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Characterisation of Particulate Epoxy Composites for Mechanical Behaviour

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Abstract— over the last century, polymers have emerged as one of the most indispensible components used in everyday life, epoxy or poly- epoxide being one such example. Until recently, synthetic filler materials have been the preffered choice for reinforcement of epoxy to improve its toughness. However, natural filler and fiber materials are emerging as suitable alternatives to synthetic materials for reinforcing polymers such as epoxy due to their environment friendliness, high abundance, renewability and cost effectiveness.

Several research efforts have been put to study the effectiveness of natural fiber based materials on the mechanical behavior of epoxy composites, focusing mainly on fibers and their weight percent's within the composites.

The present experimental study aims at investigating mechanical behavior of walnut shell powder reinforced epoxy composites. Composites bearing 10, 20, 30 and 40% weight fraction of walnut shell powder where made using hand layup method. The fabricated composite samples are prepared according to the ASTM standards for flexural testing. Three-point bending testy is carried samples and results are presented. Analytical results and experiment results are compared and they found to be very close agreement.

Keywords— Composite, Epoxy resin, Particulate, Polymer, Matrix, FEA, SEM (Scanning Electron Microscopic) & PMC (Polymer Matrix Composites).

INTRODUCTION

The development of mankind is defined in terms of advances in materials: the Stone Age, the Bronze Age, and the Iron Age. Today the development of any country is decided based on the amount of steel and concrete used. The Industrial Revolution was to a large extend made possible by advances in the use of materials in industrial equipments [1]. In the continued quest for improved performance, materials which may be specified by various criteria including less weight, more strength and lower cost, currently used materials frequently reach the limit of their usefulness [2]. In the last half century, the growth of materials technology has been explosive, and its impact on our daily lives, pervasive. In last few decades the developments in materials technology is fuelled mainly by composite material [1]. Thus material engineers and scientists are always striving to produce either improved traditional materials or completely new materials. Composites are an example of the latter category. They are developed as mixture of two distinct physical constituents and perform better than either of the constituents in its individual existence. The concept of composites is not very new. Bricks made from mud reinforced with straw, which are used in ancient civilizations, could be named composite. Also the naturally occurring materials like bone and wood are composites. But presently the same concept is used to develop man-made composite materials that perform well at a reduced weight/cost [2].

1.1 Definition of composites

A composite material is a materials system composed of a mixture or combination of two or more macro constituents differing in form and/or material composition and that are essentially insoluble in each other [3].

Composite materials represent nothing but a giant step in the ever constant endeavor of optimization in materials [4].

A structural composite is a material system consisting of two or more phases on macroscopic scale, whose mechanical performance and properties are designed to the superior to those of the constituent materials acting independently. One of the phase is usually discontinuous, stiffer, and stronger and is called reinforcement, where as a less stiff and weaker phase is continues and is called matrix (Figure 1.1). sometimes, because of chemical interactions or other processing effects, an additional phase, called interphase, exists between the reinforcement and matrix [6].

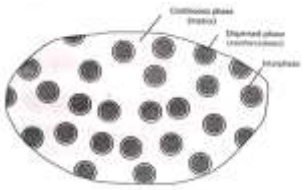

Figure 1.1 Phases of composite materials

1.2 Classification of composites

Two phase composite materials are classified into three broad categories depending on the type, geometry, and orientation of the reinforcement phase, as illustrated in the chart of Figure

