STUDY OF THE MECHANICAL PROPERTIES OF WALNUT/PISTACHIO SHELLS BASED EPOXY COMPOSITES

A DISSERTATION

SUBMITTED IN THE PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF THE DEGREE OF

MASTER OF TECHNOLOGY

IN

POLYMER TECHNOLOGY

Submitted by:

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2K17/PTE/07

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CANDIDATE'S DECLARATION

I VAIBHAV PATHAK, 2K17/PTE/07 student of M.Tech Polymer Technology hereby declare that the project dissertation titled "**STUDY OF THE MECHANICAL PROPERTIES OF WALNUT/ PISTACHIO SHELLS BASED EPOXY COMPOSITES"** submitted by me to the Department of Applied Chemistry, Delhi Technological University, Delhi in the partial fulfillment of the requirement for the award of the degree of Master of Technology, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any degree, Diploma Associate-ship, Fellowship or other similar title or recognition.

Place: Delhi **VAIBHAV PATHAK**

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CERTIFICATE

I hereby certify that the project dissertation titled **"STUDY OF MECHANICAL PROPERTIES OF WALNUT/PISTACHIO SHELLS BASED EPOXY COMPOSITES"** submitted by VAIBHAV PATHAK

(Roll No.: 2K17/PTE/07), Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Technology, is a record of the project work carried out by him under my supervision. To the best of my knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

Place: Delhi **Dr Poonam**

Date: **SUPERVISOR**

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VAIBHAV PATHAK

ABSTRACT

In this Project work, WALNUT & PISTACHIO shells were used as a reinforcement to increase the mechanical properties of Bisphenol-A based Epoxy resin. First sample of neat Epoxy, was synthesized and was then reinforced using pistachio/walnut shell based filler along with transition metal salt (Cu). Finally reinforced epoxy composites were analyzed on the basis of their mechanical properties. Mechanical test showed the excellent improvements in mechanical properties of the composites prepared using pistachio/walnut based filler and further enhancement in mechanical properties was observed upon incorporation of transition metal salt. In this work, synthesize composite samples have been analyzed using Fourier-transform infrared spectroscopy, Thermo-gravimetric analysis, tension test and compression test. Results shows the significant improvement in tensile, compression and load bearing capacity of walnut/pistachio shell reinforced Epoxy composite.

In today´s world, composites materials are replacing the traditional material attributed to their high stiffness, low thermal expansion and high strength to weight ratio. However the use of natural filler as a reinforcement in composite can result in enhancement of properties such as reduced weight, lesser density and lower cost.

The synthesized composites samples were tested as per ASTM standards to evaluate mechanical properties i.e. tensile strength and impact strength. The result of tests shows that walnut and pistachio based hybrid composite was far better mechanical properties than pure walnut/pistachio filler reinforced composite under mechanical loads. However it was observed that the incorporation of transition metal in walnut shell and pistachio shell based composites can further enhance their mechanical properties.

LIST OF ABBREVIATION

Symbol	Notation
CFRP	Carbon fiber reinforced polymer
MMC	Metal matrix composite
CMC	Ceramic matrix composite
PMC	Polymer matrix composite
SMC	Sheet molding compound
GMT	Glass fiber mat reinforced thermoplastics
RTM	Resin transfer molding
PAN	Polyacrylonitrile
TETA	Triethylenetetramine
MDI	Methylene diphenyl diisocynate
PEG	Polyethylene glycol
DMF	Dimethyl formamide
FTIR	Fourier transform infrared spectroscopy
TGA	Thermal gravimetric analysis
UTM	Universal testing machine
ASTM	American society for testing and materials

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CHAPTER 1. INTRODUCTION

In recent years, with increased environmental awareness, ecosystems and new legislations, bio wastes are getting primary importance for reinforcement in polymeric matrices. Several researchers have reported the effective utilization of bio wastes in different polymer matrices. In this way, both the polymer matrix gets improved performance characteristics while effective utilization of bio waste is also accomplished. Wastes from agro industry used for making different polymer matrices include almond shell, apricot shell, wheat straws, cotton seed hulls, walnut shells, wood flour and corn. The compositions of these materials include mostly cellulose, hemicellulose, lignin and other extractives. They are used in variety of application including marine equipment's, sporting goods, defense, electronic industry packaging, electronics, automotive, aerospace, and construction industries, because of superior mechanical properties. They are light weighted and have superior mechanical properties over monolithic materials which make them suited for vast applications. Composite materials from natural materials and polymeric matrices have been extensively investigated in composite research.

Keeping this in view the current work focus on the development of a polymer matrix composite with epoxy resin and natural filler derived from bio-waste (derived from walnut/pistachio shells as reinforcement) to study its mechanical properties and performance.

The characteristic properties of natural fibers/fillers are mostly depending on their physical and chemical configuration, such as filler structure, percentage of cellulose content, angle of fibrils, cross section area and on the degree of polymerization. Major reason behind the use of natural fiber/filler in reinforcement is their obtainability in bulk quantities.

The only reason limiting the composite use in engineering practices or applications are the lack of reliable experimental methods for their evaluation of mechanical properties of reinforced composites, difficulties in the deformation processes, mathematical modeling and evaluation of the load-carrying ability of reinforced composites, and the requirements for advance and more economical manufacturing methods of processing. However determining mechanical parameters of composites in terms of characteristics of individual constituents gives wide scope for the design of materials with desired properties. This work aims to examine how the mechanical properties of walnut/pistachio shells fillers reinforced epoxy composites shows variation when filler ratio is varied in matrix. Mostly the binding polymer used is thermoset resin i.e epoxy, but sometimes other thermoset or thermoplastic polymers, like polyester, vinyl ester or nylon, can be used to prepare composites

USE OF WALNUT & PISTACHIO SHELLS AS A FILLER?

Biomass nutshell is a large yield crop residue which is generally discarded or incinerated. This not only pollutes the environment, but also affect a large amount of resources. In order to overcome this, many researchers have undertaken research and focusing on the use of biomass nut shells to produce activated carbon. Several researchers have reported the effective utilization of bio wastes in different polymer matrices. In this way, both; the polymer matrix gets improved performance characteristics while effective utilization of bio waste is also accomplished.

Wastes from agro industry used for making various polymer matrices include almond shell, apricot shell, wheat straws, cotton seed hull, walnut shells, wood flour and corn.

Composition of cellulose, hemicellulose, lignin in shells- (w %)

The content of cellulose in walnut shells is 36.475%, which is very desirable, but higher than other shells except pistachio shell. This indicates that the mechanical properties of composite prepared by walnut shells might be higher than that of most biomass nut shells. Lignin is an amorphous substance without fixed structure; however, it is three-dimensional and highly branched. Lignin generally surrounds microfibers and large fibres. The lignin content of walnut shells is 44.54%, and pistachio contains 16.74%. Lignin may be beneficial to improve compatibility with thermoplastic, and it has a certain flame-retardant property which is important for composites being used as building materials.

Sample	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Walnut shells	36.38 $\pm$ 0.05	27.85 $\pm$ 0.31	43.70 $\pm$ 0.57
Pistachio shells	43.08 $\pm$ 0.19	25.30 $\pm$ 0.46	16.33 $\pm$ 0.41

Epoxy Resin -

Epoxy resins are the widely used thermoset resin in polymer matrix composites. Epoxy resin is itself a family of thermoset plastic materials that do not give off reaction products while they cure and also has low cure shrinkage. They have desirable properties such as excellent adhesion, highly chemical and environmental resistant, good chemical properties, and high insulating property. The epoxy resins are obtained by reacting epichlorohydrin with bisphenol. Resin with varying properties can be obtained by varying proportions of the reactants, as the proportion of epichlorohydrin is reduced the molecular weight of the resin is increased. Curing agent/hardener polymerizes the process, resulting in the very dense cross-linked network of polymer.

