STUDIES ON TRIBOLOGICAL BEHAVIOUR AND THERMAL PROPERTIES OF MARBLE POWDER-EPOXY COMPOSITES

A thesis submitted in partial fulfilment of the requirement for the degree of

Bachelor of Technology

in

Metallurgical and Materials Engineering

By

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&

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DEPARTMENT OF METALLURGICAL & MATERIALS ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

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DEPARTMENT OF METALLURGICAL & MATERIALS ENGINEERING

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CERTIFICATE

This is to certify that the work in this project report entitled "Studies on tribological behaviour and thermal properties of marble powder-epoxy composites" by SWOPNIL MOHAPATRA and DEBESH KANUNGO has been carried out under my supervision in partial fulfilment of the requirements for the degree of Bachelor of Technology in Metallurgical and Materials engineering, National Institute of Technology, Rourkela and is an authentic work carried out by them under my guidance.

To the best of my knowledge, this work has not been submitted to any other university/institute for the award of any degree or diploma.

Place:

Date:

Prof. Subash Chandra Mishra

Department of Metallurgical and Materials engineering

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Finally, we would like to extend our sincere thanks to all the staff members of the department and our fellow classmates for their help and support.

Place:

Date: **Swopnil Mohapatra (111MM0370)**

Debesh Kanungo (111MM0593)

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ABSTRACT

In this study, the usability of waste marble dust as a reinforcement material in epoxy resin, to fabricate a novel particulate composite has been evaluated. For this purpose, waste marble dust is mixed in different weight percentages (10%, 20%, 30% & 40%) with a polymer matrix (epoxy resin). Physical and chemical compositional analyses of marble powder and epoxy have been done prior to mixing. Once the samples of standard test dimensions were fabricated, their wear properties were examined using Pin-on-Disc wear testing machine. Scanning Electron Microscopy (SEM) was used to study the worn surface of the composites. Differential Scanning Calorimetry (DSC) was used for the thermal analysis of the composites, to study the effect of the reinforcement content on its glass transition temperature. The molecular structure and bonding of the particulate composite has also been evaluated using FTIR characterization. The results indicate that the wear resistance decreases with increasing weight% of marble powder. It is also concluded that the untreated samples show better wear resistance as compared to the treated samples. The thermal properties of the composite showed very little changes with increasing marble powder content.

Keywords: marble powder, epoxy, particle reinforced composite, wear, curing, glass transition temperature, SEM, DSC, FTIR

Chapter 1

INTRODUCTION

1.1 Background

Marble is a metamorphic rock produced from limestone by pressure and heat in the earth's crust due to geological processes. It is widely used cement, construction and sculpture industries. Marbles are, chemically, crystalline rocks constituting mainly calcite and dolomite. In the last 2 decades, the marble industry has gained industrial importance. Countries like USA, France, Belgium and other European countries have considerable marble reserves. During marble processing, 30% of the stone goes to scrap because of being smaller in size and/or being irregularly shaped. Millions of tons of marble powder are processed every year during extraction, around the world. As a result, significant environmental damage occurs from the large quantities of marble dust produced in these industries. Marble dust may also lead to skin allergies. However, apart from these disadvantages, marble powder is also among the most useful substances in the planet and has found uses in several applications, in a number of different forms. It is mainly used as a filler or additive in several industrial applications.

Many researches previously investigated marble waste as a potential constructional material. However, it can now also be used to make a new class of polymer-MWP composites. Epoxy resins (ER) are very useful substances as polymeric matrix system for developing such new composite materials. With growing engineering applications, epoxy based composites have been a subject of extensive research in the last few years.

1.2. Composite Materials and their Classification

Composites make up a very broad and important class of engineering materials. Over10 million tons of composite is fabricated every year worldwide and its market has been growing at 5-10% per annum. Composites are used in a wide variety of applications. With the current technologies, there is considerable scope for tailoring their structure to suit the required service conditions.

In general, composite are materials made up of two main constituents:

1. Reinforcements
2. Matrix

The matrix holds the reinforcements in their proper positions, provide toughness to the material and ensure proper load transfer to the reinforcements, which are the major load bearing constituents of any composite. The reinforcements provide adequate stiffness to the composite material.

In general, composites are material made of two or more constituents which are insoluble in each other and retain their respective physical properties in the composites too. The constituents are separated by a well-defined interface, across which the variation of properties occur.

On the basis of the reinforcements in them, composites can be grouped into:

1. Fibre Reinforced Composites
2. Whisker Reinforced Composites
3. Particulate Reinforced Composites

On the basis of the matrix material in them, composites can be grouped into:

1. Metal Matrix Composites
2. Polymer Matrix Composites
3. Ceramic Matrix Composites

1.2.1. Polymer Matrix Composites

In recent years, polymer matrix composites (PMCs) have found wide applications in a number of industries, starting from sports and automobiles to aeronautics and robotics. The main advantages of PMCs over other types of composites are as follows:

1. They are cheap.
2. They can be easily processed and engineered to get desired properties.
3. They have more chemical resistance than metals.
4. They have low density.
5. Many polymers show good ductility.
6. Polymers have strong covalent bonds and are generally poor conductors of heat and electricity.

On the other hand, polymers have low strength and modulus and lower operation temperature limits. Prolonged exposure to UV light and certain solvents can cause the degradation of polymer properties. There are two types of polymers, which are used as matrix material:

- *Thermosetting*: This includes resins like epoxy, polyester and vinyl ester. These cover a broad class of chemicals and a wide range of physical and mechanical properties can be determined. In case of thermosetting polymers, the liquid resin changes to a hard, rigid solid by chemical cross linking, which in turn forms a compact three dimensional network. This occurs when the composite is being formed. The mechanical properties depend on the molecular units making up the network and on the length and density of the cross-links.
- *Thermoplastics*: Unlike thermosetting resins, thermoplastics do not show cross linking. Their strength and stiffness comes from the inherent properties of their monomer units and their high molecular weights. During heating of amorphous thermoplastics, disentanglement occurs and there is a change from a rigid solid to a highly dense and viscous liquid. In case of crystalline polymers, heating results in the melting of the crystalline phase to give an amorphous viscous liquid.

1.2.2. Particulate Reinforced Composites

Metal and ceramic composites are usually fabricated by using their powder/particles as reinforcements. In particulate reinforced composites, particles of the reinforcement phase are embedded in the matrix phase. These reinforcement particles are known to be of different shapes, but their dimensions are, more or less, equal.

Particle reinforced composites are cheap and can be categorized based on the particle size as:

- Large-particle composites, which restrict the matrix movement and strengthen the composite.
- Dispersion-strengthened composites, which particles in the size range of 10-100nm. In these composites, the matrix is usually the major load bearing component.

The advantages of particle reinforced composites over fiber reinforced composites are:

i. These 3D reinforcements provide isotropy in properties
ii. The strength of the composite depends on the particle diameter, its distribution and volume fraction, which can be altered to get the desired mechanical properties.

However, the use of nanoscale particles may lead to agglomeration in the composite and proper precautions must be taken to avoid it.

Chapter 2

LITERATURE REVIEW

Bilgin et.al. [2-5], investigated the usability of marble powder as an additive for industrial bricks. They concluded that, the presence of marble powder had a positive effect on the mechanical, physical and chemical properties of the bricks. They have suggested that the use of marble dust can contribute to the economy and also minimize environmental pollution.

Borsellino et.al. [1] studied the behaviour of composite structures reinforced with marble powder. They researched on the variation in mechanical and physical properties of the composite with different matrix materials (epoxy and polyester resins) and different weight fractions of reinforced particles. Their work shows that a composite with 60% marble powder with epoxy resin gave much superior properties compared to monolithic marble.

Arunit et.al. [6-7] examined the effect of post curing temperature on the properties of a polymer matrix particle reinforced composite. Their aim was to draw a relationship between the post curing mode and the composite application. Their work suggests that the composite material should be cured at 60-80^{0}C. They also concluded that with increasing curing temperature, the glass transition temperature increases but the material becomes more brittle.

Imran Oral [8] studied the effects of marble powder, type of coagulant and their dosages on the ultrasonic properties of the epoxy resin/marble powder composites. His work also showed that the morphological and ultrasonic properties of the epoxy resin improved by addition of marble powder. He concluded that the ultrasonic non-destructive method is very useful for evaluating the elastic properties of epoxy/marble powder composites.

Marras et.al. [10-12] studied the effective recovery and reuse of by-products of marble processing industries. Their focus was to integrate these by-products with industrial applications. They concluded that the addition of marble dust and other traditional constituents improved the quality of industrial bricks. Their work shows that marble powder can partially replace the clay in the bricks, leading to less waste generation.

Bahar Demirel [9] has investigated the effect of using waste marble powder as a fine additive on the mechanical properties of concrete. He prepared different concrete-marble powder-sand mixture for mechanical testing. His work suggests that the addition of waste marble powder resulted in the enhancement of the compressive strength of the concrete and also a decrease in the porosity of the concrete.

Hasan et.al. studied [13-14] the abrasive wear behaviour of Al-SiC composite using Pin on disc machine. They obtained the wear rate in terms of the weight loss and also the volumetric wear rate. The characteristics of the wear surface was investigated using SEM. Their studies conclude that the material wear increased with increasing load and that the wear of the Al-SiC composite significantly lower than that of the base materials.