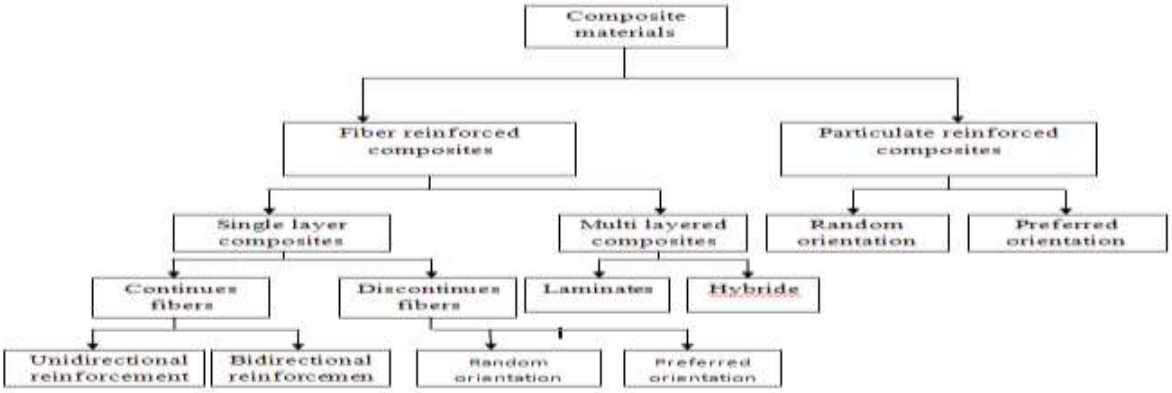

Fig 1.2 Classification of composite materials

4. Materials, Processing and Testing Methods

This chapter describes specification and properties of materials used as filler and matrix. This chapter covers the methods adopted for processing composites with varying content of the filler. In the present work walnut shell powder is used as the filler with Lapox L-12 epoxy resin as a matrix system and K-6 hardener .This chapter also covers the testing methods followed and the procedure of testing.

4.1 Materials

4.1.1Filler

Use of inorganic fillers in composites is increasing. Fillers not only reduce the cost of composites, but also frequently impart performance improvements that might not otherwise be achieved by the reinforcement and resin ingredients alone. Fillers can improve mechanical properties including fire and smoke performance by reducing organic content in composite laminates. Also, filled resins shrink less than unfilled resins, thereby improving the dimensional control of molded parts. Important properties, including water resistance, weathering, surface smoothness, stiffness, dimensional stability and temperature resistance, can all be improved through the proper use of fillers.

The thermosetting resin segment of the composite industry has taken advantage of the properties of fillers for many years. More recently, the thermoplastic industry has begun to make widespread use of inorganic fillers. Breakthroughs in chemical treatment of fillers that can provide higher filler loadings and improved laminate performance are accelerating this trend.

Figure 4.1 Walnut Shells

Figure 4.2 Walnut shell Powder

4.1.2 Filler Types

There are a number of inorganic filler materials that can be used with composites including:

Calcium carbonate is the most widely used inorganic filler. It is available at low cost in a variety of particle sizes and treatments from well-established regional suppliers, especially for composite applications. Most common grades of calcium carbonate filler are derived from limestone or marble and very common in automobile parts.

Kaolin (hydrous aluminum silicate) is the second most commonly used filler. It is known throughout the industry by its more common material name, clay. Mined clays are processed either by air flotation or by water washing to remove impurities and to classify the product for use in composites. A wide range of particle sizes is available.

Alumina trihydrate is frequently used when improved fire/smoke performance is required. When exposed to high temperature, this filler gives off water (hydration), thereby reducing the flame spread and development of smoke. Composite plumbing fixture applications such as bathtubs, shower stalls and related building products often contain alumina trihydrate for this purpose.

Calcium sulfate is a major flame/smoke retarding filler used by the tub/shower industry. It has fewer waters of hydration, and water is released at a lower temperature. This mineral filler offers a low cost flame/smoke retarding filler.

Other commonly used fillers include:

Mica
Feldspar
Wollastonite
Silica
Talc
Glass microspheres
Flake glass
Milled glass fibers
Other microsphere product

4.1.3 Using Fillers in Composites

When used in composite laminates, inorganic fillers can account for 40 to 65% by weight. They perform a function similar to silica fume in concrete. In comparison to resins and reinforcements, fillers are the least expensive of the major ingredients. These materials are nevertheless very important in establishing the performance of the composite laminate for the following reasons:

Fillers reduce the shrinkage of the composites part.
Fillers influences the fire resistance of laminates.
Fillers lower compound cost by diluting more expensive resin and may reduce the amount of reinforcement required.
Fillers can influence the mechanical strengths of composites.
Fillers serve to transfer stresses between the primary structural components of the laminate (i.e., resin and reinforcement), thereby improving mechanical and physical

Uniformity of the laminate can be enhanced by the effective use of fillers. Fillers help maintain fiber-loading uniformity by carrying reinforcing fibers along with the flow as resin is moved on the mold during compression molding.

