Properties of Epoxy Composites Reinforced with Multi-walled Carbon Nanotubes

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***Abstract*: Plastic composite material has been in to frontier of research as one of the new competitive materials in engineering. Especially, Particle reinforced plastic is a relatively new class of composite material manufactured from particles, nanoparticles and resins, and has proven efficient and economical for the development and repair of new and deteriorating structures. In this project, we report the mechanical properties of epoxy composites strengthened with Multi walled carbon nanotubes. Different composition (0.1%, 0.5%, 1%, 1.5%) of Multi Walled Carbon Nanotubes (MWCNT) are mixed with epoxy resin and castings are prepared by moulding technique. The different mechanical properties such as Tensile strength, Flexural strength and Hardness are evaluated at room temperature. One set of samples are immersed in salt water for moisture absorption and strength degradation studies. The mechanical properties are determined and are compared with that of dry specimens. The Results show that the Mechanical Properties increases with the increase in Percentage of MWCNT for the prepared Composites.**

***Keywords*- Multiwalled Carbon Nanotubes, Epoxy, resin, flexural strength, hardness , tensile strength**.

I. INTRODUCTION

Following the technological developments, industries are searching for lighter weight, higher strength and safer material to meet the demands of structural designs and for economic benefit. In order to extend the application area of plastics, plastic composites are developed by adding reinforcement materials to the polymer matrix. Some of the reinforcements used in structural and industrial applications are Carbon, Aramid, Silicon oxide nano clay and Glass fibers. Plastic composite material has therefore become one of the new competitive materials in engineering. Particulate reinforced plastic is a relatively new class of composite material manufactured from fibers, nano particles and resins, and has proven efficient and economical for the development and repair of new and deteriorating structures. Their mechanical properties make them ideal for widespread applications in various industries worldwide.

Polymer Nanocomposites (PNC) consists of a polymer or copolymer having nanoparticles or nanofillers dispersed in the polymer matrix. These may be of different shape (e.g., platelets, fibers, spheroids), but at least one dimension must be in the range of 1–50 nm. These PNC's belong to the category of multi-phase systems (MPS, viz. blends, composites, and foams) that consume nearly 95% of plastics production. These systems require controlled mixing/compounding, stabilization of the achieved dispersion, orientation of the dispersed phase, and the compounding strategies for all MPS, including PNC, are similar.

Carbon Nanotubes (CNTs) are allotropes of carbon with a cylindrical nanostructure. Nanotubes have been constructed with length-to-diameter ratio of up to 132,000,000:1significantly larger than for any other material. These cylindrical carbon molecules have unusual properties, which are valuable fornanotechnology, electronics, optics and other fields of materials science and technology. In particular, owing to their extraordinary thermal conductivity and mechanical and electrical properties, carbon nanotubes find applications as additives to various structural materials.

The term 'epoxy' refers to a chemical group consisting of an oxygen atom bonded to two carbon atoms that are already bonded in some way. The simplest epoxy is a three-member ring structure known by the term 'alpha-epoxy' or '1,2-epoxy'. The idealized chemical structure is shown in the figure below and is the most easily identified characteristic of any more complex epoxy molecule. Epoxy resins are formed from a long chain molecular structure with reactive sites at either end. The epoxy molecule also contains two ring groups at its centre which are able to absorb both mechanical and thermal stresses better than linear groups and therefore give the epoxy resin very good stiffness, toughness and heat resistant properties. The chemical name of LY556 epoxy resin is 2 (Chloromethyl)oxirane;4[2-(4hydroxyphyenl)propan2-yl]phenol And its chemical formula is C18H21ClO3.