Sudheer et.al. [15-17] have studied the wear behaviour of epoxy-metal sulphide composites. The wear tests for different compositions of the composite were carried out on a Pin on disc wear tester with changing sliding distance, but constant speed (1m/s). Their studies reveal that there is a significant decrease in the wear rate of the composite after the incorporation of the metal sulphide fillers. The wear mechanisms were investigated by SEM of the wear surface.

Singla et.al. [18-19] have investigated the effect of load and sliding speed on the wear rate of Al7075-fly ash composites. Their work concludes that with increasing load and sliding speed, the wear rate and the coefficient of friction decreases. Also, the addition of fly ash to Al7075 matrix improved the material's resistance to material loss as with increasing fly ash content the coefficient of friction and hardness increases.

Zvetkov et.al. [20] have studied the DSC kinetics of microwave curing of epoxy-poly (ethylene terephthalate) blends. The curing process of the different blends were monitored using DSC in isothermal mode. The curing data was used to estimate the temperature profile during curing. The results provide data for the efficient modification of the epoxy phase.

Sanchez-Soto et.al. [21] studied the curing FTIR and the mechanical characterization of glass beads-epoxy composites. Their work followed the curing reactions and the conversions that occurred in the functional groups present, during curing. They concluded that the introduction of rigid glass beads had a significant influence on the curing process of the composite. The mechanical properties, namely the Young's modulus and the resin strength, increased with increasing content of glass beads.

Abdul Munium Razoki Majeed Algbory [22] studied the wear rate behaviour of carbon fiber/epoxy composites with varying load and sliding speed. His study shows that the wear rate increased non-linearly with increase in load and sliding speed. There is also a decrease in the wear rate with increasing volume fraction of carbon fibers.

Antaryami Mishra [23] studied the dry sliding wear behaviour of epoxy-rubber dust composites. Pin on disc wear testing was adopted to determine the wear rate for different compositions of the composite, with varying load, sliding speed and time. His studies conclude that all the composite specimens showed very low coefficients of friction and wear rates.

Sudheer et.al. [15-17] have investigated the tribological properties of epoxy/Al_2O_3 nano-composites. The wear properties were obtained by using the pin on disc wear testing machine. His work shows that the addition of Al_2O_3 particles in the epoxy matrix provided an anti-wear mechanism and prevented the epoxy matrix from a large wear.

Volkan et.al. [24] conducted the FTIR and thermal analysis of polyester and epoxy based composites, with glass and carbon fibers as reinforcements. FTIR analysis was conducted to determine the bonding characteristics between the matrix and reinforcement. SEM was used to evaluate the strength of this interaction. Their work concludes that a strong interaction was observed between the epoxy matrix and carbon fiber reinforcements.

Majhi et.al. [25] have studied the tribological behaviour of rice husk reinforced epoxy composites. Pin on disc wear test was adopted to evaluate the wear properties of modified and unmodified rice husk-epoxy composites. Their studies conclude that the modified rice husk composites gave much better tribological properties compared to unmodified rice husk composites.

Chapter 3

EXPERIMENTAL PROCEDURE

3.1. Composite Fabrication

3.1.1. Materials Used:

The following materials were used in the preparation of epoxy- marble powder composite:

1. ***Epoxy (L12) (Bis-phenol A)***: A transparent dense fluid with high viscosity served as the matrix in the composite. It is a thermosetting resin with high molecular weight.
2. ***Hardener (K6)*****:** Epoxy curing agent K-6 is a low viscosity room temperature curing liquid hardener. Being rather reactive it gives a short pot life and rapid cure at normal ambient temperatures. It has a shelf life of least two years if stored in its original container away from humidity and heat. The mixing ratio of mixing of epoxy and hardener is fixed as 10:1 by the supplier.
3. **Marble powder:** A white fine powder with density more than that of the epoxy matrix and served as the particulate reinforcement. The compositional and physical analysis of marble powder is given further in tables 1 & 2 respectively.

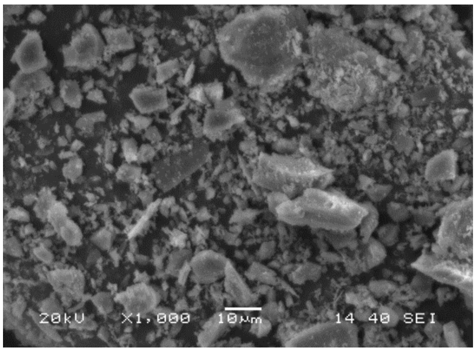
Table 1: Compositional Analysis of Marble powder

Sl. No.	Compound present	Percentage
1	SiO_2	2.76
2	Fe_2O_3	0.4
3	Al_2O_3	0.98
4	CaO	34.98
5	MgO	18.96
6	LOI	41.92

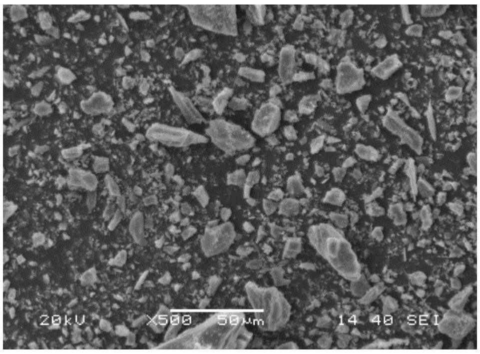
Table 2: Physical Analysis of Marble powder

Sl No.	Properties	Value
1	Specific gravity	2.67
2	Water content	49.4%
3	Liquid limit	18.05%
4	Shrinkage limit	23%

3.1.2. Scanning Electron Microscopy:



(a)



(b)

Fig 1. (a) SEM image of Marble Powder at 1000X. (b) SEM image of marble powder at 500X

The above SEM images (Fig. 2) of the marble powder show that the particles are non-uniform in sizes and shapes and it proves to be helpful during the mixing process. Due to their non-uniform shapes and sizes, during the compaction process the voids formed due to the agglomeration of larger particles, is filled up by particles of smaller sizes and thus, it results in an effective compaction. This differences in sizes also gives the compact a dense structure.

3.1.3. Particle Size Analysis

For better physical characterization of the marble powder, its grain size distribution was determined using particle size analysis. From this analysis, it has been found that the marble powder consists of medium-sized particles, with an average particle size of 20.54 µm.

3.1.4. Sample Preparation :

a) **Mixing:** The experiment has been planned for four different compositions of marble powder reinforcements in epoxy matrix i.e. (10%, 20%, 30%, and 40%). The calculated weights of marble powder are added to the epoxy as per the composition. The mixture is first stirred manually. After attaining a nearly white semi fluid like material, the mixture is transferred to another beaker and is then stirred using an ultrasonic mixer (sonicator) for 10 minutes with a pulse time of 5 seconds. We get a thin white liquid to which hardener K-6 (10 wt. % of the amount of epoxy) is mixed and stirred well manually for about 20 minutes.

Table 3: The amount of marble powder and epoxy used for a 50g batch of each composition

Sl No.	Composition (% of MP)	Wt. of Marble powder (g)	Wt. of Epoxy (g)	Wt. of Hardener K-6 (g)
1.	10	5	45	4.5
2.	20	10	40	4.0
3.	30	15	35	3.5
4.	40	20	30	3.0

b) **Casting:** From the mixed compositions, cylindrical compacts are made. A cylindrical die is made from the PVC wiring pipes of 1" length and 12mm diameter. The moulds are prepared by applying grease on the inner walls of the die and subsequently using mould release spray for easy removal of the casting after solidification. After preparing the mould, the mixture is poured into the pipes and is left to solidify for 6-8 hours. (Note: If the mixture is poured into the mould when it is still hot, it will lead to foaming of the sample and rapid solidification takes place, hence proper care on the temperature of the mixture must be taken.)

c) **Sample removal from the mould & machining:** The samples prepared are removed from the mould after solidification by cutting the pipes. The produced samples are machined by turning and facing to get the desired diameter and a flat base. The samples are turned to 10mm diameter for abrasive wear test. The specimens are then stored in airtight containers. Powder samples and small broken chips were also collected for each composition during machining, to be used during their characterization by FTIR and DSC respectively.

d) **Post-curing of samples:** 4-5 samples of each composition were post-cured in an oven for 30 minutes at 120^0C. This was done to investigate the variation in the thermal and wear properties of the composite with curing temperature.

3.2. Wear Test

***Pin on disc sliding wear test apparatus*:** Test set-up used in the study of wear test is capable of creating reproducible sliding wear situation accessing the sliding wear resistance of the prepared samples. It consists of a pin on disc, loading panel and a controller.

Fig. 2. DUCOM Wear and Friction Monitor used for the wear tests

The entire test was carried out using a "DUCOM friction and wear monitor" machine at normal atmospheric conditions. The wear test was carried out for the treated and untreated samples of each composition. The wear rate was observed from the wear distance vs time plot obtained. The variation in the coefficients of friction with curing temperature and increasing marble powder content was also investigated. Care was taken to clean up the sample surface before and after each test to prevent any form of damage to it. All the tests were carried out with a track radius of 60mm.

