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Synthesis, Characterization and Comparisons of Mechanical and Tribological Behaviour of Ortho and Meta Cresol Novalac Epoxy Resin

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Abstract

In this study ortho and meta cresol novalac epoxy resins were synthesized to study their mechanical and tribological behaviour under dry sliding conditions. The friction and wear tests were carried out on a pin on disc apparatus under applied normal load of 10, 20, 30 and 40 N and different sliding velocity of 0.9, 1.8, 2.7 and 3.6 m/s respectively. To evaluate mechanical properties of these synthesized novalac epoxy resins the tensile, flexural and compression tests have been carried out. It is found that in both novalac epoxies the coefficient of friction decreases with increase in the applied normal load and increases with increase in the sliding velocity where as specific wear rate increases with increase in the applied normal load and decreases with increase in the sliding velocity. It is also found that the mechanical properties of the ortho cresol novalac epoxy resins showed better results in comparison to the meta cresol novalac epoxy. The morphology of these novalac epoxy is studied with the help of (SEM) scanning electron microscope.

Keywords: Ortho/meta cresol novalac epoxy resin, Mechanical properties, Friction and wear.

Contents

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1. Introduction

Epoxy resins are a major class of thermosetting polymer. Since the introduction in the 1940s epoxy resins have been widely used on a large scale as matrices for fiber reinforced composite materials, adhesives, coatings, castings, composites in aerospace, painting materials, electronic industries as well as other engineering materials due to their excellent thermal and chemical resistance, superior mechanical properties, and high adhesion to many substrates [1-5]. The main reasons for this are the beneficial high mechanical strength and stiffness of epoxy resins, their high chemical resistance, their good processability and their superior performance at temperatures that occur in automotive applications. The most distinct feature of epoxy resins however is the extraordinary wide range of available monomers, which allows for tailoring coatings to meet the demands of individual applications beyond the mere choice of a single (thermoplastic) matrix [6-8].

In recent years more than 90% of the world's overall epoxy production is based on the diglycidyl ether of bisphenol-A (DGEBA). However composites for high-temperature applications are mainly based on novolac resins since their glass transition temperatures are about 50˚C higher than for bisphenol-A resins. Also novolac epoxy resins have high concentration of benzene ring and multifunctional group which are desirable to have good thermal stability as well as superior mechanical properties [8, 9].

One part of composite material for technical applications may be represented by a thermosetting polymer matrix like epoxy resin, which already covers alone some of the demanded properties. Now a day's many polymers composites are being used as sliding components which were formaly composed only of metallic materials. Further new developments are still under way to explore other fields of application for these materials and tailor their properties for extreme load bearing and environment temperature conditions [10]. Many researchers reported that the various kinds of reinforcement agents such as metal particles, clays, glass, rubber particles, thermoplastics and ceramic particles have been used to improve the mechanical strength, wear resistance and thermal properties [11-19]. Also due to the improvement in the processing methods the friction and sliding wear mechanism of polymer matrix and its composites have been studied by many researchers [20-24].

From the literature it is found that no information were availableabout mechanical properties, friction and wear behaviour of ortho and meta cresol novolac epoxy resin. That means none of the published work is directly comparable to the current work. The objective of this study is to synthesize the ortho and meta cresol novalac epoxy resin and to study their mechanical properties, friction and wear behaviour under dry sliding conditions at different applied normal loads and different sliding velocities. Therefore it is intended to establish baseline data of these novalac epoxy resin for the tribological applications.

2. Experimental Procedure

2.1. Material

Ortho/meta cresol and epichlorohydrin were purchased from Fisher Scientific Chemical Ltd. India; Toluene is purchased from Sigma Aldrich. Methanol GR grade, Acetone and sodium hydroxide pellets purified (MKOM603629) were all purchased from Merck India Ltd. And the curing agent 4, 4 Diaminodiphenylmethane was purchased from (Alfa Aesar), Pyridine AR is purchased from SD fine Chem. Ltd, Whatman filter paper no. 42 are also purchased. The ortho/meta cresol formaldehyde novolac resin and novalac epoxy was synthesized as described below.