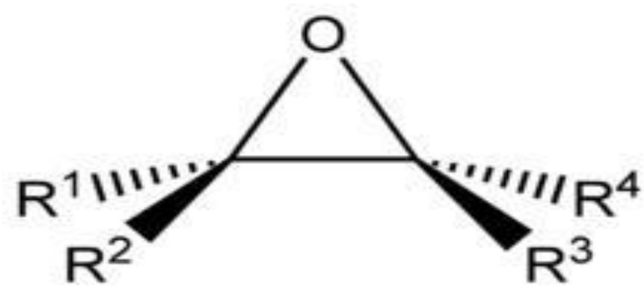


Fig: 3.1 Structure of epoxy resin

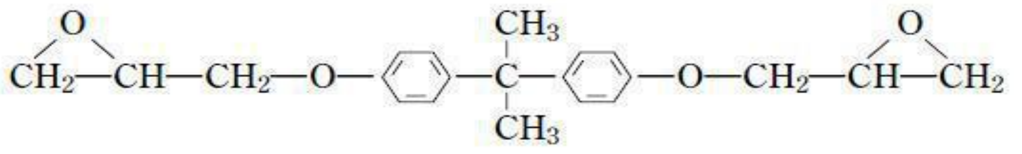
The word epoxies come from the molecules that have the epoxide ring. An epoxide ring is an oxirane structure that consists of three-member ring with one oxygen and two carbon atoms.

Epoxies consists of one or more than one epoxide ring cured by reaction with amines, acids, amides, alcohols, phenols, acid anhydrides or mercaptans. Epoxy resin is used widely in structural adhesives. The desired properties of epoxy are high strength and modulus, less volatile, excellent adhesion, low shrinkage, excellent chemical resistance, and easy processing. The major drawbacks of epoxy are brittleness and the degradation in presence of moisture, manufacturing techniques are autoclave molding, filament winding, press molding, vacuum bag molding, resin transfer molding, and pultrusion. Curing process has a range varying from room temperature to approximately 350°F (180°C). Generally curing ranges between 250°F and 350°F (120° and 180°C). Generally, properties like tensile modulus, glass transition temperature, thermal stability, chemical resistance are improved with increasing cross-linking, but brittleness is also increased.

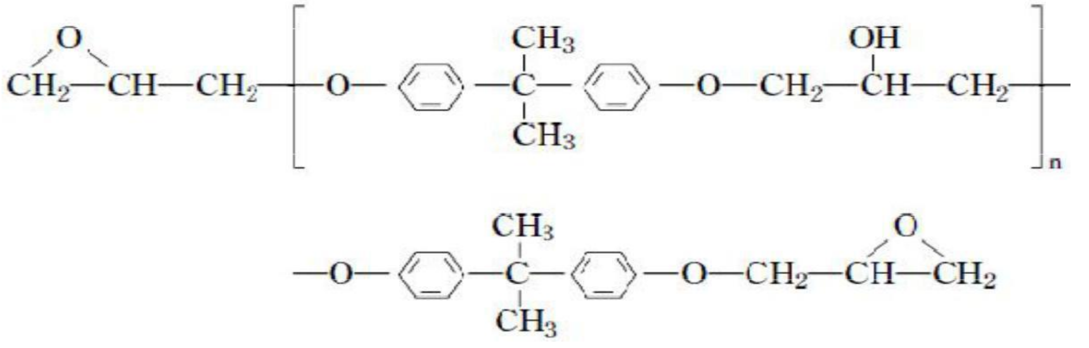
Properties of Epoxy

- Better Adhesive Properties.
- Superior Strength & Stiffness
- Improved Resistance to fatigue and micro-cracking.
- Highly Water resistant.
- Increased resistance to osmosis
- No volatile products & exothermic reaction.
- Due to polarity of aliphatic hydroxyl group and ether group promotes electromagnetic bonding.
- Extremely long pot life.
- Excellent chemical resistance
- High Strength
- Low Viscosity and low flow rates, which allow good wetting of fibers and prevent misalignment of fibers during processing.
- Low volatility during cure.
- Low shrink rates, which reduce the tendency of gaining large shear stresses of the bond between epoxy and its reinforcement.
- Available in more than 20 grades to meet specific property and processing requirements.

<u>Bisphenol A Based epoxy resin structure</u> -

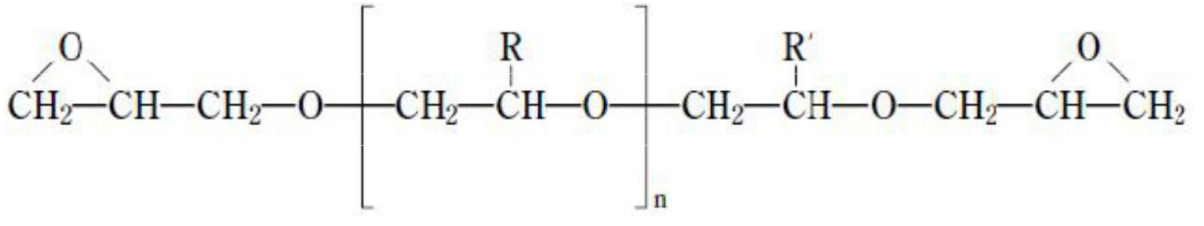


The higher molecular weight homologs are represented by the following theoretical structure:



Generic Bisphenol A Based Epoxy Resin Chemical Structure

With increasing molecular weight, another reactive site — the OH group is introduced. This group can react at higher temperatures with anhydrides, organic acids, amino resins, and phenolic resins, or with epoxide groups (when catalyzed) to give additional cross-linking



In high melting point solid resins, “n” may be as high as 18

Properties of epoxy resins:-

PROPERTY	VALUE	UNITS
Density	1.1-1.4	MgM^{-3}
Young's modulus	2-6	GNM^{-2}
Poisons ratio	0.38-0.4	ν
Tensile strength	35-100	Mpa
Compressive strength	100-200	Mpa
Elongation to break	1-6	%
Thermal conductivity	0.1	WM-1 OC
Coefficient of thermal expansion(α)	60	10^{-6} ^{o}C
Heat distortion temperature	50-300	OC
Shrinkage on curing	1-2	%

Epoxy Hardener (HY5052):

Hardener is used as solvent. Hardeners are substances that are added to polymers for aiding in curing of composites. Hardeners are used to enhance the physical properties of epoxy resins such as adhesion, impact strength and to alter the viscosity of the polymer matrix. It also improves the life, lower exotherm and reduces shrinkage. Hardener HY5052 is polyamine hardener Application of Epoxy Hardeners –

- Solvent free, High Coatings.
- Pipeline & tank coatings.
- Adhesives
- Surface tolerant primers for metallic substrate.
- Marine & industrial coatings.

