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Effect of (ZnO) Nanoparticles on The Mechanical, Antibacterial and Morphological Properties of Epoxy Resin

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Abstract
Nanocomposites are materials reinforced with fillers having nanosize that are composed from two materials, the base material and the filler material, where the filler has at least one dimension that falls within the nanoscale. The purpose of this research is to study the effect of additions nanoparticles (ZnO) on the fracture toughness, morphological and antibacterial properties of pure epoxy, nanocomposites were prepared by reinforcing epoxy matrix with two concentration (1%wt and 5 wt%) from zinc oxide (ZnO). Results show that the addition (1% wt. ZnO) gives the best fracture toughness (compact tension and single edge notched bending) properties where the values of KIC for (C.T=4.38 MPa $m^{1/2}$, SENB=5.05 MPa $m^{1/2}$). For the Infrared Fourier Transform Spectrometer (FT-IR) results show physical interaction between nanoparticles and epoxy matrix, and there is no chemical reaction between them. In the Atomic Force Microscope test (AFM) test, an increase in the nanoparticle's surface roughness could lead to the intimate interfacial adhesion between nanoparticles and matrix by mechanical interlocking. Lastly, by examining the antibacterial properties, it was found that all the tested additives were inhibitory to the gram negative bacteria (Escherichia coli) and gram positive bacteria (staphylococcal aureus), and the ratio [1 wt% ZnO)] gives the best result.
Keywords: Fracture Toughness, Morphological and Antibacterial Properties, Epoxy, ZnO

Introduction

Nanocomposites have received a lot of attention in recent decades due to their outstanding characteristics and uses. Antibacterial polymer-metallic nanocomposites outperform all other nanocomposites in terms of antimicrobial characteristics and potential applications [1–3]. Today, health care would be impossible to do without the use of polymer. Polymers are tough and can withstand sterilization, high temps, and a chemical environment. Polymeric materials are important in the infection transmission since they enable fungus and bacteria to proliferate [4, 5]. Bacteria are one-cell microorganisms, which was identified in the 1970s by Van Leeuwenhoek. In the nineteenth century concepts of bacteria have been developed that there is a strong relation between bacteria and diseases. Such consideration became of interest not only to answer some mysterious questions about infection but also to find out substance that kills, inhibits, or at least slows down the growth of bacteria [6]. Several investigations have shown that pathogenic and nonpathogenic organisms polluted hospital surfaces and a lot of old medical equipment [6]. Gram-positive bacteria are the most common cause of illness. Gram-negative bacteria such as Staphylococcus aureus (s. aureus) and Staphylococcus epidermidis The illness seems to be more severe with (s. aureus) and pseudomonas areuginosa than with Escherichia (E. coli) and pseudomonas areuginosa (E. coli). Because control systems are often ineffective, microbial infection is the world's leading cause of death. Because bacteria have the capacity to adapt to drugs over time, treating microbial infections has grown increasingly difficult [7]. As a result, one of the most pressing problems in materials and medicine is bacterial growth management. There are a variety of techniques for preventing contamination, including the use of antibacterial compounds [8]. For example, human has used Zinc Oxide (ZnO) as an antibacterial agent in several applications. Nanoparticle (ZnO NPS) is the next descent of antibacterial agent. Getting through antibiotics and medical devices. As well, studies in (ZnO Nps) application for medical use has been very active, and the achieve products allow to cure several infections and develop new infection safe device [9].

Experimental

Samples Preparation

Pure Epoxy Resin

The epoxy resin was mixed with a hardener using a mechanical stirrer for 30 minutes. the mixture was put under vacuum at room temperature for 30 minutes to remove bubbles. Then, the mixture was poured into the silicon mold, which was previously prepared, then left for 24 hours at room temperature.

Epoxy / ZnO Nanocomposites

Composites were prepared using different concentrations of nanoparticles (NPs) (ZnO), wt. % 1, and 5 "As shown in Table 1". The dispersion of NPs in Acetone was carried out using a mechanical stirrer for 30 minutes. Then the mixture was put in ultrasonic for 45 minutes at 25 °C, then the nanoparticles solution added into resin then using mechanical mixing for 30 min to get good mixing then using ultrasonic for 30 min and evaporate the solution, then the hardener was added and was mixed for 15 minutes using a mechanical stirrer. The prepared mixture put in the degassing system under vacuum at room temperature for 30 minutes to eliminate bubbles, and the mixture was then poured into the mold of silicon and left for 24 hours at room temperature.