2.2. Synthesis of Ortho and Meta Cresol Formaldehyde Novolac Resin

Ortho/meta cresol (180 gm) was dissolved in 6 ml of water and formaldehyde in the molar ratio of 1:0.7 was used for the preparation of novolac resin. The reaction scheme for the synthesis of ortho and meta cresol formaldehyde novalac resin are shown in Fig. 1 (a, b).

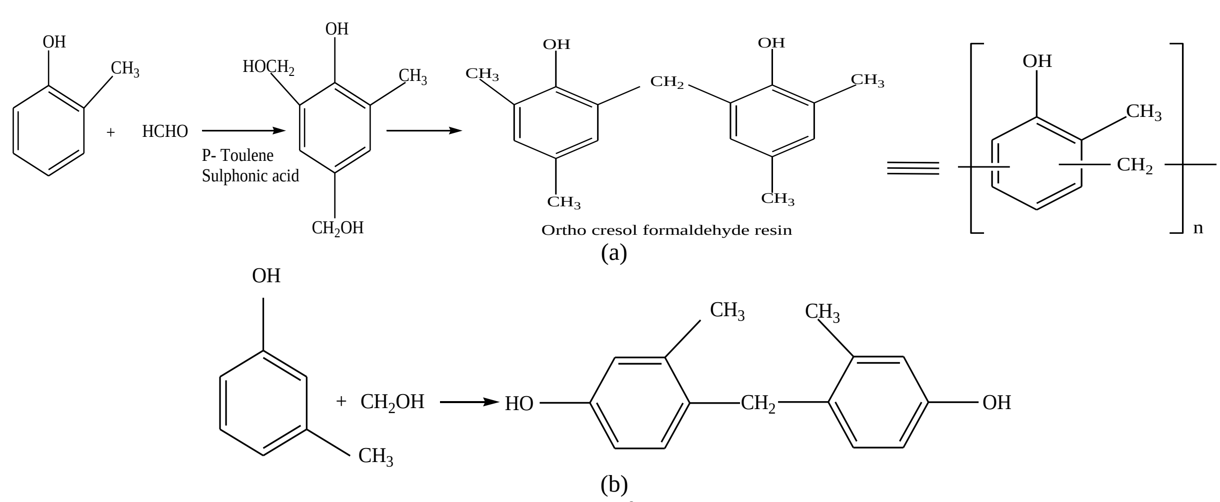


Fig-1. (a, b) Ortho and cresol formaldehyde novalac resin

The mixture was heated to 80° C and formaldehyde solution (55.3 ml) solution (55.3 ml) was added drop wise while maintaining this temperature and under constant stirring over a period of 3.5 hours. The reaction was arrested by adding 40 ml of 10 wt% sodium bicarbonate and the product was finally washed with worm water and dried at 80° C under reduced pressure (40 ± 5mm Hg).

2.3. Synthesis of Ortho and Meta Cresol Formaldehyde Novolac Epoxy Resin

Epoxy novolac resin were prepared by reacting the above synthesized novolac resin with epichlorohydrin (5 moles for every phenolic group of the novolac resins). Sodium hydroxide in the mole ratio of 0.2:1.0 with epichlorohydrin was used as a catalyst. The reaction was carried out (112°C ± 1° C) with the addition of sodium hydroxide (40 wt%) gradually over a period of 3 and 1/2 hours. The reaction scheme of synthesized ortho and meta cresol formaldehyde novolac epoxy resin are shown in Fig. 2 (a, b).