Table 4: Specifications of the DUCOM wear and friction monitor

Parameter	Unit	Minimum	Maximum
Wear disc	mm	100x6	---
Disc speed	RPM	10	800
Pin diameter	mm	2	12
Pin length	mm	10	50
Ball diameter	mm	10	---
Wear track diameter	mm	10	80
Normal load	N	0	100
Frictional force	N	0	100

The variables involved in wear test are:

- % marble powder in epoxy-marble powder composite
- Normal Load
- Disc Speed
- Track Radius

From the raw data obtained by conducting the test, the wear height δh can be obtained and it is plotted against time considering the best fit curve for each specimen.

Initial sample diameter is measured and is used to calculate the cross-sectional area A_0.

$$\text{Wear Volume} = \delta h \times A_0$$

The track diameter is set to be D_1 and let the time of run and the RPM be 't' and 'R_0', respectively. Hence,

$$\text{Sliding Distance} = R_0 \times t \times \pi \times D_1$$

The best fit curve is extrapolated from the above calculated data to get the wear volume and sliding distance. Later, the corresponding graphs are plotted using OriginPro.

In this experiment, the wear test was conducted and the plots for wear height vs time, wear volume vs sliding distance and friction coefficient vs time were obtained for each compositions.

3.3. SEM Analysis

After the wear test, the sample surface was observed under a scanning electron microscope (SEM) at 50X and 500X magnifications to determine the wear behaviour in the microscopic level. The sample surface was given a thin, conducting platinum coating prior to mounting in the microscope.

3.4. DSC Analysis

Differential scanning calorimetry (DSC) is a thermal analysis technique in which differences in heat flow into a substance and a reference are measured as a function of sample temperature, while the two are subjected to a controlled temperature program. DSC was operated under nitrogen atmosphere. For this test, small chips of 10-15 mg, cut off the samples, were placed into the aluminium crucibles. The dynamic measurements were made at a constant heat rate of 10°C/minute from 30 to 200°C to determine the effects of the increasing marble powder content on glass transition temperature, T_g of the composites. This was determined by the mid-point method.

3.5. FTIR Analysis

FTIR spectroscopy was used to study the epoxy curing process. An IRPrestige-21 spectrometer was used to monitor the variations in molecular structure as a function of marble powder content in the composite specimens. The sample was ground to powder, mixed with potassium bromide, and a thin film was prepared at room temperature. The potassium bromide was used as the background. The scanning spectra was recorded in the wavenumber interval between 4000 cm^{-1} and 500 cm-1.

The absorbance vs wavenumber plots were obtained from the transmittance vs wavenumber plot using the Beer's equation:

$$\%Absorbance = 2 - log_{10}(\%Transmittance)$$

Chapter 4

RESULTS & DISCUSSIONS

4.1. Wear Test

4.1.1 For Untreated Samples (Cured at room temperature):

The samples were expected to show more wear with increasing composition of marble powder in epoxy. Marble powder is the brittle phase and epoxy the ductile phase, so the presence of marble powder in the ductile matrix leads to fracturing and fragmentation in the composite, resulting in higher wear with increasing composition of marble powder. As expected, the wear rate was observed to increase with increasing marble content in the composite.

Table 5: Untreated composite sample specifications for wear test

Wt. % MP	Height (mm)	Diameter (mm)	Weight (g)	Density (g/mm^3)	Contact Area (mm^2)
10	21.6	9.5	2.183	1.406×10^{-3}	70.882
20	24.7	9.7	2.497	1.368×10^{-3}	73.898
30	18.5	9.8	2.096	1.502×10^{-3}	75.430
40	19.5	10.0	2.419	1.579×10^{-3}	78.540

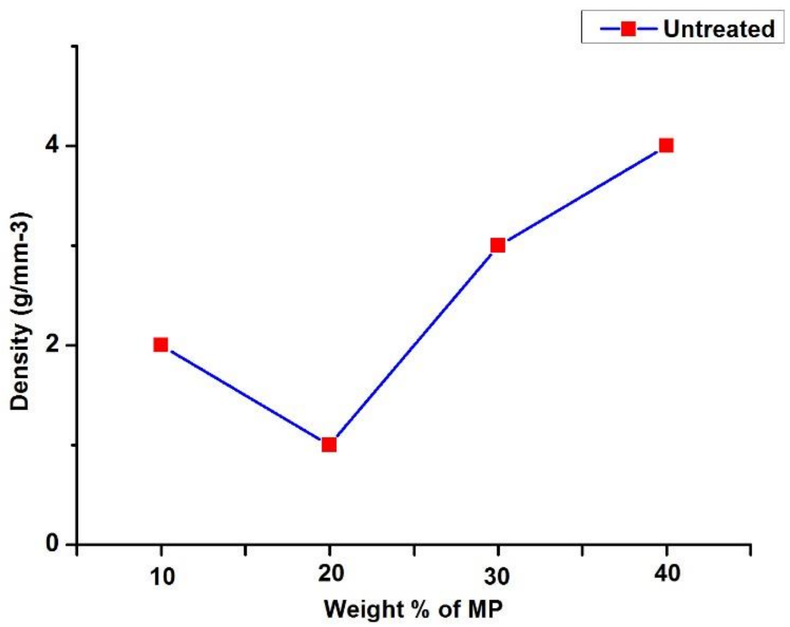
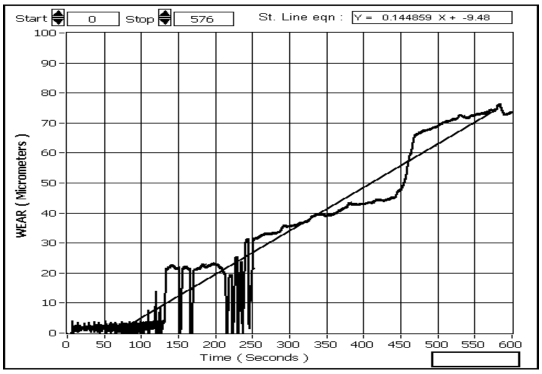
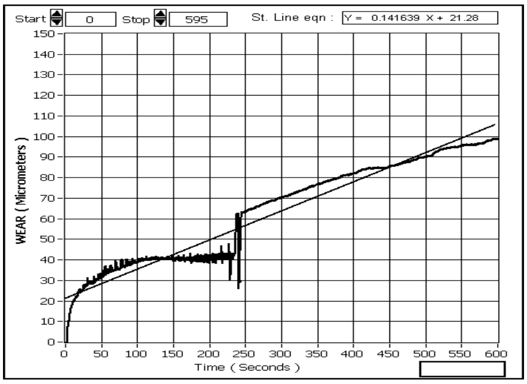
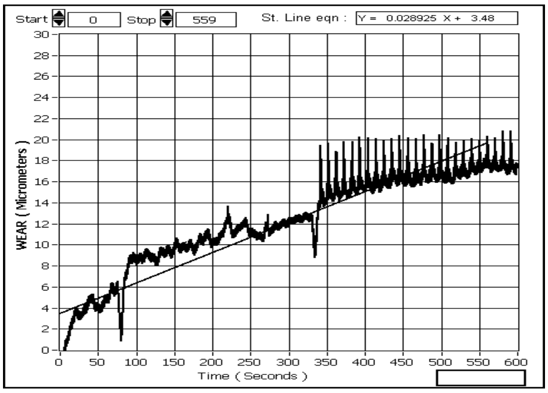


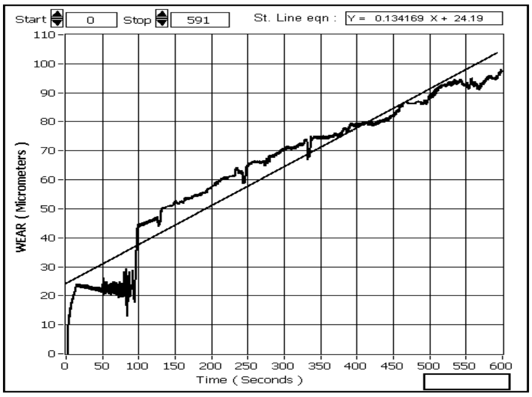
Fig.3. Variation in density of untreated composite with increasing MP content