Table 1. ZnO NPs Content in The Epoxy Matrix

Sample No.	Epoxy Resin Content (wt%)	Fillers Content (wt%)	ZnO Content (%)
Sample 1	100	0	0
Sample 2	99	1	100
Sample 3	95	5	100

Results and Discussion

Results of Fracture Toughness Test

Compact Tension

It can be observed from " Table 2 and Figure 1" that the nanocomposites with 1%wt and 5 %wt. ZnO shows the highest maximum load and KIC as compared to pure epoxy samples. Furthermore, 1% wt. addition of ZnO is more effective than 5% wt additions as a toughener.

This is due to ZnO having a small ionic radius and good dispersion in the epoxy matrix leading to prevent crack propagation and needing more energy to make the crack propagate in the tension mode, and for 1% wt. better dispersion than for 5% wt.

Table 2. Typical Load-Deformation for Stable Crack Propagation in C.T Samples of Epoxy Matrix with Fillers Content.

Samples	Load (kN)	Deformation(mm)
1	0.705	1.87563
2	0.828	1.67844
3	0.816	3.03781

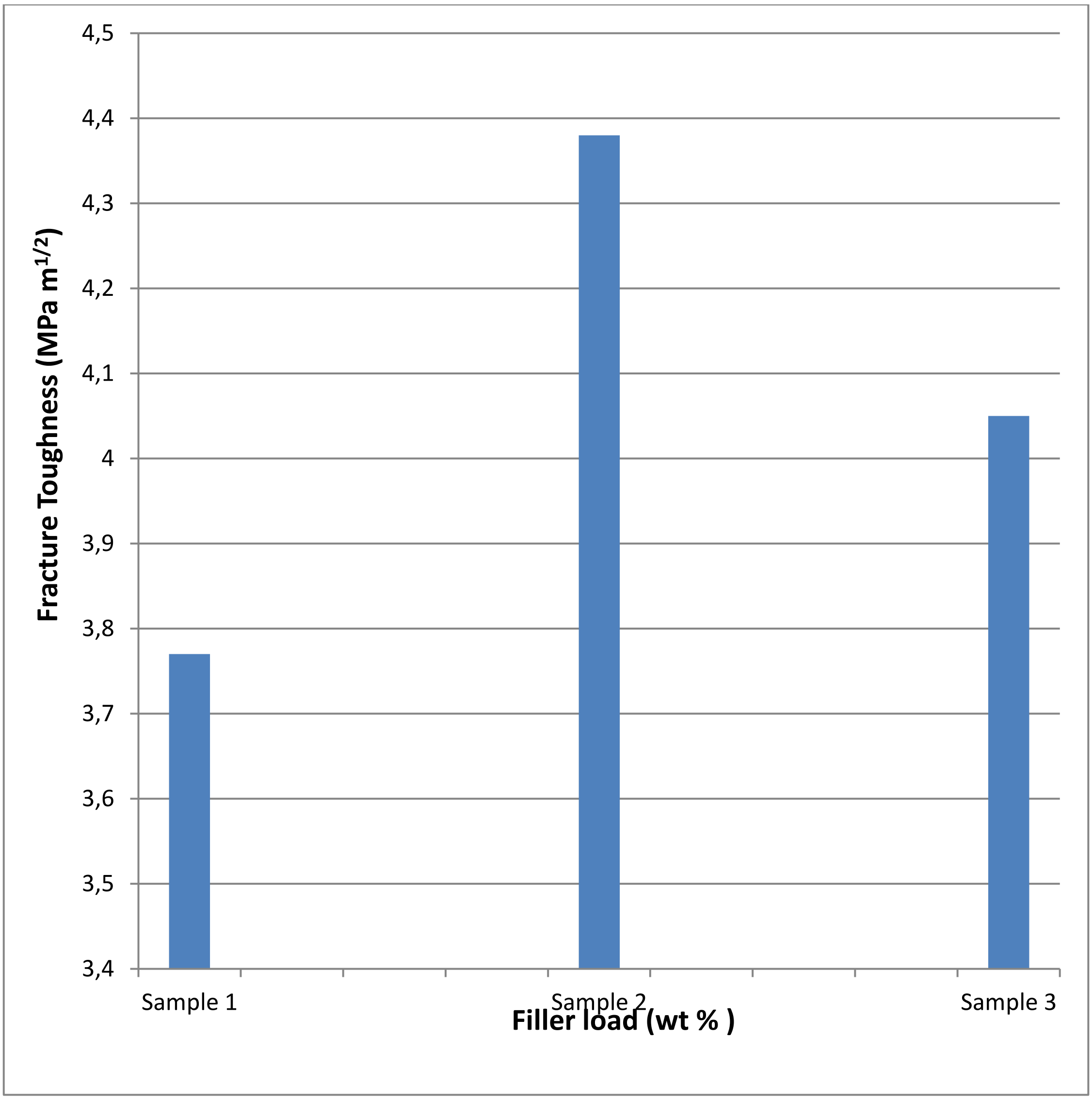

Figure 1. Effect of Fillers Content (wt%) on The Fracture Toughness (K_{IC}) of the Epoxy Matrix

Single Edge Notch Bending

