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Parametric Study of Twin Screw Extrusion for Processing Epoxy/Carbon Black Nanocomposites Using Orthogonal Array Technique

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Abstract

This paper presents the experimental investigation of the influence of process parameters of twin screw extrusion on the dispersion and electrical, thermal and mechanical properties of epoxy/CB nanocomposites. Four factors, namely, loading level of carbon black (0, 2, 4, 6, 8 and 10 wt%) in epoxy, screw speed (75, 100 and 125 rpm), temperature (5 °, 15 ° and 30 °C) and number of passes (5, 10 and 15) were selected for the design of L_{18} OA layout. Response factors such as electrical resistivity, glass transition temperature, microhardness and impact strength of epoxy/CB nanocomposites were studied. Highest electrical conductivity was observed at 10 wt% CB loading, 30 °C, 5 passes and 100 rpm. Highest microhardness, impact strength and glass transition temperature were obtained at 10 wt%, 15 passes, 75 rpm and 15 °C. From TGA, the addition of CB to epoxy resulted in improved thermal stability. ANOVA of the experimental results showed that CB loading had significant influence on all the responses, followed by number of passes. The Grey relational grade was highest for factor level combination 10 wt% CB loading, 15 passes, 15 °C and 75 rpm. Volume conductivity increased from 4.92×10^{-15}(epoxy) to 2.2×10^{-5} S-cm^{-1} (10 wt% CB/epoxy). Similarly, microhardness, glass transition temperature and impact strength increased by 29.96, 35.92 and 69.23% respectively due to the addition of 10 wt% CB to epoxy, corresponding to the best parameter combinations obtained by Grey relational analysis.

Keywords: *carbonblack (CB), orthogonal array (OA), thermo-gravimetric analysis (TGA), DOE: design of experiment, analysis of variance (ANOVA)*

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INTRODUCTION

Epoxy (LY556) has extensive application in aerospace and automotive industries for structural applications. However, it's insulating nature results in local heating and premature degradation of the structures and electronic components due to the accumulation of electrostatic charge on their surface. To avoid electrostatic charging of an insulating matrix, an electrical conductivity greater than 10^8 S-cm^{-1} is required. Conducting epoxy can be used in various applications including electromagnetic radiation shielding, electrostatic discharge (ESD) protection and electro-packaging applications to avoid electrical charges and electrical magnetic field from disturbing the communications due to its superior structural property.

Electrical conductivity can be achieved by incorporating highly conductive fillers, such as CB particles, carbon fibers, metallic fillers or intrinsically conducting polymers in thermosets and thermoplastics [1–6, 8–16]. Graphite and CB also have advantage of being compatible with many polymer systems. Though incorporating carbon nanotubes (CNT) and carbon nanofibers (CNF) in plastics exhibits better mechanical and electrical properties [8–14], the automobile and aerospace industries cannot rely on these materials for mass production due to high costs.

The nanofillers are dispersed in polymeric resins by several mechanical methods like ultrasonication, magnetic agitation, high-speed stirring, shear mixing, etc. The improvement in the end properties due to the addition of CB is dependent on filler quality, amount of CB, and the dispersion technique adopted [1–5]. Twin screw extrusion used for dispersing nanoclay in vinylester yielded superior results [6–7].

Several authors investigated electrical behavior of different grades of epoxies and found that the percolation threshold varies between 0.5 and 4% nanofiller content. Th. V. Kosmidou *et al.* investigated the effect of dispersion of carbon black in epoxy (DGEBA/TETA) on electrical and mechanical properties. In both with and without post cured cases, at lower filler contents, T_g increased up to the maximum value (at about 0.7 wt% filler). Typical dielectric behavior was observed below 1% CB. At higher CB contents conductivity increased significantly.

Though electrical property improvements of different grades of epoxy are reported, such studies involving LY556 which has extensive application in automobile and aerospace industries are scarcely available and also parametric study of twin screw extrusion for processing epoxy (LY556)/CB nanocomposites is not reported. Hence, the research was mainly focused on parametric studies of twin screw extrusion for dispersing CB in LY556.

EXPERIMENTAL

Materials

The resin used in this study was bisphenol-based epoxy of grade LY556 supplied by Huntsman, Hindustan Ciba-Geigy Ltd. The curing agent for this resin was Hardener HY951. Nanofiller used for fabrication of nanocomposites was carbon black N220 grade supplied by Philips Carbon Black Ltd.

Design of Experiments

CB was dispersed using ultrasonication followed by twin screw extrusion (Alpha 18, Steer Engineering, Bangalore). Six levels for CB loading and three levels each for number of passes, temperature and screw speed were selected. Based on the factors and levels, L_{18} Orthogonal array was selected. The physical layout for the designed experiments is shown in Table 1.

