

Dry Sliding Wear Behaviour of Micro oven Treated Fly Ash Reinforced Epoxy Composite using Extended Taguchi Approach Optimization

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Abstract

Fly ash epoxy composite has been developed in ultrasonic mixing method with the aim of reducing the impact of hazardous waste like fly ash on the environment. Post curing has been done in micro oven to modify the surface so as to have better wear resistance properties. An orthogonal array, L_{32} has been designed with the influencing parameters like % fly ash, applied normal load, speed, track diameter and time of operation. A multi objective parametric condition, TOPSIS is used to convert it into a single objective optimization technique and ANOVA helps in getting the percentage contribution of each parameter for the wear of the material. In general, this article emphasizes the importance of micro oven treatment on the surface modification of polymeric material which retards the wear.

Keywords: Fly ash; Epoxy resin; micro oven; TOPSIS; Taguchi; ANOVA

1. Introduction

High strength at low weight makes the polymer composites usable in automobile, aerospace industries and various household appliances. Particulate reinforced polymer composites are advantageous over fibre reinforced polymer composites due to low fabrication cost and less raw material cost. The choice of material for a particular application depends on the variables like material cost, thickness, quality and working conditions. Particulate reinforced polymer composites can be used for low energy transfer where wear plays an important role in determining the life of the material. The possible reason in sudden increase in wear is due to frictional heat generated at the contact surface and loss of material [1]. So, wear is an important field which has to be analysed in perspective of increasing the life span of the material. Various researchers have found the effect of various parameters like sliding distance, sliding speed, time and applied load on the wear of the material [2]. Some researchers have also concluded that the particulate filled polymer composites are mostly encouraging building materials because of their reasonable determination of lattice and fortifying strong molecule stage. It prompts a composite with a blend of quality modulus better than those of traditional metallic materials [3]. Some polymer composites find application in high wear applications like pump wear rings, bushings, line shaft bearing and pressure reducing bushings. Ceramic filler like fly ash has been introduced to epoxy resin with the aim of increasing the mechanical, electrical and thermal properties simultaneously. Fly ash is a major hazardous waste generated in million tons from the thermal power plants has silica and alumina as the major constituent. Earlier researchers have done some work on the mechanical properties of the fly ash epoxy composite but very little work has been done on tribology of the material [4-9]. Previously, effort has been made to show the effect of parameters without any curing process. A slight but effective modification has been done in the form of micro oven curing to increase the wear resistance



of the material. And it has been found that wear resistivity increases after micro oven curing. It may be due to the total modification of the polymer composite.

2. Experimental details

2.1 Fabrication of composite

Class C fly ash used in this experimental work is collected from CPP-2 of Rourkela Steel Plant, Odisha, India. SEM micrograph shown in figure 1 reveals that the particles are round in shape but having different diameters. Particle size analysis done in MALVERN MASTERIZER and a graph has been plotted between particle size and volume percentage of particle analysed as shown in figure 2. The mean diameter is found to be 27.26 μm . Detail chemical compositional analysis shown in table 1 reveals that silicon and aluminium only constitute more than 70% of the total volume which make it a good ceramic material. Epoxy resin (LY556), chemically named Bisphenol-A-Diglycidyl-ether and hardener (HY951) supplied by ATUL industries limited, Kolkata is used as a matrix material due to its easy availability and low cost. It is a high dense polymer having density 1129 g/cm^3 . The mixing ratio between epoxy resin and hardener is fixed as 10:1 by weight percentage. Fly ash is mixed with epoxy resin at four different weight percentages like 10, 20, 30 and 40. It is put under an ultrasonic sonicator with the horn dipped inside for 30 minutes. A pulse time of 5 sec is given during the mixing time. After the mixture is ready, pre weighed hardener is mixed gently by hand stirring method to avoid trapping of bubbles. Once the mixture is ready, it is poured gently to plastic moulds having diameter 10mm and allowed to harden in atmospheric condition for 24 hours. Once it is hardened, the plastic pipe is cut and solid cylindrical composites are removed and kept carefully for further use. The composites are further micro oven treated at 120 $^{\circ}\text{C}$ for 30 minutes. The micro oven treated condition is determined by trail basis.

