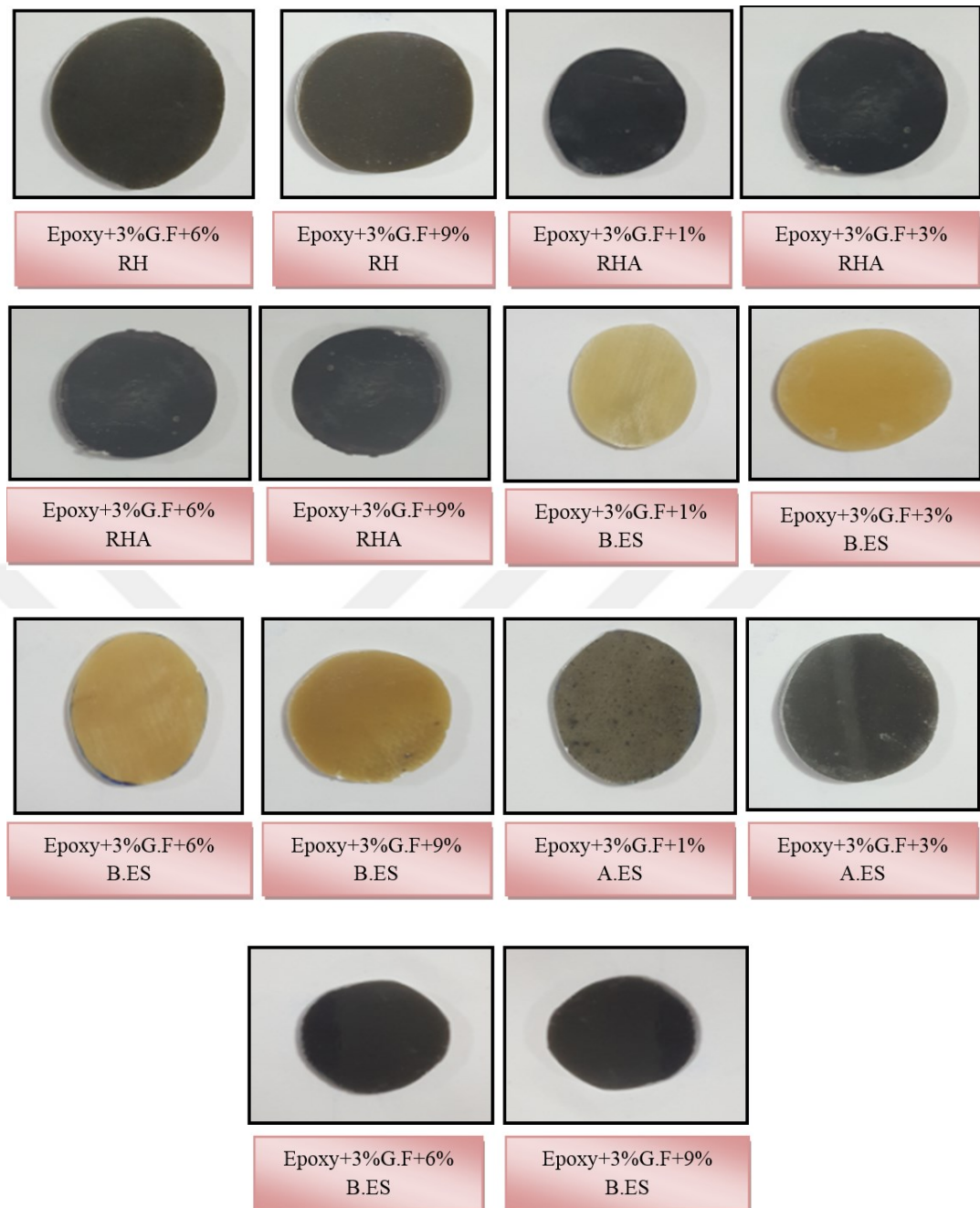
machining processes are very soft to ensure no vibration during cutting the samples, and to avoid distortions that may occur during the process.

3.10 Physical Tests

3.10.1 Density

This test is performed according to (ASTM D792) standard [75]. The samples were cut to a diameter of 40 mm and a thickness of 5 mm as shown in Figure 3.28. Samples should be tested in the air and weighed when immersed in distilled water. Density can be obtained by applying Equation (2.10)



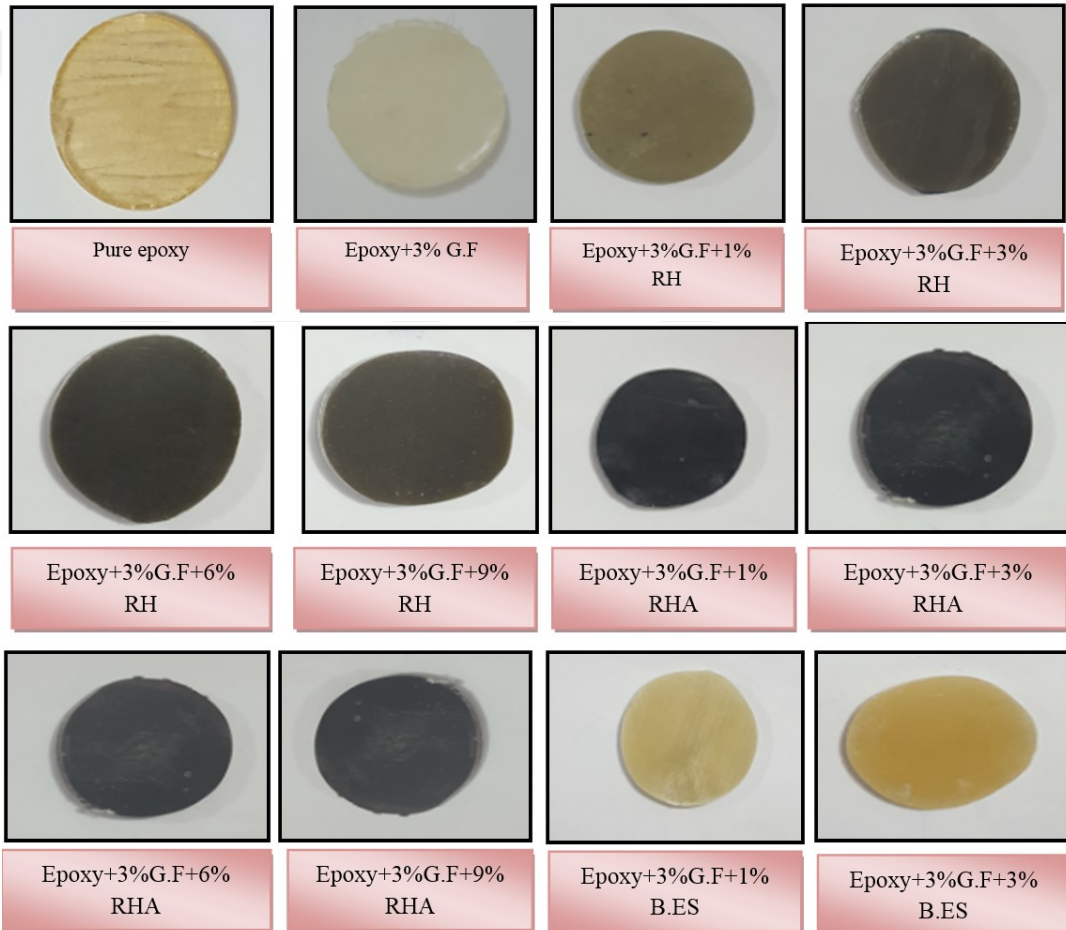
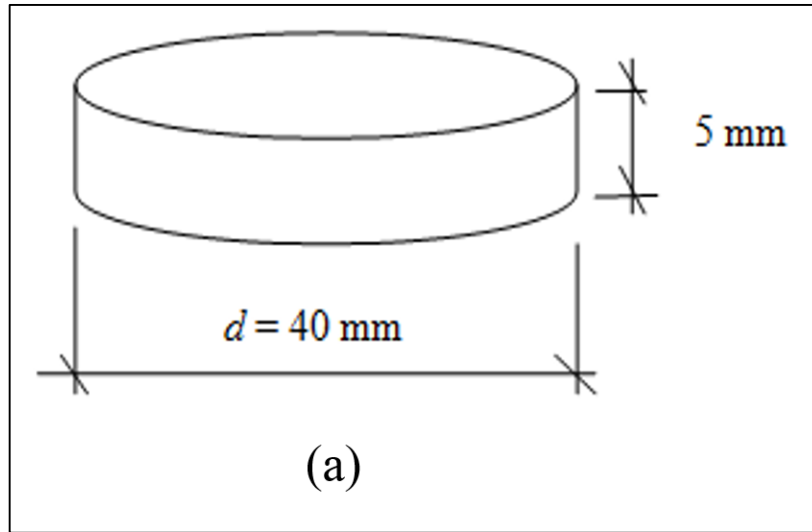


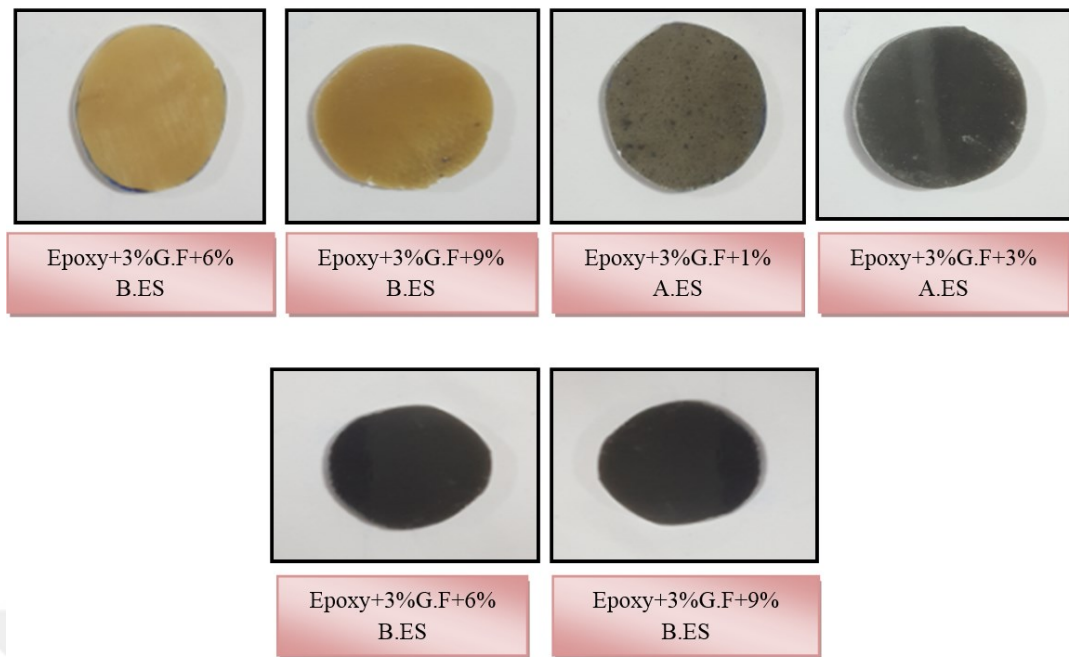
(b)

Figure 3.28: Standard specimens (a) Schematic samples [75], (b) Experimental samples.

3.10.2 Water Absorption

This test was done according to the standards (ASTM D 570) at room temperature [78]. Samples were cut in diameter (40 mm) with thickness (5 mm), the test sample is shown in Figure 3.29.



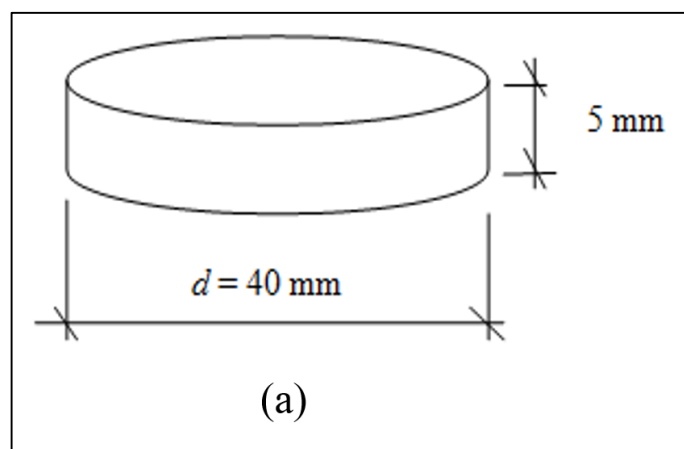


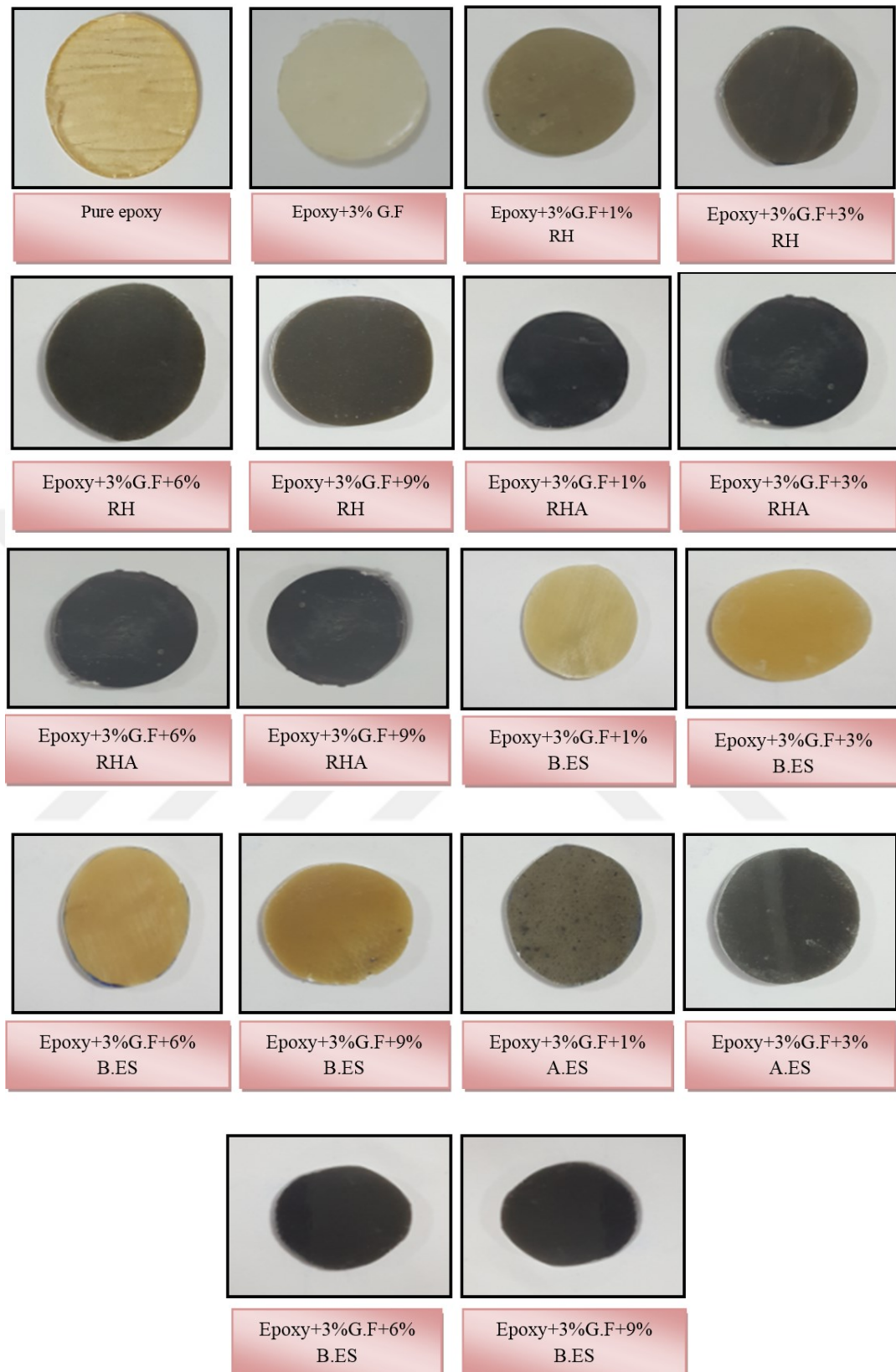
(b)

Figure 3.29: Standard specimens [78]. (a) Schematic samples, (b) Experimental samples.

3.10.3 Thermal Conductivity Test

Thermal conductivity test was carried out on test specimens shown in Figure 3.30 [98]. One of the most commonly techniques for studying thermal transport properties (thermal conductivity, thermal diffusivity and specific heat per unit volume) is the transient plane source (TPS- 500) method, were measured by using (Hot Disk Thermal Constant Analysis) Figure 3.31 shows the thermal conductivity test machine used to test polymer-based composite materials.





(b)

Figure 3.30: Specimens of thermal conductivity test: (a) Schematic specimen, (b) Experimental samples.

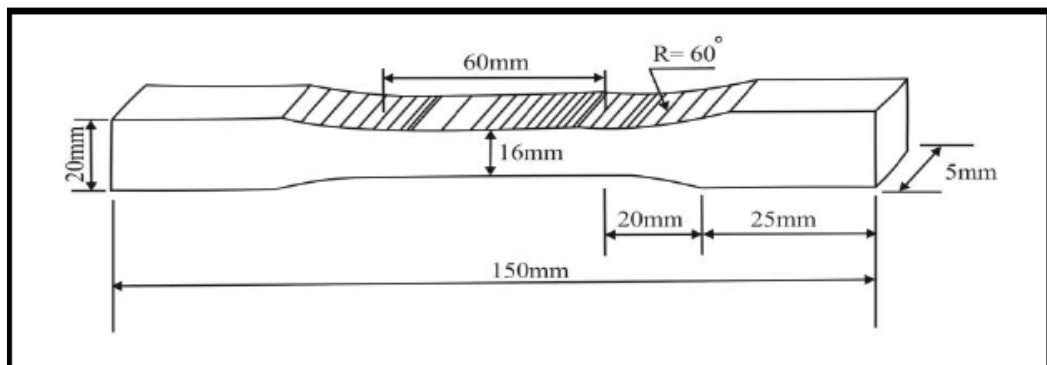


Figure 3.31: Thermal conductivity test machine.

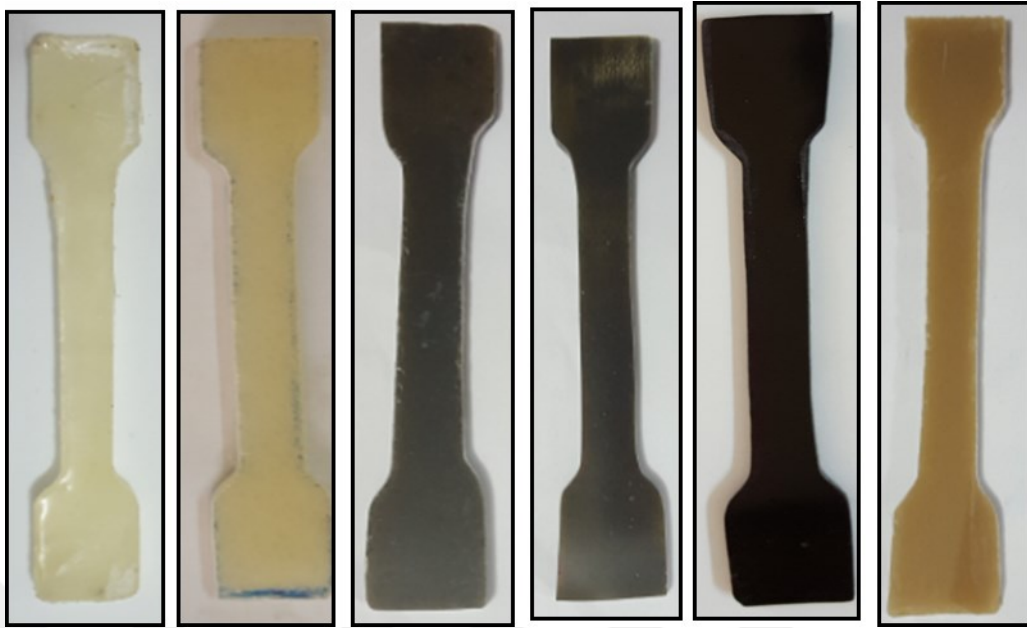
3.11 Mechanical Tests

3.11.1 Tensile Test

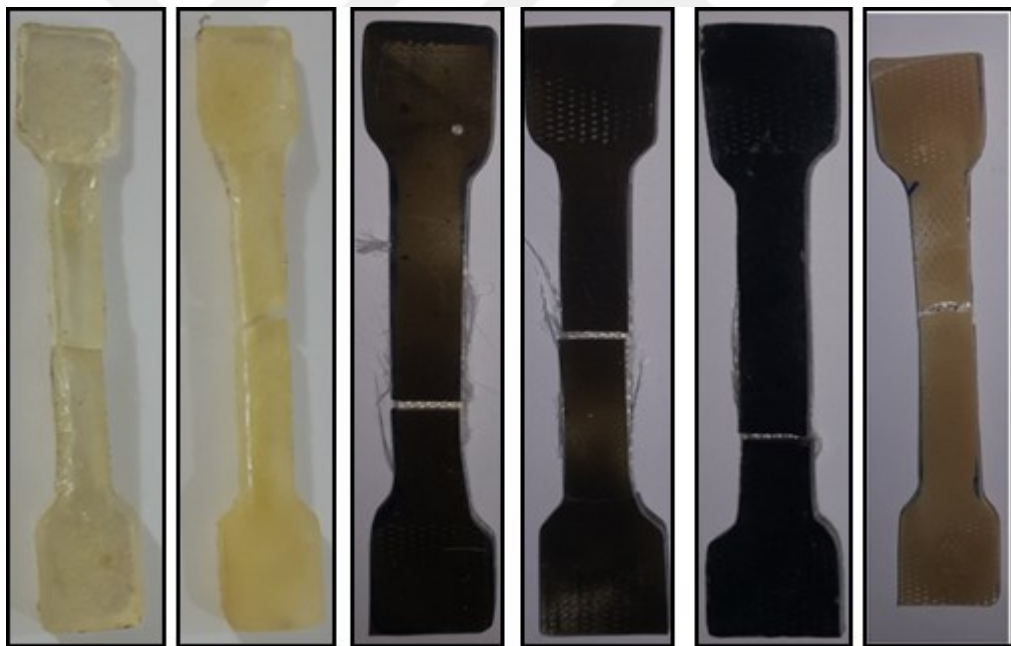
The tensile test is performed depending in (ASTM D638-03) [80] using a tensile machine universal testing machine, type (Instron) on the head of the cross speed (strain rate) (5mm/min) and load was applied equal to (5 kN) until break the specimen occur. Figure 3.32 shows the standard specimen of tensile test. Figure 3.33 shows tensile device located in (Resistance) Laboratory, Materials Engineering Department, University of Technology.



(a)



(b) Before test



(c) After test

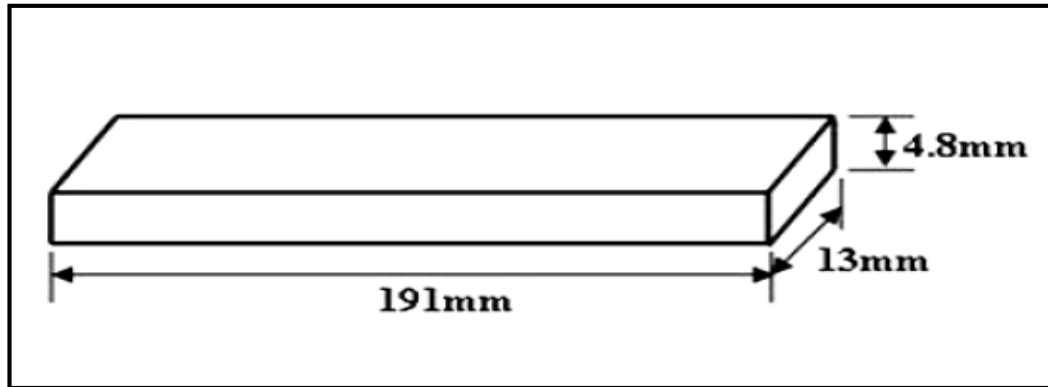
Figure 3.32: Standard specimen.
(a) Schematic samples [80].
(b) Experimental samples before test.
(c) Experimental samples after test.



