



**PREPARATION POLYMER –BASED  
MATERIALS FOR THERMAL AND ACOUSTIC  
INSULATION SUPPLICATIONS**

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MASTER THESIS  
METALLURGICAL AND MATERIALS  
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Jamal Ismael ABDULHAMEED

## **ABSTRACT**

**M. Sc. Thesis**

### **PREPARATION POLYMER –BASED MATERIALS FOR THERMAL AND ACOUSTIC INSULATION SUPPLICATIONS**

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**Karabük University**

**Institute of Graduate Programs**

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**February 2023, 44 pages**

The economic and environmental benefits of repurposing waste tyres into new products cannot be overstated, and nor can the negative impacts of storing too many tyres in one place be ignored. used Crumbed rubber tires (CRT) as filler material shows a decreasing in the mechanical properties of CRT/polymer composites, due to insufficient bonding between CRT and polymeric matrices. Trimethoxy silyl propyl methacrylate, 3-chloropropyl triethoxysilane, and silica acid, are the three types of silanes coupling agents used to prepare the CRT surface for incorporation as filler materials in an epoxy matrix. To determine the impact of CRT surface treatment on the mechanical properties of the composite, samples containing 10 wt% of both untreated and treated CRT were manufactured and tested. Results show that surface treating with 3-chloropropyl trimethoxy silane coupling agent can increase the mechanical properties of CRT/epoxy composite by 56%, 30%, and 36%,

respectively, compared to non-treated CRT/epoxy, while surface treating with other types of silanes coupling agents can produce less mechanical improvement but still better than that of non-treated.

By creating (10,20,30,40) wt.% treated CRT/epoxy composites, we may also investigate how increasing the CRT impacts the composite's thermal, acoustic insulation, and mechanical properties. The results demonstrate that as the percentage of CRT in the material increases, its tensile strength, modulus of elasticity, and impact strength drop but its acoustic insulation improves.

**Key Word** : Polymer matrix composites; crumbed rubber tires; Epoxy thermal conductivity; acoustic insulation; mechanical properties.

**Science Code** : 91524

## **ÖZET**

**Yüksek Lisans Tezi**

### **ISI VE AKUSTİK YALITIM İÇİN HAZIRLIK POLİMER ESASLI MALZEMELER EKLENTİLERİ**

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Atık lastikleri yeni ürünlere dönüştürmenin ekonomik ve çevresel faydaları göz ardı edilemez ve çok fazla lastiği tek bir yerde depolamanın olumsuz etkileri de göz ardı edilemez. Dolgu malzemesi olarak kullanılan kullanılmış lastiklerden (CRT) elde edilen kırıntı kauçuğu, CRT ile polimerik matrisler arasındaki yetersiz bağ nedeniyle CRT/polimer kompozitlerin mekanik özelliklerinde bir azalma gösterir. Trimetoksi silil propil metakrilat, 3-kloropropil trietoksisilan ve silika asit, CRT yüzeyini bir epoksi matrisine dolgu malzemeleri olarak dahil edilmek üzere hazırlamak için kullanılan üç tip silan bağlama maddesidir. CRT yüzey işleminin kompozitin mekanik özellikleri üzerindeki etkisini belirlemek için, hem işlenmemiş hem de işlenmiş CRT'nin ağırlıkça %10'unu içeren numuneler üretildi ve test edildi. Sonuçlar, 3-kloropropil trimetoksi silan birleştirme ajanı ile yüzey işleminin, CRT/epoksi kompozitin mekanik özelliklerini işlenmemiş CRT/epoksi ile

karşılaştırıldığında sırasıyla %56, %30 ve %36 artırabildiğini, diğer yandan yüzeyl işlemenin1 diğer maddelerle yapıldığını göstermektedir. silan türleril birleştirme ajanları, işlenmemiş olanlardan 1 daha az 1 mekanik iyileştirmel sağlayabilir, ancak yine de daha iyil1 sağlayabilir. (10,20,30,40) wt.% CRT/epoksi kompozitler oluşturarak, CRT'nin arttırılmasının kompozitin termal, akustik yalıtımı ve mekanik özelliklerini nasıl etkilediğini de araştırabiliriz. Sonuçlar, malzemedeki CRT yüzdesi arttıkça, çekme dayanımının, esneklik modüllerinin ve darbe dayanımının düştüğünü ancak akustik yalıtımının iyileştiğini göstermektedir.

**Anahtar Kelime:** Kompozitleri etkiler; termal, akustik yalıtım; hafif çelik; polimerik matrisler.

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## CONTENTS

	<b><u>Page</u></b>
APPROVAL.....	ii
ABSTRACT.....	iv
ÖZET.....	vii
ACKNOWLEDGMENT.....	ix
CONTENTS.....	x
LIST OF FIGURES .....	xiii
LIST OF TABLES .....	xv
SYMBOLS AND ABBREVIATIONS INDEX .....	xvi
PART 1 .....	1
INTRODUCTION .....	1
1.1. SCRAP TYRES.....	2
1.1.1. Expird Tyres (Waste Tyres).....	4
1.1.2. Tyre Disposal Options .....	4
1.1.3. New Material Produced Through Scrap Tire Recycling .....	5
1.1.4. Technology for Mechanical Grinding.....	6
1.1.5. Fine Rubber by Grinding Cryogen .....	6
1.1.6. Wet Milling Process.....	6
1.1.7. Water Jet Milling Process .....	7
1.1.8. Classification Scrap Tyres Aggregate.....	7
1.2. EPOXY.....	8
1.1.9. Epoxy Resins and its Types .....	9
1.1.10. Epoxy Resin Applications .....	10
1.1.11. Advantages/Characteristics of Epoxy.....	10
PART 2 .....	11
LITERATURE REVIW .....	11
2.1. LITERATURE REVIW USING EPOXY AND RUBBER TYRE.....	11

	<u>Page</u>
PART 3 .....	17
EXPERIMENTAL STAGE .....	17
3.1. INTRODUCTION.....	17
3.2. MATERIALS USED IN EXPERIMENTAL WORK.....	17
3.2.1. Silane .....	17
3.2.2. Crumbed <sup>1</sup> Rubber <sup>1</sup> Tires CRT .....	18
3.3. EPOXY RESIN .....	18
3.4. RUBBER SURFACE TREATMENT .....	19
3.4.1. Specimens' Preparation.....	19
3.5. MATRIX EPOXY RESIN <sup>1</sup> .....	20
3.6. TENSILE TEST .....	20
3.7. FLEXURAL TEST.....	21
3.8. ANALYSIS BY MEANS OF FOURIER TRANSFORM INFRARED SPECTROMETRY .....	22
3.9. IMPACT TEST .....	23
3.10. HARDNESS TEST .....	24
3.11. THERMAL PROPERTIES TEST .....	25
3.12. ACOUSTIC INSULATING TEST .....	26
 PART 4 .....	 28
RESULTS & DISCUSSION .....	28
4.1. INTRODUCTIONS.....	28
4.2. THE EFFECTS OF CRT SURFACE TREATMENT .....	28
4.2.1. Hardness Tests Results .....	29
4.2.2. FTIR Inspection .....	29
4.2.3. Impact Tests.....	30
4.2.4. Tensile Tests .....	31
4.3. MECHANICAL, THERMAL AND ACOUSTIC INSULATION PROPERTIES OF EPOXY/ SURFACE TREATED CRT COMPOSITES ..	32
4.3.1. Epoxy/ B-Silane Treated CRT Composite Hardness Tests .....	33
4.3.2. Epoxy/ B-Silane Treated CRT Composite Tensile Strength .....	33
4.3.3. Epoxy/ B-Silane Treated CRT Composite Flexural Strength Tests .....	35
4.3.4. Epoxy/ B-Silane Treated CRT Composite Impact Tests .....	35
4.3.5. Epoxy/ Treated CRT Thermal Conductivity .....	36

	<u>Page</u>
4.3.6. Epoxy/ Treated CRT Composite Acoustic Insulation .....	37
PART 5 .....	38
CONCLUSIONS.....	38
REFERENCES.....	39
RESUME .....	44

## LIST OF FIGURES

	<b>Page</b>
Figure 1.1. Waste tyres.....	2
Figure 1.2. Waste Hierarchy .....	4
Figure 1.3. Types of torn rubber .....	7
Figure 1.4. Epoxy hardening.....	9
Figure 1.5. Categories of Epoxy Resin .....	10
Figure 3.1. Show the Samples (a) Before <sup>1</sup> Testing <sup>1</sup> , (b) tensile <sup>1</sup> Testing <sup>1</sup> Machine, (c), (d) after <sup>1</sup> Testing.....	21
Figure 3.2. Show the Samples (a) Before <sup>1</sup> a Test, (b) Flexural Test Machine <sup>1</sup> (c)& (d) after <sup>1</sup> a Test.....	22
Figure 3.3. The FTIR analyser.....	23
Figure 3.4. (a) Samples Before <sup>1</sup> a Test, (b) The Izod impact <sup>1</sup> test device,(c)& (d) Samples <sup>1</sup> after <sup>1</sup> a Test.....	24
Figure 3.5. The (a) Shore D durometer and (b) and (c) samples of various harnesses.....	25
Figure 3.6. Prepared epoxy and composite specimens .....	25
Figure 3.7. Lee's disc method.....	26
Figure 3.8. Acoustic Isolation Gear .....	26
Figure 3.9. A- Epoxy pure. B- Epoxy +CRT. C- Epoxy + Treated CRT.....	27
Figure 4.1. Shore D hardness of epoxy, treated CRT composite, and non-treated CRT composite.....	29
Figure 4.2. FTIR transparency of untreated CTR, CTR treated with (A and B), and (C) silane <sup>1</sup> coupling <sup>1</sup> agents.....	30
Figure 4.3. Fracture <sup>1</sup> toughness <sup>1</sup> of epoxy / treated <sup>1</sup> and non-treated <sup>1</sup> CRT composite. .....	30
Figure 4.4. Stress-strain <sup>1</sup> behaviour of <sup>1</sup> epoxy resin and <sup>1</sup> epoxy /10 wt. CRT composites.....	31
Figure 4.5. Maximum tensile <sup>1</sup> strength <sup>1</sup> of epoxy <sup>1</sup> and treated <sup>1</sup> , non-treated <sup>1</sup> CRT composite.....	32
Figure 4.6. Development of composite hardness with treated CRT weight fraction. 33	
Figure 4.7. Stress-strain behaviour of epoxy and epoxy / different CRT weight fractions composite.....	34
Figure 4.8. Maximum tensile strength of epoxy and variant treated CRT weight fraction.....	34

	<b><u>Page</u></b>
Figure 4.9. Impact strength of epoxy and variant treated CRT weight fraction. ....	35
Figure 4.10. Impact strength of epoxy and variant treated CRT weight fraction. ....	36
Figure 4.11. Thermal conductivity of epoxy and epoxy/different weight fractions CRT composites. ....	36
Figure 4.12. Epoxy/ treated CRT composite acoustic insulation.....	37

## LIST OF TABLES

	<b>Page</b>
Table 3.1. Experimental work involving silane coupling agents. ....	17
Table 3.2. The crumbed rubber tires CRT properties. ....	18
Table 3.3. Epoxy Resin's Mechanical and Physical Properties.....	19
Table 3.4. Coding <sup>1</sup> of CRT / epoxy <sup>1</sup> composite <sup>1</sup> specimens. <sup>1</sup> .....	20
Table 3.5. Epoxy Resin's Mechanical and Physical Properties.....	20

## **SYMBOLS AND ABBREVIATIONS INDEX**

### **SYMBOLS**

ASTM	: American society for testing material
CRT	: Crumbed rubber tiers
FTIR	: Fourier transform infrared spectroscopy
ISO	: International standard organization
FS	: Flexural strength
E.P	: Epoxy



## **PART 1**

### **INTRODUCTION**

During the last two decades, epoxy resin toughening for improved crack resistance has been a hot topic of research [1]. The second method for rubberizing epoxy resins involves the use of prepared rubber particles, such as recycled automotive-tyres particles. Some surface treatment procedures can improve the epoxy resin's compatibility with a rubber particle. The kind of rubber, size, and dispersion of the rubber particles are all crucial factors to consider when selecting rubber to toughen. Waste tyres can be used as fillers for rigid epoxy matrices to increase the tensile strength and hardness of epoxy and rubber products. If the interface between both the epoxy composite and rubber particles is adequate, scrap tyre particles can act as toughening agents or fillers [2]. A second intriguing option for rubber recycling research is the functionalization of waste rubber tyres, which can be used as low-cost, flexible magnetic materials or reinforcing (or semi-reinforcing) fillers in polymer composites, bitumen, and concrete. They also use whole or scrap tyres as fuel for cement kilns, paper mills, and power plants. It is an approach that is pretty risk-free [3]. Waste tyre aggregate can be employed as a filler or toughening agent in hard epoxy matrices. To enhance the bond between the epoxy resin and the recycled rubber particles, silane coupling agents were used. It is used to change the surfaces of rubber particles for this purpose. After the epoxy–rubber specimens have been prepared, the silane coupling agents can strengthen the interface between the epoxy matrix and the rubber particles, resulting in increased strength and modest reductions in sample toughness [2]. Surface-treated rubber particles with a silane coupling agent can raise the viscosity of epoxy asphalt rubber (EAR), and lower the permitted construction time of epoxy asphalt rubber tack coat [4]. Rubber-soil combinations provided good thermal insulation in fly ash and soil mixture [5]. due to the low thermal conductivity of the rubber aggregate [6]. The rubber aggregate is decreasing thermal conductivity sound and enhances its long-term durability [7].