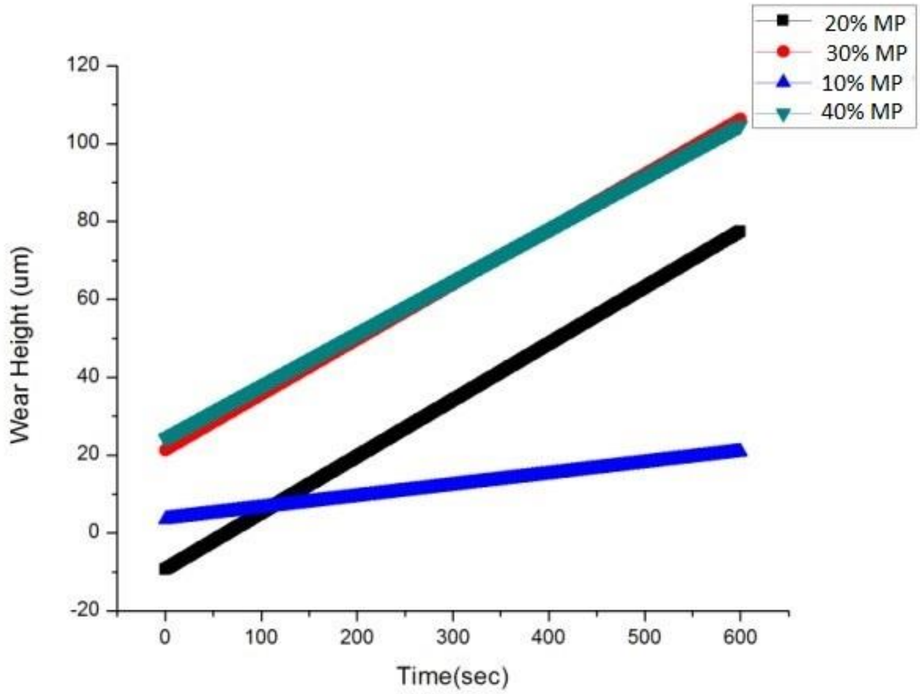
(a) **(b)**



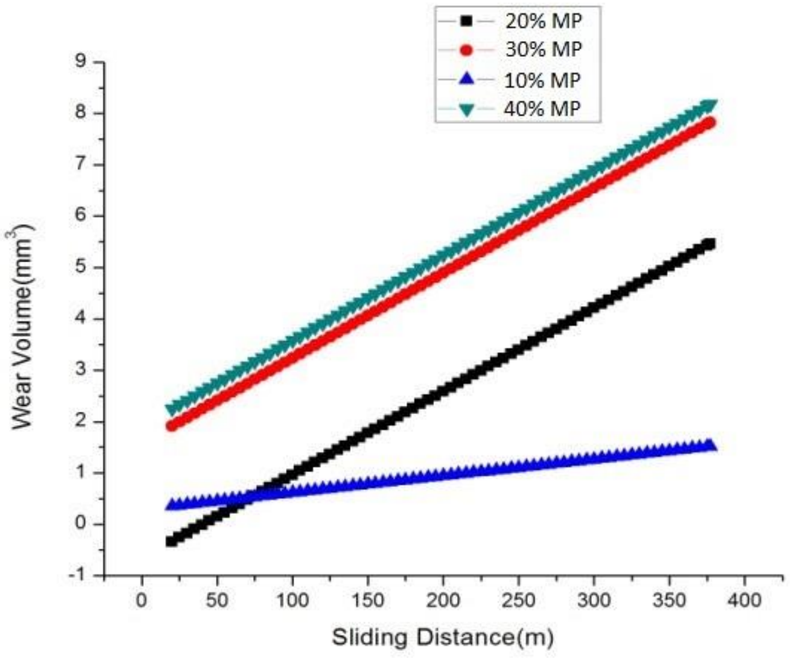


(c) **(d)**

Fig.4. Scatter Plot of wear distance vs time for untreated (a) 10% Marble Powder/Epoxy composite, (b) 20% Marble Powder/Epoxy composite, (c) 30% Marble Powder/Epoxy composite, (d) 40% Marble Powder/Epoxy composite



(a)



(b)

Fig.5. (a) Wear height vs time and (b) Wear Volume vs sliding distance for the untreated marble powder/epoxy composites

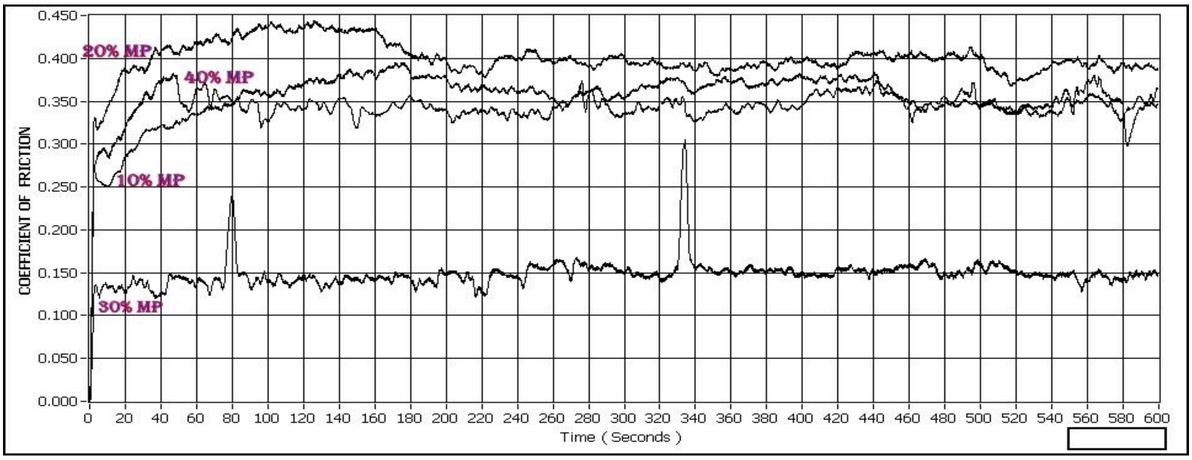


Fig.6. Comparison plot of friction coefficient v/s time for various composition of uncured Marble Powder in Epoxy composite

From the above plots it can be seen that friction coefficient follows a similar trend as that of the wear rate, but the most notable information that was collected was that the mean friction coefficient for each material was found to be less than 0.4 and hence it can be said that due to low coefficient of friction, these materials are not much affected by sliding and hence lesser is the wear compared to brittle materials like marble. The mean friction coefficient for 10%, 20%, 30%, 40% of marble powder are found to be 0.354, 0.399, 0.148, and 0.344, respectively. Due to such a low friction coefficient these materials show better tribological behaviour than pure marble.

4.1.2. For Treated Samples (Cured at 120^0C for 30mins):

The samples of required dimensions are cured at 120^0C for 30 mins in an oven and then were tested for sliding wear. In this experiment wear test was conducted and the graphs between wear height and time, wear volume with sliding distance and friction coefficient with time for various compositions were obtained.

Table 6: Treated composite sample specifications for wear test

Wt. % MP	Height (mm)	Diameter (mm)	Weight (g)	Density (g/mm^3)	Contact Area (mm^2)
10	17.1	10.2	1.820	$1.302x10^{-3}$	81.713
20	18.7	9.8	2.247	$1.593x10^{-3}$	75.429
30	17.4	10.1	1.983	$1.423x10^{-3}$	80.118
40	22	9.5	2.161	$1.386x10^{-3}$	70.882