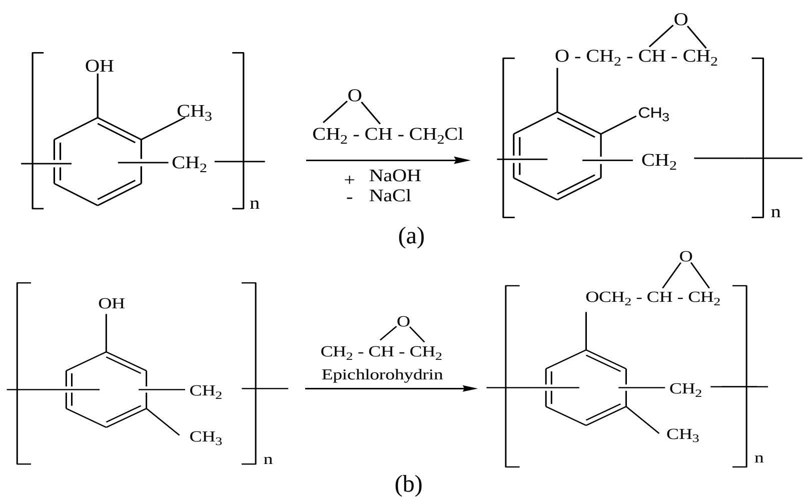


Fig-2. (a, b) Ortho and meta cresol novalac epoxy resin

The contents were dissolved in toluene and the solution was filtered using Whatman filter paper no. 42 to remove the salts. Toluene was then removed by heating under reduced pressure.

2.4. Fabrication of Test Specimens

Calculated amount of ortho/meta cresol novalac epoxy resin were mixed with the help of mechanical stirrer at temperature of 80°C and 4, 4^1 diamino diphenyl methane (DDM) as shown in Fig. 3 was used as a curing agent and which was hand mixed thoroughly.

The resin and curing agent was mixed parts by weight according to the following relation [25].

$$\text{Theoretical mixing ratios} = \frac{\text{Weight of amine}/\text{Number of available atom}}{\text{Epoxy equivalent weight}} \quad (1)$$

After mixing the mixture it was degassed in a vacuum chamber for about half an hour. Then the mixture was poured into the aluminum foil covered molds for degassing and room temperature curing for 2-3 hours. After degassing and room temperature curing the molds were placed in an oven and the temperature of the oven were first set as 100˚C for about 1 hours and after that for post curing the temperature of the oven were set as 180˚C for 2 and half hours so that the samples were cured properly.

2.5. Epoxy Equivalent Weight

The epoxy equivalent weight of ortho/meta cresol novalac epoxy resin was determined by pyridinium chloride method [26]. The epoxy equivalent was calculated using the following equation:

$$\text{Epoxy equivalent weight} = \frac{16 \times \text{Sample weight,(g)}}{\text{Grams of oxirane in sample}} \quad (2)$$

Grams of oxirane in the sample = (A-B) (N) (0.0016)

Where

A- ml of NaOH for blank

B- ml of NaOH for sample

N- Normality of NaOH.

2.6. Mechanical and Wear Characterization

The Mechanical Properties such as tensile strength, flexural strength and compression strength tests of the ortho and meta cresol novalac epoxy resin specimens were determined by performing the tests on a Hounsefield-25KN universal testing machines at a deformation rate of 2 mm/min. The hardness tests of these epoxy composites were performed on a Rockwell hardness tester as per the ASTM standards, density of these novalac epoxy resin specimen is obtained by the Archimedean principle.

The friction and wear tests were carried out by using a pin on disc friction and wear monitoring tester (DUCOM) as per ASTM G 99 at a constant sliding distance of 3000m, and the diameter of the wear track was 100 mm. The smooth and hardened ground steel disk surface (EN-32, hardness 72 HRC) served as counterpart and which was finished by abrasion against 1200 grade SiC paper which provided a surface finish of (R_a = 0.6μm to 0.7μm). The circular cross section end of these novalac epoxy resin specimen pin was also finished by 400 grade size Sic paper for sliding contact. The specimen is held stationary and the disc is rotated while a normal force being applied through a lever mechanism. During the test friction force was measured by transducer mounted on the loading arm. Applied normal loads of 10, 20, 30 and 40 N were applied to the lever arm for the four sliding velocities 0.9, 1.8, 2.7 and 3.6 m/s during the tests. The friction force readings are taken as the average of 100 readings every 40seconds for the required period. For this purpose a microprocessor controlled data acquisition system is used. The environment conditions in the testing laboratory were 30˚C and 63 % relative humidity.