## 1.1. SCRAP TYRES

Scrap tyres are producing severe environmental problems and ecological dangers as a result of the increasing development of the world population and the use of automobiles, which may be handled by recycling them [8]. The recycling of scrap rubber tyres helps to safeguard the environment while also preserving natural resources. Waste disposal is one of the most pressing issues confronting the world today. It might take more than 50 years for leftover tyre rubber to decompose [9]. The environmental and health awareness of discarded tyres has risen dramatically in recent years. This has prompted efforts to find secondary uses that will make use of tyres after they reach the end of their useful lives, reducing landfill waste. shown in Fig.1.1 [10].



Figure 1.1. Waste tyres [11].

In the beyond decades, scrap tyres were posing good sized troubles to the environment. Every year, approximately 800 million waste tyres are produced worldwide, the bulk of which can be generated in advanced nations and it's miles predicted to growth through approximately each year. This parent debts for 2% of the entire annual strong waste manufacturing through weight [12]. Just with inside the United States, over 2.5 million heaps of scrap tyres had been generated with inside the year of 2005, which changed into equal to nearly three hundred million passenger tyres refer Figure1.2. The quantity of waste tyres produced in different advanced nations is likewise huge; approximately 2 million heaps are generated with inside the European Union yearly and 0.5 million heaps are created in Japan [12]. Traditionally, maximum scrap tyres had been dumped in landfills and in monofills. It is apparent and extensively agreed that landfilling isn't a positive choice to deal with waste tyres thinking about fabric loss, land wastage and unfavorable environmental impacts. Scrap tyres also are at risk of fires from which poisonous smoke and oils are generated, inflicting good sized air and water pollution. In mild of those drawbacks, policies were laid down in many nations to save you similarly landfilling. For example, the European Commission imposed a ban at the disposal of entire scrap tyres and shredded tyres in landfills in 2003 and 2006 respectively [13]. Most of the states with inside the United States have followed sure regulations on putting scrap tyres in landfills (refer Table I), and some of them have even banned all tyres from landfills [14]. This has caused the accelerated intake of scrap tyres in power restoration and civil engineering applications, to be able to be similarly mentioned as follows. However, the scrap tyre hassle has emerged as an increasing number of approaching in latest years. With the fast growth with inside the variety of vehicles, the quantity of scrap tyres in growing international locations is developing at an alarming speed. Taking China as an example, the once-a-year manufacturing of tyres soars to over 350 million tyres in 2007, that's extra than triple of that during 2001. Following the fast financial increase of many growing international locations, it's miles envisaged that this determine will retain to upward push with inside the coming decade. Unfortunately, there may be neither any recognized law to limit the landfilling of waste tyres nor any coverage to inspire the reuse and recycling of tyres. What makes this case even worse and extra complicated is that many scrap tyres are exported from advanced international locations to growing international locations to

be able to keep disposal charges and to fulfill the more and more stringent necessities for the reuse and recycling of tyres. [13]. waste tyres have already emerge as a first-rate form of strong waste in latest years. It is consequently critical to undertake suitable waste control practices to hamper the large scrap tyre stockpiles and to address the massive quantity of waste tyres generated each year. The suitability of diverse feasible remedy strategies may be assessed in phrases of sustainability and the related environmental impacts [13].

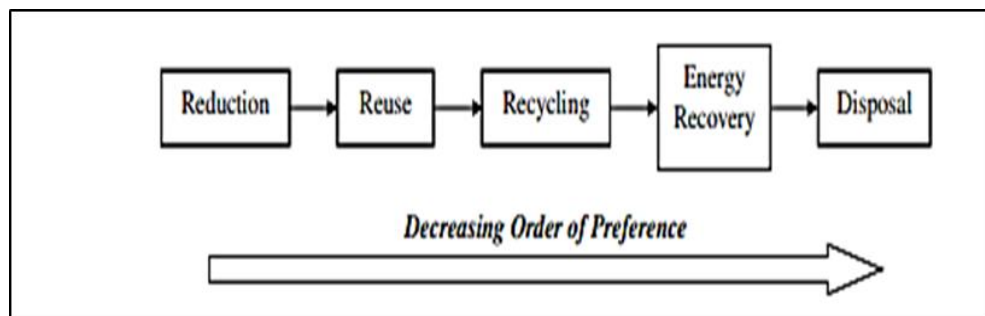


Figure 1.2. Waste Hierarchy [12].

### 1.1.1. Expird Tyres (Waste Tyres)

Tyres that have reached their expiration date or can no longer be used are referred to as waste or junk tyres. Each year, the United States and Europe manufacture six million tonnes of discarded tyres. [15]. Due to the negative effects on the environment and associated health hazards, properly disposing of the vast volume of waste tyres produced each year has become a significant problem. Pointing out that used tyres are important carbon sources and can be used as raw materials for a range of applications is beneficial. The sections that follow outline common tyre recycling garbage techniques.

### 1.1.2. Tyre Disposal Options

Tyre disposal methods such as dumping tyres in open areas or landfills are not viable and have grown increasingly unpopular in recent years. There are currently over four billion trash tyres stacked up around the planet. In Australia, 34% of used tyres are dumped on the ground. Massive heaps of used tyres and landfilling are

unsustainable management techniques that cause a never-ending list of environmental and health issues. In addition to taking up a significant amount of space (75% vacant area), they also harbor numerous vermin, mosquitoes, and other creatures that spread infectious and unidentified diseases. [14]. The dissolution of metals and other compounds poses a danger to the environment as they are hazardous to the environment and can pollute soil and water. Embedded tyre length is important. Leachate is likely to move more slowly when whole tyres are stacked than when they are shredded. Leaching is affected by water contact with the tyres. highly permeable soils may further increase permeation. [14]. The potential for a fire to break out of control is one of the biggest risks associated with waste storage that affects both the environment and human fitness. [15]. Tyres are very difficult to ignite, yet tyre fires are quite challenging to extinguish. Numerous cases were documented, and it was possible to determine how environmental contaminants and fitness issues could have an impact. The gases emitted by tyre fires contain dangerous levels of CO, CO<sub>2</sub>, and sulfur oxides, making them hazardous to humans and their surroundings. Tyre fires are expensive to put out and clean up.

### **1.1.3. New Material Produced Through Scrap Tire Recycling**

Tyre trash can also be utilised as feedcloth to make new polymeric products because tyres contain a significant amount of rubber, a polymeric cloth. The ability to mix the used tyres with a polymeric material, uncooked cloth, the scrap tyres must first be shredded or floored. Diverse grinding techniques may aid us in downsizing this hard cloth in order to organize tyres for sparkling polymeric manufacturing. A specialised mechanical device that can shred and grind tyres to an extremely small length and at a high degree of purity is needed since grinding is a difficult process and because tyres have an excessive level of mechanical strength.

Crumb rubber is created by grinding discarded tyres into extremely small pieces, and it can be utilised as a raw material to create a variety of rubber products. The main challenge with this method is the need to first remove the metal and material used in tyre production. Because the removed metal and cloth are recyclable materials that can be sold, it is profitable. Additionally, the fabric can be recovered after cleaning

by either producing insulation or using it to save electricity (fabric combustion) [18, 19]. There are specific sorts of grinding, including mechanical, cryogenic, moist, and excessive-stress grinding.

#### **1.1.4. Technology for Mechanical Grinding**

In mechanical grinding, the tyre is ground at room temperature using a mechanical device that includes a shredder, rolling generators, and granulators. Until the desired length of 0.3 mm is reached, this is repeated. Metal and material are removed from the grinding process using a separator and an electromagnetic device, respectively. The tyre debris need to be cooled so that it will save you combustion because of warmth launched all through the mechanical processes [20].

#### **1.1.5. Fine Rubber by Grinding Cryogen**

In the cryogenic method, the tyre is frozen using liquid nitrogen to a temperature between 80 and 100 C and then pounded into tiny fragments. Fabric and steel are no longer used (like with inside the mechanical grinding approach). The presence of moisture after all of the tyres have been installed is a drawback of the cryogenic method. A compressor machine may additionally update the liquid nitrogen to lower the temperature to reduce the costs associated with this machine [20]. Rubber floor debris were created in a study by Liang and Hao [21], which was done by their methods. They should utilise a cryogenic grinding machine, a separator, and a vortex mill to grind trash tyres, and they should deliver debris that is less than 50 millimetres long and has very smooth surfaces.

#### **1.1.6. Wet Milling Process**

With high floor and extent and a low level of granulate degradation, the moist grinding method may produce very nice comminuted grain. Tyres must be pre-shredded in order to be ground up using the damp grinding method. the use of grinding stones. The articles enter a drying process after being continuously cooled by the water [19–21].

### 1.1.7. Water Jet Milling Process

The water jet grinding method is suitable for large-sized tyres, such as those used on tractors, lorries, and other vehicles. Tyres are cut into thin strips using a jet of water that is accelerated and stressed above safe levels (over 2000 bar). The advantage is that the floor rubber is extremely pure because the metal fabric inside the tyres is preserved during the grinding process. It is an environmentally friendly strategy that also saves electricity. It takes trained personnel to operate high-pressure equipment [22].

### 1.1.8. Classification Scrap Tyres Aggregate

Rubber tyres come in a wide range of shapes and sizes. Fig. 1.3: The most frequent varieties of old tyre rubber are chips, crumbs, and ground [23].

- Rubber can be used as a gravel substitute if it has been shredded or diced. The tyre must be shredded twice to produce this rubber. At the end of the first step, the rubber should be 300-430 mm long and 100-230 mm wide. It is then cut down to 100-150 mm in the second stage. Shredding produces "chips particles" with diameters ranging from 5 to 76 (mm).
- Crumbs Rubber is made by shredding large rubber into tiny bits using hydraulic mills, which can be used in place of sand. Depending on the type of mills used, this process may produce rubber particles of different sizes. In contrast, the range of the particle size, from 0.425 to 4.75 (mm), makes the process of creating particles simple.

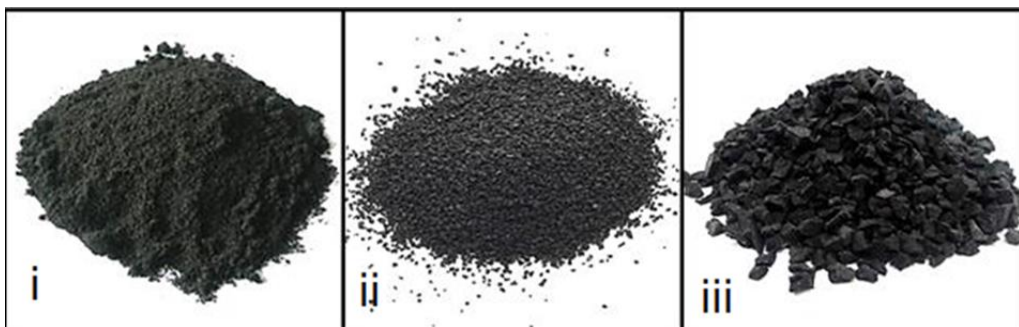


Figure 1.3. Types of torn rubber [24].

## 1.2. EPOXY

Epoxy resins are the resins that come below the class of “thermoset” own circle of relatives of resins, at the side of polyester, silicones, urethanes, melamines, acrylics, and phenolics. Epoxy resins as soon as cured can't be melted through the utility of heat. This makes epoxy resins specific amongst different plastics like polyethylene, vinyl polypropylene, etc., which may be melted and molded time and again again. The maximum generally used artificial epoxy resin is the diglycidyl ether of bisphenol A (DGEBA). The epoxy resins utilized in adhesive formulations range from aliphatic resins having simple, low molecular weight to aromatic resins which can be complicated and multifunctional. epoxy resins are used within the “potting and casting” manner to shape inflexible, lightweight, and foamy systems.

