



MECHANICAL PROPERTIES OF NANOIRON PARTICLES REINFORCED EPOXY/POLYESTER NANOCOMPOSITES

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ABSTRACT

We report on synthesis of two highly dissolve nanoparticles viz. Fe_2O_3 & $f-Fe_2O_3$ using chemical reduction method. Reaction effect was initiated to mix up solution 1 (i.e. $FeCl_3 \cdot 6H_2O$) into solution 2 (i.e. $FeCl_2 \cdot 4H_2O$) as one under the occurrence of ammonium to build up nanoiron (NI) particles. Mechanical properties as above mentioned nanoiron particles filled with polyester and epoxy nanocomposites were fabricated to assess the possibility of using this filler as a latest material. Functionalization agent as Methacryloxypropyl was used to prepare $f-Fe_2O_3$ nanoparticles. $f-Fe_2O_3$ nanocomposites of mechanical properties were improved with the help of functionalization when compared with nanocomposites of Fe_2O_3 . Nanoiron particles functionalization favours the composite fabrication with a curing temperature at low as compared to the as-synthesised nanoparticles filled polyester nanocomposites. Mechanical properties carried out are Hardness, Impact strength, Tensile strength, Flexural strength and Compression strength. Mechanical property values increased due to the homogeneous nanoparticle dispersion and chemical bonding between polyester matrix and nanoparticles. After incorporation nanoiron particles into the polyester resin matrix it becomes magnetically harder. Machines generated mechanical property values were compared and analysed with system generated software analysis of variance (ANOVA) values. Machine values and ANOVA values were measured for the

specimens of epoxy+polyester+nanoiron, where the nanoiron is varying viz. 1, 2, 3, 4, 5 and 7 wt.%.

Key words: Mechanical Properties, Nanocomposites, Epoxy/Polyester and ANOVA.

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1. INTRODUCTION

Today we know well, even though not understood completely, that nanoparticles can influence polymer properties by crystallization, electrical, thermal conductivity, mechanical strength, melt processing and visco-elasticity among others. The best two examples of the above mentioned are the large mechanical degradation stability and higher stiffness rendered to polymer matrices by nanoiron filler particles adding in small amounts. characterization of mechanical tests namely tensile, flexural, compression, hardness and impact tests on epoxy/polyester nanocomposites at different nanoiron variations viz., 1, 2, 3, 4, 5 and 7 wt.% are presented. Results and discussions are presented based on the type of test and their experimental results and graphs. In this case at least six samples are tested at classified interval. Organic materials with both conducting ferromagnetic properties have received tremendous attention due to their potential applications in batteries, electrochemical display devices, molecular electronics, nonlinear optics, sensors, electrical- magnetic shields and microwave-absorbents.

The possibility of adjusting the polymer blending offers cost concert balance and couture the technology to create products for specific use of applications which extends engineering resin's performance, improves specific properties and provides revenue for industrial and consumer plastics ravage recycling. Polymer blends combination with wood and other cellulose materials appears quite promising on the basis of balanced performance, re-utilization of plastic wastes and recyclables after the end use. Among various polymer blends and alloys, modification of epoxy and polyester matrix combinations are attractive route to promote the performance of the thermosetting matrix; because their blends are expected to improve impact, tensile, flexural and moisture resistance properties and the low cost polyester with excellent mechanical and barrier properties of epoxy. Machine values were measured for the composition of epoxy + polyester + nanoiron. In this nanocomposite we have taken epoxy + polyester as constant throughout all the nanoiron variations. Mechanical properties like tensile strength, flexural strength, compression strength, hardness, and impact strength will be validating through the system software and they are comparing with system values.