PRODUCTION SHARE OF WALNUT & PISTACHIO WORLD WIDE-

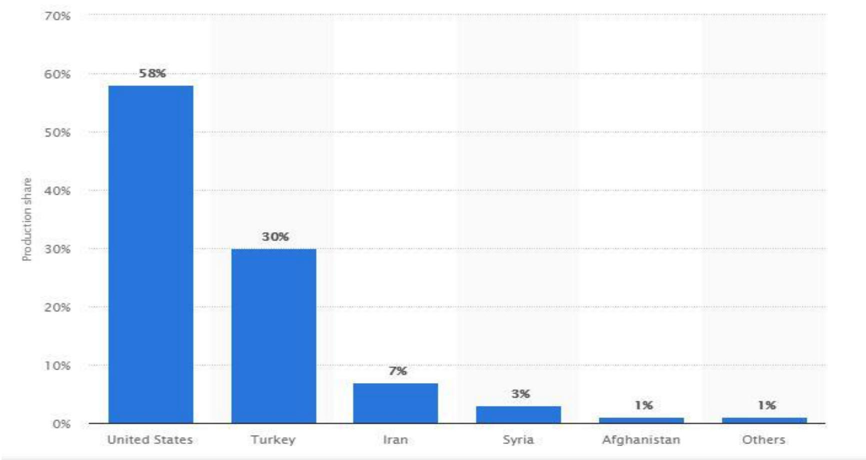


Fig.1 Worldwide production of pistachios

Other major walnut producing nations-

China and the US account for nearly three-quarters of the global walnut production. The European Union, Ukraine, Chile, Turkey, and Moldova are the other top regions/countries for walnut production.

(a)

(b)

Fig.2 Processing of Pistachio shells (A) and wallnut shells(B)

COMPOSITE

A composite is combination of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and is embedded in the other materials called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer.

According to type of matrix material they are classified as:

- Metal Matrix Composites (MMC)
- Ceramic Matrix Composites (CMC)
- Polymer Matrix Composites (PMC)

According to Reinforcement types of polymer composites are:

- Fiber reinforced polymer (FRP)
- Particle reinforced polymer (PRP)
- Structural reinforced polymer (SRP)

In this project, FRP is synthesize using epoxy resin as matrix and walnut/pistachio shell as a filler and mechanical properties are studied.

1.2 OBJECTIVE

To measure the feasibility of the epoxy and Walnut/Pistachio filler based composite on basis of cost, strength, energy and effect of chemicals on environment, which are used in treatment of the fiber.

To use Walnut/Pistachio based treated filler as reinforcement in epoxy material, which is a waste generated from agricultural. It is a challenge to make a material from waste and improve the mechanical properties and reduce the cost.

Study the effects of chemically treated Walnut/pistachio shells reinforced with epoxy resin matrix and study the effect of incorporation of transition metal ion on mechanical properties of composites.

- To develop a series of hybrid composite by varying weight percentage and varying grain sizes of filler in Epoxy matrix.

Mechanical tests performed for samples -

1. Tensile Strength
2. Tensile Modulus of Elasticity
3. Flexural Strength
4. Impact Strength
5. Hardness

Chapter 2. Literature Review

This chapter provides the general review of the relevant literature available on the mechanical properties of natural filler reinforced epoxy based composite. The purpose of this study was to explore the mechanical properties of natural filler reinforced epoxy based composite. This also present a literature on factor that influence the mechanical properties of natural filler reinforced epoxy based composite .Therefore need to review all relevant literature concerning the concept of the mechanical properties of Epoxy based natural filler polymer composite and application in the area of epoxy based natural filler polymer composite it founded a strong basis for the development of research frame work. Various Research papers were studied so that we can know what development is take place in the epoxy resin based polymer composite.

Literature survey had been done & a number of research papers had been studied and the research papers related to our study are tabulated below with their respective abstract, results & Conclusions:

<table>
<tr><th>Title Of Paper</th><th colspan="2">Study on Mechanical And tribo-performance of rice-husk filled glass-epoxy hybrid composites</th></tr>
<tr><th>Name Of Author</th><th colspan="2">Arun Kumar Rout and Alok Satapathy</th></tr>
<tr><th>Abstract</th><th>Result</th><th>Conclusion</th></tr>
<tr><td>A no. of composites were fabricated by using glass fiber and rice husk filler in the epoxy matrix.
The weight% of rice husk was varied as 5,10 and 15 wt%</td><td>Tensile strength of 15 wt% rice husk is less as compared to 10 and 5 wt%.
Tensile Modulus of 15 wt% rice husk is more as compared to 10 and 5 wt%.</td><td>Increasing Rice husk content decreases tensile strength but increases Tensile Modulus.</td></tr>
</table>

Title Of Paper	Mechanical Properties of natural fiber reinforced polymer composites	
Name Of Author	AS Singha And Vijay Kumar Thakur	
Abstract	**Result**	**Conclusion**
A no. of composites were fabricated using hibiscus sabdariffa as fiber and filler in The urea formaldehyde matrix. Hibiscus sabdariffa was used both as fiber and particulate filler Alternately.	Tensile strength of Particulate Filler Reinforcement is about 20 N/mm^2 more than the Fiber Reinforcement of Hibiscus sabdariffa.	Particle Reinforcement of Hibiscus Sabdariffa in Urea Formaldehyde was found to be more effective as compared to fiber reinforcement.

Title Of Paper	Effect Of Alkali Treated Walnut Shell (JUGLANSREGIA) on High Performance Thermosets.	
Name Of Author	A.H SHAH And S. WANG	
Abstract	**Result**	**Conclusion**
A comparative analysis of high performance thermosets based on epoxy and with reinforcement with alkali treated walnut shells.	Alkali treatment of walnut shells improves the thermal stability and increases crystallinity in composites.	Composite system can be designed based on treated walnut shells with improved thermal and thermochemical behavior.

Title Of Paper	Environment Friendly Composite Materials: Bio composites and Green Composites	
Name Of Author	B.C Mitra	
Abstract	**Result**	**Conclusion**
Preparation of bio-composite by suitable biofiber surface treatment and make it more suitable for composite application.	Tensile strength is increased by 20% after removal of amorphous lignin and hemicellulose from it.	Bio composites prepared by coconut shells and walnut shells shows excellent impact strength.

Title Of Paper	Study on Effects of the Addition of Pistachio Shell Particles on the Properties of Polyurethane Matrix Composite.	
Name Of Author	Ismail Ibrahim Marhoon	
Abstract	**Result**	**Conclusion**
A no. of composites were fabricated by using pistachio shell filler particles in the polyurethane matrix. The weight% of pistachio shell filler is varied as 5,10 and 12.5 wt%.	Tensile strength of 12.5 wt% pistachio filler composite is less as compared to 10 and 5 wt%. Tensile Modulus of 12.5 wt% pistachio filler composite is more as compared to 10 and 5 wt%.	Compression strength and impact strength increased as the weight fraction of pistachio shell particles increased to 7 and12.5 wt%., respectively the properties of Prepared composite decreased. Hardness increased as the weight fraction of pistachio shell particles increased and reached the maximum value at 12.5 wt%.

Title Of Paper	Study of Almond Shell Characteristics	
Name Of Author	Xuemin Li And Yinan Liu	
Abstract	**Result**	**Conclusion**
In this paper anatomical & chemical characteristics of almond shells are investigated in order to contribute to better utilization of these shells. The micromorphology, surface elements, thermal stability, crystallization, Chemical composition, and relative properties of almond shells are analyzed.	Cellulose content of almond shell is much more as compared to other shells so percentage crystallinity is more as compared to walnut and pistachio shells.	The thermal stability of walnut is superior to that of almond shells, but compared with other nut shells, the thermal stability of almond shells is better.

2.2 GAP IN LITERATURE

Literature survey showed that most of the study in this field is focused on the alkaline treatment of the walnut and pistachio shells in order to remove the amorphous lignin and hemicellulose from it so that reinforcement fibers only contain crystalline cellulose material. Which leads to higher adhesion to the matrix and increases its toughness and impact strength, hence mechanical properties. Further the combination of walnut shells filler is used as combination with metal salt.