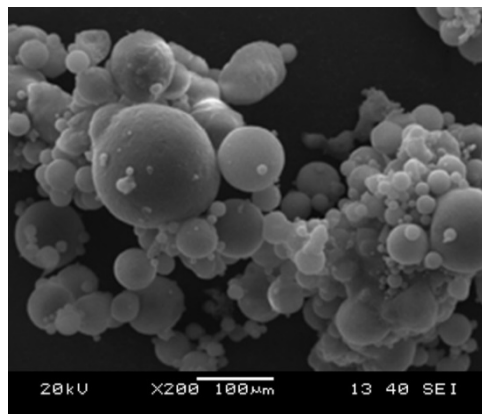


Figure 1: SEM image of fly ash.

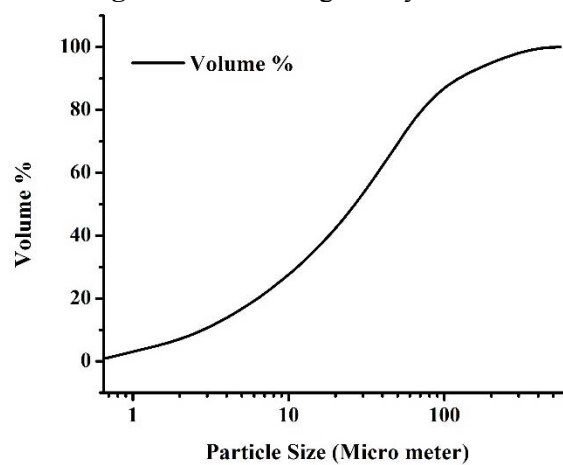


Figure 2: Particle size analysis of fly ash.

Table 1: Chemical composition of fly ash

Constituents	Percentage (%)
Fe ₂ O ₃	8.1
MgO	1.14
Al ₂ O ₃	24.98
SiO ₂	55.85
P ₂ O ₅	0.15
SO ₃	1.16
K ₂ O	0.85
CaO	2.54
Na ₂ O	0.2
TiO ₂	1.75
CO ₃	1.56
LOI	1.72

Dry sliding wear test has carried out as per ASTM G99 standard. A schematic diagram in figure 3 show various parts of a dry sliding wear test rig. Surface morphology has been carried out using JEOL 6480LV scanning electron microscope. Thermal analysis has been carried out using Mettler Toledo-822 low temperature DSC (Differential Scanning Calorimetry) with temperature -100 °C to +400 °C. Fourier transform infrared spectroscopy (FTIR) applied to measure radioactive thermal properties of thin polymer films for application in an intermediate temperature range. A “Shimadzu”, IR presige-21, automatic infrared microscope with a wavelength range 350 cm⁻¹ to 7800 cm⁻¹ is used for structural characterization of the composite.

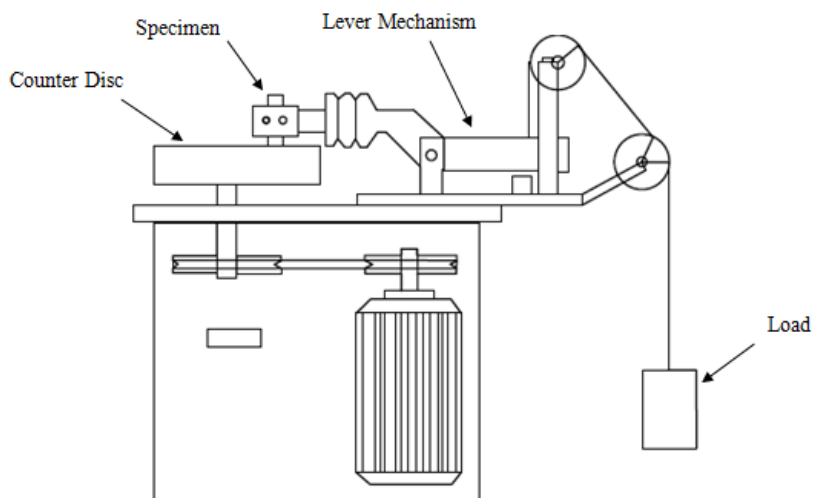


Figure 3: Schematic diagram of sliding wear test rig.