2. MATERIAL AND METHODS

In the present work, a commercially available polyester, catalyst and accelerator were purchased from the V.G.R. Enterprises, Madurai, Tamialnadu, India. Poly-ester monomers with two reactive poly end groups facilitate the crosslinking for network formation. The liquid resin has a density of 1.231 g/cm³ and a viscosity of 370 centipoises (cps) at room temperature. Nanoiron particles with an average diameter of 10-15 nm and a specific surface area of 45m²/g were functionalized and used as nanofillers for the nanocomposite fabrication. Trigonox 239-A (curing catalyst or initiator, organic peroxide, liquid) was purchased from Akzo Nobel Chemicals. Cobalt naphthenate was used as a catalyst promoter to decompose the catalyst at room temperature. Methacryloxypropyl-trimethoxysilane and tetrahydrofuran were purchased

from Sigma–Aldrich Chemical Company. All the chemicals were used as-received without further treatment. All the tests were carried at ambient conditions. In this case, six identical specimens were tested. All the tests were accomplished at a room temperature.

3. PREPARATION OF FERRITE NANOPARTICLES

Reaction was carried out by mixing two solutions, named as solution A (i.e. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and solution B (i.e. $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) to form nanoparticles. 0.1 Mole of 27.030 gms of solution A was dissolved in 1000 ml de-ionised water, whereas, 19.881 gms of solution B was dissolved in 1000 ml deionised water used were to prepare solutions. Then solution A was added in solution B drop wise by 2:1 stoichiometric ratio under vigorous stirring. Ammonium (45ml) was dropped in to the mixture; forthwith black powder was produced in less than one second. After reaction, the product particles were separated from the solution by a strong magnet and washed with deionised water. Freeze-drying overnight was utilized to dry the particles.

4. FABRICATION OF BLENDED/NANOCOMPOSITES

Then pre-calculated amount of epoxy/polyester (i.e. 85/15; w/w ratio) were mixed together in a suitable beaker. Hardener/accelerator/catalyst/promoter (100:10/2/2/2) parts by weight was added to the modified epoxy/polyester mixer. A glass mould with required dimensions was used for making sample on par with ASTM standards and it was coated with mould releasing agent enabling to easy removal of the sample. Brush and roller were used to impregnate composite. The closed mold was kept under pressure for 24 hrs at room temperature. To ensure complete curing the blended composite samples were post cured at 80°C for 1 hr and the test specimens of the required size were cut out from the sheet. Composites were prepared by compounding with extrusion and hot press machine. The processing temperature is maintained at 180°C and the pressure was almost all constant. The extruded composites were hot pressed under 10MPa for 5min at 180 °C into sheets of suitable thickness for making the specimens as per ASTM standard. Sheet size and thickness were dependent on the testing methods used in this study.

5. DETERMINATION OF MECHANICAL PROPERTIES OF THE COMPOSITES

Tensile, compression and flexural properties of the composites were determined using a UTM (Instron, Series-3369) with cross head speed of 5mm/min. In Tensile strength, three point bending tests were carried out on par with ASTM D 53455, ASTM D 690 and ASTM-53452, respectively. All the tests were performed in a displacement controlled mode on a closed-loop servo-hydraulic MTS testing machine. Impact strength of samples was measured on Zwick impact strength testing machine (ZIS 250) according to ASTM D 53433. Rockwell hardness properties were performed using Rockwell hardness testing machine (Model-2000R) according to ASTM D 256. All the tests were accomplished at a room temperature of 24 °C.

6. VALIDATION ANALYSIS OF VARIANCE

Statistical analysis (regression and ANOVA) of the responses are carried out to estimate the coefficient polynomial of the response by regression and to check the significance of the regression coefficients of independent variables and interaction variables by ANOVA. Analysis of variance (ANOVA) table is used to determine the significance of the first degree, second degree and cross-product terms of the polynomial. In this case, the adequacy of the model is confirmed when the Model Probability > F is less than 0.05. Analysis of Variance validates the results from the machine values with predicted values which are generated by system software.