Because of its low cost and simplicity of manufacturing, epoxy resin is the most frequently utilized [26]. Epoxy resins are different from other thermosetting resins in that they require less pressure to fabricate products normally used for thermosetting resins; shrinkage is much lower; and thus, the residual stress in the cured product is lower than that seen in the vinyl polymerization used to cure unsaturated polyester resins; they can be cured at a wide range of temperatures with good control over the degree of crosslinking. The resin is available in a variety of forms, ranging from a low-viscosity liquid to a tack-free solid, and so on. Epoxy resins are widely used in structural adhesives, surface coatings, engineering composites, and electrical laminates due to their unique characteristics and useful properties, such as high strength, very low creep, excellent corrosion, and weather resistance, elevated temperature service capability, and adequate electrical properties [1]. Epoxy hardening is shown in Fig. 1.4.



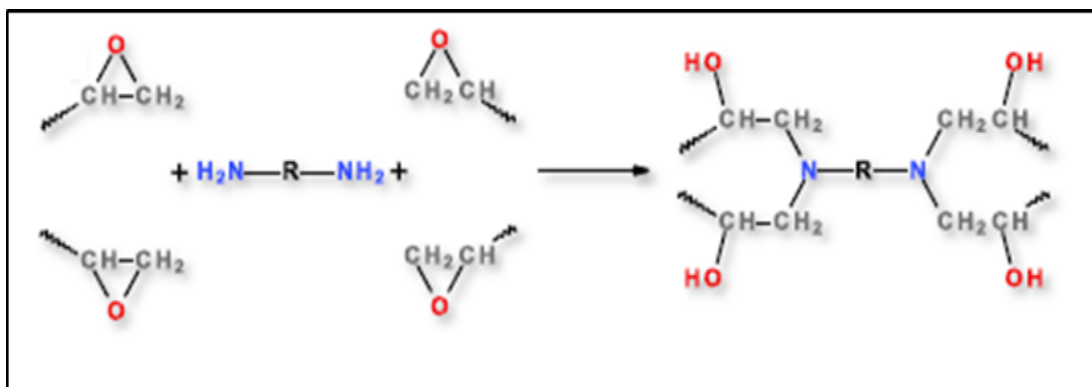


Figure 1.4. Epoxy hardening [3].

Advantages of epoxies over all different polymeric substances in coating Epoxy has the ability of adhesion firmly to maximum of the substances such as metals, concrete, glass, ceramics, stone, wood, leather, etc. These are flexible; enormously proof against chemical solvents; and ambient moisture because of their lengthy hydrophobic chains, first rate electric insulating houses, and minimal shrinkage on curing which offers desirable dimensional stability. They are widely known for excessive tensile, flexural, and modulus houses [22].

### 1.1.9. Epoxy Resins and its Types

Epoxy resins are oxirane-containing oligomers. These resins are heat-placing resin made via way of means of the chemical bonding of smaller molecules into large ones. In 1927, Schrade suggested the primary training of resins from ECH. Later in 1936, Castan produced a low-melting, amber-coloured ECH-bisphenol A resin which changed into reacted with phthalic anhydride to provide a thermoset.[25] In 1939, Greenlee explored the ECH-bisphenol A synthesis direction for the manufacturing of recent resins for coating applications. Commercially, the primary resin changed into the response fabricated from ECH and bisphenol A produced in 1947. Nowadays a extensive form of epoxy resins are available. Epoxy resins fall into sorts primarily based totally on their molecular shape and applications, specifically the glycidyl epoxy and nonglycidyl epoxy. These may be similarly divided into 3 sorts on the idea in their shape: glycidyl ether, glycidyl ester, and glycidyl amine (Fig. 1.5).

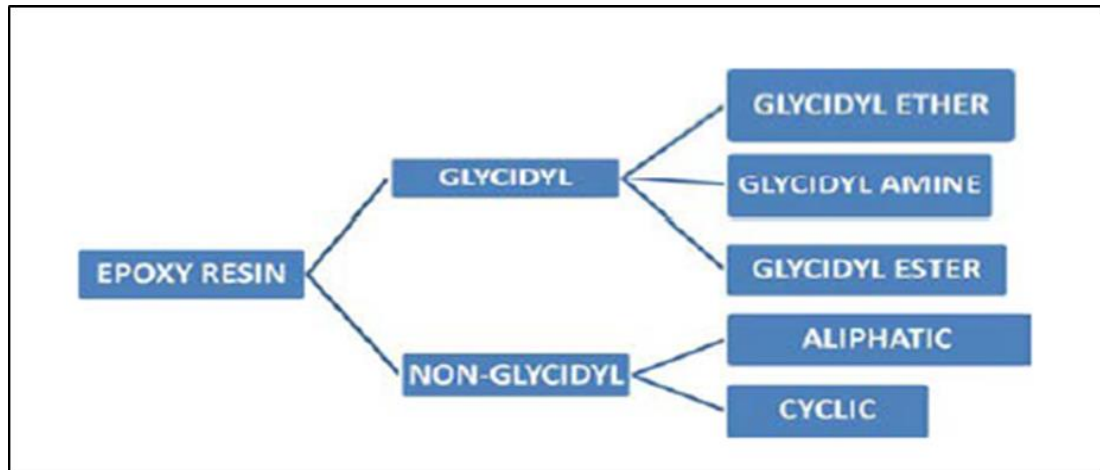


Figure 1.5. Categories of Epoxy Resin [25].

#### 1.1.10. Epoxy Resin Applications

- Water infiltration and corrosion-promoting media are effectively blocked.
- Adhesion-promoting injection resin for concrete, masonry, stone, steel, and wood.
- Used to fill and seal holes and cracks in constructions such as bridges and other civil engineering, industrial and residential buildings, and water retaining structures, such as columns, beams, foundations, walls, floors, and water retaining structures.
- Forms a structural link between concrete pieces.

#### 1.1.11. Advantages/Characteristics of Epoxy

- Hard.
- Shrinkage free hardening.
- Solvent free.
- High mechanical and adhesive strengths.
- Suitable for both dry and damp conditions. Low viscosity and injectable with single component

## PART 2

### LITERATURE REVIEW

#### 2.1. LITERATURE REVIEW USING EPOXY AND RUBBER TYRE

In this part we will discuss the previous researches that deal with crumbed rubber tires using as filler material in polymeric matrix composites. Also, we will discuss using silane coupling agents to promote matrix reinforcement phase bonding and the mechanical properties of composite materials.

- **M. Abadyan et al. (2010)** they studied the behavior of amine-terminated butadiene acrylonitrile rubber and recycled tyre particles serving as fine and coarse modifiers in a hybrid rubber-modified epoxy system, which has been examined for its behavior. Synergistic toughening in the hybrid system was demonstrated. The inclusion of 2.5-phr big particles (recycled tyre) and 7.5-phr tiny particles (ATBN) led to synergistic toughening in the hybrid system, according to the blends' fracture toughness (KIC) measurements. Different toughening processes for the blends were visible in transmission optical micrographs; the inclusion of small ATBN particles boosted toughness by expanding the damage zone and causing corresponding plastic deformation close to the crack tip. However, in the case of hybrid resin, significant stress concentrators in the form of coarse recycled rubber particles caused the initial fracture point to branch. fracture toughness (KIC measurement of the blends). Fine ATBN particles increased toughness by enlarging the damage zone and causing plastic deformation around the fracture tip. However, the original fracture tip branched because coarse recycled rubber particles acted as significant stress concentrators[26].
- **Arthanarieswaran V.P. et al. (2014)** The effect of randomly intertwining natural fibres with glass filaments was investigated. Epoxy glue and E-glass

fibre were used to encase shredded banana and sisal fibres. Composite samples were tested for mechanical qualities and subjected to scanning electron microscopy. Tensile strength is increased by 2.34 and 4.13 percent, respectively, when two or three layers of glass fibre are used. The impact strength and flexural properties were best in the sisal and three glass-fiber laminate. As a result of being sandwiched between two layers of glass fibres, banana-sisal fibre gained superior flexural capabilities. The failure of laminates is traced to cavities, insufficient fiber-matrix adhesions, and fibre pullout, as seen by scanning electron microscopy. [27].

- **Mona A. Ahmed et al. (2015)** studied Nanoclay and reactive rubber nanoparticles were tested for their ability to toughen epoxy resins without altering the resins' stiffness, strength, or glass transition temperature. High modulus, chemical resistance, and dimensional stability are just a few of the remarkable features of epoxy resin. Epoxy's high crosslink density makes the material brittle with low impact strength and poor resistance to crack propagation, limiting its wide range of potential uses. The primary goal of this work is to combine organically modified nanoclay with reactive rubber nanoparticles in an epoxy matrix. Synthesis and characterization of epoxy hybrid nanocomposites including RRNP, Cloisite-30B, and a mixture of RRNP and Cloisite-30B. When RRNP is included, a nanocomposite with improved toughness and decreased stiffness takes the place of the traditional resin. The stiffness and toughness of the epoxy and nanocomposites were improved when [o-mmt] was added to the cured epoxy, in contrast to the neat resin [28].
- **Basim Abu-Jdayil et al. (2016)** used different rubber concentrations (0–40 vol.%) to make the polymer composites. Thermal conductivity, water retention, density, and thermal stability tests were used to identify them. According to the findings, rubber particles are an excellent filler that may be combined with polyester to create an insulating composite. Researchers have demonstrated that adding rubber particles to the polymer matrix lowers the density and thermal conductivity of composites. A material with extremely little water retention and useful uses as a thermal insulator may result from this. The density and thermal conductivity decrease as rubber content and size

grow, but water retention rises concurrently. In comparison to plain polymers, the composites showed a slight decrease in heat stability [29].

- **K. Aoudia et al. (2017)** studied of the possibility use waste Tire Rubber (WTR) was devulcanized using microwave electromagnetic energy after being ground into powder using a PQ500 disc mill at room temperature. The study's ultimate goal was to create a novel composite by incorporating it into a thermoset resin. Epoxy composites filled with devulcanized ground tire rubber (DGTR) appeared to have superior mechanical qualities to those filled with untreated ground tire rubber (GTR) [30].
- **Müller et al. (2018)** Researched and assessed how adding active rubber powder (ARP) to a structural two-component epoxy resin modified the material's strength and toughness. The addition of ARP reduced the material's tensile strength and hardness. An interior strain in the adhesive bond layer can be absorbed by elastic ARP. Results from experiments showed that ARP had a beneficial effect on adhesive bonds subjected to cyclic deterioration at both high and low temperatures.[30].
- **Maryam Aliakbari et al (2019)** studied and analyzed to improve epoxy for less brittleness Epoxy use as an adhesive for metal-polymer surfaces is restricted by its brittleness and poor temperature stability. Using the L9 Taguchi experimental design technique, epoxy formulations incorporating phenolic resin, recycled tire powder, and clay nanoplatelets were optimized. The single-lap shear strength increased by roughly 39% in comparison to the blank epoxy glue formulation. Based on measurements of the degradation peak temperature, thermal stability increased by 16%. In comparison to pure epoxy, the modulus and toughness fall by 24.73% and 12.44%, respectively, and 107.18%. Tensile strength, Young's modulus, and toughness are combined into one response and compared between two samples. Sample 1 exhibits a 46 °C rise in T<sub>max</sub> and a considerable increase in thermal stability as measured by TIDT. Both crack tip blocking and crack development route variation in the vicinity of microparticles were suggestive of a significant mechanism of toughening, as shown by SEM micrographs of the fracture surface of epoxy composites[31].

