Thermo-Mechanical Characterization of Epoxy Hybrid Composite Reinforced with Chicken Feather Fiber and Fish Residue Ash Particulates

THESIS

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G.B.PANT UNIVERSITY OF AGRICULTURE & TECHNOLOGY,
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By

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Pantnagar *(Gagan Bansal)*
July, 2016 *Author*

CERTIFICATE

This is to certify that the thesis entitled **"Thermo-Mechanical Characterization of Epoxy Hybrid Composite Reinforced with Chicken Feather Fiber and Fish Residue Ash Particulates"** submitted in partial fulfilment of the requirements for the degree of **Master of Technology** in Mechanical Engineering with major in **Design and Production Engineering** of the College of Post-Graduate Studies, G. B. Pant University of Agriculture and Technology, Pantnagar, is a record of *bona fide* research carried out by **Mr. GAGAN BANSAL,** Id. No. **47053** under my supervision and no part of the thesis has been submitted for any other degree or diploma.

The assistance and help received during the course of this investigation and source of literature have been duly acknowledged.

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We, the undersigned, members of the Advisory Committee of **Mr. Gagan Bansal,** Id. No. **47053**, a candidate for the degree of Master of Technology in Mechanical Engineering with major in **Design and Production Engineering**, agree that the thesis entitled **"Thermo-Mechanical Characterization of Epoxy Hybrid Composite Reinforced with Chicken Feather Fiber and Fish Residue Ash Particulates"** may be submitted in partial fulfilment of the requirements for the degree.

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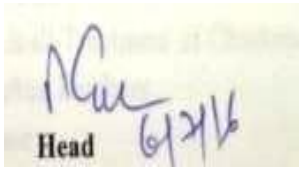

Department of Mechanical Engineering

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LIST OF SYMBOLS & ABBREVIATIONS

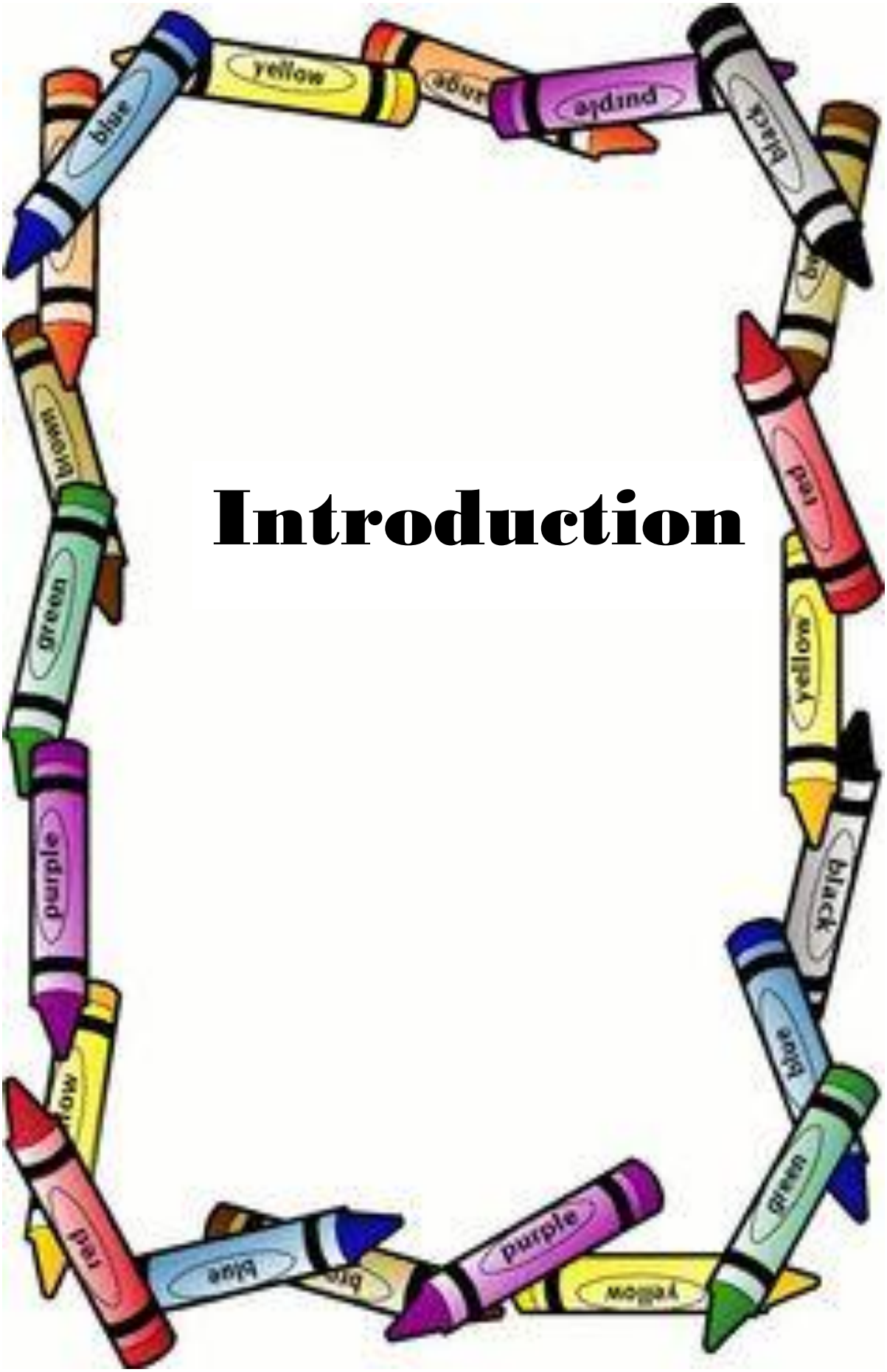
SYMBOLS

ε	: Strain
ε_f	: Flexural Strain
σ	: Stress
σ_f	: Flexural stress
σ_u	: Ultimate Tensile Strength
ρ	: Density of Material
ρ	: Density of Material
ρ_{ct}	: Theoretical density of composite
ρ_{ce}	: Experimental density of composite
ρ_f	: Density of fiber
ρ_m	: Density of matrix
ρ_p	: Density of particulate
ρ_{hct}	: Theoretical density of hybrid composite

ABBREVIATIONS

a	: Depth of notch in impact test specimen
d	: Depth of test beam
gm	: Grams
m	: Mass of Material
s	: Gradient of initial straight line of Load Deflection Curve
t	: Thickness of Impact Test Specimen
v%	: Volume %
w	: Thickness of Impact Specimen
wt%	: Weight Percentage
B	: Width of Test Beam
CFF	: Chicken Feather Fiber
D	: Maximum Deflection of the Centre of the Beam
E	: Modulus of Elasticity

E_f	: Flexural modulus
ERP	: Extracted Residue Powder
HRL	: Rockwell Hardness on L-scale
IS	: Impact Strength
L	: Support Span in Bending Test
P	: Load on a Given Point on the Load Deflection Curve
SEM	: Scanning Electron Microscopy
TS	: Thickness Swelling
V	: Volume of the Material
WA	: Water Absorption



Introduction

Chapter.1 INTRODUCTION

1.1 Introduction

Composite material can be well-defined as a material system composed of two or more unlike components, insoluble in each other, differing in forms, physically distinct and chemically inhomogeneous **(Gupta, 2006)**. The resulting output component possesses properties which are different as compared to individual constituent materials. Composite basically consists of a major constituent matrix and a reinforcing agent i.e. a fiber or any particulate. Now days the combination of fiber- fiber, fiber- particulate, nano-nano, nano-micro, sizing variations, orientation manipulations and thousands of other combinations are made along with the matrix in order to enrich the properties of the resultant composite material and to get the desired output based on the applications of the material.

In the current research work, the hybrid bio composite is fabricated using **epoxy resin (CY-230)** as matrix with **hardener (HY-951)** as curing agent, reinforced with varying weight percentages (wt%) of **chicken feather fiber (CFF)** and later **extracted residue powder (ERP)** from **Rohu fish** is used as Particulate to enhance the mechanical and physical properties of developed composite. Composite materials with their high strength to weight ratio and their diverse functionalities have attracted most of the researchers towards the advance study of the hybrid composites.

Epoxy resin was discovered in 1909 by Prileschajew **(May, 1988)**. Ciba-Geigy Ltd. in 1946 first introduced epoxy resins **(Nagarjuna *et al*., 2014)**. Epoxy resins are the thermoset plastic resin which has wide application in the polymer composite domain. These resins are synthesized by condensation of bisphenol-A. Epoxy resin CY-230 has negligible shrinkage, excellent resistance to chemical attack, non appearance of volatiles on curing, outstanding electrical insulating properties. In general epoxy resin exhibits excellent corrosion and chemical resistance, high stiffness, good dimensional stability, better surface properties, good wetting ability and shows immense characteristics at elevated temperatures. Epoxy resin is mostly used for fiber reinforcement in structural adhesives, surface coatings and electrical laminates. It has wide application in construction, automotive, sectors, commodity and aerospace industries due to these desirable properties.

Thermosetting resins like the epoxies, polyurethane, polyester, phenolic, etc. are in widespread use together with the natural fiber to form the composites with higher performance applications. They endow with high mechanical properties, in particular strength and stiffness, at suitably low prices. The broad word epoxy resin explains a set of thermosetting resins organized by the ring-opening polymerization of compounds containing an average of more than one epoxy group per molecule **(Irfan, 1998)**. Hardener HY-951 used in the current research has product name **ARADUR** is manufactured by Petro Araldite Pvt. Ltd., Chennai. It is yellowish-green colored liquid. It is used as a curing agent and to permanently solidify epoxy resin.

In the past few years, the biological fibers have turn out to be an eye-catching reinforcement for many polymeric matrixes from both the ecological and economical point of view. The main source of obtaining the biological fibers is from plants, animals and minerals. The higher volume occupied by the natural fibers as compared to the synthetic ones due to their lower weight perk up the fuel efficiency and reduced emission in automobile applications **(Wambua *et al*., 2003** and **Sahieb and Jog, 1999)**.

Chicken feather fiber is used as the reinforcement material in the current fabrication process. CFF are the waste product after processing chicken for food. In the current work various chicken's feather is used as a fiber like Kadaknath, Columbian, Guinia Fowl, RIR, Uttara Fowl, Raising Guinia, White Lagan etc. found in the Pantnagar University Poultry farm (figure 1.1). Chicken feather is approximately 91% keratin, 8% water and 1% lipids by mass **(Kock, 2006)**. Both the fiber and quill consist of hydrophobic keratin that's why CFF shows irregular surface finish during composite fabrication. The CFF quill improves the acoustical and mechanical properties in the material **(Huda *et al*., 2007).**

The addition of multi elemental particulate with various individual elements like calcium, carbon, silica, phosphorus, sodium etc. and the compounds like calcium carbonate ($CaCO_3$), SiO_2, NaOH etc. leads to separate reactive properties with epoxy. Nano sized $CaCO_3$ when added to trifunctional epoxy resin shows good thermal stability and improved cross linking properties **(Fan-Long and Soo-Jin, 2009).** The white colored ash extracted from residue of Rohu fish is used as particulate in order to improve the mechanical properties of developed composite.

Figure 1.1: Types of Chicken available in University Poultry Farm, Pantnagar

The mix feather is first alkali treated using diluted KOH to remove dirt and oil from the feathers and then dried in open atmosphere for 48 hours and then the feathers are cut in minimum length (5-12 mm) in a random manner.

1.2 General Characteristics of CFF

Addition of CFF in the thermosetting Epoxy resin polymer helps to achieve various desired outputs because of the following characteristics **(Subramani *et al.*, 2014)**:-

a) CFF acts as a toughening agent when reinforced in epoxy resin.

b) CFF possess hydrophobic nature.

c) It augments stiffness to the matrix.

d) Behaves as a co monomer for the resin.

e) CFF induces plasticity in the deformation zone near crack tip in order to improve its oughness.

f) Acting as a free radical trap to reduce radical scission effects during fracture in highly cross linked polymers.

g) CFF improves flame fighting ability.

h) CFF provides improved thermal immovability and photo resistance.

i) CFF upgrades fatigue life.

j) CFF contributes to the waste management of materials.

1.3 Research Objectives

The main objective of the current research is to develop a cost effective and environment friendly hybrid biocomposite from livestock waste i.e. chicken feathers and residue powder (extracted from fish) which can provide the composite with enhanced strength or as comparably equal to the strength and other properties of available epoxy resin with hardener. The aim behind such work is to have sustainable development in the field of material science engineering and advance material research. Variation in the properties with varying percentage of fiber and particulate in the matrix is seriously monitored and the verdict is done to get the optimum results with maximum accuracy and precession. The following research objectives are under taken in the current master degree work:-

- Extraction of fish residue powder / white ash from Rohu fish residue.
- Casting and mechanical characterization of composite with varying weight percentage (wt%) of chicken feather fiber (CFF) in epoxy resin ranging from 1 wt% - 7 wt%.
- Optimization of CFF weight percentage on the basis of mechanical strength.
- To study the effect of fish residue powder / extracted fish white ash on mechanical, physical and thermal properties.
- Characterization of mechanical/ thermal properties with scanning electron micrographs and X-Ray powder diffraction spectra.
- Suggesting the suitable applications of the output composite in the real life situations.

1.4 Approach

Firstly, the epoxy resin and 10 wt% hardener with varying weight percentage (wt%) of chicken feather fiber (i.e. from 0% to 7%) is fabricated using simple hand lay-up technique. Various mechanical tests like tensile, compression, Izod impact, and Rockwell hardness test etc. were performed on the CFF filled composites.

As per the required application criteria for manufacturing hard and impact resistance material for daily use goods and based on the obtained hardness and impact test results the optimum composition was diagnosed.

Later epoxy resin based hybrid biocomposite reinforced with constant chicken feather fiber and varying weight percentage of extracted residue powder from fish as particulate are determined on the basis of enhanced mechanical and physical properties. Various mechanical tests like tensile test, compression test, Izod impact test, Rockwell hardness test, flexural test, water swelling test, density measurement etc. are performed on the final specimens and the results were than diagnosed. Also the morphological study of the fractured surfaces of all the prepared samples is done using SEM images, FE-SEM outputs and XRD analysis. Also the thermal analysis using TGA/DTA helped us to get the complete knowledge about the variation in the properties with varying percentages of fiber and particulate.

Review Of Literature

Chapter.2 REVIEW OF LITERATURE

2.1 Epoxy Resin

Epoxy resin may be defined as the polymer in which chain extension and cross linking occurs through epoxy group reactions **(Gould, 1970** and **May and Tanaka, 1973)**. It represents step growth polymerization reaction that composed of two parts: a fluid prepolymer chain with reactive epoxide group on each end and a hardener (**Pragyan, 2013**). Highly cross-linked epoxy resin used for engineering applications is normally strong but brittle. Therefore, many attempts have been made to improve toughness of epoxy resin **(Ratna, 2001, Kong *et al.*, 2006, Kumar and Kothandaraman, 2008** and **Zhou *et al.*, 2008)**. It is widely used in electrical and electronics industries because of their good properties. It serves as the excellent matrix in composite development. Here also epoxy resin CY-230 is considered as the matrix material for the research work.

2.2 Background of Chicken Feather Fiber

Chicken feathers are considered as the left-over products of the poultry industry. Billions of kilograms of waste feathers are engendered each year by poultry processing plants, creating a stark solid waste disposal concern **(Parkinson and Schmidt, 1998).**

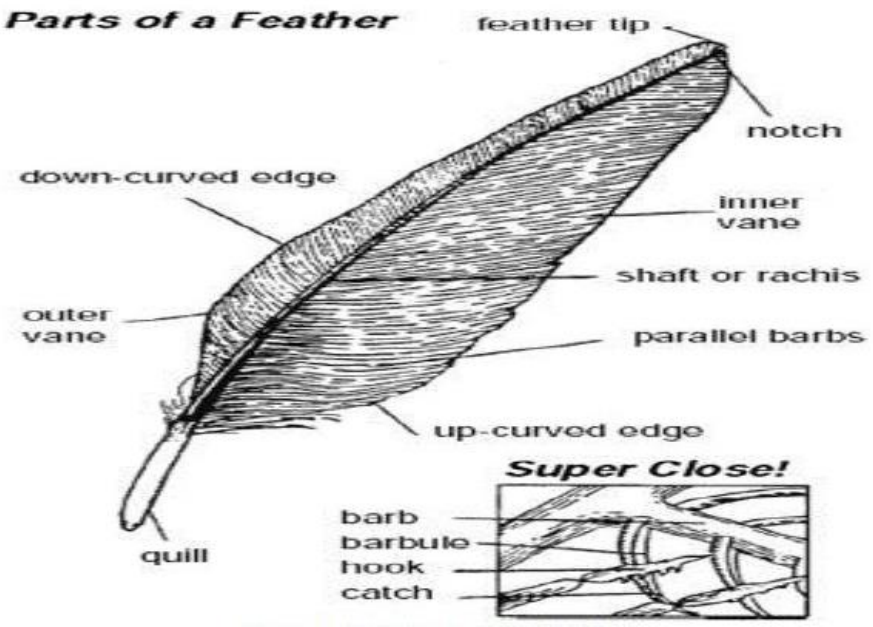


Figure 2.1: A typical Chicken feather fiber (Adetola *et al.*, 2014)

Feathers are greatly ordered, hierarchical branched structures as discussed by **Adetola *et al.*, (2014),** in figure 2.1, ranking among the most complex of keratin structures establish in vertebrates **(Yu, 2002).** Chicken feathers are roughly 91% protein (keratin), 1% lipids and 8% water **(Kock, 2006** and **Subramani *et al.*, 2014).**

Henceforth, based on **Bledzki *et al.* (1999),** all natural fibers are hydrophilic in nature and their moisture socking intensity can reach up to 3-13%. But the fact is not always true. As human hair, many CFFs (**Hernandez *et al.*, 2006**) etc. are considered as hydrophobic in nature.

2.3 Curing Agents (Hardener)

Curing agents or hardeners are the chemically active compound which converts epoxy resin into hard, infusible thermosets and promotes the crosslinking reaction either by poly addition or by homo polymerization **(Irfan, 1998)**.

Kiew *et al.* (2013) included methyl ethyl ketone peroxide (MEKP) as a curing agent in unsaturated polyester with 1% concentration by weight of matrix.

It is difficult to make generalize comment with respect to structure and properties of epoxy resins because the type of curing agent has a remarkable effect on its behavior. The curing agent largely determines the chemical resistance. Anhydride cure appears to have good thermal stability, chemical resistance (except to alkalis and electrical insulating properties).

In the current research hardener **(HY-951)** is used along with epoxy resin **(CY-230)** because of its exceptionally well binding characteristic with epoxy matrix. Here 10% of hardener by weight of epoxy resin is used during casting as per the recommendation of **Singh (2002)**.

2.4 Review based on mechanical properties of CFF based composites

Sundararajan *et al.* (1990) discussed the effects of size and shape of particle on the fracture toughness and strength based on particle-matrix adhesion. Also an increase in the flexural and tensile strength was noticed on increasing the specific surface area of the particles.

Richard *et al.* (1994) investigated the young's modulus of the feather keratin. Richard explained that variation in the flexural stiffness of the rachis along the length of a primary feather. Tensile tests on compact keratin from eight different species of birds showed similar moduli (mean E=2.50 GPa) apart from the grey heron (E=1.78 GPa). It was finally concluded that the flexural stiffness of the whole rachis is controlled by its cross-sectional morphology rather than by the material properties of the keratin.

Huda *et al*. (2007) made comparative study for acoustical and mechanical properties of composites from ground chicken quill and polypropylene (PP) and compared it with jute–PP composites. Results obtained showed that quill has better compatibility to PP compared with jute and an ideal candidate for acoustic panels and headliner substrates.

Uzun *et al.* (2011) studied mechanical actions of chicken quills and CFF reinforced polymeric composites and found that the impact properties of the CFF reinforced composites are significantly better than the control composites.

Ledezma *et al.* (2013) performed mechanical characterization on the composites of CFF and high density polyethylene (HDPE) developed for hot runner molds in injection molding applications. The composite with 20% w/w CFF and 2% w/w maleic anhydride (MA) as a compatibilizer showed similar tensile strength, impact resistance and elastic modulus as compared to HDPE but delivered better performance in flexural strength and flexural modulus.

Subramani *et al*. (2014) scrutinized the mechanical properties of the CFF reinforced with polyester and phenyl ester and presented the comparative relation between them. It was reported that the compressive strength of the CFF reinforced composites are significantly superior to the control composites. The tensile and flexural property values decreases on increasing the fiber loading percentages. Finally, it was concluded that the polyester shows better properties than phenyl ester and it can be used in any engineering application due to its improved behavior and structural applications. Also the characteristics of CFF were sequentially listed.

Adetola *et al.* (2014) investigated physical and mechanical properties of few selected chicken feathers found in Nigeria. Here the portland cement is used as binding material and calcium chloride ($CaCl_2$) is used as cement setting accelerator. It was finally concluded that the tensile strength and strain were found to be inversely proportional to

volume fraction (V_f) while the young's modulus was proportional to volume fraction up to 0.20 and inversely proportional at V_f above this value. Here the typical CFF was described pictorially as shown in figure 2.1.

Chandra *et al.* (2014) analyzed TGA, DSC and DTG properties of epoxy composites reinforced with feather fibers of 'Emu' bird. The composites were prepared using various weight percentages of Emu feathers (1 gm to 5 gm) and varying lengths of feather fiber (1 cm to 5 cm).

2.5 Reviews on thermal properties of CFF based composite

Hernandez *et al*. (2006) carried out dynamic, thermal and mechanical characterization of polymeric composites reinforced with keratin CFF. The thermal stability and transition temperature were found to be higher than standard PMMA. At higher temperature, the reinforcement provides higher stability, as reflected in the modulus behavior. Keratin bio fibers show hydrophobic nature and allow an evenly distribution within and adherence to polymers. The hydrophobic character revealed the mechanical failure occurred during testings.

Hong and Wool (2005) reported that the thermal energy required to perturb the fiber is higher than the energy required to perturb the quill. The obtained results reviled their comparative thermal analysis.

Cheng *et al.* (2009) reported the mechanical and thermal properties of CFF/PLA green composites. CFF reinforced poly lactic acid (PLA) composites were processed using a twin screw extruder and an injection molder. The morphology was evaluated by SEM images. Also the mechanical and thermal properties of pure PLA and CFF/PLA composites were compared using Dynamic Mechanical Analysis (DMA), Thermo Mechanical Analysis (TMA) and Thermo Gravimetric Analysis (TGA). The results obtained assist the development of ecofriendly composites from biodegradable polymers.

Fan-Long and Soo-Jin (2009) revealed the thermal stability occurring in the modified epoxy resin due to inclusion of calcium carbonate in the tri functional epoxy. Increasing the nano $CaCO_3$ increases the thermal stability and the cross linking density as compared to neat epoxy.

2.6 Reviews on chicken feathers as fiber in composite

Barone *et al.* (2005) investigated polyethylene reinforced with keratin fibers obtained from chicken feathers. From uniaxial tensile testing, an elastic modulus and yield stress of the composite over the virgin polymer was observed over a wide range of fiber loading. The keratin fiber had a density lower than the LDPE (low-density polyethylene) resulting in composite materials of reduced density.

Bullions *et al.* (2005) studied contributions of feather fibers and various cellulose fibers in developing polypropylene matrix (PPM) composites. PPM composites were made with varying compositions of feather fiber (FF), recycled kraft pulp fiber (PF), recycled news pulp fiber (NF), and retted kenaf bast fiber (KF). The contributions of the four different fibers to composite strength were: PF > NF > FF > KF.

Winandy *et al.* (2007) evaluated the properties of fiberboard made with different proportions of wood fiber (MDF) and CFF. Initial strength and stiffness of MDF-CFF composites were lower than that of all-wood control panels, even though no wax was used in manufacturing the fiberboard composites, the physical properties of MDF-CFF mixtures showed a marked improvement in resistance to some modes of water absorption compared with control panels made of all-wood fiber.

Menandro (2010) studied about CFF reinforcement in cement-bonded composites. Results revealed that the stiffness, flexural strength, and dimensional stability of the feather-cement boards were reduced as the proportion of feathers was increased above 10%. Higher proportions of feather, however, showed significant drop in Modulus of Elasticity (MOE) and Modulus of Rupture (MOR), and rise in water absorption and thickness swelling after 24 hours of soaking in water.

Reddy *et al*. (2014) demonstrated that chicken feathers can also be used as matrix to develop completely biodegradable composites with properties similar to that of composites having polypropylene (PP) as matrix. Utilizing feathers as matrix could enable us to develop low cost 100 % biodegradable composites containing feathers or other biopolymers as the reinforcement. Chicken feathers as matrix provided higher tensile and flexural properties to the composites compared to using PP as matrix.

Hernández *et al.* (2014) studied the performance of a protein such as feather keratin fiber over a biopolymeric matrix composed of poly saccharides. Addition of keratin enhanced the thermal stability of the composites compared to pure matrix. The

morphology indicated uniform dispersion of keratin in the chitosan-starch matrix as a result of good compatibility between these biopolymers.

2.7 Reviews on chemical properties of CFF based composite

Shalwan and Yousif (2013) studied the effect of alkaline treatment on different natural fiber and different polymer matrix and found that tensile strength, wear strength of treated fibers were better than untreated.

Chandra *et al*. (2015) tested chemical resistance and biodegradation of Emu feather fiber reinforced epoxy composites. Here, composites were prepared with epoxy (Araldite LY-556) as resin and 'emu' bird feathers as fiber. The results reveal that weight gained for the composite samples after three days, when treated with HCl, sodium carbonate, acetic acid, NaOH, nitric acid and ammonium hydroxide. Weight loss was detected for all the samples including pure epoxy when treated with benzene, carbon tetra chloride and toluene.

2.8 Reviews on filler / epoxy composite

Park and Yoon (2011) studied thermo-mechanical properties such as glass transition temperature (T_g), dynamic mechanical analysis, tensile and flexural strength. The properties of conventional epoxy/micro-silica composite were improved by the addition of nano silica and it was due to the increase of compaction via even dispersion of the nano-silica among the micro-silica particles. The storage modulus, flexural strength etc. were determined.

Arpitha *et al.* (2014) performed mechanical characterization of epoxy based hybrid composites reinforced with sisal/SIC/glass fibers. Different compositions were tested and results revealed that composites without filler showed better results compared to the composites with silicon carbide filler. Therefore the removal of carbon from the ash was considered necessary before using it as a particulate.

Jin and Park (2009) described the thermal stability of nano sized calcium carbonate ($CaCO_3$) in modified trifunctional epoxy resin. The results showed high cross linking density and decreased coefficient of thermal expansion at the region of high contamination of particles.

2.9 Reviews on application of natural fibers

Jang *et al.* (1989) found a significant improvement in the impact properties of hybrid composites incorporating either particulates or ceramic whiskers. Attempts to understand the modifications in the tribological behavior of the polymers with the inclusion of fillers or fiber reinforcements have been made by many researchers **(Wang *et al.*, 2003)**. Also the hybrid composite materials find wide applications in the field of engineering due to low cost, higher strength-to-weight ratio and ease of manufacturing **(Gururaja *et al.*, 2012).**

Chinta *et al.* (2013) investigated CFF in technical textiles applications. They added by saying that the nonwoven which is prepared by CFF has very versatile and a wide application in the field of technical textiles.

Wang *et al.* (2013) used chicken feathers to prepare high-capacity carbon for super capacitors. In this research work the author concluded that the chicken feather carbon is acted as electrode materials for the first time. Chicken feather initiates from free renewable livestock biowaste. There are abundant micro pores for the activated chicken feather carbon that shows the excellent electrochemical properties.

Giraldo *et al.* (2013) expressed the rachis of CFF for hydrogen storage application. Here the samples were characterized through nitrogen isotherms at -196^0C, FTIR, SEM and XPS.

Bhattacharyya *et al.* (2014) studied the flammability portrayals of natural fibers. Here the sequential sub classification of natural fiber is done as shown in figure 2.2. The proper explanation of the characteristics and properties of different fibers have been discussed by the author.

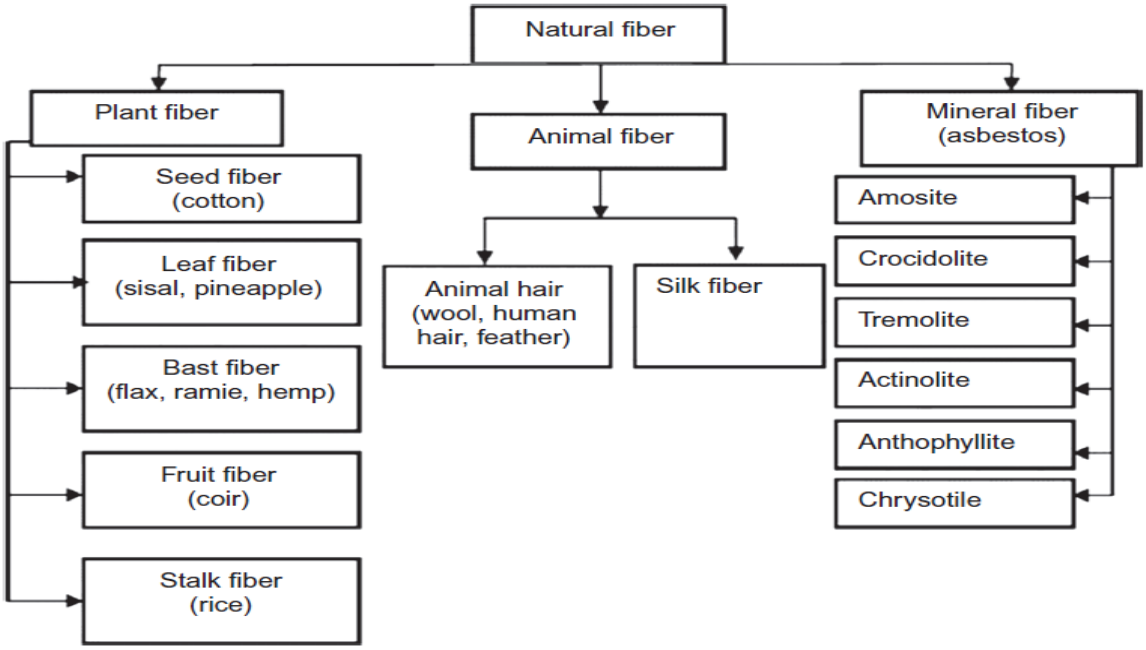


Figure 2.2: Classification of natural fiber (Mohini *et al.*, 2011 and **Bhattacharya *et al.*, 2014)**

Jagadeeshgouda *et al.* (2014) described results based on experimental study on activities of poultry CFF. In the study, reviews on the behavior of CFFs were made to understand their usability as a reinforcing material for composite fabrication.

Reddy (2015) explored the application of poultry feathers in non-food industry and described the importance of poultry industrial waste in material technology and other fields.

2.10 Conversion Methodology

Tuna *et al.* (2015) presented the technique for thermo chemical conversion of poultry CFF of different colors into micro porous fibers. Here, the carbon nitrogen fiber was derived from CFF and the role of different colors on its char characteristics was investigated.

Mittal (1997) performed the laboratory experiment on rice husk ash to get silica from ash. Rice husk ash contains 90-98% silica. Davinder performed the following two reactions under controlled condition to get silica from ash.

$$\underset{\text{(ash)}}{SiO_2} + \underset{\text{(caustic soda)}}{2NaOH} \xrightarrow[\text{6-8 atm.}]{180\text{-}200^{o}C} \underset{\text{(sodium silicate)}}{Na_2SiO_3} + \underset{\text{(water)}}{H_2O}$$

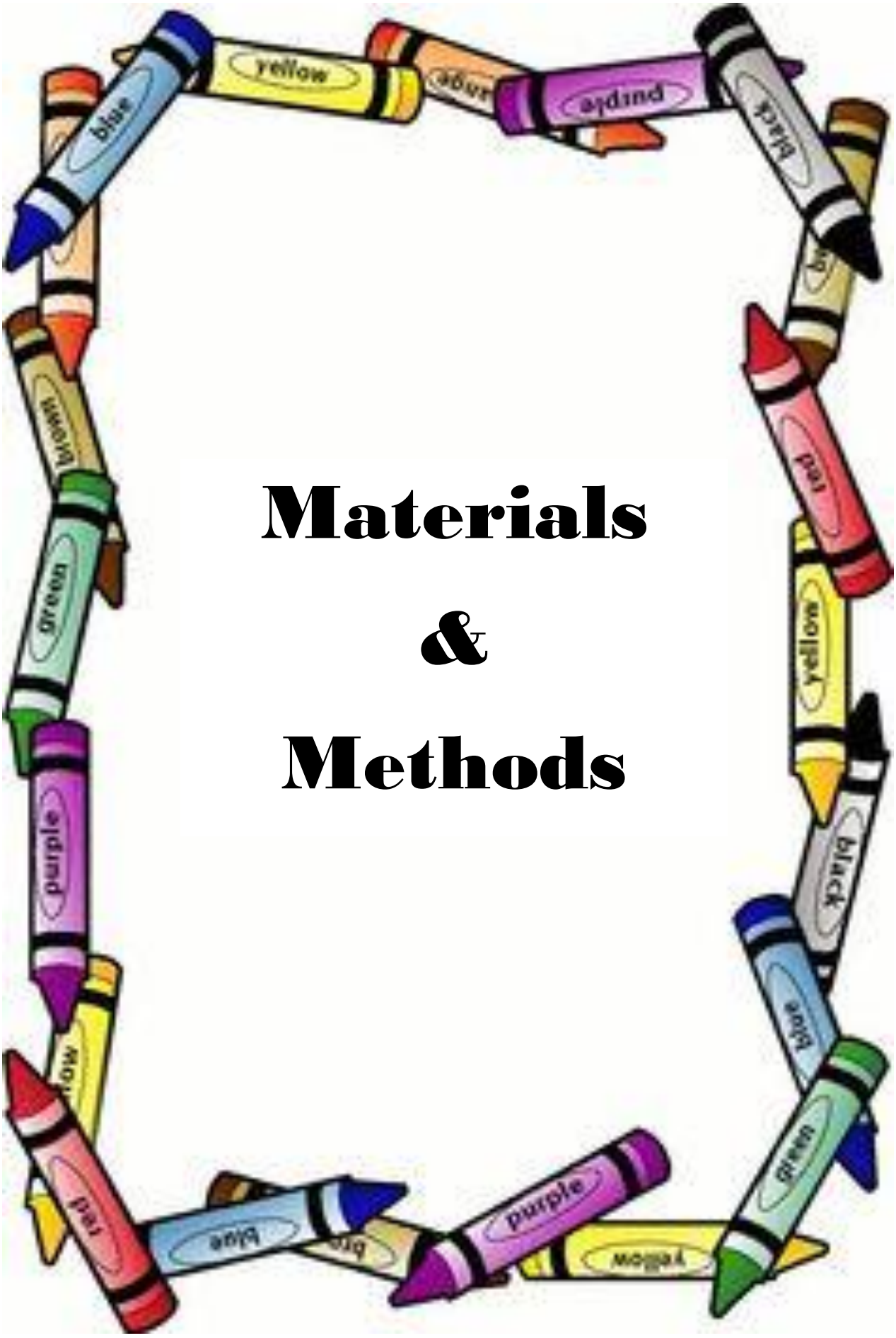
$$Na_2SiO_3 + H_2SO_4 \xrightarrow{90\text{-}100^oC,\ 1\ atm} SiO_2 + Na_2SO_4 + H_2O$$

(sodium silicate) + (sulphuric acid) → (silica) + (sodium sulphate) + (water)

Ragini *et al.* (2014) gave the detailed information about the process of preparation silica powder from rice husk. The results obtained were verified and justified using FTIR and XRD analysis. The compositions in the extracted powder are mentioned along with the weight percentages.