Table-1. Material and test conditions

Materials	Type of Epoxy	Measured Density gm/cm^3	Temperature (°C)	Humidity (%)	Load (N)	Sliding speed (m/s)
A	OCNE	1.22	33	65	10 20 30 40	0.9 m/s 1.8 m/s 2.7 m/s 3.6 m/s
B	MCNE	1.24	33	65	10 20 30 40	0.9 m/s 1.8 m/s 2.7 m/s 3.6 m/s

Weight loss method was used for finding the specific wear rate. During these experiments initial and final weight of the specimens were measured. The material loss from the composite surface is measured using a precision electronic balance with accuracy $\pm$ 0.01 mg. The specific wear rate (mm^3/Nm) is then expressed on volume loss bases.

$$W_s = \frac{\Delta M}{\rho L F_N} \tag{3}$$

Where W_s is the specific wear rate (mm^3/Nm), ΔM is the mass loss in the test duration (gm), ρ is the density of the composite (gm/cm^3) and F_N is the average normal load (N). The materials and test conditions are given in Table 1.

2.7. Scanning Electron Microscope

The Scanning Electron Microscope (SEM) was used to analyze the worn surface of the composites. The SEM analysis is performed by a FEI quanta FEG450 machine. The samples are mounted on stubs with gold coating. To enhance the conductivity of the samples, thin films of platinum are vacuum evaporated onto them before the photomicrographs were taken.

3. Results and Discussion

3.1. FTIR Analysis

The FTIR spectrums of novalac epoxy resin detected are shown in Fig. 3. The Fig. 3 shows the FTIR spectrum of cresol based novalac epoxy. The C-H stretching of the epoxy ring is formed at 2980 cm^{-1}. Epoxidation of ortho cresol formaldehyde novalac epoxy was followed by the appearance of the band due to the symmetrical stretching or ring breathing frequency of the epoxy ring at 1254 cm^{-1} and the most significant features is the appearance of C-C stretching of the epoxy ring at 917 cm^{-1} respectively.

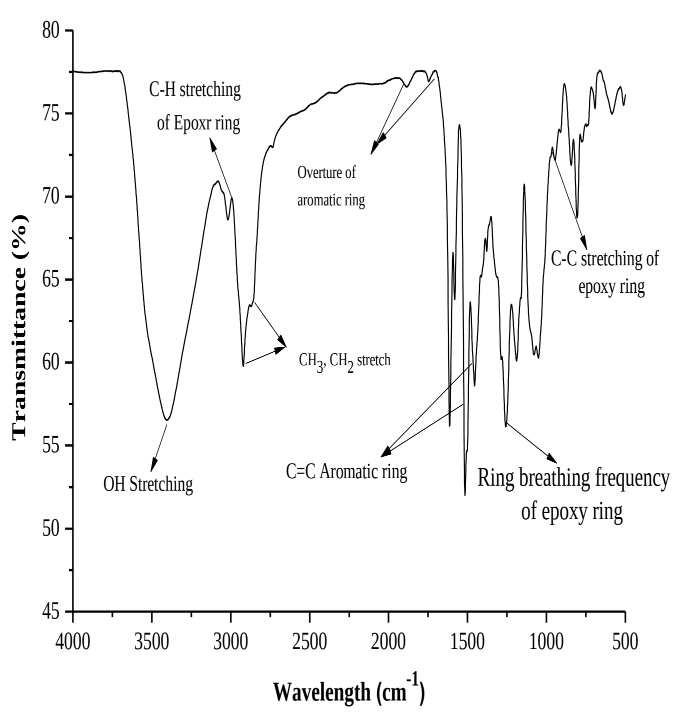


Fig-3. FTIR spectra of cresol based novalac epoxy resin

3.2. Hardness

The hardness of ortho and meta cresol novalac epoxy resin is shown in Fig. 4. From the Fig. 4 it is reported that the hardness values of ortho cresol novalac epoxy resin (OCNE) lies in the range of (40 HRC) and the hardness values of meta cresol epoxy resin (MCNE) lies in the range (44 HRC).