***Table 1:** Physical Layout for the Designed Experiment.*

Expt. No.	CB (%)	No. of passes	Temp (°C)	Screw Speed (rpm)
1	0	5	5	75
2	0	10	15	100
3	0	15	30	125
4	2	5	5	100
5	2	10	15	125
6	2	15	30	75
7	4	5	15	75
8	4	10	30	100
9	4	15	5	125
10	6	5	30	125
11	6	10	5	75
12	6	15	10	100
13	8	5	10	125
14	8	10	15	75
15	8	15	5	100
16	10	5	30	100
17	10	10	5	125
18	10	15	15	75

Nanocomposite Preparation

CB/Epoxy Specimens

The CB was dispersed by wt. 0, 2, 4, 6, 8 and 10% in epoxy resin using ultrasonicator 37 kHz for 1 h using tip sonicator and later by twin screw extrusion as per DOE. The screw extruded epoxy/CB gelcoat was mixed with hardener in 11:100 ratio as per manufacturer's recommendation and cured at room temperature for 24 h.

Electrical Conductivity

Volume and surface resistivity were measured as per ASTM D257 using Keithley 6517 model 8009 test fixture. A voltage of 200 V was applied on the specimen for a span of 60 s to measure the conductivity. The bias voltage was set to 200 V and bias time was set to 60 s. Three sets of readings were taken for each experiment. The resistivity values were calculated using the formula:

$$\sigma_v = \frac{22.9}{t} R \qquad 2.1$$

$$\rho_s = 53.4R \qquad 2.2$$

Where σ_v and ρ_s are the volume and surface resistivities, R the corresponding resistance in ohms (meter reading), 22.9 and 53.4 the apparatus constants. The resistivity and corresponding conductivity are presented in Table 2.

Microhardness

Microhardness test was carried out using Vickers microhardness tester supplied by Metatech. Vickers microhardness test was carried out by indenting the test specimen subjected to a load of 0.1 Kg-f for a period of 20 s with a pyramid-shaped diamond tip. Three replicates were performed for each experiment and results are tabulated as shown in Table 2. The average values were calculated using three replicate values, based on the relationship:

$$HV = \frac{F}{A} \approx \frac{1.854F}{d^2} \qquad 2.3$$

Glass Transition Temperature

Glass transition temperature of the epoxy/CB was studied using differential scanning calorimetry (Model – Mettler DSC – 823, temperature range: 25–500 °C). The sample weighing 5 mg sealed in a hermetic aluminum crucible was used for the characterization. For obtaining the curing heat flow pattern of the composite, a dynamic scanning experiment was conducted in the range of room temperature to 200 °C at a heating rate 10 °C/min. The experiment was carried out in nitrogen (N_2) atmosphere with a flow rate of 20 ml/min. The response (T_g) obtained after performing designed experiment is listed in Table 2.

Impact Strength

Impact strength of nanocomposite samples was evaluated as per ASTM D256, using instrumented impact testing machine (International Equipments, Mumbai, capacity: up to 25 J, release angle of pendulum: 150 °). The specimens of size 64 mm long × 12.7 mm wide × 3 mm thick were used for this investigation. The R1 scale was used as the impact load which is having a range of 0–2.71 J. Three tests were carried out to obtain average impact energy, which was then divided by the thickness of the specimens to get the impact strength. The results are listed in Table 2.

RESULTS AND DISCUSSION

Electrical Conductivity, DSC and Mechanical Properties of Epoxy/Carbon Black

The volume conductivity and surface conductivity results are plotted as a function of filler content as shown in Figure 1 which indicates that there is a significant drop in resistivity at 2 wt% CB loading due to the formation of percolation threshold. The volume conductivity increased from 4.92E-15 to 3.83E-05 S/cm at 10 wt% CB loading. The surface conductivity increased by 1.7E-14 S to 3.5E-06 S at 10 wt% CB loading. The least resistivity was observed at 10 wt% CB loading. It can be concluded that higher content of CB in the material results in reduced resistivity. The dc conductivity of epoxy/CB nanocomposites near percolation threshold follows universal scaling laws given below:

$$\sigma_{pc} = A\,(W - W_C)^t, \quad W > W_C$$

Table 2: *Response Values of Epoxy/CB Nanocomposites.*

Expt. No.	Avg. Vol conductivity (S/cm)	Avg. surface conductivity	Microhardness (VHN)			Avg microhardness	Glass transition temp °C	Impact strength N/m
			1	2	3			
1.	4.92498E-15	--	20.2	20.9	20.4	20.5	103.65	86.66
2.	4.59664E-15	1.7E-14	19.4	18.9	19.6	19.3	102.59	90.00
3.	5.01932E-15	1.5E-14	20.3	21.2	20.6	20.7	103.27	86.90
4.	1.47527E-07	1.3E-14	20.8	20.6	21.0	20.8	113.01	96.66
5.	1.35056E-07	1.1E-08	21.1	20.9	21.3	21.1	110.75	100.00
6.	4.54876E-07	9.7E-09	22.2	22.5	22.5	22.4	116.8	113.33
7.	1.05649E-07	5.4E-09	21.8	21.7	21.9	21.8	118.4	106.33
8.	5.28243E-07	3.2E-09	22.4	22.3	22.0	22.2	118.52	110.00
9.	1.20187E-06	4E-08	21.8	22.2	22.3	22.1	115.05	126.66
10.	7.93966E-07	1.7E-07	21.6	21.9	21.9	21.8	120.01	126.66
11.	1.86086E-06	2.8E-08	22.8	23.5	23.3	23.2	122.91	130.00
12.	1.84773E-06	5.4E-07	23.1	23.4	23.1	23.2	122.54	133.33
13.	1.72374E-06	3.6E-07	23.5	23.4	23.9	23.6	127.8	126.33
14.	1.72148E-07	5.8E-07	24.3	24.4	24.5	24.4	133.1	143.33
15.	2.54377E-06	2.6E-07	24.2	24.3	24.1	24.2	129.96	136.66
16.	2.18341E-05	8E-06	25.8	25.6	26.0	25.8	134.74	146.66
17.	3.86444E-05	2.2E-06	25.9	26.2	26.3	26.1	139.68	143.33
18.	3.23468E-05	4.3E-06	26.4	26.2	26.0	26.2	140.23	146.66