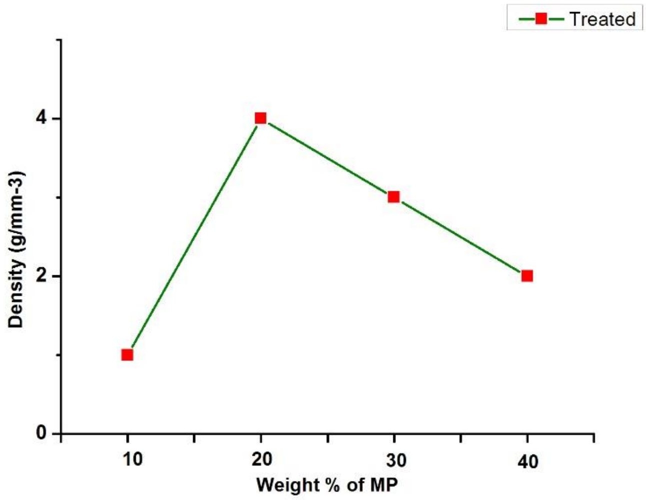


Fig.7. Variation in density of treated composite with increasing MP content

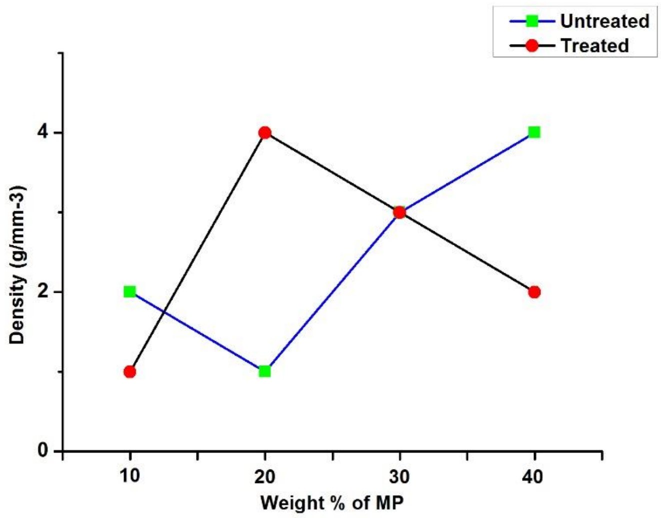
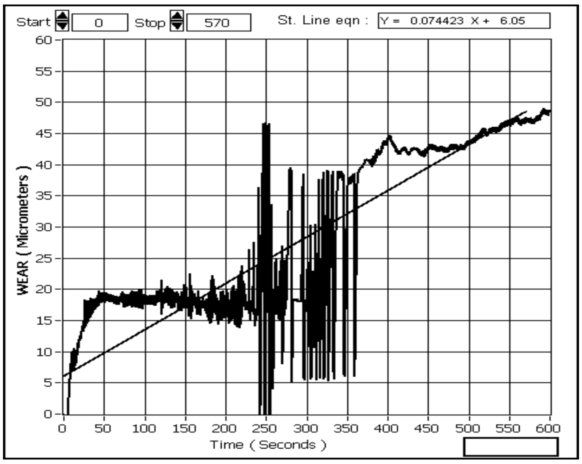
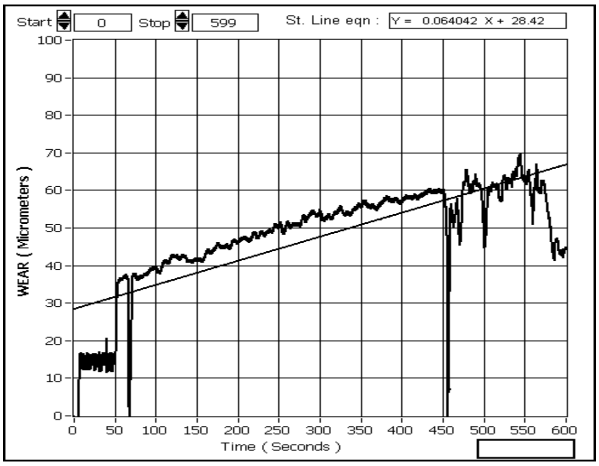


Fig.8. Comparison of density variations for treated and untreated composite samples

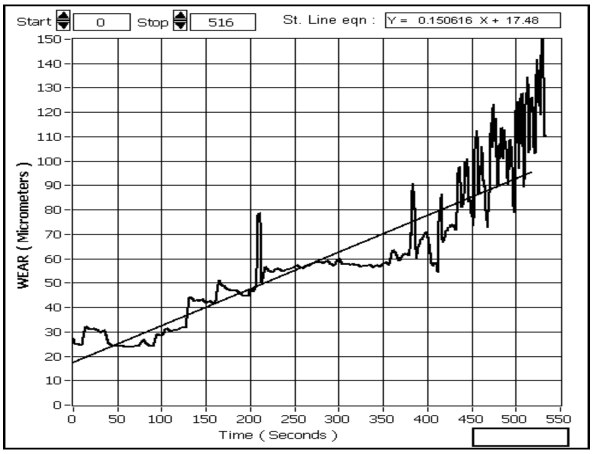
After oven treating the samples of different compositions at 120^0C for 30 mins, the wear test was conducted for samples of each of the treated compositions. It was observed that as the percentage of marble powder in the composite increased from 10-40% the wear loss volume also increased. This can be related with the fact that epoxy is ductile in nature and inclusion of brittle marble powder particles will increase the wear loss. Unlike the untreated samples, the treated samples did not show any deviation from the expected trend at any particular composition. Thus, it can be said that at high temperature the diffusivity of the particles increases and hence voids and the other inclusions diffuse to the surface and leave the sample, leaving behind a purer sample. Thus, after treatment the samples started following the expected trend.



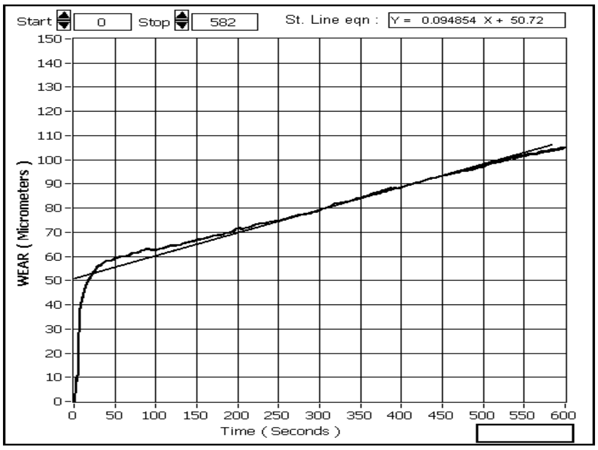
(a)



(b)



(c)



(d)

Fig.9. Scatter Plot of wear vs time for treated (a) 10% Marble Powder/Epoxy composite, (b) 20% Marble Powder/Epoxy composite, (c) 30% Marble Powder/Epoxy composite, (d) 40% Marble Powder/Epoxy composite

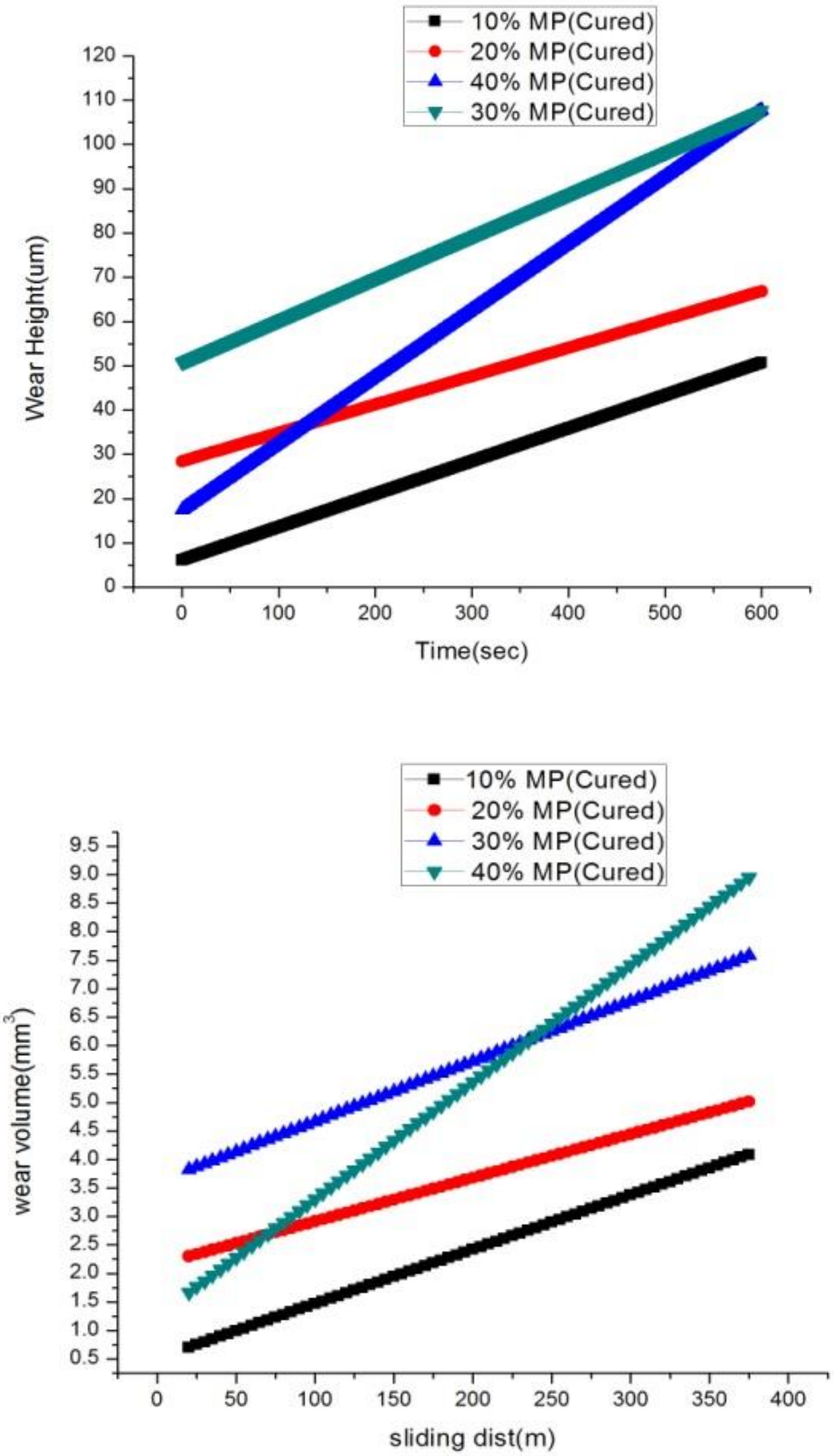


Fig. 10. Variation of (a) wear height with time, and (b) wear volume with sliding distance for treated marble powder/epoxy composites