2.11 Sum-up to Literature Review

With the elaborative study regarding the multi directional use of chicken feather as an additive in development of multi phased biodegradable hybrid and non-hybrid biocomposite, the current study can be summed up in a very fruitful manner. CFF can be used in various fields and its abundance availability is the main reason for its upliftment in material science and technology. Livestock waste is utilized in best possible way using different matrix-fiber combinations based on compatibility. The low cost and high strength materials can be developed using advance casting/ fabricating technique discussed in the various literature sited. So the most advanced and effective research is possible in the field of composite development using various livestock waste products. The reinforcement of CFF improves the acoustical, mechanical and physical properties of the polymeric composite. Also the inclusion of particulate and the elements like calcium, silica, sodium etc. shows good compatibility with epoxy resin in elementary as well as compound forms. Thermal stability and cohesive bonding is amended using silica, sodium, calcium carbonate and other micro and nano sized particulates. Therefore, the similar efforts are been made by the use of CFF and fish residue in the current research work to effectively use the livestock waste in the development of advance material.


Materials
&
Methods

Chapter.3 MATERIALS AND METHODS

The current chapter "Materials and Methods" basically composed of the detailed information about the materials used in the fabrication and processing of composite, the casting methodology, details about the various tests being performed and the procedure for testing. The current section systematically outlines the informative data about the research work carried out for the characterization and fabrication of hybrid biocomposites.

3.1 Materials

Specifically in the present research work, the development of hybrid composite requires the matrix (as the major constituent) which is CY-230 Epoxy resin, Binding agent (Hardener) which is HY-951, Reinforcing agent which is Chicken Feathers and the Particulate which is extracted residue powder from Rohu fish.

The different materials used for the casting of composite are described below.

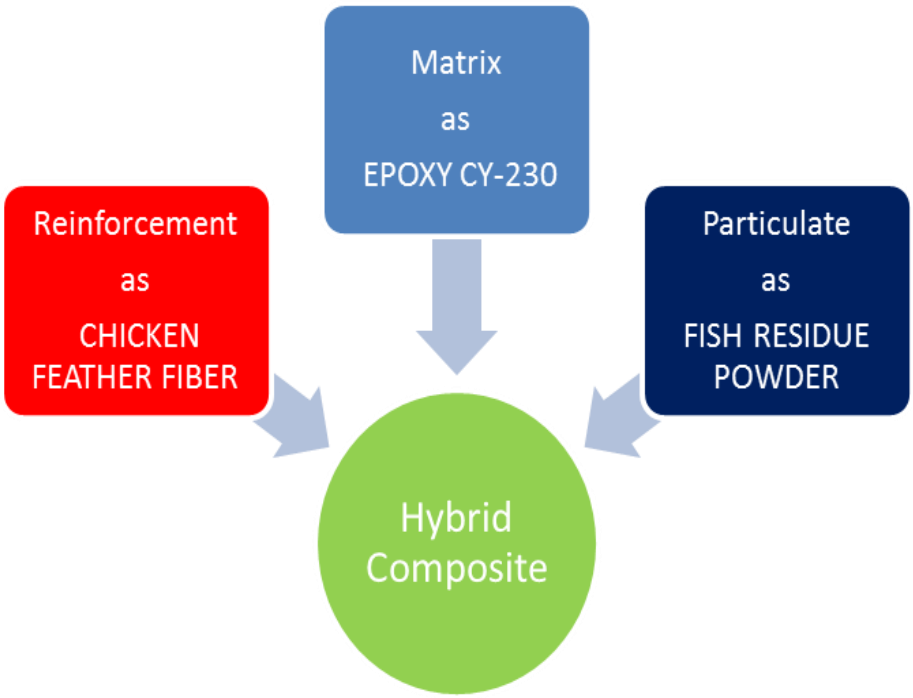


Figure 3.1: Constituents in hybrid composite

3.1.1 Matrix Material

In composite the matrix is the material with major constituent and that holds the relative position of the filler material. Here Epoxy resin (CY-230) is considered as a Matrix material. It is responsible for the environmental tolerance, surface appearance, shape and overall durability of the final product.

3.1.1.1 Epoxy Resin (CY-230)

Epoxy Resin is a thermoset plastic resin with excellent adhesion to wide variety of fibers, superior mechanical and electrical properties, excellent resistance to atmospheric attack of fully cured mixture, good performance at elevated temperatures. In addition to that they have low shrinkage upon curing and good chemical resistance. Araldite CY-230 is a liquid solvent free epoxy resin. Epoxy resin has wide range of industrial applications because of their high strength and mechanical adhesiveness characteristic. It can be easily colored. Curing takes place at atmospheric pressure and room temperature after addition of hardener. Generally, curing shrinkage is very less and may further be reduced by the addition of fillers such as bio-particles, China clay etc.

Density =1150kg/m^3 [when mixed with 10wt% (HY-951) hardener]

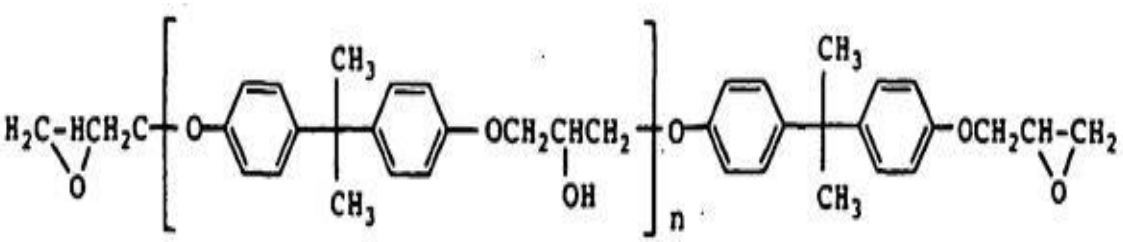


Figure 3.2: Chemical structure of Bisphenol-A based epoxy resin (Irfan, 1998).

In the present investigation CY-230 purchased from M/s CIBATUL Limited, India has been used as matrix material.

3.1.1.2 Hardener (HY-951)

Hardener HY-951 is a yellowish green transparent liquid. Hardener (HY-951) with product name **ARADUR** is manufactured by Petro Araldite Pvt. Ltd., Manali, Chennai. Three most common types of hardener are:

1) Aliphatic Amines

2) Aromatic Amines

3) Acids and Anhydrides.

The triethylenetetraamine (TETA) used here is the aliphatic amine group of hardener. It is used as a curing agent for CY-230 epoxy resin matrix. In the present investigation 10 % wt/wt of hardener has been used in all material developed. The effect of wt% of hardener on the mechanical properties is shown in figure 3.3. The weight percentage of hardener used in the present investigation is as per the suggestion and recommendation of **Singh V.K., 2002**.

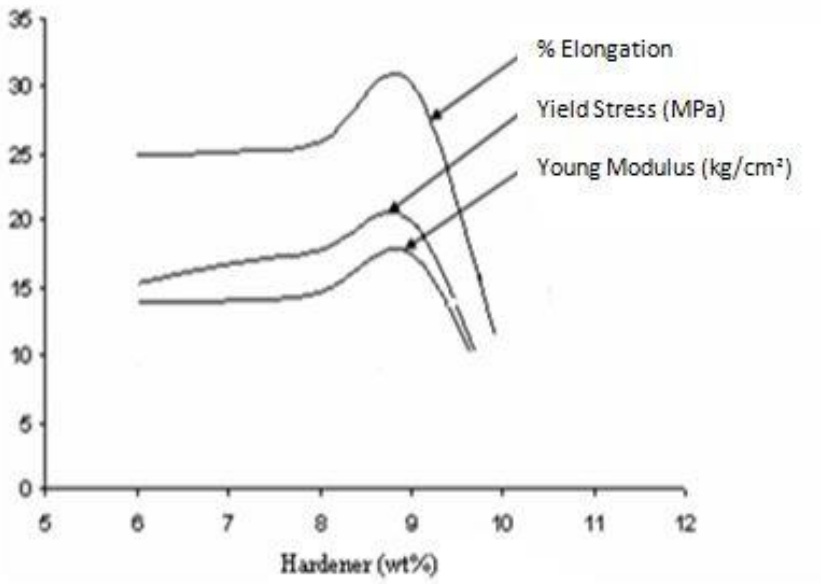


Figure 3.3: Effect of wt% of hardener (HY-951) on mechanical properties (Singh, 2002)

3.1.2 Reinforcing Element

The addition of reinforcing agents to the resin improves the properties of the material. Chicken feathers are used as reinforcing agents to mend and enhance the various properties of the composites material. The main reason for using chicken feather as fiber reinforcing element is the availability of feathers as livestock waste, its disposal problem after extraction of eatable part and various characteristics involved as discussed in section 1.2, Chapter "Introduction".

3.1.2.1 Chicken Feather Fiber (CFF)

Chicken Feathers are the waste product from the processing of chickens for food. Chicken feather initiates from free renewable livestock biowaste (**Wang *et al.*, 2013**). CFF can be considered as a waste by product that is serious causal to environmental pollution due to the disposal problems. Basically there are two main feather disposal methods that is, a burning and burying. But both of them have negative effect on the environment. Chicken feather is approximately 91% keratin, 8% water and 1% lipids by mass **(Kock, 2006)**. The typical structure of chicken feather is shown in figure 2.1. Both the fiber and quill consist of hydrophobic keratin that's why CFF shows irregular surface finish during composite fabrication. Different types of chicken feathers used as a fiber were taken from various chickens available at Pantnagar poultry farm as shown in figure 1.2.

- All the feathers used in the research analysis were collected from Poultry Farm G. B. Pant University of Agriculture and Technology

There are five generally recognized categories of feathers: contour, bristle, semi plume, down and filo plume as shown in figure 3.4.

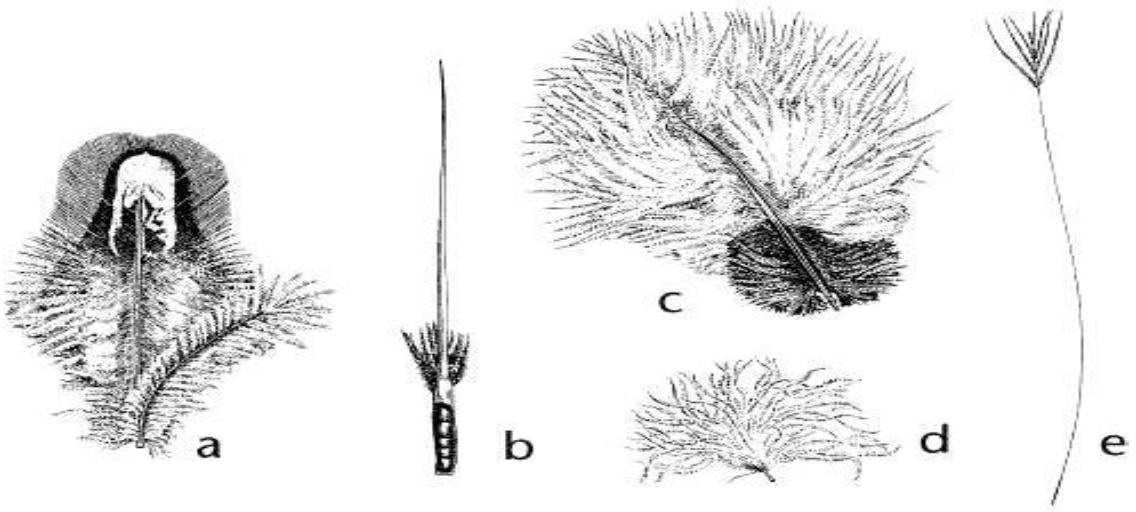


Figure 3.4: Five primary types of chicken feathers: (a) contour, (b) bristle (c) semi plume (d) down (e) filo plume (Bartels, 2003)

In our research we have basically focused on Contour type of feathers. Contour chicken feather is shown in figure 3.4 (a) and figure 3.5.

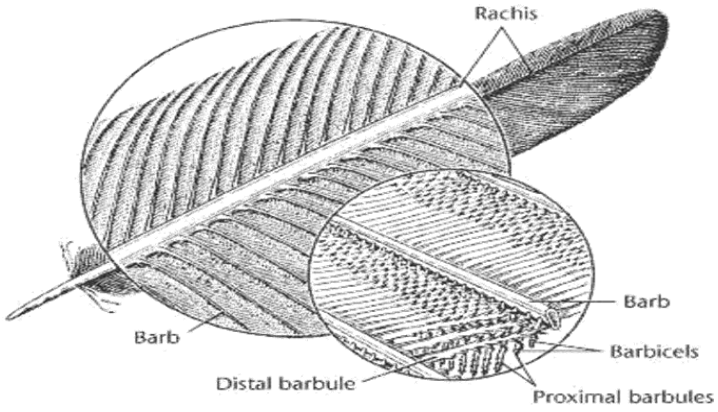


Figure 3.5: A contour feather (Bartels, 2003)

Bristles provide shielding and are found on a chicken's head, at the base of the beak, around the eyes, and covering the nostrils **(Subramani *et al*., 2014)**. The bristle is considered as the smallest feather, which is stiff and has few, if any, short barbs near the tip. Bristle is shown in figure 3.4 (b).

The semi plume mediates between the contour and down class of feather. It stakes the characteristics with both. They have a large rachis and predominantly downy vanes. Semi plume is shown in figure 3.4 (c).

Down feathers are smaller than contour feathers. They are mostly soft and fluffy, located beneath the contour feathers. It provides most of the chicken's insulation and forms a waterproof barrier for contour feathers **(Subramani *et al*., 2014)**. The powder down feather is depicted in figure 3.4 (d).

Filo plumes are smaller than semi plumes, with very less barbs at the tip of a fine shaft. These likely serve a sensory function in chickens, controlling vibrations and changes in pressure. A Filo plume is shown in figure 3.4 (e).

3.1.3 Particulate Element

Particulates are the fine micro or nano sized particles used in the composite development to uplift the properties of the homogeneous material. Here, the white semi crystalline micro sized powder prepared by treating the residue of Rohu fish under various laboratory methods is used as a particulate in the development of hybrid biocomposite. The prepared powder contains various elements as seen through Field Emission Scanning

Electron Micrography (FE-SEM) and XRD analysis has been discussed later. The systematic process of extraction of silica powder from fish is elaborated in the next session.

3.1.3.1 Extracted residue powder/white ash from Rohu fish

ERP is the white colored micro sized particle which was extracted from the Rohu fish. The various laboratory reactions were performed in the development of ERP. The developed ERP contains varying percentages of elements like calcium, sodium, and oxygen etc. in compound form which shows better compatibility with Epoxy**.** The composition in extracted powder can be seen through FE-SEM composition analysis which is discussed later.

The white powdered particulate was extracted from Rohu fish using systematic step by step processing as the silica is extracted from rice husk ash **(Mittal, 1997).** The sequential process is described below and the pectoral view of the complete process is shown in figure 3.6.

- Firstly, collect the waste Rohu fish fins, skin and shells in a beaker and wash it properly with the running water. Than dry the deposit in sun light for 30 min (optional).

- Take the residue in a metallic beaker and burn it in open atmosphere at around $200^{o}C$ till it completely burns (black colored and oily appearance). Open atmosphere is recommended as burning of fish deposite makes pungent and foul smell.

- Try to remove maximum amount of black oil from the burnt fish residue by absorbing sheet and filter paper to get dry black substantial (as much as possible) and keep it to dry for 48 hours approximately. (Black semi solid paste type appearance can be seen).

- Treat the oily ash mixture with thrice the weight of NaOH and heat it at $180^{0}C$ for 1 hour in closed oven. Use magnetic stirrer and stir the mixture at $180^{0}C$ at approximately 450 rpm for 2 hours.

Figure 3.6: Step by step processing performed during extraction of fish residue powder

- With the help of glass dropper add H_2SO_4 (sulphuric acid) drop wise and be alert as it makes accidental chances while hydrogen liberation takes place. Keep the sample for 5 hours at rest in atmospheric condition.
- Now heat the substance for 1 hour each in a high temperature furnace with the hit and trial method at various temperatures starting from 300^0C than 400^0C than 500^0C - 600^0C etc. till the desired appearance is visible and keep observing the color change.
- At around 720^0C you will observe the white crystalline solid similar to silica. Check at validate result via TGA/DTA, XRD and other analysis.
- By the composition detection (using FE-SEM) the clear view regarding the composition of extracted powder was recovered and it was found that apart from silica the extracted residue powder contains calcium, sodium, phosphorous, etc. in elemental and $CaCO_3$, SiO_2 etc. in compound form.

  The variation that occurred during the entire process was captured and is shown in figure 3.6.

3.1.4 Silica Gel Grease

The semi liquid, transparent, sticky grease is used to disassemble the final casting from the mould without causing any harm to the mould as well as the casting. It is referred to as the releasing agent between the mould and the casting. The thin layer of grease is applied on the inner surface of the mould before the casting material is filled in the mould. It separates the direct linking of steel sheet and the composite mixture. Therefore, it provides better surface finish to the casting.

3.2 Method of Casting

`Initially, we require CFF, epoxy resin (CY-230), hardener (HY-951), steel mould, thermometer, the convection oven, silica gel grease, NaOH solution, extracted silica powder, Weight measuring machine, measuring beakers and stirrer. The solution obtained by mixing chicken feather fiber in epoxy resin is kept in the oven at a temperature of 140°C for one hour as per the recommendation of **Singh and Gope, 2010**. The convection oven (temperature range 0-240°C) used for this purpose is shown in figure 3.7. The method for preparing different materials is given below.

Figure 3.7: Convection oven

3.2.1 Preparation of Fiber for Reinforcement

Fibers are prepared in order to convert it in a usable form as per the requirement. Here, we have focused on the chicken feathers as a fiber for reinforcement with epoxy resin matrix as it light weighted and easily available livestock waste. The frequent use of CFF helps us to develop cost effective composite material. Preparation of CFF helps us to overcome the hydrophobic nature of chicken feathers.

3.2.1.1 Washing or Alkali Treatment of Chicken Feathers

Chicken Feathers are cleaned and washed properly by Alkali treatment using NaOH (Sodium Hydroxide) solution. Chicken feather fibers were soaked in 5% concentration of NaOH with distilled water for 6-8 hours at 30°C and then thoroughly washed with running water (sieving method). It was then dried in the natural light for 24 hours **(Raghavendra *et al.*, 2012)**. The processing of feathers using alkali solution helps to remove dirt, impurity, stickiness, blood contamination and the oily part from the feathers. The washed feathers are separated and are kept for drying as shown in figure 3.8.

Figure 3.8: Chicken feathers kept for drying after alkali treatment

3.2.1.2 Sizing of Chicken Feathers

The dried Chicken feathers are cut into small fibers (less than 5mm) pieces one by one as shown in figure. The rashes and barbs are removed and only the barbicels and hairy part is used as fiber. The sharpened scissor is used for proper sizing of CFF. Ones the rashes are removed the feathers are further cut into smaller size. Figure 3.9 shows the image of the fibers used for composite development.

Figure 3.9: Final prepared chicken feather fiber for reinforcement

3.2.2 Preparation of Particulate

Various sized particulate is prepared as per the requirement and application in composite. Here, we have focused on micro sized powdered form of particulate in order to develop hybrid biocomposite. The systematic process of extracting white residue ash from Rohu fish is elaborated in section 3.1.3.1.

3.2.3 Preparation of Mould

For the ease of mounting and unmounting the casting, the permanent moulds were designed using thin GI steel Sheet as shown in figure 3.10. The dimension of the mould was kept as 200 x 100 x 15 (dimensions in mm). The Silica gel grease is applied on the inner layer of mould before pouring the fluid into it for the easy removal of the solidified casting. It provides better surface finish and reduces machining. Mould used in current research work is shown in figure 3.10.

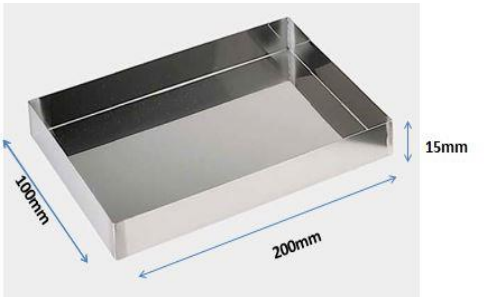


Figure 3.10: Steel mould used in casting

Calculation Involved in taking mass fraction in composite preparation.

Considered Density of Epoxy (ρ) = 1150 kg/m^3

Volume of Casting = 200*100*10

=2, 00, 000 mm^3

=2 X 10^5 X 10^{-9}

=2 X 10^{-4} m^3

Therefore,

Mass of molten material (i.e. liquid epoxy resin) = Volume*Density

= 2*10^{-4} * 1150

=0.23 kg

=230 gm.

The mass of hardener i.e. 10% of epoxy resin + mass of CFF + mass of fish residue powder is taken as extra material for grinding compensation and dimensional accuracy of the outer (upper) layer and accurate specimen preparation.

3.2.4 Preparation of Chicken Feather Fiber filled biocomposite

The process for casting of CFF filled epoxy resin based biocomposite initially requires the proper mixing of 230gms of Epoxy resin (CY-251) and the 2.53gms (1 wt% of epoxy resin + hardener) of prepared CFF in a glass beaker. The beaker is than kept in convection oven and the mixture is heated at the temperature of 140^0C for 1 hour. After one hour the beaker is placed in an open atmospheric condition till the temperature of the mixture (CFF + epoxy resin) reaches below 40^0C. Timely check the decrease in temperature using thermometer till it reaches 40^0C. Add 23gms (10 wt% of epoxy resin) of hardener (HY-951) in the beaker and thoroughly mix the entire mixture so that the hardener is properly mixed with CFF and epoxy resin.

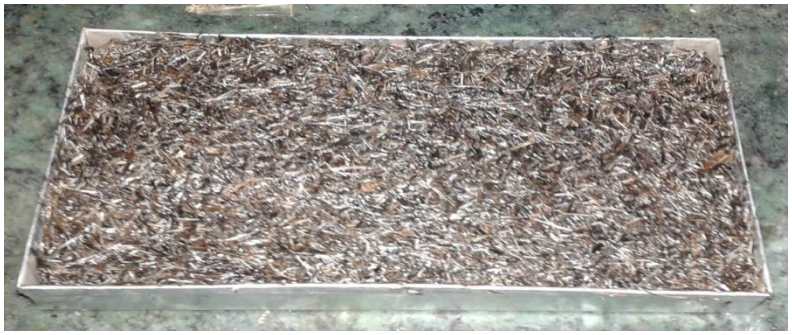


Figure 3.11: Prepared casting kept for solidification

Table 3.1 Design of experiments for CFF filled composite

Designation	Epoxy resin (grams)	Hardener (grams)	CFF (grams)
CONTROL	230	23	0.00
CFF 1	230	23	2.53
CFF 2	230	23	5.06
CFF 3	230	23	7.59
CFF 4	230	23	10.12
CFF 5	230	23	12.65
CFF 6	230	23	15.18
CFF 7	230	23	17.71

Pour the mixture in the silica greased layer steel mould and uniformly spread the casting using Hand lay-up technique for solidification as shown in figure 3.11. Keep the

casting undisturbed for minimum 48 hours before removing it from the mould. Repeat the same process with varying wt% of CFF in the mixture.

3.2.5 Preparation of extracted residue powder filled hybrid biocomposite

Similar to preparation of CFF based composite, in preparation of extracted residue powder filled biocomposite in 5wt% CFF (i.e. 11.50gm), all we need to do is the mixing of varying wt% of prepared powder in the mixture of CFF (11.50gm) and epoxy resin before convection heating. Weight measurement of ERP using digital weight measuring machine is shown in figure 3.12.

Table 3.2 Design of experiment for extracted fish powder filled hybrid composites

Designation	Epoxy resin (grams)	Hardener (grams)	CFF (grams)	Extracted Residue Powder (grams)
ESP 0	230	23	12.65	0.00
ESP 1	230	23	12.65	2.66
ESP 2	230	23	12.65	5.32
ESP 3	230	23	12.65	7.97
ESP 4	230	23	12.65	10.62
ESP 5	230	23	12.65	13.28
ESP 6	230	23	12.65	15.93

All other processing is same as elaborated in previous section 3.2.4. The compositions of different type of castings developed for characterization are listed in table 3.2.

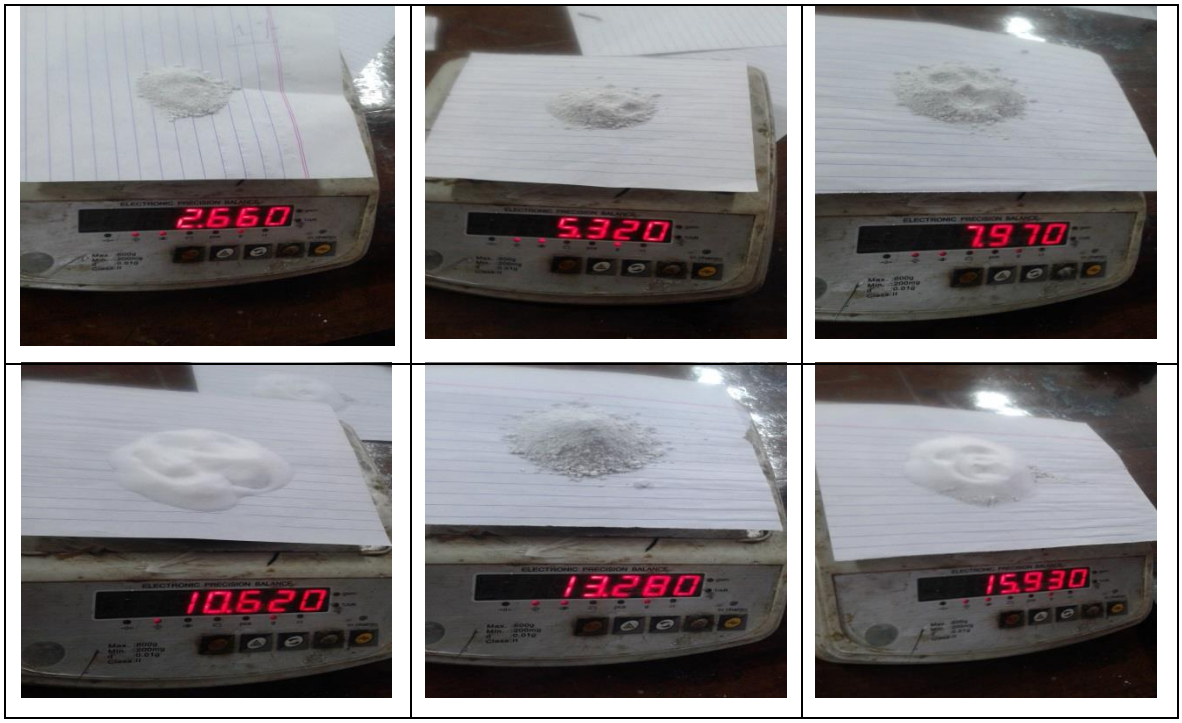


Figure 3.12: Measurement of wt% of extracted residue powder

3.3 Mechanical Tests

Different mechanical tests were performed in order to characterize the composite. The composite is designed and fabricated as per the ISO standards. The efforts are made in order to develop the maximum accuracy in terms of dimensions, variations in compositions, testing and adopted methodology.

3.3.1 Tensile Test

The tensile strength of CFF filled epoxy resin composite is experimentally determined using 25kN servo controlled universal testing machine (Manufactured by ASI Sales Pvt. Ltd., Model AMT-SC) at fixed cross head speed of 1mm/min under displacement control mode. An ISO standard ISO 527-2/1B/50 is followed in specimen preparation for tensile testing. The process of tensile testing is well explained in section B.1 (Appendix B).The tensile test is performed to determine the Stress/Strain relation, Young's Modulus, Ultimate tensile Strength, percentage elongation, proportional limits, yield point and other tensile properties in a graphical manner. UTM used in the current research work is shown in figure 3.13 (a). The specimen dimension is shown in figure 3.13 (c). All the

tests were conducted at room temperature (i.e. 30^0C as recorded) with relative humidity (RH) 50%. The results obtained from tensile test are shown in Section 4.3.1 and 4.5.1 (for hybrid composite).

Figure 3.13 (a): Universal Testing Machine

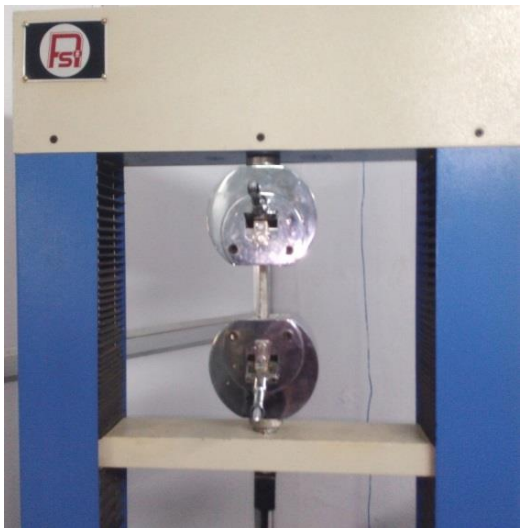


Figure 3.13 (b): Clamping of tensile test specimen on UTM

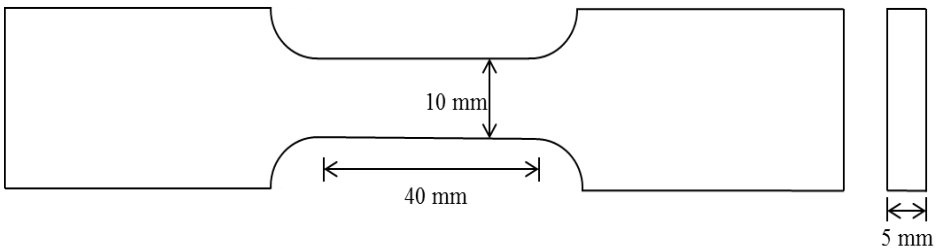


Figure 3.13 (c) Specimen dimensions based on ISO Standard for tensile testing of polymeric specimen

Figure 3.13 (d): Actual test specimen for tensile testing

3.3.2 Compression Test

The compressive properties of CFF filled epoxy resin composite is experimentally determined using 25kN servo controlled universal testing machine (AMT-SC, 2008 model, manufactured by ASI Sales Pvt. Ltd.) at fixed cross head speed of 1mm/min under displacement control mode. An ISO standard ISO 604: 2002 (E) is followed for compression testing of polymeric composite and specimen dimension is based on ISO-1708, 1960. The procedure of compression testing is well explained in section B.2 (Appendix B). Compression in just reverse of tensile loading in terms of loading. Here the cylindrical specimen with proper standard dimensions is placed at the center of the compression fixtures of UTM machine, and then the load is applied with computer control system.

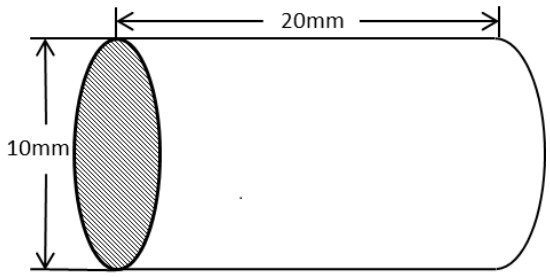


Figure 3.14 (a): Specimen configuration

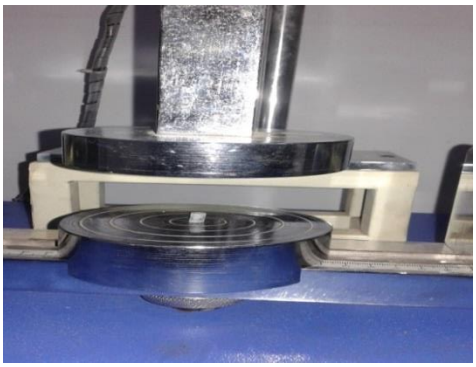


Figure 3.14 (b): Fixture for compression test

It helps to characterize various mechanical properties like variation in stress/strain, ultimate compressive strength, yield points, compressive young's modulus, strain etc. Specimen configuration is shown in figure 3.14 (a). The fixture for compression test is shown in figure 3.14 (b). All the results obtained are discussed in Section 4.3.2 and 4.5.2 (for hybrid composite).

3.3.3 Impact Test

The impact strength of any material is determined by the Izod or Charpy tests. In the current analysis we are focusing on the Izod Impact test. Impact testing machine manufactured by Advance Equipment, Maharashtra, India. (Model: AE.ICT.O, 2014) is used for the characterization of impact properties. It has 50Hz frequency, 230V supply

voltage and takes 120W load. Impact test necessitates notched specimen to determine its energy required to fracture. In this the specimen is placed as the cantilever beam. The specimen is dimensioned based on ISO standard ISO 180:2000 used for plastic components. The procedure of impact testing is well explained in section B.3 (Appendix B).The digital multi loading Izod testing machine is used in the experimentation. Firstly, we input the dimensions of specimen, type of hammer and other parameters on the machine through the keyboard available on the test machine. Then the hammer is released from the predetermined angle on the specimen to get the impact strength, impact energy, impact strength per unit thickness on the digital screen situated on the machine. The Impact testing machine used here is shown in figure 3.15 (c). The sample dimensions and actual prepared specimen is shown in figure 3.15 (a) and figure 3.15 (b) respectively. Also the graphical representations of the output results are shown in figure 4.6 and figure 4.8 (for hybrid biocomposite). The atmospheric temperature and relative humidity (RH) at the time of test were 28^0C and 49% respectively (as recorded).