2.2 Taguchi design

Wear plays an important role in characterization of any material as it is directly concerned with the weight loss of material. To reduce the number of experiments Taguchi L32 orthogonal array is applied and results have been obtained. Each wear test gives three outputs like wear in micrometre, frictional force and coefficient of friction. As it is giving three different outputs, so initial steps are taken to make it a single parametric optimization technique. To make it a single objective optimization, TOPSIS method has been applied. After making it a single objective optimization, Taguchi optimization technique has been applied to get the suitable parametric combination and ANOVA gives the percentage contribution in each situation. Confirmatory test has been done to find the percentage of error in the experiment. Percentage of reinforcement of fly ash, duration of sliding,

speed, track diameter and load are considered as five control variables and altered at different levels as demonstrated in table 2 in this experimental work.

Table 2: Levels of variables used in the experiment

Control Factors	Unit	Levels			
		1	2	3	4
Time (A)	min	5	10		
% of FA (B)		10	20	30	40
Speed (C)	RPM	200	400	600	800
Track Diameter (D)	mm	40	50	60	70
Load (E)	N	10	20	30	40

Taguchi method is used to find out the influence of each parameter. In this experiment no noise factor or interactions between parameters is considered. Table 3 shows the L32 orthogonal array with three desired outputs in the form of wear, frictional force and coefficient of friction. The three output parameters have been converted into a single relative closeness coefficient which is also mentioned in table 3 and it has been found out by using TOPSIS method. Signal to noise ratio is calculated from the output results. As we require minimum value of these results, so it is a lower is better type experiment and the result having lower value of S/N ratio is treated as the most suitable result.

The S/N ratio is calculated as, $\eta = -10 \log_{10} [1/n \sum y_i^2]$ where $1 \leq i \leq n$ (1)

Where n is the number of experiments carried out. Therefore, the ideal level of the input factors is the combination of individual parameters with levels having the maximum signal to noise ratio. Despite the performance characteristics, a noteworthy signal to noise ratio resembles to better performance with least variation. Therefore, the ideal level of the input factors is the combination of individual parameters with levels having the maximum signal to noise ratio.

2.3 Basics of TOPSIS method

The traditional TOPSIS strategy is taking into account the thought that the best option ought to have the most limited separation from the positive perfect arrangement and the best separation from the negative perfect arrangement. TOPSIS model shows the alternative ways of normalizing the data and measuring the distances from the mean position. TOPSIS method has superior advantages with regard to the adaptability of its evaluation method and the accuracy of evaluation result. The TOPSIS method consists of the following steps:

- Normalizing the decision matrix

$$r_{ij} = \frac{X_{ij}}{\sqrt{\sum_{k=1}^m X_{kj}^2}}, i = 1, \dots, m; j = 1, \dots, n \quad (2)$$

Multiplying the columns of normalized matrix with associated weights. Here similar weights have been assigned to each parameter i.e.0.33.

$$V_{ij} = W_j \times r_{ij}, \quad (3)$$

- Determine the positive ideal and negative ideal solutions respectively,

$$A^+ = \{v_1^+, v_2^+, \dots, v_n^+\} = \left\{ \left(\max v_{ij} \mid j \in K_b \right) \left(\min v_{ij} \mid j \in K_c \right) \right\} \quad (4)$$

$$A^- = \{v_1^-, v_2^-, \dots, v_n^-\} = \left\{ \left(\min v_{ij} \mid j \in K_b \right) \left(\max v_{ij} \mid j \in K_c \right) \right\} \quad (5)$$