In " Table 3 and Figure 2 " the maximum load and KIC of nanocomposites are greater than that of pure epoxy. There is no big difference in the maximum load and KIC between nanocomposites with 1% wt and 5% wt. This is attributed to the same reasons mention in the C.T section. These results are similar to the results of (Naous W, et al) [10].

Table 3. Typical Load-Deformation for Stable Crack Propagation in SENB Samples of the Epoxy Matrix with Fillers Content

Samples	Load (kN)	Deformation(mm)
Sample 1	0.211	0.71375
Sample 2	0.301	1.08375
Sample 3	0.274	0.79688

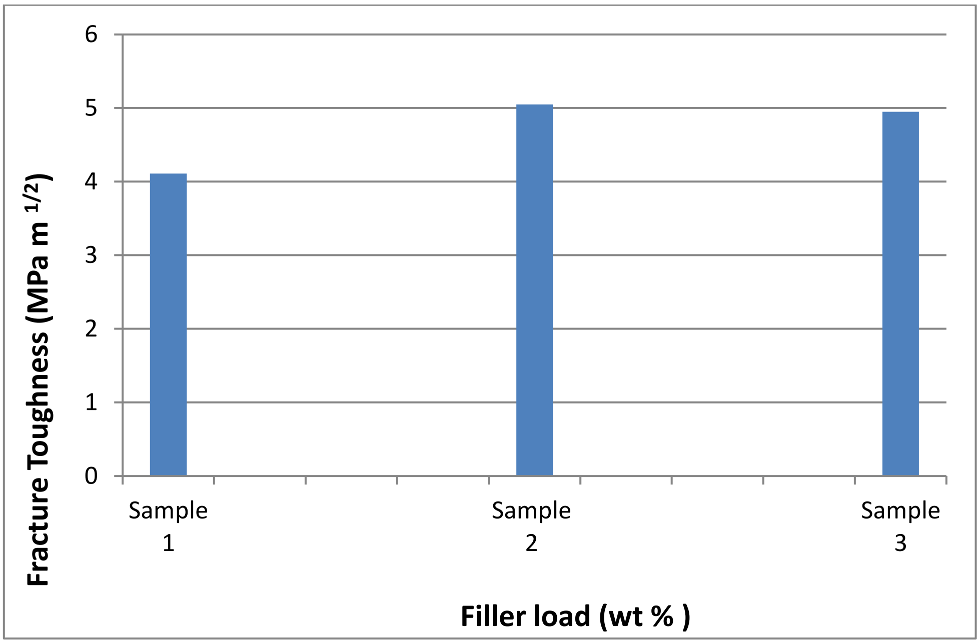

Figure 2. Effect of Fillers Content (wt%) on the Fracture Toughness (K_{IC}) of The Epoxy Matrix.

Results of Antibacterial Test

The antibacterial test was carried for EP (as control sample), EP/1% ZnO NPs and EP/5%ZnO NPs samples. It was used two types of bacteria during the test including E. coli and S. aureus. In " Figures 3 and 4" the results show that EP, EP/1% ZnO NPs and EP/5% ZnO NPs are more influence by the growth of E. coil than on the growth of S. aureus. So that, a bigger inhibition zone (IZ) (mm) of EP, EP/1% ZnO NPs and EP/5% ZnO NPs are observed in the E. coil media than in the S. aureus media. The lowest inhibition zone is seen for EP samples in both the E. coil media and S. aureus media. In contrast, the EP/1% ZnO NPs sample displays the highest inhibition zone in both the E. coil media and S. aureus media.