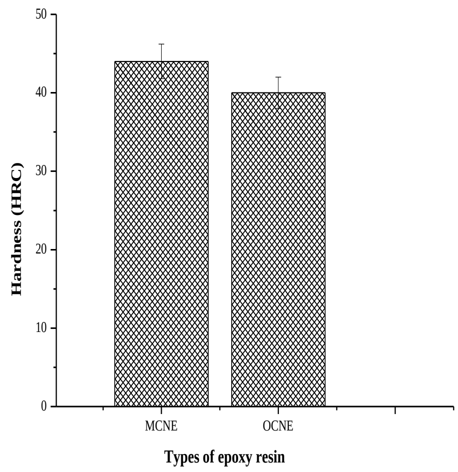


Fig-4. Hardness of ortho and meta cresol novalac epoxy (OCNE/MCNE).

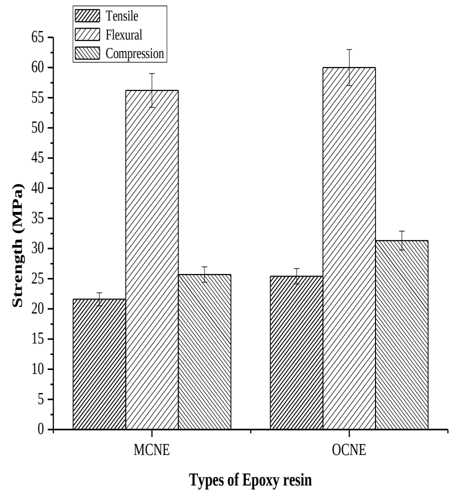


Fig-5. Mechanical properties of the ortho and meta cresol novalac epoxy (OCNE/MCNE).

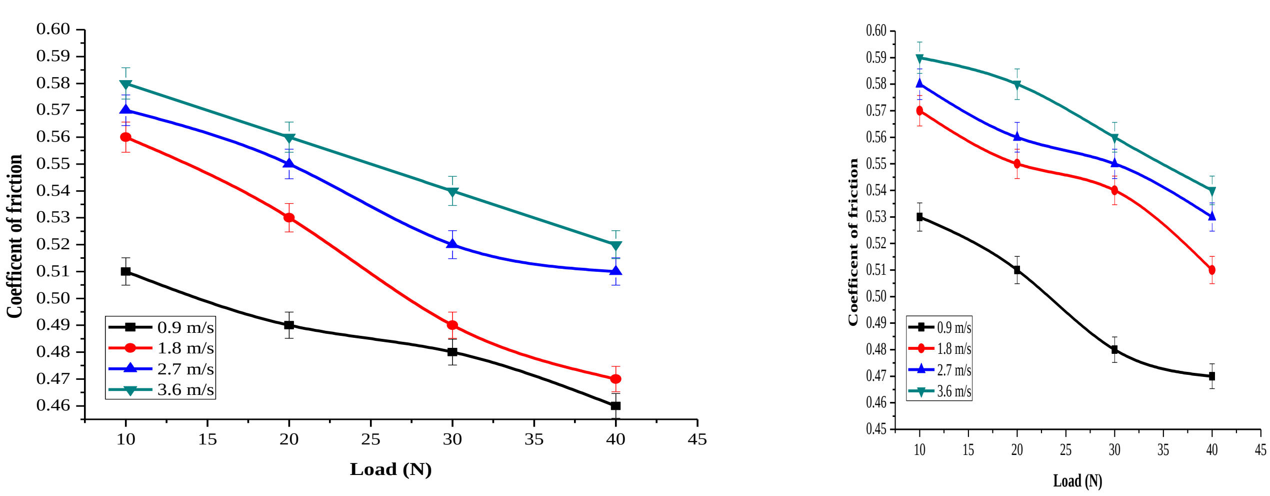
It is observed that the hardness of meta cresol novalac epoxy resin is approximately equal or slightly more than the ortho cresol novalac epoxy resin. This is due to the reason that meta cresol novalac epoxy has high density and high epoxide equivalent weight and also the formation of the three dimensional network and the high degree of cross linking which dominates the cross-linking process leading to the formation of a stronger material which exhibits better hardness. Also during curing with high temperature it becomes brittle and hence high hardness.

