Effect of surface treatment on tribological characteristic of ferrite nanoparticles epoxy composites

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Abstract. In this investigation, surface treatment of ferrite nanoparticles were efficiently prepared by using amino propyl silane (APS). XRD which is a spectral analysis technique used for investigate the morphology and chemical structure of treated and normal ferrite nanoparticles. Ferrite nanoparticles epoxy composites were prepared by Modified ferrite nanoparticles (MFNs) with varying weight percentage and ferrite nanoparticles (FNs). XRD shown that MFNs was effectively modified with highly crystalline structure and silane surface treatment decreased the average particle size of FNs to ∼16.48 nm. Surface treatment of FNs enhanced the homogeneous dispersion of ferrite in epoxy resin and improved interfacial adhesiveness between MFNs and epoxy. Tribological property of Modified ferrite nanoparticles epoxy composite (MFNEs) compare to cured epoxy and normal ferrite nanoparticles epoxy composites (FNEs) has been enhanced due to surface treatment.

Keywords. Ferrite Nanoparticles, Nano-magnetic Polymer Composites, Spectra, Tribological Characterization, Silane Coupling

1. Introduction
There is an expanding interest for multi-functional composites to encounter extraordinary prerequisites of engineering segments. [1–2]. Engineers and scientists both are facing a new problem since last decades that how to enhance the tribological properties of a composite. The necessity of metal substitution in radar, sensors, electronic device, microwave absorbers and rocket application requires stunning tribological properties for better working life cycle. There are numerous answers for taking care of the issue. The most widely recognized is to fill the polymer with different powders. [3-5].

Ferrite nanoparticles are critical class of attractive materials from application perspective and are widely utilized as lasting magnets in market in light of their low value, high attractive execution, non-toxicity, biocompatibility, high resistance to corrosion and environment-friendly qualities [6-9]. So due to decent electromagnetic properties Ferrite nanoparticles is one of the most promising and greatest...
characterized filler particles, which is united with epoxy, i.e., to record media [10] and in medical applications [11]. Numerous polymorphic forms of Ferrite nanoparticles are used in the field of aerospace, sensor and other electronic fields. Epoxy are extensively used in numerous applications due to their higher adhesion, high strength, and resistance to creep, heat and chemicals [12].

But ferrite nanoparticle epoxy composites have some limitations. Ferrite nanoparticle-strengthened properties of epoxy nano-composites but suffered low tribological problems. One of the main reason of low tribological characteristics of ferrite epoxy composites is poor dispersion of ferrite particles into epoxy resin [13]. Main concern in the fabrication of ferrite/polymer composites is to accomplish the good dispersion and interfacial strength between ferrite nanoparticles and the epoxy resin. Actually during the process when ferrite nanoparticles dispersed in to epoxy resin slurry, powerful magnetic attraction between the ferrite nanoparticles started agglomeration of ferrite nanoparticles. Agglomerates of hydrophilic ferrite nanoparticles could not be dispersed homogeneously in organic epoxy normally assisted to establishment of imperfections belongs to micro-structural in the ferrite nanoparticles epoxy composites. This types of imperfection affect the composite’s tribological properties [14-16].

In current study point of view, Ferrite particles were surface treated by APS coupling agent. Modified ferrite nanoparticle epoxy were fabricated through dispersion of modified ferrite nanoparticles and normal ferrite nanoparticles into epoxy matrix. For examination the surface treatment effect on ferrite nanoparticles with APS by tribological properties of composite; microstructural characterization was analyzed using XRD and tribological characterizations by Pin on Disc wear test.

2. Experimental

2.1. Materials
The commercially available epoxy (CY230) which is based on DGEBA and hardener (HY 951) were procured from M/S Huntsman India Limited. APS were purchased by Himedia Laboratory Pvt. Ltd. India. Ferrite nanoparticles (FNs) (M.W.159.69, 95%) were purchased from Research-Lab Industries, Mumbai, India. All chemicals and solvents used without further purifications. Deionized water (conductivity10^{-8} S/cm) was used in the preparation.