Composite material is used as commodity product, not as the engineering product and does not require specific mechanical properties. Chemical treatment may not require at all. In this study, the mechanical properties and characterization are analyzed and composites are equated on the basis of different composition.

2.3 World market of composites-

The global high performance composites market size was valued at USD 40.50 billion in 2017-2018. Composites are a combination of two distinct components namely, fiber and resin. The new product, which exhibits completely different properties from fiber and resin, is characterized by high strength & stiffness and low weight. These properties have increased the product demand in a variety of applications including aerospace, defense, and automobile.

Application of composites in various industry are as follows-

(1) **The automobile industry**- Composite materials can remarkably reduce fuel consumption, and are anti-corrosion and shows high damping, so their large application of composite materials in the automobile industry. Polymer composite materials are also used in automobile components like glass fiber unsaturated polyester. In the past, they were produced by hand lay-up and spray-up molding, but in recent years, sheet molding compound (SMC), glass fiber mat reinforced thermoplastics (GMT) compression molding and resin transfer molding (RTM) are also used. The method of making SMC is by chopped glass fiber roving or glass fiber mat is impregnated using the mixture like paste resin, of unsaturated resin system, fillers and other additives, and then wrapped with polyethylene or polypropylene film on both the sides of itself to have the sheet type compound. SMCs are widely used because of continuous production, easy application and low cost.

In addition to glass fiber reinforced composite materials, the application of advanced composite materials in the automobile industry is also there. Due to high cost limits the use of advanced composite materials, but the good news is that the present cost of carbon fiber has dropped close to the price that automobile industry can accept.

2) The shipping industry- From a long time composite material are used in fishing boats, tugs, yachts, boats and minesweepers. The composite materials are lightweight and can bear high external pressure, development of composite materials submersibles is in progress, which will further enhance the application of composite materials in submarines.

3) The construction industry- In the construction industries, composite materials are used in variety of lightweight structural houses, decorative materials, sanitary ware and cooling towers, storage tanks and many more, besides, in present year, they are now being used in the application of the reinforced in concrete and the bridge. Composite materials are used as reinforcement in concrete instead of steel to make new type of concrete, which have attractive prospects, because it can increase the earthquake resistance and diamagnetism of building structure. The glass fiber, carbon fiber and aramid fiber or hybrid fibers for two-dimensional or three-dimensional woven or braiding are also used as reinforcement material. Now a day, such types of concrete has been used in the foundation of bridges and buildings, such as magnetic observatories, high-frequency electrical room and also many other applications, that extends service life of buildings and always improves performance. The application of composite materials in the construction of houses, bridges, tunnels, culverts, subways and also in related structural applications of concrete and also in various infrastructure projects, also in recent years it has become the fastest-growing civil and one of the most promising areas, and it has become keen area of the research and development now a day.

CHAPTER 3. EXPERIMENTAL

3.1 **Materials**

3.1.1 Raw materials/Chemicals used -

- Walnut Shell/Pistachio shell
- Epoxy resin Araldite LY 5052
- Mixture of polyamine hardener 5052
- Standard silicone rubber for mold preparation
- NaOH for treating fillers.
- Wax for mold preparation.

1. Walnut Shell/Pistachio shell based filler –

(a) (b) (c)

Fig.3 CHEMICALLY TREATED FILLERS –

(a) Walnut filler (powder form)
(b) Pistachio filler (coarse size)
(c) Pistachio filler (finer size)

Crushed walnut shell is a hard fibrous material ideal as abrasive. Its grit is extremely durable, angular & multi-faceted, yet considered a soft abrasive. Very fine particle size passes through 40 mesh (sieve size of 0.42mm). Fine light-brown powder, characteristic Oder. Insoluble in water.

The major constituents of shells are as follows-

Sample	Cellulose	Hemicellulose	Lignin
Walnut shells	36.38 ± 0.05	27.85 ± 0.31	43.70 ± 0.57
Pistachio shells	43.08 ± 0.19	25.30 ± 0.46	16.33 ± 0.41

Properties:

Soft but effective abrasive. It is natural, non-toxic, biodegradable, reusable & compatible with anionic, non-ionic & cationic surfactants

Applications-

Body scrubs, shower gels, peeling creams & lotions.

Advantages of natural filler over synthetic filler:

- Availability of renewable natural resources.
- Satisfactory high specific strength and light weight.
- Low Cost and bio-degradability
- Eco-friendly.

3.1.2 Specification and Sources of Raw Materials/Chemicals

Specification and sources of raw materials/chemicals used in the studies are given in Table 3.1

Sr. No.	Chemicals	Source	Specification
1	Epoxy resin Araldite LY 5052	Huntsman Corporation United States,	Industrial Grade
2	Polyamine hardener 5052	Huntsman Corporation United States,	Industrial Grade
3	Silicone Rubber RTV 1010 chemicals	Aditya Genuine Products	Industrial Grade
4	WAX	Indian wax pvt. limited	Paraffin Wax

3.2. MOLD MAKING

Silicon rubber was used to prepare the specimen.

Petri dish was used for making the silicon mold as outer surface controller in which resin was poured to take the shape of mold.

Silicon was mixed with hardener in Ratio of 100:4 (in ml).

3 replicas of specimen were made by wax.

Then it will fix on surface of petri dish by suitable adhesives.

It will take 24 hours to solidification of silicon, then mold was taken out from petri dish.

(a) **(b)**

Fig 4 Preparation of mold & silicon rubber used

(a) ASTM size wax replica is made and fixed in petri dish.
(b) Removal of solidified silicon rubber i.e silicon mold.

3.3 Methodology

The systematic methodology highlighting how the progress of proposed work towards achieving desired objectives was proceeded, involves the following steps:

- Selection of raw materials
- Washing and drying of shells in oven. (Temperature range 60-70 degree Celsius)
- Preparation of fillers after drying.
- Treating fillers with 5% NaOH solution.
- Preparation of composite with 10%, 15% and 20% composition.
- Characterization and testing of composites

3.3.1 Preparation of fillers by shells

In this project Walnut and Pistachio shells were used for preparation of filler then added with Epoxy resin.

They are crushed with grinder and after that they are treated with 5% NAOH solution and then filtered and dried in oven (65-70 degree centigrade)

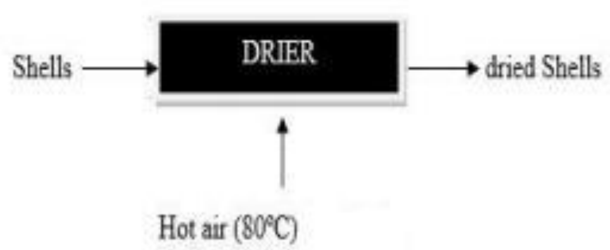


Fig: 5 Filler treatment with NaOH sol.

Fig .6 Fillers obtained after treatment

3.3.2 Preparation of samples

To prepare the samples, Fillers were first mixed in the epoxy resin Araldite LY 5052 at room temperature. A stoichiometric ratio (2:1) of the hardener 5052 was added and mixed with mechanical stirring. Epoxy need 24 hours solidify after that specimen was taken out and trimmed.

Fig: 7 Prepared samples

3.3.3 FINAL SPECIMENS:

The tensile specimen after curing in mold was look like-

Figure 8 Curing of samples

After molding impact specimen are in rectangle shape but for tensile specimen should be in dumbell shaped so for that we grind the specimen on grinding machine. After grinding operation specimen was ready to perform tensile test. And for impact test we make a notch on specimen by knife.