Where, K_b = Set of benefit criteria and K_c = Set of cost criteria

Obtaining the distances of the existing alternatives from the positive ideal and negative ideal solutions, two distances for each alternative are, respectively, calculated as follows:

$$S_i^+ = \sqrt{\sum_{j=1}^n (v_{ij} - v_j^+)^2}, i = 1, 2 \dots m \quad (6)$$

$$S_i^- = \sqrt{\sum_{j=1}^n (v_{ij} - v_j^-)^2}, i = 1, 2 \dots m \quad (7)$$

- Calculate the relative closeness to the ideal alternatives;

$$RC_i = \frac{S_i^-}{S_i^+ + S_i^-} \quad i=1, 2, \dots, m \quad 0 \leq RC_i \leq 1 \quad (8)$$

- Rank the alternatives according to their relative closeness to the ideal alternatives, the bigger RC_i , the better alternative A_i .

3. Results and discussion

3.1 Statistical Analysis

For each treatment condition 32 numbers of tests have been carried out and results in the form of wear, frictional force and coefficient of friction are obtained with accordance to the change in parameters as demonstrated in table 3. In all the three cases, these are multi objective parametric conditions are converted to single objective condition by applying TOPSIS. A relative closeness value is obtained for each condition which is the combined output of all the outputs. The optimal parametric combination is then assessed which would come about most noteworthy relative closeness value.

Table 3: L32 Orthogonal array with results for micro oven curing condition

Sl no	% FA	Time (Min)	Speed (RPM)	Load (N)	Track diameter (cm)	Wear (μm)	Frictional force (N)	COF (μ)	Relative closeness coefficient (P)
1	10	5	200	10	40	80.32	4.41	0.2	0.052048
2	10	5	400	20	50	126.66	11.16	0.3	0.080975
3	10	5	600	30	60	103.55	13.47	0.2	0.115109
4	10	5	800	40	70	110.72	20.83	0.4	0.158343
5	10	10	200	40	40	98.13	17.01	0.3	0.147733
6	10	10	400	30	50	70.91	12.32	0.3	0.148024
7	10	10	600	20	60	280.53	7.07	0.3	0.024583
8	10	10	800	10	70	41.8	6.82	0.6	0.140271
9	20	5	200	10	50	102.74	1.98	0.17	0.018908
10	20	5	400	20	40	39.79	3.99	0.23	0.091138
11	20	5	600	30	70	198.63	13.93	0.5	0.065534
12	20	5	800	40	60	143.25	18.63	0.72	0.115085
13	20	10	200	40	50	147.05	21.27	0.3	0.126366

14	20	10	400	30	40	82.43	16.91	0.22	0.170223
15	20	10	600	20	70	67.92	9.03	0.24	0.117349
16	20	10	800	10	60	58.89	5.02	0.39	0.078548
17	30	5	200	20	60	50.68	11.92	0.35	0.190415
18	30	5	400	10	70	23.77	5.88	0.38	0.198314
19	30	5	600	40	40	54.41	17.73	0.21	0.245772
20	30	5	800	30	50	72.43	12.37	0.18	0.145873
21	30	10	200	30	60	181.22	15.06	0.17	0.076727
22	30	10	400	40	70	221.49	17.92	0.11	0.074851
23	30	10	600	10	40	183.17	5053	0.8	0.965018
24	30	10	800	20	50	163.77	3.46	0.6	0.02069
25	40	5	200	20	70	84.55	5.22	0.2	0.058149
26	40	5	400	10	60	41.57	4.04	0.32	0.088577
27	40	5	600	40	50	138.49	11.36	0.4	0.075809
28	40	5	800	30	40	23.98	10.11	0.1	0.296568
29	40	10	200	30	70	58.63	10.85	0.23	0.15616
30	40	10	400	40	60	84.72	14.1	0.28	0.142684
31	40	10	600	10	50	35.8	4.46	0.34	0.11078
32	40	10	800	20	40	36.87	10.15	0.41	0.215866

The average wear values for diverse levels of selected parameters are indicated in tables 4. From the table4 below, the average value of time at level 2 is higher than the average value of time at level 1 which indicates that level 2 is better than level 1. Similarly, better choices have been found out in other parameters.