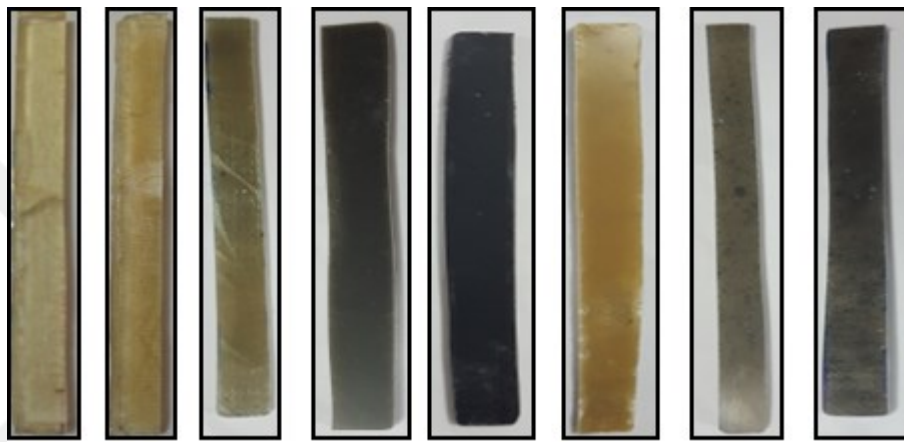
Figure 3.33: Tensile device.

3.11.2 Flexural Test

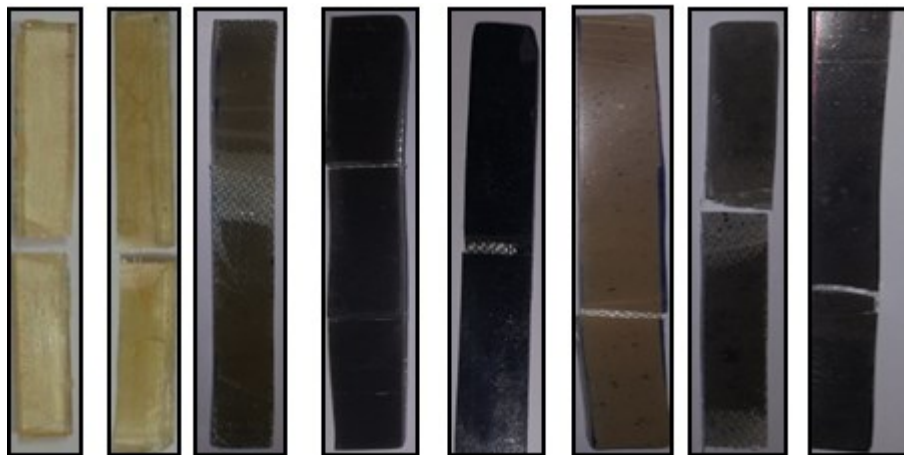
This test was performed according to ASTM D790 [99] at room temperature. Standard specimen for flexural test as show in Figure 3.34. Figure 3.35 show the device used in this test (applied load 0-45 KN by using the machine type laryee Chinese made model 1031), located in (Resistance) Laboratory, Materials Engineering Department, University of Technology.



(a)



(b)



(c)

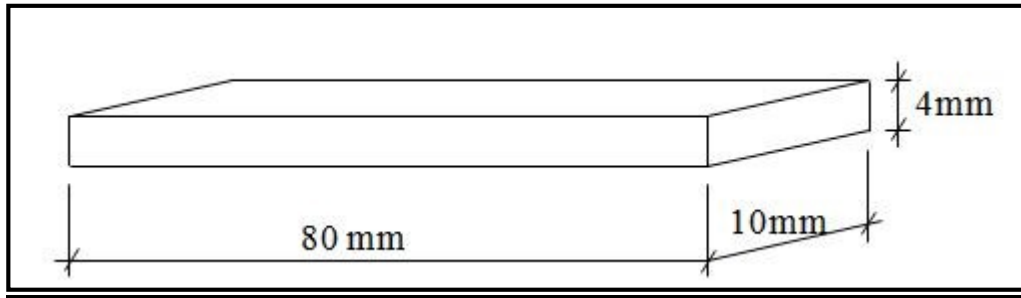
Figure 3.34: Standard specimens.
(a) Schematic samples [99].
(b) Experimental samples before test.
(c) Experimental samples after test.



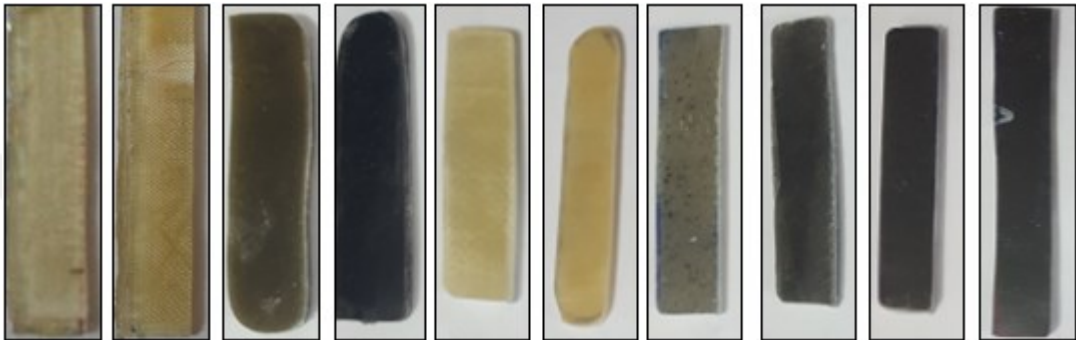
Figure 3.35: Flexural strength and shear stress device.

3.11.3 Impact Test

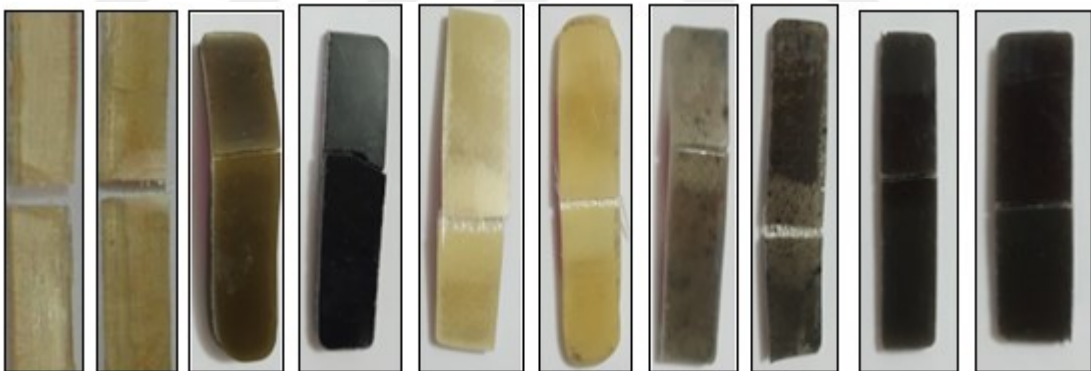
This test was performed according to ISO-180 at room temperature. Standard specimen for impact test as shown in Figure 3.36 [100]. Calculate the impact strength and fracture toughness depends on the calculation of the energy required for the break. Figure 3.37 show the Izod impact test machine used for testing polymeric materials.



(a)



(b) Before test



(c) After test

Figure 3.36: Standard specimens.
 (a) Schematic samples [100].
 (b) Experimental samples before test.
 (c) Experimental samples after test

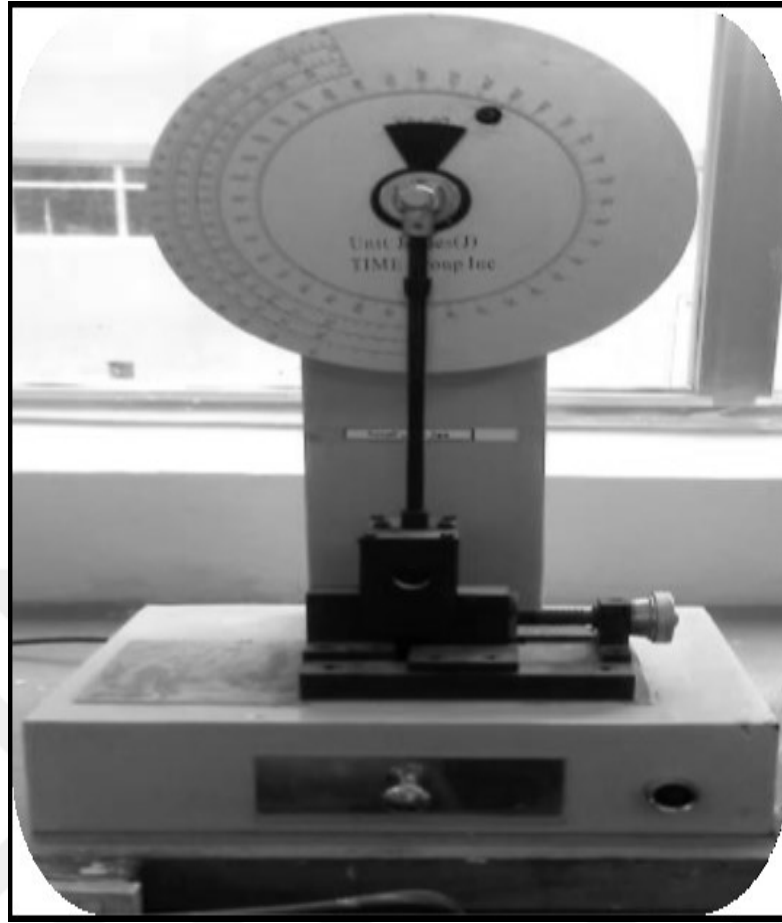
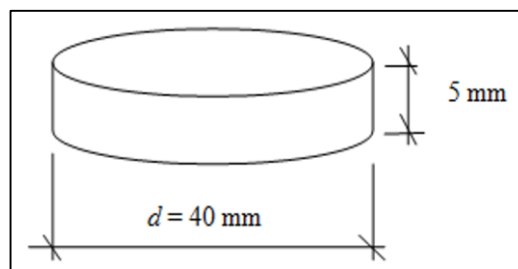


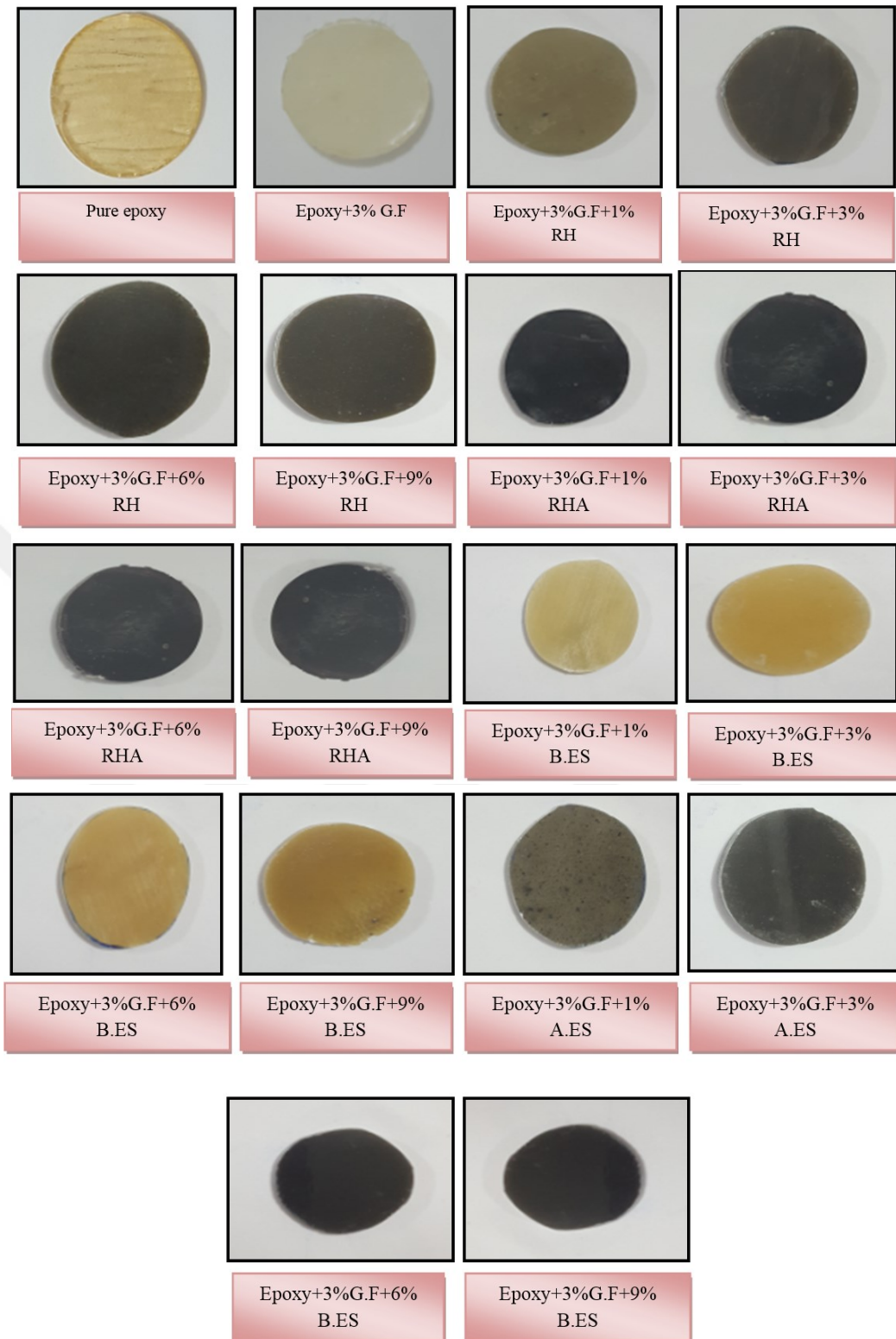
Figure 3.37: Impact test device.

3.11.4 Hardness Test

This test is performed using hardness (shore D) according to ASTM standard (D-2240) at room temperature. The samples were cut in diameter (40MM) with a thickness (5mm). Standard specimens for this test as shown in Figure 3.38 [101]. Figure 3.39 shows hardness shore (D) device located in (Polymer) Laboratory, Material Engineering Department, and University of Technology. The average hardness was calculated by taking five measurements hardness (shore D).



(a)



(b)

Figure 3.38: Hardness test specimen.
 (a) Schematic specimen [101],
 (b) Experimental specimens.



Figure 3.39: Hardness shore (D) device.

CHAPTER FOUR

DISCUSSION OF RESULTS

4.1 Introduction

This chapter contains the results of different physical and mechanical tests for all the prepared composites, and discusses the effect of each material on the obtained values of density, water absorption, thermal properties, tensile properties, flexural properties, maximum shear stress, impact strength, fracture toughness and hardness shore (D).

4.2 Physical Tests

4.2.1 Density

Density is an important physical property in many weight sensitive applications. Polymer-matrix composite materials have been used in many applications to replace metals and conventional materials primarily for low densities. The density of the composite depends on the relative ratio of the matrix resin and reinforcing materials. The voids and pores have a significant effect on mechanical properties and even on composite performance [102]. More voids were found in composite materials with fiber addition as well as filler [21]. Figure 4.1 shows the result value density for specimens (pure epoxy and epoxy +3% glass fiber). From Figure 4.1 it can be seen that the specimen (epoxy +3% glass fiber) has a higher density value than the specimen (pure epoxy), due to the density of glass fiber (2.58 gm/cm^3) is higher when compared to the density of pure epoxy where the density of epoxy resin is (1.05 gm/cm^3) [103].

Figure 4.2 and 4.3 show the result value density for specimens (Ep+3% G.F+1%, 3%,6%,9% rice husk, rice husk ash and before, after burning eggshells). In Figure 4.2 the specimen (epoxy+3% glass fiber +9% RH) has a lower density value than the specimen (epoxy+3% glass fiber +9% eggshells), because the eggshells have (1.041 gm/cm^3)

higher individual density when compared with the density of the rice husk (0.654 gm/cm^3). In Figure 4.3 the specimen (epoxy+3% glass fiber +9% RHA) has lower density value than (epoxy+3% glass fiber +9% eggshells), because the eggshells have (0.7416 gm/cm^3) higher individual density when compared with the density of the rice husk ash (0.462 gm/cm^3). In the preparation of composite materials of one or more of the two substances is difficult and effect directly on the shrinkage of the base material. Where increasing the weight fraction of the reinforcement material creates more voids that represent the locations for stress concentration. Also the value density depends on the good distribution and bonding of all constituents. Figure 4.4 shows the specimen (ep+3% g.f+9% RHA) has a lower value density than other specimens because the value density of RHA lower than (RH, Eggshells).

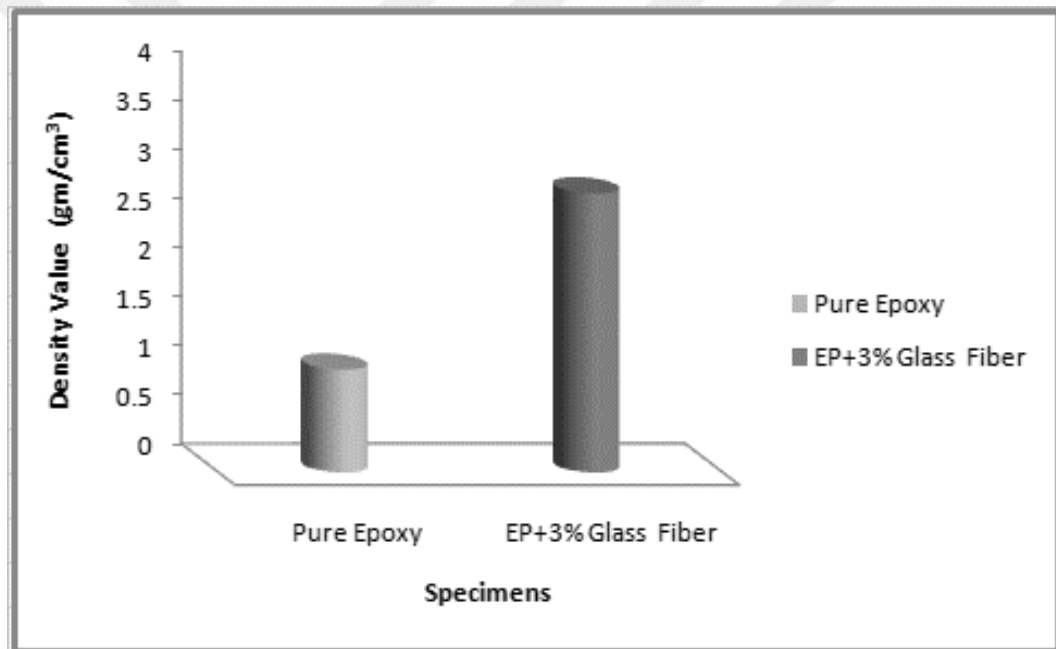


Figure 4.1: Density value of pure epoxy and epoxy +3% glass fiber.

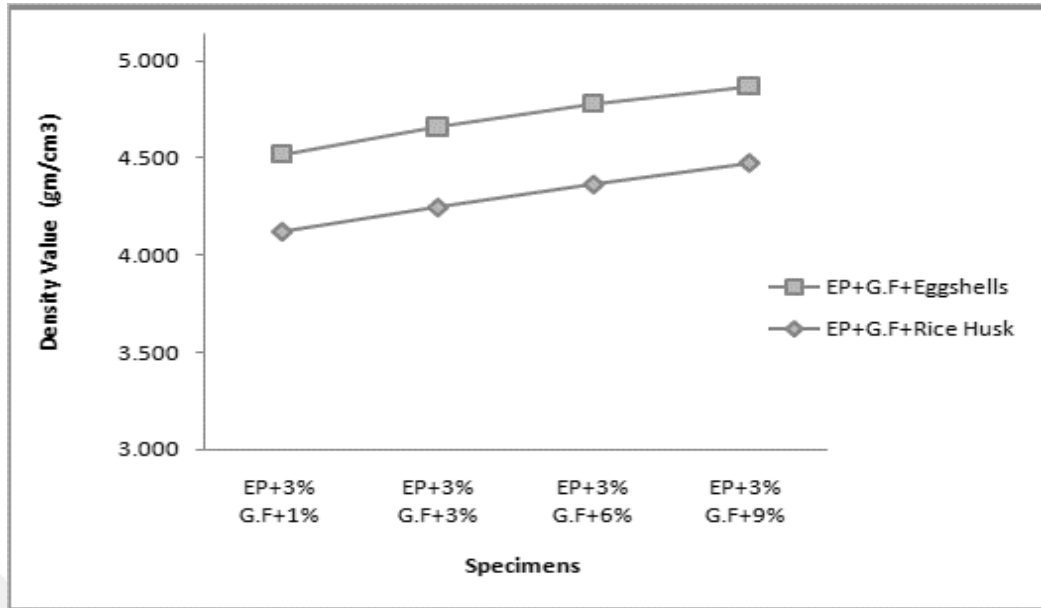


Figure 4.2: Density value of specimen reinforced with (glass fiber, before burning rice husk and eggshells).

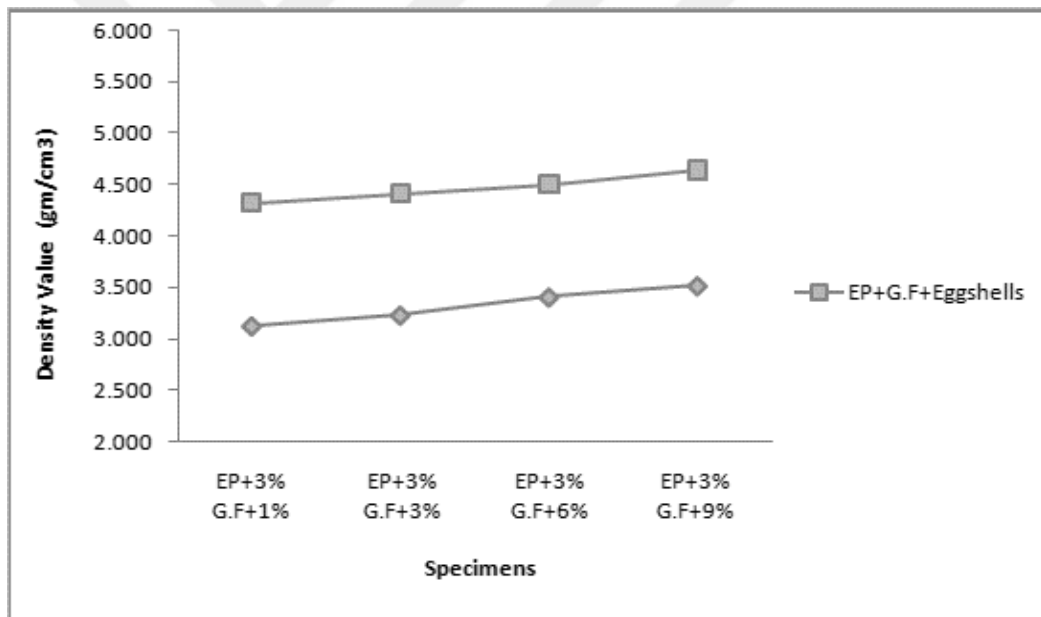


Figure 4.3: Density value of specimen reinforced with (glass fiber, after burning rice husk ash and eggshells).