Fig: 9 Final specimen

3.5 Mechanical test

Mechanical properties of different composite samples were measured according to standard ASTM standards as follows

Tensile Testing (ASTM D 638)

Flexural Testing (ASTM D790)

Impact Testing (ASTM D256)

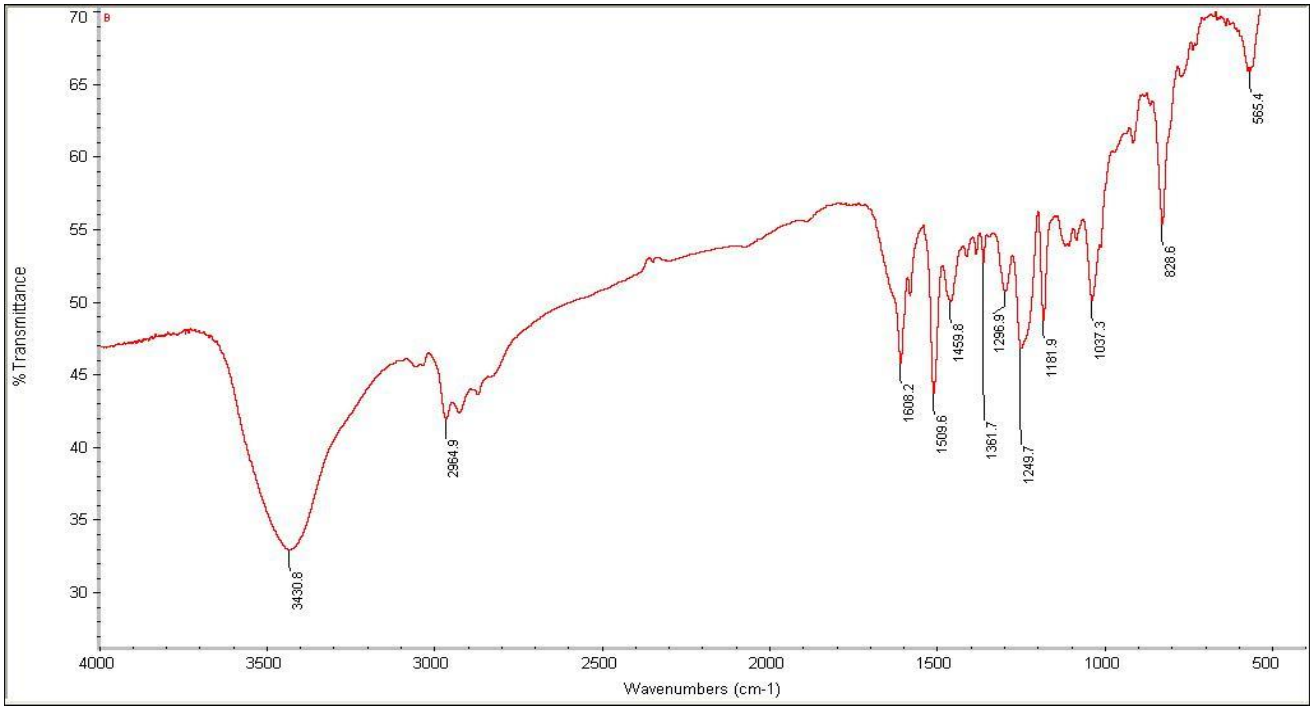
Hardness Testing (ASTM D 2240)

CHAPTER 4. RESULTS AND DISCUSSION

4.1

FTIR

Neat Epoxy

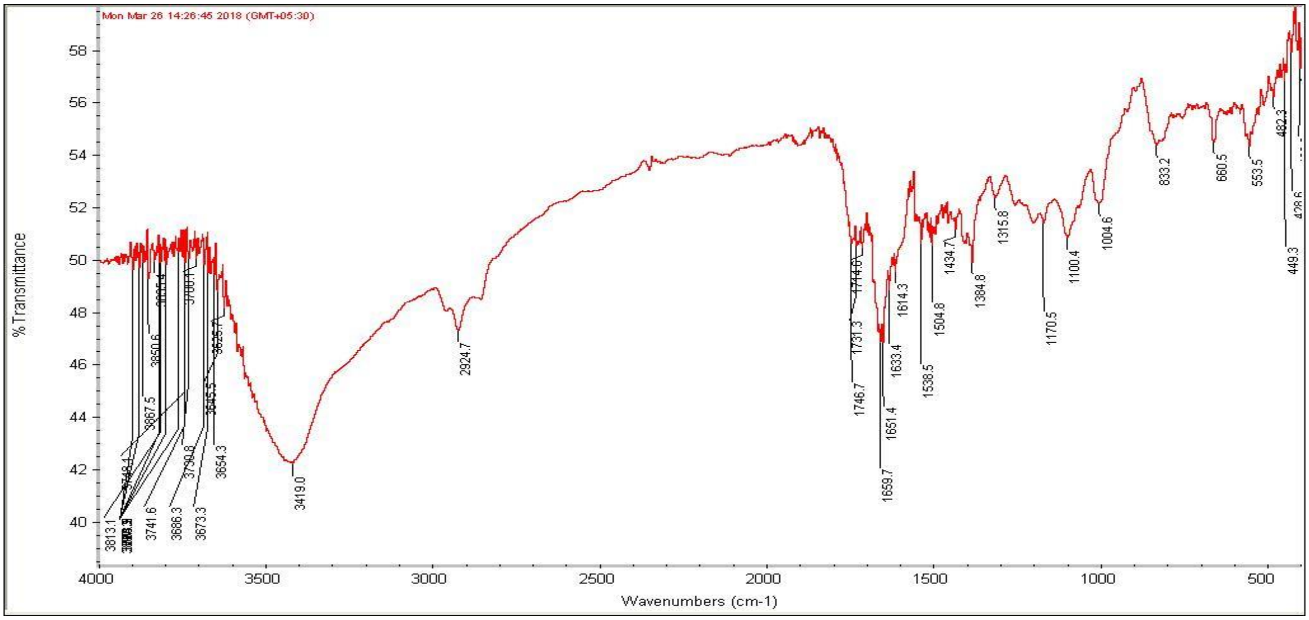


Graph 1

Absorption band assignment in the FTIR spectrum of neat epoxy:

Range(cm^{-1})	Observation
3430	OH Stretch
3057	Stretching of C-H of the oxirane ring
2964-2857	Stretching of C-H of CH_2 and CH aromatic &aliphatic
1608	Stretching C=C of aromatic rings
1509	Stretching of C-C of aromatic
1037	Stretching of C-O-C of ether
828	Stretching of C-O-C of oxirane ring