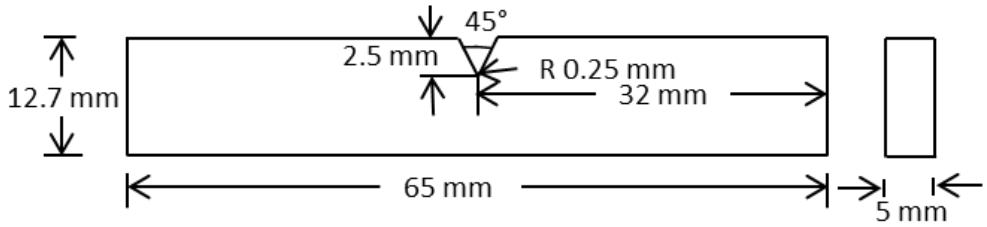


Figure 3.15 (a): Dimensions for impact testing specimen

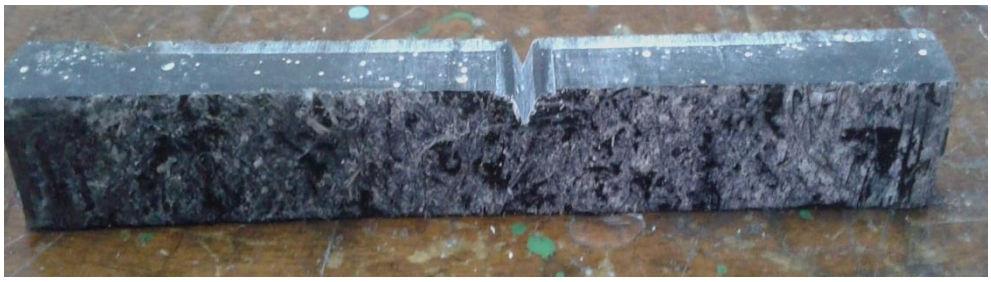


Figure 3.15 (b): Actual specimen for impact testing

Figure 3.15 (c): Impact testing machine

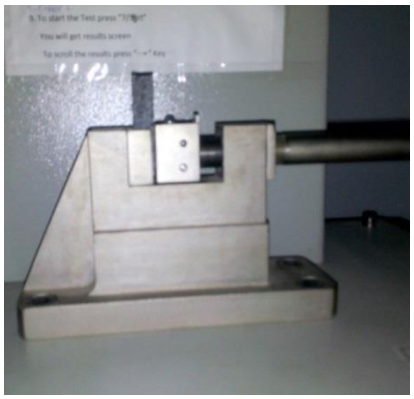


Figure 3.15 (d): Clamping of notched impact specimen

3.3.4 Rockwell Hardness Test

Hardness is the surface property that provides resistance to scratching. The digital hardness testing machine is used to get the Rockwell hardness value of the prepared composites. The scale L, with ball type indenter of size ¼" (6.35mm) is used for the testing. The Rockwell hardness test is most preferred hardness testing method as it provides direct reading on the digital screen and no calculation is needed. The procedure of hardness testing is well explained in section B.4 (Appendix B).The hardness value at various positions is taken and then the average of reading is considered as the final recorded value measured in HRL. The atmospheric temperature and relative humidity at the test Centre was 27^0C and 46% respectively. The test results are discussed in Chapter 4. Figure 3.16 shows the method of placing the hardness testing specimen on the fixture for hardness test.

Figure 3.16: Placing of finished specimen for hardness testing

3.3.5 Flexural Test

To identify the nature of specimen during bending we perform 3-point bending flexural test. The flexural strength of the prepared composite is experimentally determined using 25kN servo controlled universal testing machine **(AMT-SC, 2008 model, Manufactured by ASI Sales Pvt. Ltd.)** at fixed cross head speed of 1mm/min under displacement control mode. The flexural properties are determined using ISO 178: 2001 standard. The process of flexural testing is well explained in section B.5 (Appendix B).The bending characteristic like flexural strength, flexural strain and flexural modulus are determined by flexural test using the load v/s displacement curve generated during the test. The atmospheric temperature and relative humidity during the test were 33^0C and 50%

respectively. Figure 3.17 (b) shows the positioning of flexural specimen on the UTM. The flexural stress (σ_f), flexural modulus (E_f) and flexural strain (ε_f) for rectangular cross section are determined by the analytical formula:

$$\sigma_f = \frac{3PL}{2bd^2} \quad \text{... (3.1)}$$

$$E_f = \frac{L^3 m}{4bd^3} \quad \text{... (3.2)}$$

$$\varepsilon_f = \frac{6Dd}{L^2} \quad \text{... (3.3)}$$

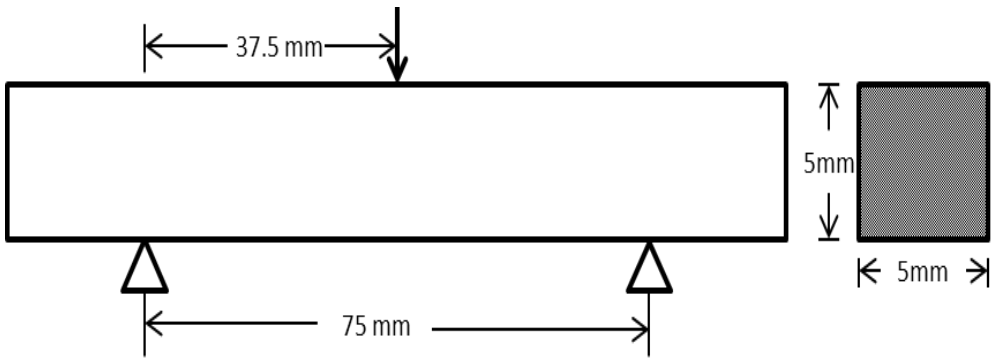


Figure 3.17 (a): Schematic diagram of 3-point flexural test

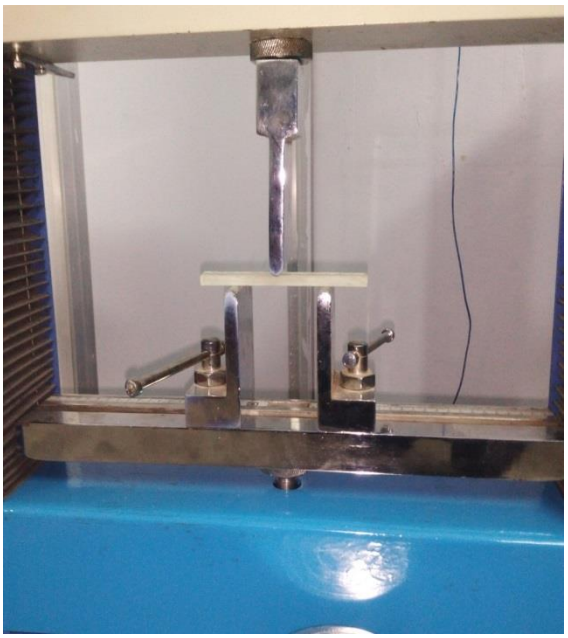


Figure 3.17 (b): Positioning of flexural specimen on the UTM

3.4 Morphology

In the current research morphological analysis of the hybrid composite material is done using various advance techniques like Scanning Electron Micrograph (SEM) , Field Emission Scanning Electron Micrograph, X-ray Powder Diffraction (XRD) technique etc. Also the thermal analysis is carried out using Thermo Gravimetric Analysis (TGA), Differential Thermal Analysis (DTA). The detailed information about these advance methods of micro and nano level analysis of composites are discussed below.

3.4.1 Scanning Electron Microscopy (SEM)

SEM is an electron micrograph that provides pectoral morphological image of the micro sized irregularity at various levels of magnifications with the focused beam of electrons. It provides high resolutions to captured images. SEM provides the clear cut view about the fractured surface occurring due to various mechanical tests performed on the prepared specimens. The images were obtained through micrographic investigation with LEO435V6. For SEM analysis the tensile fractured specimen with the fractured surface on the top was cut in small square pieces (approx. 5mm). It was first gold plated to avoid the artifacts associated with the sample charging and then kept in SEM machine where the beams of electron fall over it. Maximum 8 samples can be placed in the chamber at a time. Image generation is suitable after 15 minutes of keeping the specimen in the chamber as it generates vaccum atmosphere around the samples inside SEM chamber in 15 minutes. The acceleration voltage was kept at 15kV. In the current work, SEM images of tensile fractured surface of various samples (with different wt% of Particulate in 5wt% CFF) is taken at various magnifications (i.e. 100X, 500X, 1000X, 2000X and 5000X). The micrographs obtained are shown in figure 4.14 (a-f). The test was conducted at IIC Department, IIT Roorkee.

3.4.2 Field Emission Scanning Electron Microscope (FESEM)

Field Emission Scanning Electron Microscope (FESEM) is a microscope that works with electron (i.e. negatively charged ions). It provides topographical as well as elemental information about the entire or fractioned object under investigation in various magnifications ranging from 2X to 3,00,000X and with unlimited virtual depth of cut. As compared to SEM it is much more superior and provides clearer, least distorted images with spatial resolution down to 1 ½ nanometers. FESEM can also be used as a tool that

can provide information about nano size particle. Therefore, through FESEM we can get the elaborative information about the structure, composition, morphology, uniformity determination and small contamination in the material.

3.4.3 X-Ray Powder Diffraction (XRD)

X-Ray Powder Diffraction (XRD) technique can be defined as a fast analytical technique used for phase identification of the crystalline material and it is used to get the crystalline information about the unit cell of the material. It is used to identify the crystallographic density and the crystal structure of the unknown crystalline solid. It diagnoses the lattice imperfections, defects and provides the best failure analysis of the structure occurring at microscopic level. XRD helps to characterize the compound through varying diffraction pattern. The effect occurs due to incident of monochromatic x-ray beam on the specimen.

It follows Bragg's equation

I.e. $2d\sin Q = n\lambda$ … (3.4)

Where, d= Interplaner Spacing

λ = wave length

Q = diffraction angle

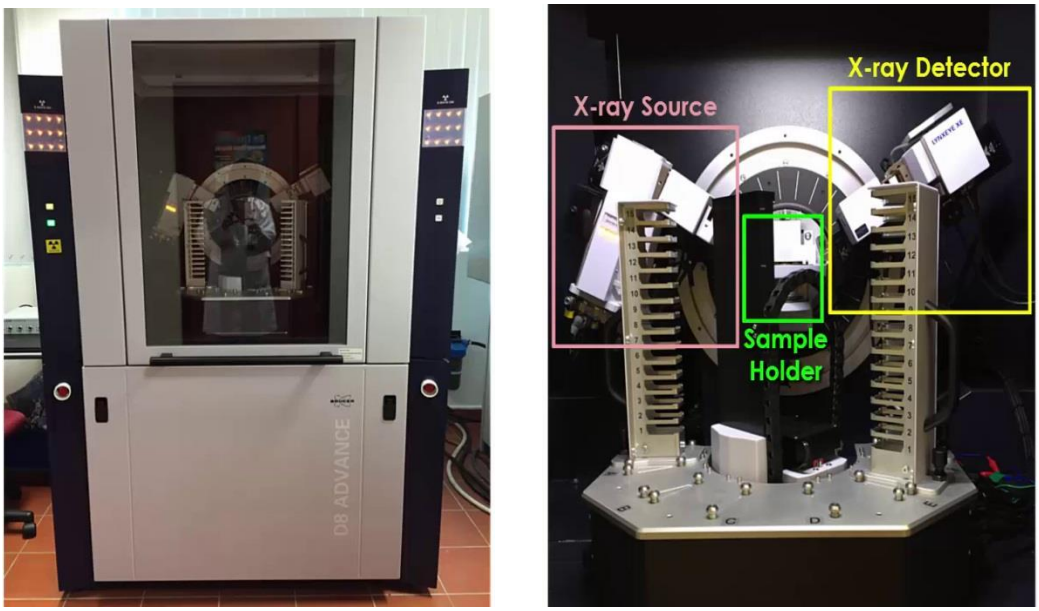


Figure 3.18: XRD machine

Bruker D8 Advance XRD machine shown in figure 3.18 is used for the XRD analysis. During the analysis the count v/s 2 theta curve generated. The results gives Interplaner distance or gallery spacing (d), value of 2 theta (legend or thread angle) that corresponds to d value, intensity percentage. For the current research work, the XRD analysis using powdered form of various samples is performed at IIC, IIT Roorkee. The results are graphically represented in figures 4.16 (a-f).

3.5 Density

Density can be defined as an intensive property of the material which is termed as the ratio of mass of the body to the volume of the body.

I.e. $$Density = \frac{Mass\ of\ the\ Body}{VOlume\ of\ the\ Body} \quad \ldots (3.5)$$

Its SI unit is kg/m^3

It is denoted by the symbol "ρ" (pronounced as roh)

Through the existing literature reviews it was noticed that the approximate density of Epoxy resin (CY-230) with hardener (HY-951) is $1150kg/m^3$, ash powder is 2130 kg/m^3 and Chicken feather is $800kg/m^3$.

The theoretical density of composite samples in terms of weight fraction can be obtained easily by the following equations suggested by **Agarwal, B.D. and Broutman L. J., 1990.**

$$\rho_{ct} = \frac{1}{\left(\frac{W_f}{\rho_f}\right) + \left(\frac{W_m}{\rho_m}\right)} \quad (3.6)$$

$$\rho_{hct} = \frac{1}{\left(\frac{W_f}{\rho_f}\right) + \left(\frac{W_m}{\rho_m}\right) + \left(\frac{W_p}{\rho_p}\right)} \quad (3.7)$$

where, *W* and ρ symbolizes the weight fraction and the density respectively. The suffix *ct*, *hct*, *f*, *m* and *p* stands for the composite, hybrid composite, fiber, matrix and particulate respectively.

The actual/experimental density (ρ_{ce} and ρ_{hce}) of the composite or hybrid composite material, however, can be firmed experimentally by the uncomplicated toluene immersion technique. The volume fraction of voids (V_v) for CFF filled composite and hybrid composite (V_{hv}) is calculated by the help of the subsequent equations 3.8 and 3.9 respectively:

$$V_{\upsilon} = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \tag{3.8}$$

$$V_{h\upsilon} = \frac{\rho_{hct} - \rho_{hce}}{\rho_{hct}} \tag{3.9}$$

3.6 Water Absorption and Thickness Swelling Test

The design of material is based on the type of application it has to perform. Mostly, the composite materials are subjected to different loading conditions and environmental factors such as intensity of loading, magnitude of force applied, heating effect, cryogenic environment, flow velocity, humidity, etc. So it is necessary to study the effect of environmental change on the material developed so that it can be used in various applications.

Figure 3.19: Experimental setup for water absorption and thickness swelling test

The water absorption and thickness swelling test helps us to diagnose the moisture socking intensity of the composite developed. It is necessary to diagnose the rigidity, molecular compactness, adhesion between the matrix and the particles (fiber or particulate) in composite material and the surface strength of the material. The greater is the water absorption capacity of the material the more is the amount of pores/voids in the material and lesser is the attraction bonding between the combining molecules and therefore lesser is the strength of the material. The water with pH 7.1 was used to perform the water absorption test at 33.2^{0}C ambient temperature and 51% RH. For the water absorption test, the weight was measured using electronic weight measuring machine (least count 0.01gm) in every 6 hours for 48 hours. And for thickness swelling test, the thickness was measured using Vernier calliper (least count 0.05mm). Before reimmersing the sample in water wipe it properly to remove water from the surface using tissue paper. The difference in weight and thickness is recorded and thus the conclusion is made.

Water absorption, WA is calculated by:

$$WA(\%) = \frac{(W_2 - W_1)}{W_1} * 100 \quad \ldots (3.11)$$

W_1 = initial weight of specimen, gm.

W_2 = specimen weight after N hours of water soaking, gm.

Similarly,

Thickness Swelling, TS is calculated by:

$$TS(\%) = \frac{(T_2 - T_1)}{T_1} * 100 \quad \ldots (3.12)$$

T_1= Initial thickness of specimen, mm.

T_2= Specimen thickness after N hours of water socking, mm.

3.7 Thermal Analysis

Thermal analysis is very indispensable investigating tool for determining the relative change in properties of material with change in temperature. In thermal analysis we generally apply TGA and DTA analysis (elaborated below is section 3.7.1 and 3.7.2). For the current research thermal analysis TG analyzer EXSTAR TG/DTA 6300 is used at IIT Roorkee. The DTA, DTG and TG curve corresponding to change in time for various

samples are graphically represented in figures 4.13 (a-f). The analysis is performed in airy atmosphere with flow rate of 200mL/min and temperature rate of 10^0C/ min from 0^0C to 1000^0C.

3.7.1 Thermo Gravimetric Analysis (TGA)

Thermo Gravimetric Analysis is a thermal analysis method that provides information about the change in physical and chemical properties of the material as a function of temperature (with constant heat transfer), or as a function of time (with constant mass flow or temperature i.e. Isothermal mode) under control atmosphere. It is also represented in graph as Derivative Thermo Gravimetric curve (DTG curve). It provides information about the decomposition and rate of decomposition of the material. TGA provides selective characteristics that exhibit either mass loss, decompositions or loss of volatiles (moisture). TGA analysis is performed by gradually raising the temperature of the sample in the furnace. The mass loss is observed using analytical weight balance that is situated outside the furnace. Later the decomposition rate is plotted against time change or temperature change. It provides complete knowledge about the physical phenomenon like vaporization, sublimation, absorption, adsorption and desorption. Similarly it can provide information about chemical changes like decomposition, oxidation or reduction etc.

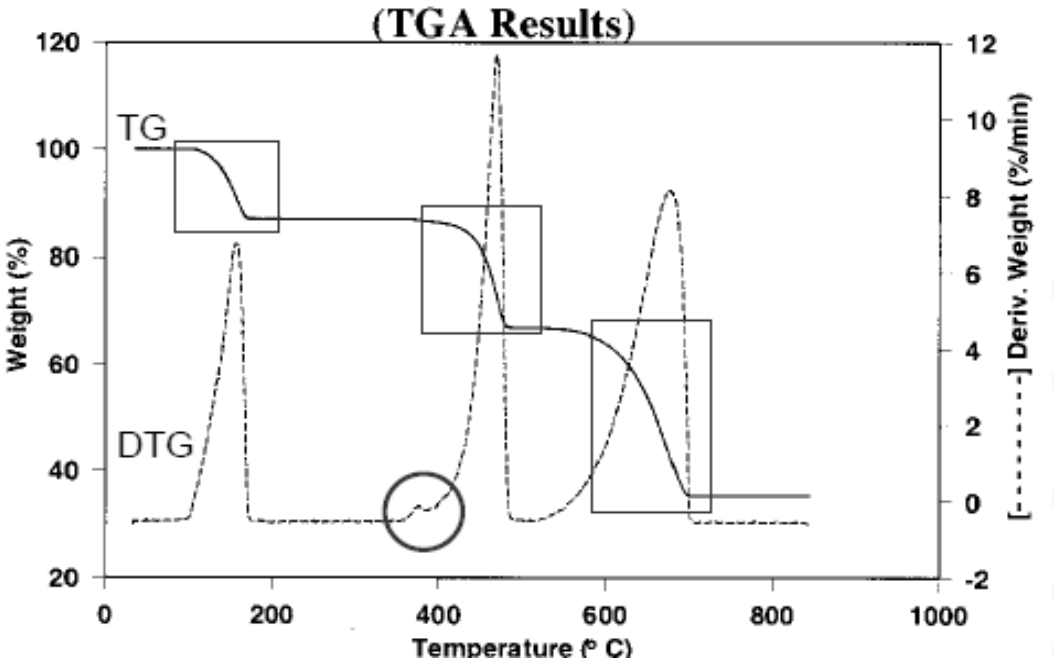


Figure 3.20: Graph representing TGA & DTA curve

3.7.2 Differential Thermal Analysis (DTA)

Differential Thermal Analysis (DTA) is a thermo analytic technique for thermal characterization of a sample. In DTA, the material under investigation and an inert reference are made to undergo under identical thermal cycles with fixed rate of deformation and monitoring the temperature difference between sample and reference. The change in temperature is then plotted against time. The relative difference either endothermic or exothermic is detected between the sample and the reference. The DTA curve provides information like glass transition temperature, crystallization temperature, melting temperature and sublimation temperature and also the curve peak depicts the enthalpy change during the deformation. The peaks in graph shows the energy dissipation or energy absorption means whether the reaction in the material is exothermic (energy dissipation shown by EX) or endothermic (energy absorption shown by EN). The area under the endotherm or exotherm is related to the enthalpy of the thermal event, ΔH.

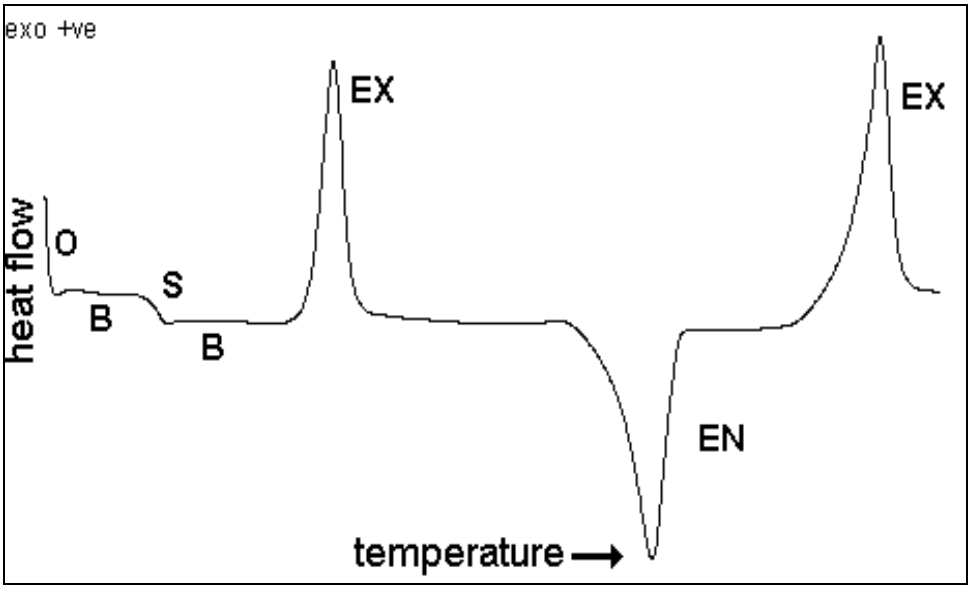


Figure 3.21: Example of DTA curve

Results

&

Discussions

Chapter.4 RESULTS AND DISCUSSION

4.1 Results and Discussion

The systematic fabrication and processing of various composite's samples with varying weight percentages of CFF in epoxy resin had led us to achieve the finest CFF – Epoxy Resin composition which are based on the results of mechanical tests performed at the Dynamics Lab, Collage of Technology, G. B. Pant University of Agriculture and Technology, Pantnagar.

Various mechanical tests like tensile test, compression test, impact test and hardness test are performed on the CFF based epoxy composite to achieve the most feasible mixing ratio. Later, based on the optimum results achieved **with maximum hardness and impact strength composition**, the hybrid composite with extracted residue powder from fish as particulate is fabricated and characterized. Different mechanical, physical, thermal and morphological tests were finally performed.

The results and discussions of various hybrid and non-hybrid composite is graphically pictured below and are efficiently explained with valid reasons for all the trends and exceptions.

4.2 Physical Properties

In order to diagnose the physical appearance, weight density, water absorption, thickness swelling etc. of the developed composite various tests were performed.

4.2.1 Appearance

Exterior appearance of Chicken Feather Fiber based Epoxy resin composite is multicolored (color based on color of chicken feathers), rough surfaced, semi transparent and hard plastic type. Its transparency decreases (i.e. becomes more and more opaque) with increasing weight percentage of CFF in epoxy resin. It requires surface machining for smoother surface. At low fiber percentage in epoxy resin the casting appears as the brittle glass material. The attractive appearance, use of livestock waste in the formation of chicken feathers filled biomaterial and the cost effective manufacturing has compelled the researchers to focus on the advancement of such materials.

4.2.2 Density

Elaborative portrayal of density measurement as explained in section 3.5.1 has helped us to get the systematic trend of experimental and theoretical density along with volume void fraction (as listed in table 4.1) with increasing wt% of CFF in epoxy resin (figure 4.1(a)). Also the variation in density and volume void fraction in hybrid composite (as listed in table 4.2) with increasing wt% of extracted residue powder in the optimum composition of biocomposite i.e. 5 wt% CFF + epoxy resin + 10 wt% hardener (figure 4.1(b)).

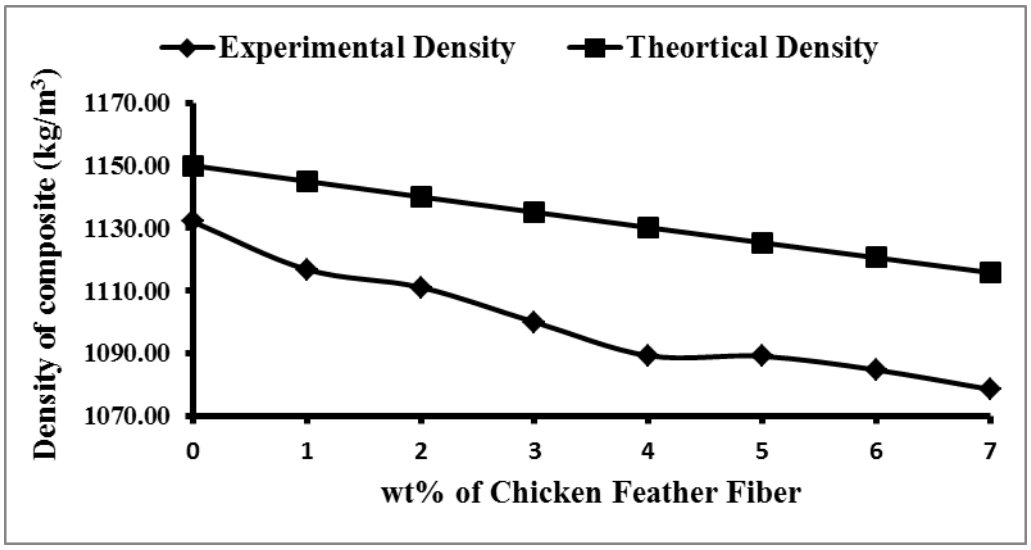


Figure 4.1 (a): Effect of wt% of CFF on density of composite

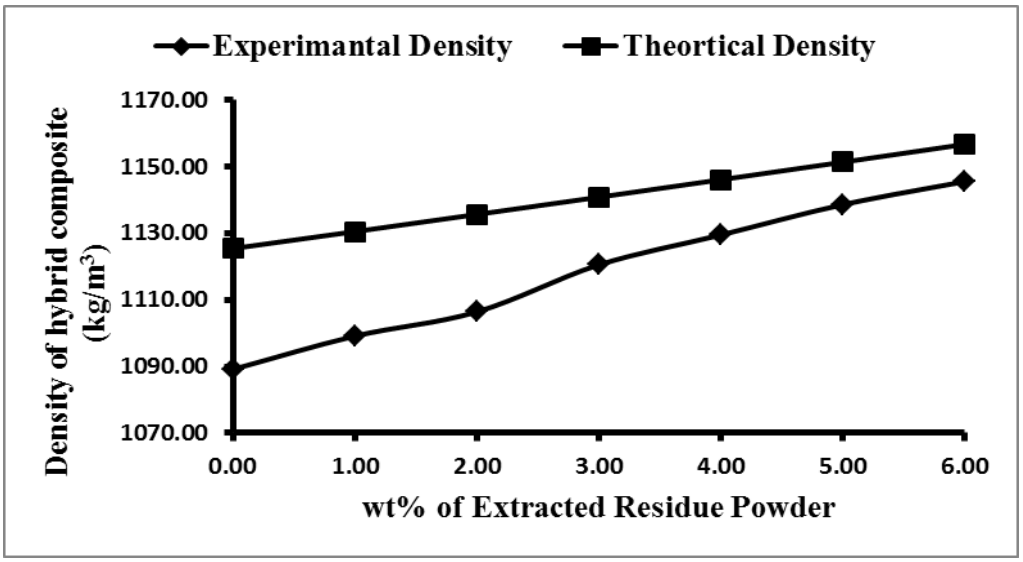


Figure 4.1 (b): Effect of wt% of extracted residue powder (in 5 wt% CFF) on density of composite

The theoretical density was calculated using equation 3.6 and 3.7 and the volume void fraction was obtained using equation 3.8 and 3.9 for CFF based composite and ERP filled hybrid composite respectively. The measure of the difference between the actual/experimental density and the theoretical density of the prepared samples gives the volume void fraction which is shown in figure 4.1(c).

Experiments showed that the neat epoxy has the weight density of 1132.08 kg/m^3 i.e. 1.56% less than the theoretical density (1150 kg/m^3). From figure 4.1 (a) it can be seen that the addition of CFF in epoxy resin decreases the weight density of the prepared composite as chicken feather is lighter in weight (density = 800 kg/m^3 approx.) as compared to pure epoxy. Also the decrease of 3.79% in density is recorded from 0 (i.e. 1132.08 kg/m^3) to 5 wt% (i.e. 10893.12 kg/m^3) of CFF in fabricated composite. The density at 7 wt% of CFF in epoxy resin is 1078.51 kg/m^3. Almost 5.19% decrease in density was recorded during the total composites development from 0 to 7 wt% of CFF. The main reason for the linear decrease in the density was due to commutatively adding of CFF in epoxy that decreases the specific weight of the prepared composite.

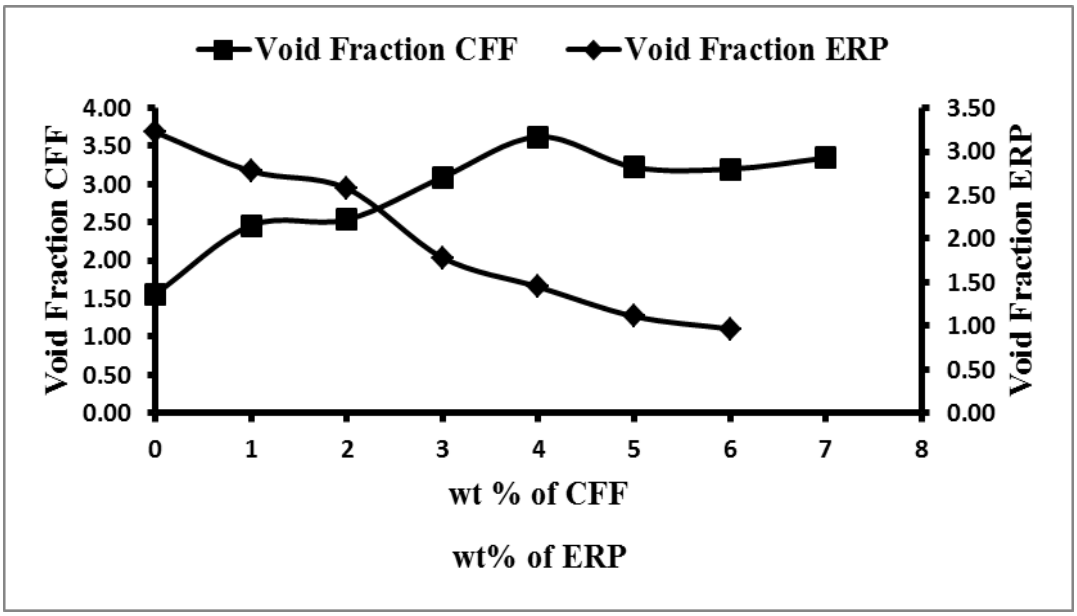


Figure 4.1 (c): Effect of varying wt% of CFF/ERP on percentage of volume void fraction

Table 4.1 (a): Density and volume void fraction percentage for various composition of CFF - epoxy resin composite

Composition	Experimental Density (kg/m^3)	Theoretical Density (kg/m^3)	Volume Void Fraction (%)
CFF 0	1132.08	1150	1.56
CFF 1	1116.82	1144.991	2.46
CFF 2	1111.11	1140.025	2.54
CFF 3	1100.00	1135.102	3.09
CFF 4	1089.29	1130.221	3.62
CFF 5	1089.12	1125.382	3.22
CFF 6	1084.75	1120.585	3.20
CFF 7	1078.51	1115.828	3.34

Table 4.1(b): Density and volume void fraction percentage for various compositions of ERP and 5 wt% CFF filled epoxy resin hybrid composite

Composition	Experimental Density (kg/m^3)	Theoretical Density (kg/m^3)	Volume Void Fraction (%)
ERP 0	1089.12	1125.38	3.22
ERP 1	1099.10	1130.47	2.78
ERP 2	1106.36	1135.61	2.58
ERP 3	1120.46	1140.79	1.78
ERP 4	1129.44	1146.02	1.45
ERP 5	1138.53	1151.30	1.11
ERP 6	1145.45	1156.63	0.97

Further in 5 wt% of CFF (5 wt% of CFF is considered as optimum composition for fabricating hybrid composite as discussed later in section 4.4) varying wt% of extracted residue powder was added and it was interesting to note that the weight density of hybrid composite (figure 4.1(b)) was continuously rising as the wt% of ERP was increased. It was 1089.12 kg/m^3 at 0 wt% of ERP and raises to 1145.45 kg/m^3 at 6 wt% of ERP in composite. The rise of 5.17 % is monitored during the experimentation from 0 to 6 wt%.

The rise in density may be due to the upright attraction bonding between the matrix and the particles at 3 wt% of ERP in 5 wt% CFF filled composite which can be clearly seen in SEM image shown in figure 4.14 (c).The decrease in volume void fraction with inclusion of ERP as shown in figure 4.1 (c) depicts the improved strength and adhesion bonding in the hybrid composite.

4.2.3 Water Absorption and Thickness Swelling Test

Precise readings of weight change and thickness swelling was recorded at equal interval of 6 hours for 48 hours continuously. Proper systematic method was followed for the testing. The efforts were made to achieve exact readings through weight measuring machine and Vernier caliper so that the manual error is decreased. Using equation (3.7) and (3.8) the percentage of water absorption and thickness swelling was calculated. The graphical representation of water absorption and thickness swelling is shown in figure 4.2(a-d) and figure 4.3(a-d) respectively.