3.3. Mechanical Test

In the mechanical test the tensile strength, flexural strength and compression strength of the ortho and meta cresol novalac epoxy resin were carried out. The tensile, flexural and compression strength of these novalac epoxy resin is shown in Fig. 5. It is reported from the Fig. 5 that in case of meta cresol novalac epoxy resin (MCNE) the values of tensile, flexural and compression strength lies in the range of (21.6MPa, 56.2MPa and 25.7MPa) and in case of ortho cresol novalac epoxy resin (OCNE) the values of tensile, flexural and compression strength lies in the range of (25.4MPa, 60MPa and 31.3MPa) respectively. Also it is found that the mechanical properties of meta cresol novalac epoxy resin is less this is due to the presence of a large number of epoxy rings and their tight, rigid and brittle structure. Where as in case of ortho cresol novalac epoxy resin shows slightly high because of the large number of hydrogen groups thus more epoxy rings would be opened making the material tougher and hence higher mechanical properties.

3.4. Coefficient of Friction and Sliding Wear

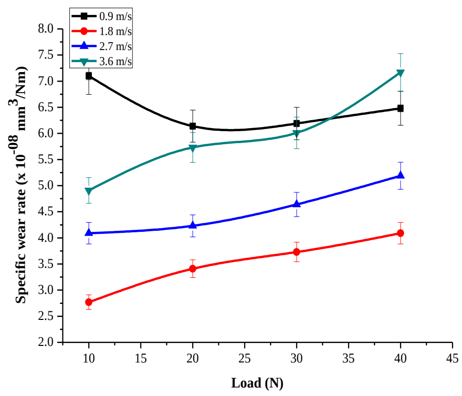
The coefficient of friction and the specific wear rate of the ortho and meta cresol novalac epoxy resin are shown in Figs. 6 and 7 respectively. The Fig. 6 presented the variation of coefficient of friction for (a) ortho cresol novalac epoxy (OCNE) and for (b) meta cresol novalac epoxy (MCNE) with applied normal load values of (10, 20, 30 and 40N) at different sliding velocity of (0.9, 1.8, 2.7 and 3.6m/s). Also Fig. 7 presented the variation of the specific wear rate for (a) ortho cresol novalac epoxy (OCNE) and for (b) meta cresol novalac epoxy (MCNE) with applied normal load values of (10, 20, 30 and 40 N) at different sliding velocity of (0.9, 1.8, 2.7 and 3.6m/s) respectively.



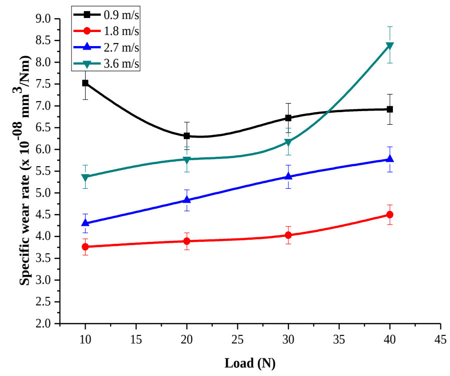
(a) (b)

Fig-6. Variation of coefficient of friction for (a) ortho cresol novalac epoxy (OCNE), for (b) meta cresol novalac epoxy (MCNE) with applied normal load values of (10, 20, 30 and 40N) at different sliding velocity of (0.9, 1.8, 2.7 and 3.6m/s).