Where σ_{pc} is the volume conductivity of the composite, W is the weight fraction of CB in the composite, W_C is the critical volume fraction (percolation threshold), A and t are fitted constants. Theoretical predictions of the critical exponent, t ranges from 1.5 to 2.0, while experimental values between 1.3 and 3.1 have been reported. The experimental values of 'A' as in Table 3 was greater compared with the similar works reported in the literature [2, 4].This might be due to better physical contact between the adjacent CB. At concentrations above 2 wt% CB, the resistivities were observed to be low and decrease marginally with increasing CB content. The temperature, numbers of passes and screw speed had very little effect on the conductivity when compared to that of CB loading in Figure 1.

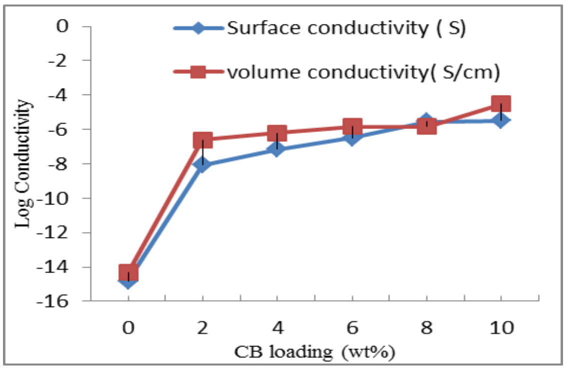

Fig. 1: *Volume and Surface Resistivity for Epoxy/CB Nanocomposites.*

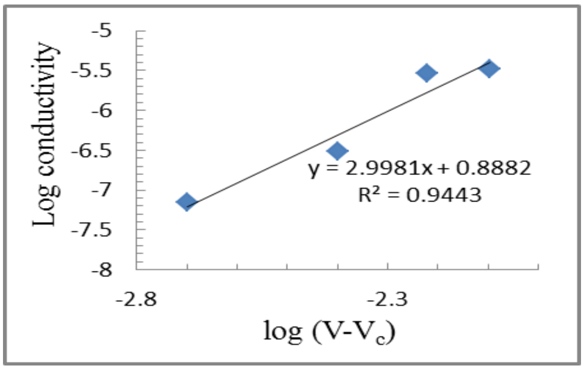

Fig. 2: *Log Conductivity v/s Log (V-V_c) for Epoxy/CB Nanocomposite.*

Table 3: *Power Law Equation Parameters.*

Parameter	Epoxy/CB Nanocomposite
t	2.998
A	2.41

Microhardness

Figure 3 shows that the microhardness has increased with increased CB content in the material and attained maximum value at 10 wt% CB. The microhardness increased with increase in number of passes. The maximum microhardness was found in 10 wt% CB loading which showed 30% increase in microhardness when compared to that of epoxy without any reinforcement. The increase of the CB content resulted in increase of the number of high strength reinforcements inside the composites, thus increasing their microhardness property. Good dispersion due to twin screw extrusion resulted in better interfacial bonding causing enhancement in microhardness. The microhardness increased with increase in number of passes as it provided higher residence time for processing and level 1 and level 3 of temperature had similar effect but level 2 resulted in reduced microhardness. Lower screw provided greater residence time for processing the mixture hence micro-hardness increased at lower screw speed.

T_g of Epoxy/CB Nanocomposites

Differential scanning calorimetry was used to determine the T_g of the nanocomposites using the measurement of heat flow versus change in temperature. Figure 4 shows the variation of T_g with increase in CB loading. The T_g was highest at 10% wt CB loading and resulted in 37% increase in T_g. The number of passes and temperature had very little effect on T_g but the same decreased with increase in screw speed. The level 1 of number of passes resulted in reduced T_g when compared to other two levels.

Impact Strength of Epoxy/CB Nanocomposites

Figure 5 shows the variation of impact strength of the nanocomposite with CB content. The impact strength was maximum at 10 wt% processed at 15 °C, 15 passes and 75 rpm and showed 50% increase in impact strength at this combination. The increase in impact strength may be due to efficient stress transfer between the matrix and the filler as a result of the interfacial interaction. The impact strength increased with increase in number of passes. The lower screw speed provided greater residence time for extrusion, hence resulted in increased impact strength. Levels 1 and 3 of temperature resulted in similar effect but level 2 resulted in reduced impact strength.