4.2.3.1 Water Absorption Test

After the continuous progression for two days, the data recorded outlined the curve shown in figure 4.2 (a-d). Figure 4.2 (a & b) shows the results for water absorption with varying wt% of CFF. The results revealed that the water absorption was rising in the beginning with the increasing CFF wt% till 4% of CFF and then decreases considerably. The percentage of water absorption was maximum (i.e. 1.49%) at 4 wt% CFF. The rise in weight of the sample may be due to the filling of water in the free space and void that occurs due to increase in large amount of feather in the matrix. Also the water soaking takes place at the gap between the fiber and the matrix or between the randomly oriented fibers. The sudden declination of water absorption % at 5 wt% of CFF may be because of the improved compactness and fiber matrix bonding.

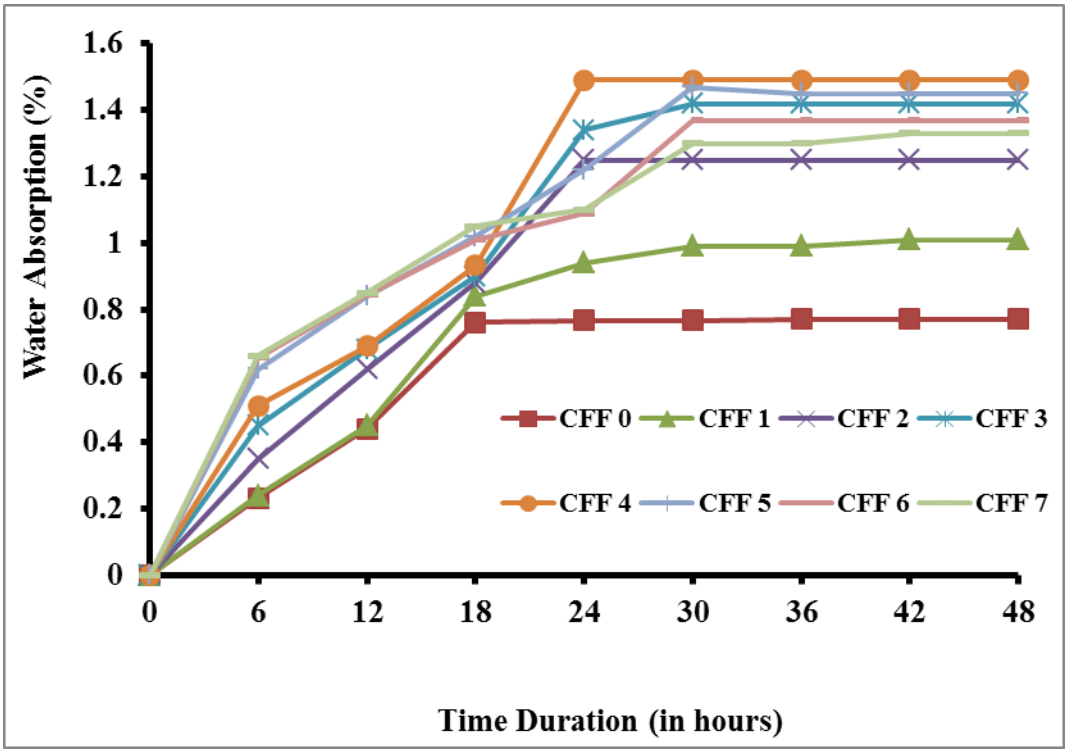


Figure 4.2 (a): Variation in water absorption % with time for CFF filled composites

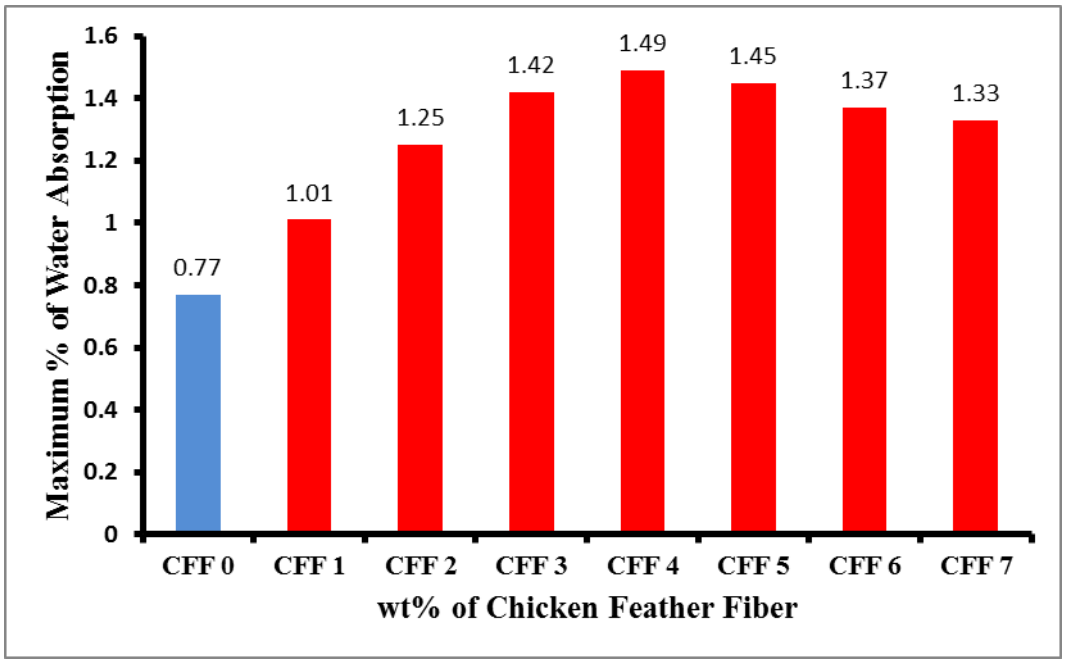


Figure 4.2 (b): Effect of wt% of CFF on maximum water absorption

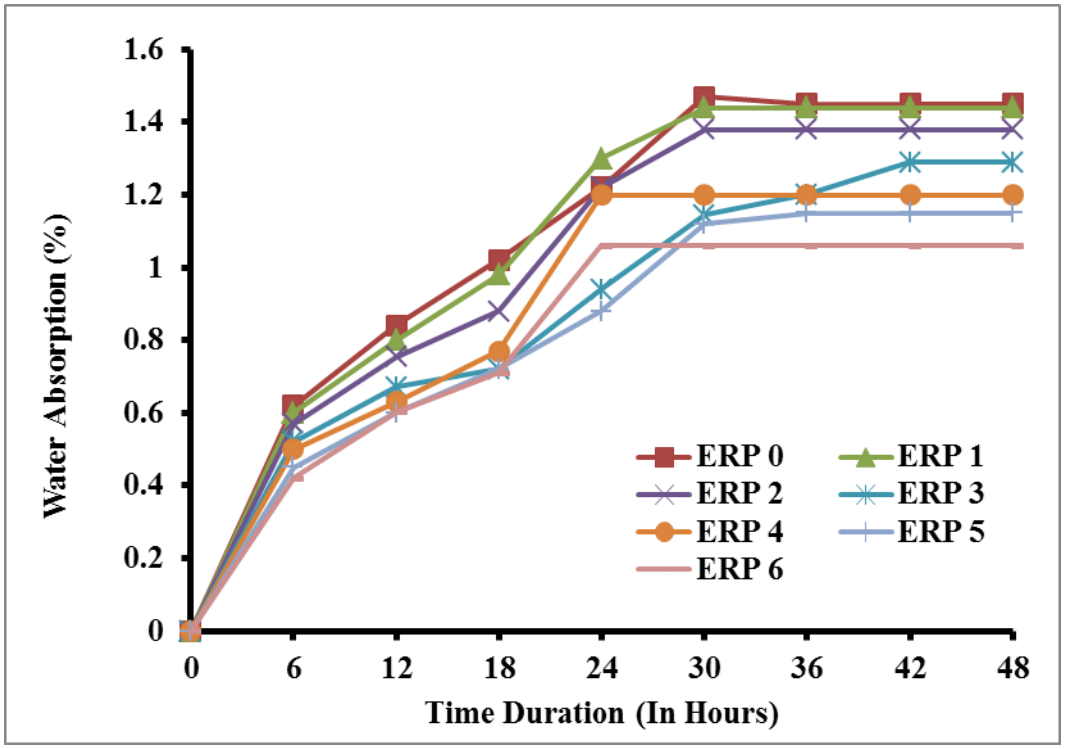


Figure 4.2 (c): Variation in water absorption % with time for ERP filled composites

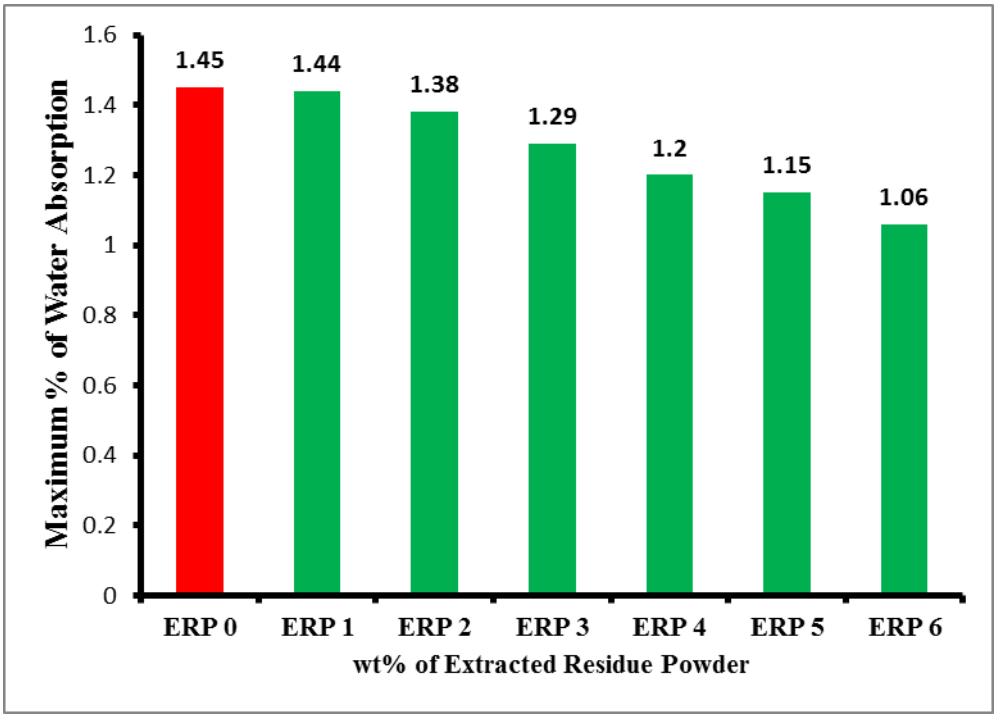


Figure 4.2 (d): Effect of wt% of ERP on Maximum Water Absorption

It was interesting to observe that by the addition of ERP in 5 wt% of CFF the amount of water absorption was continuously declining as shown in figure 4.2 (c) and (d). The decrease of 26.89% was observed between the 0 wt% ERP (1.45% water absorbed) and 6 wt% ERP (1.06% water absorbed) based hybrid composite. The falling of percentage of water absorption may be due to void filling by micro sized particulate and better binding of matrix with extracted residue powder which in turns improved the mechanical properties of the hybrid composite.

4.2.3.2 Thickness Swelling Test

Along with water absorption the variation in thickness of the samples were also recorded for 48 hours in the gap of 6 hours duration. The results obtained from the recorded data are pictured graphically in figure 4.3 (a-d). Figure 4.3 (a) and (b) shows the thickness swelling characteristics of CFF based composite which depicts that the percentage of thickness swelling increases significantly with the increasing % of CFF in epoxy based composite.

After 48 hours, it is 0.45% for epoxy and 0.66% for 7 wt% CFF filled composite. The rise in thickness can be due to the soaking of water by the composite mixture and water filling in the voids present in the sample. Similarly figure 4.3 (c & d) shows results for ERP filled hybrid composite with 5 wt% CFF (fiber wt% is kept constant). The rising change in the wt% of ERP decreases the % of thickness swelling. The high density particulate present on the outer surface of the sample restricts the movement of molecules and thus controls dimensional stability. Therefore adding particulate in the fiber based composite helps to attain better surface properties and dimensional tolerances.

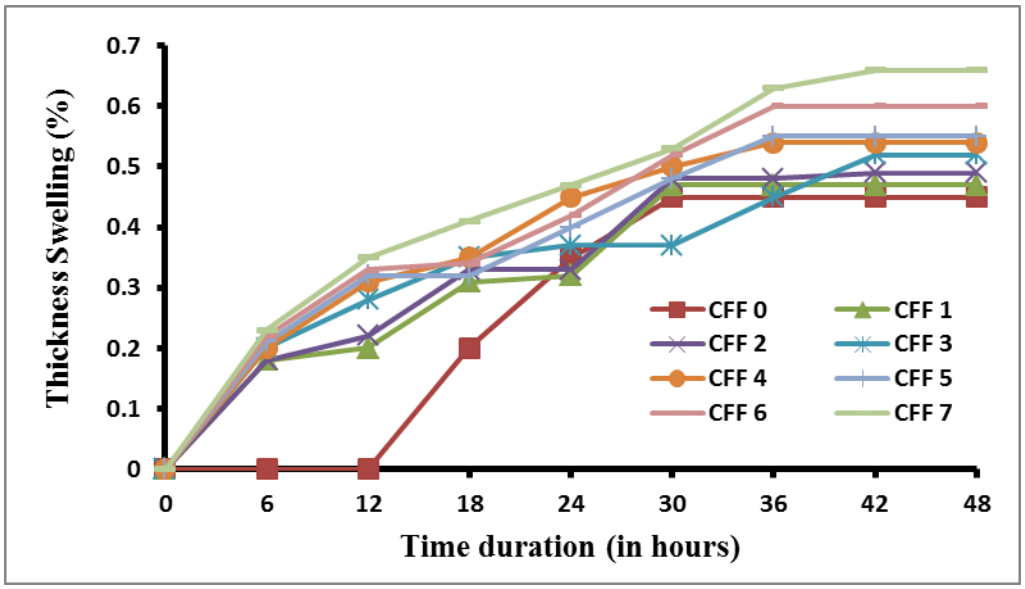


Figure 4.3 (a): Variation in thickness swelling % with time for CFF filled composites

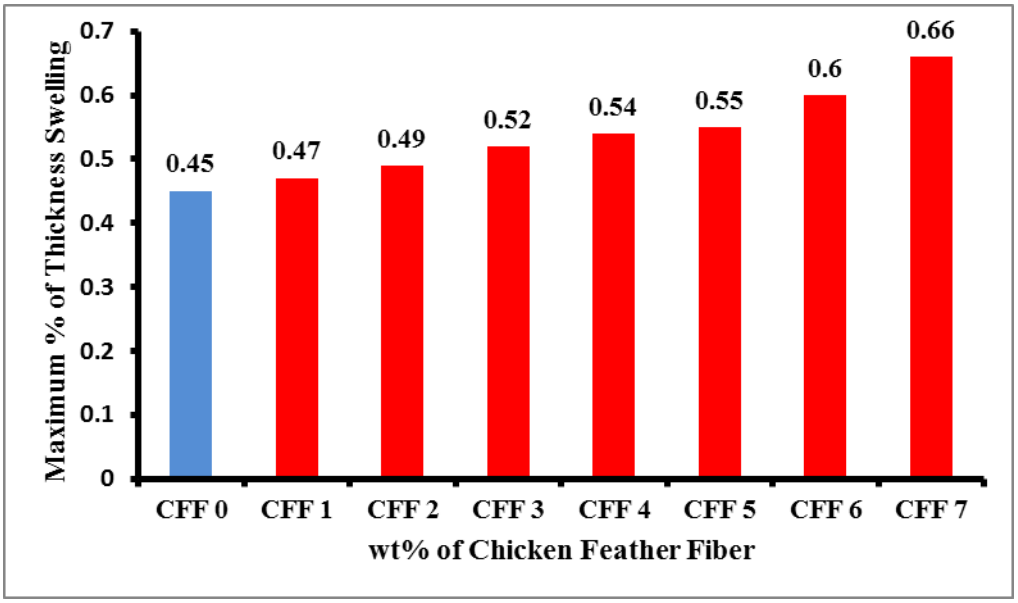


Figure 4.3 (b): Effect of wt% of CFF on maximum thickness swelling

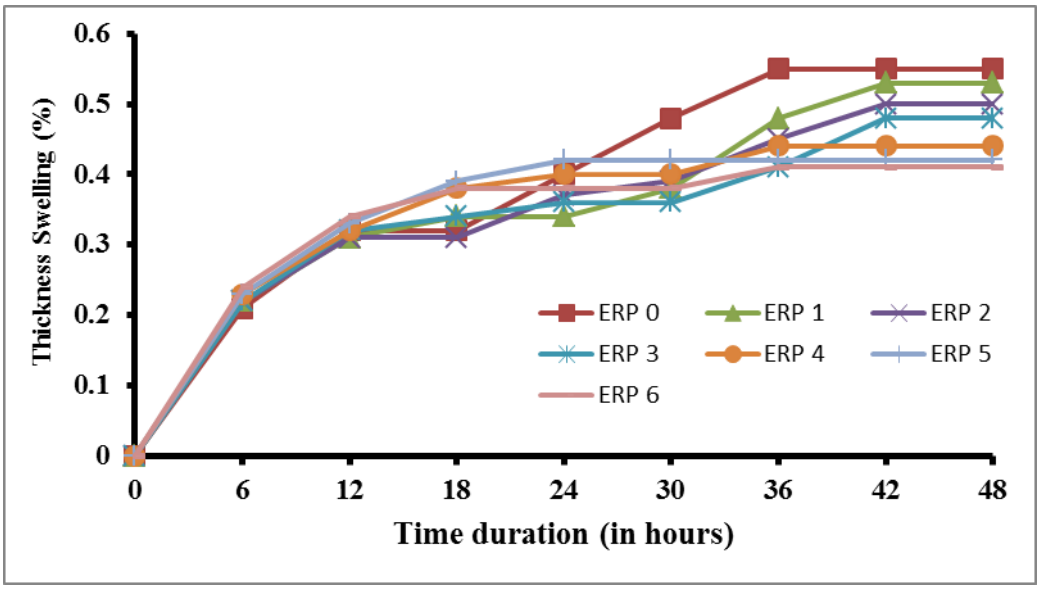


Figure 4.3 (c): Variation in thickness swelling % with time for ERP filled hybrid composites

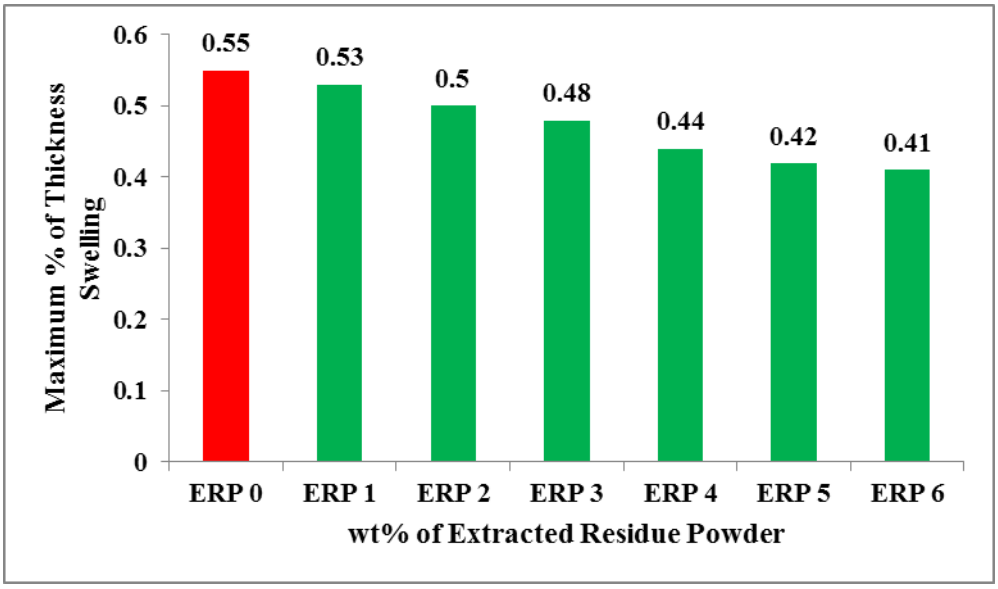


Figure 4.3 (d): Effect of wt% of ERP on maximum thickness swelling

4.3 Mechanical Tests

Various mechanical tests like tensile test, compression test, impact test, hardness test were performed on the CFF filled composite at different weight percentages of CFF in the epoxy resin matrix in order to scrutinize the optimum CFF-epoxy resin composition.

4.3.1 Tensile Test

The tensile strength of CFF filled epoxy resin composite is experimentally determined using 25kN servo controlled Universal Testing Machine (UTM) at fixed cross head speed of 1mm/min under displacement control mode. An ISO standard **ISO 527-2/1B/50** is followed in specimen preparation for tensile testing. From figure 4.4 (a) – 4.4 (c) it can be observed that, with increasing weight percentage of chicken feather fiber in Epoxy resin, the tensile strength of the composite is unceasingly decreasing. Also the maximum tensile strength of epoxy resin (CY-230) with hardener (HY-951) was observed to be 30.14 MPa.

The clear justification for the decrease in tensile strength on adding CFF in epoxy matrix could be the poor strength of fiber itself. The another reason is the irregular shape of the fibers due to which the strength of the composite decreases as they become unable to support the stress transferred from the matrix **(Yang *et al*., 2007).** The same trend of decreasing tensile strength with emu feather fiber was diagnosed by **(Sekhar *et al*., 2015).** So, it can be concluded that the addition of chicken feather fiber into epoxy is unacceptable from strength point of view.

The lower values of percentage elongation with increasing CFF wt% displays the brittle nature of the fabricated composite. From figure 4.4 (a) the remarkable difference in the strain can be observed between the control and the CFF based composite. The strain in different compositions of composite has different random values and it may be due to the non-uniform distribution of fiber in the matrix. The decrease in tensile strength monitored here might be due to the random orientation of fiber or the hydrophilic nature of chicken feathers which may decrease the adhesion characteristics between liquid epoxy and feathers at higher temperature during processing.

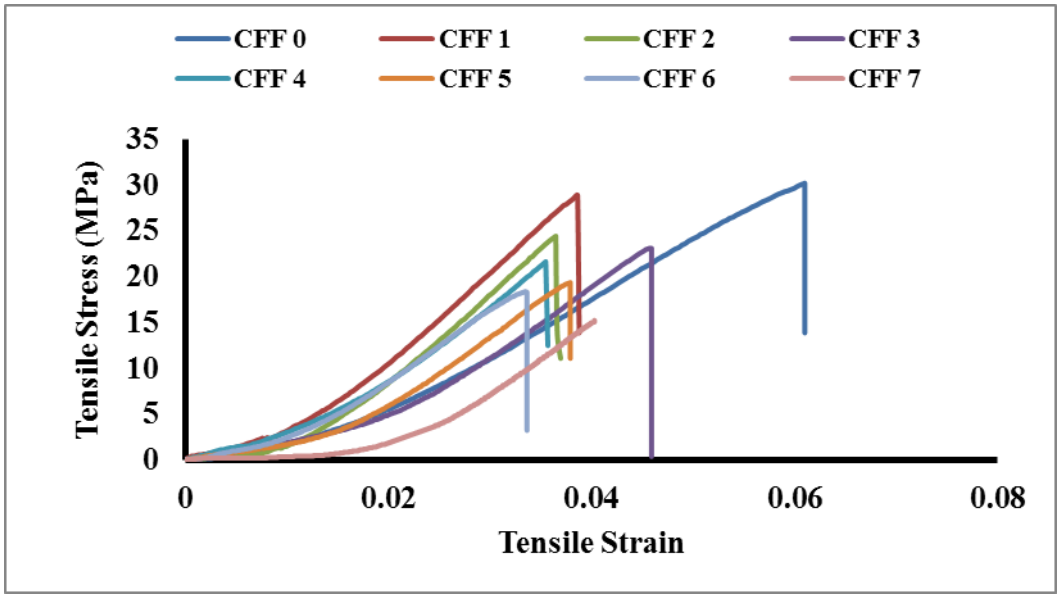


Figure 4.4 (a): Tensile stress-strain diagram with varying wt% of CFF in epoxy resin based composite

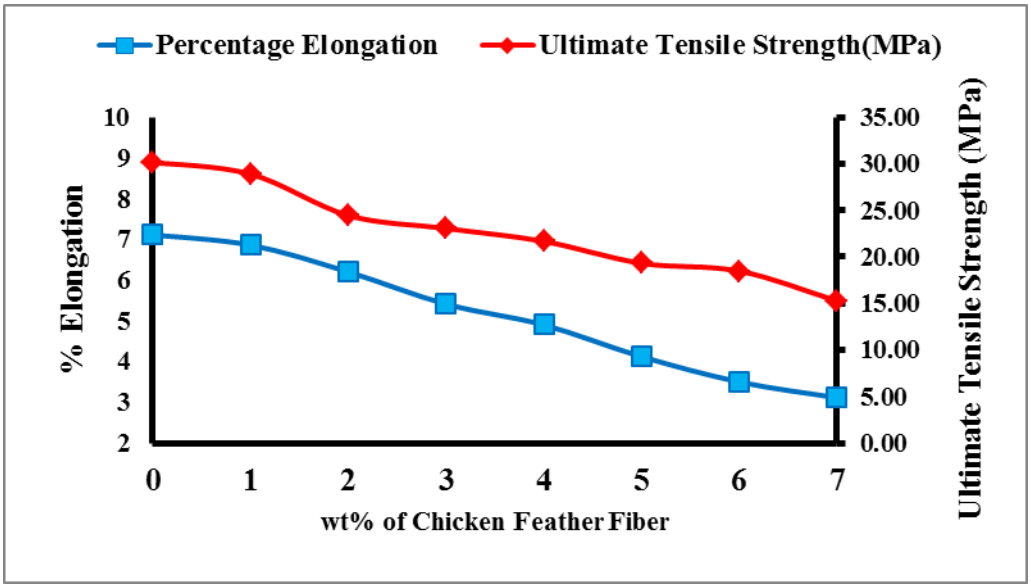


Figure 4.4 (b): Effect of CFF wt% on ultimate tensile strength (MPa) and percentage elongation in epoxy resin based composite

Figure 4.4 (d) shows that the value of young modulus is increasing with increasing wt% of CFF in epoxy and it is maximum between 1% - 2% of CFF in composite. The young modulus becomes almost constant after 3 wt% of CFF. Approximately 49.60% of

rise in the value of young modulus is monitored between the control and the composite with 1 wt% of CFF. The value of young modulus of neat epoxy is 667.6 MPa with almost 0.9999 reliability factor as shown in table 4.2.

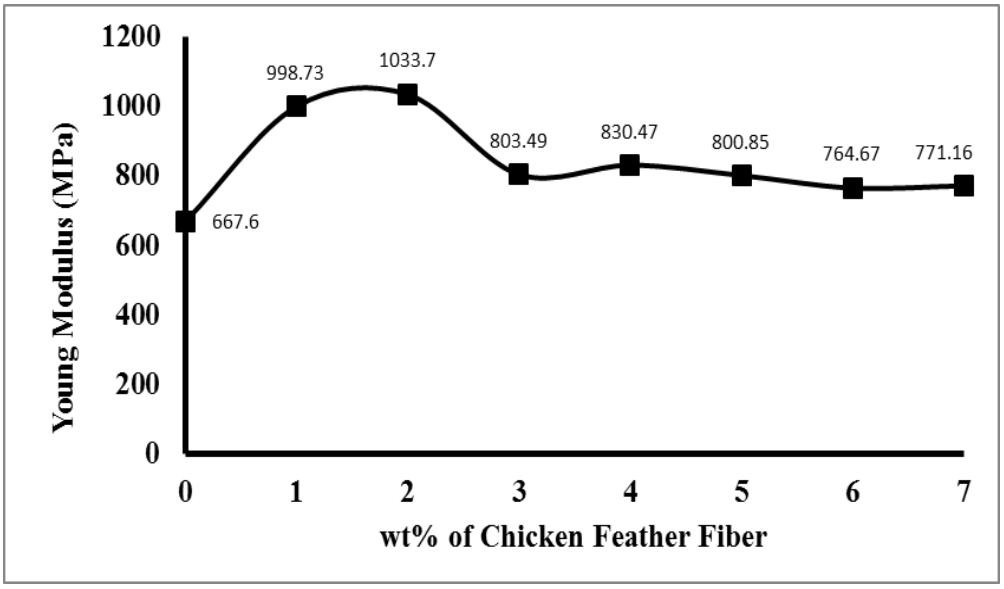


Figure 4.4 (c): Effect of CFF wt% on young modulus of epoxy resin based composite

Table 4.2: Young modulus and reliability factor (R^2 value) of various CFF-epoxy resin composite

Percentage of CFF	Young Modulus (MPa)	R^2 Value
0	667.60	0.9999
1	998.73	0.9996
2	1033.7	0.9998
3	803.49	0.9998
4	830.47	0.9995
5	800.85	0.9994
6	764.67	0.9995
7	771.16	0.9994

4.3.2 Compression Test

The compressive strength of CFF filled epoxy resin composite is experimentally determined using 25kN servo controlled Universal Testing Machine (UTM) at fixed cross head speed of 1mm/min under displacement control mode

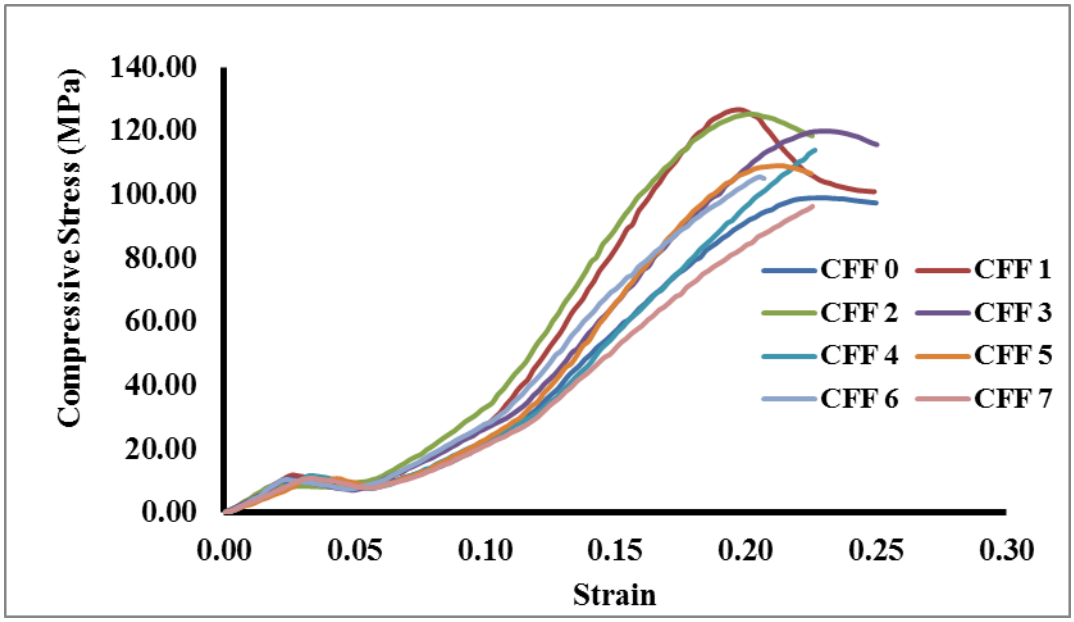


Figure 4.5 (a): Compressive stress-strain diagram for different wt% of CFF in epoxy resin based composite

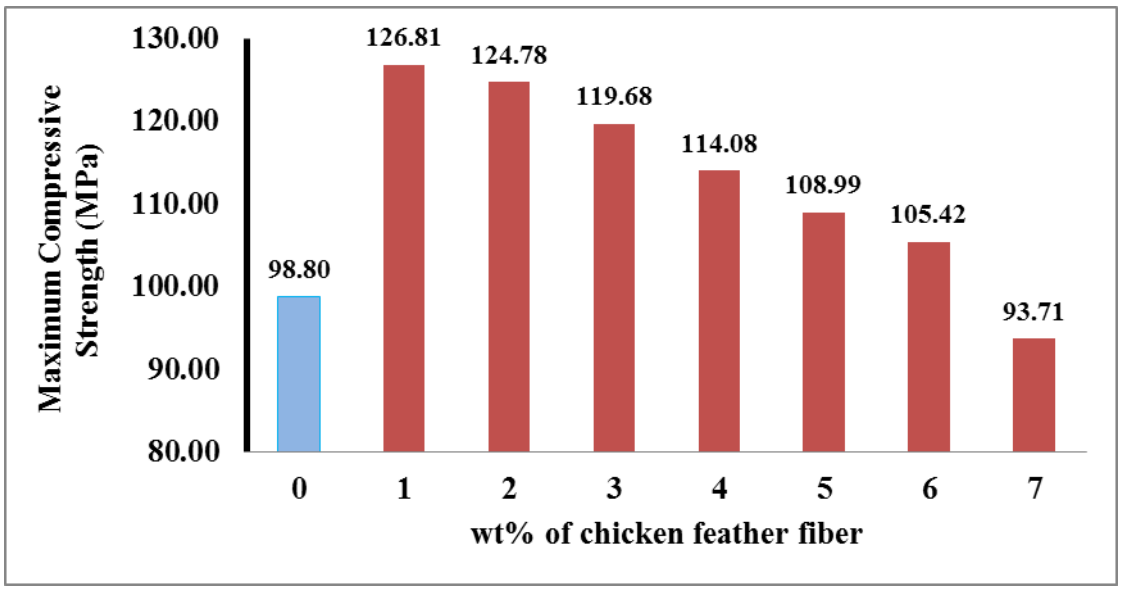


Figure 4.5 (b): Effect of CFF wt% on ultimate compressive strength (MPa)

. An ISO standard **ISO 604: 2002 (E)** is followed for compression testing of polymeric composite. Figure 4.5 (a) shows the variation in the stress with strain for different composition of CFF and epoxy resin ranging from 0 wt% (neat epoxy) to 7 wt% of CFF in epoxy resin. The decreasing trend can be seen in the figure with the increase in CFF weight percentages. It can be clearly observed from figure 4.5 (b) that the ultimate compressive strength of CFF filled composite is greater than the neat epoxy sample but it is decreasing with rise in CFF wt% in epoxy resin till 6 wt%. Approximately 28.35% increase in UTS is observed at 1 wt% CFF filled composite as compared to neat epoxy with hardener. The maximum UTS were observed to be 126.81 MPa at 1 wt% CFF composition.