7. RESULTS AND DISCUSSIONS

Mechanical properties

Table 1 shows the obtained experimental results for the effects of different nanoiron variations viz., 1, 2, 3, 4, 5 and 7 wt.% on all the properties of Hardness, Impact strength, Tensile strength, Flexural strength and Compression strength. Tensile properties such as tensile strength and elongation at break of the nanoiron composites containing 1%, 2%, 3%, 4%, 5% and 7% nanoiron as filler were measured and the results are presented in the **table.1**

Table 1 Mechanical properties (Experimental results for epoxy + polyester + nanoiron)

Filler (Wt. %)	Hardness number	Impact strength	Tensile strength	Flexural strength	Compression strength
1	95.50	160.71	42.65	20.11	121.63
2	100.83	175.04	46.20	24.80	124.53
3	104.03	182.96	49.09	26.05	130.63
4	105.41	182.45	50.00	23.85	132.45
5	103.98	180.96	44.09	22.42	128.20
7	96.02	178.86	45.02	24.33	125.36

Analysis of Hardness

Table 2 ANOVA for Hardness

Source	Sum of squares	df	Mean square	F-value	p-value
Model	92.47197	2	46.236	1285.42	0.0000398
A-NI	17.26864	1	17.2686	480.09	0.00021
A ²	92.3372	1	92.3372	2567.09	0.0000169
Residual	0.107909	3	0.03597		
Cor Total	92.57988	5			

From the above Table.2, it has been observed that, the Model F-value of 1285.42 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of “Prob > F” less than 0.0500 indicate model terms are significant. In this case A, A² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Analysis of Impact Strength

Table 3 ANOVA for Impact Strength

Source	Sum of squares	df	Mean square	F-value	p-value
Model	350.3641	3	116.788	332.319	0.003002
A-NI	18.38593	1	18.3859	52.317	0.018583
A ²	21.24668	1	21.2467	60.4573	0.016141
A ³	39.95484	1	39.9548	113.691	0.008681
Residual	0.702866	2	0.35143		
Cor Total	351.0669	5			

From the Table.3, it has been noticed that, the Model F-value of 332.319 implies the model is significant. There is only a 0.30% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, A², A³ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Analysis of Tensile Strength

Table.4 ANOVA for Tensile Strength

Source	Sum of squares	df	Mean square	F-value	p-value
Model	47.75638	4	11.9391	83.8997	0.081678
A-NI	23.58546	1	23.5855	165.742	0.049351
A ²	10.31843	1	10.3184	72.5107	0.074421
A ³	19.24028	1	19.2403	135.207	0.054615
A ⁴	10.20175	1	10.2018	71.6908	0.074841
Residual	0.142302	1	0.1423		
Cor Total	47.89868	5			

From the Table.4 it shows that the Model F-value of 83.8997 implies there is 8.17% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A is a significant model term. Values greater than 0.1000 indicate the model terms are not significant.

Analysis of Flexural Strength

Table 5 ANOVA for Flexural Strength

Source	Sum of squares	df	Mean square	F-value	p-value
Model	21.32017	3	7.10673	49.0187	0.020059
A-NI	12.08639	1	12.0864	83.3661	0.011784
A ²	2.160906	1	2.16091	14.9049	0.061016
A ³	15.32088	1	15.3209	105.676	0.009331
Residual	0.28996	2	0.14498		
Cor Total	21.61013	5			

From the Table.5 indicates that the Model F-value of 49.0187 implies the model is significant. There is only a 2.01% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, A³ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Analysis of Compression Strength

Table.6 ANOVA for Compression Strength

Source	Sum of squares	df	Mean square	F-value	p-value
Model	81.83864	4	20.4597	13684.03	0.006411
A-NI	13.46936	1	13.4694	9008.71	0.006707
A ²	21.53121	1	21.5312	14400.7	0.005305
A ³	12.74912	1	12.7491	8526.99	0.006894
A ⁴	11.87629	1	11.8763	7943.22	0.007143
Residual	0.001495	1	0.0015		
Cor Total	81.84013	5			