10% Walnut epoxy composite



Graph 2

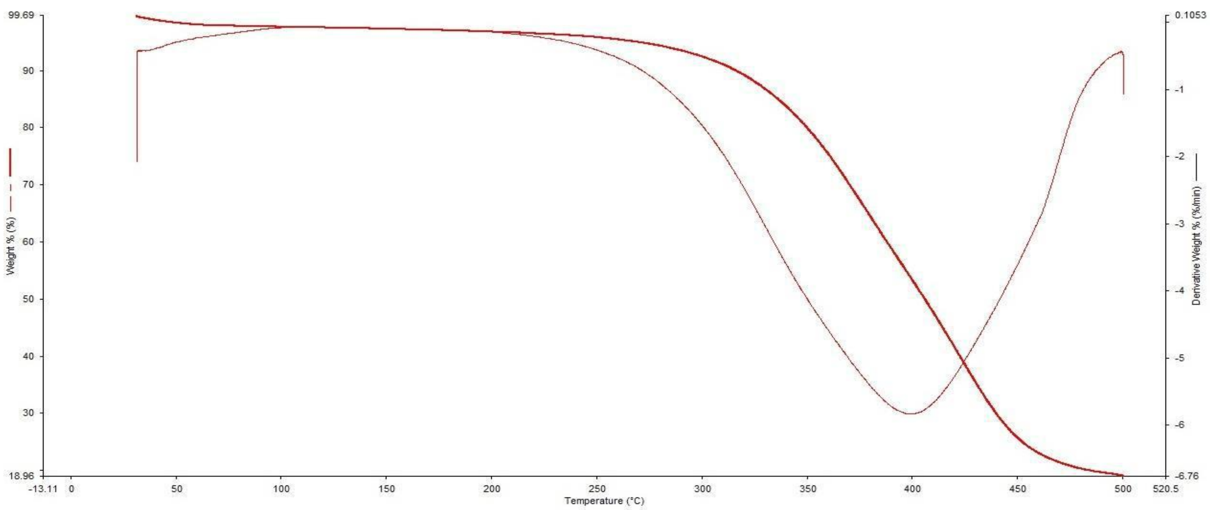
Absorption band assignment in the FTIR spectrum of 10% walnut epoxy composite:

Range cm-1	**Observation**
3300-3500	-OH cellulose, hemicellulose
2900-2950	CH asymmetric stretching
1746-1730	C=O stretching lignin, hemicellulose
1633-1614	C=O Lignin Stretching and NH stretching
1605-1580	Benzene Ring stretching lignin
1221-1230	C-O-C stretching lignin
1100	C-C stretching
1004	C-O stretching cellulose, hemicellulose, and lignin

FTIR studies of neat epoxy and after alkali treatment were compared for structural changes which show increased functionalization of shell particles after the alkali treatment. FTIR studies also confirmed the increased hydrogen bonding of Treated walnut with Epoxy resin. Increased hydrogen bonding along with sufficient methylene linkages were observed in 10 % walnut based epoxy composites containing Treated walnut.

4.2 Thermogravimetric Analysis

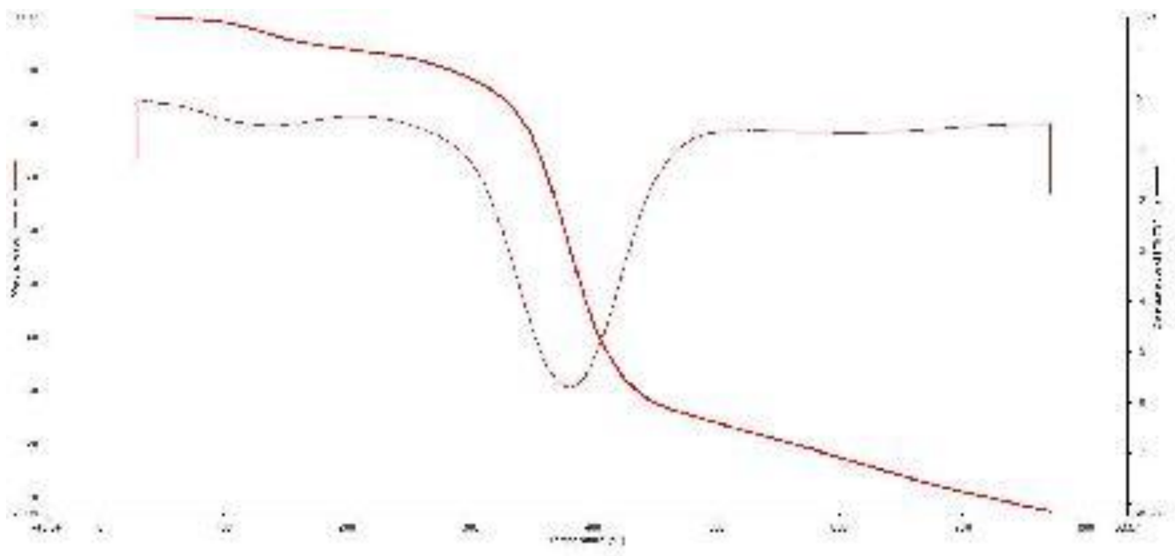
Neat Epoxy



Graph 3

Degradation occurs in one step , the onset temperature for decomposition of composite is nearly 270˚C and the derivative of thermogram represent at 400˚C max. loss in weight.

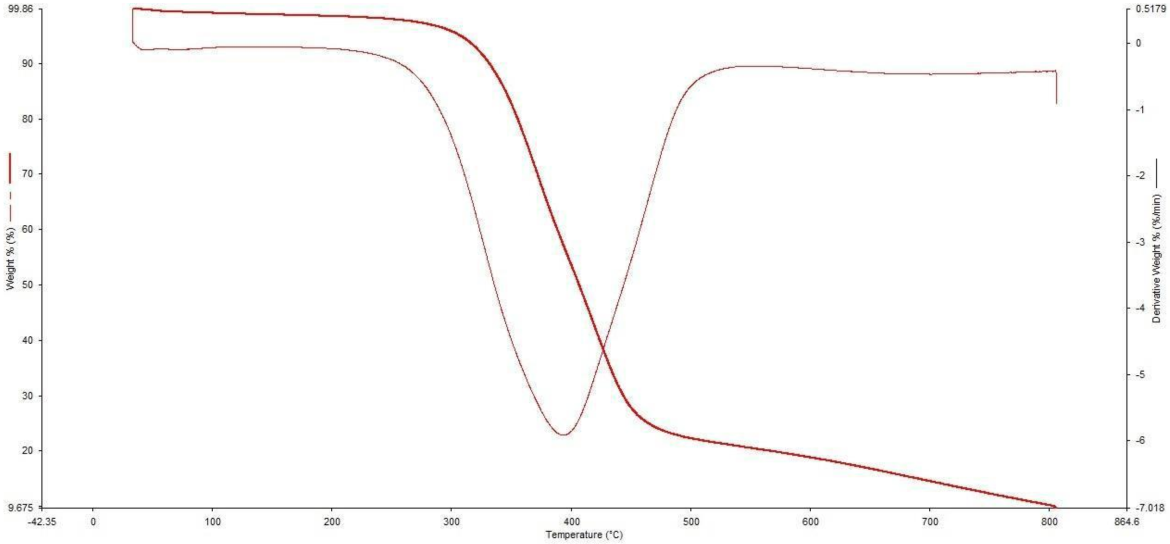
10% Walnut Epoxy composite



Graph 4

Degradation occurs in two steps, due to modify with walnut fillers in composite. For first step degradation the onset temperature for decomposition of composite is nearly 290˚C and the derivative of ther mogram represent at 350˚C max. loss in weight. It shows the evenly distribution of the walnut filler in the composite and phase separation.