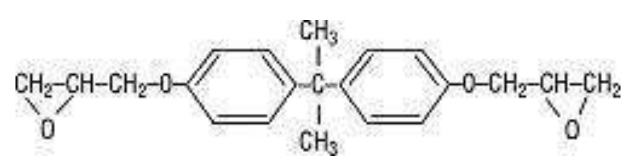

Figure.1. Chemical Structure of Epoxy

TABLE I
Properties of MWCNT

SL.NO	MWCNT	Description
1	Production method	Chemical Vapour Deposition
2	Available form	Black powder
3	Diameter	Av. Outer Diameter: 20nm Av. Inside Diameter: 16 nm
4	Length	Av 20 µm
5	Nanotubes purity	>97%
6	Metal particles	<2%
7	Amorphous carbon	<1%
8	Specific Surface area	189 m2/g
9	COOH-Content	COOH-MWNTs contain 1.8% COOH groups
10	Epoxy	Chains containing epoxy group 19%of functionalization

The hardener, often an amine, is used to cure the epoxy by an 'addition reaction' where both materials take place in the chemical reaction. The chemistry of this reaction means that there are usually two epoxy sites binding to each amine site. The chemical name of the hardener HY951 is Triethylenetetramine and is chemical formula is $C_6H_{18}N_4$.

In general, *uncured epoxy resins* have poor mechanical, chemical and heat resistance properties. However, good properties are obtained by reacting the linear epoxy resin with suitable curatives to form three-dimensional cross-linked thermoset structures. This process is commonly referred to as curing. *Curing of epoxy* resins is an exothermic reaction and in some cases produces sufficient heat to cause thermal degradation if not controlled. Curing may be achieved by reacting an epoxy with itself (homopolymerisation) or by forming a copolymer with polyfunctional curatives or hardeners. In principle, any molecule containing a reactive hydrogen may react with the epoxide groups of the epoxy resin. Common classes of hardeners for epoxy resins include amines, acids, acid anhydrides, phenols and alcohols.

II. METHODOLOGY

A. Materials

Following are the materials used

1. Epoxy Resin : LY556
2. Hardener: HY951
3. Multiwalled Carbon Nanotubes
4. Releasing Agent: Mansion Wax
5. Sealant: Anabond Silicone Sealant
6. Cleaning Agent: Acetone
7. MWCNT Dispersal Medium: Acetone

B. Preparation of Mould

1. The mould plates are cleaned with acetone.
2. Mansion wax is applied to mould for easy removal of the mould after curing.
3. Spacer plates are inserted based on the thickness of casting (here 3 mm) of the required dimensions between the two mould plates and sides of the mould are sealed with silicon sealant.
4. The sides of the mould are clamped with C- clamps.

Figure.2. Mould plate preparation

C. Preparation of Castings

1. Two beakers are washed with acetone and allowed to dry
2. As per the ratio of resin and hardener, that is 100:20, the weight of resin and Hardener is calculated. Resin is weighed in one beaker and hardener in the other.
3. The hardener is added to the beaker containing resin. It is mixed thoroughly for about 5 minutes.
4. The resin-Hardener mixture is poured into the prepared mould.
5. The mould is allowed for 48 hours for curing and the mould is released.

D. Dispersion of MWCNT in Matrix System

To achieve homogenous dispersion of Multi walled Carbon Nanotubes a Magnetic Stirrer is used. The required amount of epoxy is taken in a beaker and placed on the stirrer. The MWCNT is weighed and is added to the resin. A small amount of Acetone is added to reduce the viscosity and also for easy dispersal. The stirrer is switched on and the mixture is stirred for about 30 minutes at speed of about 1000 rpm.

Figure.3. Dispersion of MWCNT using Magnetic Stirrer

E. Cutting of specimens

Specimens were cut according to ASTM Standards using fabric cutting machine.