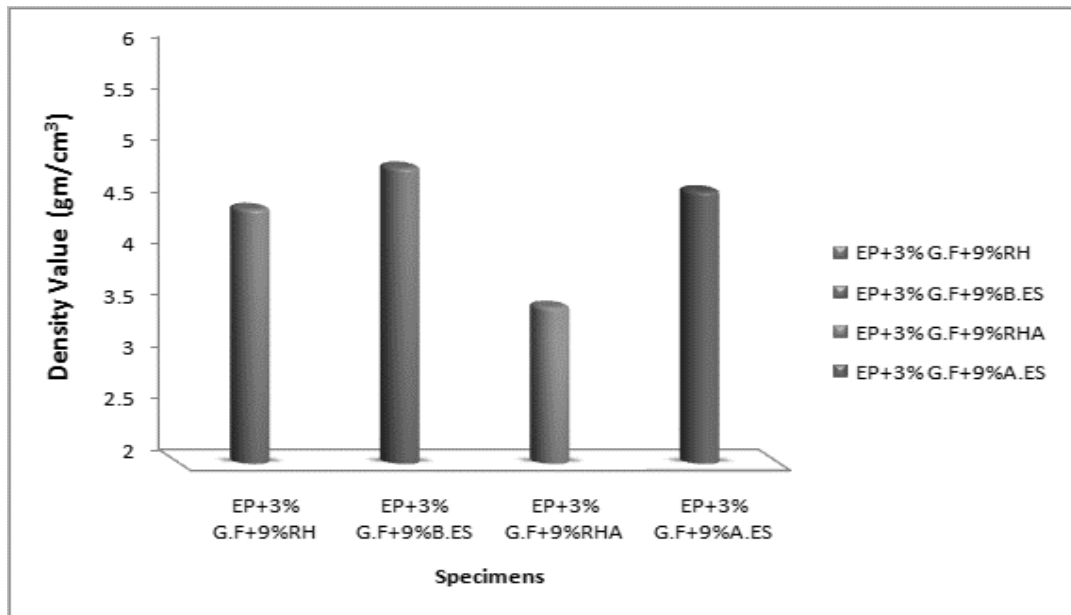


Figure 4.4: Comparing the density value of composite materials reinforced with 9% for all specimens.

4.2.2 Water Absorption

The mechanical properties such as resistance and durability decrease when the sample is exposed to moisture, or after absorption of a certain amount of water. Figures (4.5),(4.6),(4.7) and (4.8) show the values of water absorption for the prepared specimens (pure epoxy, epoxy +3% glass fiber, 1%,3%,6%&9% before and after burning for rice husk, rice husk ash, eggshells) composites. In Figure 4.5 can be seen the specimen (epoxy +3% glass fiber) has value water absorption high than specimen (pure epoxy), due to the absorption of water is increased with increasing weight fraction of the reinforcement. Water absorption also depends on the rule of mixture theory, Where reinforced has a higher water absorption ratio than the resin matrix. Water absorption attacked interface between the matrix and fibers its causes de - connectivity between the fiber and matrix, where porosity and voids cause failure of composite materials [104]. Figure 4.6 shows the water absorption values of the specimens (Ep+3% g.f+1%,3%,6% and 9% before burning rice husk and eggshells) , where the specimen (epoxy +3% glass fiber +9% RH) has a higher water absorption value than specimen (epoxy + 3% glass fiber +9% eggshells) . Figure 4.7 shows the water absorption for specimens (Ep+3% g.f+1%, 3%,6% and 9% after burning rice husk ash and eggshells). From Figure 4.7 can be seen the specimen (epoxy +3% glass fiber +9% eggshells) has a high water absorption percentage than specimen (epoxy+3%glass fiber+9%RHA).

When comparing the values of water absorption for all of the prepared specimens reinforced with 9% (B.RH, RHA, B.ES and A.ES) as indicates in Figure 4.8 find the specimen (epoxy+3%glass fiber+9%RHA) has value of water absorption lower than other specimen. This is due the reinforcing material absorbs large amounts of water compared to the matrix resin material, thus causes a weak interface between the matrix and strengthening and leads to a reduction in the transfer of stress to the reinforced materials and decrease resistance and toughness [105].

In this work the increase or decrease in water absorption values of composite materials depends on the average particle size of the powder enhanced. Because the saturation level of filler matrix composition influenced by agglomeration which will affect the water absorption ratio of the composite material. Where mean particle size of the RH is (53.7 μm), mean particle size of the RHA is (10.4 μm), mean particle size of the Eggshells before burning is (19.4 μm) and eggshells after burning is (16 μm), so the specimen is reinforced with (RHA) has lower water absorption values.

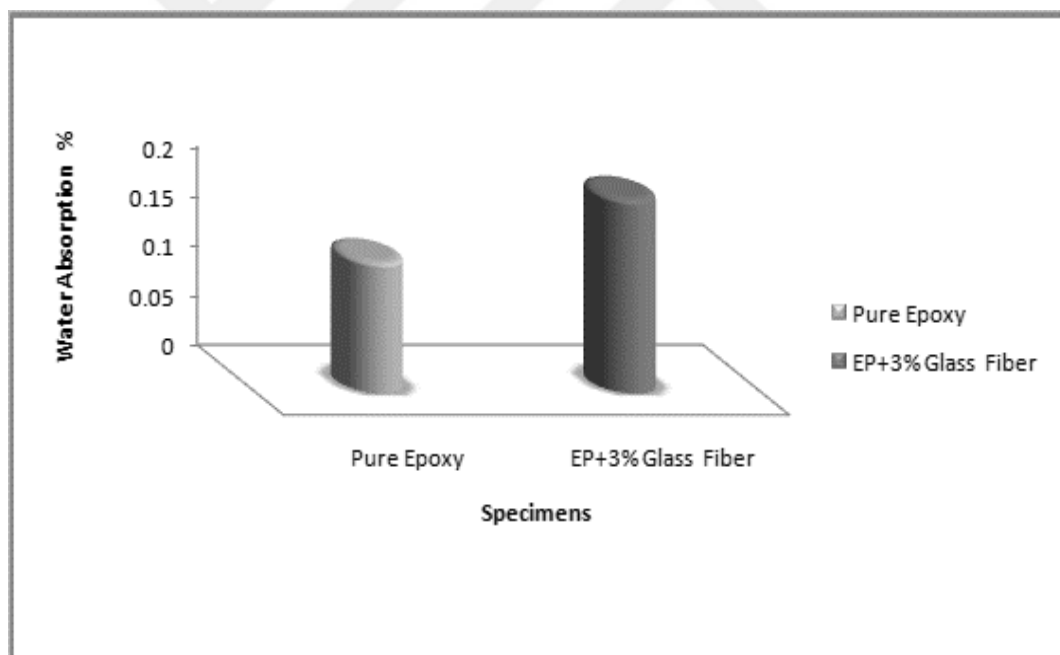


Figure 4.5: Water absorption of pure epoxy and epoxy +3% glass fiber.

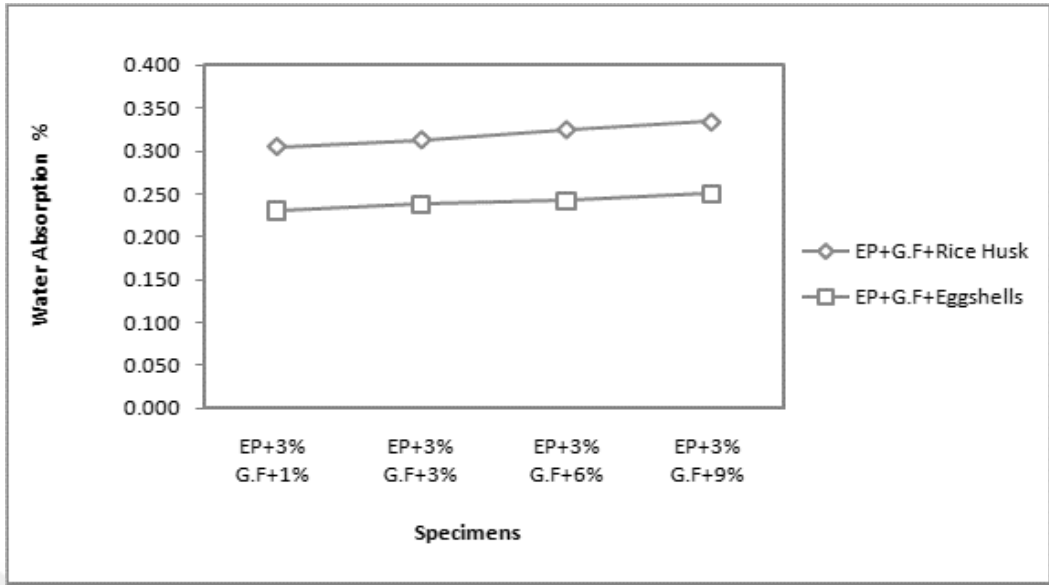


Figure 4.6: Water absorption of specimen reinforced with (glass fiber, before burning rice husk and eggshells).

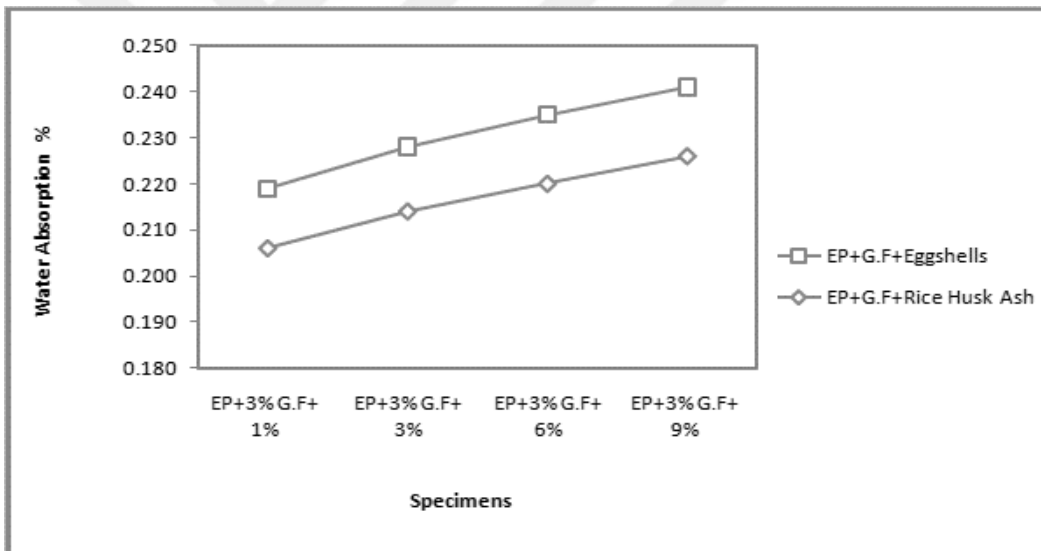


Figure 4.7: Water absorption of specimen reinforced with (glass fiber, after burning rice husk ash and eggshells).

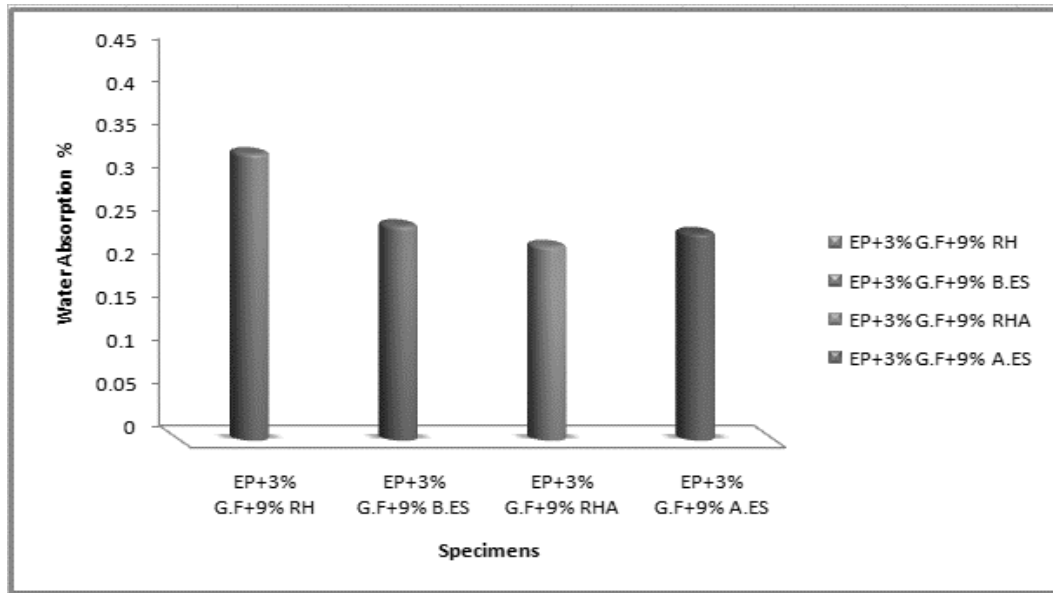


Figure 4.8: Comparing water absorption of composite materials reinforced with 9% for all specimens.

4.2.3 Thermal Tests

4.2.3.1 Thermal Conductivity

Thermal conductivity of composite materials depends on the frequency of interactions between the lattice atoms, particle size of the airspace and the nature of bonding in solid material. They also depend on the amounts and properties of the component composite. Figures 4.9, 4.10, 4.11 and 4.12 shows the values of thermal conductivity for the prepared specimens (pure epoxy, epoxy +3% glass fiber, before, after burning rice husk, rice husk ash and eggshells) composite material. It can be observed that the thermal conductivity values increased with increasing weight fraction for reinforcing materials (fibers and powders). That is due to the fact these natural powders have high thermal conductivity value compared with the epoxy resin matrix [79]. In addition, the presence of these particles work to fill or reduce the spaces and voids that were inside with epoxy resin matrix, thus facilitate the process of the heat transfer through composite specimens. From these figures can also be observed that the addition of eggshells powder before burning have a noticeable influence on the thermal conductivity of composite specimens more than rice husk ash powder. Thus, thermal conductivity values increased from (0.21W / m.K) to epoxy resins to (0.61 W/m.K) for (epoxy+3%g.f+9% A.ES) composite. This is due to the thermal conductivity of eggshells powder is greater than the thermal conductivity of RHA powder.

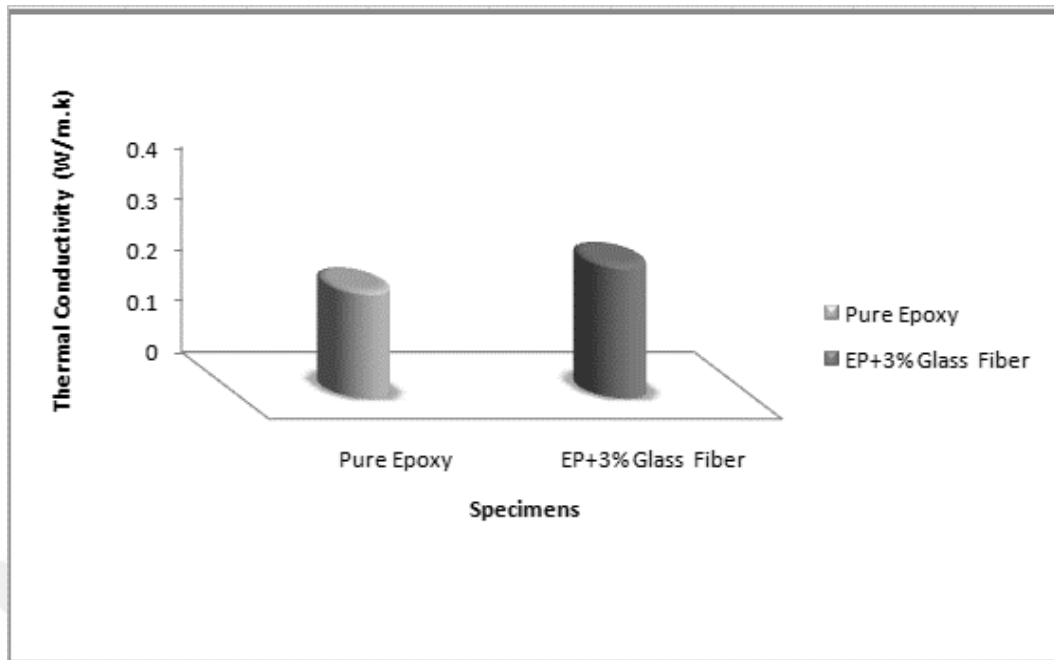


Figure 4.9: Thermal conductivity of pure epoxy and epoxy +3% glass fiber.

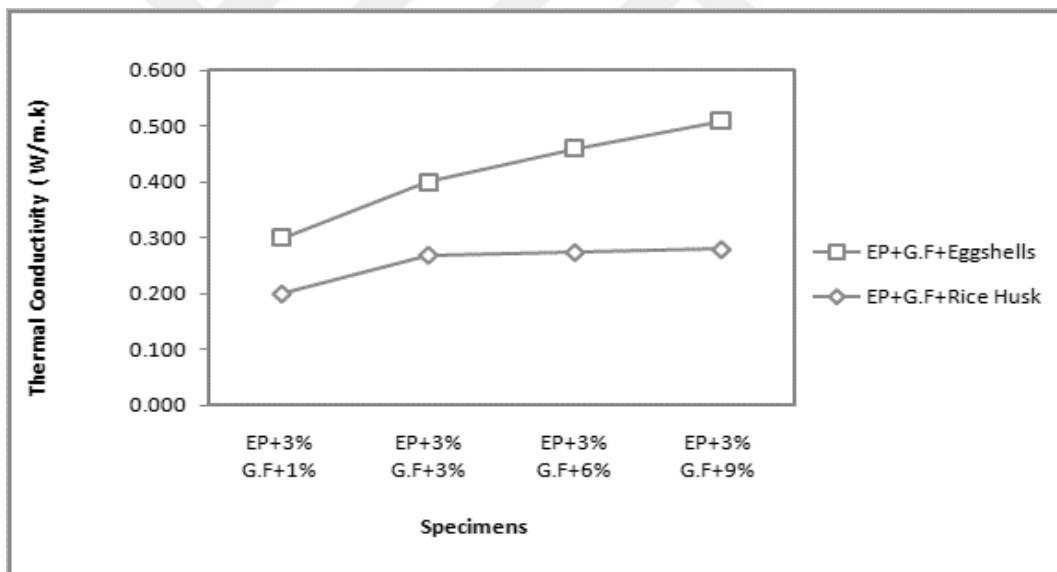


Figure 4.10: Thermal conductivity of specimen reinforced with (glass fiber, before burning rice husk and eggshells).

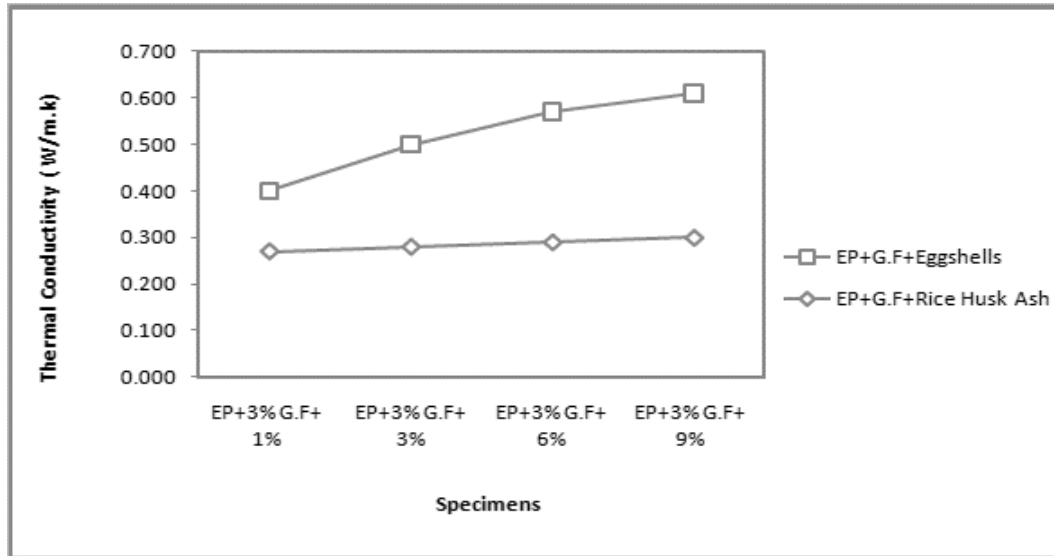


Figure 4.11: Thermal conductivity of specimen reinforced with (glass fiber, after burning rice husk and eggshells).

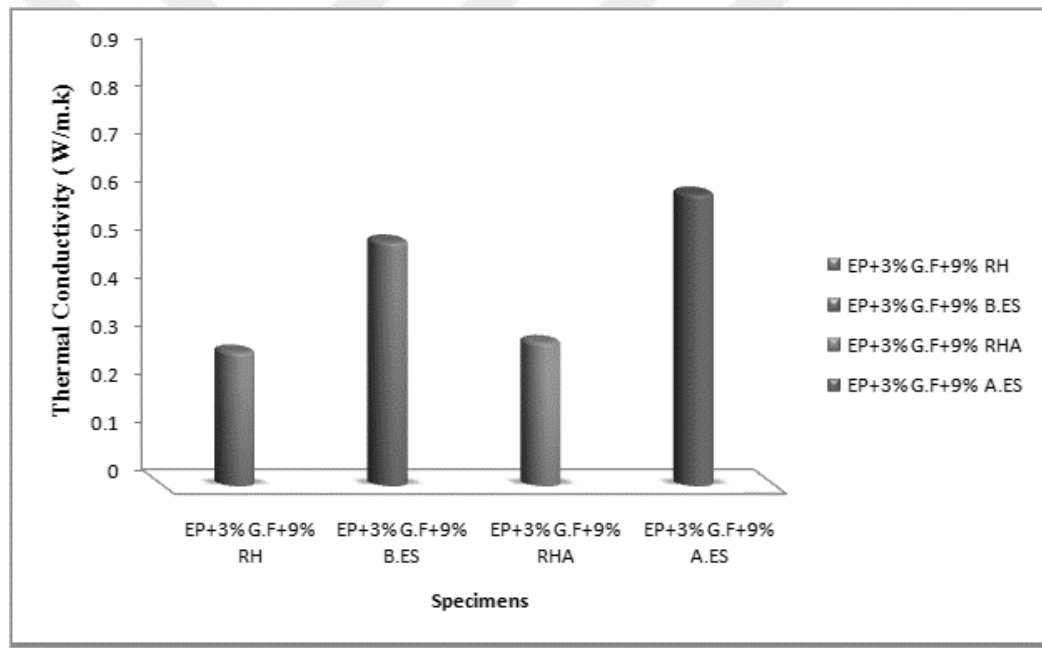


Figure 4.12: Comparing thermal conductivity of composite materials reinforced with 9% for all specimens.

4.2.3.2 Thermal Diffusivity

The thermal diffusivity values as shown in the Figures 4.13, 4.14, 4.15 and 4.16. Thus, the higher values of thermal diffusivity reach to (0.7341 mm²/Sec) for specimens (epoxy +3% Glass fiber+9%A.ES) .Thermal diffusion increasing with increases weight fraction of the reinforced material, because of the thermal diffusion of composite material depends on the weight fraction of the reinforced [79].

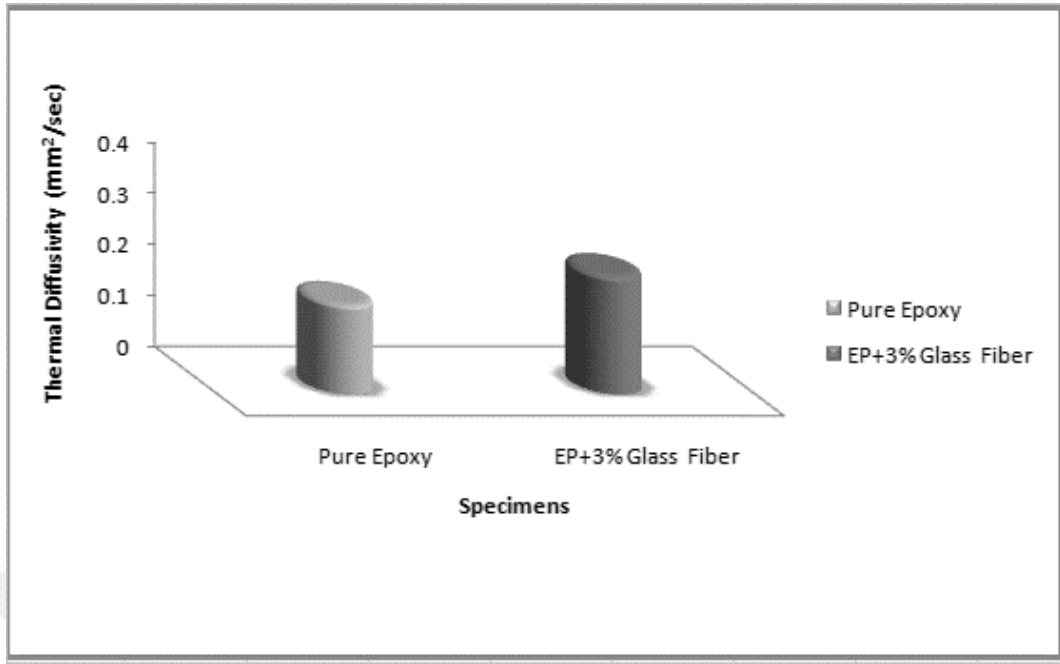


Figure 4.13: Thermal diffusivity of pure epoxy and epoxy +3% glass fiber.