Table 4: Mean response table for relative closeness coefficient for micro oven curing

Level	TIME	FLY ASH	RPM	LOAD	TRACK DIA
1	-1.770	-2.058	-1.562	-3.967	-3.485
2	-3.088	-2.052	-1.693	-2.039	-1.965
3		-4.020	-4.127	-1.526	-2.189
4		-1.586	-2.334	-2.184	-2.078
Delta	1.317	2.434	2.565	2.441	1.520
Rank	5	3	1	2	4

Figure 4 shows the main effects plots for mean values which is the output result of table 4. Taguchi suggested a robust method having a minimum sensitive to all noise factors. In this, loss function is used to find out the deviation between the experimental value and desired value. Moreover, this loss function can be converted from signal to noise ratio. Generally, the loss functions are categorized into lower-the-better, higher-the-better and nominal-the-better [10,11].

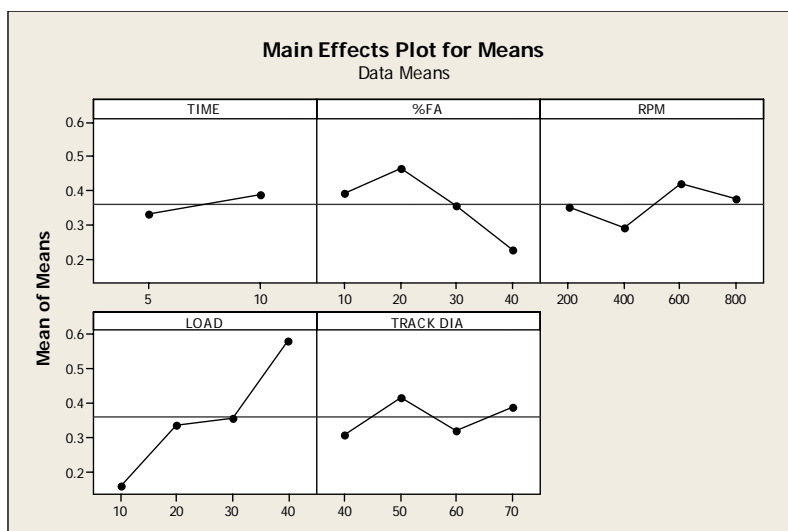


Figure 4: S/N ratio plot for relative closeness coefficient.

The above main effect plots ultimately show the optimum parametric condition to get minimum wear, frictional force and coefficient of friction.

In order to find out the contribution of each parameters like time, % of reinforcement, speed, load and track diameter on wear rate ANOVA has been carried out and results have been shown in table 5. The procedure is to a great degree accommodating to reveal the level of essentialness of effect of factor(s) on a specific reaction. It includes the total variability of the reaction (total of squared deviations about the mean) into commitments rendered by each of the parameter and the blunder [12]. The analysis has been carried out with at 95% level of confidence. The last column in table below show the significance of each parameter having very lower value. At the same time it also shows which parameter has no direct impact on the outputs.

Table 5: ANOVA table with adjusted sum of square for tests (micro oven treatment)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Time	1	0.039258	0.039258	0.039258	214.81	0.000
Fly ash	3	0.044797	0.044797	0.014932	81.71	0.000
RPM	3	0.023814	0.023814	0.007938	43.44	0.000
Load	3	0.031945	0.031945	0.010648	58.27	0.000
Track diameter	3	0.004484	0.004484	0.001495	8.18	0.001
Error	18	0.003290	0.003290	0.000183		
Total	31	0.147587				

From Table 5 only track diameter has the least significance while other parameters have influence on the output.

3.2 Confirmatory Experiment for Minimum Wear Rate

The confirmatory experiment helps in determining and analyzing the actual experimental value with the predicted value. In the wake of assessing the optimal parameter settings, the following step is to anticipate and check the enhancement of quality characteristics utilizing the optimal parametric combination. The estimated relative closeness coefficient $\hat{\gamma}$ using the optimal level of the design parameters can be calculated as:

$$\hat{\gamma} = \gamma_m + \sum_{i=1}^n (\bar{\gamma} - \gamma_m) \quad (9)$$

Where, γ_m = total is mean grey relational grade

$\bar{\gamma}$ = mean grey relational grade at the optimal level

n = number of the main design parameters

Table 6 compares the value between predicted value of input parameters and actual value using optimal parametric condition. Error is calculated by the difference in actual value and the predicted value. In all the three conditions, a minimum percentage of error is obtained which confirms that the results are least varied.