The dimensions of the different test specimens as per ASTM (American Society for Testing Materials) standard is given below

TABLE II
ASTM standards for Tensile and bending Test

Sl.No	Specimen Type	ASTM Std.
1	Tension	D638
2	Bending	D790
3	Hardness	

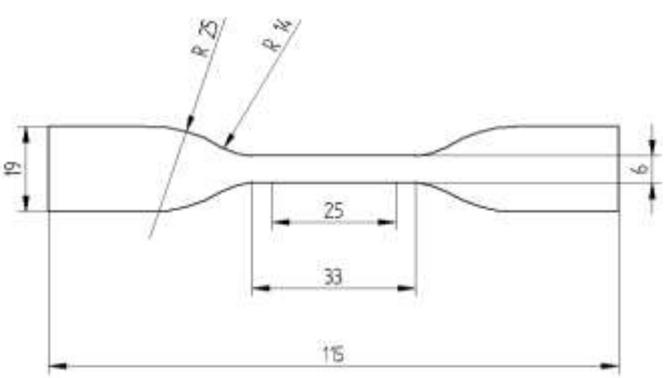

Figure.4. Dimensions of Tensile Test specimen according to ASTM standard.

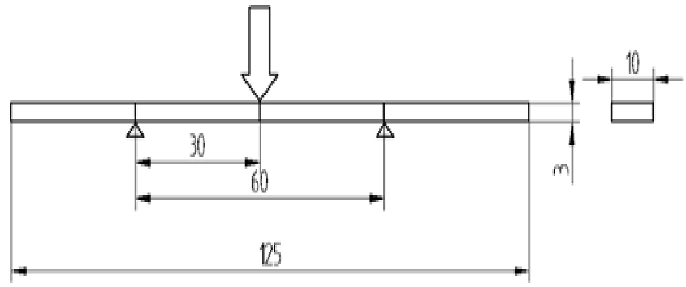

Figure.5. Dimensions of Bending Test specimen according to ASTM standard

E. Calculations

Density of epoxy resin (LY556) = 1.2g/cm3
Dimensions of the mould plate used = 25 × 25 × 0.3 cm
Thickness of Spacer Plate = 0.3 cm
Dimensions of Mould Cavity= 20 × 15 × 0.3 cm
Total volume available = 90cm3

Without MWCNT:

Mass of the matrix = 1.2 × 90 = 108g
Ratio of the matrix (Resin: Hardener) = 100:20
Mass of resin in the matrix = (100/120) × 108 = 90g
Mass of Hardener in the matrix = (12/120) × 108 = 18g

With MWCNT:

0.1% of total weight= (0.1 × 108)/100 = 0.108g
0.5% of total weight= (0.5 × 108)/100=0.54g
1% of total weight= (1 × 108)/100=1.08g
1.5% of total weight= (1.5 × 108)/100=1.62g

III. RESULTS

The Tensile Strength, Flexural Strength and Hardness of the test specimens are determined by subjecting it to Tensile Test, 3 point Bending Test and Rockwell hardness test.

A. Testing of Dry Specimens

To study the mechanical properties in dry conditions, 5 samples of composites were prepared with varying compositions of Multi Walled Carbon Nanotubes

a. Sample 1-(epoxy resin or Neat Resin Casting)
b. Sample 2-(epoxy resin + 0.1% MWCNT)
c. Sample 3- (epoxy resin + 0.5% MWCNT)
d. Sample 4-(epoxy resin + 1% MWCNT)
e. Sample 5-(epoxy resin + 1.5% MWCNT)

From each of these samples, three test specimens for tensile test and three test specimens for Bending test were cut according to ASTM standards. One test specimen for hardness test was cut and the hardness was determined at three different locations.

Tensile test

Sample 1- Neat Resin Casting(Pure Epoxy) – Dry condition

Sample 1 is Neat resin casting containing pure epoxy. Three test specimens namely 1 A, 1 B and 1 C were cut as per the ASTM standards from sample 1 and were subjected to Tensile

Test. The Result of the Specimen 1 C shows the maximum Tensile strength among the three specimens. The load v/s displacement results and tensile test results of this specimen are shown below.