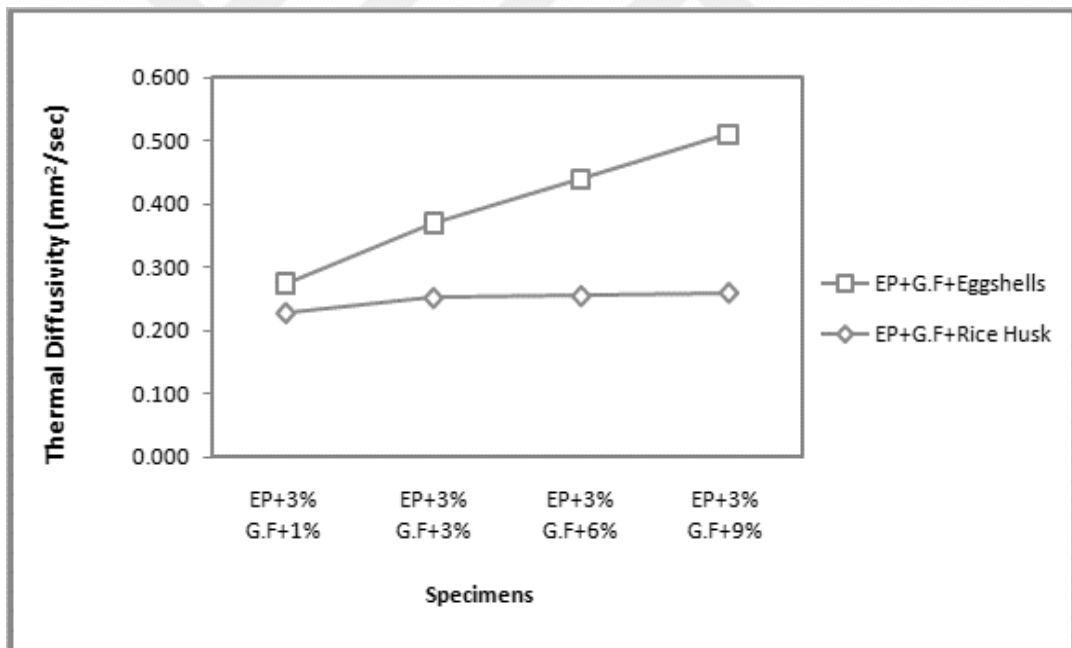


Figure 4.14: Thermal diffusivity of specimen reinforced with (glass fiber, before burning rice husk and eggshells).

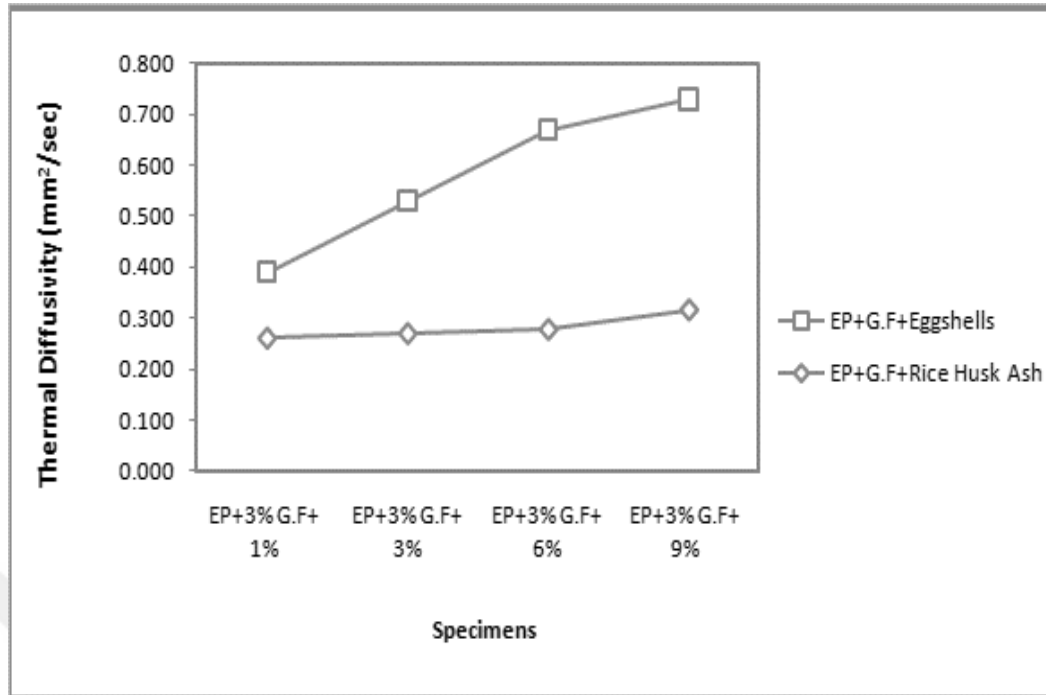


Figure 4.15: Thermal diffusivity of specimen reinforced with (glass fiber, after burning rice husk and eggshells).

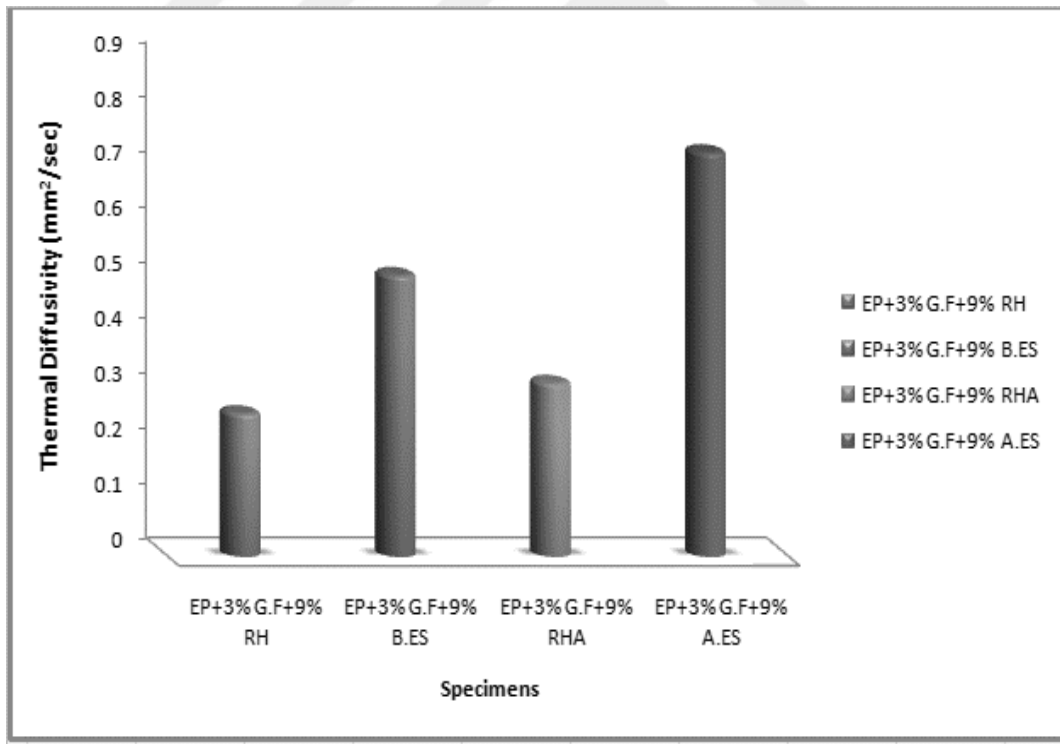


Figure 4.16: Comparing thermal diffusivity of composite materials reinforced with 9% for all specimens.

4.2.3.3 Specific Heat

Figure 4.17 to 4.20 shows the relationship between the values of specific heat and weight fraction of specimens. It can be observed that the specific heat values decrease with increase weight fraction of all samples and these values are *inversely* proportional with values the thermal conductivity and diffusivity. It also can be noticed in these figures that the values of specific heat for specimens before burning are higher than the values of specific heat for specimens after burning. Thus, the specific heat value decreased from (1.2 kJ/kg.K) for epoxy resin matrix (as referenced) to (0.8 kJ/kg.K) for the specimen (epoxy +3% glass fiber+9%A.ES) composite. The composite materials reinforced with particles and fibers have low specific heat when compare with matrix resin ,the reason is that the particle of (rice husk ash and eggshells) is ceramic material when combined with polymer these particle works good bond between the matrix material and reinforced materials [86].

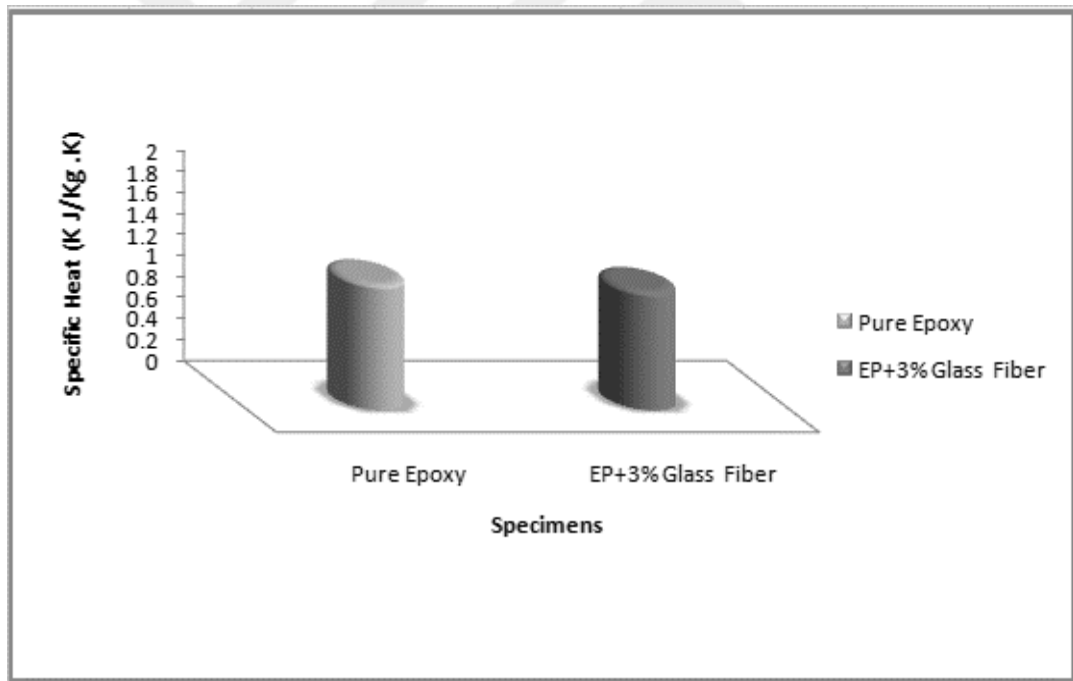


Figure 4.17: Specific heat of pure epoxy and epoxy +3% glass fiber.

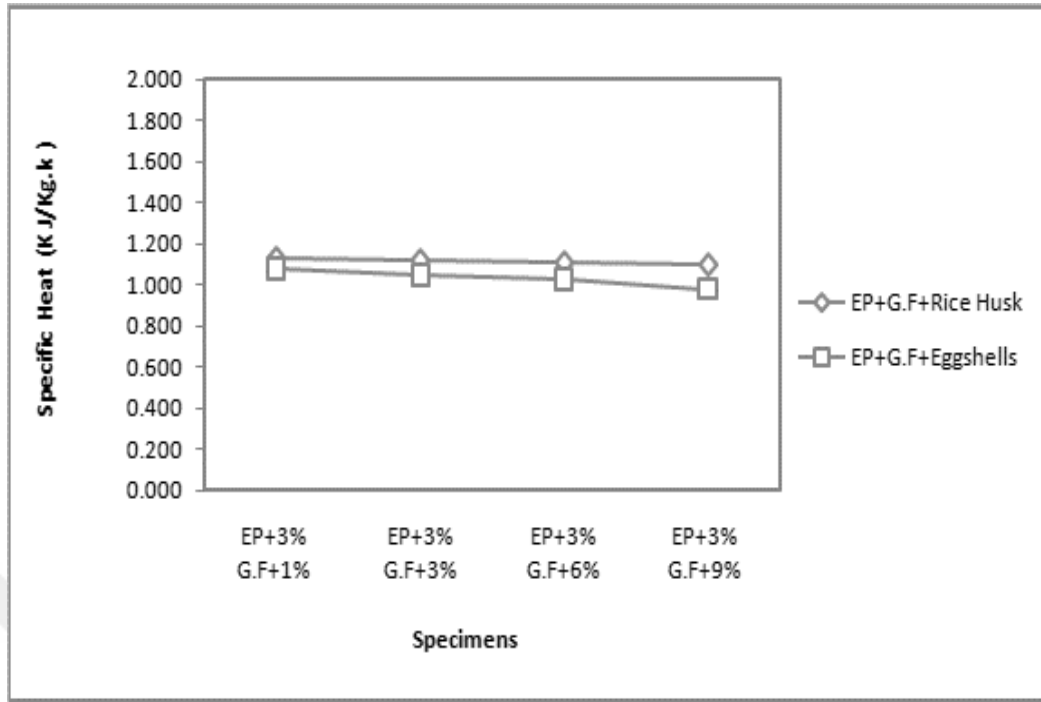


Figure 4.18: Specific heat of specimen reinforced with (glass fiber, before burning rice husk and eggshells).

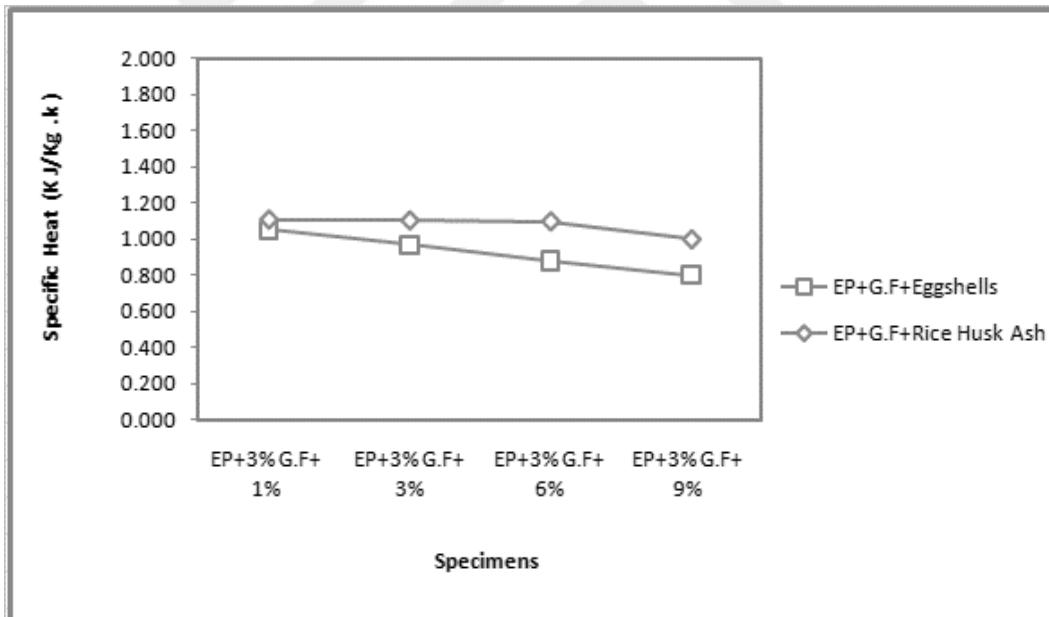


Figure 4.19: Specific heat of specimen reinforced with (glass fiber, after burning rice husk and eggshells).

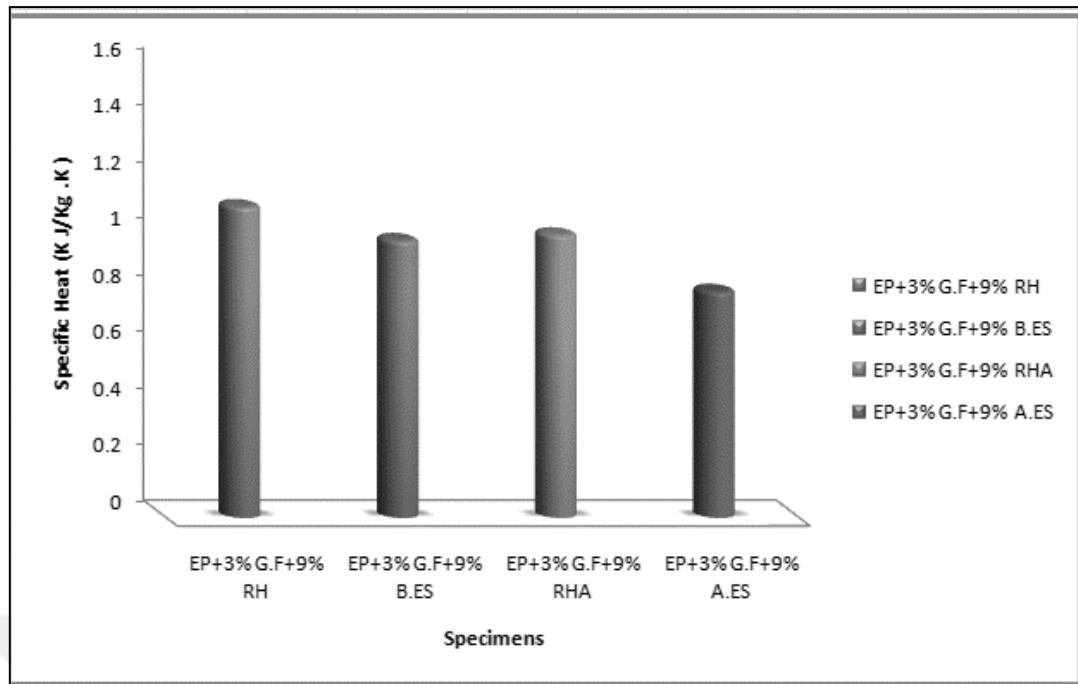


Figure 4.20: Specific heat of composite materials reinforced with 9% for all specimens.

4.3 Mechanical Tests

4.3.1 Tensile Properties

4.3.1.1 Stress – Strain Curve

The stress- strain curve for the all specimens reinforced with natural powder (RH, RHA, B.ES and A.ES) before and after burning is presented as shown in figures 4.21 to 4.26 respectively. It is evident that characteristic stress- strain results are trend growing from linearity and eventually develops into nonlinear. The characteristic linear slope predominantly reflects the deformation of glass fibers, while the characteristic nonlinear is basically attributed to the deformation of the matrix resin [106]. The tensile strength is increased with the fraction weight increase, the reasons behind this behavior is that the presence of fiber and the amount of powder enhancement plays an important role in preventing the increased slip of epoxy resins chains . It is worth mentioning that the chains require high stress to flex in a narrow space between the powders. The maximum value of strengthening is found in the weight fraction (9 %). As well as the nature of the relationship between particles and matrix resin plays an important role in increasing the bond force between particles and matrix, so the composite material needs high stress to break the physical bonding and in turn the composite material will have high tensile strength. The composite materials

supported by particles not only depend on the characteristics of the components, but also on the nature of the interface between the weight fraction and the components and sometimes depend on particle geometry [107].

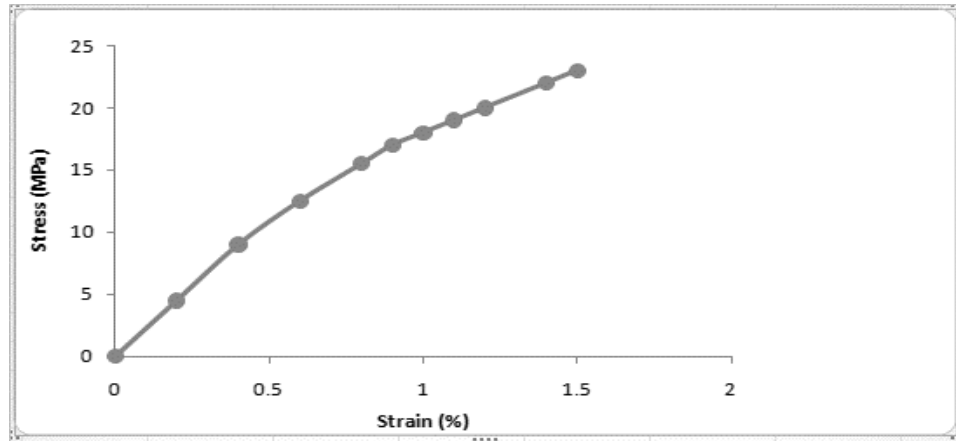


Figure 4.21: Stress- strain curve for specimen pure epoxy resin.

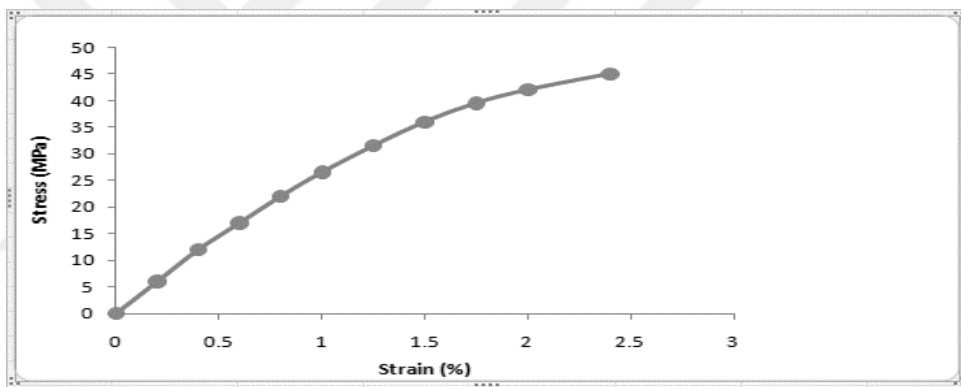


Figure 4.22: Stress- strain curve for specimen epoxy +3% glass fiber.

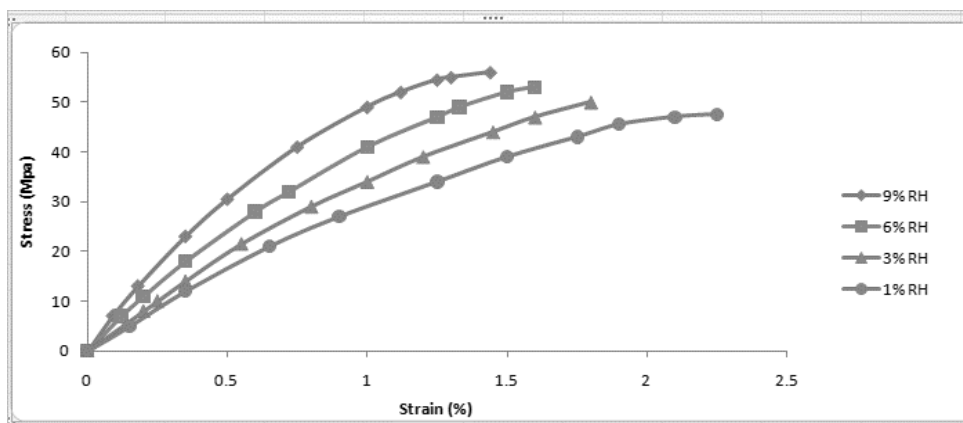


Figure 4.23: Stress- strain curve for specimens strength with (glass fiber, before burning rice husk).