Table 6: Confirmatory test results

	Initial parameter setting	Optimal parameter condition	
		Predicted value	Experimented value
Level of factors	A2B3C1E3D2	A1B4C1D3E2	A1B4C1D3E2
Time	5		5
% FA	20		40
Speed	200		200
Load	10		30
Track diameter	50		50
Relative coefficient value	0.870022		
S/N ratio of relative coefficient value		1.30663	1.24129
Improvement in relative coefficient value = 0.95%			

The main difference in atmospheric curing and micro oven curing is the treatment condition. In micro oven curing, the temperature generated inside the micro oven is transferred to the material. The heat moves through the material uniformly. Due to this inward movement, there may be modification in the internal structure of the material. As the surface is modified its hardness value increases as compared to normal atmospheric treated sample.

3.3 Morphology of wear surfaces

Morphology of worn out surfaces reveals the actual cause of wear during the process of sliding. Figure 5 show the various wear tracks and plastic flow of material. In the dry sliding wear test, frictional force plays an important role for the wear of the material. This frictional force is responsible for the loss of material and generation of heat at the contact surface. In case of polymeric material the heat generated helps in plastic flow of material which lairizes the material removed at the initial rotations of the disc. These regions will be called the primary energy dissipation zones. It is noted that the wear increases with increase in normal applied load and sliding distance [13,14]. Rate of wear is maximum in case of abrasion wear and the mechanism of wear is completely localised and the plastic deformation plays an important role in it [15]. Each experiment is carried out thrice and mean values have been taken into consideration for the calculation of relative closeness coefficient. White marks in the figure5 indicate the plastic flow of the material. The plastic flow of material is due to the unidirectional rotation of the sample and also may be due to heat generated at the sliding surface. Although there is no micro-crack was found around any void, continuity of the specimen was deteriorated so as to decrease the wear resistance [16].

