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EXPERIMENTAL INVESTIGATION OF CuO NANOPARTICLES ON THERMOMECHANICAL PROPERTIES OF NANOBASED POLYMER COMPOSITES

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Abstract—The present research work demonstrates the preparation of Copper Oxide Nanoparticles (CuO NPs) and investigates the thermo mechanical properties of the CuO NPs embedded in the polymer composites experimentally. In this study, CuO NPs were produced by aqueous precipitation method and morphology of the NPs was studied using Field Emission Transmission Electron Microscope (FESEM). Epoxy resin and glass microsphere were considered the base material for the preparation of the Nano based polymer composites. In order to fabricate the Nano based polymer composites, CuO NPs with 1.0wtpercentage were embedded in the base material by means of compression moulding press. Nano composites proved higher thermal conductivity enhancement rather than the base material. While comparing to the base material, the maximum four-point bending strength of 415 MPa was obtained from the Nano based polymer composites. The test results obtained from the TG study revealed that an addition of CuO NPs had acted as the thermal retardant and CuO NPs had delayed thermal degradation of the Nano based polymer composites. Based on the test results, it can be suggested that the newly fabricated nanocomposites have achieved the improved thermal and mechanical properties.

Key words: CuO NPs, FESEM, Epoxy, Compression, Glass Microsphere

I. INTRODUCTION

Composites are one of the most widely used materials because of their adaptability to different situations and the relative ease of combination with other materials to serve specific purposes and exhibit desirable properties. The high strength to weight ratio of the composite materials makes it an important material in Aerospace and Marine applications. [1-3]

syntactic foam include buoyancy modules for marine riser tensioners, boat hulls, deep-sea exploration, autonomous underwater vehicles (AUV), parts of helicopters and airplanes, and sporting goods such as soccer balls. Structural applications include the use of syntactic foams as intermediate layer (that is, the core) of sandwich panels. [4-6]

- Deep sea buoyancy foams
- Thermoforming plug assist
- Radar transparent materials
- Acoustically attenuating materials

The modelizations at the macroscopic level are rather scant. The spectrum of engineering applications of syntactic foams is quite broad. First and more significant applications of syntactic foams date back to the 50 in the area of naval and marine engineering, where they have been used for structural elements such as hulls, ribs and decks, for components at deep depth such as submarines, submerged buoys, deep-sea platforms and pipe joints, and for shielding and repairing submerged apparatuses. Syntactic foams are often used in civil and industrial engineering, in construction and as an imitation of wood and marble; often they are employed as core materials due to good shear stiffness and strength, fatigue and impact resistance. [7-8]

From the literature survey done so far it can be seen that syntactic foam has been a vital composite material in the making of buoyancy aid materials. Syntactic foam has been developed using chopped strand glass fibers and in another case using epoxy and polyester resins with microspheres to develop syntactic foams. In addition, the uses of glass hollow microspheres were used to develop the syntactic foam. The use of linear low-density polyethylene (LLDPE) has been very rare in the formation of syntactic foams. In the present work, the use of LLDPE with glass microsphere as microspheres to fabricate syntactic foam has been analyzed.

LLDPE in the development of syntactic foam has not been studied and its behavior to form syntactic foam composites has yet to be analyzed. The work done on fabrication and estimating the characteristics of Linear Low density Polyethylene with Glass microsphere as additive to form syntactic foam can be implemented in buoyancy aid applications, marine applications, cushioning and package material. The work done on fabrication and estimating the characteristics of CuO NPs and Linear Low density Polyethylene with Glass microsphere as additive to form syntactic foam can be implemented in buoyancy aid applications, marine applications, cushioning and package material.

2. Experimental procedure

The systematic approach was adapted to arrive at the results of the project work. The syntactic foam was prepared using LLDPE and Glass Microsphere. These chemicals were heated to 180˚C in a Compression moulding machine for nearly 45 minutes resulted in the formation of syntactic foam. Using ASTM standards the various specimens were fabricated

for the testing of tensile, compression, flexural and indentation tests. The results and values obtained from all the different testing are consolidated and analyzed.

Linear Low density Polyethylene and microspheres were taken in different ratios. The mixture of LDPE and microspheres was then placed in a clean stainless steel die. The stainless steel die used was with the following dimensions 140x140x10 mm as shown in fig.1.(a)

Figure .a) Stainless Steel Die b) Die with aluminum foil c) Syntactic foam

Aluminum foil was placed inside the die before the mixture was placed in the die fig.1 (b). This is done so that the foil acts as a remover after developing the syntactic foam. The mixture placed evenly in the die was placed in a Compression moulding machine.