- **Xiaojie Guo et al (2019)** analyzed a process for producing epoxy materials from lignin with well-balanced mechanical and thermal properties. By presents a practical strategy for producing high-performance epoxy resins as well as a green technique for synthesizing polyacids. The attributes of succinylated lignin (SA-KL), which were equivalent to those of various anhydride-cured epoxies, included modulus of 3.62 GPa and flexural strength of 48.8 MPa but poor impact strength of 6.7 kJ/m<sup>2</sup>. It was looked into whether the SA-L might be made tougher by using a liquid rubber with carboxyl-terminated butadiene acrylonitrile (CTBN). Although flexibleural strength decreased as a result of the inclusion of CTBN rubber, impact strength and fracture toughness rose dramatically. At a CTBN latex concentration of 5–12 wt%, a ductile–brittle transition was seen[32].
- **Bernardeta Dębska et al (2019)** studied epoxy mortars with waste rubber tyre, in which tyre-derived granules were employed as a replacement for sand in the amounts of 0, 20, 40, 60, 80, and 100% vol., respectively. The values of the strength parameters of composites decreased together with an increase in the waste content. But it was possible to create a material that has a very low heat conductivity, is lightweight, and has a very low water absorption rate. Researchers from the University of Aberdeen in Scotland have devised a novel method for assessing composites that have been modified using rubber waste. A rubber-mortar-based composite's excellent thermal insulation qualities, according to the simulation findings, proved the material's viability for practical use[33].
- **L.M. Gil-Martín et al (2019)** they did study cycle behavior of epoxy resin and ground tire rubber used as a partial cement substitute in RC beam-column junctions. Two polymer cement concrete (PCC) samples were created using ground tire rubber and epoxy, respectively, to substitute 5% and 15% of the cement (measured in terms of polymer/cement mass ratio (p/c)). The experimental behavior of Reinforced Concrete (RC) beam-column joints constructed with conventional and PCC and subjected to quasi-static reversed cyclic stress was examined in this study. The specimens included a reference specimen built of conventional concrete as a reference, a control specimen, and two further examples in which ground rubber and epoxy resin were used

as partial cement replacements in the joint zone, respectively. Load-bearing capacity, strength loss, ductility, stiffness loss, energy dissipation capacity, equivalent viscous damping ratio, joint damage level, and pinching width ratio were all studied to see how the two polymer-cement concretes affected the overall structural behavior of the RC joints. Epoxy resin concrete was shown to have favorable structural properties.[34].

- **Bidyut Prava Jena et al. (2019)** studied and analyzed the fabrication of composites from epoxy–waste tyre rubber crumb by the hand lay-up method with varying fiber volume fractions (10%, 20%, 30%, 40%, and 50%), followed by an investigation of their physical and mechanical properties. Epoxy resin was filled with rubber crumb to optimize the strength of the composite for structural applications, and WTRC, when added to epoxy, has lessened or improved the polymer matrix composite's hardness, tensile strength, and water absorption properties. Finally, it contributes to a healthier and pollution-free environment[35].
- **Akeem Yusuf Adesina et al. (2020)** they studied micronized waste tire rubber's effects on the tribological and mechanical characteristics of epoxy composite coatings. We looked at epoxy coatings reinforced with 1–20 weight percent micronized waste tire rubber (WTR) for better mechanical and tribological qualities. At an ideal loading of 5 wt% WTR, the hardness increased by 22% relative to the original epoxy coating. After being reinforced with WTR for all loadings, the epoxy composite coatings' coefficient of friction significantly decreased. Similarly, up to a loading of 10% WTR, the wear resistance was increased by over 70%. At greater WTR loadings, however, the mechanical and wear attributes worsened. At an ideal loading of 5 wt%, the WTR reinforced epoxy coatings showed promising characteristics [36].
- **S. Yogeshwaran et al. (2020)** The purpose of this investigation was to develop a hand layup technique for preparing hybrid composites out of jute and abaca fibres reinforced with particles made from recycled tyres. Tire particles and H<sub>2</sub>SO<sub>4</sub> treatment to improve their mechanical qualities have been used in the testing. As a point of departure, we used a range of fiber-to-matrix ratios (80%, 50%, 30%, 40%, 40%, and 30%). Rubber's interaction with epoxy was enhanced by the addition of recycled tyres, leading to greater

stiffness and tensile strength. Epoxy matrix reinforcement made from recycled tyres that had been treated with high-performance polyurethane to increase their elastic capabilities. Some of its mechanical qualities, like tensile strength, flexural toughness, and impact resistance, were measured by testing produced samples. The mechanical parameters of jute fibre, abaca fibre, and tyre particle reinforced epoxy hybrid composites were found to be optimal at a weight ratio of 30:50:20. These composites exhibited a tensile strength of 74.29 MPa, a tensile modulus of 1582.74 MPa, and an impact load of 16.5 J. When compared to untreated composites, pre-treatment composites demonstrated increases in Tensile strength of 128%, Tensile Modulus of 144%, and Flexural Strength of 132% [37] .

- **J. Shao et al. (2020)** evaluated the effects of adding fine, medium, and coarse rubber particles to epoxy concrete at (5, 10, 15, 20%), and 25%, testing their compressive strength, flexural strength, spilt strength, deflection, and strain. In a composite beam test, rubber epoxy concrete is utilised as a repair material to "glue" two ordinary concrete short beams together, and a rubber particle of a medium size is selected for further study. A deformation compatibility parameter is implemented to evaluate the composite beam's pliability via the measurement of two strains. To counteract the rigidity of epoxy concrete, it has been shown that incorporating rubber into the mix increases deformability while yet retaining the interfacial strength necessary for use as a repair medium. [39].
- **Navid H. Arani et al. (2020)** studied how angular silicon carbide particles affected epoxy composites supplemented with rubber particles. For all of the test settings, the erosion rates were discovered to be much lower than those of the neat epoxy. Erosion rates were accurately predicted utilizing the erosion rates of the two constituents using an unique mixing rule based on the reinforcement area coverage to within 0.5%-10% of those measured . The areal coverage was dependent on the volume fraction and reinforcement size and could be predicted with accuracy if the reinforcements were distributed randomly. The method could be useful for predicting how quickly other composites reinforced with elastomers will deteriorate [40].



## PART 3

### EXPERIMENTAL STAGE

#### 3.1. INTRODUCTION

Material used, sample preparation procedures, and test instruments used will all be described in depth in this part.

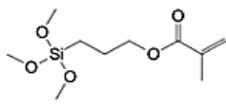
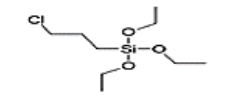
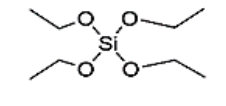
#### 3.2. MATERIALS USED IN EXPERIMENTAL WORK

- Silane
- Crumbed rubber tires (CRT)
- Epoxy

##### 3.2.1. Silane

There are three different silane coupling agents utilised to alter the surface of CRT particles, as illustrated in Table 3-1.

Table 3.1. Experimental work involving silane coupling agents.

Type	Name	Formula	purity	company	Structural
A	Trimethoxy silyl propyl methacrylate	$C_{10}H_{20}O_5$ Si	98%	Sigma-Aldrich USA	
B	3-chloropropyl triethoxysilane	$SiC_9H_{21}ClO_3$	95%	Sigma-Aldrich USA	
C	Silicic acid	$H_4SiO_4$	98%	Glenham life sciences UK	

### 3.2.2. Crumbed Rubber Tires CRT

Experiments were conducted using CRT particles made by an Iraqi firm, Abraj Alkut, specialising in the production of rubber goods. Listed in table (3-2) below is an illustration of the many CRT constructions and a few of their physical features.

Table 3.2. The crumbed rubber tires CRT properties.

No.	Items	specification	Test method
1.	Heating loss	1 % max.	D1509
2.	Sieve analysis	90% min.	D5603
3.	Specific gravity	1.1 – 1.3 gm/cm <sup>3</sup>	D297
4.	<u>Fiber</u> content	0.5% max.	D5603
5.	Steel content	1% max.	D5603
6.	Ash content	5.0 – 15 %	D297

### 3.3. EPOXY RESIN

The epoxy used in the studies was manufactured by the Swiss firm SIKA AG; it consisted of two components (labelled A and B) that were combined to produce a resin with an injection density of about 1.06 Kg/L and a low viscosity. The table (3-3) shows that a ratio of 2:1 between the two components is optimal.

Table 3.3. Epoxy Resin's Mechanical and Physical Properties.

Property	Value
Flexural Strength (MPa)	61
Elongation Percent (%)	2.6
Elastic modulus (GPa)	1.8
Tensile strength (MPa)	25
Thermal conductivity (w/m.k)	0.165-0.17
Density ( gm /cm <sup>3</sup> )	1.1

### 3.4. RUBBER SURFACE TREATMENT

First, the CRT powder washed by washing in distilled water to get rid of dirt and grime. Next, the CRT particles were subjected to an hour-long chemical treatment at room temperature involving the addition of 4 grammes of silane to 100 millilitres of ethanol (95%). Finally, the CRT washed with distilled water and dried in an oven at 50 C° for 1 hour to complete the epoxy matrix modification bonding process.

#### 3.4.1. Specimens' Preparation

For preparation epoxy specimens, Epoxy has two components (A + B) have been mixed with 2:1 ratio respectively. The two parts were mixed by glass stick to get homogenous mixture before pouring into special silicon mould. For preparation composite specimens, predefined CRT weight fractions powder was added to the mixture, then addition mixing by stick was done for three minutes to ensure good dispersion of CTR powder within epoxy matrix before casting the mixture<sup>1</sup> into silicon mould where the specimens it produces in tensile and impact tests were the intended purpose of the mould. Epoxy and epoxy/CRT composite specimens are coding according to CRT weight fraction and type of silane as in table (3-4) below

Table 3.4. Coding of CRT / epoxy composite specimens.

No	CRT wt.%	Epoxy%	Silane treated type	Notation
1	0	100	non	E
2	10	90	Non treated	N
3	10	90	Trimethoxy silyl propyl methacrylate	A
4	10	90	3-chloropropyl triethoxysilane	B
5	10	90	Silicic acid	c

### 3.5. MATRIX EPOXY RESIN

Epoxy resin of the Sikadur - 52 (A+B) kind was manufactured by a firm known as ( Egyptian Swiss Chemical Industries ). The addition of a hardener at a ratio of (2:1). The epoxy resin's measured properties are listed in Table (3-5).

Table 3.5. Epoxy Resin's Mechanical and Physical Properties.

Property	Value
<b>Flexural Strength (MPa)</b>	61
<b>Elongation Percent (%)</b>	2.6
<b>Elastic modules (GPa)</b>	1.8
<b>Tensile strength (MPa)</b>	25
<b>Thermal conductivity (w/m.k)</b>	0.165-0.17
<b>Density ( gm /cm<sup>3</sup>)</b>	1.1

### 3.6. TENSILE TEST

All samples were subjected to a tensile test in order to determine their modulus of elasticity , final tensile strength , and ratio of elongation at fracture, among other tensile parameters. This analysis was performed in accordance with worldwide norms (ASTM-D638). This inspection was carried out using a testing machine ((universal tensile instrument)) with a load capacity of (50 KN; it was manufactured in China and designated as a type (LARYEE). To create the stress-strain curve, we first adjusted the crossover speed head to extrude resin at a rate of 2 millimeters per minute (mm/min), and then we progressively applied tensile loading until the model

cracked. Three samples were examined in each run, and the average results were used for planning purposes [43]. Fig. (3.1) depicts the models used in the experiments.

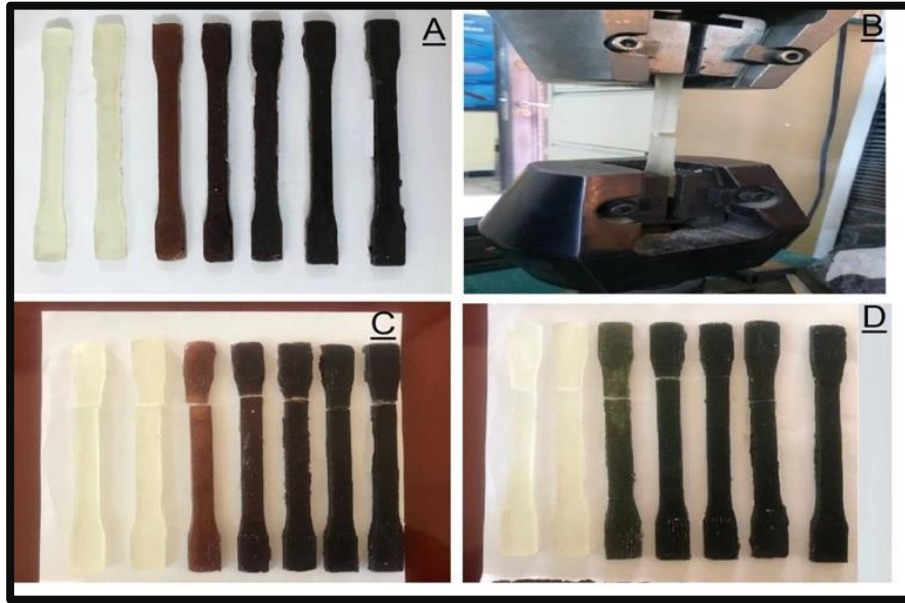


Figure 3.1. Show the Samples (a) Before Testing , (b) tensile Testing Machine, (c), (d) after Testing.