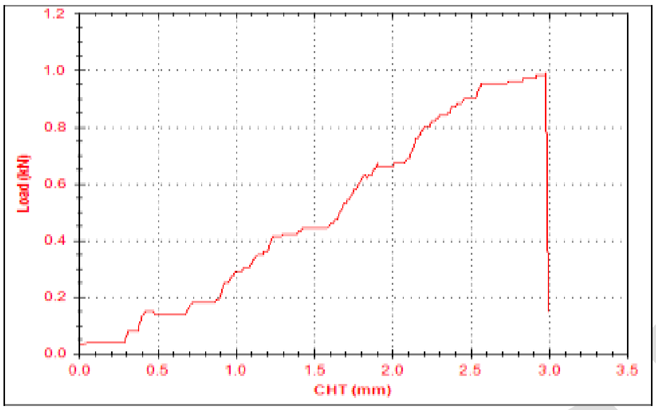

Figure.6. Load v/s Displacement for specimen 1

SPECIMEN 1
Load At Yield: 0.82 kN
Yield Stress: 34.254 N/mm
Load at Peak: 0.990 kN
Tensile Strength: 41.355 N/mm
% Elongation: 1.00 %

Sample 5 (Epoxy + 1.5% MWCNT) – Dry Condition

Sample 5 is epoxy resin reinforced with 1.5% MWCNT. Three specimens namely 5 A, 5 B and 5 C were cut from this sample and were subjected to Tensile Test.

The Result of the Specimen 5 C shows the maximum Tensile strength among the three specimens. The load v/s displacement results and tensile test results of this specimen are shown below.

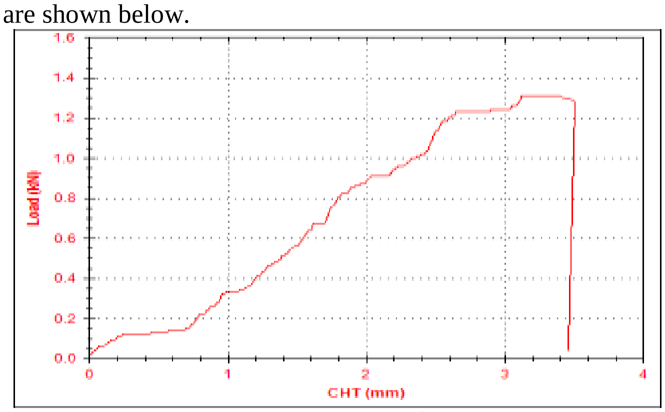

Figure.7. Load v/s Displacement for specimen 5

SPECIMEN 5
Load At Yield: 1.14 kN
Yield Stress: 63.330 N/mm
Load at Peak: 1.335kN
Tensile Strength: 71.670N/mm
% Elongation: 1.40 %

TABLE III
Final Tensile Test Results for dry samples

SAMPLES	TENSILE STRENGTH(N/mm2) Average Values DRY
Pure Epoxy casting	43.185
Epoxy + 0.1% MWCNT	55.363
Epoxy + 0.5% MWCNT	58.840
Epoxy + 1% MWCNT	63.996
Epoxy + 1.5% MWCNT	69.799

Bending Test

Sample 1 (Neat Resin Casting – Pure Epoxy) – Dry condition

Sample 1 is Neat resin casting containing pure epoxy and no reinforcements. Three specimens namely 1 D, 1 E and 1 F were cut from this sample and were subjected to 3 point bending Test. The Result of the Specimen 1 F shows the maximum Flexural strength among the three specimens. The load v/s displacement results and Bending test results of this specimen are shown below.