The Compression moulding machine has a temperature range from 0˚C to 180˚C.The stainless steel die with a mixture is heated to a temperature of 180˚C so that the mixture reaches reaction temperature and syntactic foam is formed. The foam thus formed is allowed to cool and then sent for machining fig.1(c). The synthesized foam is the cut and machined to required dimensions as per ASTM standards for various mechanical testing.

2.2 Testing of Specimens

The LLDPE & Microsphere composites which are going to be analyzed for mechanical behavior where prepared with following compositions as shown in Table.1 to find out the Tensile, Compression and Flexural tests as per the following standards.

Table .1. Specimen Composition

Specimen	LLDPE (grams)	Glass Microspheres (grams)	% of Microsphere
Specimen 1	100	0	0
Specimen 2	100	1	1
Specimen 3	100	3	3
Specimen 4	100	5	5

Tests to be carried are

Test A – Tensile test – ASTM D638

Test B– Compression Test – ASTM D695

2.3. Mechanical testing

The tensile test and compressive test were carried out to find the breaking tensile strength and compressive strength of Syntactic foams. The tensile properties were determined using a universal tensile testing machine (FIE Pvt. Ltd, India) according to ASTM D-3039 standard.

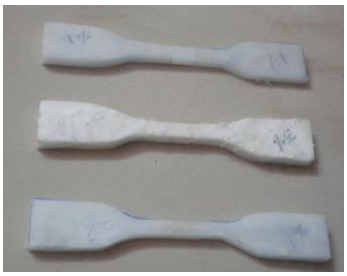

Fig.2. Specimens for Tensile Test

2.4. Micro structural analysis

Transmission electron microscopic studies were conducted on Syntactic foams samples with optimized mechanical property using transmission electron microscope (JEOL JEM 1200EX), operated at the operating voltage of 200 KV. It gives information about the dispersion of Syntactic foams and so it is a direct evidence for the formation of nanocomposites. Scanning Electron Microscopy studies were carried on CGRP samples, to analyze the delamination during machining by drilling using HITACHI-S3400N Scanning Electron Microscope.

III Experimental Results

3.1 Tensile Test Results

The fabricated specimens for tensile testing as per ASTM D638 were tested using Universal testing machine (UTM). The syntactic foam specimens with different compositions were tested and the tensile strength was determined. The tensile strength can be obtained by using the formula.

$$\sigma = \frac{P}{A}$$

Where σ = Tensile strength, P= Breaking Load, A= Cross sectional Area

Consolidated Test Results of Tensile Strength

The tensile strength of syntactic foam with various composition of microsphere was obtained and is given in Table 2. The composition of LLDPE has been kept constant but the addition of glass microsphere with LLDPE has been varied from 0% to 5% and corresponding change in tensile strength has been observed. The change in the tensile strength decreases as the percentage of microsphere in LLDPE increases.

Table.2.Comparison of Tensile Strength for various specimens

Specimen	LLDPE (grams)	Glass Microspheres (grams)	% of Glass Microsphere	Tensile strength in MPa
Specimen 1	100	0	0	14.8
Specimen 2	100	1	1	12.6
Specimen 3	100	3	3	11.6
Specimen 4	100	5	5	10.2

3.2 Compression Test Results

The specimens for compression testing were fabricated as per ASTM standards. The compression testing was carried out using UTM and the strengths were determined. The values of compression strength were measured from the stress – strain curve developed. The Compression Strength was taken from the linear section of the stress-strain curve. 10% values of the strain values with respect to stress values have been taken to calculate the compression strength

Test Results of Compression Strength

The compression strengths of various percentage of microsphere addition are shown in Table. Three the microspheres are added the compression strength vary from four Mpa to 11 Mpa for 0% microsphere addition to 5% microsphere addition respectively. The compression strengths increase with the addition of microsphere. This is due to the fact that as microsphere content increase in the composite matrix they form bubble shaped structures that aid in the cushioning effect which show good elastic deformation during compression. This shows that when compression takes place the specimen acts like a sponge with little deformation after the compression.