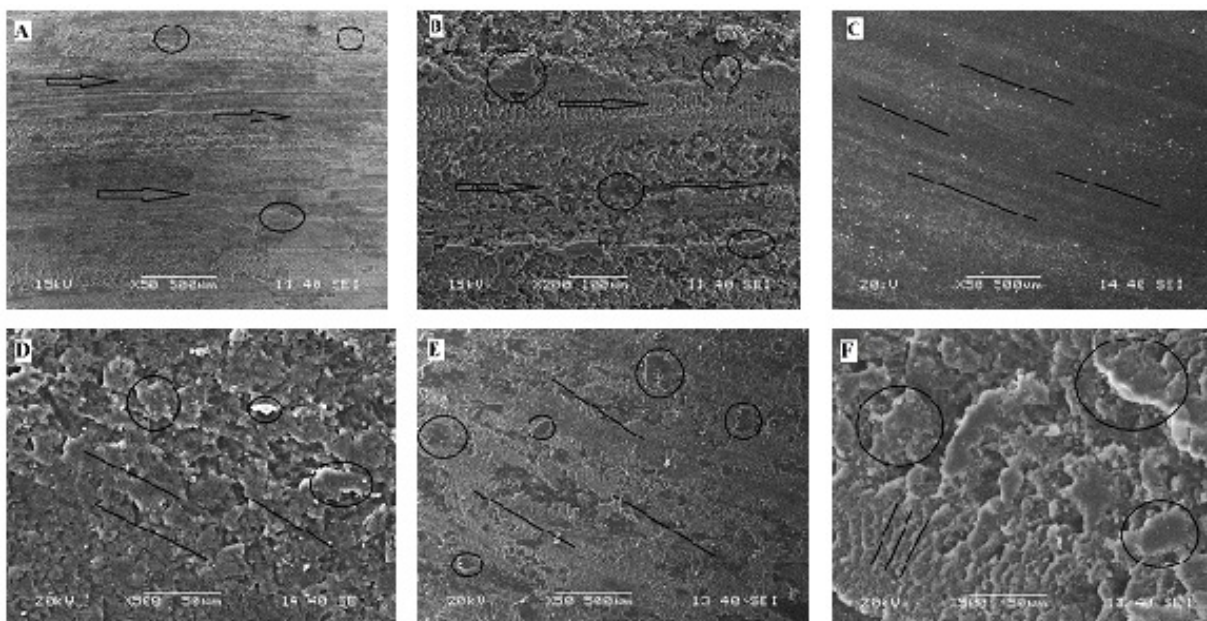


Figure 5: SEM micrographs of wear surfaces.

The straight lines in the above figures show the direction of sliding. The circular marks indicated the protruded surfaces where only matrix epoxy material is accumulated due to overheating. Figure 5 C seems to be smoothest of all as it is micro oven treated. In figure 5 B and D crack propagation and erosion of reinforcement material is clearly visible. Track broadening has taken place due to the secondary erosion that is due to the entrapment of eroded fly ash particle at the initial stages of wear. Figure 5 F which is at 500X magnification clearly shows that matrix material is highly responsible for the erosion of the material. River pattern which is clearly visible in figure 5 F is due to the overheating and overlapping of material. Regardless of material, temperature and load, more or less tribo-layers existed on the worn surface [17].

3.4 DSC Analysis

Figure 6a and figure 6 b show the T_g curve of the polymeric material before and after micro oven curing. The graphs have been plotted between temperature and amount of heat absorbed. The protruded portion represents the enthalpy of that material. From the figure it is clear that, the enthalpy of the material decreases with curing. It indicates that, the material property has been modified and strength has been enhanced. The hardness of a polymer increases with crystallinity so as the mechanical strength.

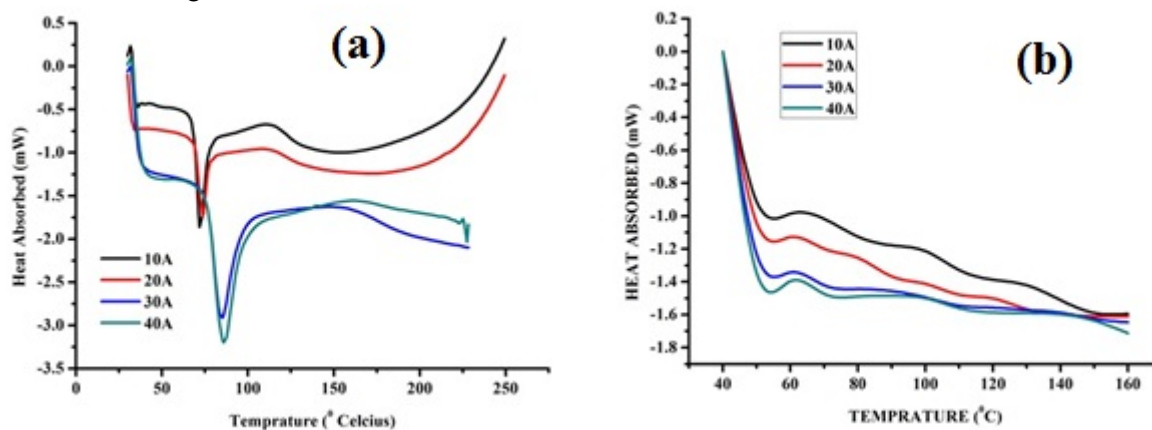


Figure 6: Comparison of DSC curves (a) for untreated and (b) micro oven treated samples.

3.5 FTIR Analysis

Figure 7 show the comparison of graphs plotted between wave number vs. % transmission for atmospheric treated and micro oven cured samples.