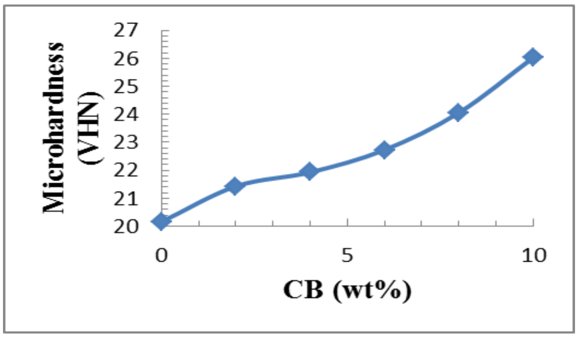

Fig. 3: *Microhardness for Epoxy/CB Nanocomposite.*

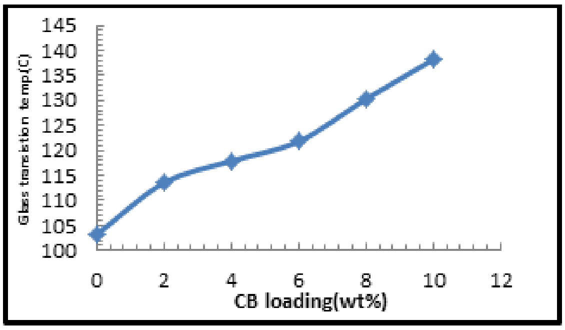

Fig. 4: *Tg for Epoxy/CB Nanocomposite.*

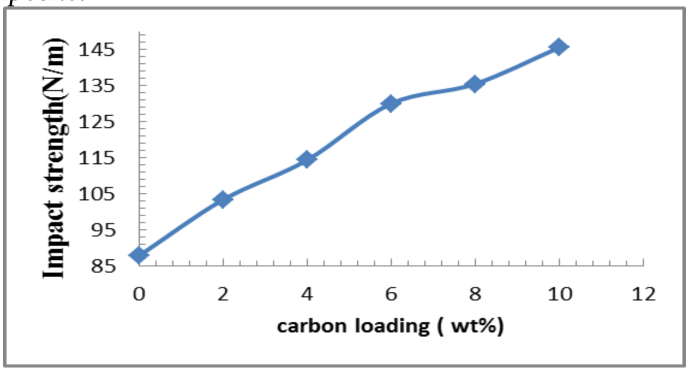

Fig. 5: *Impact Strength of Epoxy/CB Nanocomposite.*

Signal-to-Noise Ratio

To determine the effect each variable on the output, the signal-to-noise ratio needed to be calculated for each experiment conducted. Taguchi recommends analyzing data using the S/N ratio that will offer two advantages; it provides guidance for selection of the optimum level based on least variation around on the average value, which is closest to target, and also it offers objective comparison of two sets of experimental data with respect to deviation of the average from the target. Average S/N ratio for each response of the experiment were calculated based on the relation smaller the better, larger the better and nominal the best.

a) Smaller the better: This is usually the chosen S/N ratio for all undesirable characteristics like defects, damages, instability, etc., for which the ideal value is zero. Also, when an ideal value is finite and its maximum or minimum value is defined then the difference between measured data and ideal value is expected to be as small as possible. The generic form of S/N ratio then becomes

$$SN_S = -10\log\left(\frac{1}{n}\sum_{i=1}^{n} Y_i^2\right) \qquad 4.1$$

where Y is the observed data and n is the number of observations.

b) Larger the better: The larger-the-better characteristic should be non-negative, and its most desirable value is infinity. For a maximum of non-negative heat efficiency, yield, or non-defective product rate, the larger the better value is merely 1 (100%).

Therefore, they are not larger the better characteristics. On the other hand, amplification rate, power, strength, and yield amount are larger-the-better characteristics because they do not have target values and their larger values are desirable.

$$SN_L = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Y_i^2}\right) \qquad 4.2$$

c) Nominal the best: This case arises when a specified value is most desired, meaning that neither a smaller nor a larger value is desirable. Its target value is non zero and finite. For these problems when the mean becomes zero, the variance also becomes zero.

$$SN_T = 10\log\left(\frac{\bar{Y}^2}{S^2}\right) \qquad 4.3$$

Table 4: *S/N Ratio for Epoxy/CB Nanocomposite.*

Expt. No.	S/N ratio of volume conductivity	S/N ratio of surface conductivity	S/N ratio of microhardness	S/N ratio of T_g	S/N ratio of impact strength
1.	−286.152	−275.566	−24.9857	−39.062	−37.507
2.	−286.751	−276.558	−24.4618	−38.9727	−37.8355
3.	−285.987	−277.897	−25.07	−39.0553	−37.507
4.	−136.623	−159.312	−25.1119	−39.813	−38.4555
5.	−137.39	−160.233	−25.2363	−39.6375	−38.7506
6.	−126.842	−165.327	−25.7556	−40.0995	−39.8375
7.	−139.523	−169.992	−25.5197	−40.2176	−39.2837
8.	−125.543	−147.937	−25.6777	−40.2264	−39.5785
9.	−118.403	−135.535	−25.6385	−39.9706	−40.8034
10.	−122.004	−151.186	−25.5197	−40.335	−40.8034
11.	−114.606	−125.325	−26.0604	−40.5424	−41.0295
12.	−114.667	−128.929	−26.0604	−40.5162	−41.2492
13.	−115.271	−124.746	−26.2089	−40.8812	−40.7807
14.	−135.282	−131.752	−26.4984	−41.2342	−41.8774
15.	−111.89	−101.954	−26.4269	−41.0268	−41.4634
16.	−93.2173	−113.094	−26.983	−41.3476	−42.0768
17.	−88.2583	−107.271	−27.0834	−41.6533	−41.8774
18.	−89.8034	−109.079	−27.1166	−41.6874	−42.0768