Crack resistance and crack prevention properties are improved with filled resin systems. This is particularly true in sharp corners and resin-rich areas where smaller particles in the filler help to reinforce the resin in these regions.

The combination of small and medium filler particles helps control compound rheology at elevated temperatures and pressures, thereby helping to ensure that compression molded parts are uniform.

Low-density fillers are used extensively in marine putty and the transportation industry. They offer the lowest cost of filled systems, without the increases of weight that affect the performance of the final product.

The Walnut shell powder is used as a filler material in this work.

4.1.2 Matrix system:

The matrix system consists of a medium viscosity epoxy resin (LAPOX L-12) and a room temperature curing polyamine hardener (K-6) supplied by Yuje marketing, Malleswaram, Bangalore. Epoxy resin was selected as the material for the matrix system because of its wide application, good mechanical properties, excellent corrosion resistance and ease of processing. Some details including density of the constituents of the matrix system are listed in Table 4.1.

Lapox L–12: is a liquid, unmodified epoxy resin of medium viscosity which can be used with various hardeners for making glass fiber reinforced composites. The choice of hardener depends on the processing method to be used and on the properties required of the cured composite.

Hardener K–6: is a low viscosity room –temperature curing liquid hardener. It is commonly employed for hand layup applications. Being rather reactive, it gives a short pot-life and rapid cure at normal ambient temperatures. Details of constituent

properties as supplied by manufacturer are presented in Table [4.1].

Constituent	Trade name	Chemical name	Epoxide equivalent	Density (kg/m^3)	Supplier
Resin	LAPOX L-12	Diglycidyl Ether of bisphenol A (DGEBA)	182-192	1162	Yuje Marketing Bangalore
Hardener	K-6	Tri ethylene Tetra amine (TETA)	--	954	Yuje Marketing Bangalore

Table 4.1 Details of constituent properties as supplied by manufacturer

4.2 Moulds Used for Testing PRC's

4.2.1 Flexural Test

A mould of size 85 mm X 85 mm X 30mm (Figure 4.1) was prepared of mild steel for preparing compression samples. Mould cons of a base plate, frame that could be dismantled to facilitate easy removal of casting after the curing. All the surfaces of the mould w coated with wax. All the inner surfaces of mould, coming in contact with surfaces of composite to be cast are smeared with unifc coating of wax in order to facilitate the release of the cast slab.

Figure 4.3 Bending Sample mould

4.3 Processing

4.3.1 Particulate Reinforced Epoxy Composite:

Walnut shell powder is reinforced by mechanically mixing measured quantities of walnut shell powder in epoxy resin. The mixture is stirred using mechanical mixture until a slurry of uniform viscosity is obtained. K6 hardener in 12% by volume of resin was added to epoxy in the container with gentle stirring to minimize the formation of air bubbles. The slurry is cast in mild steel mould and allowed to cure at room temperature for about 24 hours, after which cast sample is withdrawn and trimmed to required shape.

The samples were made by considering different percentages of walnut shell powder by the weight fraction of the epoxy i.e. 10%, 20%, 30% and 40%.

Figures 4.4 Bending samples used for testing.

4.3.2 Post curing of samples:

All the samples are post cured at about 75`c for 3hrs in an oven. The samples are cured below 75'c because above 75'c the epoxy resin present in the samples starts melting. Then the samples are cut into ASTM standard using hacksaw.

"Archimedes principle states that the buoyant force on an object is going to be equal to the weight of the fluid displaced by the object or the density of the fluid multiplied by the submerged volume times the gravitational constant".