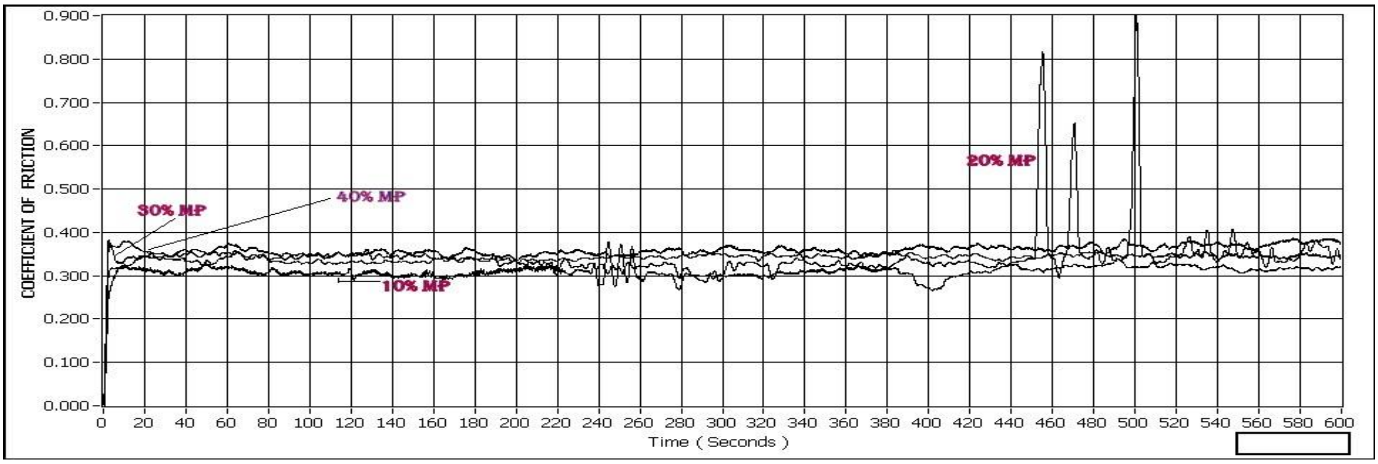


Fig. 11. Comparison plot of friction coefficient v/s time for various composition of cured Marble Powder in Epoxy composite

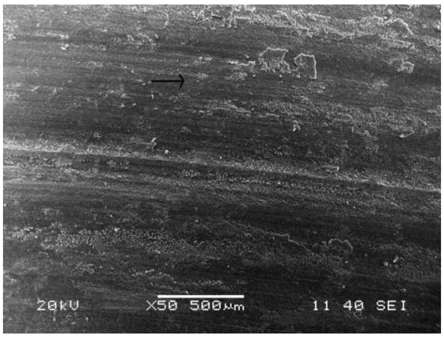
From the above plots it can be seen that friction coefficient follows similar trend as that of wear properties. But the most notable information that was collected was that the mean friction coefficient for each material is found to be less than 0.4 and hence can be found that due to low coefficient of friction these materials are not much affected by sliding and hence lesser is the wear compared to brittle materials like marble. The mean friction coefficient for 10%, 20%, 30%, 40% of marble powder are found to be 0.309, 0.342, 0.343, and 0.358 respectively. Due to such a low friction coefficient these materials show better tribological behaviour than pure marble.

4.2. Scanning Electron Microscopy:

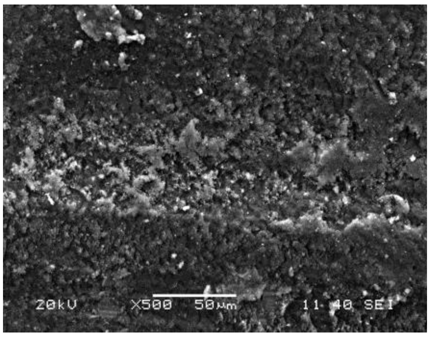
The SEM images of the worn surfaces of both the treated and untreated samples were observed at 50X and 500X magnifications.

4.2.1 For Untreated Samples (cured at room temperature):

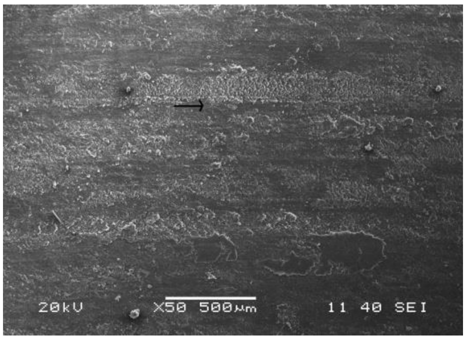
As can be seen from the SEM images in Fig. 10, as the percentage of marble powder in the composite increases more number of particles can be seen on the worn surface. The wear tracks are clearly visible and their width increased with increasing marble powder content in the composite sample. It was also observed previously from the SEM images of the marble powder used that the powder particles were of non-uniform shape and sizes, as a result of this the marble particles in the composite wears off easily and exposes the epoxy on the surface. A few cracks were also observed on the worn surface with increasing marble powder content. This can be clearly seen from the 500X magnification images of the various composites.



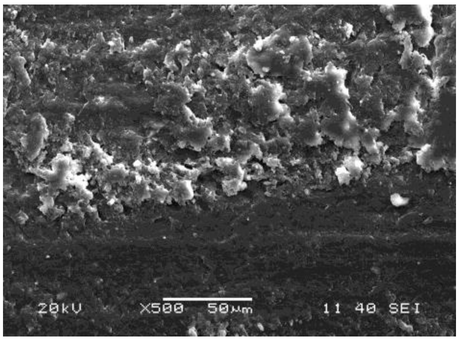
(a)



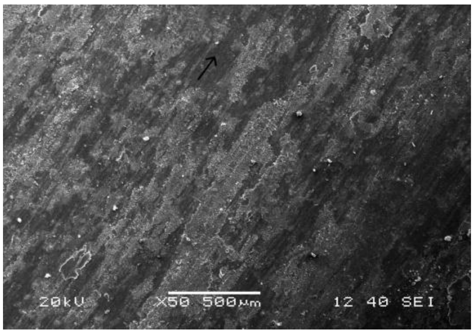
(b)



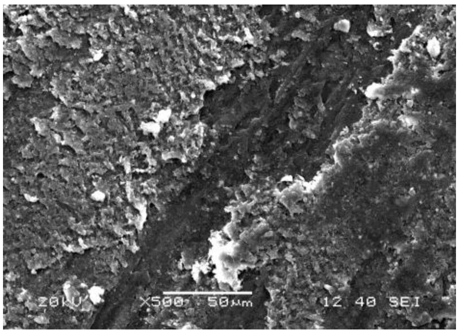
(c)



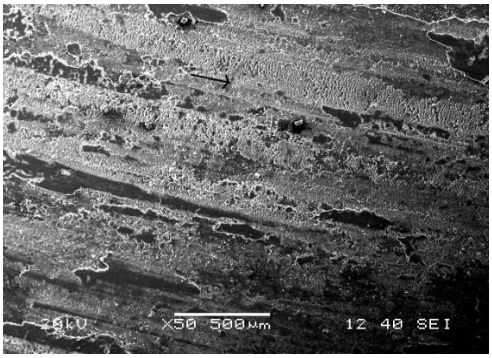
(d)



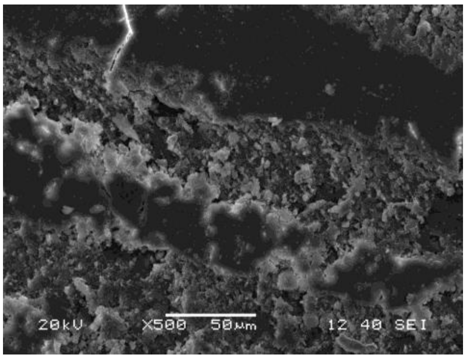
(e)



(f)



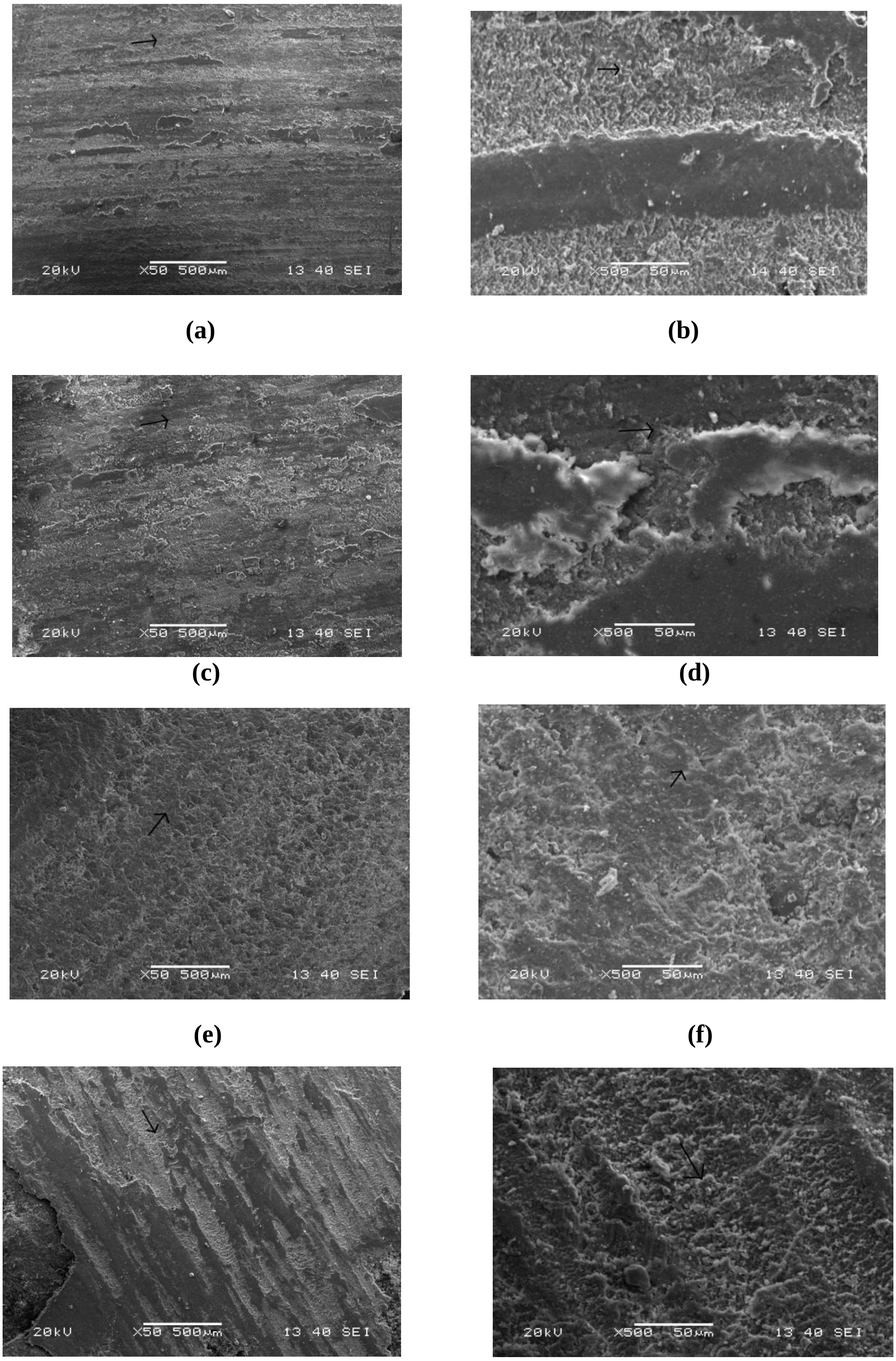
(g)