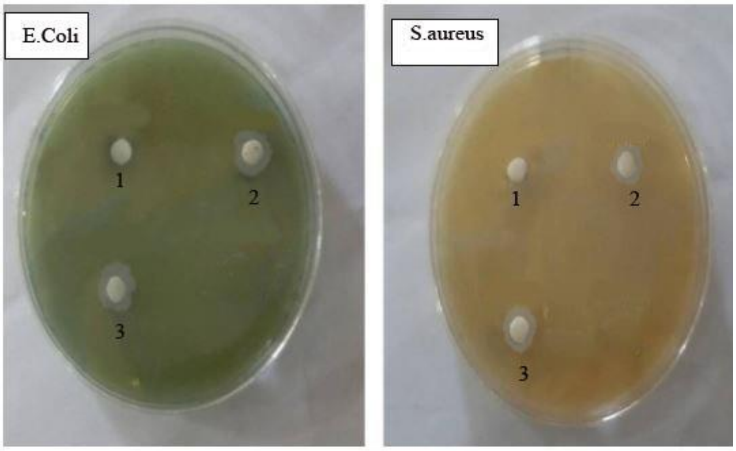

Figure 3. Inhibition Zone Diameter (mm) for (1) EP, (2) EP/1% ZnO NPs and (3) EP/5%ZnO NPs.

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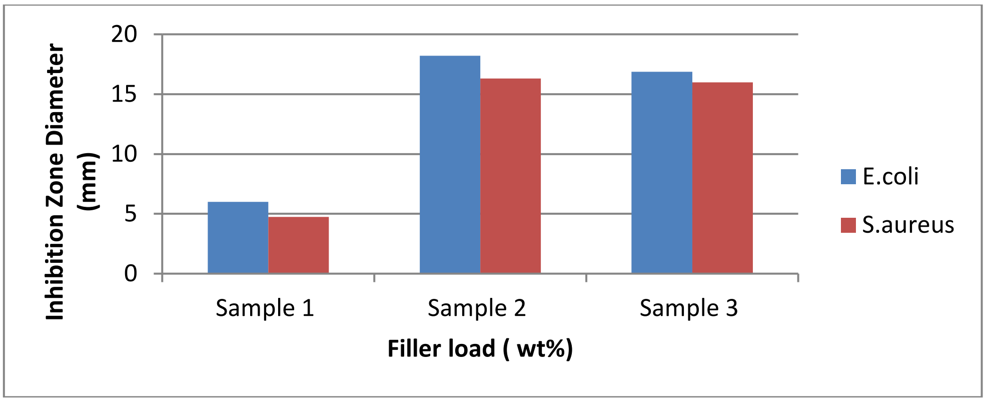

Figure 4. Antibacterial Activity for EP, EP/1% ZnO NPs and EP/5% ZnO NPs.

Atomic Force Microscopy (AFM)

The surface morphology of EP/Filler was analyzed using an atomic force microscope. " Figures 5" show a typical three-dimensional AFM image for the four types of the prepared composites. We notice through these pictures in " Figures 5" that the surfaces of the pure EP membranes are composed of clusters and that they have smooth surfaces, and that this roughness is slowly decreasing with the addition of nanoparticles and that the grain size also decreasing with this addition. From " Table 4" summarizes the results of roughness average and grain size for the surface of the prepared samples. Note that average surface roughness (Sa) is decreased after adding nanoparticles from 18 nm (epoxy pure) to 2.781nm [1% ZnO]. The changes in size thickness are most likely to blame for the shift in surface topography. The thickness variations might become due to the impact of nanoparticles. By mechanical interlocking, increasing the surface roughness of nanoparticles may lead to close interfacial adhesion between nanoparticles and matrix. We also note that the grain size decreases when adding nanoparticles to 1% ZnO and hybrid. This corresponds to the good mechanical properties of the prepared material and the findings of the researchers [11].