The density of the specimen is determined by keeping the cantilever beam as shown in Figure 4.4 over the weighing balance and then suspending the sample in air by means of thread, on the notch provided. An electronic weighing balance was used to determine the

weight of the sample. The weight of the cantilever beam and the thread is initially set to zero by using the tare option of the weighing balance.

The weight of the sample is determined in air and then the sample is dipped in water, to determine its weight in water as shown in Figure 4.5. Then the density of the sample is determined by using the formula given below.

$$\rho = \frac{W_a}{W_a - W_w} \quad (4.1)$$

Where; ρ = density of the composite material (g/cm^3)
W_a = weight of sample in air (g/cm^3)
W_w = weight of sample (g/cm^3)

Figure 4.6 Sample weighed in

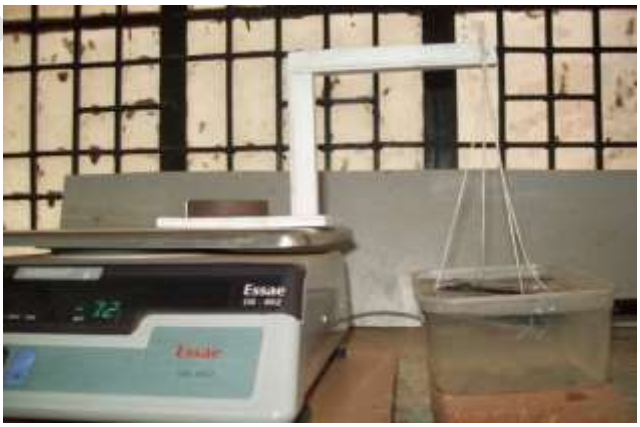

Figure 4.7 Sample weighed in distilled water

4.4.2 Mechanical Test

4.4.2.1 Bending Test

The bending test is used for studying the properties are flexural modulus, strength and maximum mid span deflection.
Testing is done on 15kN digitally controlled servo hydraulic test system equipped with load cell, stroke transducer. It is computer controlled for setting test assignments, acquiring data online and with provision to store the test results on computer media. Load cell has capacity to measure the load up to 15kN. Stroke has capacity to measure the displacement up to 60mm.

The testing machine setup is showed in below Fig 4.6. 3 point bending setup is showed in Fig 4.7.

Figure 4.8 Bending setup

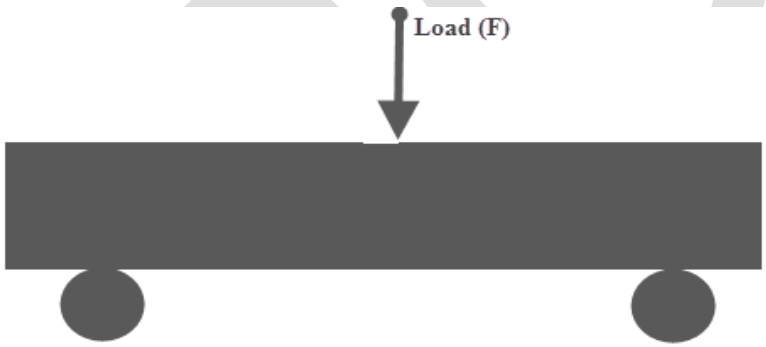

Figure 4.9Sample loading for 3 point bending test

5. Results and discussion

The experimental study presented in this chapter gives the results of both physical testing methods i.e density of all samples, and also the mechanical testing methods.

5.1.1 Physical Testing

Physical testing consists of density.

Density Test of bending

The density of all samples was calculated using the equation (4.1):
The following table 5.1 shows the densities of all PRC's