### 3.7. FLEXURAL TEST

Three-point flexural test samples have been depending on to measuring flexural strength according to ASTM (D790), the tests were performed at room temperature and by used universal test machine. The models were placed on the instrument's supports, and a vertical force was applied to the specimen's centre, gradually increasing until the specimen broke, producing a curve showing the connection between force (in newton-meters) and displacement (in millimetres) for each form. Every composite sample's flexural modulus and flexural strength are learned characteristics[44]. Experimental test specimens are depicted in Figure (3.2).

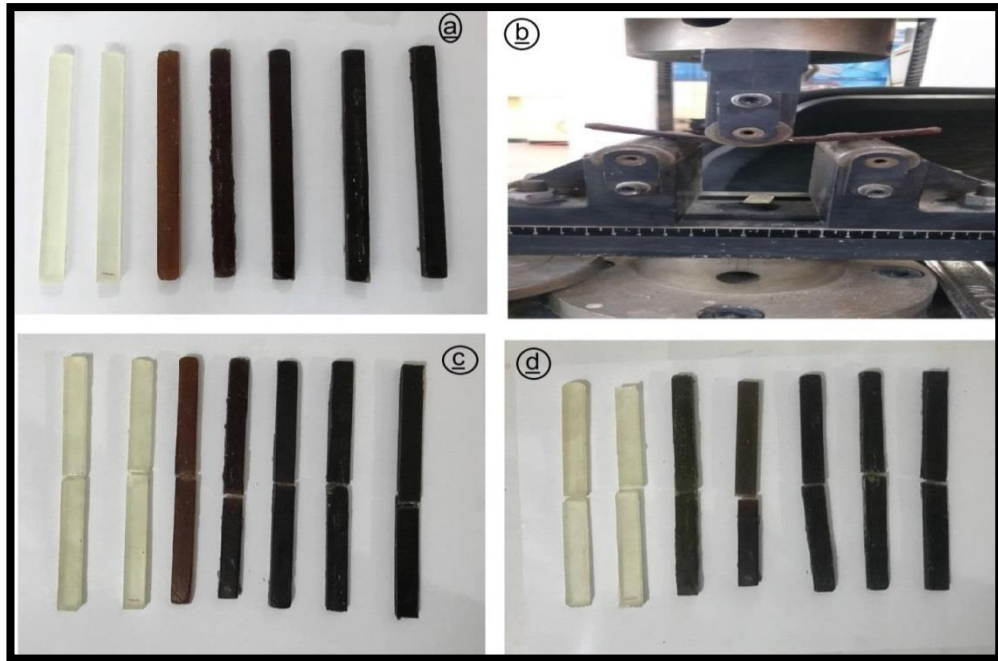


Figure 3.2. Show the Samples (a) Before a Test, (b) Flexural Test Machine (c) & (d) after a Test.

### 3.8. ANALYSIS BY MEANS OF FOURIER TRANSFORM INFRARED SPECTROMETRY

The Bruker Optics Company FTIR-D830 spectrometer is used for the FTIR analysis; it has been calibrated in line with ASTM E1252 (TENSOR-27). The sample was tested in air using a device that operates in the (4000-400  $\text{cm}^{-1}$ ) mid-infrared IR band. This test compared untreated CRT powder to treated powder that had been treated with silane. The samples were held in a specialised FTIR holder, which focused the beam through the sample and onto the detector. The x-axis of the output chart indicates the wave number, while the y-axis indicates the absorbance or intensity percentage. Figure (3.3) demonstrates the FTIR analyser [45].



Figure 3.3. The FTIR analyser.

### **3.9. IMPACT TEST**

According to the regulations, we performed a battery of impact tests using an Izod Impact Test equipment (XJU series pendulum Izod/Charpy impact testing system) (ISO-180). The model was able to catch the cantilevered beam at one end and hold it plumb during the Izod impact test. An impact from a 5.5J pendulum travelling at an impact velocity of 3.5 m/s was enough to shatter it. The amount of kinetic energy necessary to initiate a fracture and propagate it until the sample is fractured. As a result of the impact test, the samples did not break.

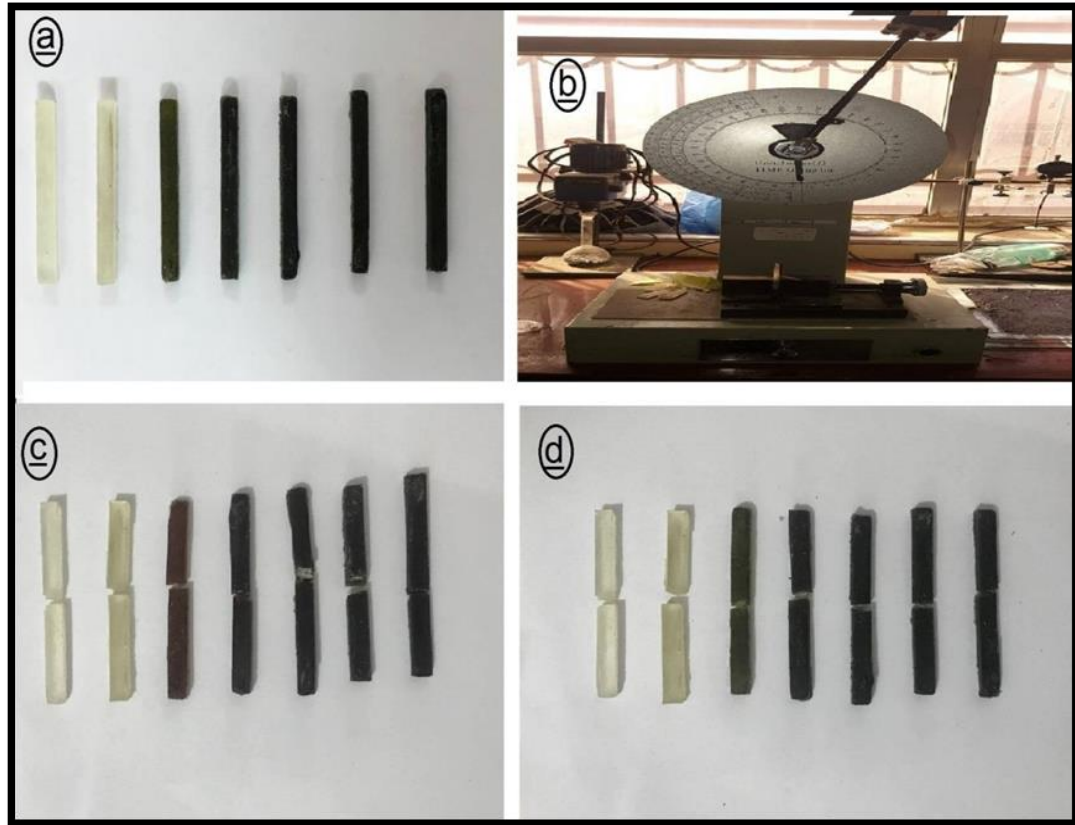


Figure 3.4. (a) Samples Before a Test, (b) The Izod impact test device, (c) & (d) Samples after a Test.

### 3.10. HARDNESS TEST

The hardness tests were conducted in accordance with (ASTM D2240), Specifically, type (Shore D), with a load applied and a measuring time that equates to (15 sec). The sample needs to be at least 3 mm thick and have a perfectly smooth, even surface. If the distance from the edge is more than 12 mm, the sample thickness is more than 12 mm, or the model diameter is more than 12 mm, the hardness value is particularly vulnerable to mechanical vibrations and must be protected. Sample utilised in this study was a circle with a thickness of 5 mm and a diameter of 50 mm, as shown in Figure 3.5. All samples were examined five times at various places before an average was determined.



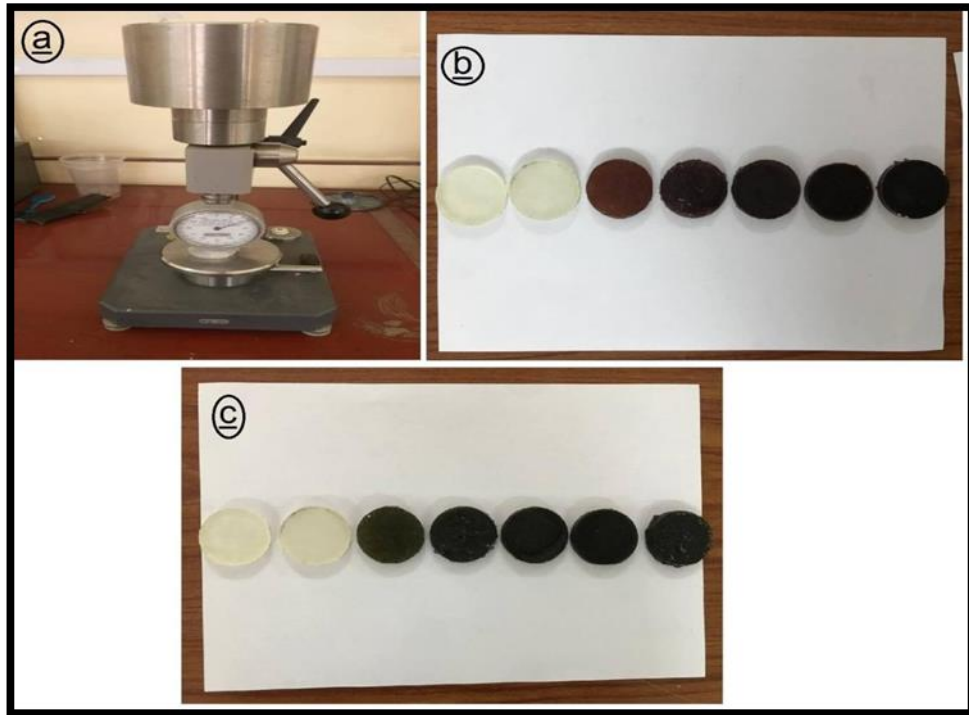


Figure 3.5. The (a) Shore D durometer and (b) and (c) samples of various harnesses.

### 3.11. THERMAL PROPERTIES TEST

By using the Lee disk method, the thermal conductivity was evaluated. The two metal discs (often brass), a sample, and two thermometers needed for Lee's Disc method of temperature gradient measurement. The temperature of the sample and the brass base are both recorded by thermometer T2. In this method we can see the difference between the temperatures in the small disk. Figure [3.6] shows prepared epoxy and composite specimens, figure 3.7 shows Lee's disc device

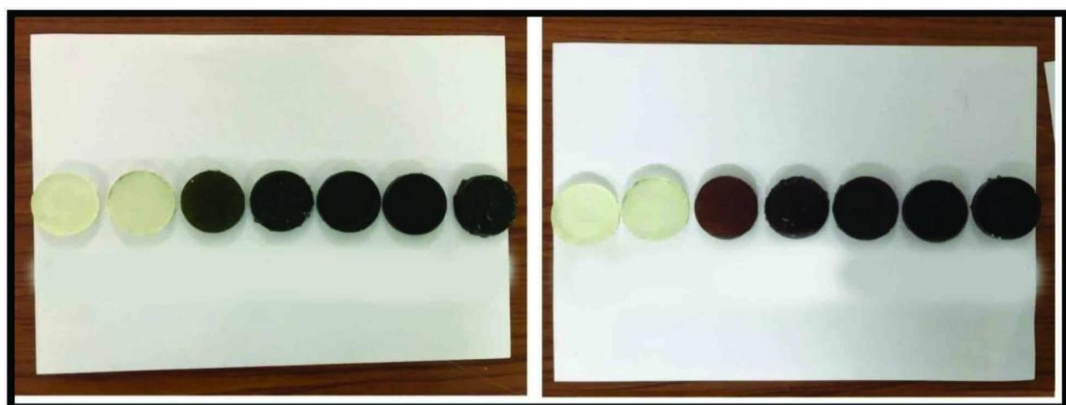


Figure 3.6. Prepared epoxy and composite specimens



Figure 3.7. Lee's disc method.

### 3.12. ACOUSTIC INSULATING TEST

According to the American standard (ASTM - E336) the sound insulation test was carried out through a locally made device, which is a wooden box as shown in the figure (3. 9). As illustrated in Figure (3.10), the test begins when a (25250.6) cm<sup>3</sup> sample is positioned in the center of the frame box and sound waves are generated by the wave generator and sent to the loudspeaker, which is in contact with the wooden box. When the box was sealed, waves were generated at a frequency 15 Hz and then sent to the receiver of the sound or wave. [46].