(h)

Fig. 12. Wear Surface of 10% Untreated Marble Powder Epoxy Composite at (a) 50X magnification and (b) 500X magnification, Wear Surface of 20% Untreated Marble Powder Epoxy Composite at (c) 50X magnification, and (d) at 500X magnification, Wear Surface of 30% Untreated Marble Powder Epoxy Composite at (e) 50X magnification, and (f) 500X magnification, Wear Surface of 40% Untreated Marble Powder Epoxy Composite at (g) 50X magnification, and (h) 500X magnification.

4.2.2. For Treated Samples (post cured at 120^0C for 30 mins):



(a) (b)

(c) (d)

(e) (f)

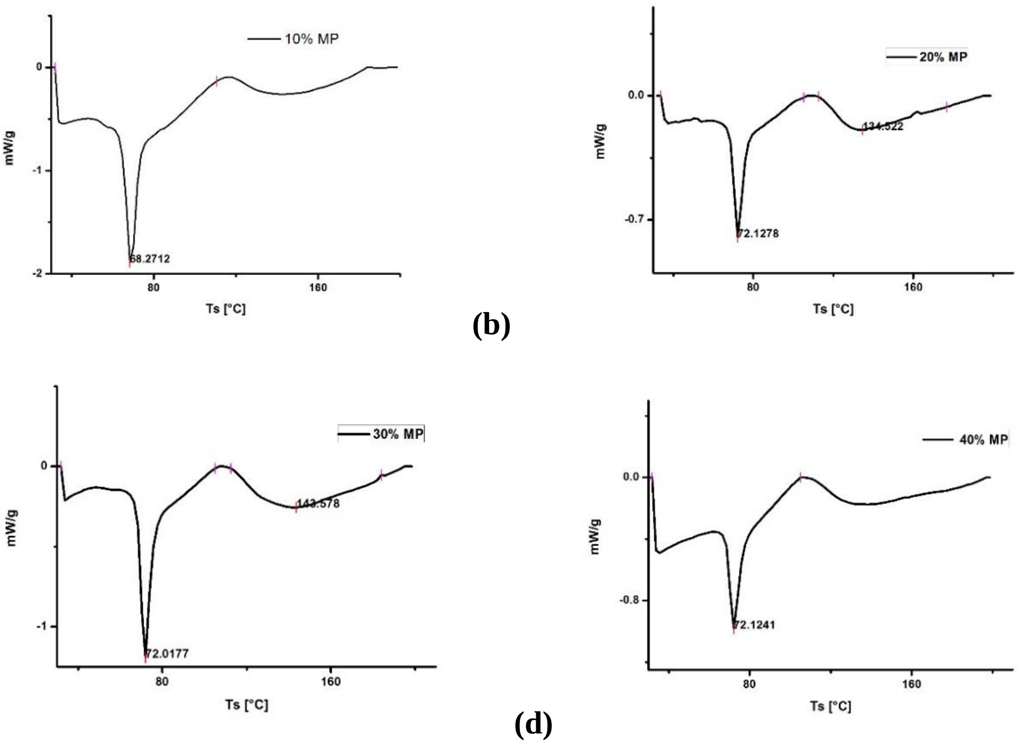
(g) (h)

Fig. 13. Wear Surface of 10% treated Marble Powder Epoxy Composite at (a) 50X magnification and (b) 500X magnification, Wear Surface of 20% treated Marble Powder Epoxy Composite at (c) 50X magnification, and (d) at 500X magnification, Wear Surface of 30% treated Marble Powder Epoxy Composite at (e) 50X magnification, and (f) 500X magnification, Wear Surface of 40% treated Marble Powder Epoxy Composite at (g) 50X magnification, and (h) 500X magnification.

Similar to the untreated composite specimens, the wear track width also increased as the percentage of marble powder in the treated composite increased. Thus more marble powder particles de-bonded from the epoxy and were observed on the worn surface. This can be clearly observed from the 500X magnification images of the various composites. It was observed that after post curing the sample at 120^{o}C for 30 minutes, the worn surfaces showed wider wear tracks compared to that in untreated samples. Thus, the wear rate was observed to be more in case of the post cured specimens.

4.3. DSC Analysis

The DSC plots shown in the above figure suggest that there is negligible change in the glass transition temperatures of the marble powder-epoxy composites with increasing marble powder content. The endothermic peak for the transition was observed between 68^{0}-72^{0}C for each composition.



(a) **(b)** **(c)** **(d)**

Fig. 14. DSC curves for untreated composite sample with (a) 10% MP (b) 20% MP (c) 30% MP (d) 40% MP

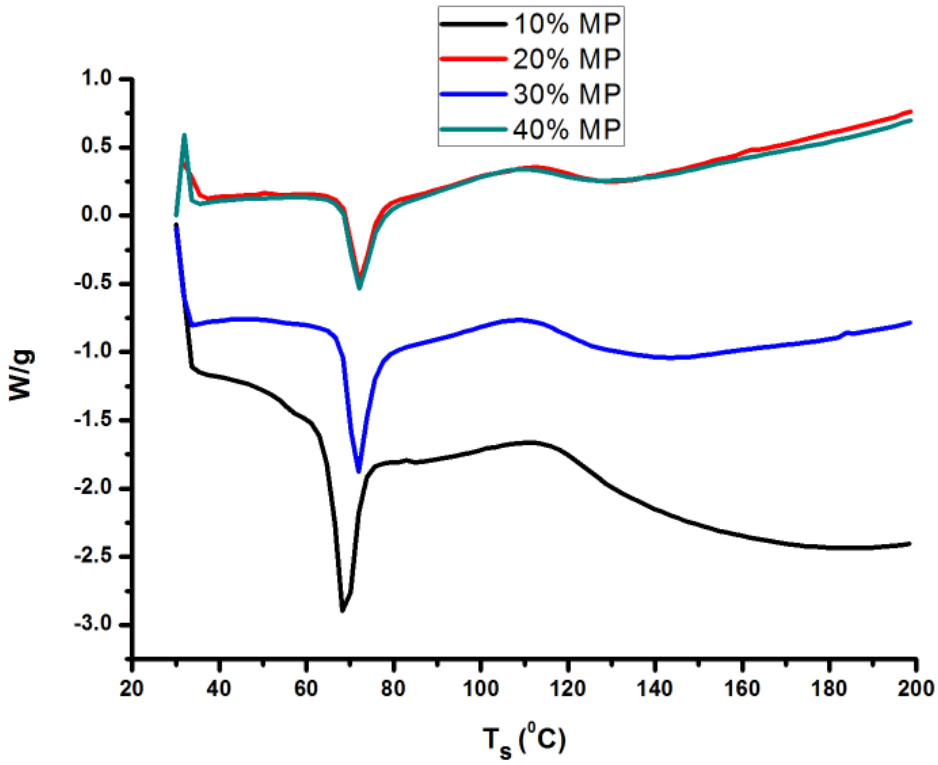


Fig. 15. Comparison of Glass Transition Temperature (Tg) from DSC plots for different composite composition (untreated)

Table 7: Glass Transition Temperatures (T_g) for MP/Epoxy composites with different weight fractions of marble powder

Sample	T_g (^0C)
10% MP+90% Epoxy	68.27
20% MP+80% Epoxy	72.12
30% MP+70% Epoxy	72.01
40% MP+60% Epoxy	72.12

4.4. FTIR Analysis

The FTIR data gave an idea about the molecular structures, chemical bonds present and their strength in the sample. The different bonds present give information about the properties of the composite and how these bonds can be modified to improve the same. Fig. 15. compares the FTIR plots of % transmittance vs the wavenumber of the incident IR beam. It is observed that the plot trend was almost similar for every composition of the composite. Similar peaks were obtained at any particular wavenumber for each sample. The wavenumber, the types of bonds and the bond strength of the observed peaks were determined using the IR chart and have been tabulated in Table 6.