Table 5.1 Densities of all Bending PRC samples

Density test of Samples					
Serial Number	**Sample Coding**	**Weight in Air (gm)**	**Weight in Water (gm)**	**Density (gms/cc)**	**Average**
1	**10% - 1**	3.55	0.51	1.1666	1.16834
2	10% - 2	3.73	0.53	1.1774	
3	10% - 3	3.35	0.49	1.18456	
4	10% - 4	3.78	0.56	1.20066	
5	10% - 5	3.59	0.53	1.20192	
6	10% - 6	3.44	0.47	1.19808	
7	10% - 7	Broken Samples			
8	10% - 8				
9	**20% -1**	3.59	0.54	1.16776	1.18633
10	20% - 2	3.75	0.53	1.16562	
11	20% - 3	3.88	0.62	1.17132	
12	20% - 4	3.64	0.60	1.17391	
13	20% - 5	3.76	0.58	1.17320	
14	20% - 6	3.70	0.60	1.15224	
15	20% - 7	3.76	0.62	1.19745	
16	20% - 8	3.60	0.57	1.18811	
17	**30% - 1**	3.50	0.50	1.16666	1.19177
18	30% - 2	3.45	0.52	1.17741	
19	30% - 3	3.53	0.55	1.18456	
20	30% - 4	3.59	0.60	1.20066	
21	30% - 5	3.75	0.63	1.20192	
22	30% - 6	3.75	0.62	1.19808	
23	30% - 7	3.80	0.63	1.19873	
24	30% - 8	3.92	0.67	1.20615	
25	**40% - 1**	4.16	0.69	1.19873	1.20240
26	40% - 2	3.93	0.67	1.20552	
27	40% - 3	3.26	0.57	1.21189	
28	40% - 4	3.81	0.65	1.20569	
29	40% - 5	3.88	0.63	1.19384	
30	40% - 6	4.27	0.70	1.9607	
31	40% - 7	4.22	0.71	1.20227	
32	40% - 8	4.23	0.72	1.20512	

From the above tables (Table 5.1), and Figure 5.1 is clearly observed that with increase in weight fraction of filler density increases. Obvious reason for this could be, influences the weight of the products.

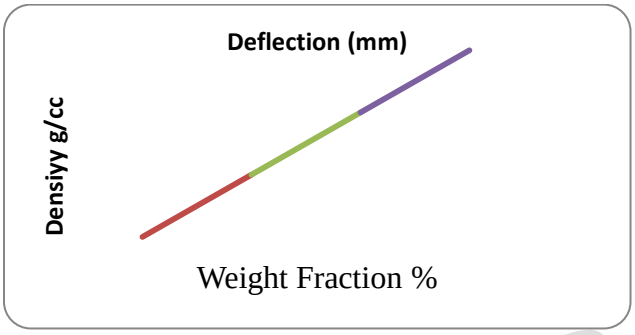

Figure 5.1 Densities of PRC Samples

5.1.2 Mechanical

Bending Test

The stress-deformation curves obtained from the flexural testing of PRCs. Below figures show that all types of PRCs fail in the brittle fracture mode at the end of the linear region in their stress-deformation curves. The failure starts at the tensile side of the specimen, in-line with the central loading anvil, and grooves towards the compressive side. Hence, the deformation and fracture behavior are governed by the tensile properties of these specimens. Figure 5.13 shows the fracture pattern of four different configuration specimens, which are selected randomly.

Table5.2 Flexural property of 10% reinforced composite

Specimen identifier	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
10% -1	2320	2510	47.9	1.9	3.1	12.4
10% -2	2280	2410	47.4	1.9	3.1	12.4
10% -3	3020	3140	59.4	1.9	3.1	12.3
10% -4	3410	3520	50.0	1.4	3.1	12.2
10% -5	3170	3420	38.9	1.1	3.1	12.3
10% -6	1040	2140	48.8	2.2	3.1	12.2
10% -7	2990	3260	49.6	1.5	3.1	12.4
10% -8	2900	3170	50.5	1.6	3.2	12.6

The Table 5.2 represents the flexural property of 10% weight fraction of particulate reinforced composites.

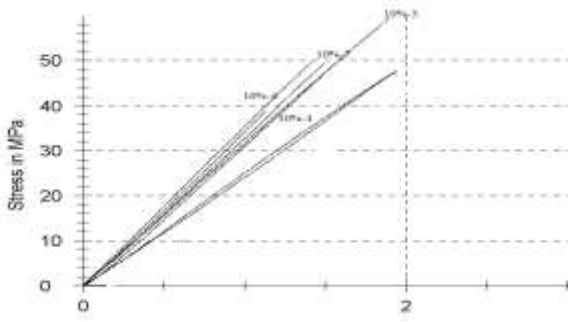

Figure5.2 Stress v/s Deflection in for curve 10% PRC from the experiment

The above Figure5.2 represents the stress v/s deflection curve for 10% weight fraction of PRCs; these results are obtained from 3-point bending test.