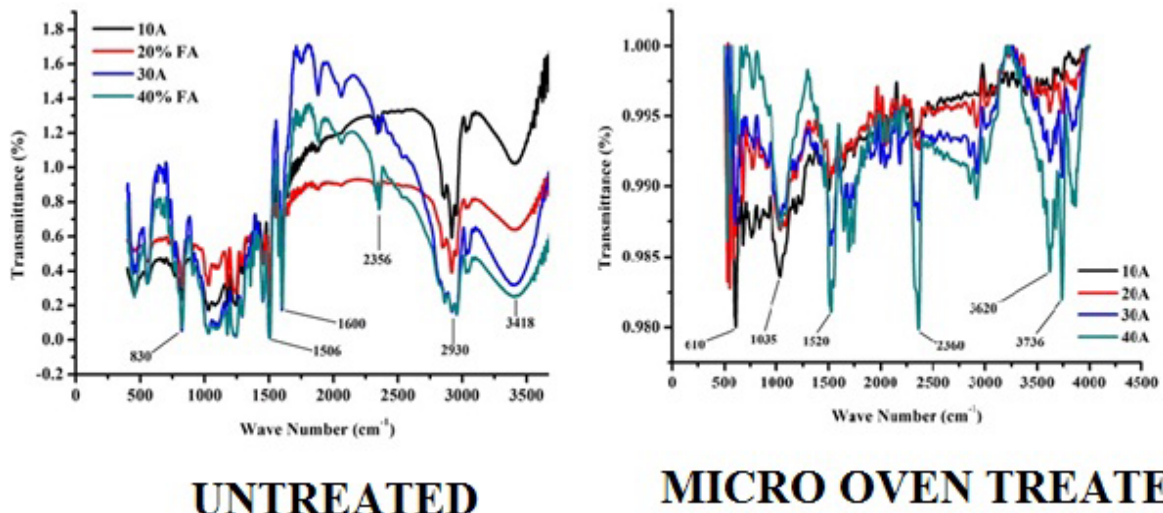


Figure 7: Comparison of FTIR spectroscopy for untreated and micro oven treated samples

From the above comparison, it is clear that more number of sharp peaks are available in micro oven cured samples. It may be due to the strengthening of bonds after curing. There is change in wave numbers also. Table show the most possible bonding that may be available before and after curing.

Table 7: Wave numbers showing possible bonding in curing samples

SI No	Wave number	Probable chemical bonding	Existence in 10% reinforced FA composite	Existence in 40% reinforced FA composite
i.	610	C-Cl / C-Br	Absent	Present
ii.	830	C-Cl / C-Br	Present	Absent
iii.	1035	=C-O-C=	Absent	Present
iv.	1506/1520	C-N=O	Present	Present
v.	1600	-NH ₂	Present	Absent
vi.	2356/2360	=NH ₂ ⁺	Present	Present
vii.	2930	-CH ₃	Present	Absent
viii.	3418/3736	=CONH ₂	Present	Absent

Due to these variations of bonding, there is structural strengthening of the material. This may be the possible reason of increase in wear resistivity of the material.

4. Conclusion

In wear, some part of work is transmitted to heat energy and some part is dissipated as frictional loss. As it is a composite material, both matrix and the reinforcement material share the loss due to the wearing of the material. As the fly ash is ceramic filler, it is easier to erode out the matrix material. Below is the list of findings after the experiment is carried out-

- Fly ash is a hazardous industrial waste which can be used as a polymeric composite which is novel method of utilizing the waste material.
- TOPSIS method can be utilized for converting the multi objective parameters into a single objective parameter and Taguchi optimization technique can be used to find out the best possible with less number of experiments.
- ANOVA helps in determining the influencing parameter. The sequence of influencing parameters is different for different treatment condition. It is in order of RPM, load, %fly ash, track diameter and time for the micro oven treatment condition.
- From the above conclusion, time is found to be the most negligible parameter which affects the wear. It may be due to the initial over heating which cause the plastic deformation and layer formation of the material.
- Taguchi optimization technique is helpful as there is minimum percentage of error.

- f. Micro oven treatment condition give wear resistant property as the treatment is related to structural modification.
- g. FTIR analysis suggests the change in bonding of the material, so this may be the possible reason of change in strength and wear resistivity.

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