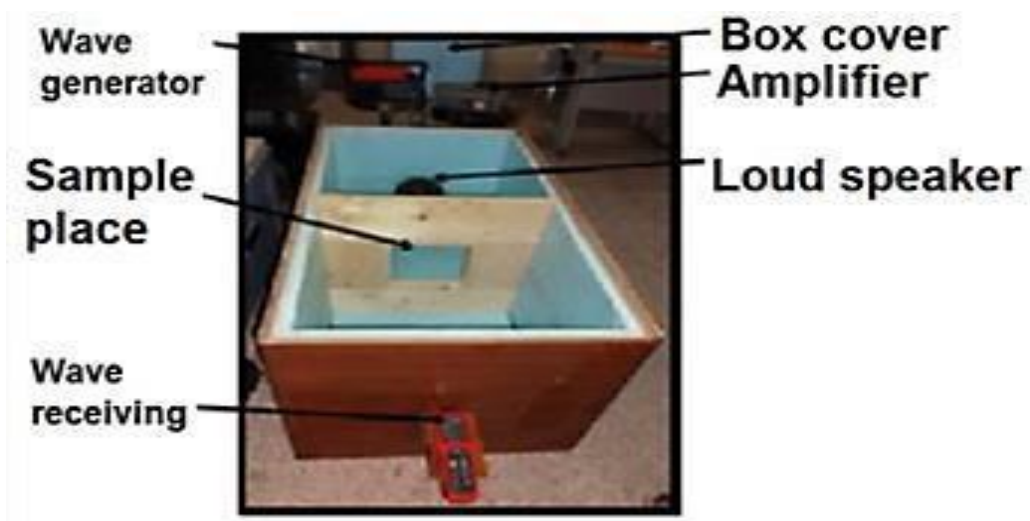


Figure 3.8. Acoustic Isolation Gear

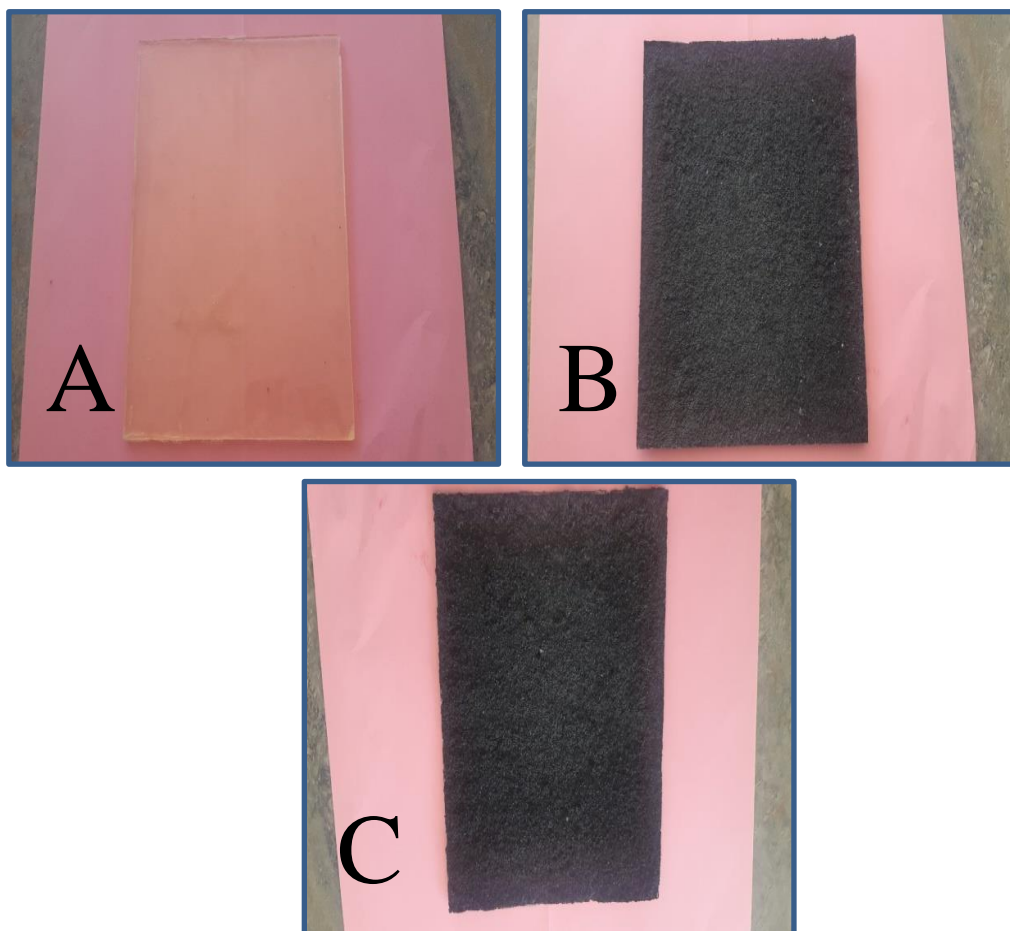


Figure 3.9. A- Epoxy pure. B- Epoxy +CRT. C- Epoxy + Trated CRT.

## **PART 4**

### **RESULTS & DISCUSSION**

#### **4.1. INTRODUCTIONS**

This chapter will show and discussion the results of experimental part of this work. The chapter will divided the results into two parts; first one is related to evaluate and compare performed mechanical tests that done to explore the effects of CRT surface treatment by different kinds of silane coupling agents before using to preparing 10 wt. % CRT/epoxy composites materials, whereas the second part will demonstration and evaluate the mechanical, thermal properties addition to acoustic insulation epoxy/CRT composite materials with variant weight fraction (10%, 20%, 30%, 40%) of CRT in epoxy matrix.

#### **4.2. THE EFFECTS OF CRT SURFACE TREATMENT**

As previously mentioned, three kinds of silane have been used to enhance bonding force between CRT and epoxy matrix. The mechanical tests that implemented to evaluate the development in mechanical properties are: hardness, tensile test, and flexural bending test.

#### 4.2.1. Hardness Tests Results

The effect of silane surface treatment into the hardness of pure epoxy and epoxy /10 wt. % treated CRT composite are shown figure (4-1).

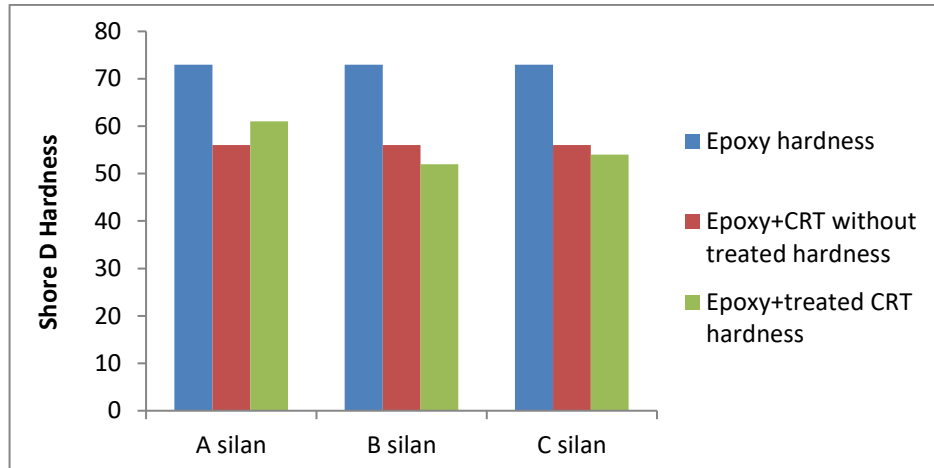


Figure 4.1. Shore D hardness of epoxy, treated CRT composite, and non-treated CRT composite.

It can be notice that hardness value of epoxy, blue columns, is greater than that of composite as low hardness value of CRT comparing with epoxy matrix, and weak bonding between these phases. Also, the results show in general that treated CRT by silane coupling agents could slightly lowering hardness as development of composite structure as result of promoting bonding between matrix and CRT filler.

#### 4.2.2. FTIR Inspection

FTIR analysis has been utilised for both treated and untreated CRT powder to investigate the adhesion of silane coupling agents to the particle surfaces In figure (4-2), we see the transmittance bands of treated and untreated rubber particles over (400- 3000)  $\text{cm}^{-1}$  wavenumber. Important FTIR transmittance peaks in treated CRT powder include Si-O-C stretching vibration at 1080  $\text{cm}^{-1}$  and conjugated C=C at 1600  $\text{cm}^{-1}$  in addition to those seen in non-treated CRT powder, which include C-H asym./sym. stretch of aliphatic groups at 2915, 2847  $\text{cm}^{-1}$ , C-C skeletal in plane vibrations at 1539  $\text{cm}^{-1}$ , and that agree with reported FTIR in [17]. The presence of organic siloxane or silicone (Si-O-C) in the 1180-1110  $\text{cm}^{-1}$  range

indicates that the three types of silanes coupling agent (A, B, & C) have successfully attached to the CRT powder via the performed treatment, whereas the absence of this regain in the untreated CRT, chart (A), is almost devoid of significant peaks [13, 14].

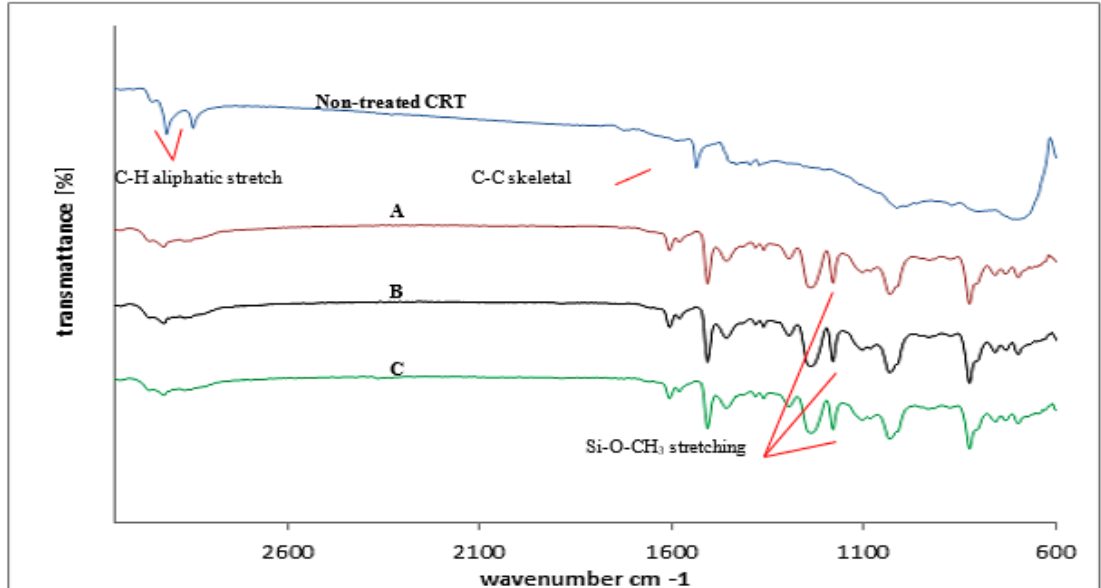


Figure 4.2. FTIR transmittance of untreated CRT, CRT treated with (A and B), and (C) silane coupling agents.

### 4.2.3. Impact Tests

The effect of silane surface treatment into impact tests results of pure epoxy resin and epoxy / 10 wt. % treated and non-treated CRT composite are shown figure (4-3).

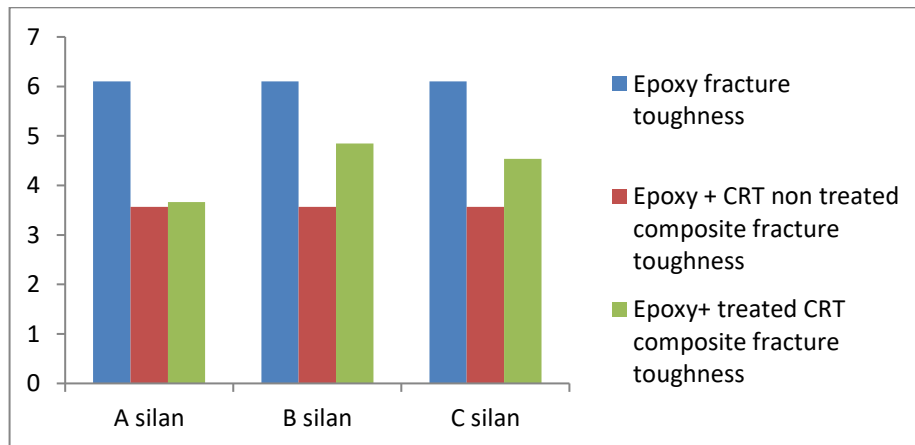


Figure 4.3. Fracture toughness of epoxy / treated and non-treated CRT composite.