Table 5.3 Statistical table for 10% weight fraction of PRCs

Series n = 8	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
$\bar{x}$	2640	2950	49.1	1.7	3.113	12.35
s	755	516	5.57	0.35	0.03536	0.1309
ν	28.58	17.52	11.36	20.75	1.14	1.06

Table 5.3 shows the statistical representation of 10% weight fraction of PRCs.

Note: $\bar{x}$ = arithmetic mean of the set of observation
n = number of observation
s = estimated standard deviation
ν= value of single of single observation

Table5.4 Flexural property of 20% reinforced composite

Specimen identifier	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
20% -1	2200	2350	35.7	1.5	3.1	12.1
20% -2	3220	3400	44.7	1.3	3.1	12.4
20% -3	-20.4	1380	39.9	2.6	3.1	12.3
20% -4	2500	2870	40.8	1.4	3.2	12.4
20% -5	2470	2680	42.4	1.6	3.2	12.4
20% -6	1970	2110	26.5	1.2	3.3	11.82

The Table 5.8 represents the flexural property of a 40% weight fraction of particulate reinforced composites.

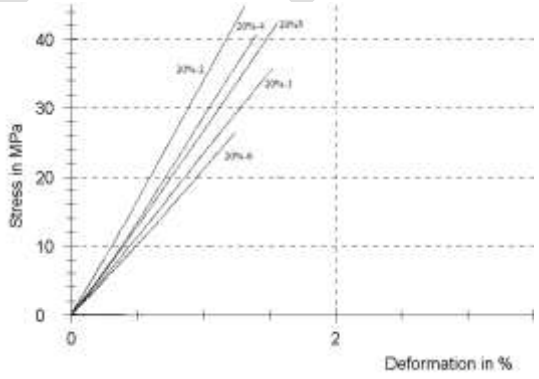

Figure5.3 Stress v/s Deflection in for 20% PRC from the experimental

The above Figure5.3 represents the stress v/s deflection curve for 20% weight fraction of PRCs; these results are obtained from 3-point bending test.

Table 5.5 Statistical table for 20% weight fraction of PRCs

Series n = 6	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
$\bar{x}$	2060	2460	38.3	1.6	3.167	12.24
s	1100	691	6.52	0.49	0.08165	0.2351
ν	-	28.06	17.02	30.55	2.58	1.92

Table 5.5 shows the statistical representation of 20% weight fraction of PRCs

Table5.6 Flexural property of 30% reinforced composite

Specimen identifier	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
30% -1	3230	3560	39.8	1.1	3.14	12.3
30% -2	2490	2640	27.1	1.0	3.44	12.55
30% -3	2550	2730	38.1	1.4	3.25	12.3
30% -4	2970	3250	38.2	1.2	3.12	12.3
30% -5	2830	3050	39.4	1.3	3.1	12.31
30% -6	3230	3410	49.0	1.4	3.04	12.4
30% -7	3060	3480	39.6	1.1	3.12	12.35
30% -8	3070	3270	46.6	1.4	3.12	11.9

Table 5.6 shows that the flexural property for the 30% weight fraction of particulate reinforced composites.