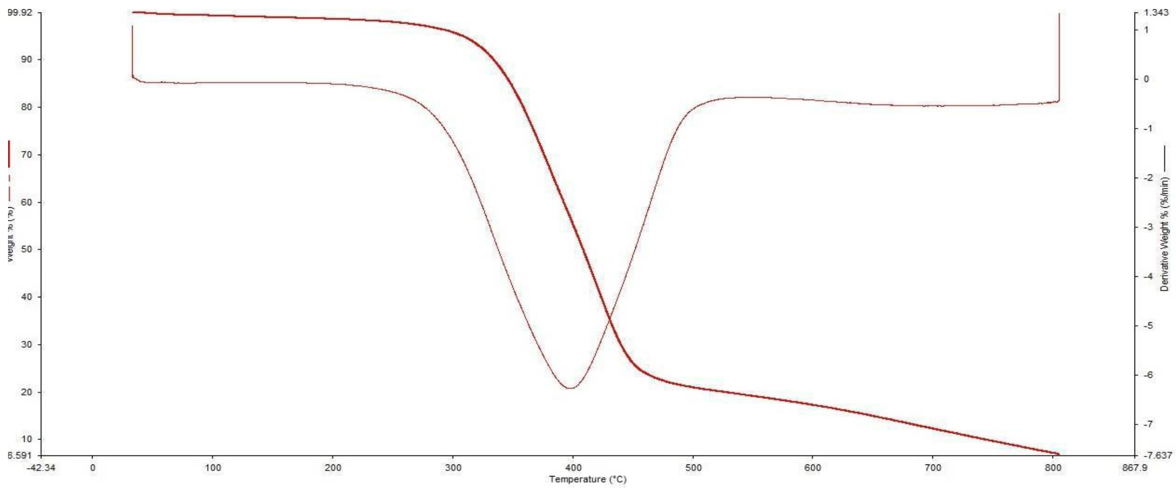
10% Walnut/Pistachio composite



Graph 5

The onset temperature for decomposition of composite is nearly 310°C and the derivative of thermogram represent at 470°C max. loss in weight. It shows that the composite phase has agglomerated and loses its property and degraded at with epoxy. Thus, these results show that chemical treatment is not required if the treated walnut/ pistachio filler based composite material is processed and used at temperature range from 0 °C to 250°C temperature.

10% Walnut/Metal salt composite

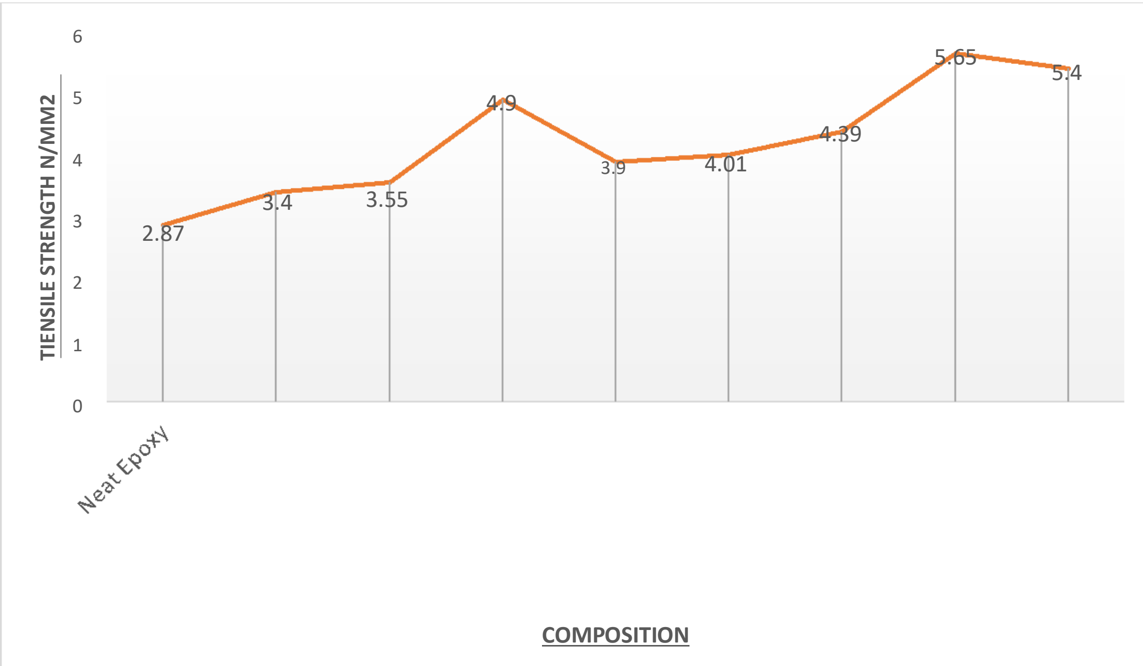


Graph 6

The onset temperature for decomposition of composite is nearly 310˚C and the derivative of thermogram represent at 470˚C max. loss in weight. It shows that the walnut/metal salt has agglomerated and loses its property and degraded at with epoxy.

The above results also show that the thermal stability of the Metal salt/Walnut filler reinforced epoxy composite increased as compared to the neat epoxy sample. TGA analysis also shows that Metal salt/Walnut filler contains more inorganic minerals in comparison to treated chemically treated fillers sample. However, it may not affect the end use of composite material, if they are not used in adverse chemical environment.

4.3 Tensile test

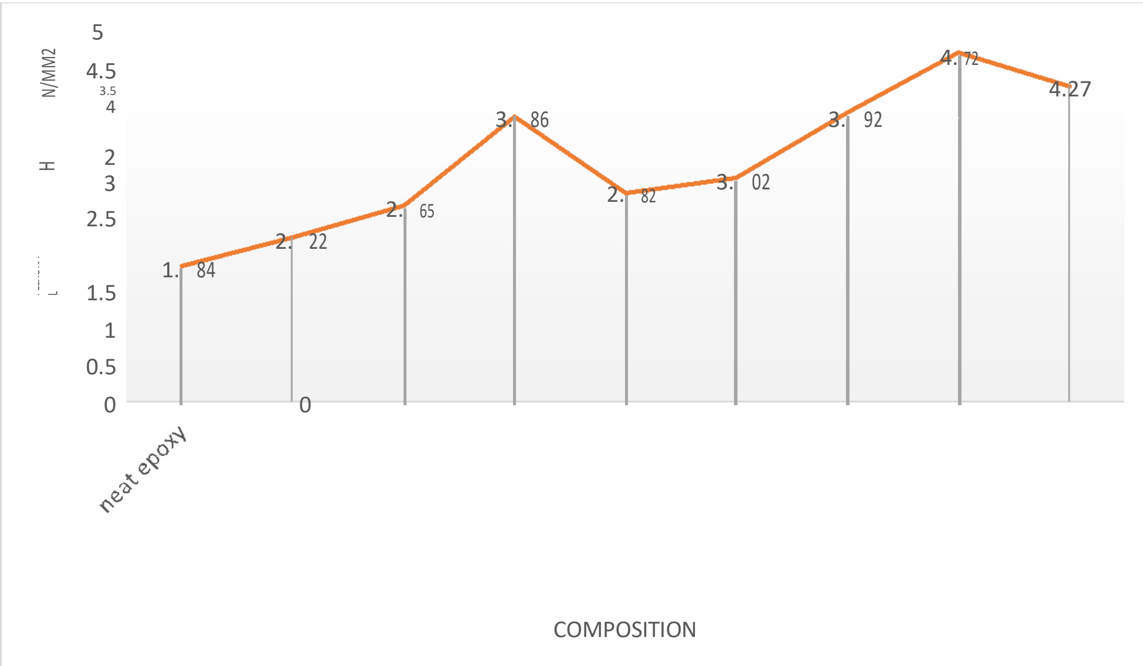


Tensile strength (N/sq.mm) of different composition

Graph 7

Above graph shows that the composite material of reinforced filler of walnut/pistachio/metal salt has increased tensile strength as compared to the neat epoxy sample, because the fillers has increased the ductility of the epoxy (brittle) and also increased its toughness. Further increasing the metal ion content decreased the tensile strength because the content forms agglomeration and made the material brittle, due to which the samples with 15% walnut & metal ion composition show less tensile strength as compared 10%.