The results showed that the impact strength of epoxy resin, blue columns, is higher than that of epoxy / CRT composite materials. CRT surface treated by silane coupling agents could improve fracture toughness of composites, green columns, comparing with those did not treat, red columns, the explanation of this development is related to improvement in bonding strength between matrix and CRT particles, so better participate of higher ductility (CRT) in absorbing applied load during impact test. The highest obtained fracture toughness is for epoxy / B-silane CRT composite.

#### 4.2.4. Tensile Tests

The stress-strain behaviour of epoxy resin and epoxy / 10 wt. % treated CRT composite is shown figure (4-4).

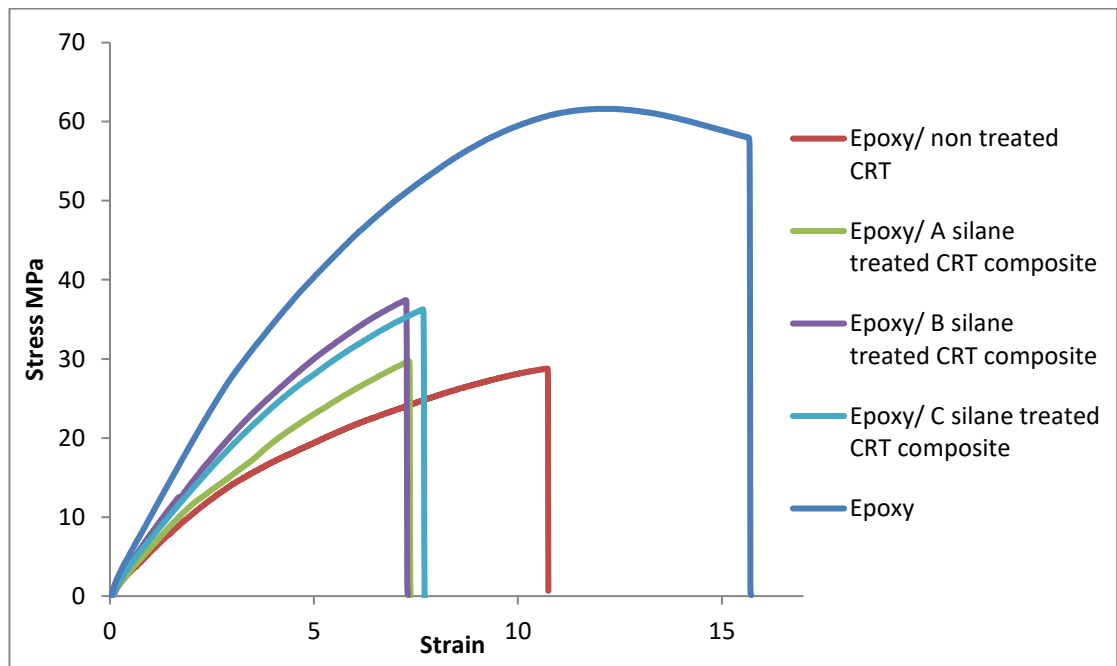


Figure 4.4. Stress-strain behaviour of epoxy resin and epoxy /10 wt. CRT composites.

The results showed that the modules of elasticity, toughness and maximum tensile strength of epoxy resin is much higher than that of composites materials, this related to low strength of rubber, addition to weak bonding between epoxy and CRT particles where CRT particles acts as defects inside structure causing declining the

mechanical properties. Tensile strength of epoxy resin and epoxy / 10 wt. % treated CRT composite shown in figure (4-5).

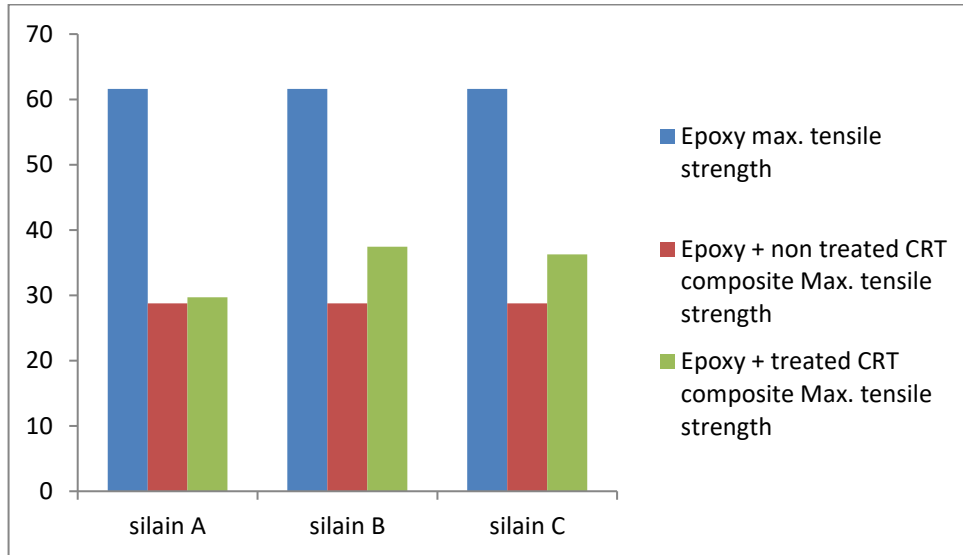


Figure 4.5. Maximum tensile strength of epoxy and treated, non-treated CRT composite.

In general, surface treating by silane agents, green columns, show higher strength comparing with epoxy / non-treated CRT composite, this related to enhancing of bonding between phases as mention before. However composite contained CRT treated by B-type silane coupling agent shows again highest strength comparing with other CRT agents treated composite.

#### **4.3. MECHANICAL, THERMAL AND ACOUSTIC INSULATION PROPERTIES OF EPOXY/ SURFACE TREATED CRT COMPOSITES**

Depending to previous tests results, surface treated CRT silane B / epoxy composite show the best mechanical results, so B-type silane coupling agents depends to treated CRT for next step of work. Variant weight fraction of surface treated CRT, (10, 20, 30, 40) wt. %, have been used to evaluate the development of composite properties with increasing treated CRT weight percentage, the tests results are shown below.



### 4.3.1. Epoxy/ B-Silane Treated CRT Composite Hardness Tests

The effect of variant treated CRT weight fractions into hardness of composite is shown figure (4-6).

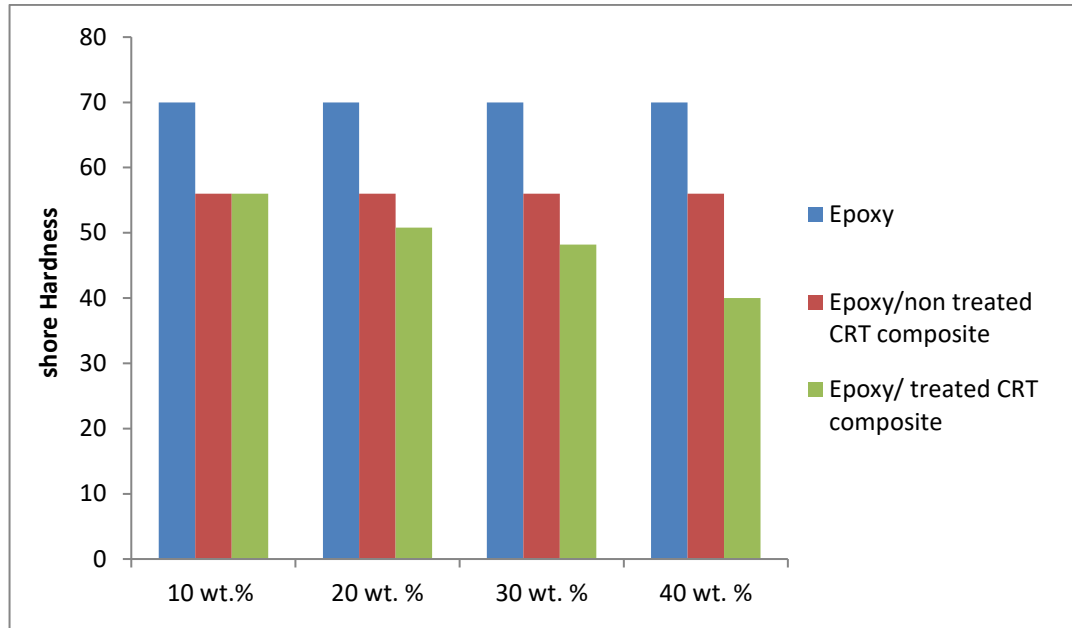


Figure 4.6. Development of composite hardness with treated CRT weight fraction.

In general, the results show that shore hardness of epoxy / treated CRT, green columns, are less than those corresponding of epoxy / non-treated CRT composite, red columns. an increase in CRT weight fraction percentage of rubber leads to a decrease in the hardness. The reason is due to the weakness of the hardness of the rubber compared with Epoxy.

### 4.3.2. Epoxy/ B-Silane Treated CRT Composite Tensile Strength

The stress-strain behaviour of epoxy and different weight fractions epoxy/treated CRT composite are shown in figure (4-7).

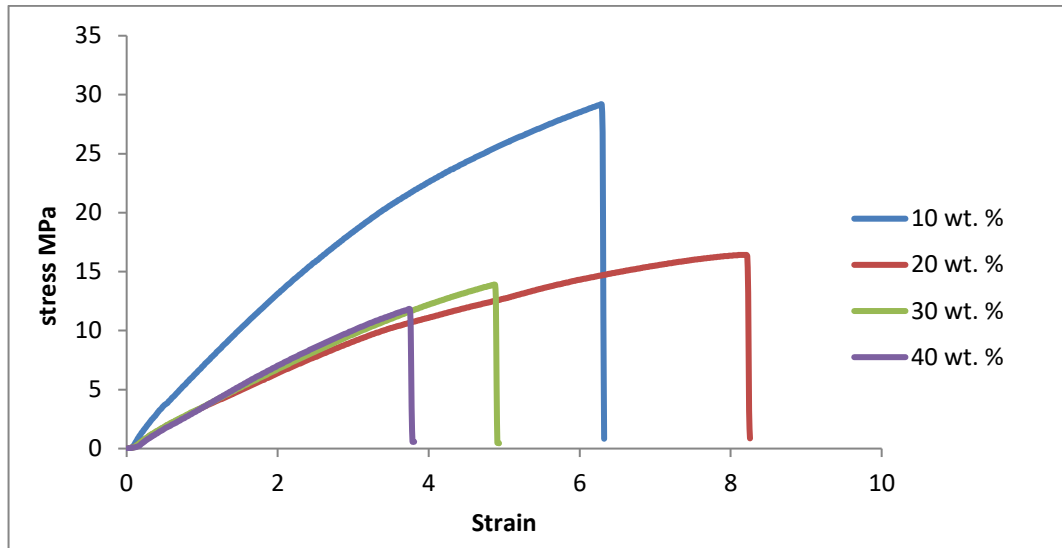


Figure 4.7. Stress-strain behaviour of epoxy and epoxy / different CRT weight fractions composite.

It can be noticed increasing the CRT weight fractions lead to decline toughness, elongation at fracture percentage and maximum tensile strength, as shown in figure (4-8).