From Table 4, 10% CB (level 6), 10 passes (level 2), 5 °C (level 1) and 125 rpm of experiment 17 has the highest S/N ratio with respect to both volume conductivity and surface conductivity and resulted in the best performance with least resistivity in both cases as shown in Table 4.

The S/N ratio for the response microhardness is highest for the combination 10% CB (level 6), 15 passes (level 3), 15 °C (level 3) and 75 rpm (level 1) in the experiment 18 and has the best performance glass transition temperature and impact strength from Table 4, it is clear that experiment 18 has the highest S/N Ratio with best performance with the factors 10% CB (level 6), 15 passes (level 3), 15 °C (level 3) and 75 rpm.

ANALYSIS OF VARIANCE (ANOVA)

ANOVA is mainly carried out to analyze the statistical significance of different factors at different levels on the response variables. It is performed based on the DOE for all S/N ratios. MINITAB module was used to perform ANOVA choosing general linear model (GLM). This module was used to analyze the effect of factors on the responses and their significance on the responses of designed experiment. It has been performed for 0, 2, 4, 6, 8 and 10 wt % epoxy/CB nanocomposite to examine the effect of process parameters on the twin screw extrusion. Form analysis of variance from Table 5 to Table 8, it is evident that CB is the most significant factor followed by number of passes, temperature and screw speed respectively for influencing the conductivity, microhardness, Tg and impact strength.

Table 5: *ANOVA Results for Volume Conductivity.*

Source	DF	Sum of squares (SS)	Adj SS	Adj mean square	F (variance ratio)	P (probability)	rank
CB (%)	5	74175.9	74175.9	14835.2	301.62	0.000	1
No. of passes	2	204.8	204.8	102.4	2.08	0.206	2
Temp.	2	103.9	103.9	51.9	1.06	0.405	3
Screw speed	2	65.3	65.3	32.6	0.66	0.549	4
Residual error	6	295.1	295.1	49.2			
Total	17	74844.9	**R^2 = 99.6%**				

Table 6: *ANOVA Results for Surface Conductivity.*

Source	DOF	Sum of squares (SS)	Adj SS	Adj mean square	F	P (probability)	Rank
CB loading	5	55395.9	55395.9	11079.2	298.41	0.000	1
No. of passes	2	476.7	476.7	238.4	6.42	0.032	3
Temperature	2	624.6	624.6	312.3	8.41	0.018	2
Screw speed	2	204.4	204.4	102.2	2.75	0.142	4
Residual error	6	222.8	222.8	37.1			
Total	17	56924.5		**R^2 = 99.6%**			

Table 7: *ANNOVA Results for Microhardness.*

Source	DOF	Sum of squares (SS)	Adj SS	Adj mean square	F	P (probability)	Rank
CB loading	5	9.17093	9.17093	1.83419	49.09	0.000	1
No. of passes	2	0.25565	0.25565	0.12783	3.42	0.102	2
Temperature	2	0.07472	0.07472	0.03736	1.00	0.422	4
Screw speed	2	0.15937	0.15937	0.07968	2.13	0.200	3
Residual error	6	0.22419	0.22419	0.03737			
Total	17	9.88485	**R^2 = 93.6%**				

Table 8: *ANOVA for S/N Ratio of Glass Transition Temperature.*

Source	DOF	Sum of squares (SS)	Adj SS	Adj mean square	F (variance ratio)	P (probability)	Rank
CB loading	5	12.7273	12.7273	2.54546	37.02	0.000	1
No. of passes	2	0.1237	0.1237	0.06185	0.90	0.455	2
Temperature	2	0.0421	0.0421	0.02106	0.31	0.747	3
Screw speed	2	0.2128	0.2128	0.10638	1.55	0.287	4
Residual error	6	0.4125	0.4125	0.06876			
Total	17	13.5184	**R^2 = 96.9%**				

Table 9: *ANOVA for S/N Ratio of Impact Strength.*

Source	DOF	Sum of squares (SS)	Adj. SS	Adj. mean square	F	P (probability)	Rank
CB loading	5	40.5138	40.5138	8.10276	33.62	0.000	1
No. of passes	2	1.3536	1.3536	0.67678	2.81	0.138	2
Temperature	2	0.2525	0.2525	0.12624	0.52	0.617	3
Screw speed	2	0.1174	0.1174	0.05871	0.24	0.791	4
Residual error	6	1.4460	1.4460	0.24100			
Total	17	43.6833	**R^2 = 96.7%**				

S/N Ratio analysis and ANOVA revealed that CB loading was the most significant factor on the resistivity, microhardness, T_g and impact strength. The resistivity decreased as loading of CB increased. A percolation threshold less than 2 wt% was obtained in both volume and surface resistivity. Resistivity decreased with increase in number of passes and with decrease in temperature. The screw speed had little effect on resistivity.