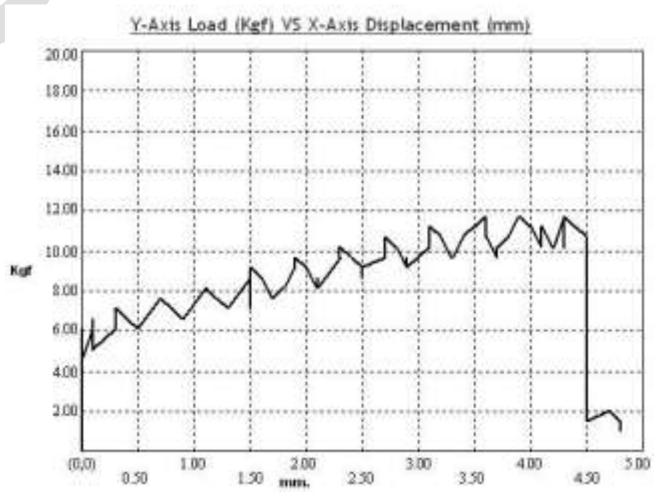

Figure.8. Load v/s Displacement for specimen

Sample 5 (Epoxy + 1.5% MWCNT) – Dry Condition

Sample 5 is Epoxy resin reinforced with 1.5% MWCNT. Three specimens namely 5 D, 5 E and 5 F were cut from this sample and were subjected to 3 point bending Test. The Result of the Specimen 5 F shows the maximum Flexural strength among the three specimens. The load v/s displacement results and Bending test results of this specimen are shown below.

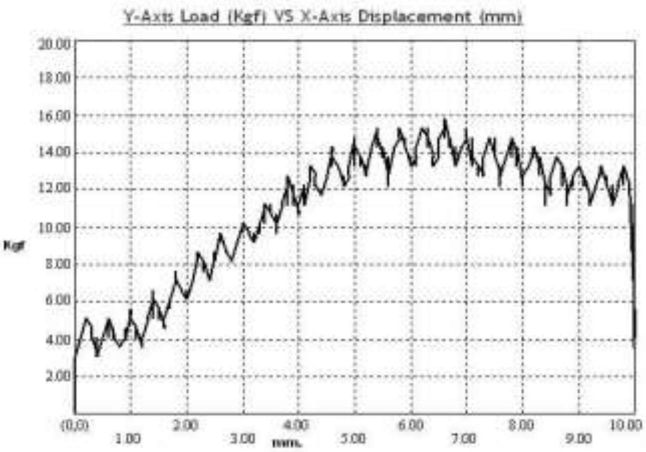

Figure.9. Load v/s Displacement for specimen

SPECIMEN 5
Ultimate Load: 154.998 N
(15.8 kgf)
Flexural Stress: 140.173 N/mm
Maximum Displacement: 6.30 mm

TABLE IV
Final Bending Test Results for Dry samples

SAMPLES	FLEXURAL STRENGTH(N/mm2) Average Values
	DRY
Pure Epoxy casting	89.978
Epoxy + 0.1% MWCNT	107.62
Epoxy + 0.5% MWCNT	112.230
Epoxy + 1% MWCNT	121.848
Epoxy + 1.5% MWCNT	134.535

Hardness Test

HRM values for the prepared specimens are tabulated in the Table

TABLE V
Hardness test Results for Dry Samples

SAMPLES	HRM
	DRY
Pure Epoxy casting	60
Epoxy + 0.1% MWCNT	73
Epoxy + 0.5% MWCNT	79
Epoxy + 1% MWCNT	83
Epoxy + 1.5% MWCNT	87

B. Testing of Wet Specimens

To study the mechanical properties in wet conditions, 3 samples of composites were prepared with varying compositions of Multi Walled Carbon Nanotubes.

a. Sample 1-(epoxy resin or Neat Resin Casting)
b. Sample 2-(epoxy resin + 0.1% MWCNT)
c. Sample 3- (epoxy resin + 0.5% MWCNT)

From each of these samples, three test specimens for Tensile test and three test specimens for Bending test were cut according to ASTM standards. They were immersed in saline water for 15 days and were subjected to testing.

Tensile Test

Sample 1 (Neat Resin Casting- Epoxy) – Wet condition

Sample 1 is Neat resin casting containing pure epoxy and no reinforcements. Three specimens namely 1 A, 1 B and 1 C were cut from this sample, immersed in saline water and were subjected to Tensile Test.

The Result of the Specimen 1 C shows the maximum Tensile strength among the three specimens. The load v/s displacement results and Tensile test results of this specimen are shown below.