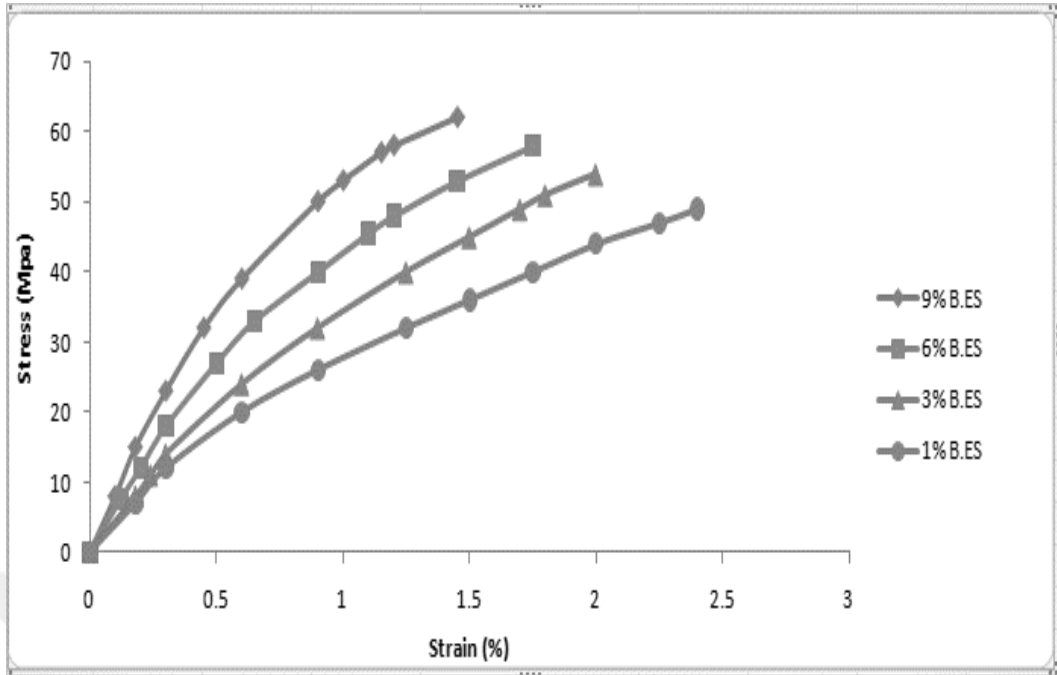


Figure 4.24: Stress- strain curve for specimens strength with (glass fiber, before burning eggshells).

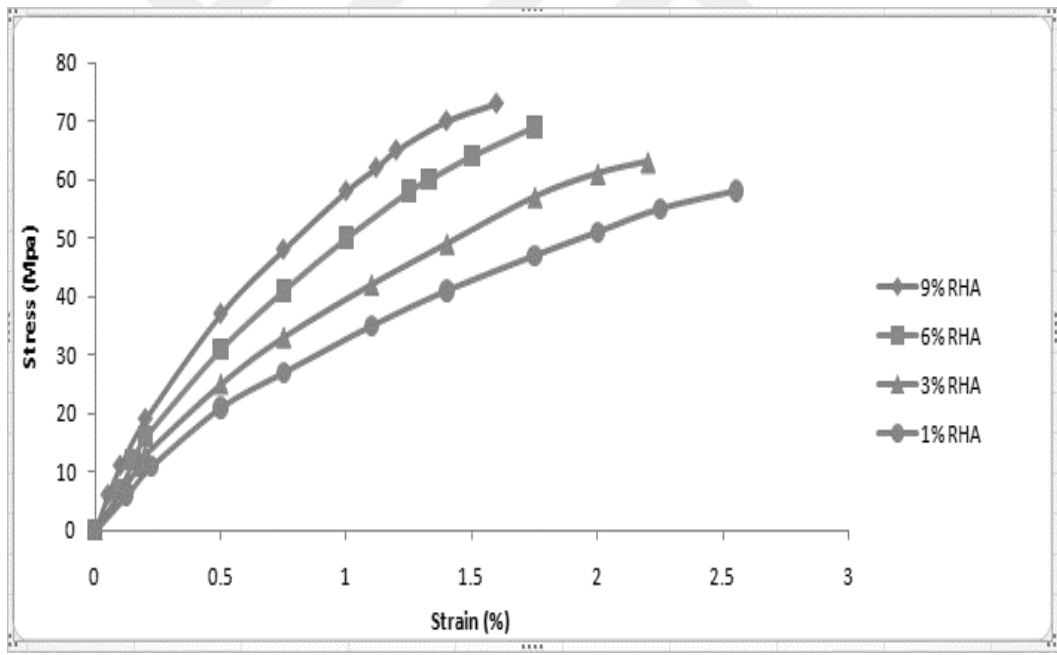


Figure 4.25: Stress- strain curve for specimens reinforced with (glass fiber, after burning RHA).

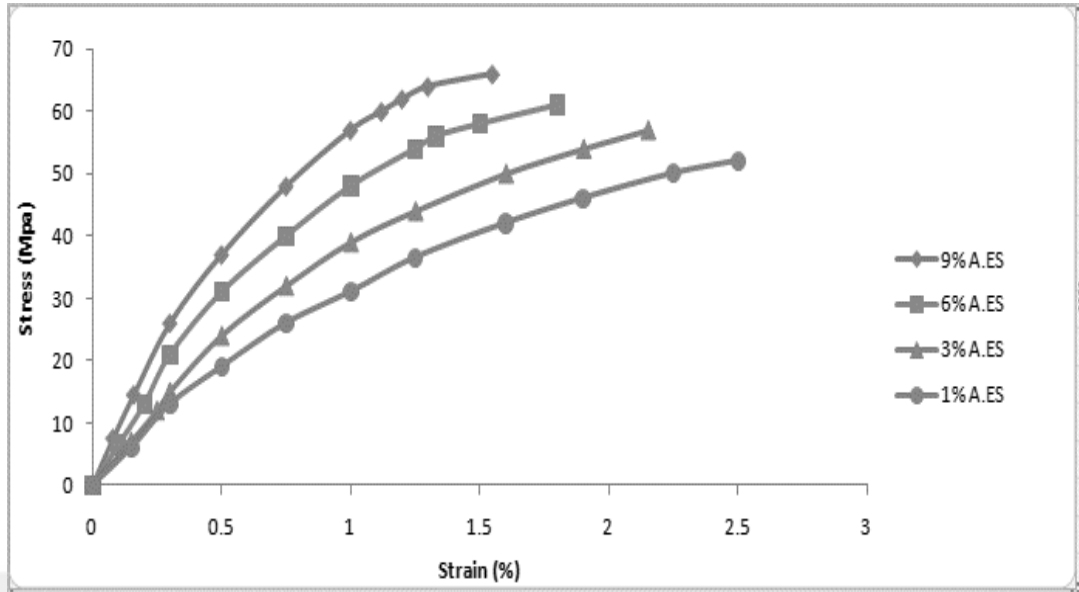


Figure 4.26: Stress- strain curve for specimens reinforced with (glass fiber, after burning eggshells).

4.3.1.2 Modulus of Elasticity:

Figures 4.27, 4.28, 4.29 and 4.30 shows the relationship between the weight fraction of the filler powder (RH, RHA, B.ES and A.ES) which were added to the glass fiber with epoxy resin with the modulus of elasticity. It can be observed with increase weight fraction the modulus of elasticity increases. Therefore, the greatest value of the modulus elasticity was (9.5 Gpa) at weight fraction (9%) for the specimen (epoxy +3%g.f+9% RHA) with mean particle size of (10.4 μm). This is due to the nature of the correlation and the mechanism of reinforcement between them. From these Figures 4.28, 4.29 and 4.30 also it can be seen the small mean particle size of RHA powder improves the modulus of elasticity more than other powders, this is due to the reinforcement mechanism as follows:

Firstly: Large particle reinforcement for (RH) powder tend to restrain the movement of the matrix phase in the vicinity of each particle, while the matrix transfers several of the applied load to the particles and a bear fraction of it.

Secondly: Small fillers impede slipping of matrix chains and requirement high stress to bow them in narrow space amongst particles compared with large particles and the matrix bears the large portion of the applied load [107 & 108].

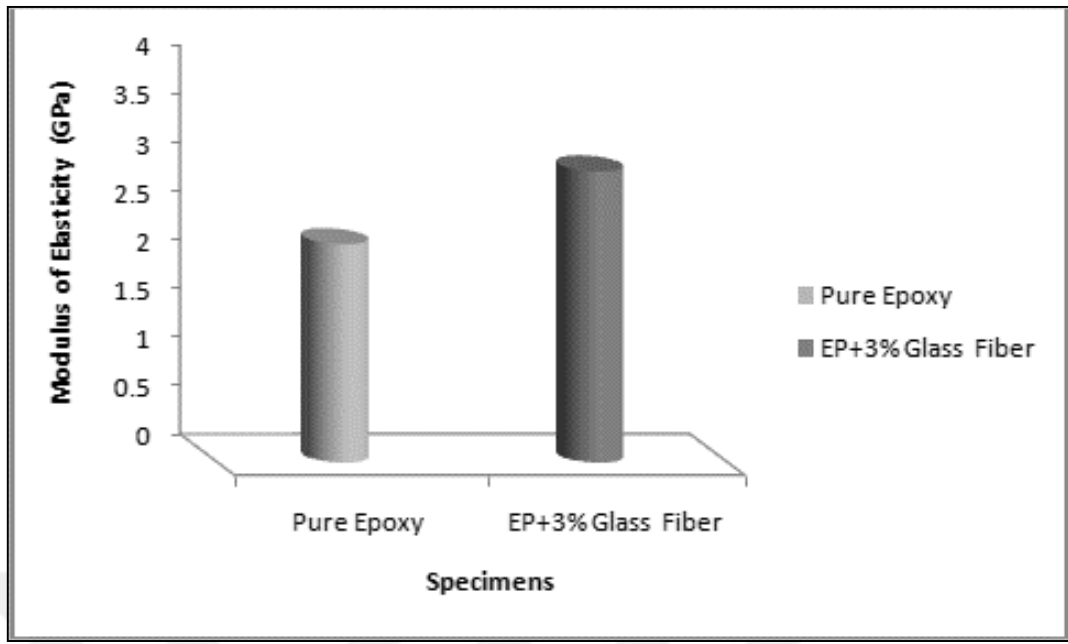


Figure 4.27: Modulus of elasticity of specimen pure epoxy and epoxy +3% glass fiber.

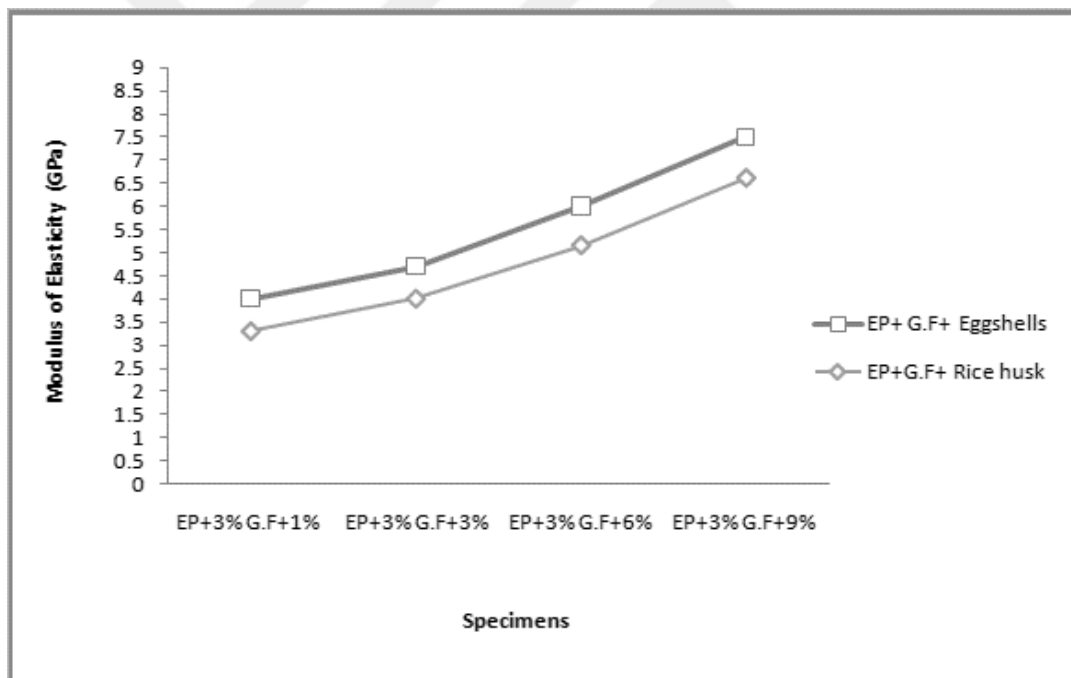


Figure 4.28: Modulus of elasticity of specimens reinforced with (glass fiber, before burning rice husk and eggshells).

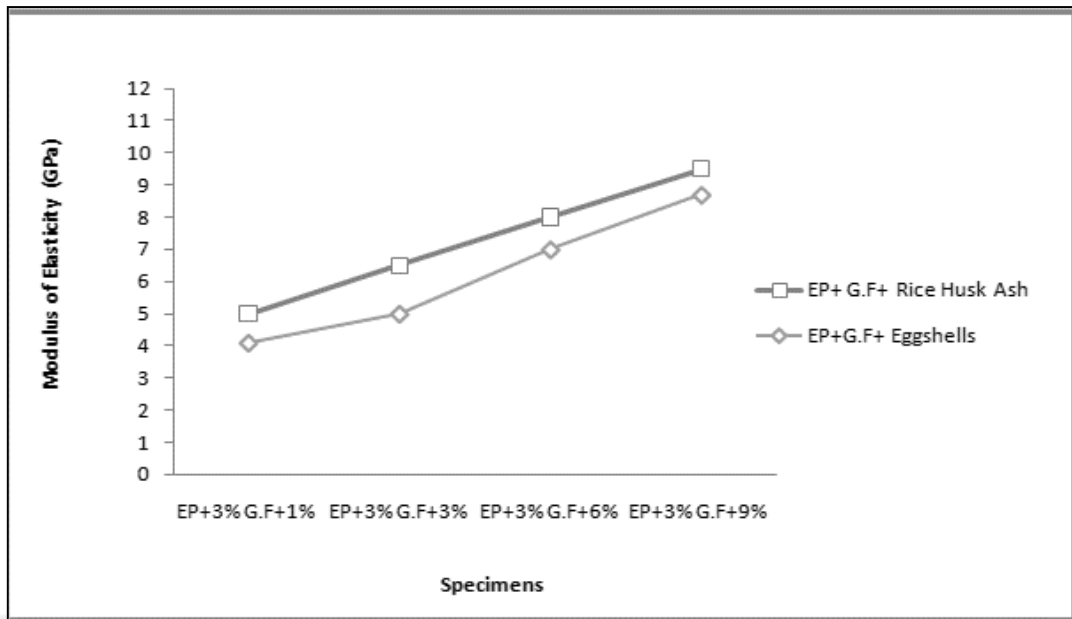


Figure 4.29: Modulus of elasticity of specimens reinforced with (glass fiber, after burning rice husk ash and eggshells).

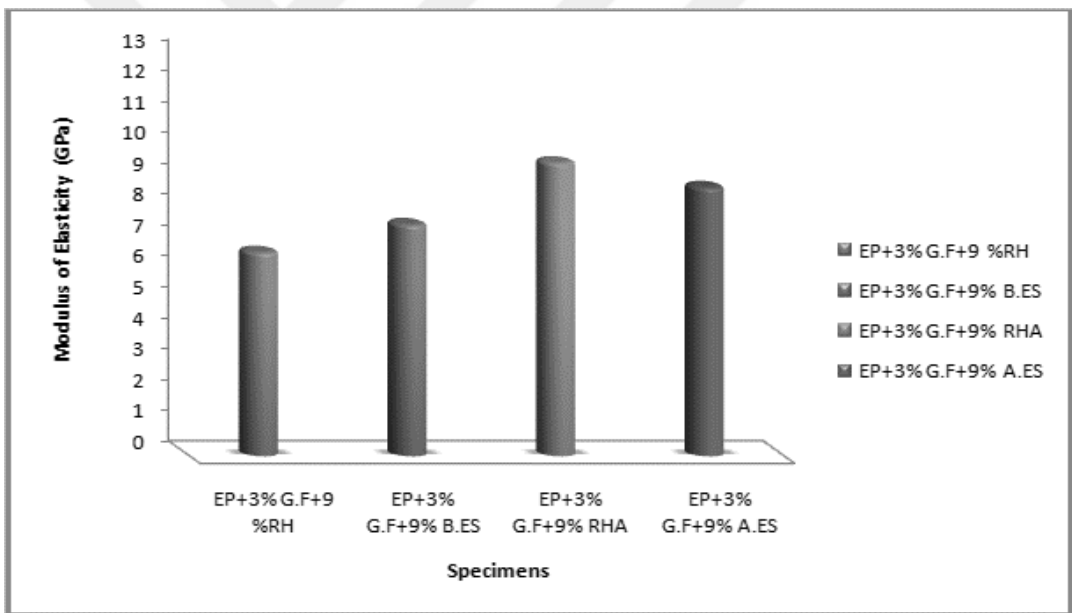


Figure 4.30: Comparing value of Modulus of elasticity of composite materials reinforced with 9% for all specimens.

4.3.1.3 Tensile Strength

The values of tensile strength are illustrated in Figures 4.31 to 4.34 for specimens prepared from (pure epoxy, epoxy + 3% glass fiber and before, after burning rice husk, rice husk ash, egg shells). Figure 4.31 shows the value of tensile strength of pure epoxy and epoxy reinforced with (3%w.f) glass fiber. From the figure can be seen the specimen (ep +3% glass fiber) has value tensile strength higher than specimen pure

epoxy, This can be attributed to the fact that glass fiber is characterized by high hardness and high strength (resistance to crack propagation); therefore, the composite can sustain higher loads compared to the unreinforced epoxy [109&110]. Figures 4.32 & 4. 33 shows the tensile strength value for specimens (Ep+3% g.f+1%, 3%,6% and 9% before , after burning of RH, RHA and ES). From these figures, it was found that the specimens before burning (epoxy + 3% glass fiber + 9% eggshells) and the specimen after burning (epoxy + 3% glass fiber + 9% RHA) have higher tensile strength than other specimens. Also can be seen from Figure 4.34 when comparing values of eventual tensile strength for all specimens composites the specimen (epoxy + 3% glass fiber +9% RHA) gave a higher value of tensile strength than other specimens. Such demeanor is due to the nature of bonding force between the matrix resin and reinforced particles which is strong bonding in case of small particle size that does not allow forming internal defects cracks in a quick manner and in turn the composite polymer material will have high tensile strength. Composite polymer material supported by powder does not based only on the properties of components, however also on the nature of the interface between the components and weight fraction also in sometimes on the geometry of the particulate [107].

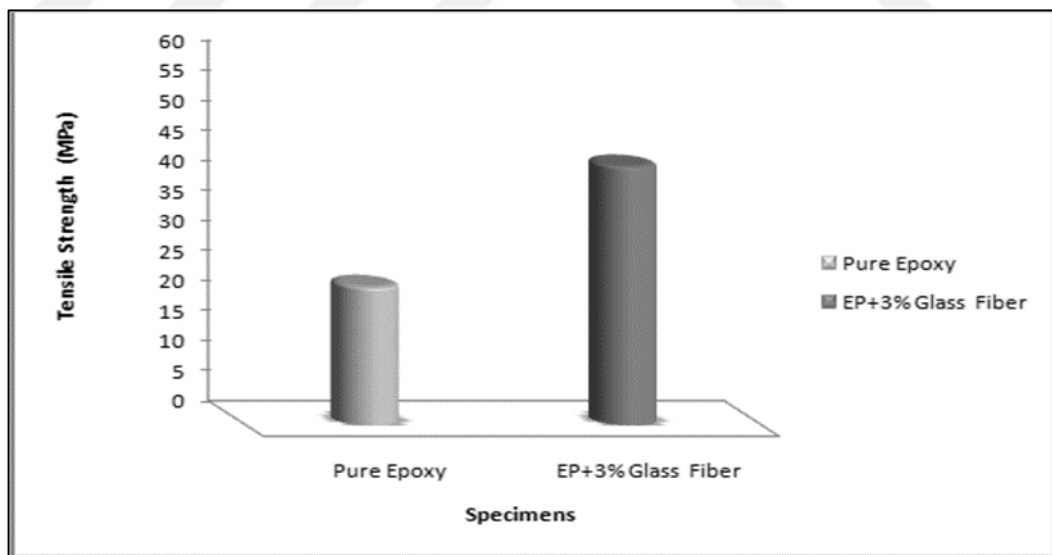


Figure 4.31: Tensile strength of specimen pure epoxy and epoxy +3% glass fiber.

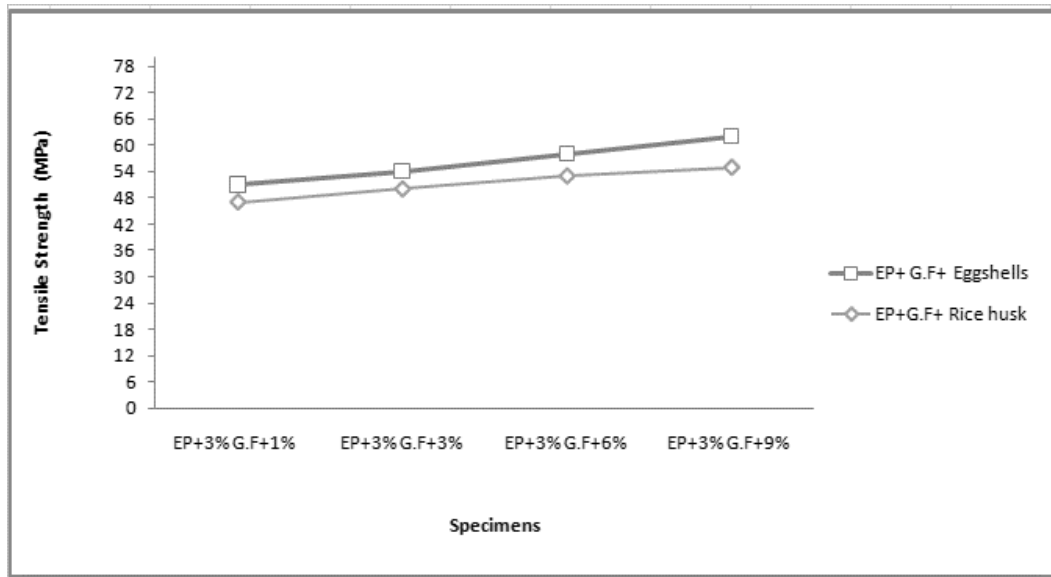


Figure 4.32: Tensile strength of specimens reinforced with (glass fiber, before burning rice husk and eggshells).

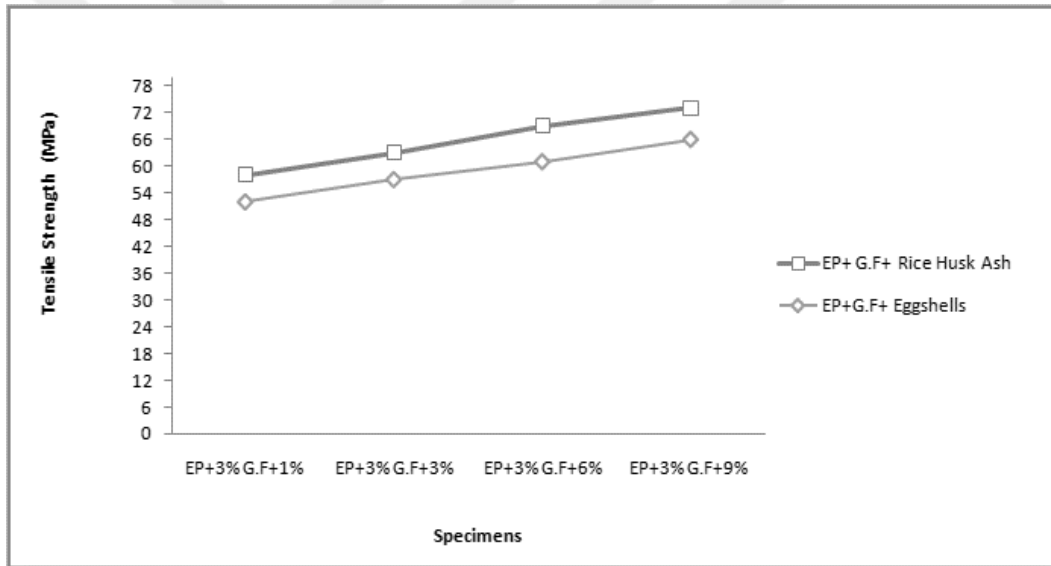


Figure 4.33: Tensile strength of specimens reinforced with (glass fiber, after burning rice husk ash and eggshells).