Table.3. Comparison of Compression Strength for various specimens

SI.NO	Test Conducted	%	Sample	Strength in Mpa	Standard Deviations sample	Strength of composite with 1.0wt% NPs
1	Tensile strength D638	0	Sample 1	4.45	0.6527	15.1
			Sample 2	15.56		
			Sample 3	14.41		
		1	Sample 1	12.6	0.3917	
			Sample 2	13.37		
			Sample 3	13.11		
		3	Sample 1	11.6	0.4366	
			Sample 2	12.25		
			Sample 3	12.43		
		5	Sample 1	9.53	0.3534	
			Sample 2	10.2		
			Sample 3	9.67		
2	Compressive stress D 695	0	Sample 1	15.95	0.1955	16.7
			Sample 2	16.12		
			Sample 3	15.73		
		1	Sample 1	12.95	0.0793	
			Sample 2	12.98		
			Sample 3	13.1		
		3	Sample 1	7.6	0.2379	
			Sample 2	7.9		
			Sample 3	7.43		
		5	Sample 1	4.6	0.3404	
			Sample 2	3.92		
			Sample 3	4.23		

3.3 *Consolidated Test Results of Syntactic Foam*

The Table 4 shows the values obtained for various percentages of microsphere addition from 0% to 5% respectively. The values of Tensile strength, Flexural strength and Compression strength variation have been tabulated. The Tensile strength varies from 14.8 MPa to 10.2 MPa. While flexural strength varies from 12.6 MPa to 39.5 MPa. The compression strength increases from 0% with 15.95 MPa to 4.6 MPa at 5% addition of microspheres.

Table.4.Consolidated Test Results of Syntactic Foam

Specimen	LDPE (grams)	Microspheres (grams)	% of Microsphere	Compressive Strength in Mpa
Specimen 1	100	0	0	15.95
Specimen 2	100	1	1	12.9
Specimen 3	100	2	2	7.6
Specimen 4	100	3	3	4.6

3.4 Thermal stability of composite

The TG curves for the pure composite and composite with 1.0wtpercentage CuO NPs are illustrated in Fig. The results show that there is only one-step of weight loss for the pure composite and CuO based nanocomposite. The weight loss of the nanocomposite is smaller than the pure composite and this is mainly due to dispersion of the CuO NPs in the base material. As seen in Fig., the first step occurs at a temperature between 520 °C and 810 °C and this thermal degradation is due to the molecular chains breakage of the LLDPE. The paraffin acted as the thermal retardant and consequently, they had delayed the decomposition of the composite. Thermal retardant property of the CuO NPs would deter the process of conversion of flammable molecules to the gas phase. This result proves that CuO based achieved the better thermal stability, rather than the pure composite.[4]

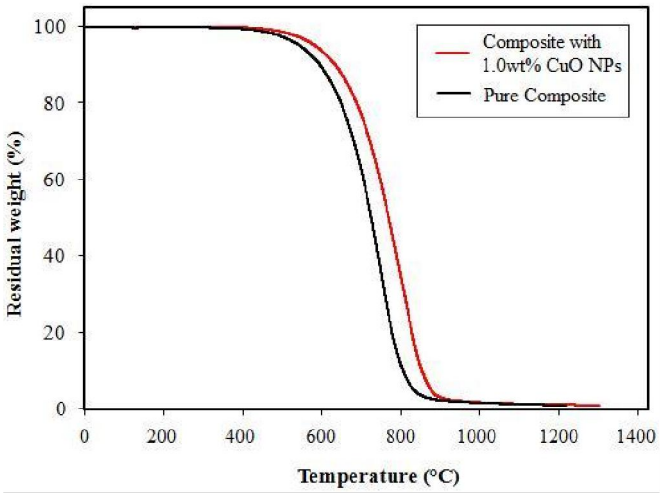

Fig.3.TG curves for composite and nanocomposite

3.5FESEM Image of CuO Nanoparticles

The FESEM image as shown in Figure. The shape of the CuO particles synthesized by the aqueous precipitation method is found to be rod-like. The rod structure would provide more contacting area to the base material, compared to the rectangular and spherical shapes.