From the above Table.6 it is clearly indicated that the Model F-value of 13684.03 implies the model is significant. There is only a 0.64% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, A², A³, A⁴ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Table 7 Actual and predicted values of hardness

Run	A: NI in %	Hardness (actual values) MPa	Hardness (predicted values) MPa	% Deviation
1	1	95.5	95.6641	0.1715
2	2	99.53	101.028	1.4827
3	3	107.53	104.254	3.1423
4	4	105.41	105.353	0.0541
5	5	101.88	104.295	2.3155
6	7	96.52	95.7858	0.7665

From the above Table.7 it indicates that the actual and predicted hardness values for the nanocomposites with nanoiron as an input parameter. The percentage deviation between actual and predicted values indicates accurate prediction within the limits of $\pm 4\%$.

Table.8 Actual and predicted values of impact strength

Run	A: NI in %	Impact Strength (actual values) MPa	Impact Strength (predicted values) MPa	% Deviation
1	1	160.71	160.091	0.3866
2	2	173.04	175.315	1.2976
3	3	184.96	182.000	1.6263
4	4	181.45	182.919	0.8030
5	5	180.96	180.845	0.0635
6	7	178.86	178.810	0.0279

It indicates in the table.8 that the actual and predicted impact strength values for the nanocomposite with nanoiron as an input parameter. The percentage deviation between actual and predicted values indicates accurate prediction within the limits of $\pm 2\%$.

Table.9 Actual and predicted values of tensile strength

Run	A: NI in %	Tensile Strength (actual values) MPa	Tensile Strength (predicted values) MPa	% Deviation
1	1	42.65	42.6783	0.0663
2	2	46.20	46.0643	0.2945
3	3	50.09	50.3444	0.5053
4	4	50.00	49.7739	0.4542
5	5	44.09	44.1743	0.1908
6	7	45.02	45.0143	0.0126

It indicates from the table.9 that the actual and predicted tensile strength values for the nanocomposite with nanoiron as an input parameter. The percentage deviation between actual and predicted values indicates accurate prediction within the limits of $\pm 1\%$.

Table.10 Actual and predicted values of flexural strength

Run	A: NI in %	Flexural Strength (actual values) MPa	Flexural Strength (predicted values) MPa	% Deviation
1	1	20.11	20.086	0.1150
2	2	24.80	24.945	0.5840
3	3	26.05	25.711	1.3169
4	4	23.85	24.209	1.4829
5	5	22.42	22.263	0.7024
6	7	24.33	24.343	0.0546

It indicates from the table.10 that the actual and predicted flexural strength values for the nanocomposites with nanoiron as an input parameter. The percentage deviation between actual and predicted values indicates accurate prediction within the limits of $\pm 2\%$.

Table.11 Actual and predicted values of compressive strength

Run	A: NI in %	Compression Strength (actual values) MPa	Compression Strength (predicted values) MPa	% Deviation
1	1	121.63	121.633	0.0024
2	2	124.53	124.516	0.0112
3	3	130.63	130.656	0.0199
4	4	132.45	132.427	0.0173
5	5	128.2	128.209	0.0070
6	7	125.36	125.359	0.0007

It indicates from the table.11 that the actual and predicted compression strength values for the nanocomposite with nanoiron as an input parameter. The percentage deviation between actual and predicted values indicates accurate prediction within the limits of $\pm 1\%$.

8. CONCLUSIONS

In the present work two highly nano disperse Fe_2O_3 & $f-Fe_2O_3$ nanoparticles were synthesized through chemical reduction method and then dispersed into the epoxy/polyester polymers. Tensile strength, Flexural strength, compression strength, hardness and impact strength mechanical properties were studied on machine generated values and software (ANOVA) generated values. These values were compared and evaluated to percentage deviation to test the results might be accurate. In the case of these values, ANOVA seemed to reduce p-values but the machine generated values were more as expectations.

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