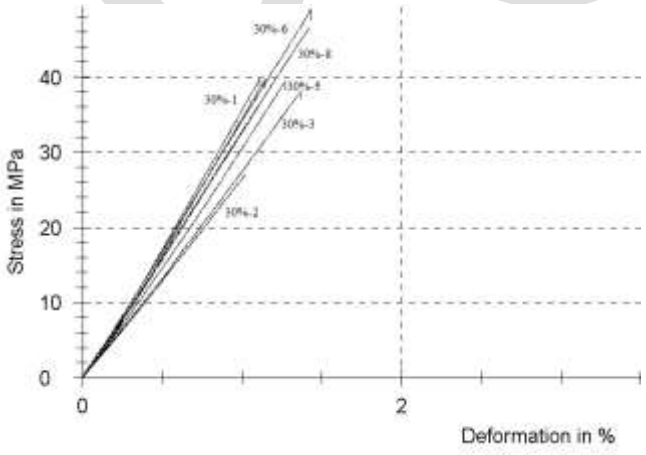

Figure5.4 Stress v/s Deflection curve for 30% PRC from the experimental

The above Figure5.4 represents the stress v/s deflection curve for 40% weight fraction of PRCs; these results are obtained from 3-point bending test.

Table 5.7 Statistical table for 30% weight fraction of PRCs

Series n = 8	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
$\bar{x}$	2930	3170	39.7	1.2	3.166	12.3
s	285	341	6.53	0.15	0.125	0.1833
ν	9.72	10.74	16.44	12.42	3.95	1.49

Table 5.7 shows the statistical representation of 30% weight fraction of PRCs.

Table5.8 Flexural property of 40% reinforced composite

Specimen identifier	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
40% -1	3310	-	26.9	0.78	3.15	13.08
40% -2	3090	-	24.3	0.74	3.22	12.44
40% -3	3240	3430	45.2	1.3	3.31	10.39
40% -4	3170	3330	40.5	1.2	3.25	12.42
40% -5	3120	3190	46.3	1.5	3.3	11.75
40% -6	3010	3250	48.4	1.5	3.35	13.15
40 % -7	3210	3460	42.9	1.2	3.32	12.9
40% -8	3310	3490	53.3	1.5	3.28	13.08

The Table 5.8 represents the flexural property of a 40% weight fraction of particulate reinforced composites.

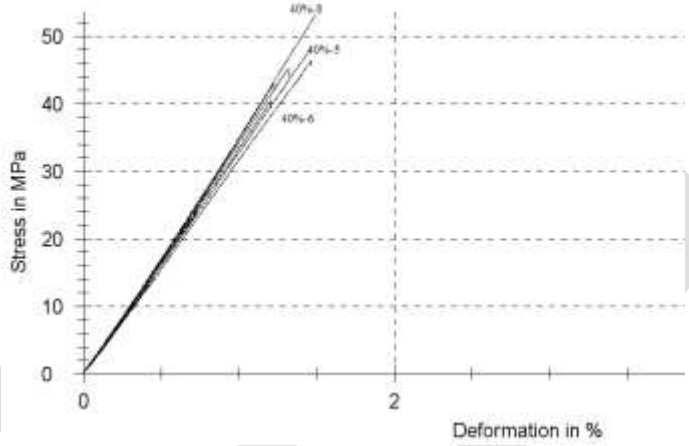

Figure5.5 Stress v/s Deflection curve for 40% PRC from the experimental

The Figure 5.5 shows the stress v/s deformation curve for the 40% weight fraction of PRCs, which is obtained from the 3- point bending experiment.

Table 5.9 Statistical table for 40% weight fraction of PRCs

Series n = 8	modulus MPa	sec mod MPa	flex strngth MPa	deflection %	thickness mm	spec width mm
$\bar{x}$	3180	3360	41.0	1.2	3.273	12.4
s	106	120	10.2	0.30	0.06409	0.9413
ν	3.34	3.57	24.97	24.77	1.96	7.59

The above Table 5.9 shows that the statistical representation of the 40% weight fraction of the particulate reinforced composites.