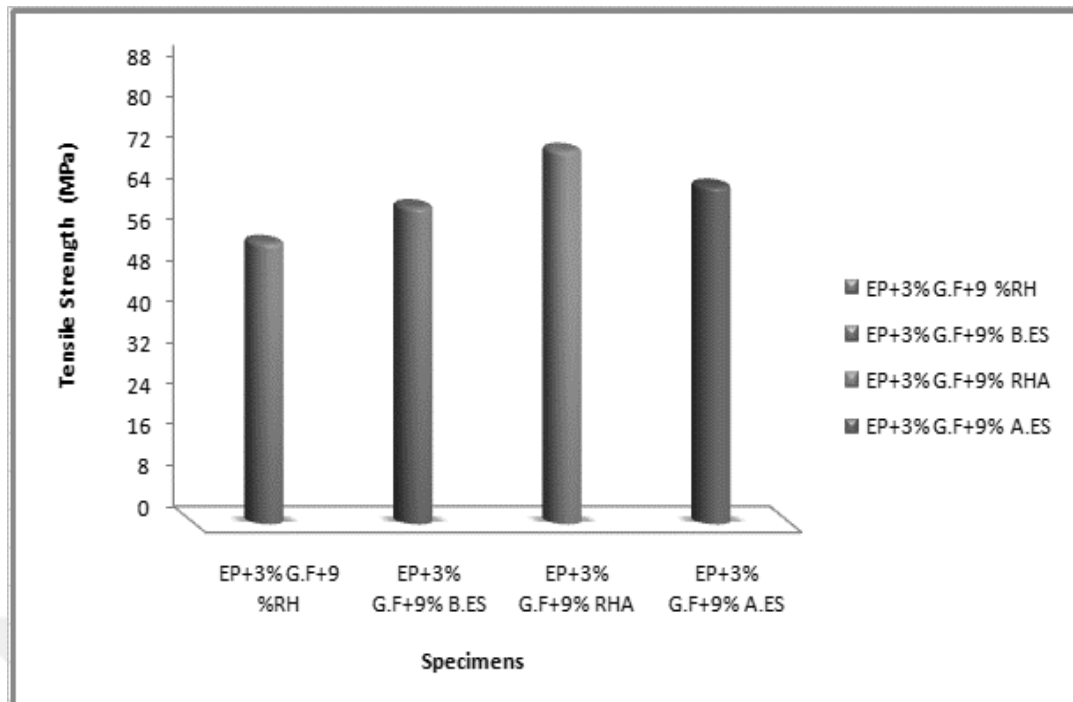


Figure 4.34: Comparing value of tensile strength of composite materials reinforced with 9% for all specimens.

4.3.1.4 Percentage Elongation

The results of percentage elongation at break for all specimens reinforcing with epoxy resin are shown in Figures 4.35, Figures 4.36, Figures 4.37 and Figures 4.38. It is shown that the percentage elongation at break of the composite specimens decreases with increasing weight fraction of (fiber and natural powder). This is due to the presence of fiber lends the stiffening effect within the matrix, thus imposes a mechanical constraint on the composite. As well as decrease of percentage elongation depends on the interference between fibers, natural powders and matrix resin. This behavior may be attributed to the formation of strong structures [111&112].

The minimum value of percentage elongation obtained for specimens before burning rice husk and eggshells powder is (1.35 %) for the specimen (epoxy +3%g.f+9%RH) and minimum value of percentage elongation obtained for specimens after burning rice husk ash and eggshells powder is (1.5%) for (epoxy +3%g.f+9% eggshell).

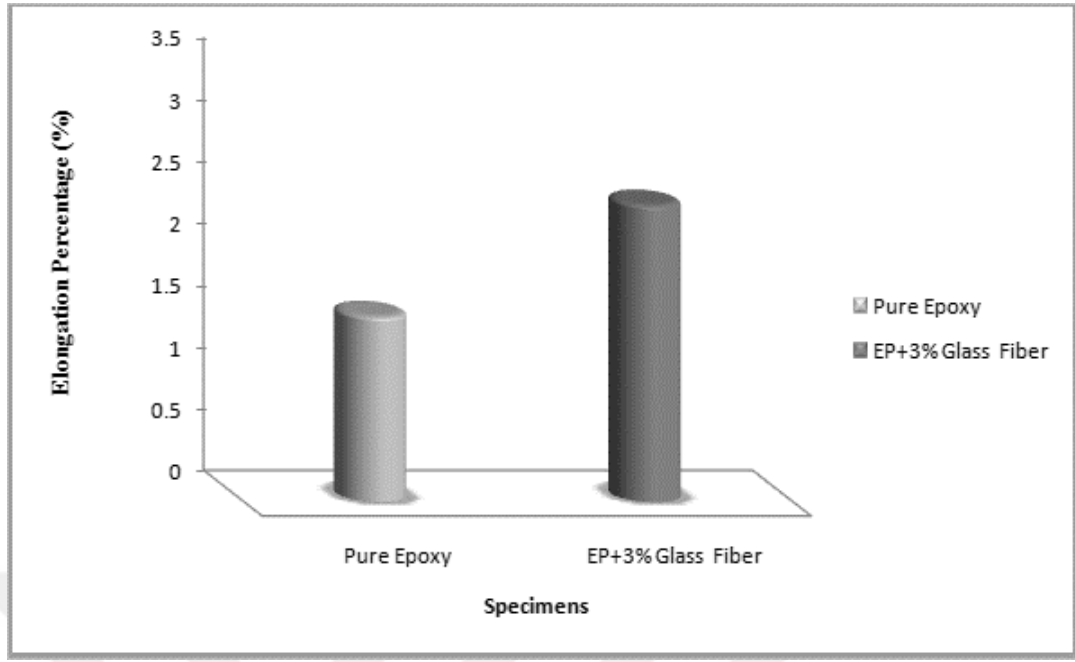


Figure 4.35: Elongation percentage at break of specimen pure epoxy and epoxy +3% glass fiber.

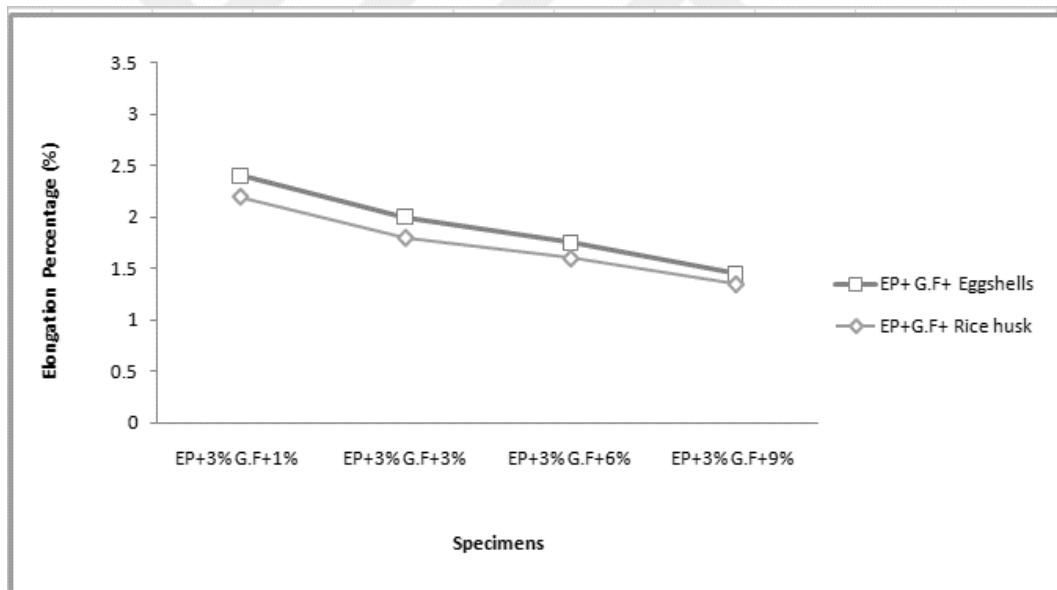


Figure 4.36: Elongation percentage at break of specimens reinforced with (glass fiber, before burning rice husk and eggshells).

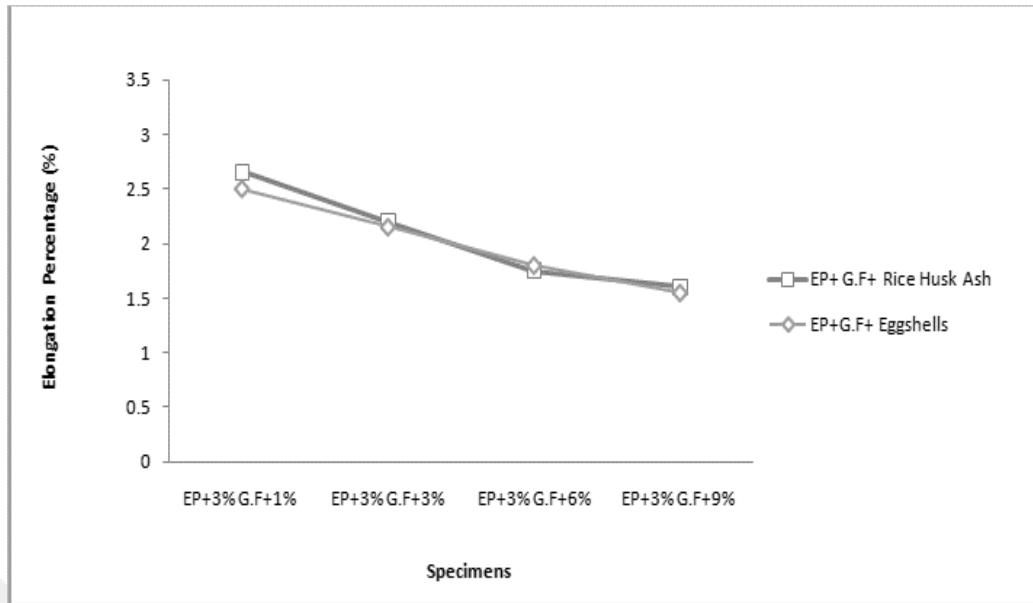


Figure 4.37: Elongation percentage at break of specimens reinforced with (glass fiber, after burning rice husk ash and eggshells).

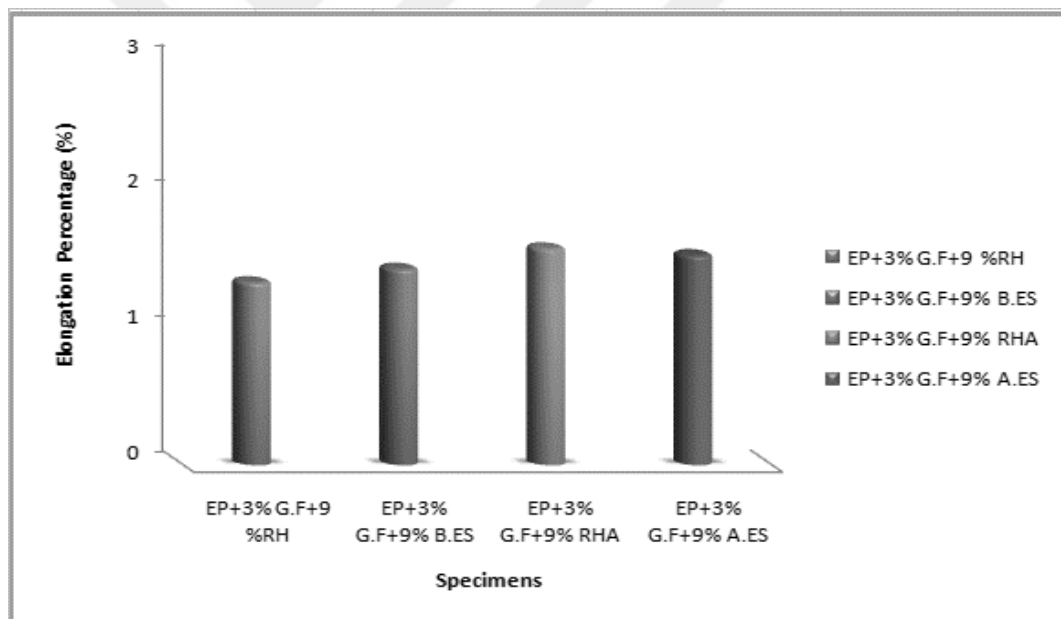


Figure 4.38: Comparing value of Elongation percentage at break of composite materials reinforced with 9% for all specimens.

4.3.2 Flexural Strength

The values flexural strength of the prepared specimens (pure epoxy, epoxy +3% glass fiber) composite materials as shows in Figures 4.39. From the Figures 4.39, the addition of 3% g.f weight fractions has a clear effect on the value of flexural strength. Where the specimen (epoxy +3% glass fiber) has a higher flexural strength than specimen pure epoxy due the addition of glass fiber in the epoxy resin and also the

specimen epoxy +3%glass fiber has value hardness higher than specimen pure epoxy. The relationship between the flexural strength and specimens of the filler powder (before and after burning rice husk, rice husk ash and eggshells) as shown in Figures 4.40 & Figures 4.41. The Figures illustrate that the flexural strength increases with an increasing weight fraction of the filler natural powder. Where the specimen (epoxy +3% glass fiber+9% before burning eggshells) has a higher flexural strength than specimen (epoxy +3% glass fiber+9% before burning rice husk),also the specimen (epoxy +3% glass fiber+9% rice husk ash) has a higher flexural strength than specimen (epoxy +3% glass fiber+9% after burning eggshells). The reason for increased bending strength is to increase the elasticity modulus of composite materials reinforced by fibers and particles compared to the matrix resin material.

Figure 4.42 shows the comparing the values of flexural strength for all of the prepared specimens composites. From this figure can be seen the specimen (epoxy + 3% glass fiber + 9% RHA) gave a higher value of flexural strength than other specimens reinforced 9% weight fractions. Also the composite materials reinforced with small mean particle fillers can improve the flexural strength more than large particle filler. This is due the small mean particle size provide highly strong bonding between the reinforced and matrix material.

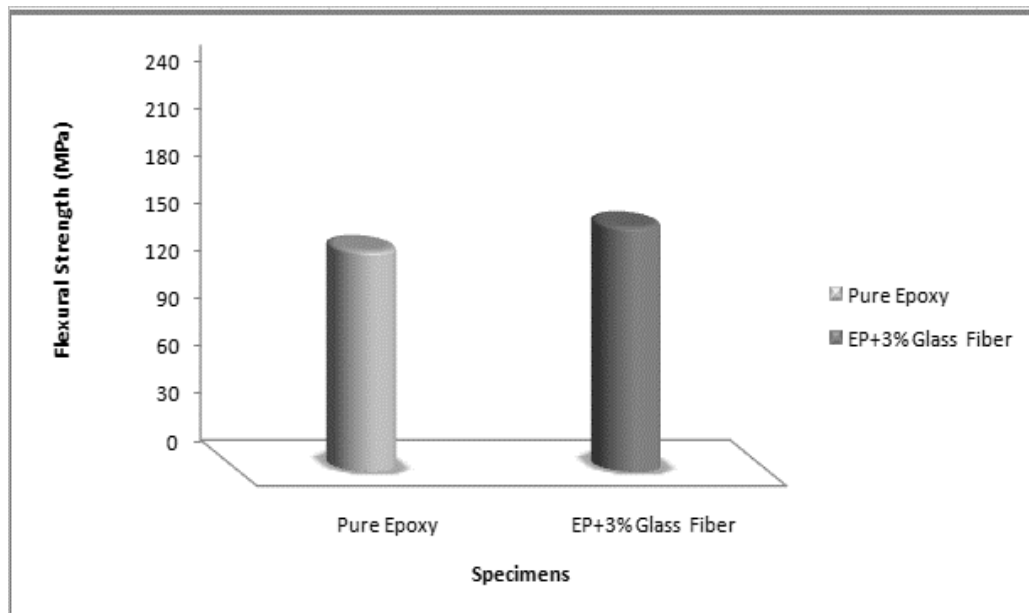


Figure 4.39: Flexural strength of specimen pure epoxy and epoxy +3% glass fiber.

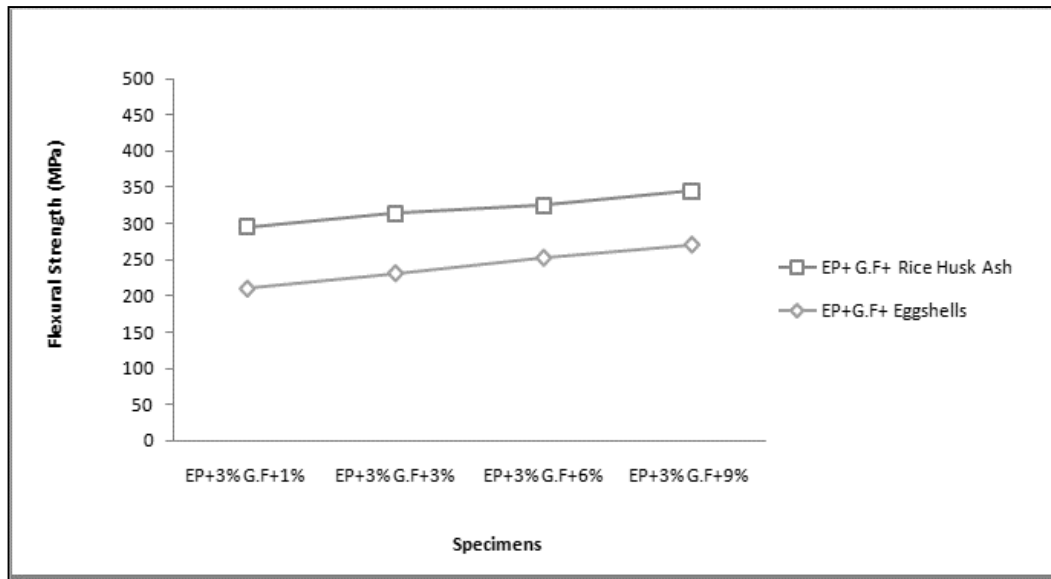


Figure 4.40: The flexural strength of specimens reinforced (glass fiber, after burning rice husk ash and eggshells).

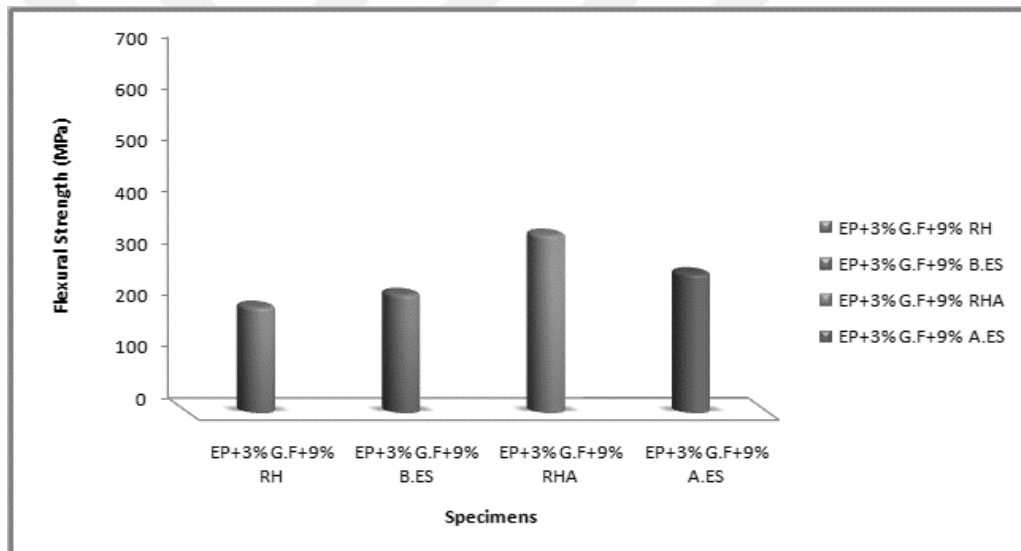


Figure 4.41: Comparing value of flexural strength for composite materials strengthened with 9% for all specimens.

4.3.3 Flexural Modulus

Flexural modulus is calculated from Equation 2.17 as shown in Figures 4.43, Figures 4.44, Figures 4.45 and Figures 4.46 for all specimens. Specimens after burning have higher flexural modulus than specimens before burning. This is because the presence of a natural powder with glass fiber in the matrix resin gives higher mechanical properties than the epoxy resin matrix alone [3]. Epoxy resin has a lower flexural modulus been (1.76 GPa), than other specimens. That is related to the nature of epoxy with high extension compared with specimen reinforcement materials.

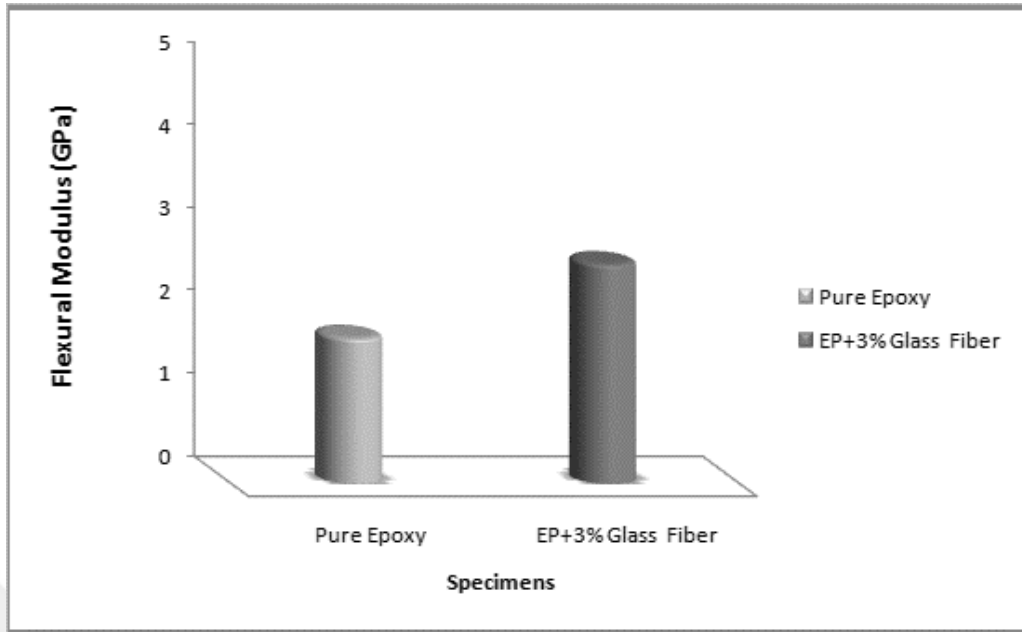


Figure 4.42: Flexural modulus of specimen pure epoxy and epoxy +3% glass fiber.

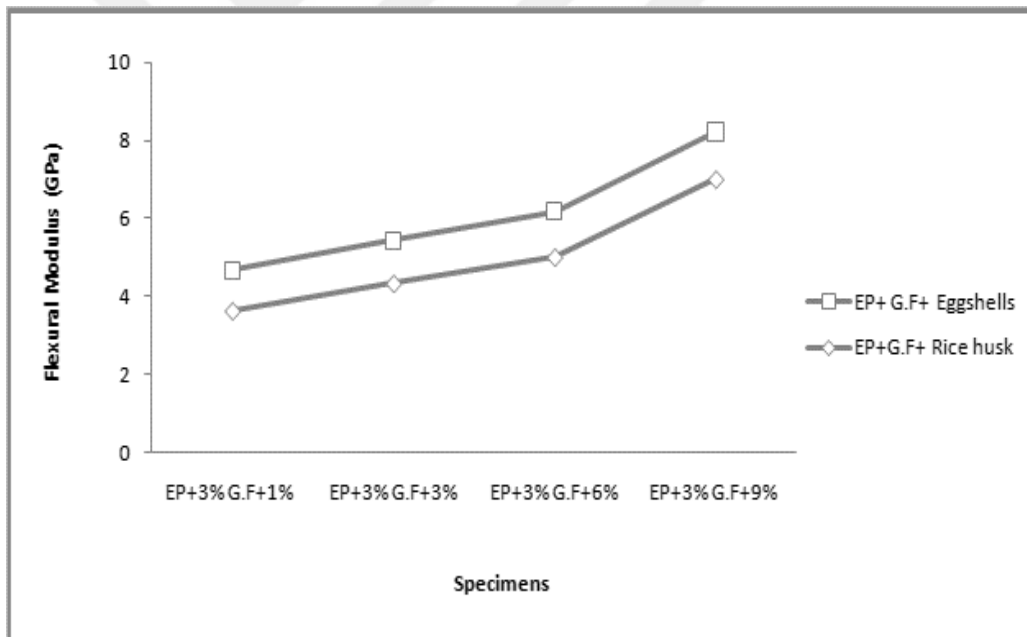


Figure 4.43: Flexural modulus of specimen reinforced with (glass fiber, before burning rice husk and eggshells).