2.2. Synthesis of modified ferrite nanoparticles epoxies (MFNEs)
The synthesis of MFNEs has been implemented into two phases. Primarily, ferrite nanoparticles (FNs) modified by surface treatment. In this process firstly, 10gm FNs was mixed into 1 liter deionized water in a round bottom glass flask using ultra-sonication for 15 min. The surface modification of ferrite particles were started with the help of 9 ml APS which added drop wise into ferrite deionized slurry. For vigorous stirring a Teflon-coated powerful stirring bar has been used for 12 hour at 45±5 °C. The achieved centrifuged modified nanoparticles was cleaned by distilled water- ethanol solution (1:1) four times and then dried at 90±5 °C to acquire MFNs and on secondary stage, Epoxy composites was filled with loading different weight percentage of modified ferrite nanoparticle and normal ferrite nanoparticles. Separately, both ferrites particles were firstly mixed properly into resin (CY 230) and then applied ultra-sonication in ice bath for 15 min. The acquired mixture was firstly 1 hour heated at 90 ±10 °C in microwave and then cooled down to 45±5 °C. After that addition of the curing agent (HY 951), the epoxy- modified ferrite nanoparticle and epoxy - normal ferrite nanoparticles were set aside for curing at normal room temp. for 72 hours in teflon moulds to avoid stacking.

2.3. Characterization
The crystallinity of the MFNEs and FNEs samples were characterized using XRD analysis. The diffraction patterns were recorded by a Rigaku-Geiger-flex diffracto-meter using Cu–Kα radiation at 25°C. The investigation was performed though“40 mA current, 40 kV voltage and 1.54 Å wavelength” The 20 range was 4–80 degree with a rate of 1degree /min. Tribological tests of MFNEs,
FNEs and cured epoxy were performed on a pin-on-disk tribometer procured by DUCOM, India. ASTM G 99 standard was used for Specimen’s dimension. The tribometer had a hardened alloy steel disc working as a counter body. Each pin shaped epoxy composite with dimension 20 mm in length and dia10 mm has taken three reading using a pin-on-disc tribometer at two different steady state conditions i.e. 200 RPM and 400 RPM, by keeping other constraints constant.

3. Results and discussion
Figure 1 shows the XRD for MFNs and FNs samples. The found XRD patterns attributed for the “rhom-bohedral phase” for “α-Fe2O3” conferring to ICDD ref. card04-008-7623. The shown XRD patterns exhibit high crystalline nature and pure structure of “α-Fe2O3” specimens [17]. Also, the expansion in heights of the intensity peaks approves enchasing the intensity of crystallization. The average crystalline size of MFNs was found to be 16.48 nm using Scherrer’s formula as explained in “Eq. (1)”.

\[ T = \frac{K\lambda}{\beta \cos \theta} \]  

Here, “T” (average crystalline size), “K” (0.89), “θ” (Bragg’s angle) and “λ” (wavelength) and “β” (FWHM of intense XRD peak at θ).

Figure 1. XRD pattern of MFNs and FNs

Figure 2 – Figure 3 shows the variations in different wear rate of cured epoxy, FNEs and MFNEs with different concentration of MFNs (wt%). Figure 2 represents the variations in wear rate \(W_R \times 10^{-2}, \text{mm}^3/\text{m}\) of cured epoxy and MFNEs with different concentration of MFNs (wt %) at two different rpm 200 and 400 respectively. The variations in decrement in \(W_R\) of MFNEs over cured epoxy have been represented in the Figure 2 – Figure 3. The cured epoxy has shown \(W_R\) by 3.16 and 3.39 at 200rpm and 400rpm respectively. With increasing concentration of MFNs a notable reduction in \(W_R\) has been shown for each of MFNEs. Figure 3 has shown \(W_R\) comparison between cured epoxy, ferrite nanoparticle epoxies (FNEs) and modified ferrite nanoparticle epoxies (MFNEs).

At 200 rpm, MFNEs-I shows a decrease in \(W_R\) over cured epoxy by 22.15%. Increase in concentration of MFNs to 1wt%, MFNEs-II, has further contributed decrease in \(W_R\) of MFNEs over cured epoxy by 34.17%. Similarly at MFNs concentration 1.5wt%, MFNEs-III and MFNs concentration 2wt%, MFNEs-IV shows a decrement in value of \(W_R\) by 41.4 and 51.26 respectively over cured epoxy. At 400 rpm, MFNEs-I shows a decrease in \(W_R\) over cured epoxy by 15.92%.
Increase in concentration of MFNs to 1wt%, has further contributed decrease in \( W_R \) of MFNEs over cured epoxy by 25.07%. Similarly at MFN concentration 1.5wt%, MFNEs-III and MFNs concentration 2wt%, MFNEs-IV shows a decrement in value of \( W_R \) by 28.31 and 33.03 respectively over cured epoxy (Figure 2).