4.3.3 Rockwell Hardness Test

Digital Rockwell hardness testing machine manufactured by OM Engineering Instruments, Delhi, India was used to test the hardness of the composite. For the current test scale L is selected for measurement of hardness with ¼" ball indenter diameter. Scale L is basically used for plastic materials: vulcanized, fiber, Bakelite etc. Figure 4.6 shows the variation in Rockwell hardness value with change in CFF wt% in epoxy based composite. Ten readings per sample were taken for the evaluation of exact Rockwell hardness of the prepared composite samples. From the results obtained (Table 4.3) it can be seen that with increase in wt% of CFF the hardness of the composite is increasing and it is maximum (i.e. 140.57 HRL) at 5 wt% of CFF in epoxy resin. The hardness value of neat epoxy (control) is 130.45 HRL.

Hardness is considered as a suitable parameter for optimizing the composite and 5 wt% is considered as an optimum composition for fabrication of hybrid composite. The hardness values of various composition shows minimum variation with varying wt% of fiber because hardness is the surface property and it is observed that the non uniform mixing of fiber in matrix leads to almost similar surface property (except surface irregularity) when average reading of hardness is recorded.

Table 4.3: Effect of wt% of CFF on Rockwell hardness value

% of CFF	Hardness Value (in HRL)										
	R1	**R2**	**R3**	**R4**	**R5**	**R6**	**R7**	**R8**	**R9**	**R10**	**Avg.**
0	130.00	131.45	130.90	130.16	131.10	130.85	130.41	130.00	131.14	130.20	130.62
1	131.50	132.00	132.75	130.90	133.00	132.00	131.80	131.98	132.00	133.25	132.12
2	131.97	133.00	133.85	134.00	132.85	133.00	134.18	134.00	131.00	133.52	133.14
3	138.50	137.00	137.45	134.00	134.12	135.00	133.89	133.00	137.02	134.52	135.45
4	136.75	136.00	137.89	138.00	138.25	137.52	136.99	139.54	139.00	136.11	137.61
5	140.15	141.00	142.50	142.66	143.00	141.00	139.88	141.20	137.90	142.60	141.19
6	133.50	141.00	137.80	140.00	136.85	136.00	138.00	141.59	138.02	140.00	138.28
7	133.50	134.00	137.00	138.50	135.00	136.20	134.00	138.00	135.00	136.00	135.72

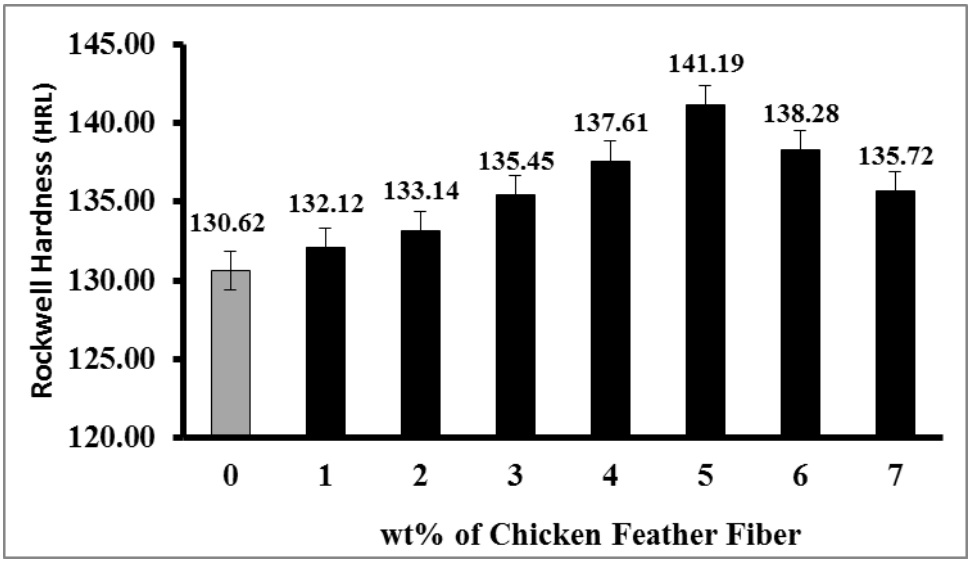


Figure 4.6: Effect of CFF wt% on Rockwell hardness value (HRL)

4.3.4 Impact Test

Izod impact test was performed using Impact testing machine. The results of impact test are shown in figure 4.7. It shows variation in impact strength and impact energy of the composite with increase in wt% of CFF and it is observed that the maximum Impact strength is occurring at 5 wt% (i.e. 1880 J/m^2).

Table 4.4: Effect of wt% of CFF on Impact Energy, Impact Strength and Energy/ thickness of epoxy based composite

% of CFF	Geometric Dimension (In mm)			Area (mm^2) T*B	Energy (J)	Energy/Thickness (J/t)	Impact Strength (kJ/m^2)
	L	T	B				
0	60.0	8.00	12.7	101.6	0.1253	0.0157	1.23
1	60.0	8.00	12.7	101.6	0.1360	0.0170	1.34
2	60.0	8.00	12.7	101.6	0.1400	0.0175	1.38
3	60.0	8.20	12.7	104.14	0.1485	0.0181	1.43
4	60.0	7.20	12.7	91.44	0.1590	0.0221	1.74
5	60.0	7.90	12.7	100.33	0.1886	0.0239	1.88
6	60.0	8.00	12.7	101.6	0.1666	0.0208	1.50
7	60.0	7.20	12.7	91.44	0.1250	0.0174	1.48

Also the impact strength is continuously increasing with wt% of CFF in epoxy resin till 5% and then suddenly falling to 1480 J/m^2 at 7 wt% of CFF. Experimentally it is determined that the impact strength of neat epoxy resin (CY-230) with hardener (HY-951) sample is 1230 J/m^2. The major reason for this hike is the flexible nature of the fibers. The fibers when added into the epoxy forms layers and absorbs energy just like fabrics. Table 4.4 lists the elaborative results obtained during Impact testing performed at 29^{0}C atmospheric temperature and 49% RH.

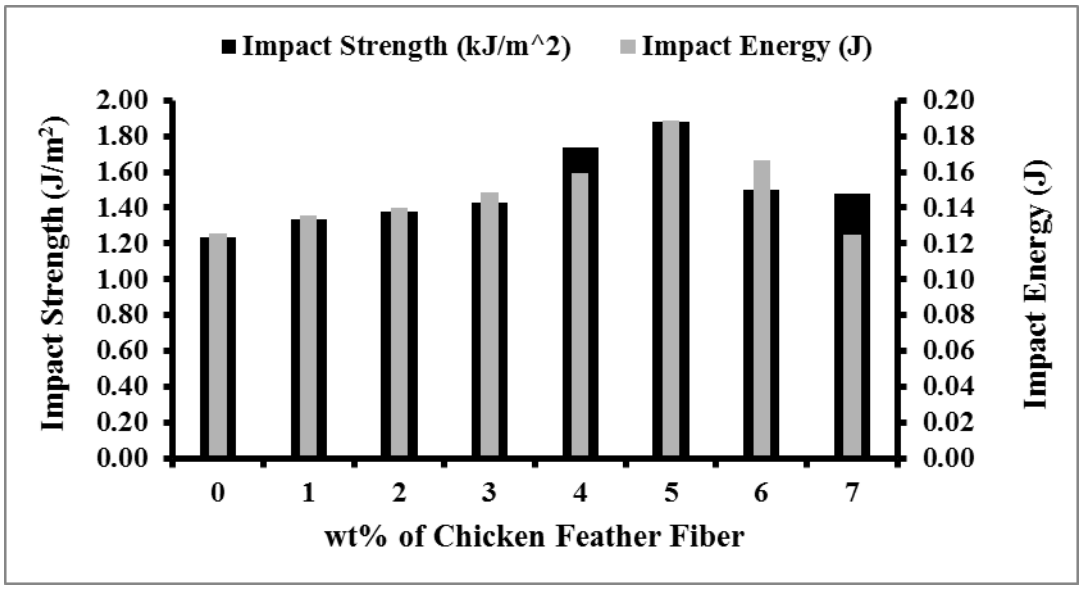


Figure 4.7: Effect of wt% of CFF on impact strength and impact energy

4.4 Pre Final Output

From the mechanical tests performed it was observed that:

- Impact strength and hardness of prepared composite increases with increasing wt% of CFF in epoxy resin.
- The maximum value of Impact strength (i.e. 1880 J/m^2) occurs at 5 wt% of CFF which is 52.84% (approximately 1.5 times) greater than the control of epoxy resin and hardener.
- The maximum value of Rockwell Hardness (i.e. 141.19) also occurs at 5 wt% of CFF which is 8% greater than the Control of Epoxy resin and hardener.

- Also the value of Compressive strength is greater than the value of control sample for 1 to 6 wt% of CFF in epoxy resin.

Therefore, 5 wt% of chicken feather fiber in epoxy resin is considered as the most feasible composition for fabrication of hybrid composite with extracted residue powder (from fish) as particulate.

The results of various composition of hybrid composite i.e. varying wt% of particulate (extracted residue powder) in 5 wt% CFF and epoxy resin + hardener is discussed in the next sections.

4.5 Mechanical Tests results for hybrid composite

4.5.1 Tensile Test

Figure 4.8 (a-c) shows the results obtained using 25 kN servo controlled Universal Testing Machine (UTM) for tensile testing. The fixed cross head speed of 1mm/min was selected for the generation of load v/s displacement curve.

Here different wt% of ERP in 5 wt% CFF filled hybrid biocomposite samples were tested and the obtained stress/strain relation is shown in figure 4.8 (a).

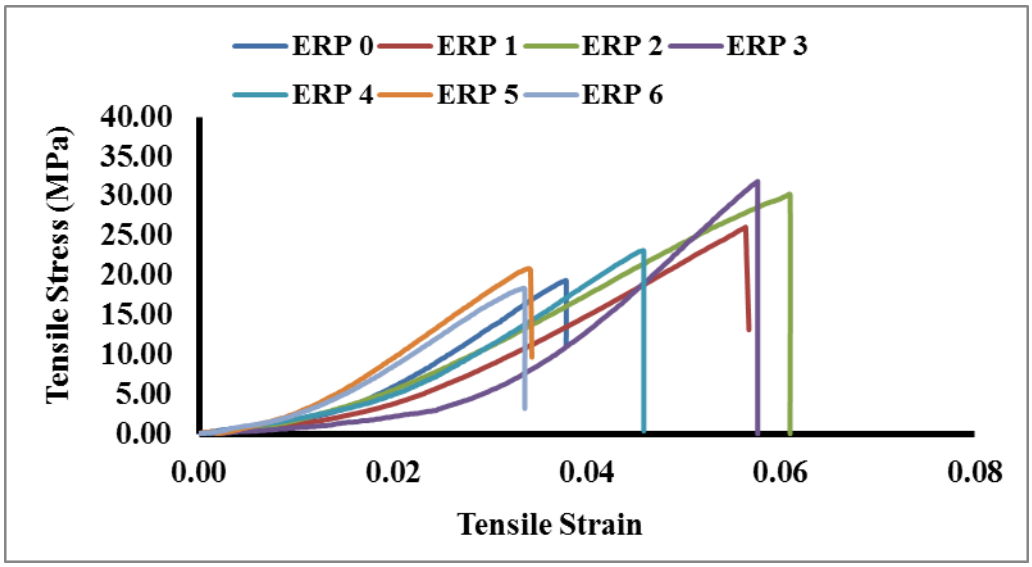


Figure 4.8 (a): Tensile stress-strain diagram with varying wt% of extracted residue powder in 5 wt% CFF filled composite

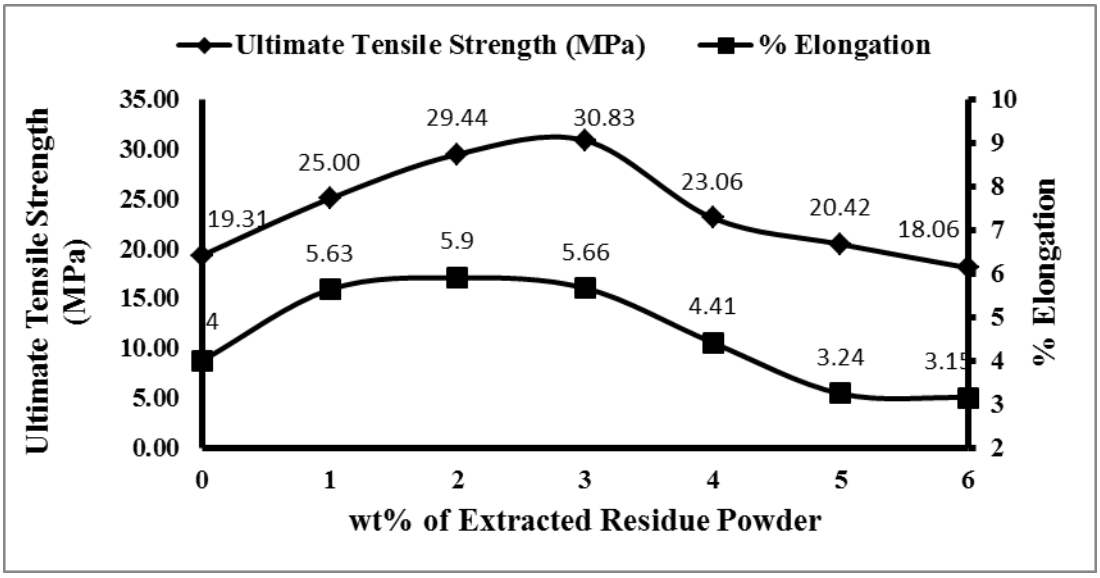


Figure 4.8 (b): Effect of ERP wt% (with 5wt% CFF) on ultimate tensile strength and percentage elongation on hybrid composite

The maximum tensile strength (i.e. 30.83MPa) was observed at 3 wt% ERP which is 70.71% greater than 0 % ERP sample. Also the percentage elongation was rising with increasing wt% of ERP till 3% (% elongation at 3% ERP was 5.78). Later with increasing wt% of ERP, percentage elongation decreases down to 3.5% which shows its brittle characteristic.

Table 4.4: Young modulus and reliability factor (R^2 value) of various hybrid composites with extracted residue powder as particulate in 5 wt% CFF filled epoxy resin

wt% of ERP	Young Modulus (MPa)	R^2 Value
0	800.85	0.9994
1	667.88	0.9997
2	666.08	0.9997
3	1115.8	0.9999
4	804.86	0.9998
5	855.33	0.9996
6	775.63	0.9993

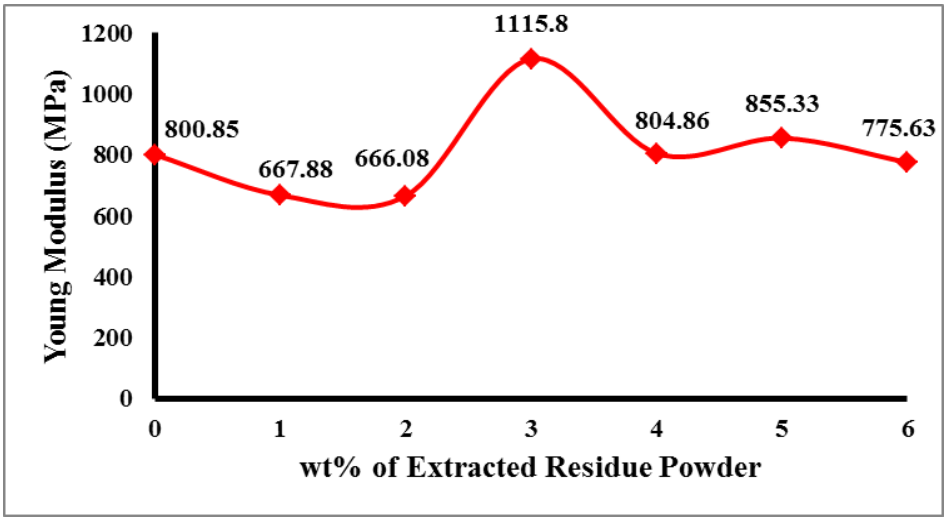


Figure 4.8 (c): Effect of ERP wt % on young modulus of hybrid composite

The increase in the young modulus (figure 4.8 (c))and ultimate tensile strength (figure 4.8 (b)) might be due to the better interfacial bonding and matrix- reinforcement cross linking as shown in SEM micrograph and XRD analysis. This also enhanced the toughness characteristics of the hybrid composites.

4.5.2 Compression Test

Figure 4.9 (a & b) systematically characterizes the compressive properties of ERP filled hybrid biocomposite with 5 wt% CFF reinforcement. The curve (figure. 4.9 (a)) shows steep and constant stress till 0.05 strain value for all the compositions but later it shows drastic rise in the stress value.

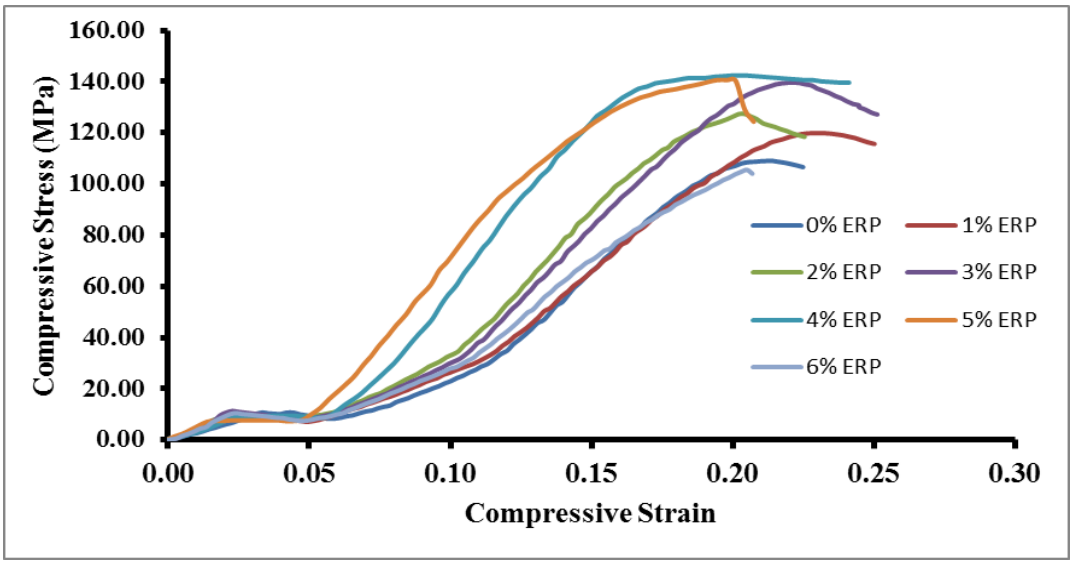


Figure 4.9 (a): Compressive stress-strain diagram for different wt% of ERP in 5 wt% CFF filled hybrid composite

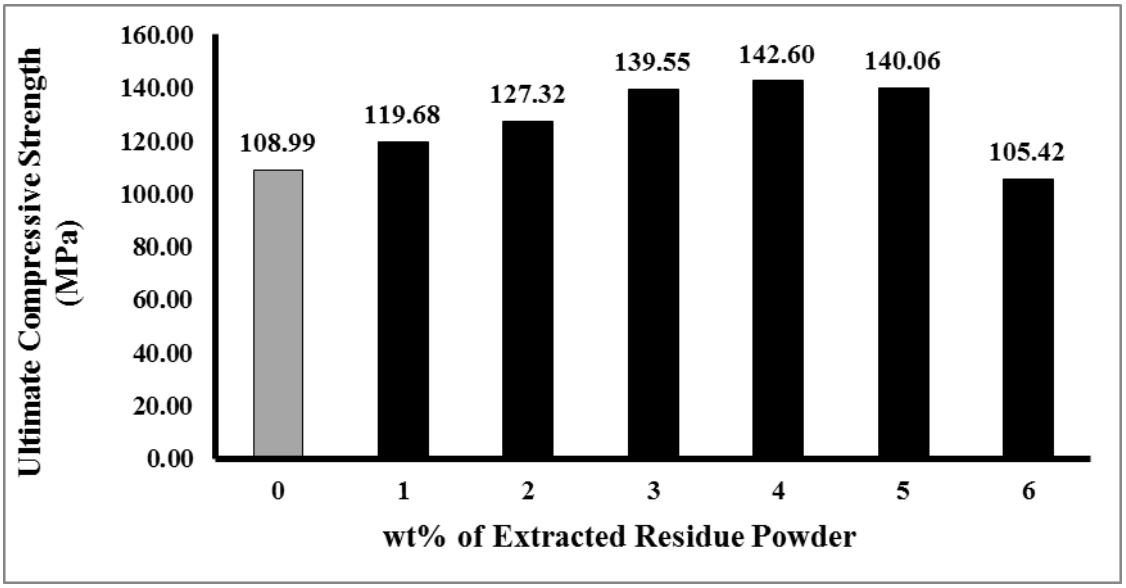


Figure 4.9 (b): Effect of ERP wt% (with 5 wt% CFF) on ultimate compressive strength of hybrid composite

This type of curve trend is usually seen in multi reinforced composite materials. Here, 4 wt% ERP sample shows maximum strength (i.e. 142.60 MPa) which is almost 30.84% greater than the compressive strength of 0 wt% ERP sample kept under same testing conditions. The low fiber volume fraction helps to attain the better strength. Also the rise of 41.24% was observed between the CS of neat epoxy that was 98.90 MPa and 3 wt% ERP filled hybrid sample (with 5 wt% CFF) which has 139.55 MPa CS. The enhancement in strength on application of compressive load might be due to the strong molecular and interfacial bonding between the matrix and the reinforcing material as clearly visible in SEM micrographs. At higher the inclusion of ERP in the voids

4.5.3 Izod Impact Test

Table 4.5 characterizes the impact properties of the ERP filled hybrid composite in 5 wt% CFF reinforcement. The variation in impact strength and energy is shown in figure 4.10. The optimum composition obtained was the hybrid composite with 3 wt% ERP. The impact strength (i.e. 1908.2 J/m^2) which was enhanced by 4.08 % as compared to composite with 0 wt% ERP (i.e. 1856.3 J/m^2) and 54.72% as compared to neat epoxy. The increase in impact strength might be due to the proper blending of ERP and better cross linking between the matrix and the reinforcing agents. The decrease in the impact properties after 3 % ERP composition may be due to fiber breakoff (as shown in SEM micrograph with 4% ERP), presence of voids or dimples as shown in SEM micrograph with 5% ERP, Figure 4.14 (e) and clustering of particulates at higher weight percentages.

Table 4.5: Effect of wt% of Extracted Residue Powder on Impact Energy, Impact Strength and Energy/ thickness of Epoxy based composite

% of ERP	Geometric Dimension (In mm)			AREA (mm^2) T*B	Energy (J)	Energy/Thickness (J/m)	Impact Strength (kJ/m^2)
	L	T	B				
0	60.0	8.00	12.7	101.6	0.1886	0.0236	1.8563
1	60.0	8.00	12.7	101.6	0.1900	0.0238	1.8701
2	60.0	8.00	12.7	101.6	0.1920	0.0240	1.8898
3	60.0	8.20	12.7	104.14	0.1963	0.0242	1.9082
4	60.0	7.20	12.7	91.44	0.1891	0.0241	1.8992
5	60.0	7.90	12.7	100.33	0.1845	0.0234	1.8389
6	60.0	8.00	12.7	101.6	0.1800	0.0225	1.5000

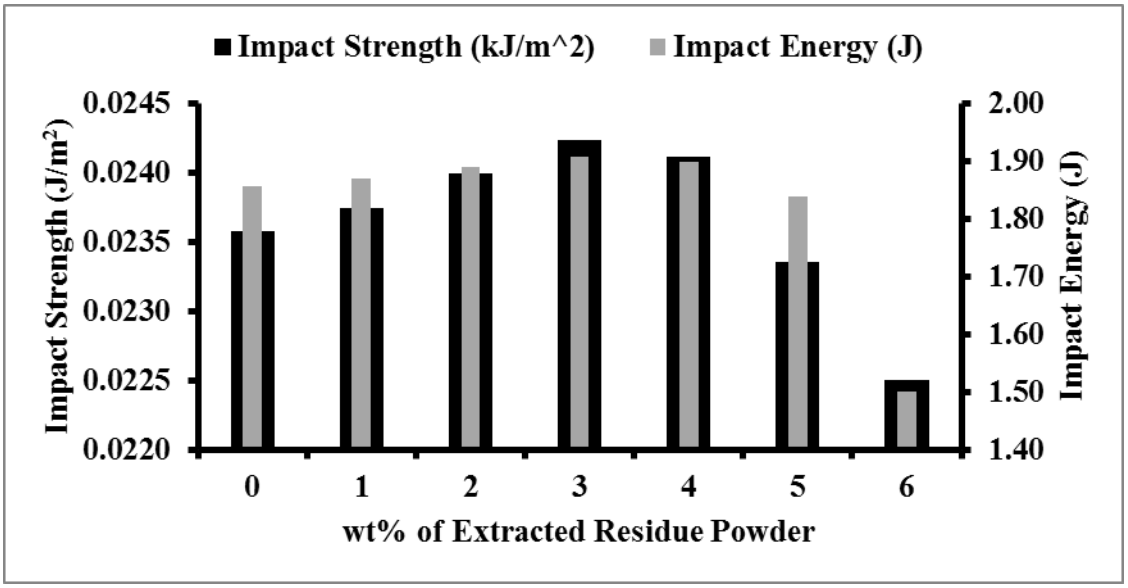


Figure 4.10: Effect of ERP wt% (with 5 wt% CFF) on impact strength and impact energy

4.5.4 Rockwell Hardness Test

Hardness being considered as the surface property, therefore the effective hardness of the prepared sample was arbitrated by the average value obtained from the testing at various positions on the same surface. From figure 4.11 it can be seen that almost same hardness value (HRL value) is been observed with varying wt% of ERP in 5 wt% fiber

reinforced composite. The maximum value can be seen between 3 to 4 wt% of ERP. The sample with 5% filled CFF and without ERP shows 141.19 HRL hardness value. The random variation in the hardness value of hybrid biocomposite is due to the non uniform mixing of particulate in the epoxy resin. The point of indentation at white ash contamination zone would be showing comparatively higher HRL value as compared to point of neat epoxy on the surface. Therefore average value of sample is considered as appropriate output for hardness measurement. The continuous decrease in fiber volume fraction with increasing ERP percentage also scrutinized better surface hardness. At 3 wt% of ERP (141.8 HRL) the total of 8.55% rise in hardness value can be monitored as compared to neat epoxy. The hardness value is 142.1 HRL at 4 wt% of ERP which is the highest value recorded for hybrid biocomposite.

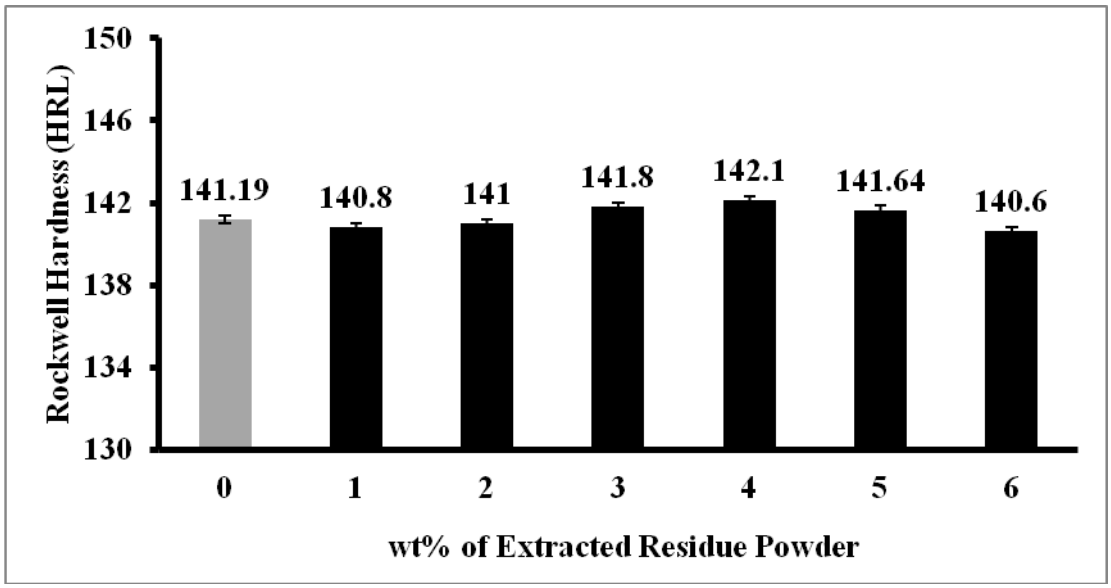


Figure 4.11: Effect of ERP wt% (with 5 wt% CFF) on Rockwell hardness value (HRL) on L scale

4.5.5 Flexural Test

Figure 4.12 (a-c) characterizes the flexural properties of the hybrid biocomposite that outlines the flexural strength, flexural strain and flexural modulus respectively of the ERP filled biocomposite.

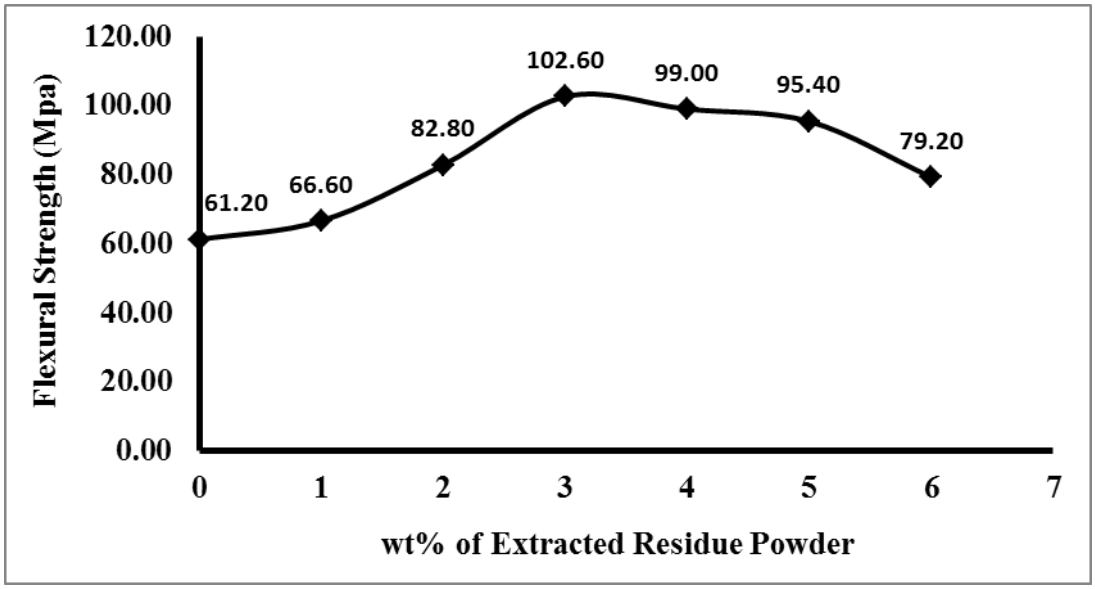


Figure 4.12 (a): Effect of wt% of extracted residue powder on flexural strength of hybrid composite

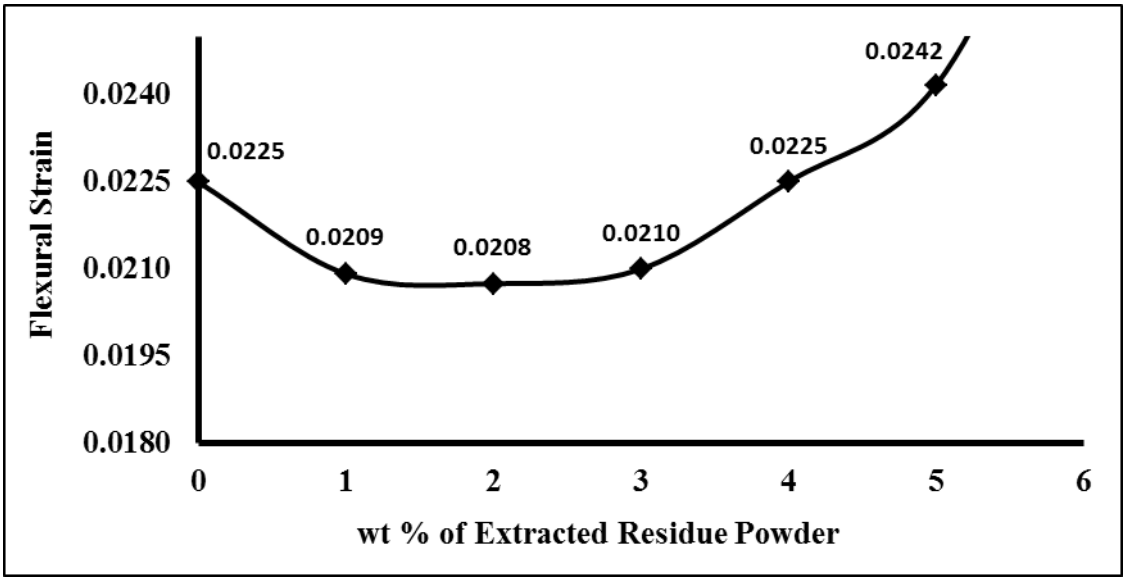


Figure 4.12 (b): Effect of wt% of extracted residue powder on flexural strain of hybrid composite

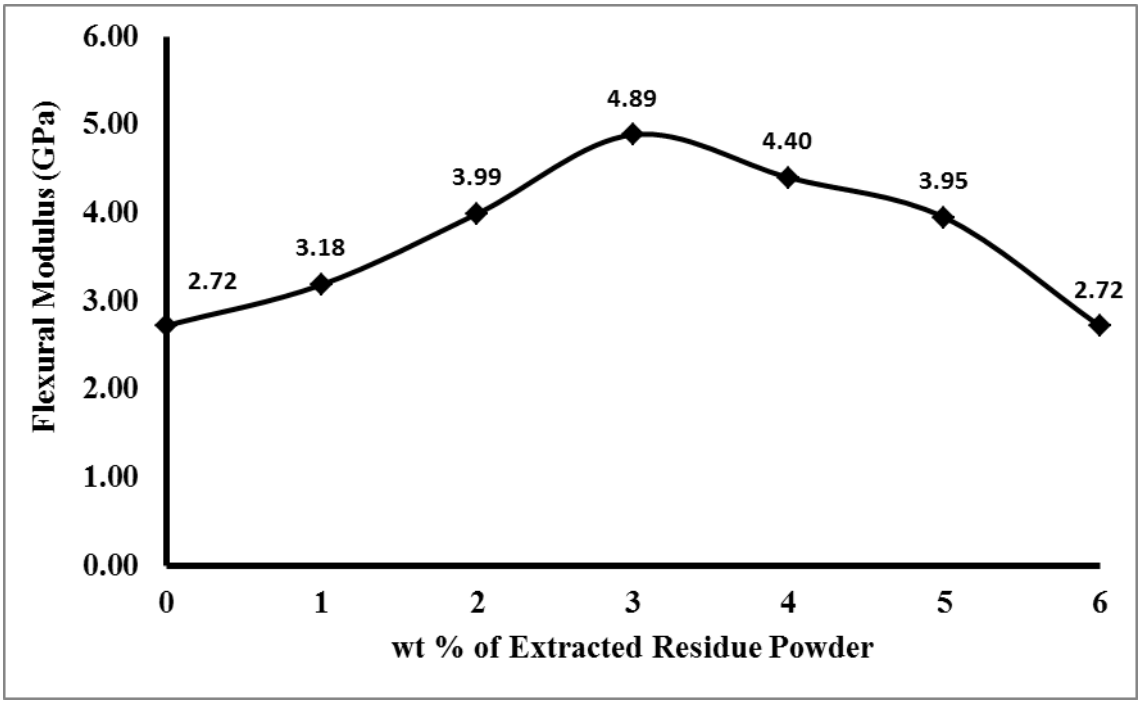


Figure 4.12 (c): Effect of wt% of extracted residue powder on flexural modulus of hybrid composite

From figure 4.12 (a & c) it can be seen that the flexural strength and flexural modulus rises with the increasing wt% of ERP in 5 wt% CFF (CFF is kept same in all the samples) till 3 wt% of ERP. The recorded rise in flexural strength was almost 67.65% at 3wt% ERP (i.e. 102.60 MPa) and flexural modulus rises by 79.78% (i.e. 4.89 GPa at 3% of ERP). From the results obtained it was seen that the flexural modulus at 0 and 6 wt% ERP was almost identical. The main cause for the decrease in the strength and modulus of the hybrid composite at higher wt% of ERP is the improper bonding and over contamination of ERP as could be seen through SEM image. The flexural strain shown in figure 4.9(b) follows the reverse trend and it is minimum (i.e. around 0.021) between 2 to 3wt% of ERP. Very less variation in strain was observed during flexural test.