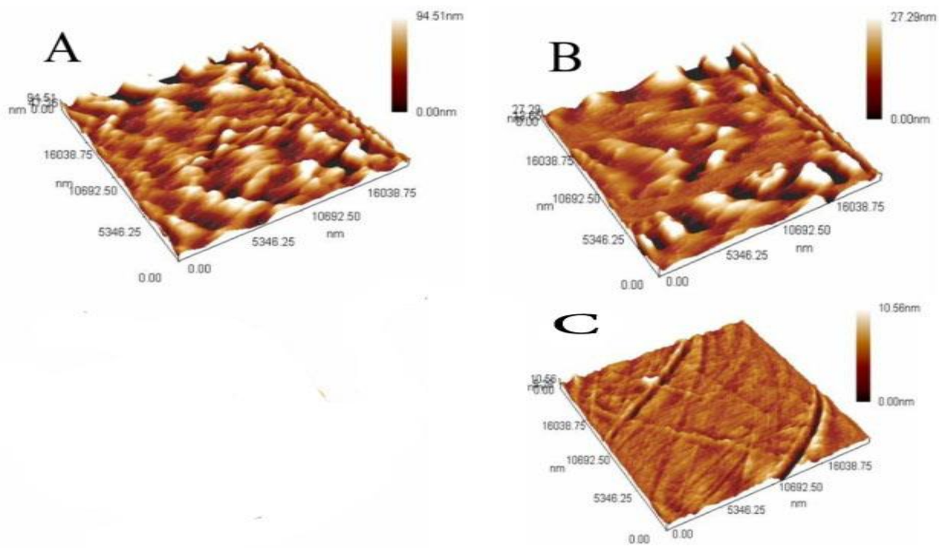

Figure 5. 3D images from AFM of (A) EP pure. (B) 1% ZnO. (C) EP/5% ZnO.

Table 4. Grain size and roughness average of the samples.

Samples No.	Grain size (nm)	Roughness Average(nm)
Sample 1	1320.51	18
Sample 2	1280.60	2.781
Sample 3	1272.67	4.32

FTIR Results

FTIR technique used to study the change in the chemical structure of composites because it provides comprehensive information and rich experimental data that helps the development and progress of research. The FTIR spectra of pure EP, EP/1 wt% ZnO NPs, and EP/5 wt% ZnO are shown in “ Figures 6 and 7”.In these spectra, the hydroxyl group (O-H) was found at (3425.2 cm-1). The bonds, which appeared in the (2924 cm-1) belong to (C-H) group. The two bonds observed in (1612 and 1458 cm-1) refer to the (C=C) and (C-C) groups respectively. The bond at (1087 cm^{-1}) belongs to the (C-O) group. Also, it is noticed that the presence of a band at a wavenumber of (2368 cm^{-1}) is for (Nanoparticles), which meaning that there is a physical interaction between the active surface of Nanoparticles and the polar matrix of EP polymer. The existence of a nitrile group in the acrylonitrile phase causes this polarity. EP is, in fact, a terpolymer made up of three components: acrylonitrile, styrene, and butadiene.