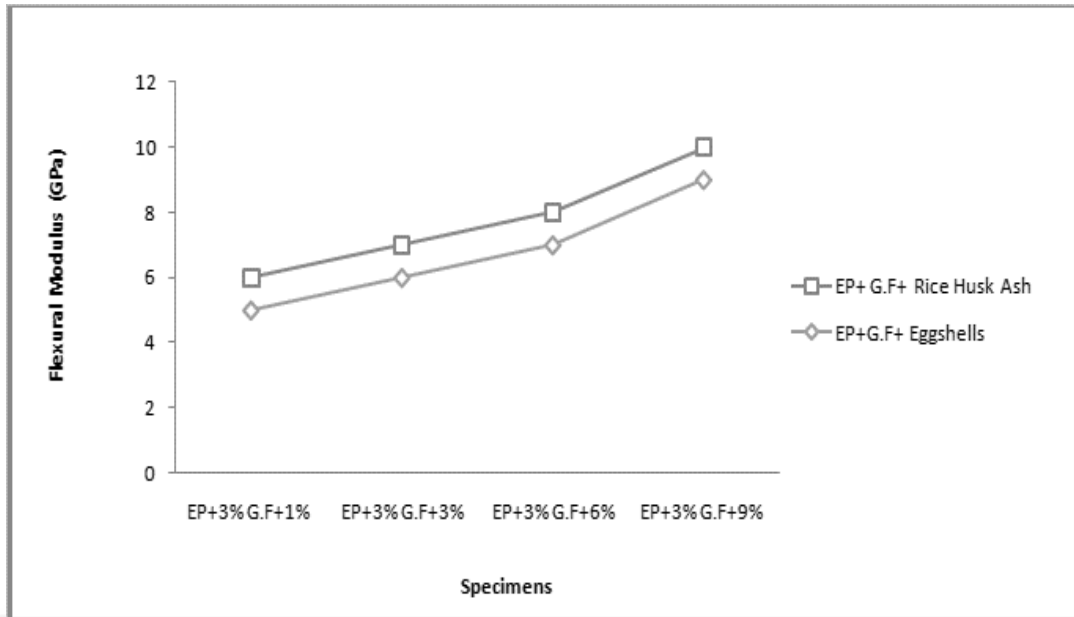


Figure 4.44: Flexural modulus of specimen reinforced with (glass fiber, after burning rice husk and eggshells).

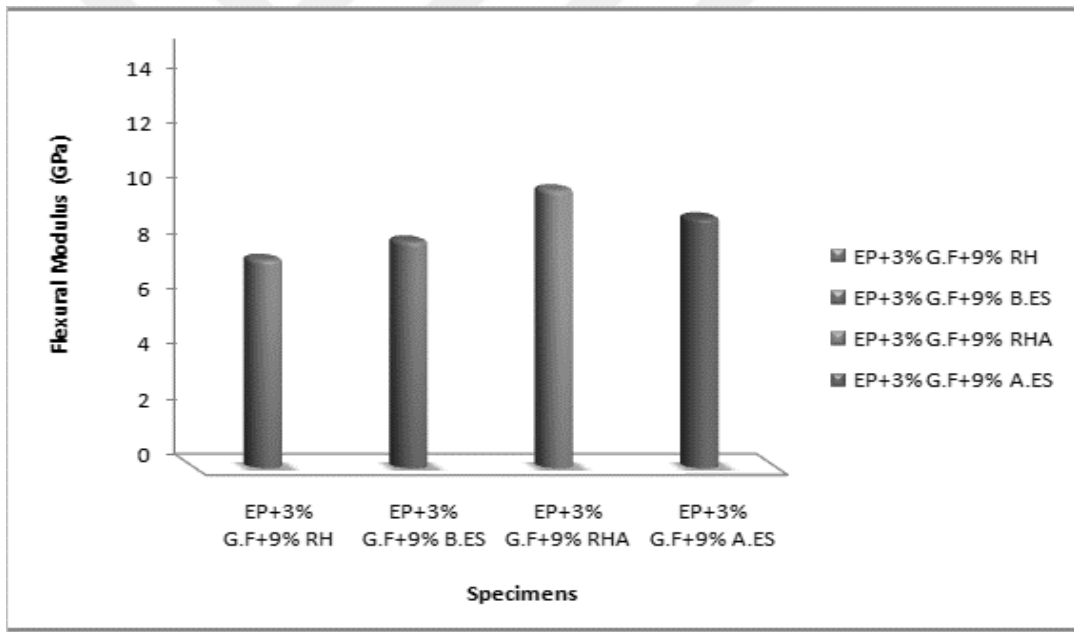


Figure 4.45: Comparing value of flexural modulus of composite materials reinforced with 9% for all specimens.

4.3.4 Maximum Shear stress

Figure 4.47, Figure 4.48, Figure 4.49 & Figure 4.50 shows the value shear stress of all specimen composite material used in this study. From the Figure 4.47 can be seen the specimen (epoxy +3% glass fiber) has a maximum shear stress than specimen pure epoxy due the addition of 3% weight fraction of glass fiber.

The relationship between the shear stress and specimens of the filler powder (before and after burning rice husk, rice husk ash and eggshells), which were added to the epoxy resin as shown in Figure 4.48 & Figure 4.49. The maximum value shear stress is obtained at (9 %) weight fraction with (10 μm) mean particle size of rice husk ash powder. Figure 4.50 shows the comparative values of maximum shear stress for all of the prepared specimens composites, the specimen (epoxy + 3% glass fiber + 9% RHA) gave a higher value of shear stress than other specimen reinforced with (9% weight fractions). The highly (reinforcing- matrix) bonding of the composites has a remarkable effect on increasing the maximum shear stress. The reason for the improved properties of shear stress is the interconnection between the matrix resin material and reinforcing, also in addition the ability of these particles to hinder the crack propagation [59].

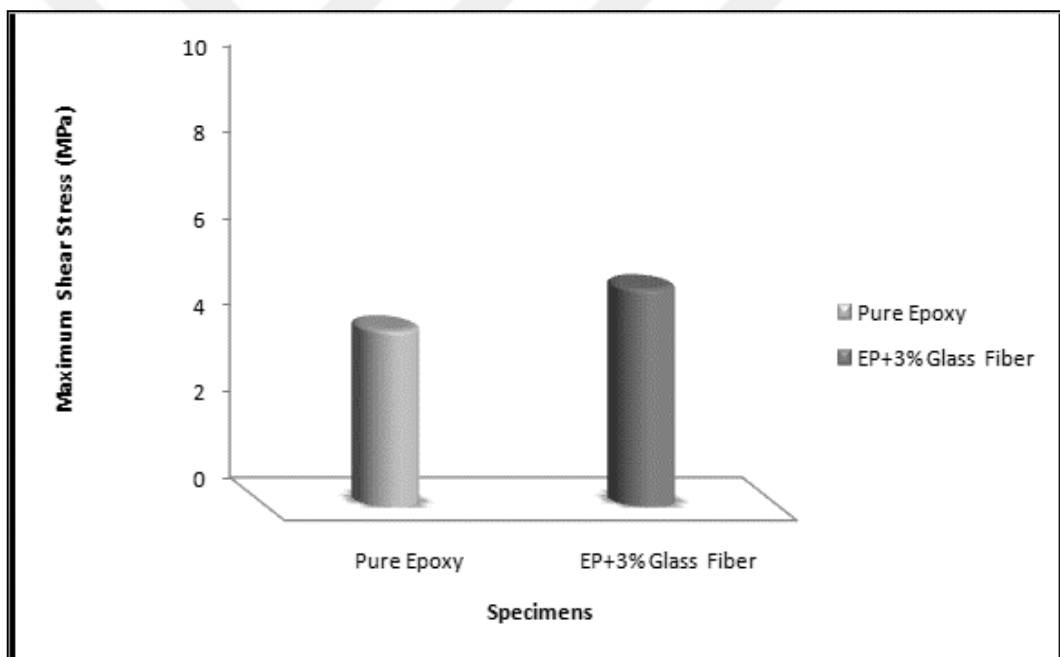


Figure 4.46: Maximum shear stress of specimen pure epoxy and epoxy +3% glass fiber.

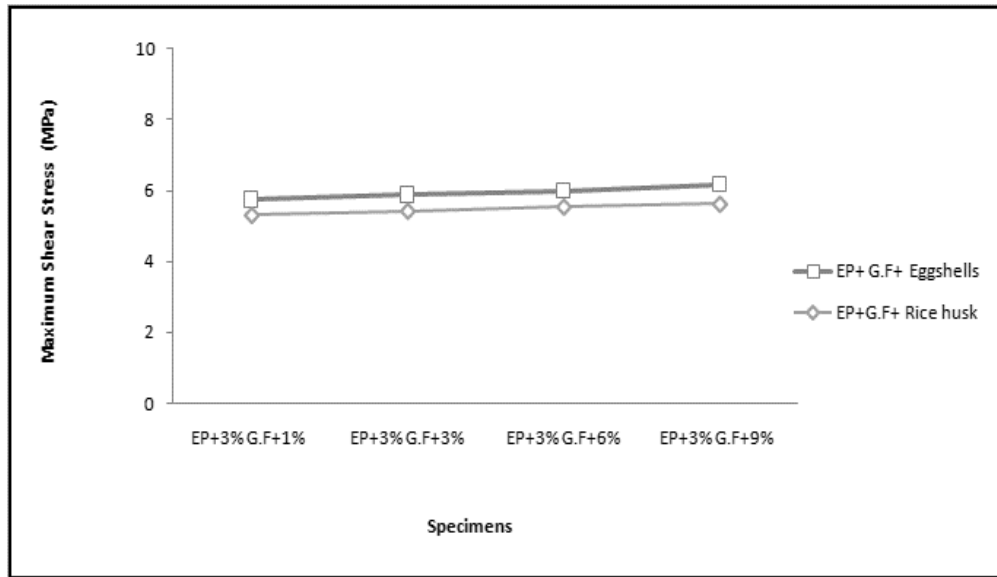


Figure 4.47: Maximum shear stress for specimens reinforced with (glass fiber, before burning rice husk and eggshells).

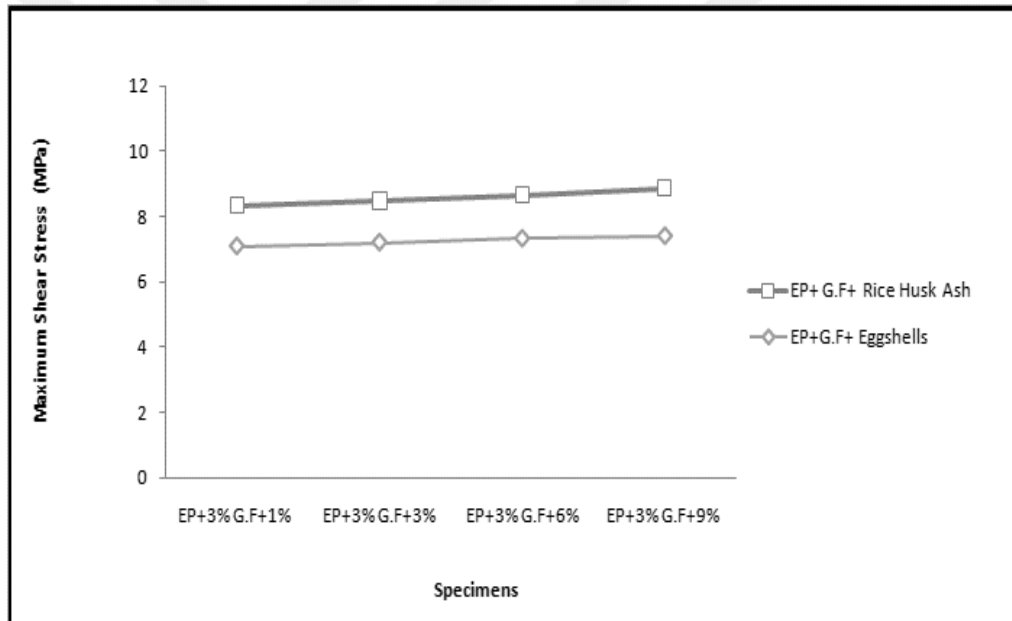


Figure 4.48: Maximum shear stress for specimens reinforced with (glass fiber, after burning rice husk ash and eggshells).

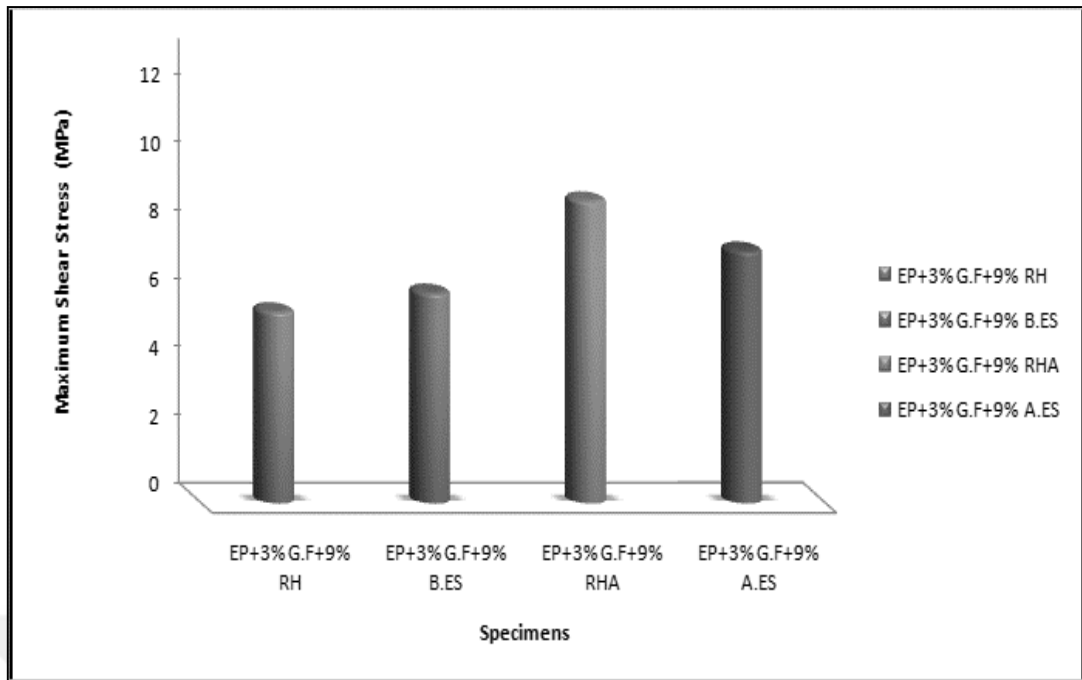


Figure 4.49: Comparing value of maximum shear stress of composite materials reinforced with 9% for all specimens.

4.3.3 Impact Test:

4.3.3.1 Impact Strength:

Impact test is utilized for determination of possibilities range composite material to impact force through the absorbed energy of these materials. Impact test different from other mechanical tests due it is very fast, where the sample is exposed to stress rapid, leading to changes in the disposal of materials. Figure 4.51 shows the result of impact strength for specimens (pure epoxy and epoxy + 3% glass fiber).The figures illustrate that the impact strength increasing with the addition 3% weight fraction from glass fiber, the reason is that the presence of fiber with epoxy resin gives the sample a good ability to absorb ability absorb a large share of impact stresses and kinetic energy. The relationship between the impact strength with specimens (epoxy + 3% glass fiber+ 1%, 3%,6% & 9% before burning rice husk and eggshells) as shown in Figure 4.52.These figures illustrate the impact strength at 9% weight fraction of rice husk and eggshells filler powder of composite materials decreases, but the specimen (epoxy + 3% glass fiber +6% eggshells) has higher values of impact strength than specimen (epoxy + 3% glass fiber +6% rice husk). The relationship between the impact strength with specimens (epoxy + 3% glass fiber+ 1%,3%,6% & 9% after burning rice husk ash and eggshells) as shown in Figure 4.53.These figures illustrate the impact strength

at 9% weight fraction of rice husk ash and eggshells filler powder of composite materials decreases, but the specimen (epoxy + 3% glass fiber +6% rice husk ash) has higher values of impact strength than specimen (epoxy + 3% glass fiber +6% eggshells), this is due to the reinforcing materials represent points of concentrated stress which cause the onset of failure and thus the structure of the composite materials will be weak. As well as an increase in concentration of reinforced reduces the ability of matrix resin to absorb energy and reducing the toughness, thus impact energy decrease [113]. From the Figure 4.54 can be seen when comparing the values of impact strength for all of the prepared specimens composites, the specimen (epoxy + 3% glass fiber + 6% RHA) gave a higher value of impact strength and fracture toughness than other weight fractions.

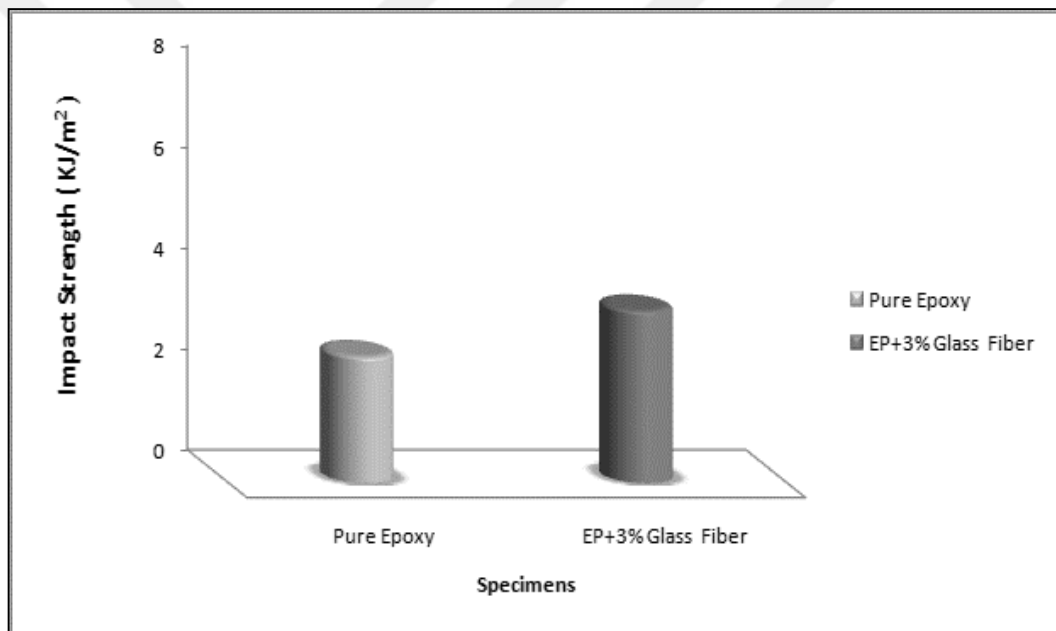


Figure 4.50: Impact strength of specimen pure epoxy and epoxy +3% glass fiber.

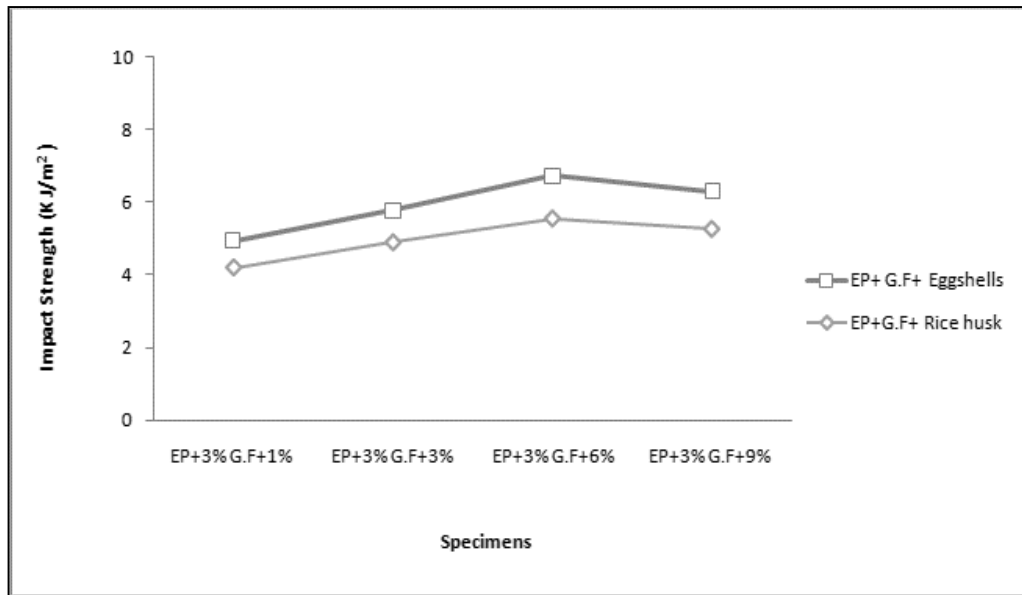


Figure 4.51: The impact strength of specimens strengthened with (glass fiber, before burning rice husk and eggshells).

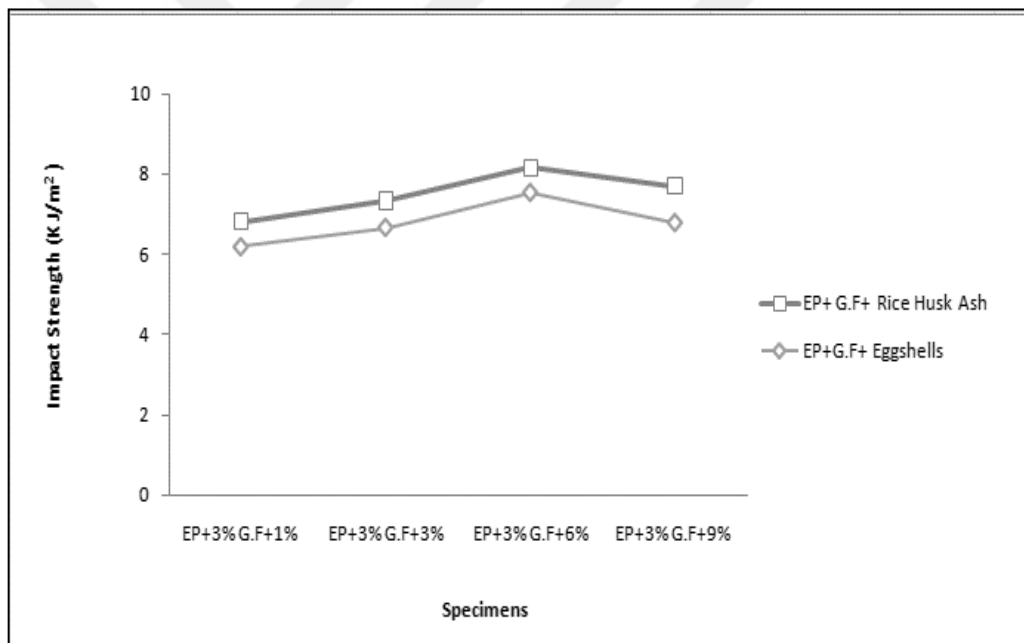


Figure 4.52: The impact strength of specimens strengthened with (glass fiber, after burning rice husk ash and eggshells).