4.6 Thermal Analysis

The thermal analyses of the prepared samples were diagnosed using TGA/DTA analysis at the IIC Laboratory, IITR. Figure 4.13 (a-f) shows the rate of decomposition and variation in the specimen with temperature and time through DTA, DTG and TG curve of the hybrid composite samples performed in Air environment.

Figure 4.13 (a) shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and **1 wt% Extracted Residue Powder** from fish residue. The initial sample weight was 10.210 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 251°C to 459°C with corresponding rate of decomposition ranging 2.64 mg/min to 2.0 mg/min. Prior to 233°C, the weight loss of 18.30% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 2.64 mg/min was observed at 251°C. Such decomposition has been supported with the heat of fusion of -1.43 J/mg at 261°C and -1.30 J/mg at 464°C with DTA signal of 190.2 µV (0.1902mV) to 275.1 µV (0.2751mV) between temperature range of 261°C and 464°C respectively. The decomposition of the material had been concluded at about 592°C leaving carbonize residue 1.7% of initial weight.

Figure 4.13 (b) shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and **2 wt% Extracted Residue Powder** from fish residue. The initial sample weight was 10.18 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 262°C to 426°C with corresponding rate of decomposition ranging 4.3 mg/min to 3.10 mg/min. Prior to 239°C, the weight loss of 15.30% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.3 mg/min was observed at 262°C. Such decomposition has been supported with the heat of fusion of -2.06 J/mg at 277°C and -3.85 J/mg at 452°C with DTA signal of 265 µV (0.265 mV) to 648 µV (0.648 mV) between temperature range of 277°C and 452°C respectively. The decomposition of the material had been concluded at about 518°C leaving carbonize residue 2.2% of initial weight.

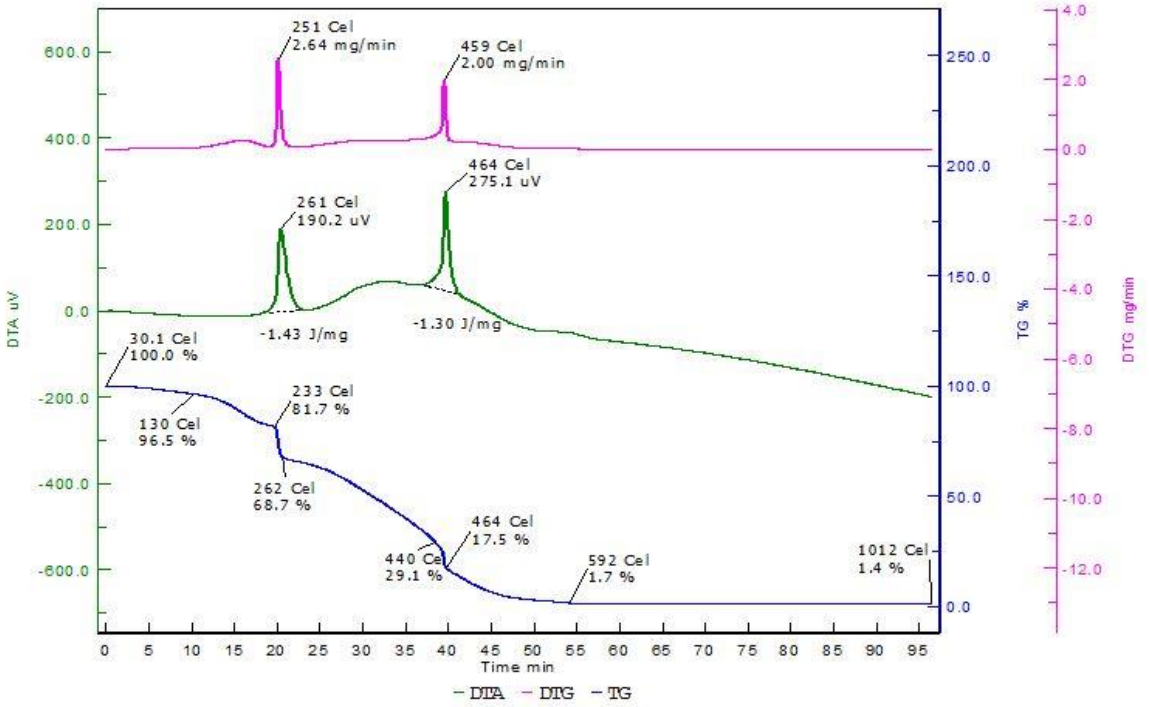


Figure 4.13 (a): Thermal analysis of 1% extracted residue powder + 5% CFF in epoxy resin hybrid composite

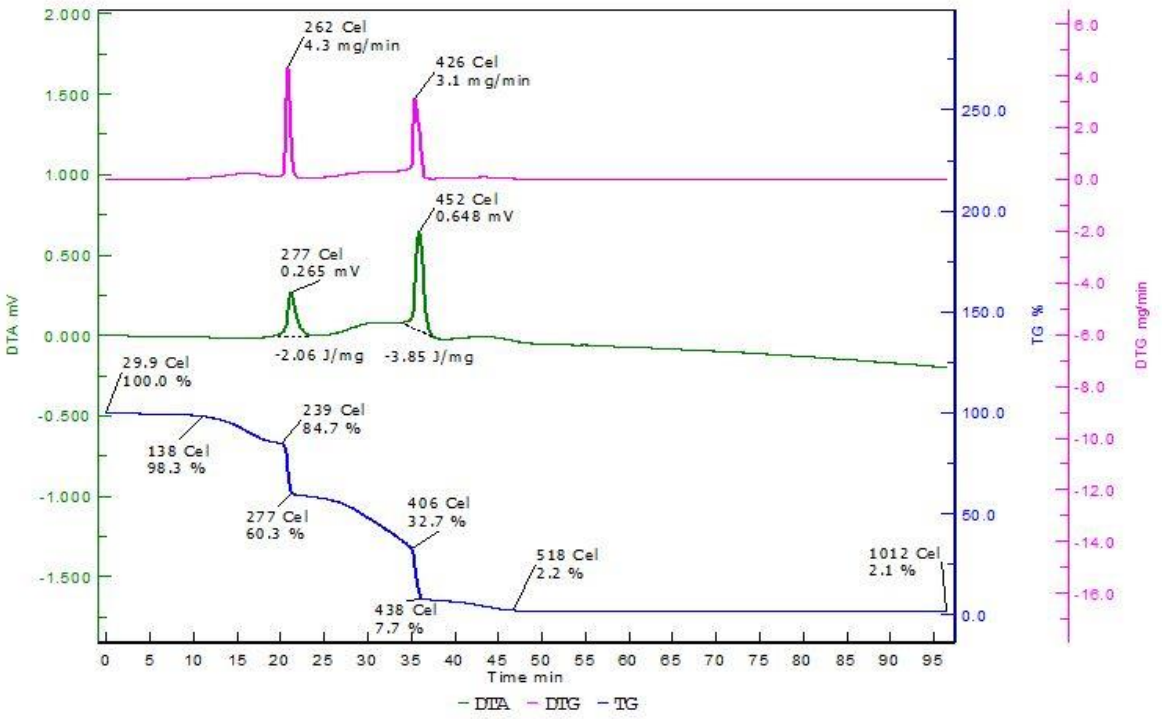


Figure 4.13 (b): Thermal analysis of 2% extracted residue powder + 5% CFF in epoxy resin hybrid composite

Figure 4.13 (c) shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and **3 wt% Extracted Residue Powder** from fish residue. The initial sample weight was 10.32 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 262°C to 425°C with corresponding rate of decomposition ranging 4.4 mg/min to 2.90 mg/min. Prior to 239°C, the weight loss of 15.60% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.4 mg/min was observed at 262°C. Such decomposition has been supported with the heat of fusion of -1.91 J/mg at 278°C and -3.65 J/mg at 450°C with DTA signal of 272 µV (0.272 mV) to 633 µV (0.633 mV) between temperature range of 278°C and 450°C respectively. The decomposition of the material had been concluded at about 522°C leaving carbonize residue 2.9% of initial weight.

Figure 4.13 (d) shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and **4 wt% Extracted Residue Powder** from fish residue. The initial sample weight was 10.27 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 258°C to 422°C with corresponding rate of decomposition ranging 5.0 mg/min to 3.0 mg/min. Prior to 245°C, the weight loss of 16.20% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 5 mg/min was observed at 258°C. Such decomposition has been supported with the heat of fusion of -2.33 J/mg at 283°C and -4.89 J/mg at 446°C with DTA signal of 306 µV (0.306 mV) to 724 µV (0.724 mV) between temperature range of 283°C and 446°C respectively. The decomposition of the material had been concluded at about 505°C leaving carbonize residue 0.9% of initial weight.

Figure 4.13 (e) shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and **5 wt% Extracted Residue Powder** from fish residue. The initial sample weight was 10.20 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 257°C to 409°C with corresponding rate of decomposition

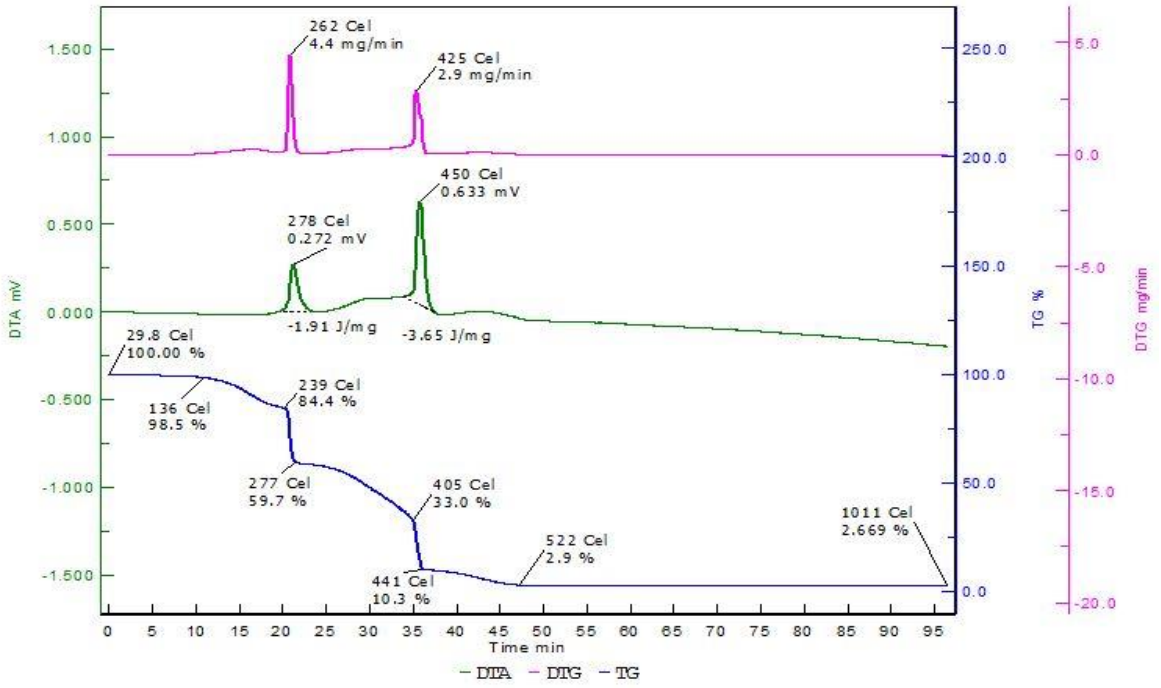


Figure 4.13 (c): Thermal analysis of 3% extracted residue powder + 5% CFF in epoxy resin hybrid composite

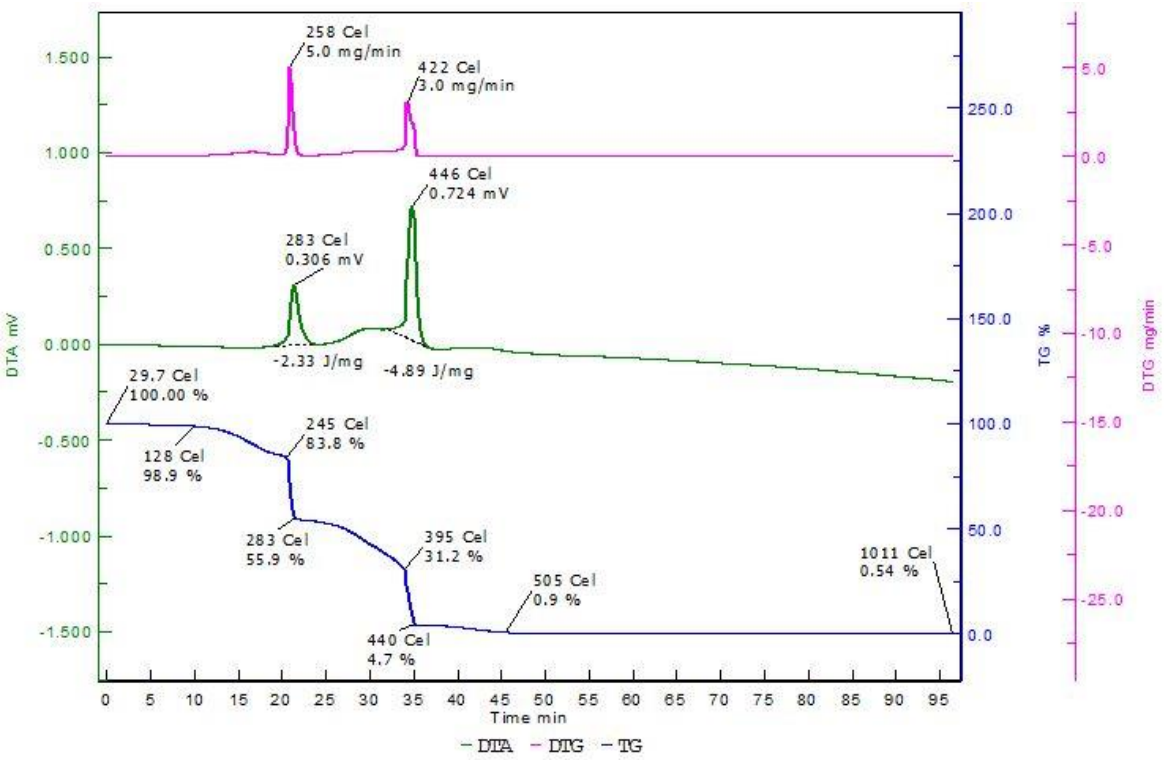


Figure 4.13 (d): Thermal analysis of 4% extracted residue powder + 5% CFF in epoxy resin hybrid composite

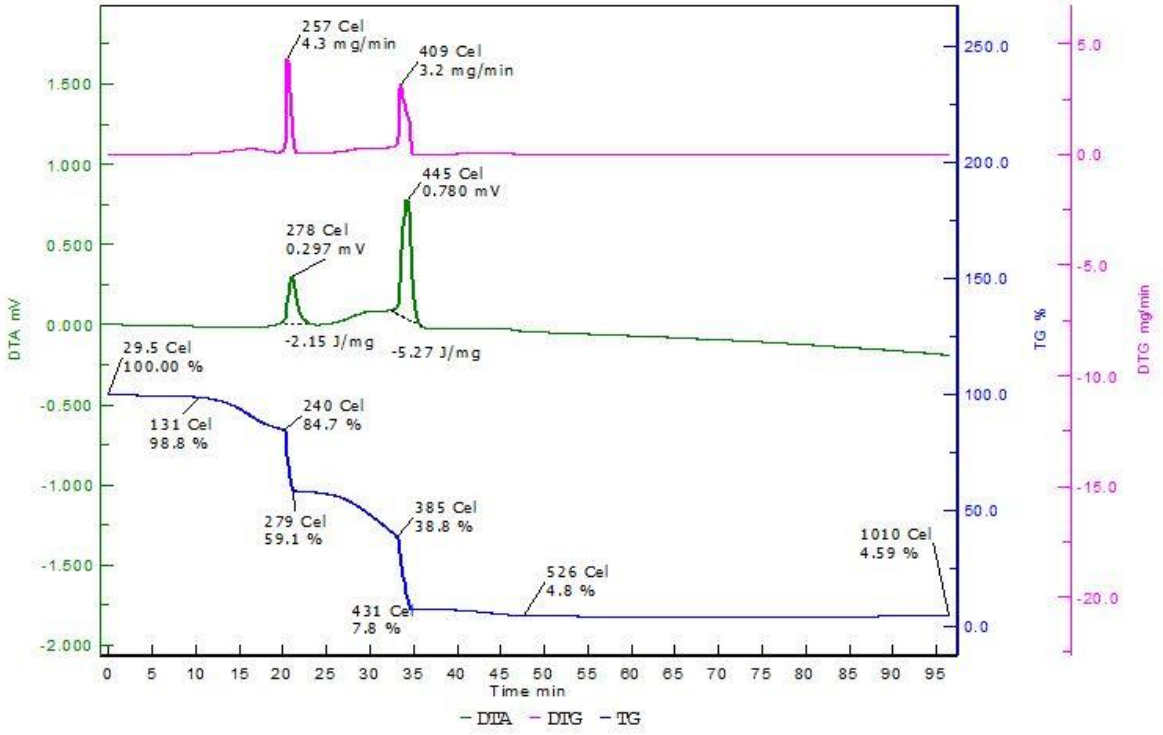


Figure 4.13 (e): Thermal analysis of 5% extracted residue powder + 5% CFF in epoxy resin hybrid composite

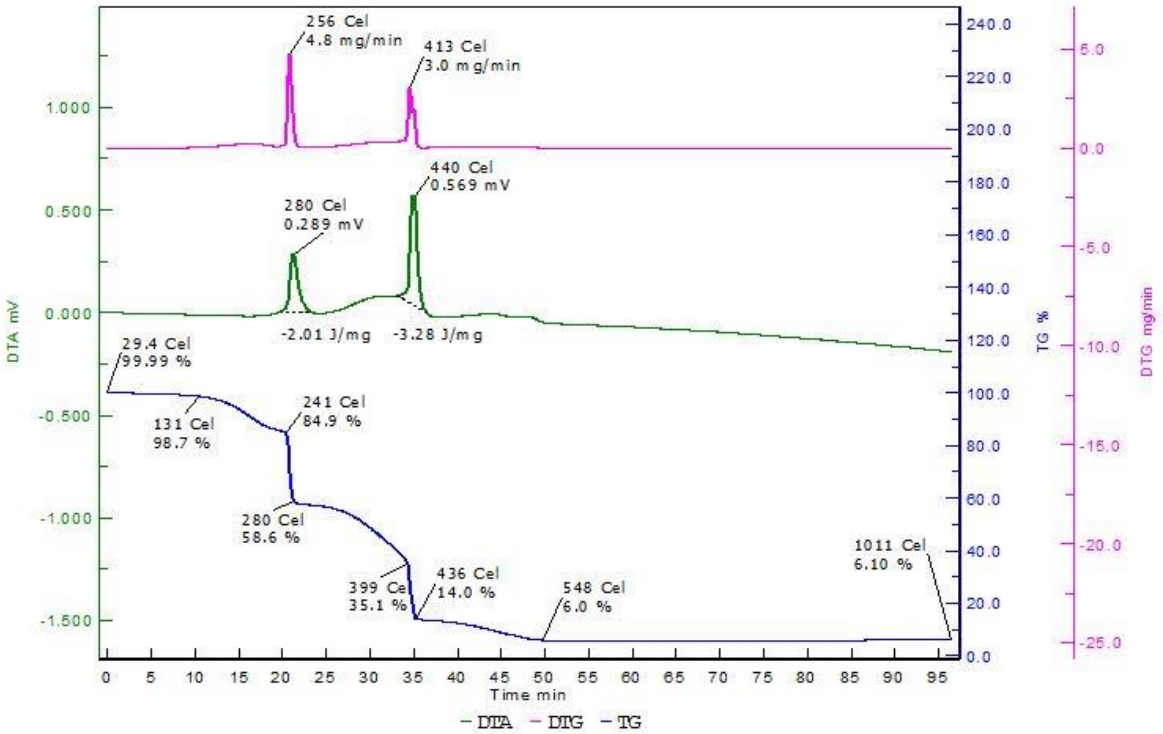


Figure 4.13 (f): Thermal analysis of 6% extracted residue powder + 5% CFF in epoxy resin hybrid composite

ranging 4.3 mg/min to 3.2 mg/min. Prior to 240°C, the weight loss of 15.30% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.3 mg/min was observed at 257°C. Such decomposition has been supported with the heat of fusion of -2.15 J/mg at 278°C and -5.27 J/mg at 445°C with DTA signal of 297 µV (0.297 mV) to 780 µV (0.780 mV) between temperature range of 278°C and 445°C respectively. The decomposition of the material had been concluded at about 526°C leaving carbonize residue 4.8% of initial weight.

Figure 4.13 (f) shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and **6 wt% Extracted Residue Powder** from fish residue. The initial sample weight was 10.18 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 256°C to 413°C with corresponding rate of decomposition ranging 4.8 mg/min to 3.0 mg/min. Prior to 241°C, the weight loss of 15.10% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.8 mg/min was observed at 256°C. Such decomposition has been supported with the heat of fusion of -2.01 J/mg at 280°C and -3.28 J/mg at 440°C with DTA signal of 289 µV (0.289 mV) to 569 µV (0.569 mV) between temperature range of 280°C and 440°C respectively. The decomposition of the material had been concluded at about 548°C leaving carbonize residue 6% of initial weight.

4.7 Scanning Electron Microscopy (SEM)

Figure 4.14 (a) shows the SEM image of the tensile fractured surface of the hybrid composite (with 1 wt% extracted residue powder from Rohu fish as particulate and 5wt% CFF in epoxy resin matrix). The unidirectional river line shows the brittle fracture. The proper accumulation of the chicken feather is visible with dispersed residue part. The Shiny squared region shown in figure signifies the clear separation of bonding which leads to sudden brittle fracture. The powdered particulate is non-uniformly mixed and it has hurdled the river lines and the insufficient filling of particulate around the matrix leads to early failure. Feather's reinforcement near point of failure, matrix flow and minute voids are visible in 500X magnification.

Figure 4.14 (b) shows the SEM image of the tensile fractured surface of the hybrid composite (with 2 wt% extracted residue powder from Rohu fish as particulate and 5wt% CFF in epoxy resin matrix). Here we can observe the improved contamination of ERP and Fiber. Hackles and ribbons are moving in different planes therefore it leads to better impact and compressive strength. Proper bonding between the matrix with fiber and the ERP is visible which shows good tensile strength behavior of the composite. Certain voids and randomly oriented fibers (shown in enlarged view) occurred using hand lay-up technique is seen and these could be the cause of material failure. Therefore improvement can be made in mixing method. The shiny views are due to sudden brittle fracture.

Figure 4.14 (c) shows the SEM image of the tensile fractured surface of the hybrid composite (with 3 wt% extracted residue powder from Rohu fish as particulate and 5wt% CFF in epoxy resin matrix). The Smother river lines show the brittle behavior at the hackle zone. Improved toughness can be observed. Ribbons and hackles are moving in different planes and therefore improved impact strength can be detected. Though the powder particulate is non uniformly distributed but it is increasing the bond strength between the matrix and the particulate as the extracted powder composed of multiple charged elements as been seen through FE-SEM analysis. It seems to be the most appropriate combination for the composite development. Here minute air voids and plucking off of matrix which can be seen at 180x zoom could be the appropriate reason for material failure.

Figure 4.14 (d) shows the SEM image of the tensile fractured surface of the hybrid composite (with 4 wt% extracted residue powder from Rohu fish as particulate and 5wt% CFF in epoxy resin matrix). Here the proper parting zone i.e. crack initiation zone, crack propagation zone and fracture zone is visible. We can observe from the SEM image that at higher accumulation of fiber and the particulate, the matrix shows enhanced bonding but the breaking of fiber and over accumulation of particulate leads to comparative decrease in the mechanical strength of the composite.

Figure 4.14 (e) shows the SEM image of the tensile fractured surface of the hybrid composite (with 5 wt% extracted residue powder from Rohu fish as particulate and 5wt% CFF in epoxy resin matrix). The decrease in the mechanical strength occurs due to stress concentration at the voids area and visibility of dimples at the fracture zone. The debonding of matrix with the fiber and the particulate may be scrutinized due to

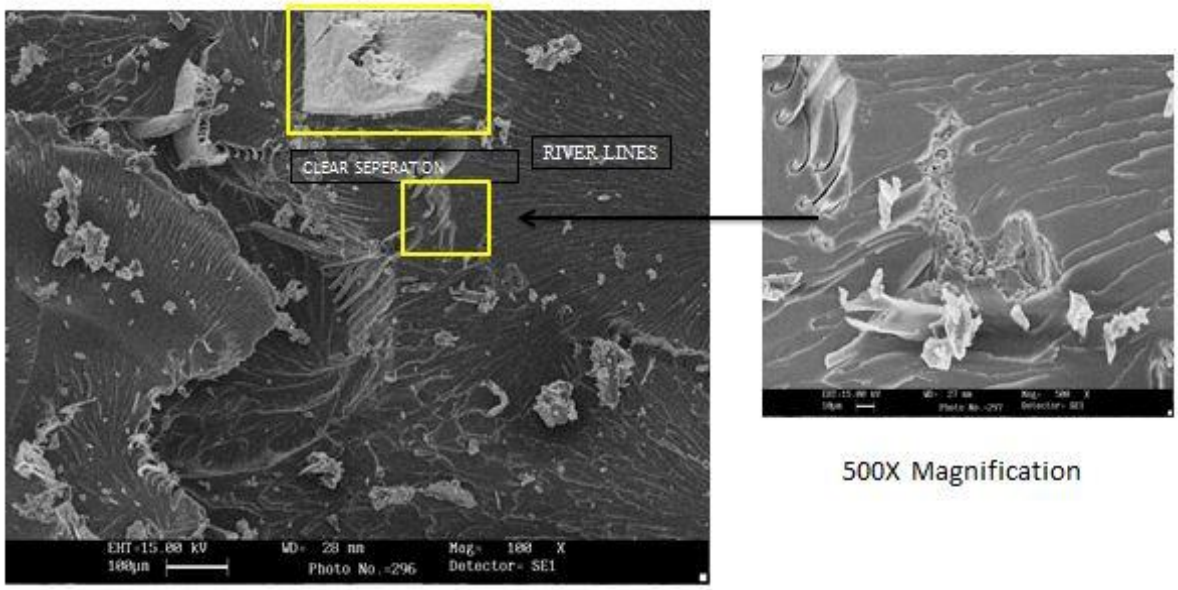


Figure 4.14 (a): SEM image of hybrid composite with 1 wt% extracted residue powder and 5 wt% CFF in epoxy resin at 100X magnification

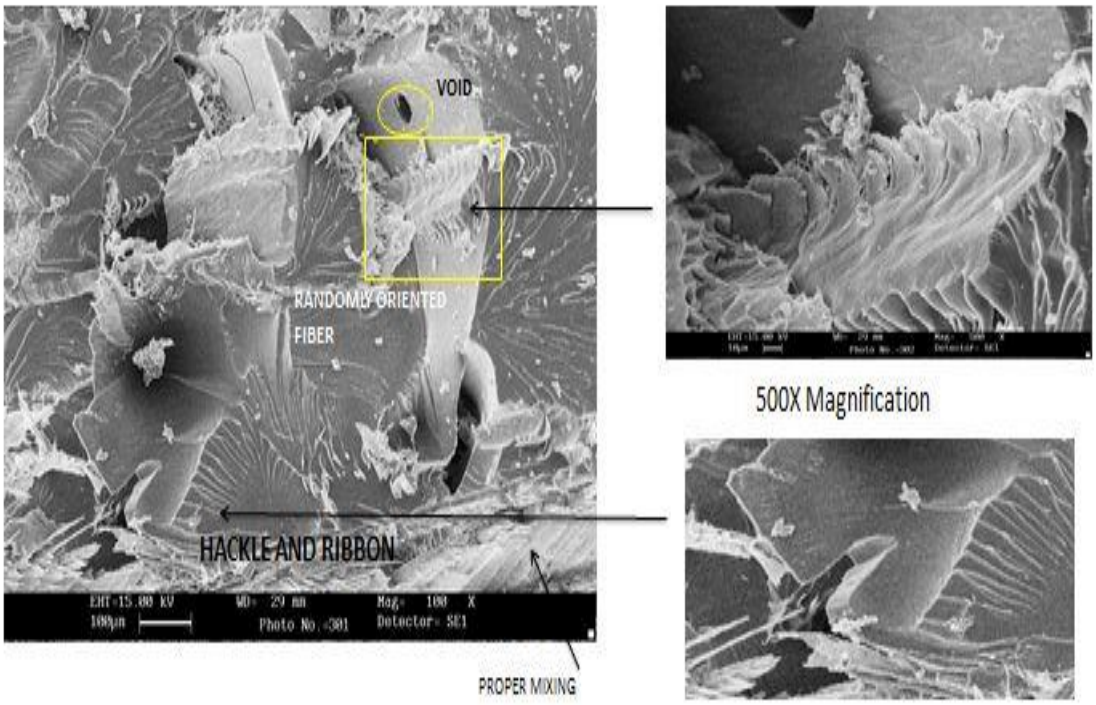


Figure 4.14 (b): SEM image of hybrid composite with 2 wt% extracted residue powder and 5 wt% CFF in epoxy resin at 100X magnification

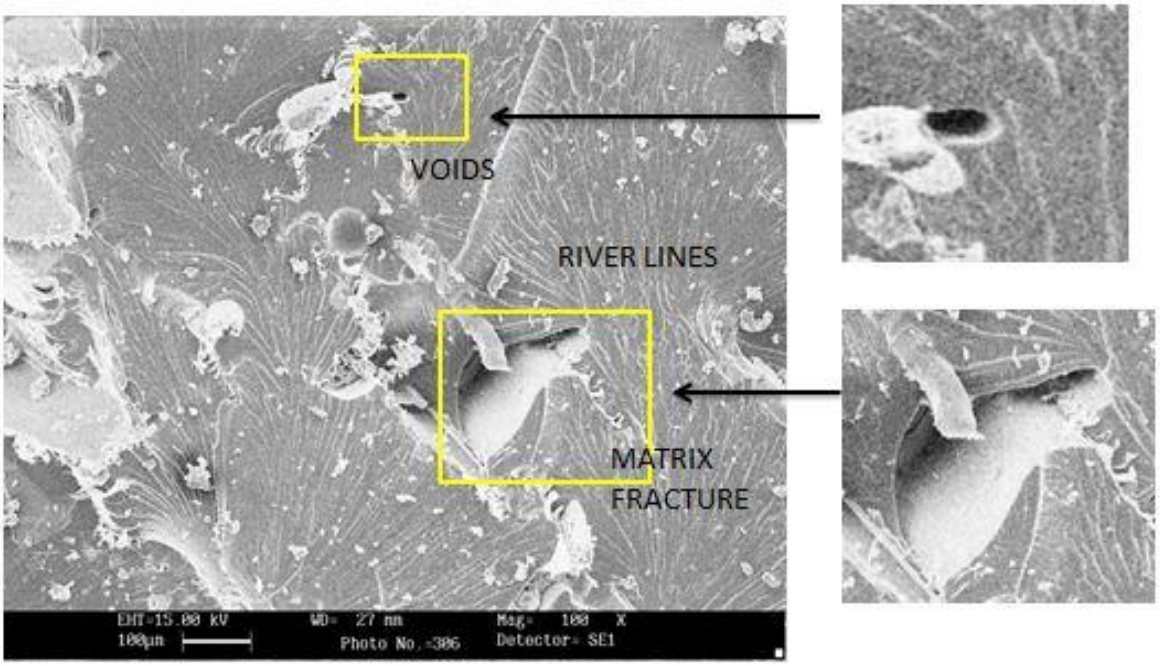


Figure 4.14 (c): SEM image of hybrid composite with 3 wt% extracted residue powder and 5 wt% CFF in epoxy resin at 100X magnification