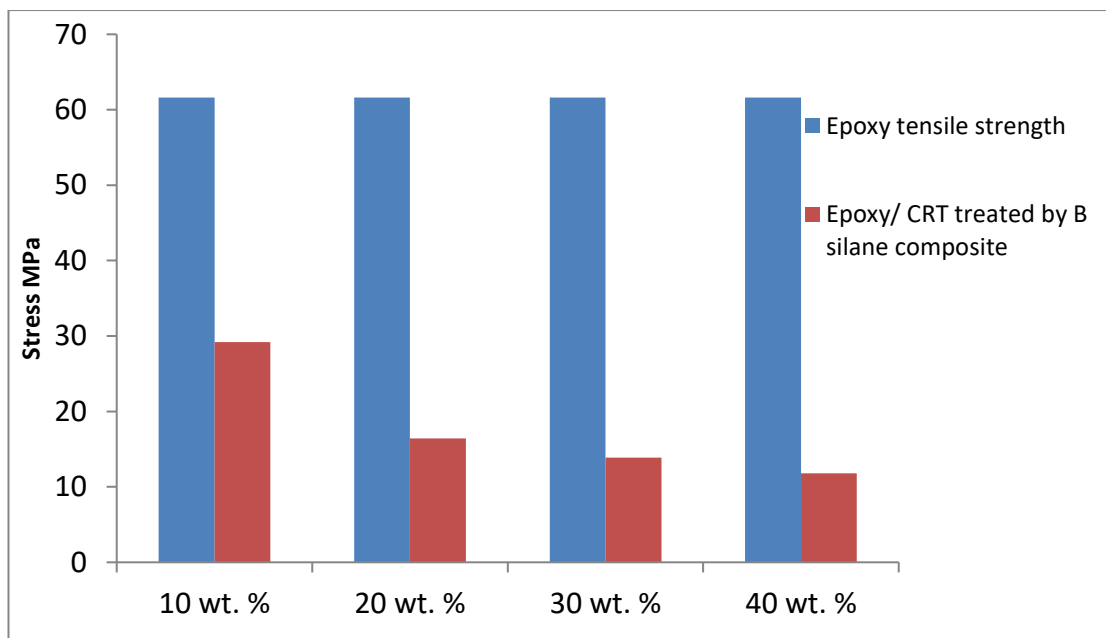


Figure 4.8. Maximum tensile strength of epoxy and variant treated CRT weight fraction.

This attitude may be related to low strength nature of rubber comparing with epoxy so increasing CRT weight fraction leading to deterioration strength according to rule of mixture.

#### 4.3.3. Epoxy/ B-Silane Treated CRT Composite Flexural Strength Tests

The results of pure epoxy and epoxy / CRT composite are shown figure (4-9).

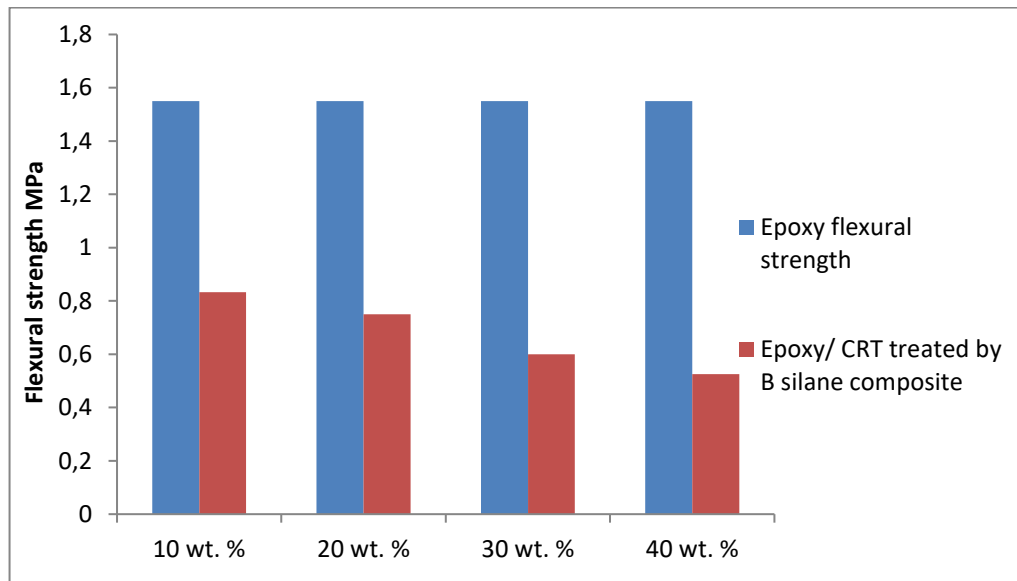


Figure 4.9. Impact strength of epoxy and variant treated CRT weight fraction.

According to chart flexural strength decrease with increasing CRT weight fractions, these behaviour agree with tensile strength tests, and can be justified by same explanations.

#### 4.3.4. Epoxy/ B-Silane Treated CRT Composite Impact Tests

The results of pure epoxy and epoxy / CRT composite are shown figure (4-10). The best results were obtained with 10% of the rubber. The increase in rubble reduces the mechanical properties of the composite material.

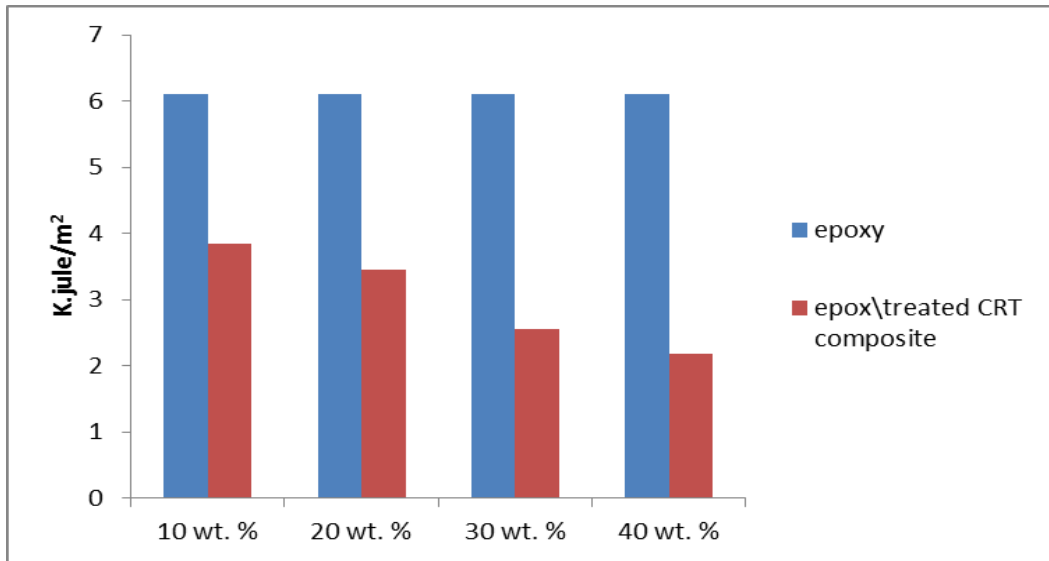


Figure 4.10. Impact strength of epoxy and variant treated CRT weight fraction.

#### 4.3.5. Epoxy/ Treated CRT Thermal Conductivity

The effect of CRT weight fraction into epoxy / treated CRT composite thermal conductivity are shown figure (4-11).

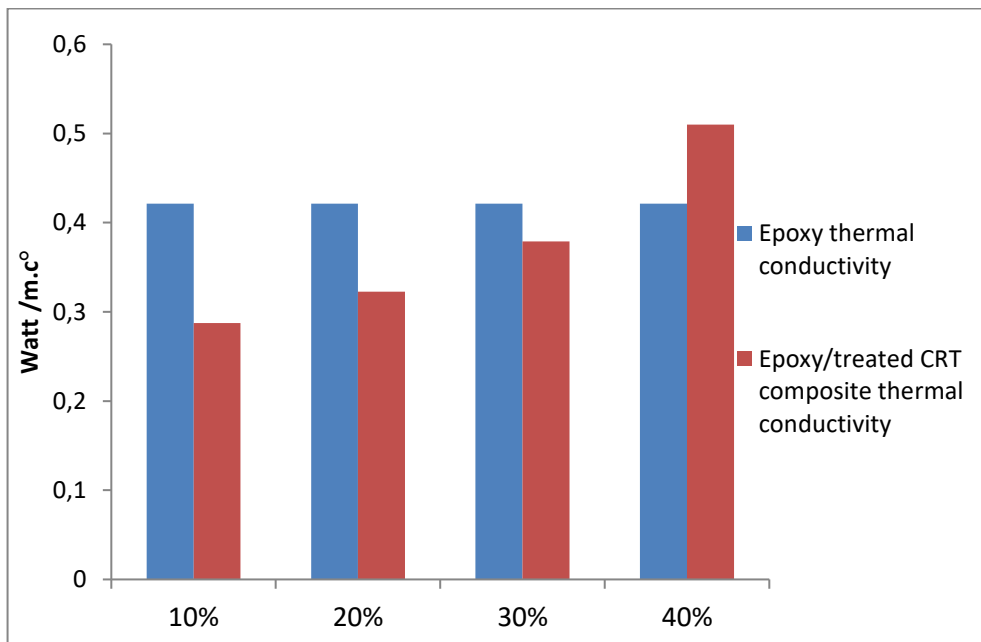


Figure 4.11. Thermal conductivity of epoxy and epoxy/different weight fractions CRT composites.

Thermal conductivity in polymeric materials depend into lattice vibration (phonons). The thermal energy associated with phonons or lattice waves is transported in the direction of their motion, where the net movement of phonons from high- to low temperature. The present of CRT within matrix hander phonons movement especially with weak connection between two phases. This explain the low thermal conductivity of composite comparing with pure epoxy resin, on other hand CRT thermal conductivity is higher than that of epoxy so increasing weight fractions improve thermal conductivity of composite according to rule of mixture.

#### 4.3.6. Epoxy/ Treated CRT Composite Acoustic Insulation

The results of acoustic insulation of epoxy, epoxy / 40 wt. non-treated CRT, and epoxy / 40 wt. treated composite are shown figure (4-12).

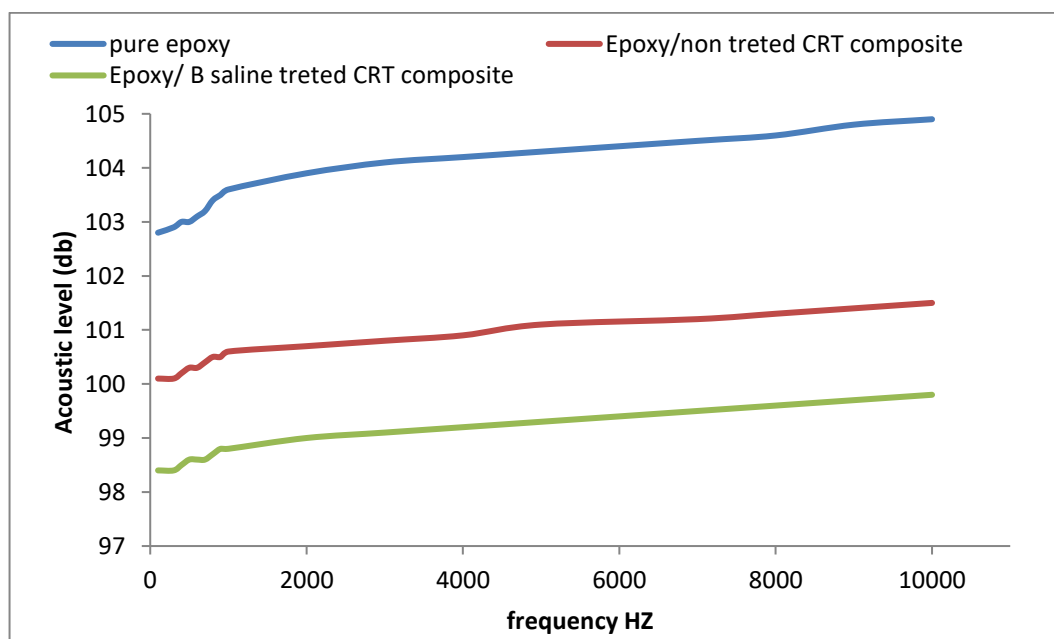


Figure 4.12. Epoxy/ treated CRT composite acoustic insulation.

According to figure epoxy has highest acoustic level, blue line, over wide range of frequency (0-10000) Hz, followed by epoxy/non-treated CRT composite, whereas epoxy /treated CRT show the lower acoustic level.

## **PART 5**

### **CONCLUSIONS**

The mechanical properties of the CRT/epoxy composite are significantly enhanced after treatment with various silane coupling agents. When compared to similarly treated composites made without CRT, those made with CRT and 3-chloropropyl triethoxysilane show the greatest improvement in mechanical qualities.

When compared to CRT or epoxy that hasn't been treated, a coupling agent with 3-chloropropyl triethoxysilane can greatly improve the mechanical properties of the composite. The modulus of elasticity, tensile strength, and fracture toughness can all be raised by 56%, 30%, and 36%, respectively, while the elongation at the point of fracture can be lowered.

The effects of increasing CRT in to thermal, acoustic insulation, and mechanical properties of CRT/epoxy composite are also tests by preparing (10,20,30,40) wt. % CRT/epoxy composites. The results show that tensile strength, modulus of elasticity and impact strength generally have decreased with increasing CRT weight percentage meanwhile acoustic insulation got improvement.

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## **RESUME**

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