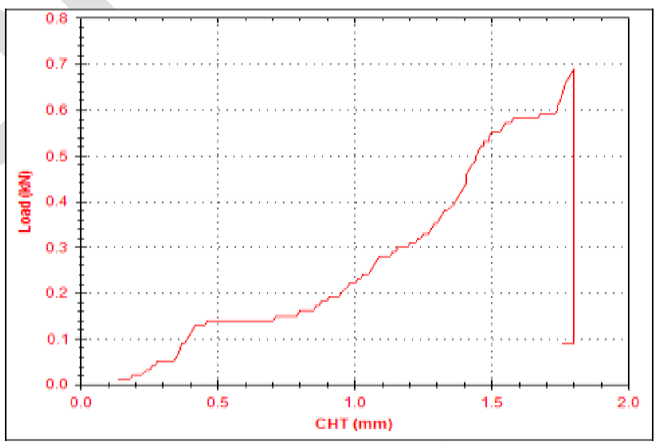

Figure.10. Load v/s Displacement of specimen

SPECIMEN 1 (Wet)
Load At Yield: 0.53kN
Yield Stress: 28.374N/mm
Load at Peak: 0.690kN
Tensile Strength: 36.939 N/mm
% Elongation: 0.56 %

The average Tensile Test Result for Sample 1 (Wet) is shown in the Table 5.14.The average Tensile Strength is 35.896 N/mm for Sample 1.

Sample 3(Epoxy + 0.5% MWCNT) – Wet Condition

Sample 3 is epoxy resin reinforced with 0.5% MWCNT. Three specimens namely 3 A, 3 B and 3 C were cut from this sample, immersed in saline water and were subjected to Tensile Test.

The Result of the Specimen 3 C shows the maximum Tensile strength among the three specimens. The load v/s displacement results and Tensile test results of this specimen are shown below.

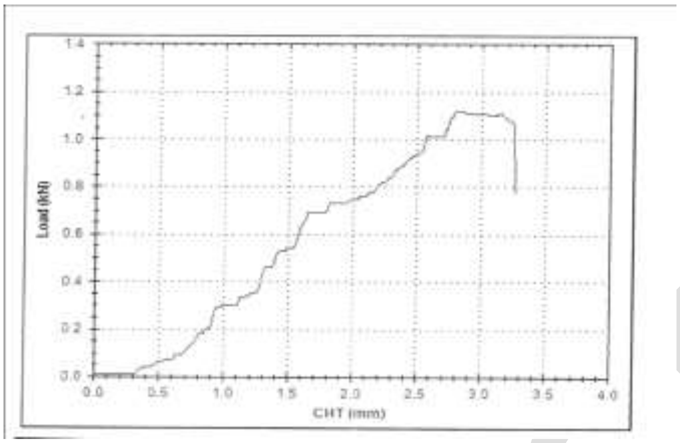

Figure.11. Load v/s Displacement of specimen

SPECIMEN 3 C (Wet)
Load At Yield: 0.95 kN
Yield Stress:49.953N/mm2
Load at Peak: 1.120 kN
Tensile Strength: 58.892N/mm
% Elongation: 1.64%

The average Tensile Test Result for Sample 3 (Wet) is shown in the Table 5.16.The average Tensile Strength is 55.252 N/mm2 for Sample 3.

TABLE VI
Final Tensile Test results for Wet Samples

SAMPLES	TENSILE STRENGTH(N/mm2) average values
	WET
Pure Epoxy casting	35.896
Epoxy + 0.1% MWCNT	49.560
Epoxy + 0.5% MWCNT	55.252

Bending Properties of Wet Specimens

Sample 1(Neat Resin Casting- Pure Epoxy) – Wet condition

Sample 1 is Neat resin casting containing pure epoxy and no reinforcements. Three specimens namely 1 D, 1 E and 1 F were cut from this sample, immersed in saline water and were subjected to 3 point beding Test. The Result of the Specimen 1 D shows the maximum Flexural strength among the three specimens. The load v/s displacement results and Bending test results of this specimen are shown below.