The microhardness, T_g and impact strength increased with increase in CB loading and number of passes and with decrease in temperature. The screw speed had little effect on T_g and impact strength.

Figure 6 shows the effect of process parameters on the response volume conductivity. It is clear that the conductivity increases with increase in CB loading. 0 wt% CB loading resulted in lowest conductivity and 10 wt% CB loading resulted in highest conductivity. A significant increase in conductivity is observed at 2 wt% due to the formation of percolation threshold. Also from Figure 7, it is observed that conductivity increases with increase in number of passes. Level 3 of the factor number of passes had more effect when compared to level 1 and level 2. Level 1 of the factor temperature had an influential effect compared to other two levels. Twin screw speed has a very little effect on conductivity when compared to other factors.

Figure 8 shows the main effect plot for S/N ratio values for microhardness. The microhardness has increased with increasing CB loading. The continuous increase of the nanopowder content resulted in increasing the number of high strength reinforcements inside the composites, increasing their microhardness property. The highest microhardness is obtained at 10 wt% CB loading and resulted in 27% increase in microhardness. The epoxy/CB nanocomposites showed better microhardness at 15 passes as more shear force was exerted on the mixture while processing.

Temperature has very little effect on microhardness when compared to other factors. The epoxy/CB nanocomposite processed at 75 rpm exhibited higher microhardness compared to those processed at 100 and 125 rpm.

Similar trend was observed in main effect plots for S/N ratio values of glass transition temperature and impact strength. The 10 wt% CB has resulted in highest T_g and impact strength resulting in 37 and 69% increase in T_g

and impact strength respectively. Here also level 3 (15 passes) was having more effect on the response when compared to other two levels. The 75 rpm screw speed resulted in better response in both Tg and impact strength.

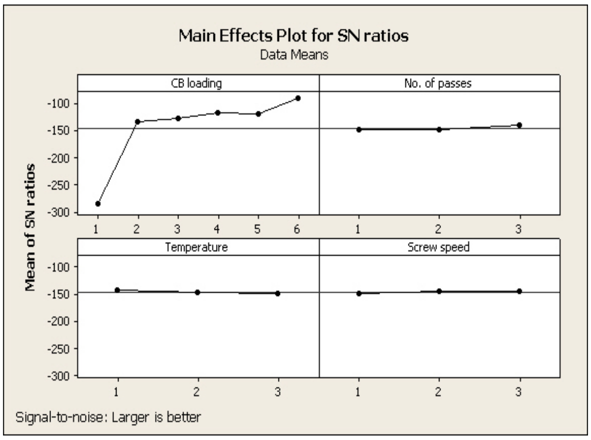

Fig. 6: *Main Effects Plot for S/N Ratio Values of Volume Conductivity.*

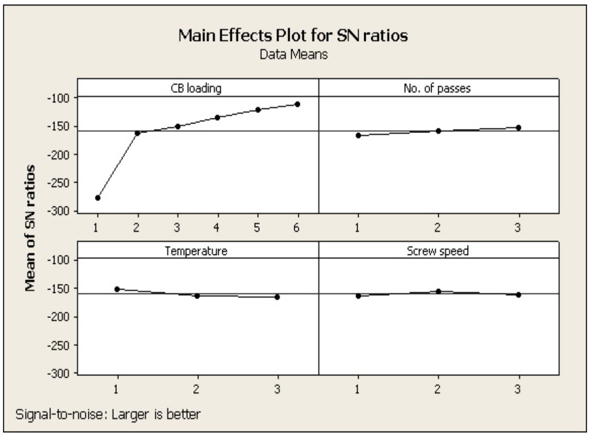

Fig. 7: *Main Effects Plot for S/N Ratio Values of Surface Conductivity.*

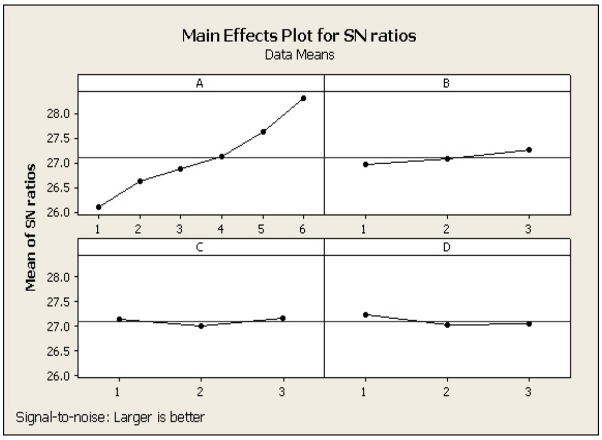

Fig. 8: *Main Effects Plot for S/N Ratio Values of Microhardness.*

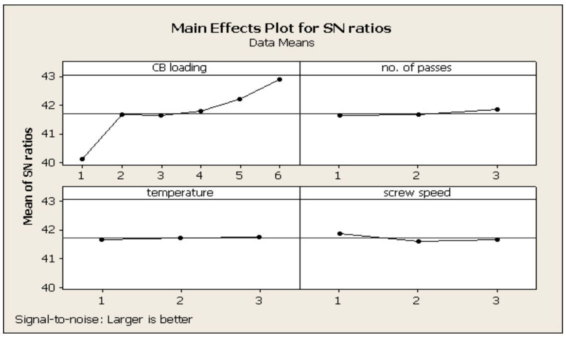

Fig: 9: *Main Effects Plot for S/N Ratio Values of Glass Transition Temperature.*