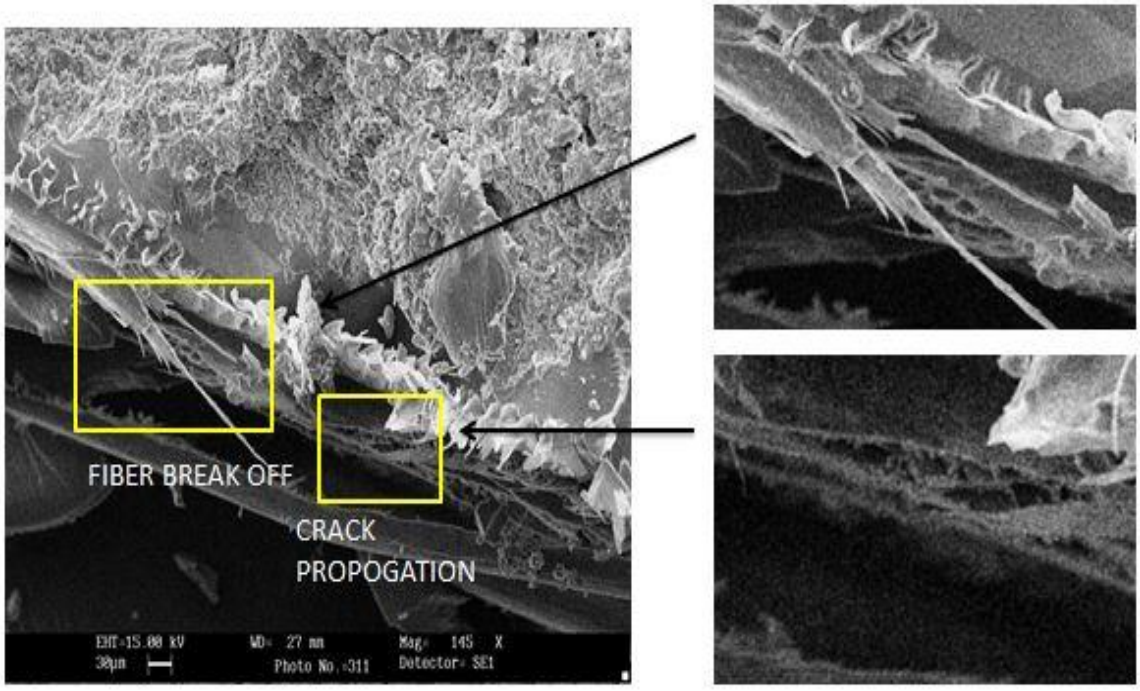


Figure 4.14 (d): SEM image of hybrid composite with 4 wt% extracted residue powder and 5 wt% CFF in epoxy resin at 100X magnification

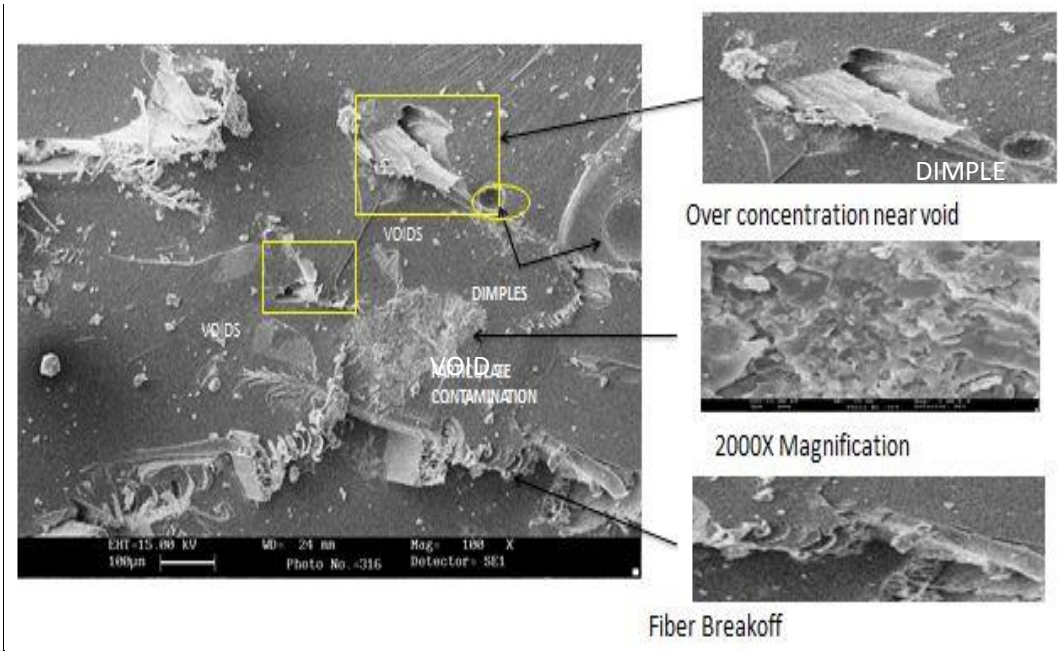


Figure 4.14 (e): SEM image of hybrid composite with 5 wt% extracted residue powder and 5 wt% CFF in epoxy resin at 100X magnification

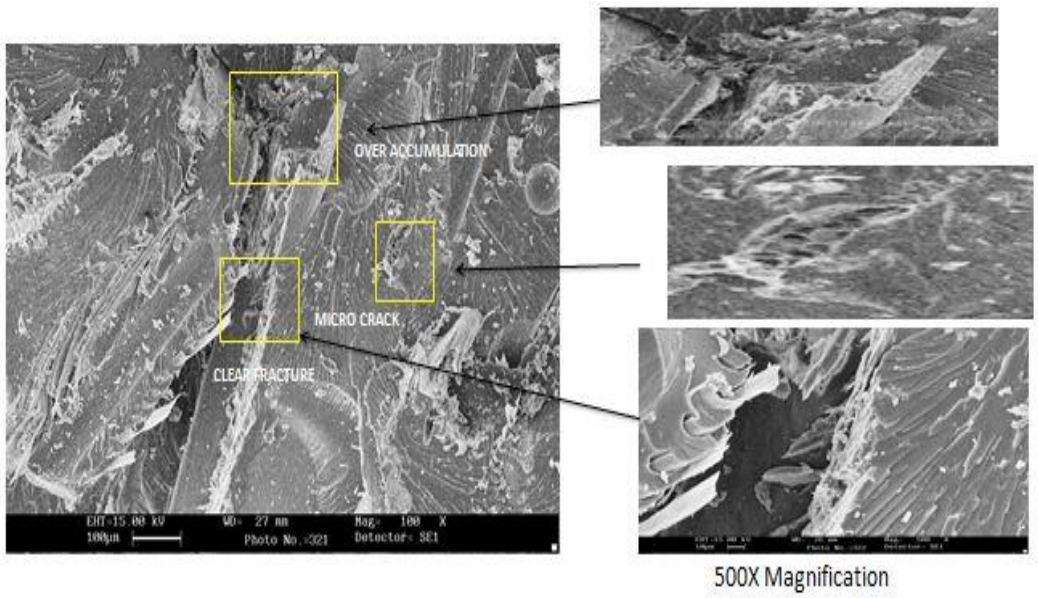


Figure 4.14 (f): SEM image of hybrid composite with 6 wt% extracted residue powder and 5 wt% CFF in epoxy resin at 100X magnification

generation of dimples and fiber breakoff near brittle Fracture zone. At 2000x magnification it was very well observed that the ERP is contaminated at one place and thus leads to material failure. Therefore with higher particulate % the strength starts decreasing.

Figure 4.14 (f) shows the SEM image of the tensile fractured surface of the hybrid composite (with 6 wt% extracted residue powder from Rohu fish as particulate and 5wt% CFF in epoxy resin matrix). Here we can observe that at higher concentration of mixture, the chemical reaction between the matrix and the particulate is showing improper bonding and reactivity decreases. The clear hackles and ribbon zones are visible and therefore reduce the strength of the composite. Accumulation of powder at one place and the generation of micro cracks are visible from the SEM image, which could also be the reason for sudden and brittle fracture at low loads. Therefore reduces bonding attraction and load bearing strength. So finally we can conclude that the 6 wt% ERP in 5 wt% CFF is the saturation state for the current epoxy based hybrid composite fabrication.

4.8 Field Emission Scanning Electron Microscopy (FE-SEM) Test

FE-SEM analysis was conducted to identify the presence of Silica powder and other minerals in the extracted powder from Rohu fish residue. The success of the process was clearly visible through the results obtained after composition analysis through FESEM. The output revealed that the certain percentage of silica powder was present in the extracted powder kept under investigation. From the table 4.6 (a-d) it can be noted that along with silica other elements like Carbon, Oxygen, Sodium, Calcium, and Phosphorous etc. were also present in the obtained powder. The reinforcing use of the extracted powder as particulate in CFF filled epoxy based hybrid composite has improved the cross linking reactivity and bond strength of the composite. The presence of multiple charged elements (like Na +1, Ca +2, P +3, C +4, O -2 etc.) has amended the proper mixing and thus strengthens the developed hybrid composite. Certain compounds like calcium carbonate ($CaCO_3$), silica oxide (SiO_2), sodium hydroxide (NaOH) etc. were also diagnosed. $CaCO_3$ increases the thermal stability and the cross linking density as compared to neat epoxy **(Fan-Long and Soo-Jin, 2009)**. SiO_2 in epoxy resin improves the modulus of elasticity by 10-20% and toughness by 25-30%**.** Therefore it can be undoubtedly concluded that the ERP is a very prestigious material as a reinforcing particulate for epoxy resin. The FE-

SEM images and the composition analysis at different positions and spectrums can be seen in figure 4.15 (a-d).

Table 4.6 (a): ERP composition at spectrum 1 (through FESEM at 685 cts)

Element	Weight%	Atomic%
C	8.86	14.05
O	44.73	53.62
Na	24.00	19.89
Si	2.78	2.53
P	4.20	2.58
Ca	15.43	7.33
Total	100.00	

Table 4.6 (b): ERP composition at spectrum 2 (through FESEM at 1515 cts)

Element	Weight%	Atomic%
C	9.43	14.46
O	46.40	53.72
Na	28.71	23.00
Si	2.61	2.40
P	2.22	1.32
S	1.90	1.09
Ca	8.72	4.01
Total	100.00	

Table 4.6 (c): ERP composition at spectrum 2 (through FESEM at 1515 cts)

Element	Weight%	Atomic%
C	2.82	5.65
O	34.33	52.60
Na	6.36	6.65
Si	2.69	2.59
P	1.37	1.07
Ca	52.42	31.44
Total	100.00	

Table 4.6 (d): ERP composition at spectrum 3 (through FESEM at 1515 cts)

Element	Weight%	Atomic%
C	10.16	16.10
O	40.59	48.67
Na	28.45	23.55
Si	2.47	2.32
P	4.63	2.85
Ca	13.70	6.50
Total	100.00	

4.9 X-Ray Powder Diffraction (XRD) Analysis

Figure 4.16 (a-f) shows the results obtained for X Ray Powder Diffraction test performed at IIC, IITR under 29^0C atmospheric temperature and 51^o RH. The powdered form of the sample is used for XRD testing. Here the output curve displays counts in ordinate axis and two theta scale in abscissa. The variation in gallery spacing (d) with thread angle (2Q) signifies the crystalline or amorphous nature of the sample put under investigation. The sharp and steep peak shows the crystalline nature whereas the smooth and less steep peak shows the amorphous nature of the material.

Table 4.7: XRD characterization of various ERP compositions in hybrid biocomposite

Designation	Counts at Peak	Gallery Spacing "d" (in Å)	Thread Angle "2Q" (in degrees)
ERP 1	980	4.57	19.41
ERP 2	1070	4.50	19.56
ERP 3	1048	4.36	19.70
ERP 4	1025	4.34	19.51
ERP 5	1080	4.77	18.58
ERP 6	880	4.75	18.67

From the obtained XRD spectra, we can observe that the curve between 17^0 to 22^0 thread angles has the smooth trend that shows the non crystalline nature of the CFF and ERP filled, epoxy based hybrid composite. The decrease in gallery spacing and the increase in thread angle together results in the better interface bonding between the matrix and the reinforcing materials. The minimum value of d between 2-4 wt% of ERP shows the optimum cross linking characteristic of the developed hybrid composite which results in better tensile strength, impact strength and flexural strength as well (figure 4.16(g)).

Therefore, the XRD analysis was termed fruitful to identify the morphological nature and the exact reason for the trends obtained for mechanical characterization of the prepared samples. The XRD characterization with number of counts at peak, gallery spacing and corresponding thread angle is shown in table 4.7.

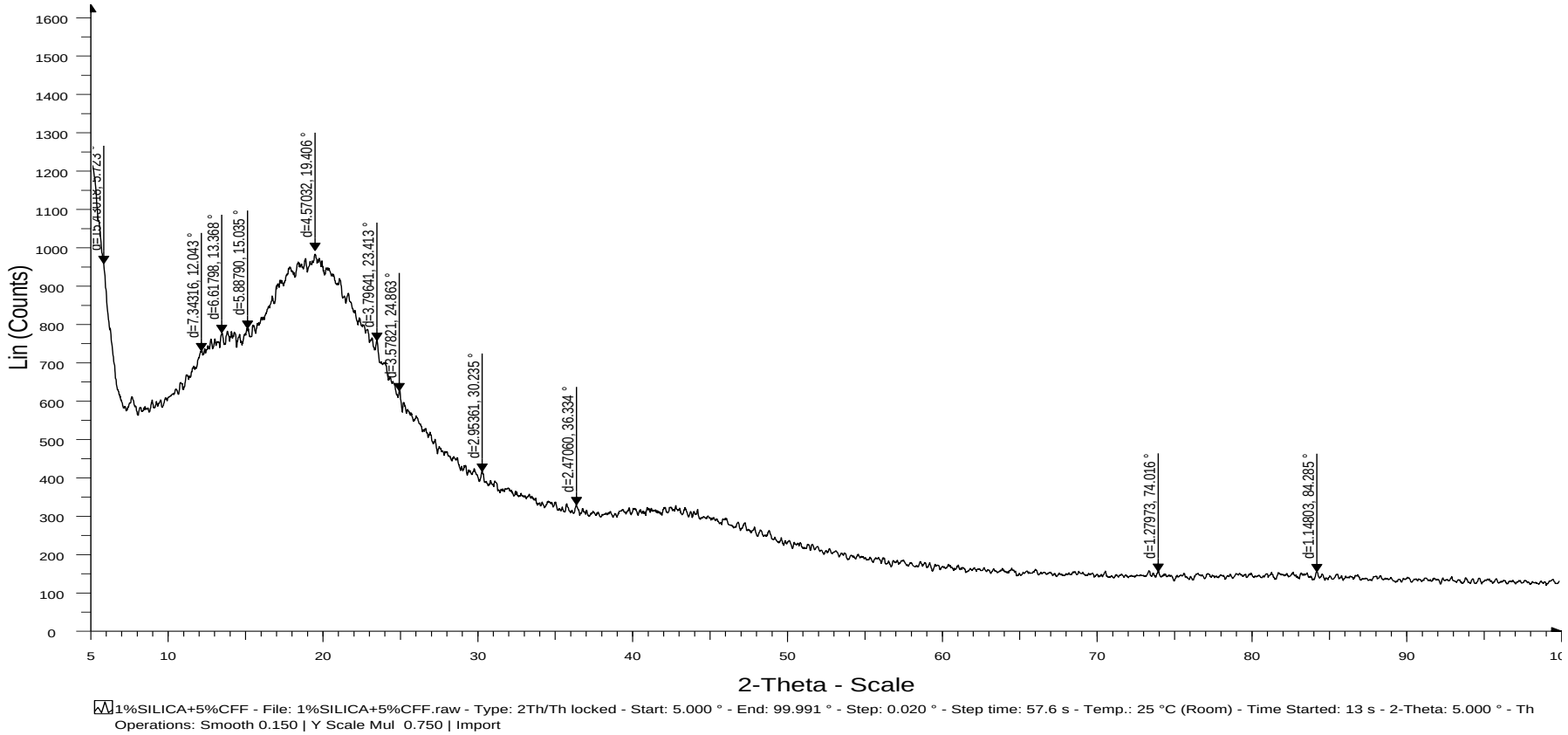


Figure 4.16 (a): XRD Curve for 1 wt% ERP in 5 wt% of CFF reinforced hybrid Biocomposite

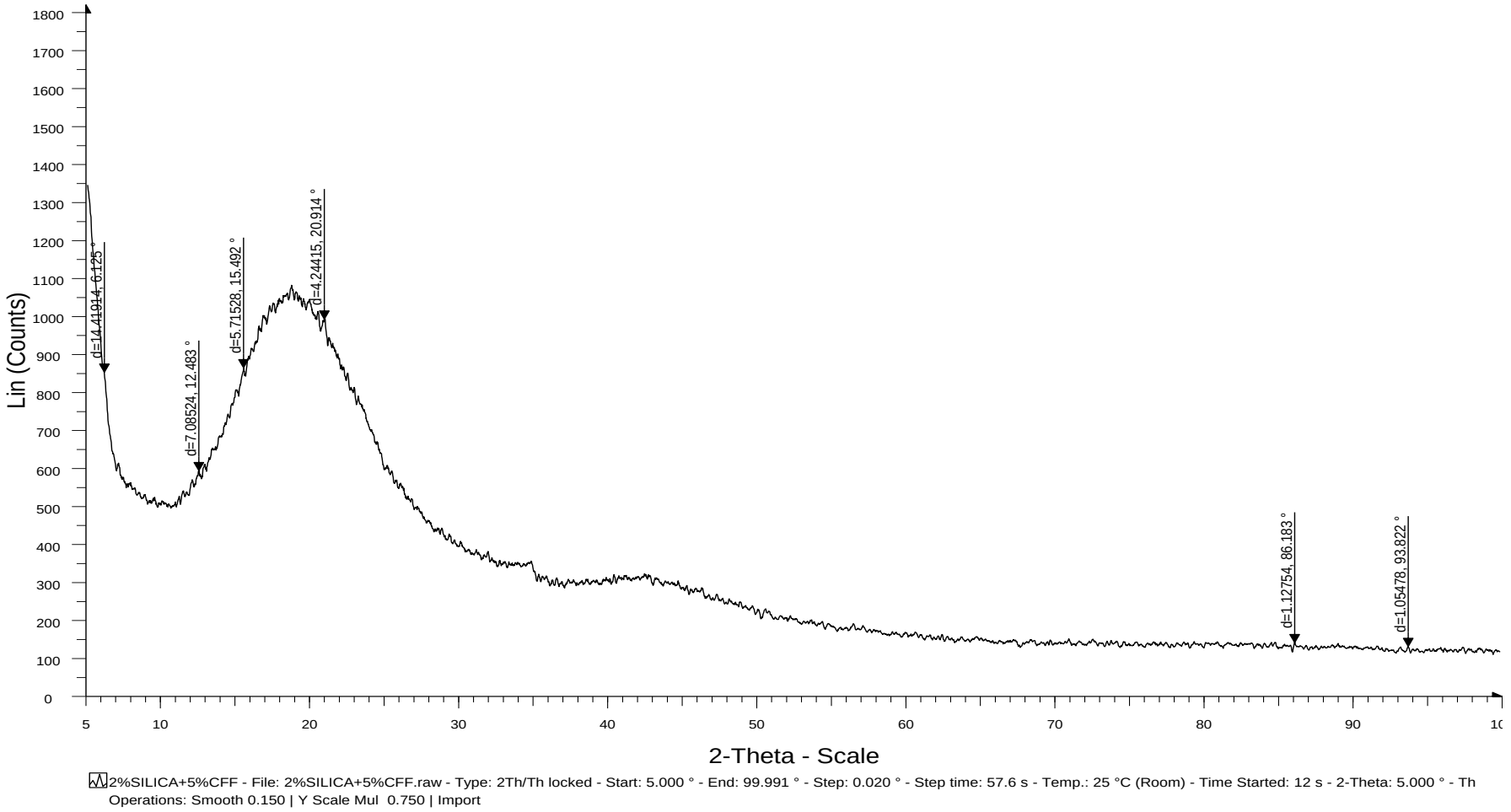


Figure 4.16 (b): XRD Curve for 2 wt% ERP in 5 wt% of CFF reinforced hybrid Biocomposite

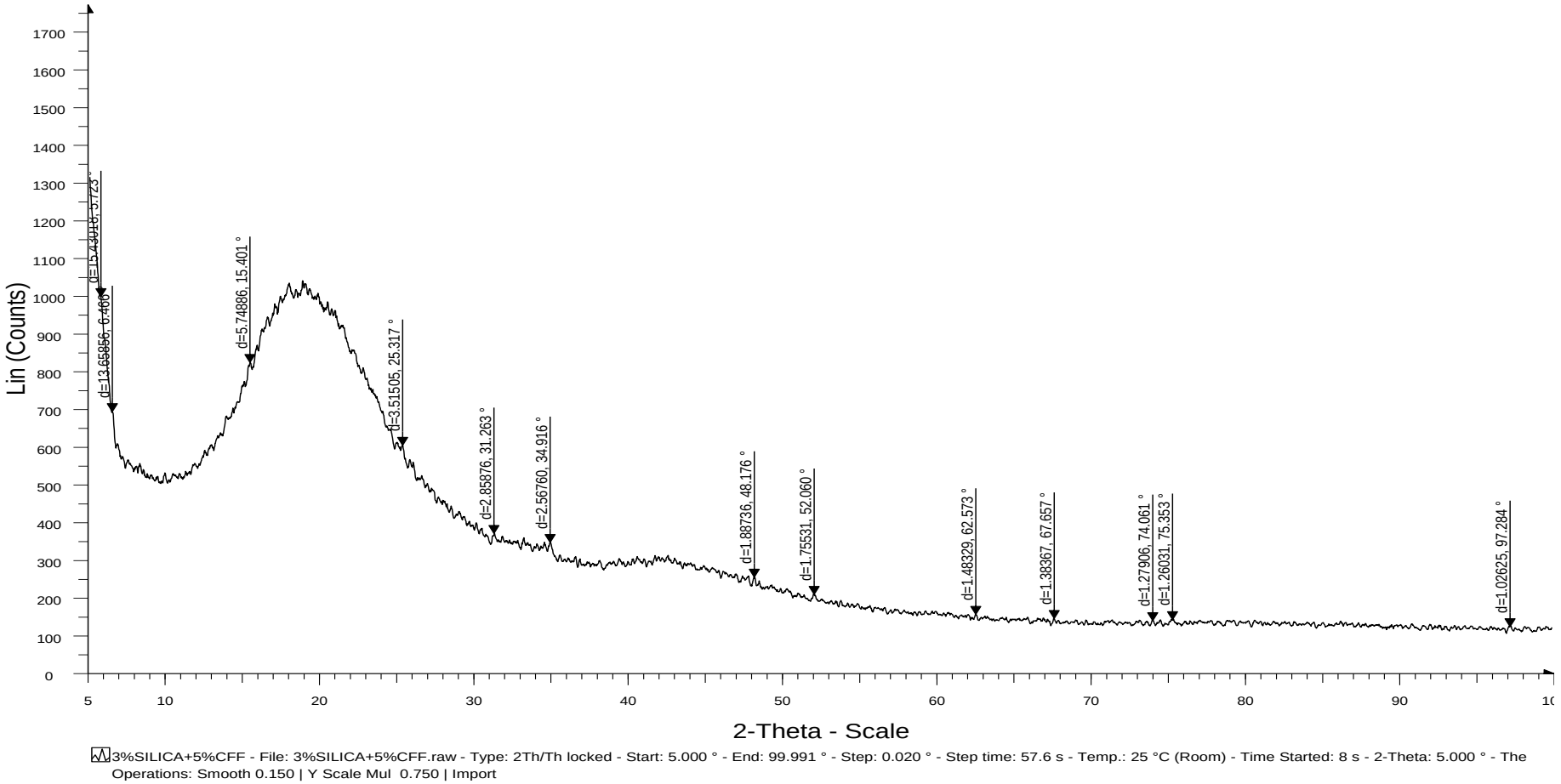


Figure 4.16 (c): XRD curve for 3 wt% ERP in 5 wt% of CFF reinforced hybrid Biocomposite

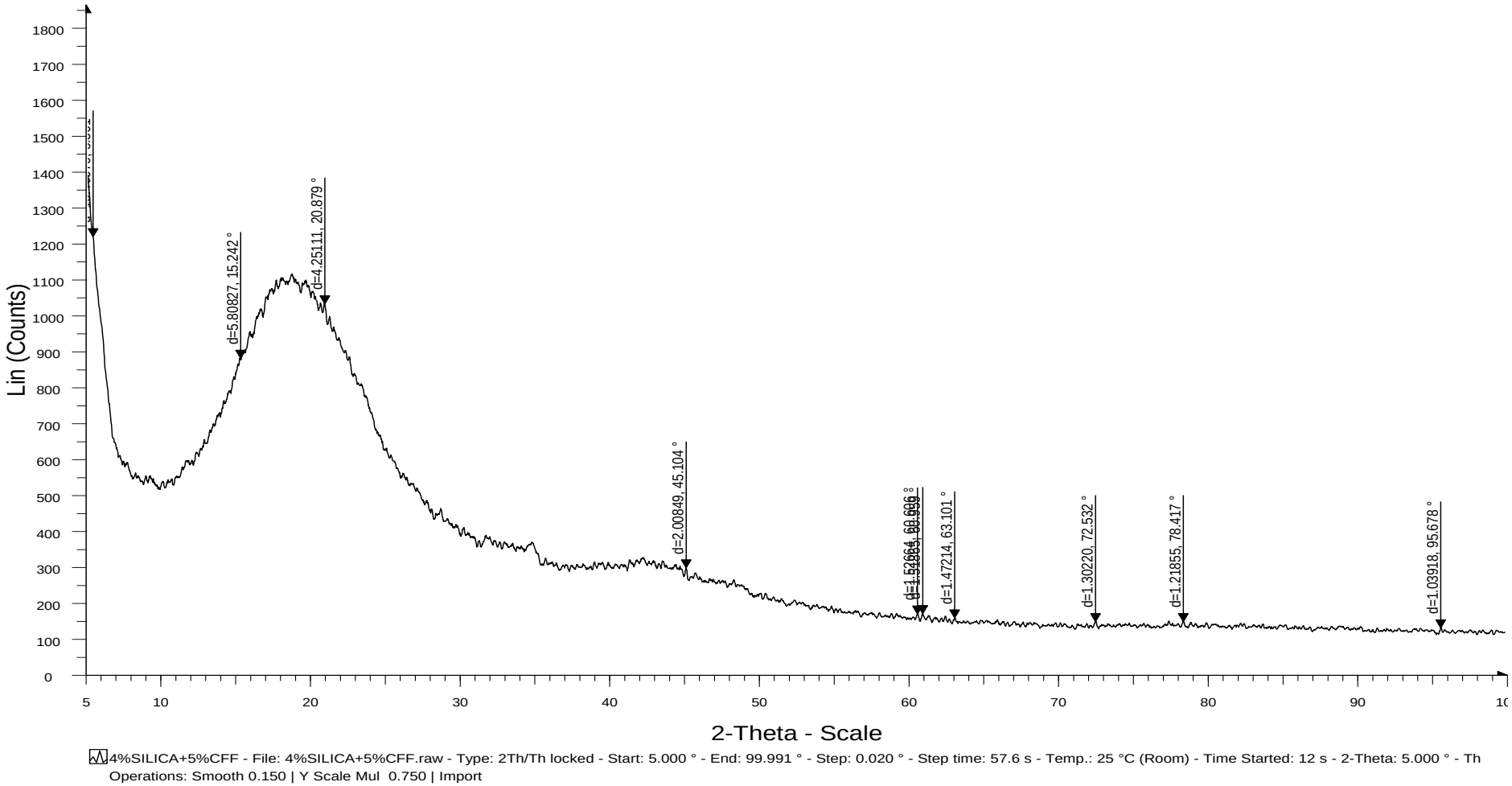


Figure 4.16 (d): XRD Curve for 4 wt% ERP in 5 wt% of CFF reinforced hybrid Biocomposite

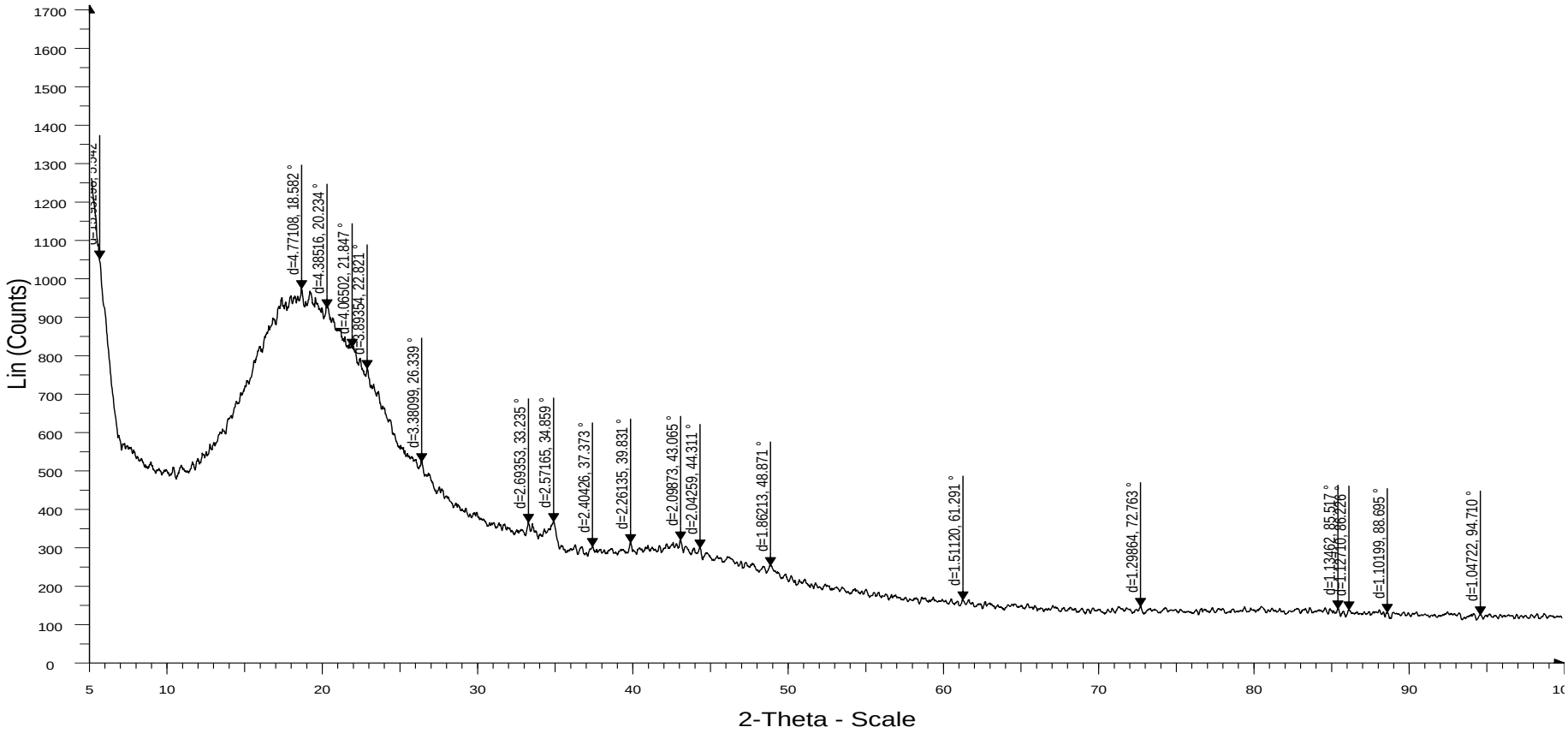


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Figure 4.16 (e): XRD Curve for 5 wt% ERP in 5 wt% of CFF reinforced hybrid Biocomposite

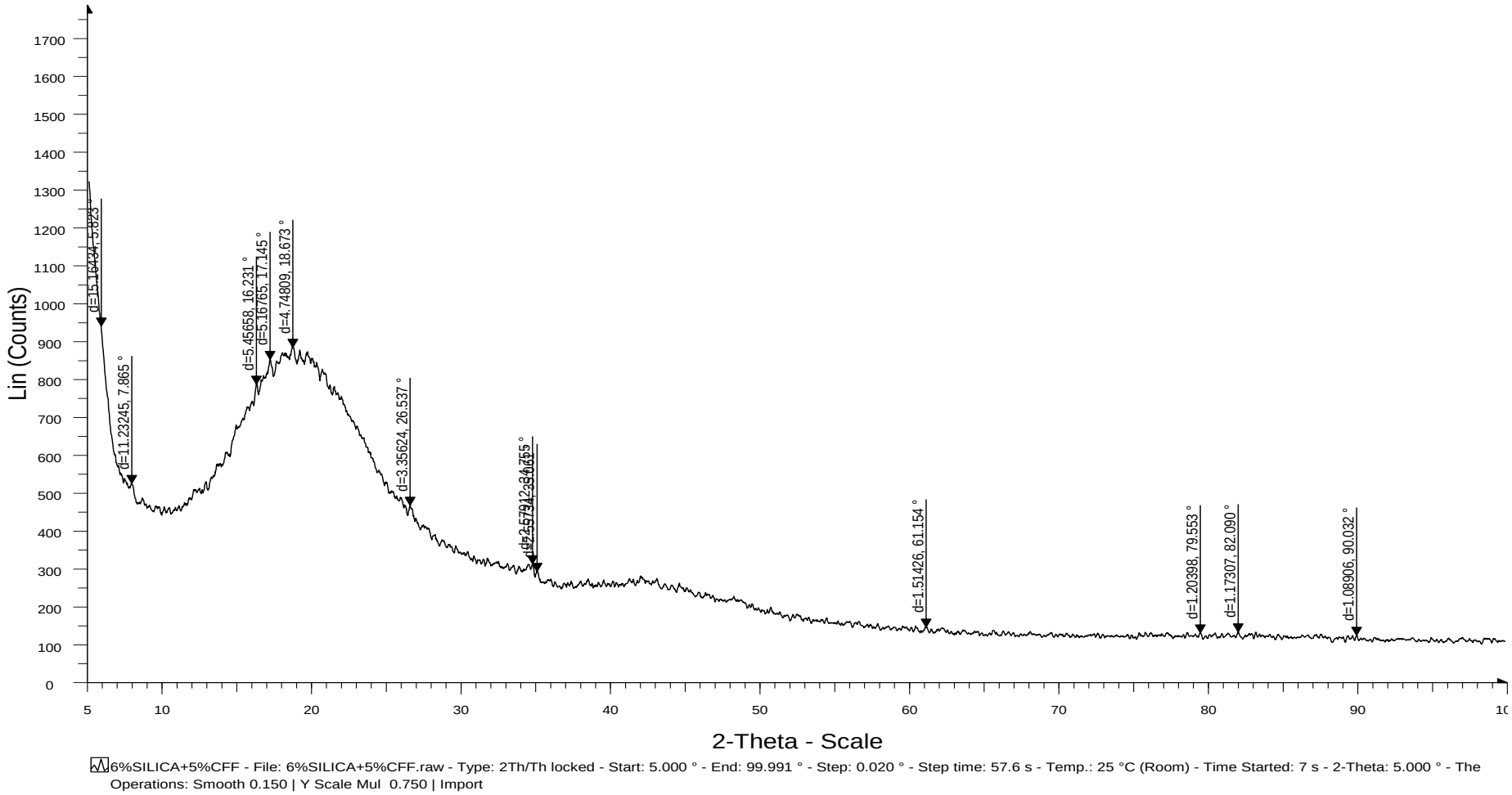


Figure 4.16 (f): XRD Curve for 6 wt% ERP in 5 wt% of CFF reinforced hybrid Biocomposite