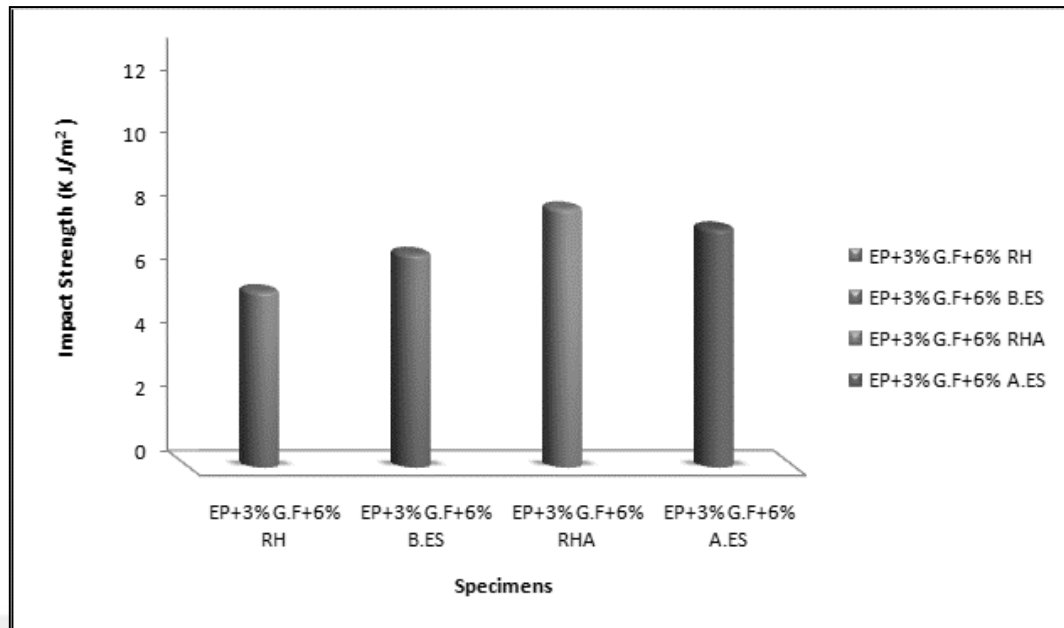


Figure 4.53: Comparing value of impact strength of composite materials reinforced with 6% for all specimens.

4.3.3.2 Fracture toughness

The fracture toughness is a property that determines the materials ability to resist when there is a crack [3]. Fracture toughness depends on the flexural modulus and impact strength for each specimen composite. A specimen of epoxy resin has a fracture toughness equal (2.1MPa.m^{1/2}). Fracture toughness results for all samples with epoxy resin matrix are shown in Figure 4.55 to 4.58. Fracture toughness of specimens composite materials increases with increase weight fraction of fiber and natural powder. That is due these specimens have the ability to crack prorogation and higher flexural modulus. The higher value of fracture toughness was (6.3 MPa.m^{1/2}, 7.2 MPa.m^{1/2}, 8.8 Mpa. m^{1/2} and 8.2 MPa.m^{1/2},) for the specimens (9% RH, 9%B.ES, 9%A.ES and 9%RHA) than other specimen.

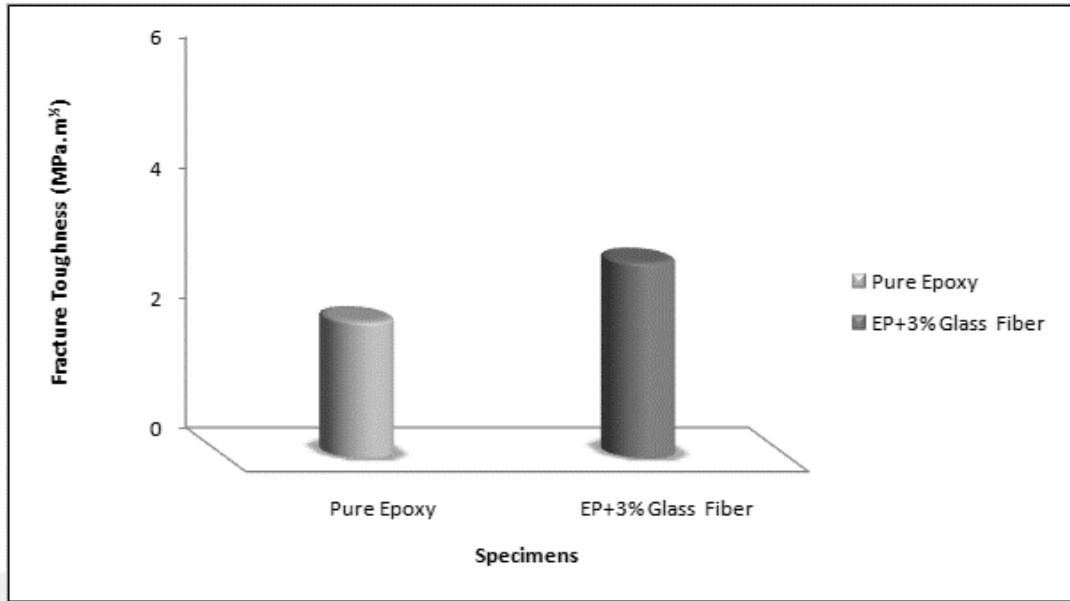


Figure 4.54: Fracture toughness of specimen pure epoxy and epoxy +3% glass fiber.

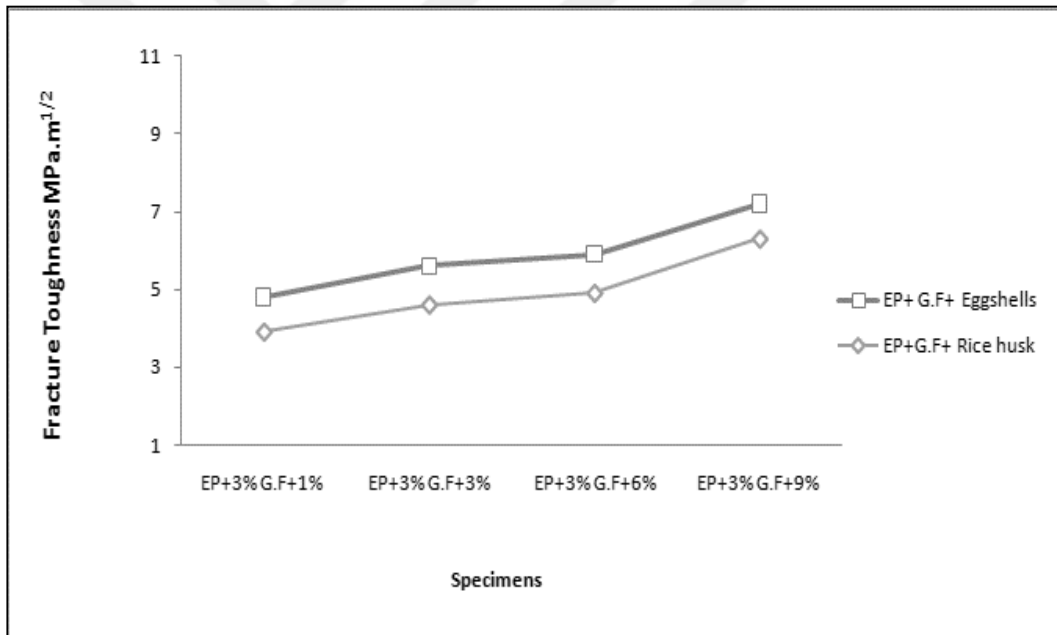


Figure 4.55: Fracture toughness of specimens reinforced with (glass fiber, before burning rice husk and eggshells).

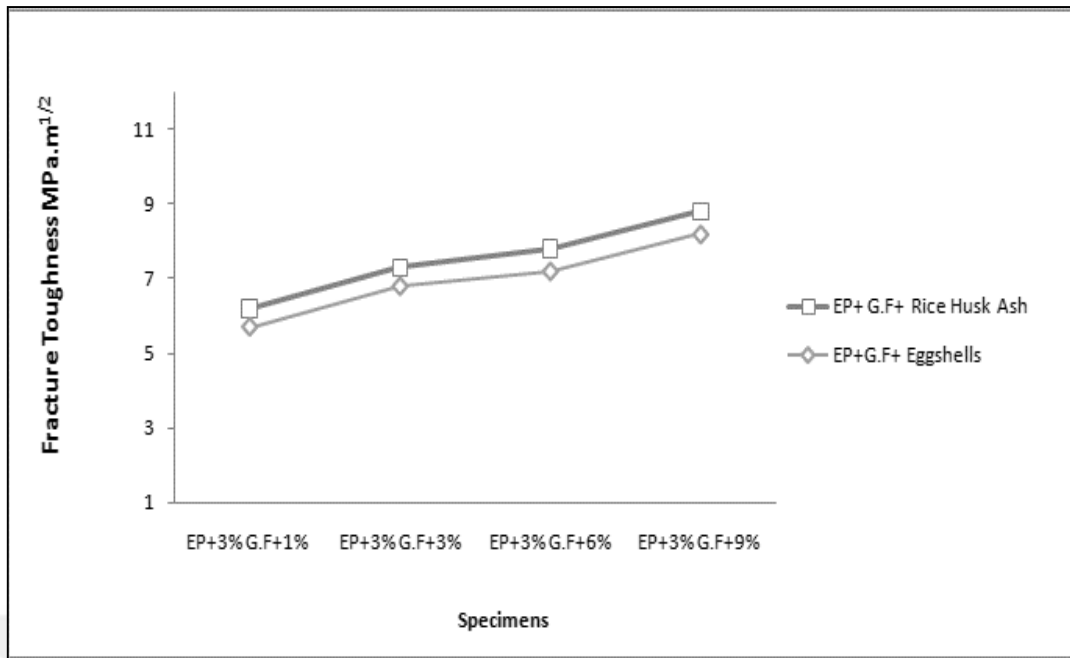


Figure 4.56: Fracture toughness of specimens reinforced with (glass fiber, after burning rice husk ash and eggshells).

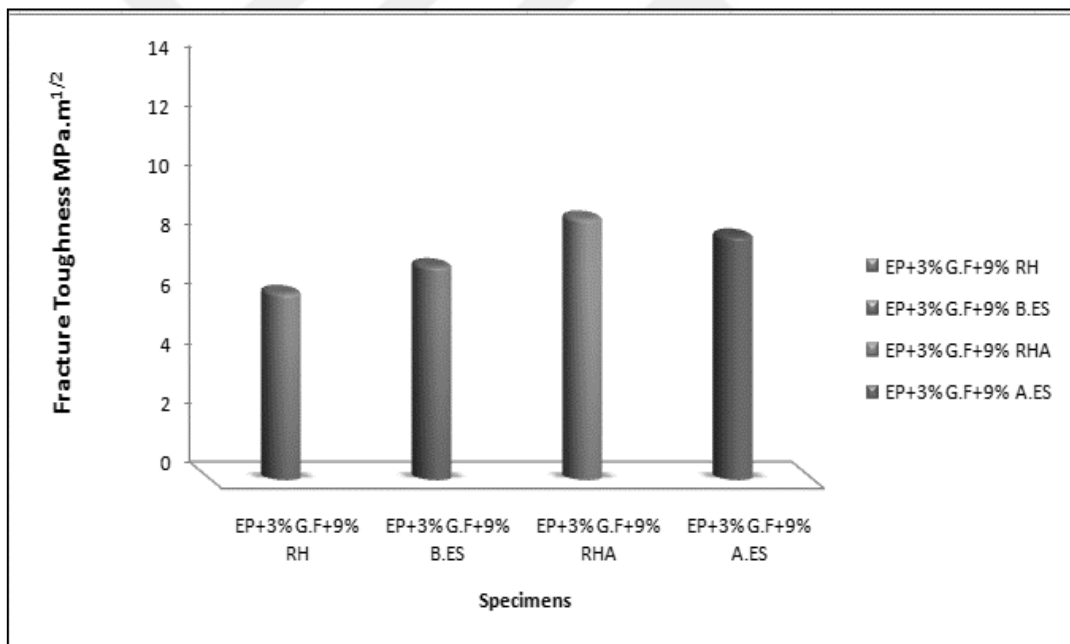


Figure 4.57: Comparing value of fracture toughness of composite materials reinforced with 9% for all specimens.

4.3.4 Hardness (Shore D)

Hardness (shore D) was performed on all samples of natural composite materials. In order to obtain hardness results in high accuracy, has taken an average of five readings for each sample. The values of hardness shore D for the specimens (EP, EP +3% G.F) as shows in Figure 4.59, the presence of fiber glass with epoxy resin

improves the hardness value of the composite materials, this is because hardness is generally considered a surface property, therefore this behavior of hardness is expected. The addition of the fiber leads to decrease in the matrix resin surface resistance to the indentation and an increase in the elasticity and [114], therefore specimen (Ep +3% G.F) has a higher hardness than specimen epoxy resin matrix. Figure 4.60 and 4.61 indicate the effect of adding 3% glass fiber with 1%,3%,6% and 9% weight fraction from (before, after burning of rice husk, rice husk ash and eggshells) powder on the hardness shore D for composite material. From these figures it can be observed that hardness is increased with increasing weight fraction. The result had revealed that the hardness of pure epoxy alone was (75 shore D) compared to maximum value (83) at weight fraction of (9%) RHA with mean particle size is (10 μ m), the reason for the increase in hardness is that RHA contains any elements harder than the pure epoxy that lead to an increase in hardness. These results become matched with our work because the RHA has a particle size smaller than other powder. From the Figure 4.62 can be seen when comparing the values of hardness shore D for all of the prepared specimens composites the specimen (epoxy + 3% glass fiber +9% RHA) gave a higher value of hardness shore D than other specimens. Generally in this work increased hardness with increased weight fraction, the reason is due to increased cross-linking and stacking, which reduces the movement of polymer molecules and making it to become more resistant to the penetration of scratching [115].

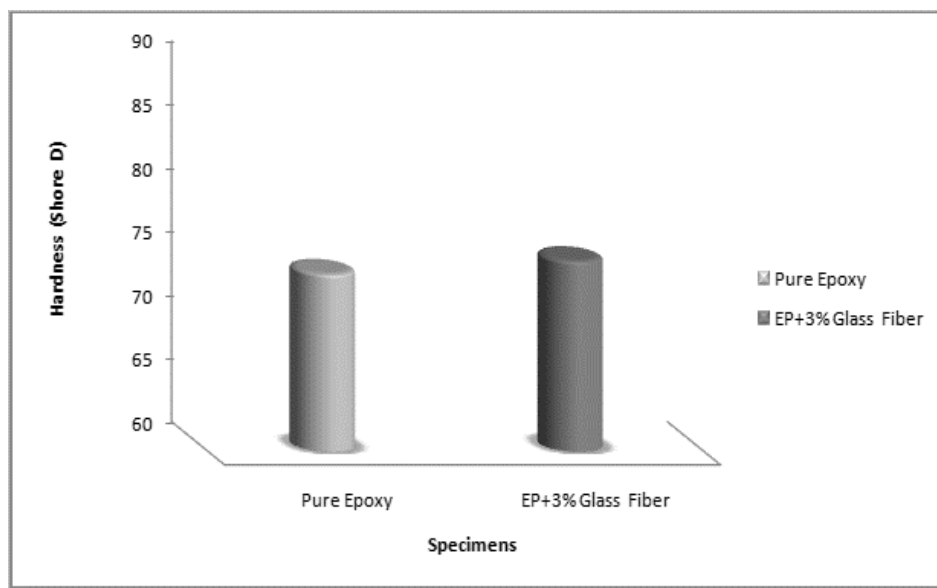


Figure 4.58: Hardness shore (D) for specimen pure epoxy and epoxy +3% glass fiber.

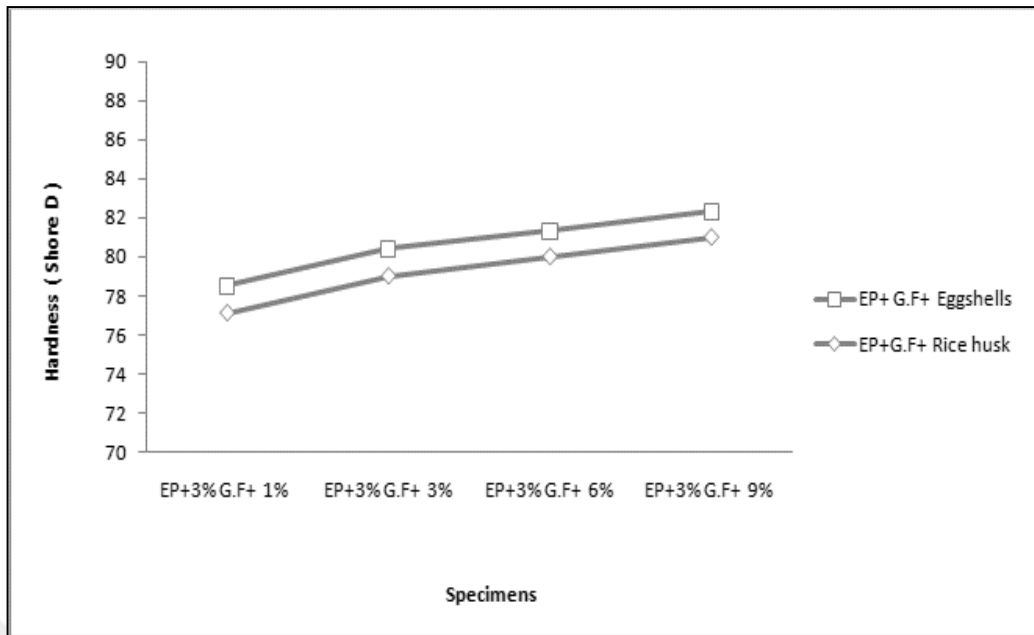


Figure 4.59: Hardness shore (D) for specimens reinforced with (glass fiber, before burning rice husk and eggshells).

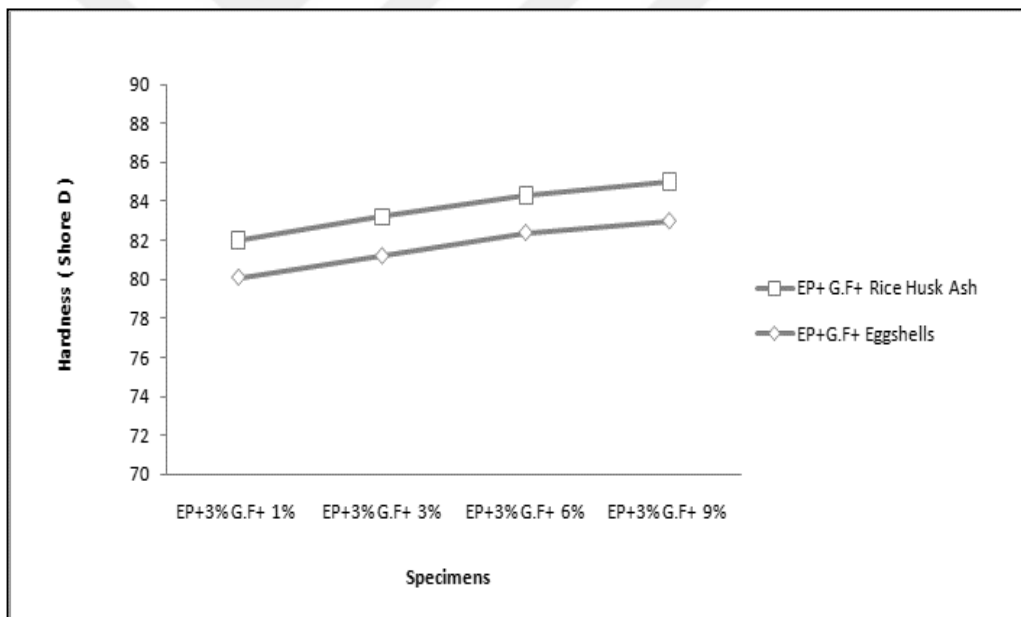


Figure 4.60: Hardness shore (D) for specimens reinforced with (glass fiber, after burning rice husk ash and eggshells).

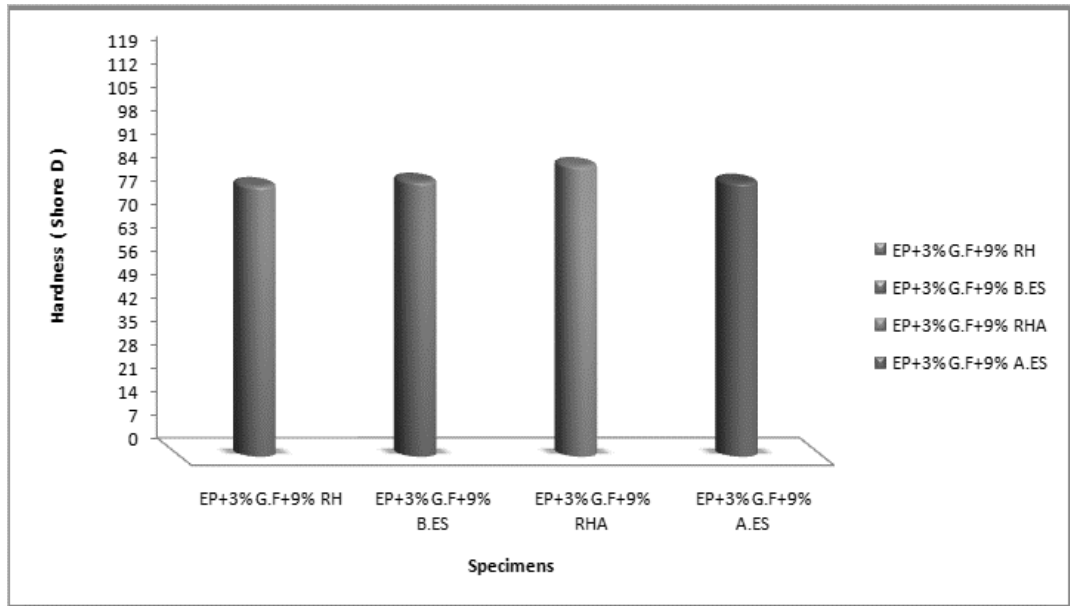


Figure 4.61: Comparing value of hardness shore (D) of composite materials reinforced with 9% for all specimens.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDED FUTURE WORKS

5.1 Conclusions

Listed below the conclusions drawn from the present work are:

1. Non – reinforced epoxy has lower physical and mechanical properties than natural powder composite specimens.
2. Natural powder composite with 3% glass fiber and 9% weight fraction of the powder have the highest density when compared with other weight fraction. Specimen (epoxy + 3% glass fiber +9% B.ES) has the maximum density value (4.871 gm/cm^3) when compared with other specimens.
3. The water absorption of natural powder composites has given higher percentage than pure epoxy and (epoxy+3% glass fiber) composites. Specimens (epoxy+3% glass fiber +9% RH) have the maximum value water absorption (0.334) % when compared with other specimens.
4. The largest values of thermal conductivity and thermal diffusivity at 9 % weight fraction for specimen (epoxy +3%glass fiber+9%A.ES).
5. The largest values of specific heat at 1 % weight fraction for specimen (epoxy +3%glass fiber+9%RH).
6. Increase the modulus of elasticity with an increase in the weight fraction of the reinforcing natural powder for the epoxy resin matrix polymer. The small particle size for natural powder improves the tensile properties of pure epoxy resin matrix more than large particle size.
7. The largest values of elongation percentages at break at 1 % weight fraction for specimen (epoxy +3% glass fibers+1%RHA).
8. The largest values of tensile strength at 9 % weight fraction for specimen (epoxy +3%glass fiber+9%RHA).

9. Flexural strength, flexural modulus and maximum shear stress increase with increasing weight fraction and reach the maximum amount at addition of the weight fraction of (9% weight) and (10 μm) particles size of powder .
10. The impact strength and fracture toughness of the specimen composite materials decrease with weight fraction of 9% natural powder.
11. The natural composite has a higher hardness than other, due to the combined effect of glass fiber and natural powder. Specimen (epoxy+3% glass fiber +9% RHA) has the maximum hardness of (85) shore (D) than with specimens.

5.2 Recommended Future Works

It is recommended that future works could be:

1. A study other mechanical properties for the natural composites prepared in the current research, such as (fatigue, creep, wear and corrosion) under different environmental conditions.
2. A study of the erosion wear behavior for the natural composites prepared in the current search in different solutions like (acids or water) and studies the resulted mechanical and physical properties.
3. Studying the characterization of epoxy resin matrix / glass fiber and natural powder composite by making FT-IR and DSC analyses.
4. Using nano-powder and study its influence on the physical and mechanical characteristic of the resulting materials.

Using other polymer resin matrix like (polyester resin or PMMA) with carbon fiber and study the mechanical and physical properties.

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