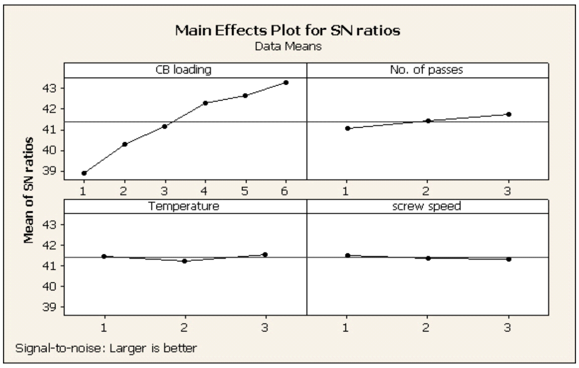

Fig. 10: *Main Effects Plot for S/N Ratio Values of Impact Strength.*

Grey Relational Analysis

Grey relational analysis was carried out to optimize the parametric combination of twin screw extrusion for processing epoxy/CB nanocomposites. The multiple performance characteristics included conductivity, microhardness, T_g and Impact strength.

In Grey relational analysis, normalization of experimental data was performed. Linear normalization of the experimental results was performed in the range between zero and unity. The normalized data processing corresponds to larger the better type for all the responses.

$$X_i(k) = \frac{Yi(k) - MinY(k)}{MaxY(k) - MinY(k)} \qquad 6.1$$

where,
$\Delta_{oi} = \| X_o(K) - X_i(K) \|$ is the difference of the absolute value between $X_o(K)$ and $X_i(K)$ and ξ is the distinguishing coefficient between zero and one.
In this study ξ value is taken as 0.5
Δ_{min} = smallest value of Δ_{oi}
Δ_{max} = largest value of Δ_{oi}

Table 10: *Rank Assignments to Experiments Based on Grey Relational Grades.*

Expt. No.	Conductivity	Micro-hardness	T_g	Impact strength	Ξ				Gray relational grade	Rank
					Res	Micro-hardness	Impact	T_g		
1	4.92E-15	20.5	103.65	86.66	0.250	0.250	0.250	0.264	0.275	17
2	4.60E-15	19.3	102.59	90.00	0.250	0.250	0.278	0.250	0.257	18
3	5.02E-15	20.7	103.27	86.66	0.250	0.351	0.250	0.259	0.278	16
4	1.48E-07	20.8	113.01	96.66	0.252	0.359	0.333	0.388	0.333	15
5	1.35E-07	21.1	110.75	100.00	0.252	0.380	0.361	0.358	0.338	14
6	4.55E-07	22.4	116.8	113.33	0.256	0.475	0.472	0.439	0.410	11
7	1.06E-07	21.8	118.4	106.33	0.251	0.431	0.414	0.460	0.389	13
8	5.28E-07	22.2	118.52	110.00	0.257	0.460	0.445	0.462	0.406	12
9	1.20E-06	22.1	115.05	126.66	0.265	0.453	0.583	0.416	0.429	10
10	7.94E-07	21.8	120.01	126.66	0.260	0.431	0.583	0.481	0.439	9
11	1.86E-06	23.2	122.91	130.00	0.274	0.533	0.611	0.520	0.484	8
12	1.85E-06	23.2	122.54	133.33	0.274	0.533	0.639	0.515	0.490	7
13	1.72E-06	23.6	127.8	126.33	0.272	0.562	0.581	0.585	0.500	6
14	1.72E-07	24.4	133.1	143.33	0.252	0.620	0.722	0.655	0.562	4
15	2.54E-06	24.2	129.96	136.66	0.283	0.605	0.667	0.614	0.542	5
16	2.18E-05	25.8	134.74	146.66	0.532	0.721	0.750	0.677	0.670	3
17	3.26E-05	26.1	139.68	143.33	0.672	0.743	0.722	0.743	0.720	2
18	3.83E-05	26.2	140.23	146.66	0.746	0.750	0.750	0.750	0.749	1

From Table 10, it can be concluded that the combination 10 wt% CB, 15 passes, 15 °C temperature and 75 rpm screw speed had the highest value of grey relational grade and thus gets the highest rank. This indicates that this factor combination of experiment 18 is the optimal.