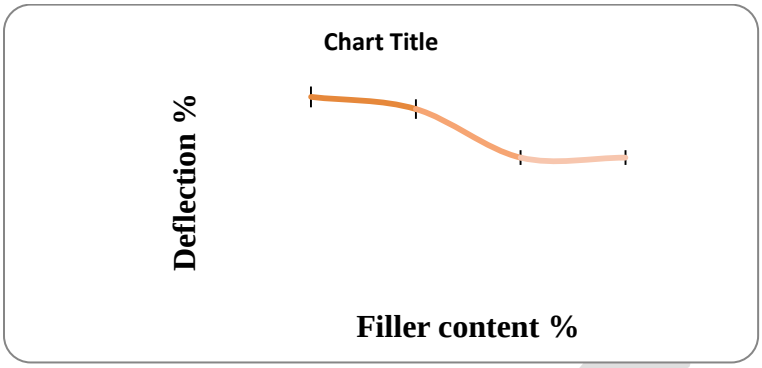

Figure 5.6 Deflection vs. weight fraction of particulate reinforced composite

Figure 5.6 shows the crack pattern of the samples for different weight fraction of walnut shell powder, it is clear that in all the cases of samples of pure composites which are randomly selected the crack has initiated near the mid-span of the specimen and propagated vertically.

5.2 Analytical Approach

The load–displacement data is used in calculating the flexural modulus and strength of bending samples. The flexural modulus and flexural strength are calculated by using Equation 5.1.

Mid span deflection (*D*) of PRC bending sample is calculated by the following equation [18].

$$D = \frac{rL^2}{6d} \qquad (5.1)$$

Where 'r' is the strain and 'L' is the span length'd' is depth of beam. The mid span deflection obtained from analytical calculation is presented in table 5.2 to 5.5.

Table 5.10 MOM approach for 10% PRCs

Load (N)	15.76	31.52	47.28	63.04	78.08	94.56
Deflection mm	0.4451	0.8902	1.3354	1.7805	2.2056	2.6708

Table 5.11 MOM for 20% PRCs approach

Load (N)	15.88	31.77	47.67	63.56	78.08
Deflection mm	0.4172	0.8347	1.2525	1.6700	1.8786

Table 5.12 MOM approach for 30% PRCs

Load (N)	15.27	30.55	45.87	61.16	74.92
Deflection mm	0.4382	0.8768	1.3165	1.7554	2.1503

Table 5.13 MOM approach for 40% PRCs

Load (N)	15.76	31.52	47.28	63.04	78.08	94.56
Deflection mm	0.4451	0.8902	1.3354	1.7805	2.2056	2.6708

Table 5.10 to 5.13 shows the results of theoretical calculation which is calculated by using Equation 5.1

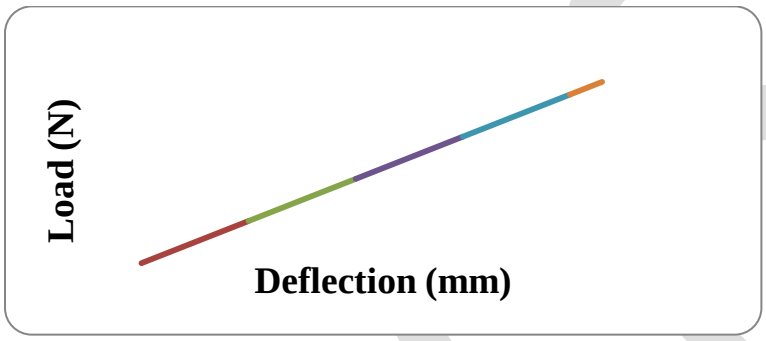

Figure 5.7 Load v/s Deflection curve from MOM approach

Figure 5.7 shows load v/s deflection curve which is obtained from the theoretical calculation using the equation 5.1. The graph shows that the deflection increases with increase the load.

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CONCLUSION

The present work deals with the preparation of characterization of waste walnut shell powder reinforced epoxy composite. The successful fabrication of a new class of epoxy based composites reinforced with walnut shell powder. In this work, flexural properties of walnut shell powder reinforced composites are analyzed under three-point bending test. Experimental results on these composites show that the specific modulus of these lightweight composite is higher. The composite flexural modulus can be effectively tailored by varying the weight fraction. On the other hand, the flexural strength is primarily influenced by the resin content of the composite. It is found that the composite strength decreases as the inclusion weight fraction increases. The flexural strength of the composite is found to be maximum with 40% weight fraction of walnut shell powder.

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