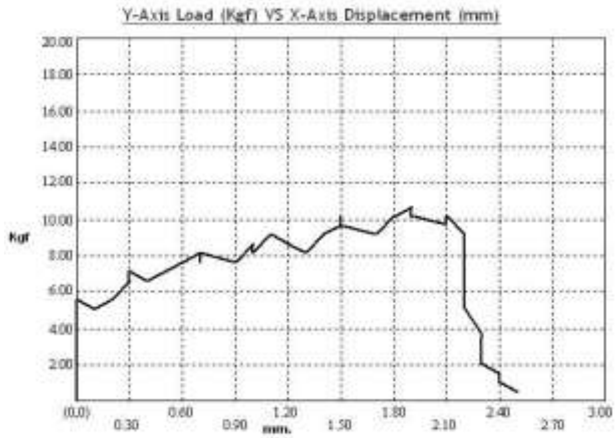

Figure.12. Load v/s Displacement for specimen

SPECIMEN 1 D (Wet)
Ultimate Load: 104.967 N
(10.70 kgf)
Flexural Stress: 84.818 N/mm2
Maximum Displacement: 2.50 mm

The average Bending Test Result for Sample 1 (Wet) is shown in the Table .The average Flexural Strength is 82.472 N/mm2 for Sample 1.

Sample 3(Epoxy + 0.5% MWCNT) – Wet Condition

Sample 3 is Epoxy resin reinforced with 0.5% MWCNT.. Three specimens namely 3 D, 3 E and 3 F were cut from this sample, immersed in saline water and were subjected to 3 point Bending Test. The Result of the Specimen 3 E shows the maximum Flexural strength among the three specimens. The load v/s displacement results and Bending test results of this specimen are shown below.

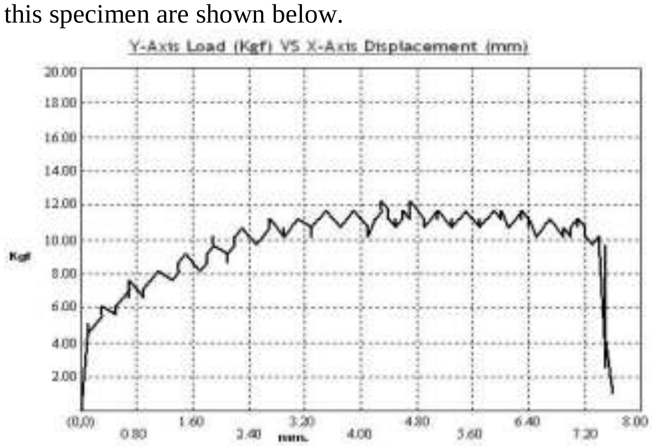

Figure.13. Load v/s Displacement for specimen

SPECIMEN 3 E (Wet)
Ultimate Load: 119.976 N
(12.23 kgf)
Flexural Strength: 117.53 N/mm2
Maximum Displacement: 7.60mm

The average Bending Test Result for Sample 3 (Wet) is shown in the Table 5.19.The average Flexural Strength is 110.490 N/mm2 for Sample 3.

TABLE VII
Final Bending Test Results for Wet Samples

SAMPLES	FLEXURAL STRENGTH (N/mm2) average values
	WET
Pure Epoxy casting	82.472
Epoxy + 0.1% MWCNT	103.744
Epoxy + 0.5% MWCNT	110.490

Hardness Test for Wet Specimens

HRM values for the Wet specimens are tabulated in the table.

TABLE VIII
Hardness test Results for Wet specimens

SPECIMENS	HRM
	WET
Pure Epoxy casting	58
Epoxy + 0.1% MWCNT	72
Epoxy + 0.5% MWCNT	78

C. Moisture Gain in Composites

Neat resin casting, Epoxy Composite reinforced with 0.1% MWCNT and 0.5% MWCNT were prepared and was immersed in Saline water for about 15 days. To study the moisture absorption behavior, specimens were placed in a constant temperature saline water bath after taking its initial dry weights. The specimens were periodically taken out from the water bath to measure their weight gain as outlined below.