Thermogravimetric Analysis

Thermogravimetric analysis for the best parametric combination of twin screw process variables on dispersion of CB in epoxy was analyzed and compared with that of neat epoxy. TGA testing was carried out in TGA TA instruments Q500 V20.2 Build 27 (Central Power Research Institute, Bangalore) in controlled nitrogen atmosphere. The sample weighing 5 mg was taken in platinum furnace for the characterization. For obtaining the thermal stability and heat flow pattern of the composite, test was conducted in the range of 0 °C to 800 °C at a heating rate 20 °C/min. The decomposition of the neat epoxy and epoxy/CB composite is shown in Figure 11. From Figure 11, it is clear that addition of CB to epoxy resulted in improved thermal stability. 10 wt% CB/epoxy nanocomposites processes at 10 °C, 15 passes at 75 rpm had 4.7% more residual at 800 °C than 0 wt% epoxy/CB nanocomposites. The addition of CB to epoxy had resulted in 50 °C increase in thermal stability.

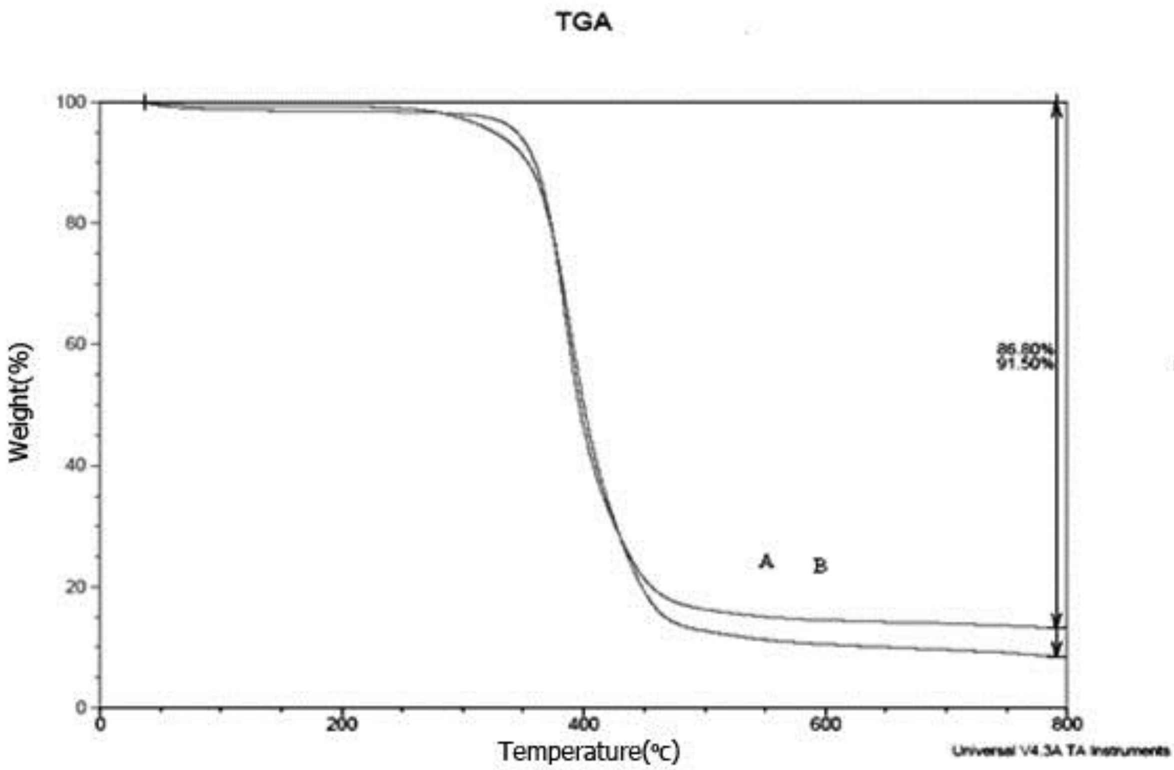

Fig. 11: *TGA Curves Epoxy Nanocomposite (Expt.3 and18).*

CONCLUSIONS

Epoxy/CB nanocomposites were fabricated using ultrasonication and twin screw extrusion to study the effect of CB loading on the electrical, thermal and mechanical properties of the nanocomposites. The effect of process parameters on the responses such as volume and surface conductivity, microhardness, T_g and impact strength were analyzed. Based on the experimental results, the following conclusions were drawn:

S/N ratio analysis and ANOVA revealed that CB loading was the most significant factor on the resistivity, microhardness, T_g, and impact strength. The resistivity decreased as loading of CB increased. A percolation threshold at less than 2 wt% was obtained in both volume and surface resistivity. Resistivity decreased with increase in number of passes and decrease in temperature. The screw speed had little effect on resistivity.

The microhardness, T_g and impact strength increased with increase in CB loading and number of passes and with decrease in temperature. The screw speed had little effect on T_g and impact strength.

The Grey relational analysis helped to arrive at the best parameter combination 10 wt% CB loading, 15 passes, 15 °C and 75 rpm, to achieve the best responses of resistivity, microhardness, T_g and impact strength collectively. The resistivity decreased by 1.0E10, microhardness increased by 29.96%, T_g increased by 35.92% and impact strength increased by 69.23% at the best parameter combination as per Grey relational analysis. From TGA, the addition of CB to epoxy resulted in improved thermal stability.

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