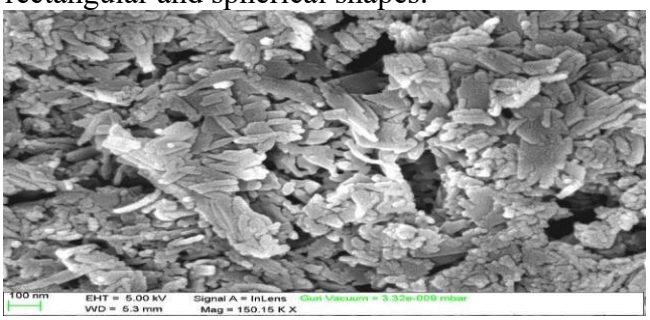

Fig.4. FESEM Image of CuO Nanoparticles

3.6 TEM Image of CuO nanoparticles

The TEM image shows the size of the CuO particles lying in the range from 12 to 50 nm, and the nanoparticles seem to have a rod-like structure. These Nano rods would certainly be beneficial to ascertain for improved adhesion. [5]

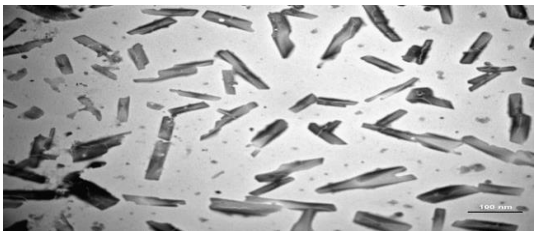

Fig.5.TEM Image of CuO Nanoparticles

4. Results and Discussion

The methodology has been developed for manufacturing of syntactic foam using LLDPE as base material with microspheres as additive in formation of bubbles in it.

Tensile Strength

The mass percentage of microsphere when gradually added to the LLDPE in the formation of syntactic foam has revealed a decrease in the tensile strength. When there was no mass percentage of microspheres added to LLDPE the tensile strength obtained was 14.48 MPa.

As the mass percentage of microsphere was added at 1%, 3%, and 5% respectively the tensile strengths obtained where 12.6 MPa, 11.6 Mpa, and 10.2 Mpa respectively and the addition of microsphere has caused the reduction in the tensile strength of the syntactic foam. When the mass percentage of microsphere has increased from 1% to 5%, the tensile strength has been reduced

Compression Strength:

The mass percentage of microsphere when gradually added to the LLDPE in the formation of syntactic foam has revealed an increase in the Compressive strength. When there was no mass, percentage of microspheres added to LLDPE the Compressive strength obtained was 15.95 Mpa. As the mass percentage of microsphere was added at 1%, 3%, and 5% respectively the flexural strengths obtained were 12.9 Mpa, 7.6 Mpa and 4.6 MPa respectively and the

Addition of microsphere has caused the reduction in the compression strength of the syntactic foam. When the mass percentage of microsphere has increased from 1% to 5%, the flexural strength has been decreased

Tensile and compression tests of Nano based composites:

The tensile stress–strain curves of the composite and Nano composite using 1.0wtpercentage CuO NPs. While comparing with pure composite, tensile strength of increased substantially and this is mainly due to dispersion of NPs.The mean tensile strength was all increased by homogeneous dispersion of NPs (+48 in the base material. On the other hand, elongation at break was slightly decreased but this reduction is negligible due to the high standard deviations observed for this property (10–20%).The compression stress–strain curves of the pure composite and Nano composite using 1.0wt% CuO NPs are given in the Fig. While comparing with pure composite, composite strength of the Nano composite was augmented notably and this is solely due to dispersion of NPs. The mean compression strength was also increased by homogeneous dispersion of NPs (36%) in the base material. To conclude on this part, CuO NPs dispersed in the pure composite have provided the larger surface area for given mass and as a result; this larger surface area ensures the strong adhesion between the glass microsphere and LLDPE. An improved tensile and compression strengths of the Nano composite are due to the size, shape and mass fraction of the nanoparticles to be dispersed in the base material.

5. Conclusion

In this study, the preparation of CuO nanoparticles (NPs) and investigation on the thermomechanical properties of the CuO NPs embedded in the polymer composites were demonstrated experimentally. LLDPE and glass microsphere were considered the base material for the preparation of the Nano based polymer composites. Surface morphology of the CuO NPs as synthesized was carried out by FESEM.

Fabrication of the composites was accomplished by means of compression moulding press. An improvement of the tensile and compression properties of the composite was obtained by dispersion of the CuO NPs. This can be explained by better adhesion between base material and flake-like structure of the CuO NPs.The test results obtained from the TG study revealed that an addition of CuO NPs had acted as the thermal retardant and CuO NPs had delayed thermal degradation of the Nano based polymer composites. Based on the test results, it can be understood that the newly fabricated nanocomposites have achieved the improved thermal and mechanical properties.

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