1. The specimens were first taken out of the bath and were placed on a filter paper. Each specimen was wiped with the filter paper to remove the free moisture adhering to its surfaces as well as the edges.
2. The wiped specimens were weighed on an electronic balance and immediately returned to the water bath. This was done in order to minimize any possible loss of moisture from the specimen at room temperature conditions.
3. The measured weights of the wet specimen were tabulated along with the immersion time.
4. The percentage moisture gain M by the specimen was calculated by the equation as follows

$$M = \frac{Ww - Wd}{Wd} \text{ X } 100$$

a.

Where Ww = Weight of moist(wet) specimen
Wd = Weight of dry specimen

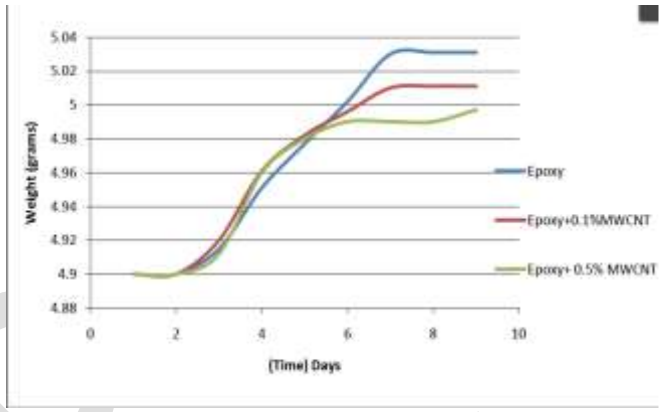

Figure.14. Specimen weight v/s Immersion Time for Wet specimens

TABLE IX
Percentage Moisture Absorption

Specimens	Initial Weight (grams)	Final Weight (grams)	% Moisture Absorption
Neat Resin Casting(Pure Epoxy)	4.9	5.033	2.71
Epoxy+ 0.1% MWCNT	4.9	5.012	2.28
Epoxy + 0.5% MWCNT	4.9	4.997	1.979

Figure.15. Immersion of specimens (with and without MWCNT) in water

IV. CONCLUSIONS

The study was conducted to determine the Tensile properties, Flexural Properties and Hardness values of the composites which has different Percentages of MWCNT reinforced with epoxy resin base.

Five samples were cast by varying the percentage of MWCNT

1. Sample 1-(epoxy resin or Neat Resin Casting)
2. Sample 2-(epoxy resin + 0.1% MWCNT)
3. Sample 3- (epoxy resin + 0.5% MWCNT)
4. Sample 4- (epoxy resin + 1% MWCNT)
5. Sample 5- (epoxy resin + 1.5% MWCNT)

In each of these five samples, three specimens for tensile test, three specimens for bending test and one specimen for hardness test were cut and subjected to mechanicaltesting. Totally there were 15 tensile specimens and 15 bend specimens and one hardness specimen. Hardness was determined at three different locations. Another set of samples (Pure epoxy composite, Composite reinforced with 0.1% MWCNT, Composite reinforced with 0.5% MWCNT) were casted. In each of these three samples, three specimens for tensile test, three specimens for bending test and one specimen for hardness test were cut and subjected to mechanicaltesting. Totally there were 9 tensile specimens and 9 bend specimens and one hardness specimen. Hardness was determined at three different locations. From the experiments conducted on the specimens, the following conclusions are drawn.

1. For the prepared composites, with the increase in the percentage of MWCNT the tensile strength, Flexural Strength and hardness increases.
2. The Tensile Strength increases by 61.62% at 1.5% MWCNT reinforcement, when compared with pure epoxy composite.
3. The Flexural Strength increases by 49.52% at 1.5% MWCNT reinforcement when compared with pure epoxy composite.
4. The Mechanical Properties of Epoxy composites deteriorate when subjected to moisture absorption due to swelling of epoxy and weakening of bonds.
5. The moisture absorbed by composites reinforced with 0.1% and 0.5% MWCNT is less compared to that of pure epoxy composite.
6. The Percentage Degradation of Tensile Strength, Flexural Strength and hardness due to moisture absorption is less for the composites reinforced with 0.1% and 0.5% MWCNT when compared to pure epoxy composite.

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