![Figure 2. Wear Rate of different MFNEs with varying speed at 200rpm and 400rpm](image)

In Figure 3 has shown a comparison of \( W_R \) which shows that due to good dispersion and enhance adhesion between MFNs and epoxy, lower \( W_R \) of MFNEs comparison to FNEs and cured epoxy. The percentage decrease in the \( W_R \) of FNEs and MFNEs by 43.98 and 51.26 respectively as compared to
cured epoxy at 200rpm and similarly 28.32 and 33.03 respectively as compared to cured epoxy at 400 rpm.

Figure 4. Wear Resistance of different MFNEs with varying speed at 200rpm and 400rpm

Figure 5. Variation in Wear Resistance of Cured Epoxy, FNEs and MFNEs at 200 rpm and 400rpm

Figure 4 represents the variations in wear resistance ($W_{RE}$ mm$^3$) of MFNEs with different concentration of MFNs (wt %) at two different rpm 200 and 400 respectively. The variations in increment in $W_{RE}$ of MFNEs over cured epoxy have been represented in the Figure 4 – Figure 5. The cured epoxy has shown $W_{RE}$ by 31.6 and 29.45 at 200 rpm and 400 rpm respectively. With increasing concentration of MFNs a notable increase in WRE has been shown for each of MFNEs. Figure 5 has shown $W_{RE}$ comparison between cured epoxy, ferrite nanoparticle epoxies (FNEs) and modified ferrite nanoparticle epoxies (MFNEs).
At 200 rpm, MFNEs-I shows an increase in $W_{RE}$ over cured epoxy by 28.16%. Increase in concentration of MFNs to 1 wt%, MFNEs-II, has further contributed increase in $W_{RE}$ of MFNEs over cured epoxy by 51.89%. Similarly at MFNs concentration 1.5 wt%, MFNEs-III and MFNs concentration 2 wt%, MFNEs-IV shows an increment in percentage value of $W_{RE}$ by 70.88% and 105.06% respectively over cured epoxy. At 400 rpm, MFNEs-I shows an increase in $W_{RE}$ over cured epoxy by 18.91%. Increase in concentration of MFNs to 1 wt%, has further contributed increase in $W_{RE}$ of MFNEs over cured epoxy by 33.34%. Similarly at MFNs concentration 1.5 wt%, MFNEs-III and MFNs concentration 2 wt%, MFNEs-IV shows an increment in percentage value of $W_{RE}$ by 39.69% and 49.16% respectively over cured epoxy (Figure 4).

In Figure 5 has shown a comparison of $W_{RE}$ which shows that due to good dispersion and enhance adhesion between MFNs and epoxy, higher $W_{RE}$ of MFNEs comparison to FNEs and cured epoxy. The percentage increase in the $W_{RE}$ of FNEs and MFNEs by 78.29 and 105.06 respectively as compared to cured epoxy at 200 rpm and similarly 39.69 and 49.16 respectively as compared to cured epoxy at 400 rpm.

4. Conclusions

The surface treatment of FNs was efficiently accomplished by using APS as coupling agent to achieved MFNs. XRD pattern confirmed that the achieved MFNs has high crystalline structure and their size reduced to 16.48 nm. Improved dispersion of MFNs into epoxy resin and enhanced adhesiveness between filler and epoxy resin was gained due to APS coupling. Due to APS surface treatment agglomeration of ferrite particle has been reduced and ferrite particles homogeneously dispersed in epoxy resin which has been shown better tribological behaviour of ferrite nanoparticles epoxy composites compare to normal ferrite nanoparticles epoxy composite. Results has been Shown that percentage decrease in the wear rate of FNEs and MFNEs by 43.98 and 51.26 respectively as compared to cured epoxy at 200 rpm and similarly 28.32 and 33.03 respectively as compared to cured epoxy at 400 rpm whereas percentage increase in the wear resistance of FNEs and MFNEs by 78.29 and 105.06 respectively as compared to cured epoxy at 200 rpm and similarly 39.69 and 49.16 respectively as compared to cured epoxy at 400 rpm.

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