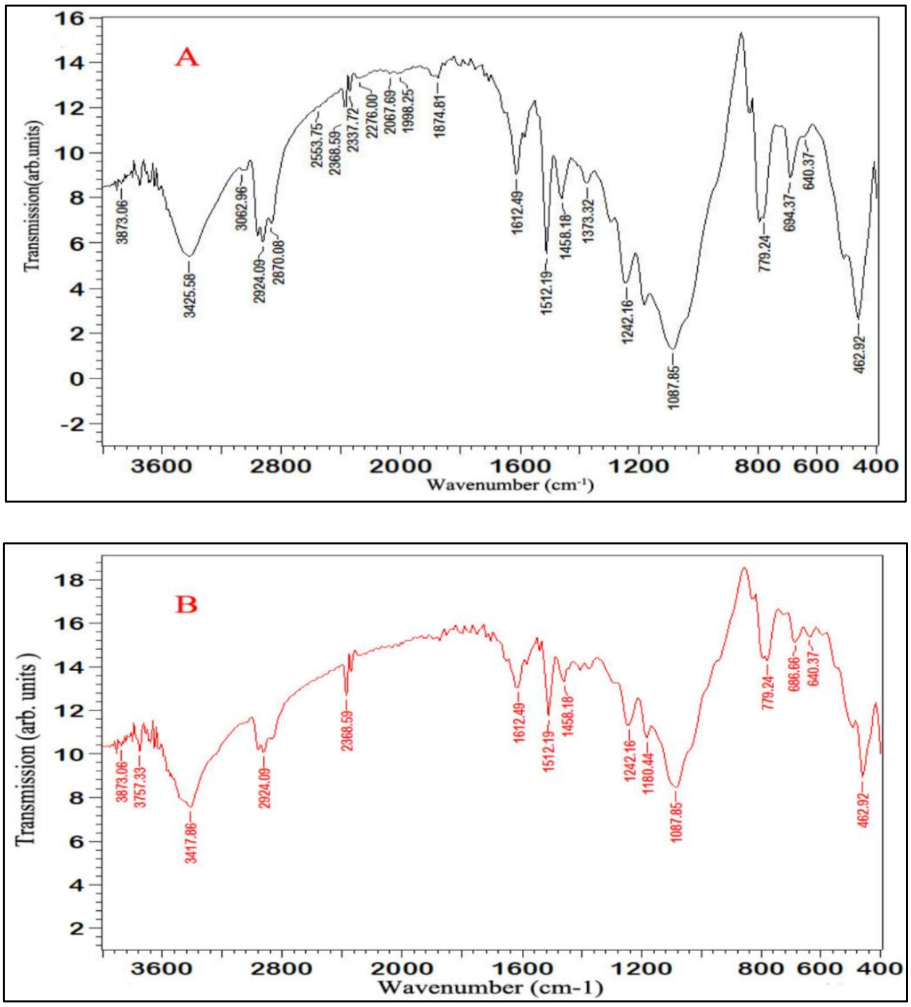

Figure 6. FTIR Spectra of A: Sample 1, B: Sample 2

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" Figures 6, 7" and " Table 5" show the FTIR spectrum of the polymeric mixture, where we note the presence of all the peaks and bonds for all components of the mixture and this agrees with the experimental results well and confirms the occurrence of a physical reaction as a result of a slight shift in the number of wavelengths while the shape of the spectrum remains the same. The spectral region focused on 400 and 3600 cm^{-1} which contained more appropriate information on the chief chemical functional groups.

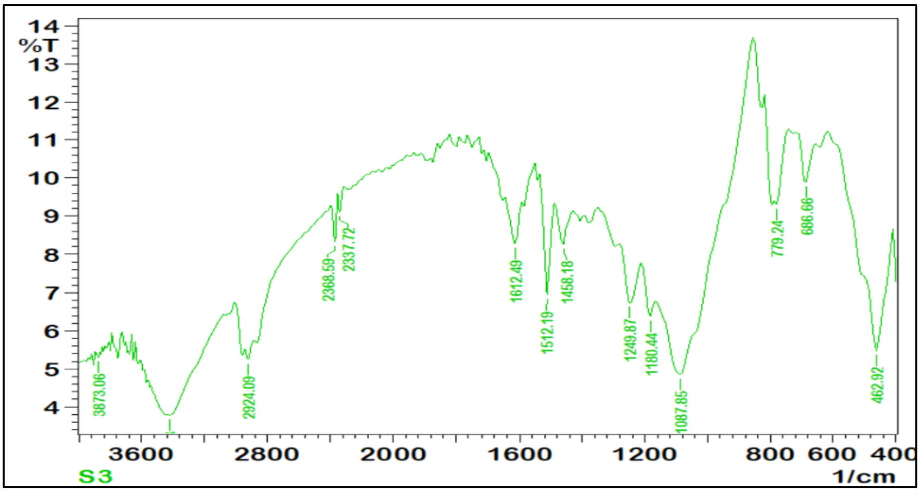

Figure 7. FTIR Spectra of: Sample 3.

Table 5. Wavenumber Assignments of FTIR Spectra of Epoxy Resin.

Wavenumber (Cm^{-1})	Assignments
3390	(O-H)
2924	(CH_2)
2860	(O-H)
2075	(-C≡C-)
1647.5	(C=O)
1153	(-C-O-C)
863	(=C-H)

Conclusion

The ZnO, filler was successfully incorporated into the epoxy matrix using a mechanical mixing process. The dispersion of nanoparticles (ZnO) was enhanced significantly using the ultrasonic. 1%wt of ZnO gives a better dispersion in the epoxy and increases the intercalation effect. The nanocomposites with 1 %wt. (ZnO) show the highest maximum load and KIC as compared to pure epoxy. FT-IR results show physical interaction between nanoparticles and epoxy matrix, and there is no chemical reaction between them. In the AFM test, the surfaces of the pure EP membranes are composed of clusters and that they have smooth surfaces, and that this roughness is slowly decreasing with the addition of nanoparticles, and that the grain size also decreasing with this addition. The increase of the nanoparticles' surface roughness could lead to the intimate interfacial adhesion between nanoparticles and matrix by mechanical interlocking. While the addition [1% (ZnO)] displays perfect anti-bacterial properties. We suggest some recommendations for future studies; (1) Using other nanoparticles such as SiO_2 or Al_2O_3; (2) Using another polymer matrix like thermoset polyurethane.; (3) Using low concentration of NP less than 1%; (4) More test like mechanical tests (tensile and bending); (5) Study the effect of nanoparticles on wear or corrosion.

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