It can be seen from Fig. 6 (a, b) that the coefficient of friction of ortho and meta cresol novalac epoxy resin decreases with increase in the applied normal load and increases with increase in the sliding velocity. The maximum and minimum coefficient of friction of ortho and meta cresol novalac epoxy lies in the range of (0.32, 0.52 and 0.41, 0.57) respectively.



(a)



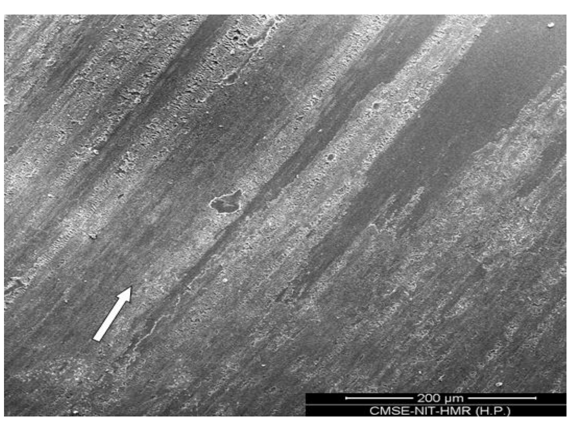
(b)

Fig-7. Variation of specific wear rate for (a) ortho cresol novalac epoxy (OCNE), for (b) meta cresol novalac epoxy (MCNE) with applied normal load values of (10, 20, 30 and 40N) at different sliding velocity of (0.9, 1.8, 2.7 and 3.6m/s).

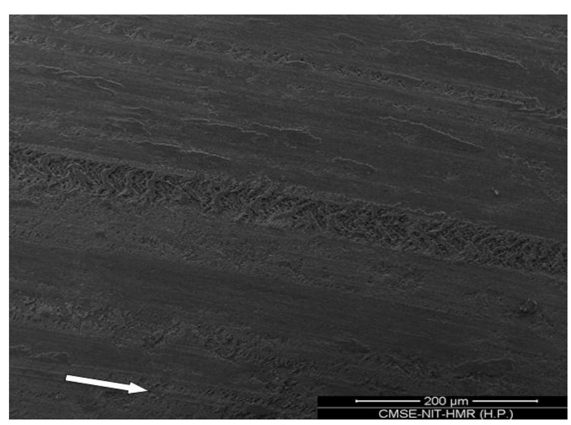
From Fig. 7 (a, b) it is found that with increase in the applied normal load the specific wear rate of the ortho and meta cresol novalac epoxy resin increases where as with increase in the sliding velocity the specific wear rate of these novalac epoxy decreases . The maximum and minimum specific wear rate of ortho and meta cresol novalac epoxy lies in the range of (7.17 × 10^{-8} and 2.77 × 10^{-8} mm^3/Nm and 8.40 × 10^{-8} and 3.76 × 10^{-8} mm^3/Nm) respectively. It was also found that the specific wear rate of the meta cresol novalac epoxy resin is higher in comparisons to the ortho cresol novalac epoxy resin. This is because of the high brittleness of the meta cresol epoxy resin, and during testing because of this high brittleness behaviour of the meta cresol epoxy resin more cracks were formed perpendicular to the sliding direction. Therefore more material removal and wear debris was produced and hence high specific wear rate.