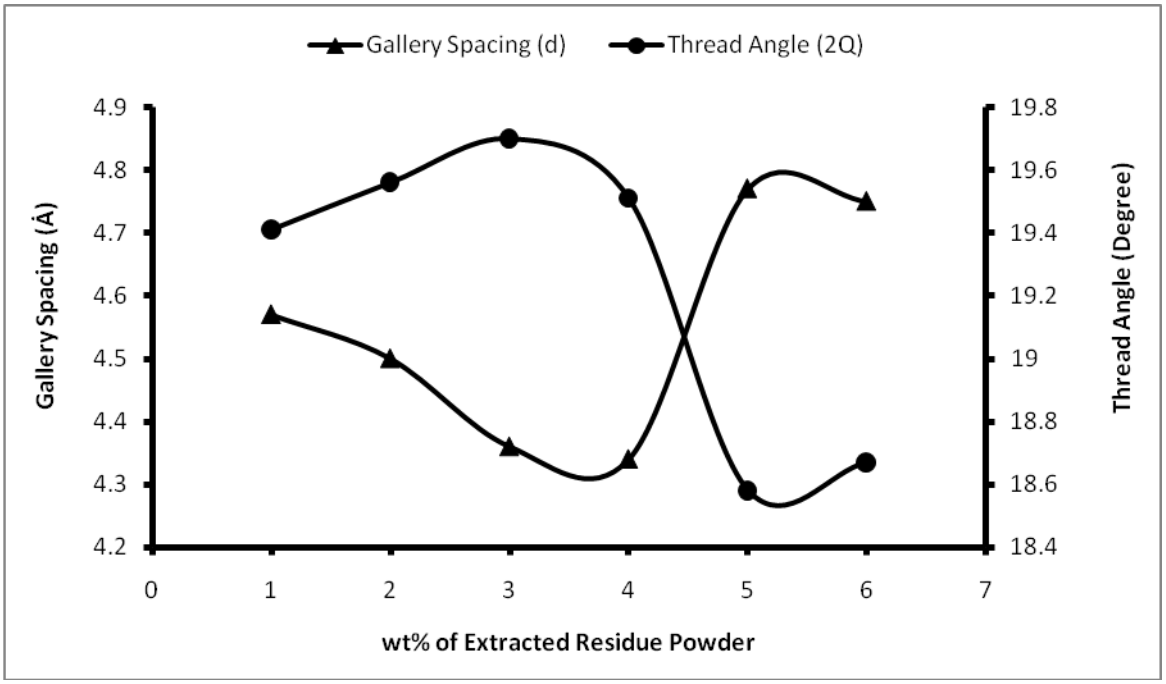
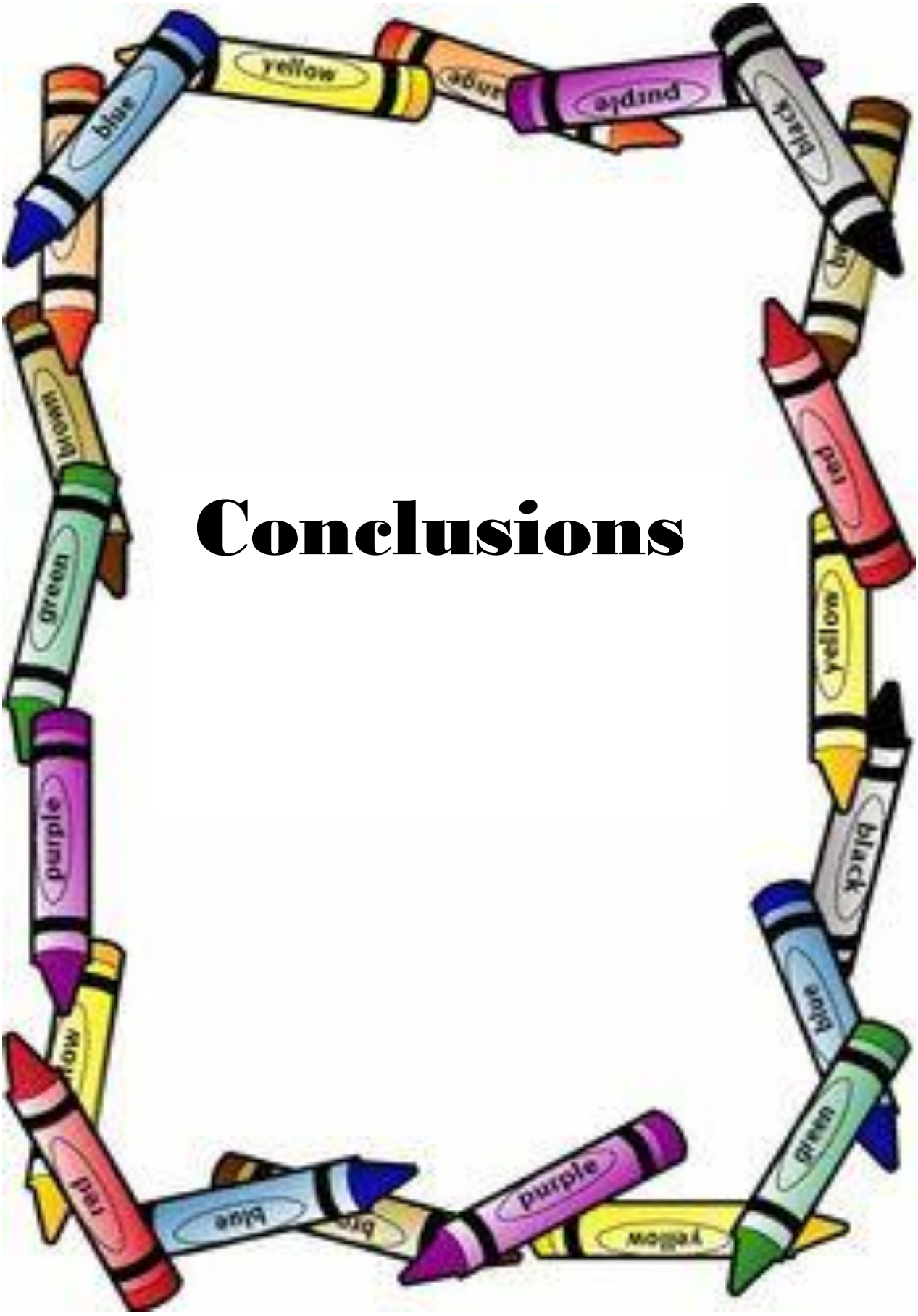


Figure 4.16 (g): XRD results analysis for varying wt% of ERP filled hybrid composite


Conclusions

Chapter.5 SUMMARY AND CONCLUSION

5.1 Summary

Fabrication and thermo-mechanical characterization of epoxy based composites was performed in the present research work. Among all the Epoxy Resin based composites developed with varying weight percentage of chicken feather fiber reinforcement i.e. 0 wt% (control sample) to 7 wt% of CFF, the 5 wt% of CFF in cured epoxy resin was the most appropriate composition based on the output results of hardness and impact strength and for hybrid biocomposite development with extracted powder from Rohu fish by varying wt% of particulate from 0 wt% (i.e. 5 wt% CFF) to 6 wt% of reinforcing particulate the 3 wt% of particulate gave the most optimized results during the entire investigation of hybrid composite characterization. The depiction of mechanical properties is done through various tests like tensile test, compression test, flexural test, hardness test, impact test etc. the thermal analysis is done using TGA/DTA. For morphology SEM, XRD and FE-SEM were appreciated. The entire thesis helps to get the deep knowledge about the material used, methods adopted, process required for complete development of material, its testing procedure and the systematic standardization.

5.2 Conclusions to present Research Work

1. The appearance of CFF based composite can be altered in color by the use of color variant of chicken feather in the composition. But the properties may vary. Therefore the developed composite can be used as decorative light weight material.

2. The density i.e. weight per unit volume of CFF based composite decreases with the increase in weight percentage of CFF in epoxy resin and thus provide light weight application. Also with the rise in wt% of particulate (extracted residue powder from fish) the density of the hybrid composite rises and it reaches close to density of cured epoxy resin at 3 wt% of ESP in 5 wt% CFF filled composite.

3. Water absorption and thickness swelling test revealed that the developed CFF based material has good adhesion fiber-reinforcement bonding on hybrid composite development. The results showed rise in absorption and thickness with increasing wt% of cff and the values showed linear decreasing trend on inclusion

of extracted residue powder (particulate). The rate of water absorption and thickness swelling becomes almost constant after certain interval of time. The water absorption is due to void generation which was unavoidable during hand ley-up mixing technique and it was seems to be reduced by particulate reinforcement.

4. The mechanical testings of CFF filled composite showed poor tensile strength and was continuously decreasing with cumulative CFF wt%. The compression strength was also decreasing but showed better results as compared to neat epoxy sample. The enhanced impact and hardness characteristics were achieved for initial samples (varying wt% of CFF in cured epoxy resin) with maximum value at 5 wt% CFF jam-packed composition. It was therefore termed as the judging parameter before fabricating hybrid composites. Later, the enhanced mechanical properties were detected with improved tensile, Compression, flexural, impact and hardness strengths close to 3 wt% of Extracted Powder as particulate reinforcement.

5. The thermal analysis of hybrid composites (1 to 6 wt% ESP) was performed using output graphical representation of TGA/DTA Curve. The separate DTA, DTG and TG curve for every sample gave the complete information about the decomposition rate, thermal characteristics and thermal stability. The 1 wt% ESP reinforced hybrid composite showed maximum burying temperature of 592^0C for complete combustion. The initial decomposition temperature and time required for complete concealing of the composite was spotted through the exploration.

6. The elaborative and clear morphological investigations of the prepared hybrid composite samples were done using Scanning Electron Microscopy (SEM) analysis. The SEM images of tensile fractured surface gave the appreciable interpretations for the cause of tensile failure and the output trends of various mechanical Tests. The fiber orientation, inclusion uniformity, grain flow, voids, denseness, fiber failure (breaking, tearing, pull-off etc.) can be understand using SEM test.

7. The Field Emission Scanning Electron Microscopy (FESEM) test was performed on the extracted white residue ash from Rohu Fish. The obtained results revealed the presence of inert element silica along with various other elements like calcium, phosphorous, sodium, carbon etc. The main aim of performing FESEM was to

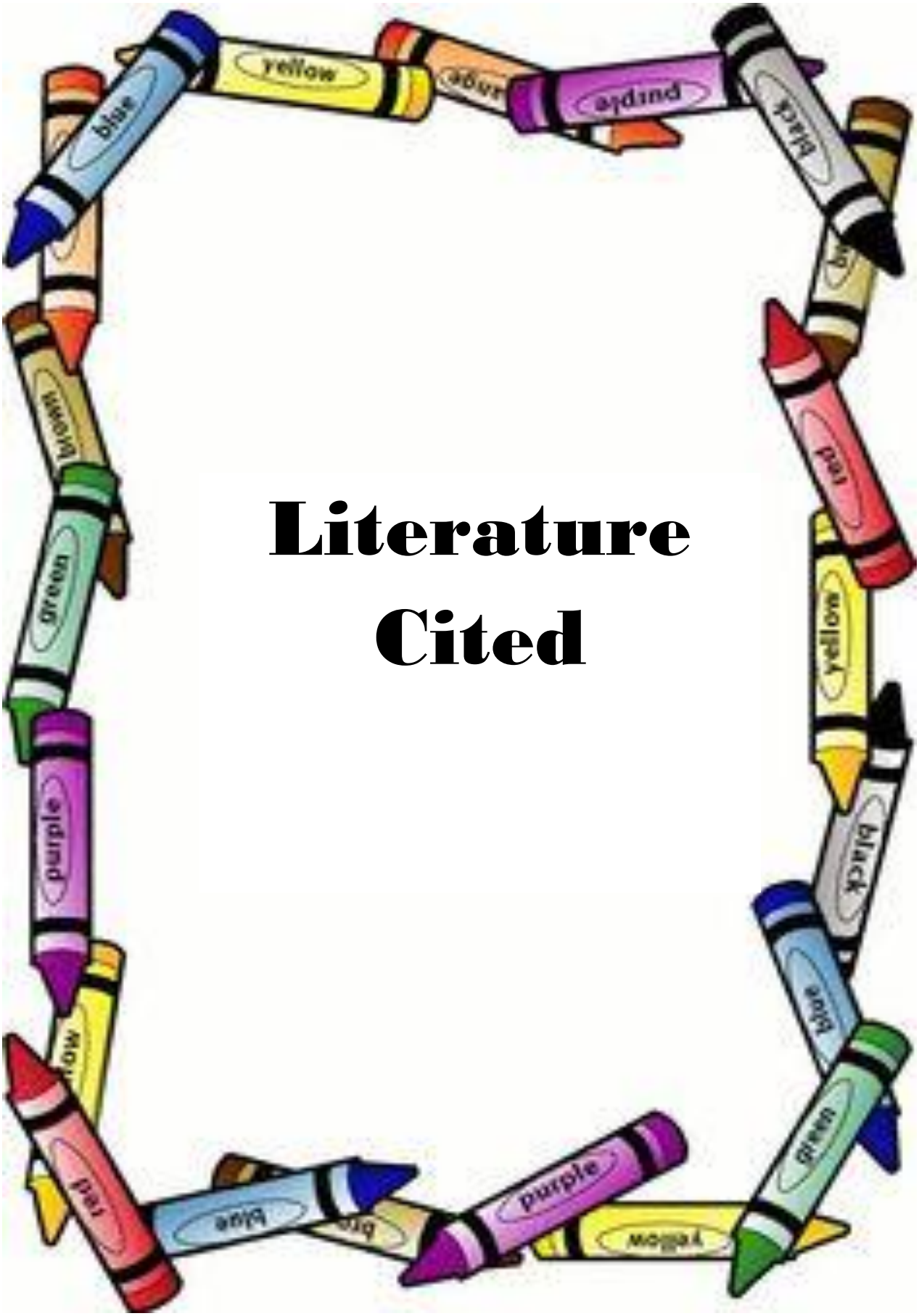
identify the composition of extracted powder and its bonding characteristics on treating it as reinforcing material in hybrid composite fabrication.

8. The CFF and multi elemental powder particulate filled polymeric composite was diagnosed for crystallography using XRD Technique. The gallery spacing, thread angle and the nature of peak (curve movement) obtained concludes the clear amorphous character of the developed material. The nature of the XRD results showed almost the similar trend for all the compositions.

5.3 Recommendations for Future Scope in the current research

From the conclusions drawn above and the entire brain storming on the Epoxy Resin-CFF-Fish Residue based research, much more extension in the present investigation can be done. Some of the future scope may include the following:

- Nano particulate extraction can be done for developing more compact and highly rigid composites.
- Apart from chicken's hairy feathers the barbs, raches and other parts of the feather can be utilized to enhance livestock waste management.
- Matrix manipulation can be done to characterize the fiber compatibility.
- Characterization based on tribology, aerology and some specific application can be done.
- Size of fiber and particulate, orientation of feathers, particulate uniform dispersion, mixing, and fabrication techniques can be modified and then characterize the developed hybrid composite.



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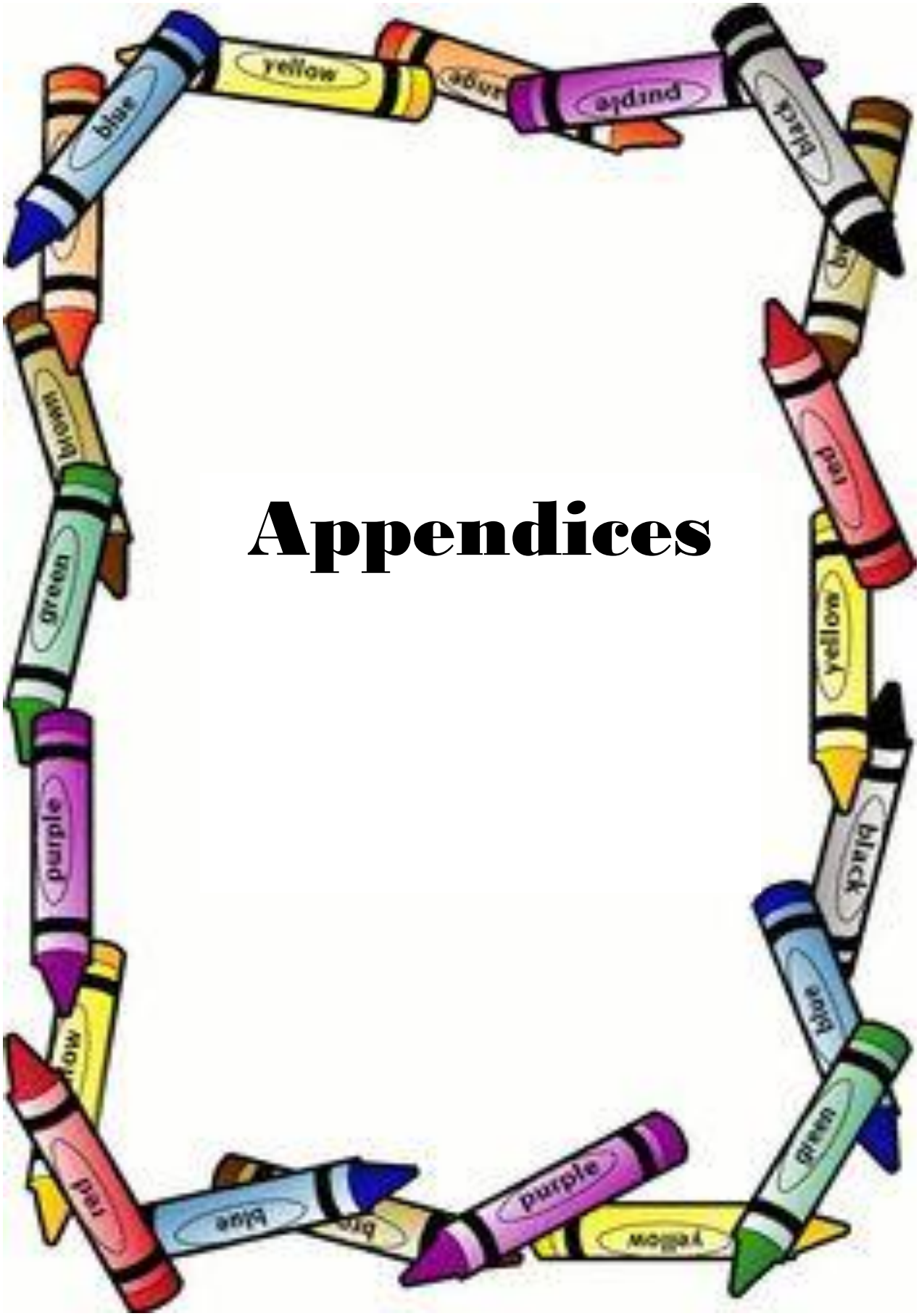
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Appendices

APPENDIX A

Table A1: Density variation with varying wt% of chicken feather fiber

Percentage of CFF	Weight of Specimen(gm)	volume of water displaced(mL)	Density (kg/m^3)
0	116.88	101.2	1154.940711
1	115.21	105	1097.238095
2	112.11	107	1047.757009
3	110.5	109	1013.761468
4	109.23	110	993
5	105.99	112	946.3392857
6	103	113	911.5044248
7	100.48	113	889.2035398

Table A2: Density variation with varying wt% of extracted fish powder

Percentage of silica	Weight of Specimen(gm)	volume of water displaced(mL)	Density (kg/m^3)
0.00	105.99	112	946.34
1.00	110	111	990.99
2.00	112.11	109	1028.53
3.00	115.42	99.12	1164.45
4.00	118.25	98.7	1198.07
5.00	121.78	97	1255.46
6.00	124	95.9	1293.01

Table A3: Thermal Analysis of hybrid composite with 5 wt% CFF and varying ERP wt%

Sample	DTG		DTA Endogram		TG weight loss % at 200°C	TG weight loss % at 400°C
	peak temperature, °C	Rate, mg/min	°C	Heat of fussion (-ΔH), J/mg		
ERP 1	251	2.64	464(275.1μV)	1.30	16	65
ERP 2	262	4.3	452 (648μV)	3.85	13	67.3
ERP 3	262	4.4	450(633μV)	3.65	13	67
ERP 4	258	5.0	446(724μV)	4.89	12.5	70
ERP 5	257	4.3	445(780μV)	5.27	12	80
ERP 6	256	4.8	440 (569μV)	3.28	12	74.9

APPENDIX B

B.1 Procedure of tensile testing Using (AMT-SC Model) UTM

- Prepare the flat tensile specimen based on desired standard.
- Switch on the servo UTM machine to the tensile testing mode and the computer to the servo test platform.
- Clamp the specimen at the tensile fixture as shown in figure 3.13 (b).
- In the tensile platform in the computer, make fresh stock entry with separate name say "TENSILE" and add all the required information in the space provided.
- Select stock icon on the task bar and open the stock "TENSILE" and click update.
- Now click on refresh button on the UTM or computer and then press start button. The test will start and the displacement v/s load variation can be seen on the desktop screen.
- After tensile fracture occurs, save the test for future use and characterization using EXCEL.

B.2 Procedure of compression testing Using (AMT-SC, 2008 model) UTM

- Prepare the compression specimen based on desired standard.
- Switch on the servo UTM machine to the compression testing mode and the computer to the Servo test platform.
- Place the specimen between the compression fixture as shown in figure 3.14 (b).
- In the compression Platform in the Computer, make fresh stock entry with separate name say "COMPRESSION" and add all the required information in the space provided.
- Select stock icon on the task bar and open the stock "COMPRESSION" and click update.
- Now click on refresh button on the UTM or computer and then press start button. The test will start and the displacement v/s load variation can be seen on the desktop screen.
- After compression failure occurs, save the test for future use and characterization using EXCEL.

B.3 Procedure for impact testing using IMPACT TESTING MACHINE (Manufactured by AE, India)

- Prepare the impact testing specimen based on desired standard.
- Clamp the specimen at the fixture as shown in figure 3.15(d).
- Switch on the impact testing machine.
- Press "8" button the machine and select hammer, length, width and thickness.
- Press "4" to return and then you are ready for the test.
- Press "7" to start the hammer movement and the sudden impact will break the specimen.
- The impact energy, impact strength, impact gradient etc. will be visible on the screen.

Note the readings and characterize the results for various compositions.

B.4 Procedure for Rockwell hardness test using digital hardness testing machine.

- Prepare the good surface finished specimen based on desired standard.
- Select scale L, initial load 60 kgf, ball type indenter of dia ¼" for the indentation.
- Place the surface finished specimen on the plate as shown in figure 3.16.
- Initially, keep the lever in the unloading condition and rotate the wheel arm till the HRL value reaches 300.
- Wait for 3 sec and then apply major load with the help of lever.
- The dwelling time starts and decrease in the HRL value is recorded.
- At the beep sound unload the specimen with the lever.
- Re-rotate the arm lever very slowly and then the final HRL value is recorded.
- Repeat the same procedure at different locations of the same specimen and select the mean value as an optimum result.

B.5 Procedure of flexural testing using (AMT-SC, 2008 model) UTM

- Prepare the flexural specimen based on desired standard.
- Switch on the servo UTM machine to the flexural testing mode and the computer to the Servo test platform.
- Place the specimen on the fixture like the simply supported beam as shown in figure 3.17 (b).

- In the flexural platform in the Computer, make fresh stock entry with separate name say "FLEXURAL" and add all the required information in the space provided.
- Select stock icon on the task bar and open the stock "FLEXURAL" and click update.
- Now click on refresh button on the UTM or computer and then press start button. The test will start and the displacement v/s load variation can be seen on the desktop screen
- After bending failure occurs, save the test for future use and characterization using EXCEL.

B.6 Procedure for determining experimental density of the composite material.

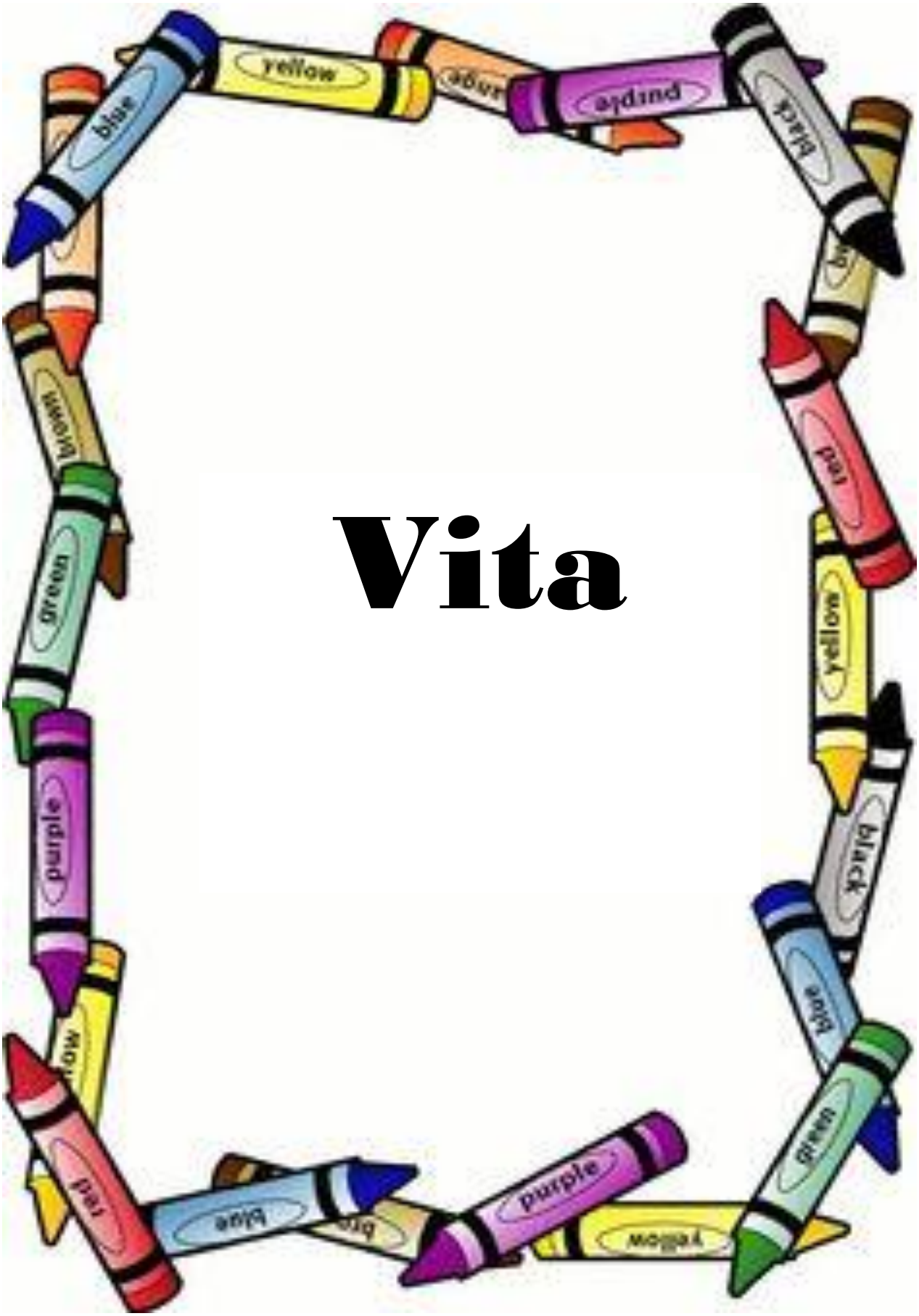
- Take the small amount of sample (irregular shape) and weigh it using digital weight measuring machine.
- Take a measuring flask of 500mL and add around 250mL of water in it.
- Dip the weighted sample inside the beaker which will raise the height of water inside the flask.
- Exactly record the rise in the volume of water inside the flask.
- The volume of water displaced is equal to the volume of the sample dip in the flask.
- Use the formula given in equation 3.10.

$$\boldsymbol{Density = \frac{W*10^{-3}}{V*10^{-6}} = \left(\frac{W}{V}\right) * 10^3\, kg/m^3} \qquad \textbf{... (3.10)}$$

Where, W = weight of Sample (gm)

V = Volume of water displaced (mL)

Vita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VITA

The author, Gagan Bansal, was born on 2nd September 1991 in Rudrapur (U. S. Nagar), Uttarakhand. He belongs to businessman family. He passed his High School in 2004 and Intermediate in 2006 from Holy Child School (CBSE Board). He earned his Bachelor's degree (With Honours,) in Mechanical Engineering from Graphic Era University, Dehradun in 2012. He Joined Graphic Era University in 2012 and served the university for 2 years. During his tenure at GEU the author pursued the MBA in Human Resource Management from SHIATS University (Former Allahabad Agriculture Institute) by distance education. In 2014 he took admission in the College of Post Graduate Studies at G. B. Pant University of Agriculture and Technology, Pantnagar for Master's degree in Mechanical Engineering with major in Design and Production Engineering.

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Thesis Title	**:** Thermo-Mechanical Characterization of Epoxy Hybrid Composite Reinforced with Chicken Feather Fiber and Fish Residue Ash Particulates		
Advisor	**:** Dr. V. K. Singh		

ABSTRACT

In the current thesis work the experimental investigation for the thermo-mechanical characterization of the developed composite is done. Here the effect of Chicken feather fiber in Epoxy Resin is diagnosed with varying weight percentages (0 to 7 %). Later 5 wt% CFF in varying wt% of extracted residue powder form fish was used as a particulate to develop hybrid composite.

The different wt% of ERP in 5 wt% CFF filled epoxy based hybrid composite was casted. Tests have been conducted on 100 kN servo-hydraulic universal testing machine under displacement mode of control, digital Rockwell hardness testing machine and impact testing machine were used for mechanical testing, TGA/DTA have been conducted for thermal properties. XRD was used for crystallography analysis.

Mechanical test results indicate that the mechanical properties of the composite, including compression strength, flexural modulus, impact strength, flexural strength and hardness etc. are enhanced by the increment of the weight percentage of ERP. The rate of water absorption increases with the increase in weight percentage of ERP. Thickness swelling property is increased initially with time and it becomes almost constant for all wt% of ERP. On the basis of overall study of the epoxy resin as matrix, ERP as particulate and CFF as fiber, the 3 wt% ERP and 5 wt% CFF combination is found to be better than all other combinations.

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Advisor

(Gagan Bansal)
Author

नाम : गगन बंसल **परिचायंक संख्या** : ४७०५३

षठमास एवं प्रवेश वर्ष : प्रथम, २०१४-१५ **उपाधि** : स्नातकोत्तर
(यांत्रिकी अभियांत्रिकी)

मुख्य विषय : परिकल्पना एवं उत्पादन अभियांत्रिकी **विभाग** : यांत्रिकी अभियांत्रिकी

सलाहकार : डा० विनय कुमार सिंह

शोध ग्रंथ शीर्षक : थर्मो - यांत्रिक निस्पण विशेषता इपोक्सी संकर समग्र प्रबलित मुर्गो के पंख के रेशे और मछली अवशेष राख कर्ण के साथ ।

सारांश

वर्तमान शोध में प्रयोगात्मक जांच द्वारा विकसित समग्र के थर्मा-यांत्रिक लक्षण जांचे गए है । यहाँ मुर्गो के पंख का प्रभाव इपोक्सी रेसिन में अलग-अलग वजन प्रतिशत (0-7%) में निदान किया गया है। बाद में 5 भार% सीफफ में निकाले गए मछली के अवशेष पाउडर कण के रूप में बदलते भार% को संकर समग्र विकास के लिए इस्तेमाल किया गया है।

ईआरपी के विभिन्न भार% को 5 भार% सीफफ में इपोक्सी रेसिन आधारित हाइब्रिड समग्र की ढलाई मैं इस्तमाल किया गया । 100 केएन इमदादी-हाइड्रोलिक यूनिवर्सल परीक्षण मशीन द्वारा डिस्प्लेसमेंट कंट्रोल मोड पर परीक्षण आयोजित किया गया है । डिजिटल रॉकवेल कठोरता परीक्षण मशीन और प्रभाव का परीक्षण मशीन यांत्रिक परीक्षण के लिए इस्तेमाल किया गया, टीजीए / डीटीए थर्मल संपत्तियों के लिए आयोजित किया गया है । अक्सआरडी क्रिस्टलोग्राफी विश्लेषण के लिए इस्तेमाल किया गया है ।

यांत्रिक परीक्षण के परिणाम से संकेत मिलता है कि समग्र के यांत्रिक गुणों , संपीड़न ताकत , फ्लेक्सुरल मापांक, प्रभाव शक्ति, फ्लेक्सुरल शक्ति और कठोरता आदि सहित ईआरपी का वजन प्रतिशत की वेतन वृद्धि से बढ़ा रहे हैं । जल अवशोषण की दर ईआरपी का वजन प्रतिशत में वृद्धि के साथ बढ़ जाती है। मोटाई सूजन गुण ईआरपी के सभी भार% समय के साथ शुरुआत मैं बढ़ती है तथा बाद में लगभग स्थिर हो जाती है। संपूर्ण अध्ययन के आधार पर इपोक्सी रेसिन मैट्रिक्स, ईआरपी कण और सीफफ के रेशे के रूप में 3 भार% ईआरपी और 5 भार% सीफफ संयोजन अन्य संयोजन की तुलना में बेहतर हो पाया है ।

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