4.4 Flexural Strength

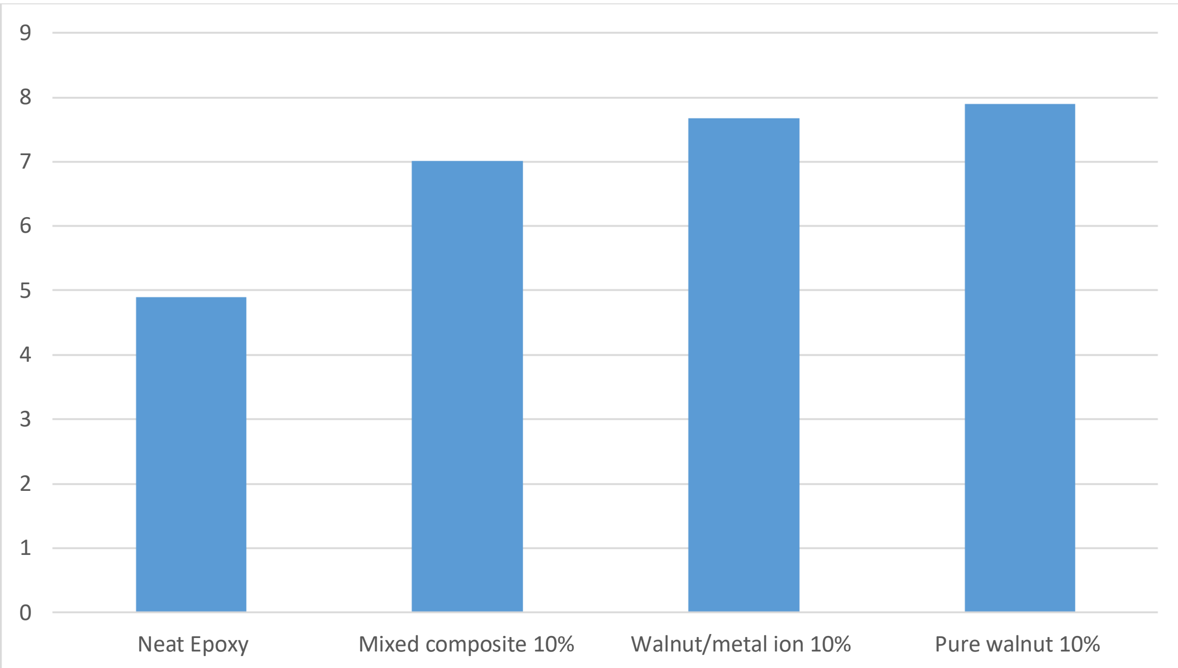


Flexural strength (N/sq.mm) of different composition

Graph 8

Above graph shows that the composite material of reinforced filler of walnut/pistachio/metal salt has increased flexural strength as compared to the neat epoxy sample, because the fillers has increased the flexibility of the epoxy (brittle) and also increased its toughness. Further increasing the metal ion content decreased the flexural strength because the content forms agglomeration and made the material brittle, due to which the samples with 15% walnut & metal ion composition show less flexural strength as compared 10%.

4.5 Impact strength



Graph - 9

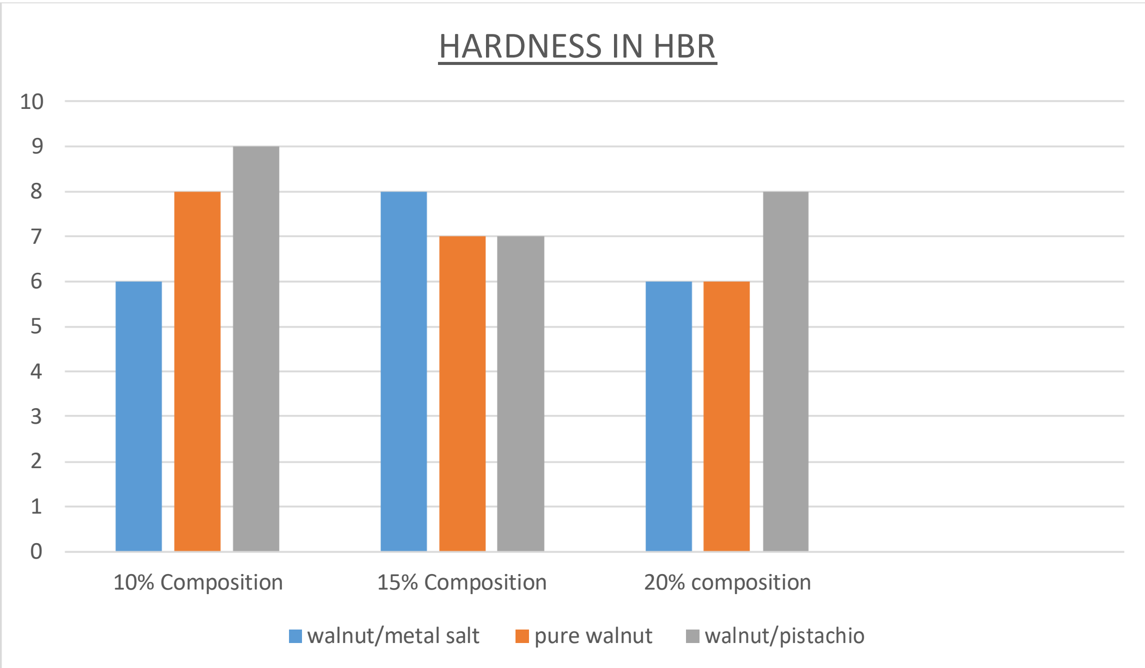
Impact strength in joule (j/m) of different composition

Above graph shows that the composite material of reinforced filler of walnut/pistachio/metal salt has increased impact strength as compared to the neat epoxy sample.

Impact Strength of composition in increasing order is –

Neat Epoxy < Mixed composite 10% < Walnut/metal ion 10% < Pure walnut composite 10%

4.6 HARDNESS TEST



Maximum hardness (up to 9HBR) is present in 10% composition of composites

10 % Walnut/metal salt composition < 10% pure walnut composition < 10% Walnut/pistachio combination possess maximum hardness (9 HBR).

In 15% composition maximum hardness possess by walnut/metal salt composition (8 HBR) but less than that of 10 % composition.

In 20% composition maximum hardness possess by walnut/pistachio composition up to 8 HBR.

CHAPTER 5. CONCLUSION

- A series of Hybrid composites has been developed using Pistachio shell, walnut shell & metal salt with varying weight percentage (10%, 15%&20%) filler.
- Tensile test and Impact Tests were successfully carried as per the ASTM Standard using Universal testing Machine and Impact tester on developed hybrid composite.
- The test results had been tabulated in Tables as well as Graphical Representation of test results were also shown using appropriate graphs.
- As filler content % increases the tensile strength, young's modulus & impact strength of the hybrid composite increases.
- But after 15% it's going to decreases.
- But fine grain filler make composites more brittle.
- Maximum Tensile Strength of 5.67 N/mm2 & Maximum Impact Strength of 8 J/m were observed in metal salt/walnut composite of 10 wt%.

CHAPTER 6: FUTURE SCOPE

Different materials (reinforcement fillers) or the combination of different materials can be used for increasing the mechanical properties of the epoxy resin. Other reinforcement materials can also be used to increase the properties of the composite. Different materials can be incorporated in the composite material to increase the desired property. We have tried pistachio and walnut shell powder combination in epoxy resin and prepared a hybrid polymer composite. In Future different natural fibers like hibiscus sabdariffa, sisal, jute, rice husk, aloe vera etc. can be added to get better strength of the material. Several other operations like drilling, turning, vibration testing, cutting, etc. can also be performed to know the composite material behavior better. Reinforcement material in form of filler as well as in powder can also be incorporated.

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