3.5. Worn Surface Morphology

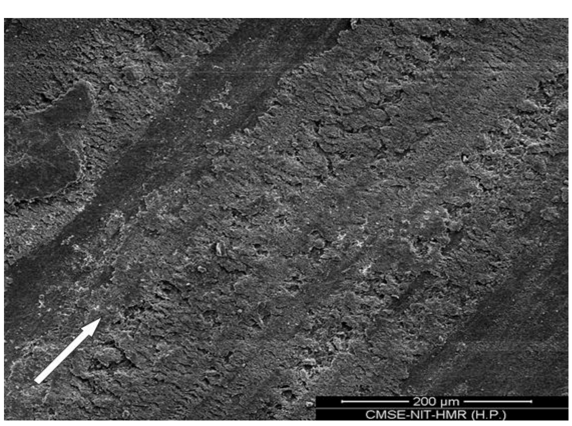
To understand the morphology and the corresponding sliding wear mechanism of ortho and meta cresol novalac epoxy resin, the worn surface were inspected with the help of scanning electron microscope (SEM).Fig. 8 (a, c) shows the SEM micrographs of ortho cresol novalac epoxy under sliding speed and applied normal load (0.9 m/s, 10 N and 3.6 m/s, 40 N) and Fig. 8 (b, d) shows the SEM micrographs of meta cresol novalac epoxy under sliding speed and applied normal load (0.9 m/s, 10 N and 3.6 m/s, 40 N) respectively. The arrow shows the sliding direction. The worn surface of ortho and meta cresol novalac epoxy as shown in Fig. 8 (a-d) is caused by the fatigue wear mechanism for the material loss in the form of thin layers.



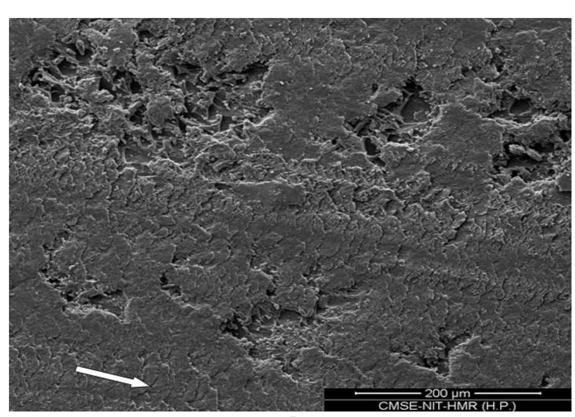
(a)



(b)



(c)



(d)

Fig-8. (a, d) SEM micrographs of ortho cresol novalac epoxy under sliding speed and applied normal load (0.9 m/s, 10N and 3. m/s, 40N) and (b, d) of meta cresol novalac epoxy under sliding speed and applied normal load (0.9 m/s, 10N and 3.6m/s, 40N) respectively.

From Fig. 8 (a, b) it is clear that at low applied normal load and sliding velocity (10 N, 0.9m/s) the worn surface were found much smoother in comparison to the high applied normal load and sliding velocity (40 N, 3.6m/s) shows much rough surfaces as shown in Fig. 8 (c, d). It is also found that when the applied normal load and sliding velocity increases up to (40 N and 3.6m/s) the contact temperature and the friction force were gradually increased which caused the breakage of the matrix at the interfacial region therefore the surface damage was remarkably as shown in Fig. 8 (c, d) respectively.

4. Conclusions

The present work deals with the synthesize of ortho and meta cresol novalac epoxy resin and to study their comparison on the mechanical properties, friction and wear behaviour of these novalac epoxy resin under dry sliding conditions. The main conclusions drawn from this are listed below:

- The hardness of the meta cresol novalac epoxy resin is approximately equal or slightly more than the ortho cresol novalac epoxy resin. Whereas on the other hand the mechanical properties such as tensile, flexural and compression strength of the ortho cresol novalac epoxy resin is found more in comparison to the meta cresol novalac epoxy resin.
- The coefficient of friction of these novalac epoxy resin is decreases with increase in the applied normal load and increases with increase in the sliding velocity. Whereas the specific wear rate increases with increase in the applied normal load and decreases with increase in the sliding velocity. The maximum and minimum coefficient of friction of ortho and meta cresol novalac epoxy lies in the range of (0.32, 0.52 and 0.41, 0.57). Similarly maximum and minimum specific wear rate of these novalac epoxy lies in the range of (8.40×10^{-8}, 3.76×10^{-8} mm^3/Nm and 7.17×10^{-8}, 2.77×10^{-8} mm^3/Nm) respectively.
- From SEM analysis it is found that at low applied normal load and sliding velocity (10 N, 0.9 m/s) the worn surface of these novalac epoxy resin were found much smoother in comparison to the high applied normal load and sliding velocity (40 N, 3.6 m/s) shows much rough surfaces.

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