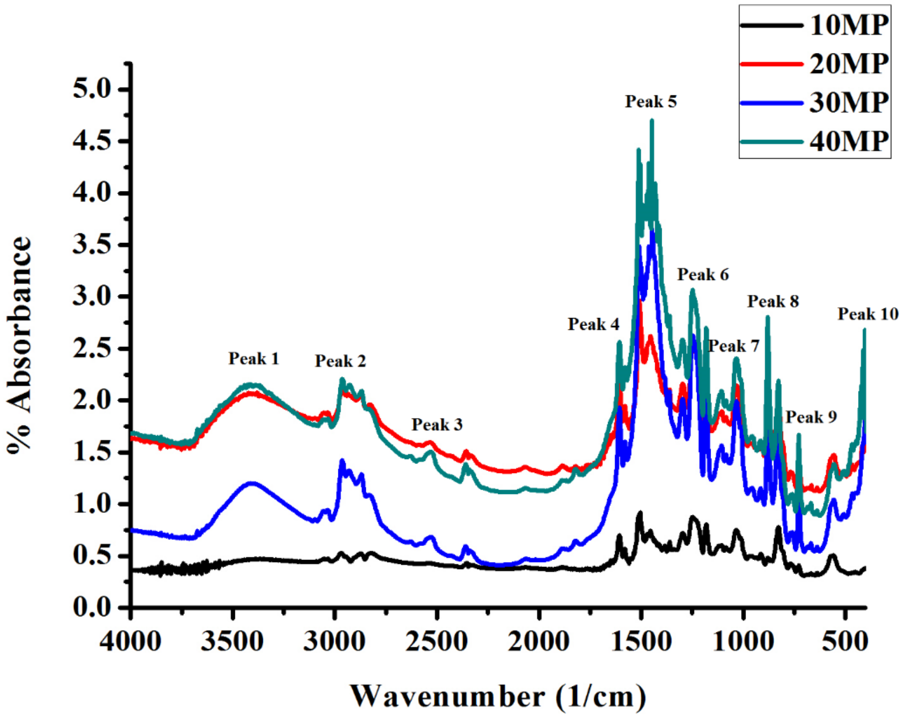
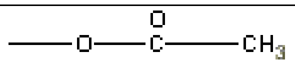
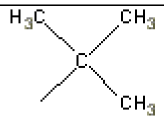
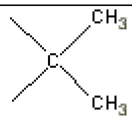
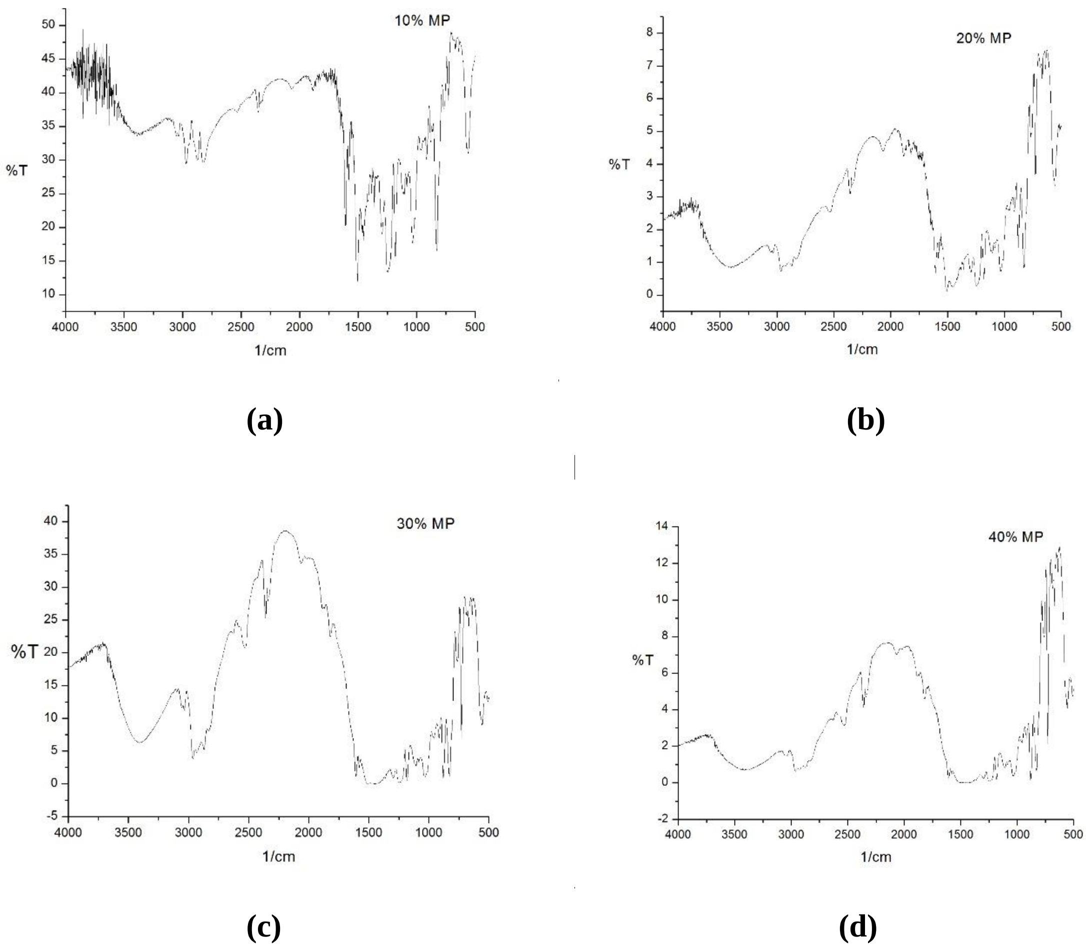


Fig. 16. Comparison of FTIR peaks for the MP/Epoxy composites of different compositions

Table 8: Observed FTIR peaks for the MP/Epoxy composites

Peak	Wavenumber	Bond	Comments
Peak 1	3500-3700	O-H STRETCH free	strong, sharp
Peak 2	3200-3600	O-H (stretch, H-bonded)	strong, broad
Peak 3	2100-2260	Alkynes stretch	variable, not present in symmetrical alkynes
Peak 4	1800-1830 & 1740-1775	Anhydride C=O stretch Ketone cyclic stretch	two bands strong
Peak 5	1740-1720 1700-1725	Aldehyde C=O stretch Acid C=O stretch	strong strong
Peak 6	1340-1385	 acetates	Medium strength
Peak 7	1165-1225		Medium strength
Peak 8	790-840		Weak
Peak 9	600-800 675-1000	C-Cl stretch =C-H bending	strong strong
Peak 10	485-540	Straight-chain/branched alkanes	weak



(a) (b)

(c) (d)

Fig. 17. %Transmittance vs wavenumber for untreated MP/Epoxy composites with (a) 10 wt. % MP, (b) 20 wt. % MP, (c) 30 wt. % MP, and (d) 40 wt. % MP

Chapter 5

CONCLUSIONS

The following conclusions can be drawn from the investigations conducted on the marble powder/epoxy composites:

1. Marble powder reinforced epoxy matrix composites can be easily fabricated with different weight fractions of reinforcements.
2. The untreated marble powder/epoxy composites showed increasing wear with increasing weight fraction of marble powder.
3. The presence of hard and non-uniform marble powder resulted in their fragmentation and thus it was the major contributor to the composite wear.
4. On post-treating the composites at 120^0C for 30 minutes, the wear resistance of the composites was found to decrease.
5. The wear rate of the treated marble powder/epoxy composites increased with increasing marble powder content.
6. The SEM images of the untreated marble powder/epoxy composites revealed that the wear track width increased with increasing marble powder content in the composite. More number of marble powder fragments were observed on the wear surface.
7. The SEM images of the treated (post-cured) marble powder/epoxy composites revealed that the wear track width increased with increasing marble powder content in the composite. More number of marble powder fragments were observed on the wear surface.
8. The DSC analysis of the marble powder/epoxy composites showed that there is negligible change in the glass transition temperature of the composite with increasing marble powder composite.
9. The glass transition temperature for each composition of the composite lies between 68^0C-72^0C.
10. The FTIR analysis shows the different bonds that exist between the marble powder and the epoxy matrix, in the composite. Very similar nature of bonding is observed for each composition of the composite.

SCOPE FOR FUTURE WORK

- The wear behaviour of the composite with varying wear parameters like load and sliding velocity can be evaluated.

- The effect of post-curing on the glass transition temperature and the chemical bonding in the composites can be investigated using DSC and FTIR analyses, respectively.

- Mechanical properties like hardness, three point bend test and Charpy impact test are to be conducted for evaluating the mechanical properties of the composites.

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