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RESEARCH ARTICLE

CHARPY IMPACT TEST FOR SiO2 (NANO-MICRO) PARTICLES/EPOXY COMPOSITES

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ABSTRACT

Epoxy with different mix ratio of volume fractions 1, 3, 5, 7, and 10% of SiO_2 nanoparticles and microparticles were used to prepare epoxy nanocomposites and microcomposites in order to examine and study their Impact strength. The molding method was used to prepare epoxy and their nanocomposites and microcomposites specimens using shearing mixer, ultrasonic homogenizer and vacuum system. Charpy impact test was used to test epoxy and their nanocomposites and microcomposites.

Results obtained from charpy impact test shows that Impact strength of EP/SiO_2 nanocomposites was increased with increasing volume fractions of SiO_2 nanoparticles in random manner. It was noticed the Impact strength reach to maximum values (20.11 KJ/m^2) at 3% vol. of SiO_2 nanoparticles, then decrease with increasing volume fraction of SiO_2 nanoparticles, but the results of Impact strength still higher than that of neat epoxy. Brittle fracture still appears at specimen surface obtained for EP/SiO_2 nanocomposites. Also the results showed that the specimen of EP/SiO_2 microcomposites reach to maximum values (6.74 KJ/m^2) at 3% vol. of SiO_2 microparticles than the other specimens, then Impact strength values decrease with increasing volume fraction of SiO_2 microparticles.

In comparison the composites with silica nanoparticles exhibits higher mechanical properties than the composites containing micro size silica particles. However, the fatigue life is generally better for nanocomposites than for micorcomposites. However, the Impact strength is generally better for nanocomposites than for micorcomposites.

INTRODUCTION

Nanotechnology refers to the structure and properties of materials and devices at the nanometer level. Developments in synthesis and characterization methods have resulted in materials that are designed from the ‘‘bottom up”, rather than from the ‘‘top down”. These terms were first used by the famous physicist Richard Feynman [1]. Nanotechnology is directed towards the formation of various nanomaterials such as nanocomposites, nanofiber, nanoparticulate fillers and nanoporous [2].

The use of organic or inorganic filler has become ubiquitous in polymeric systems, Polymer composites are manufactured commercially for many diverse applications such as sporting goods, aerospace components, automobiles, etc. In the last 20 years, there has been a strong emphasis on the development of polymeric nanocomposites where at least one of the dimensions of the filler material is of the order of a nanometer. The final product does not have to be in nanoscale, but can be micro- or macroscopic in size [3].

Polymer nanocomposite shows unique properties, combining the advantages of the inorganic nanofillers (e.g. rigidity and thermal stability) and the organic polymers (e.g. flexibility, dielectric, ductility, and process ability) [4-6]. The inorganic nanofillers (nanoscale building blocks) include nanotubes, layered silicates (e.g. montmorillonite and saponite), nanoparticles of metals (e.g. Au and Ag), metal oxides (e.g.TiO_2 and Al_2O_3), semiconductors (e.g. PbS and CdS), and so forth, among which SiO_2 is viewed as being very important inorganic nanofillers. Adefining feature of polymer nanocomposites is that the small size of the fillers leads to a dramatic increase in interfacial area as compared with traditional composites. This interfacial area creates a significant volume fraction of interfacial polymer with properties different from the bulk polymer even at low loadings [5]. In general, these nanofillers have sizes in the range of 1~100 nm, and they are well-dispersed in matrix materials to create nanocomposites, even at very low concentrations, can strongly change the macroscopic properties of the polymer [7].

Polymer nanocomposites show major improvements in mechanical properties, gas barrier properties, thermal stability, fire retardancy, and other areas [8-10]. There are many factors that affect the polymer nanocomposite properties; synthesis methods, polymer nanocomposite morphology, types of nanoparticles and Polymer matrix such as crystalli- nity, molecular weight, polymer chemistry, and whether thermoplastic or thermosetting [4].

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MATERIALS AND METHODS

Raw Materials

Raw materials used in this experimental work are listed below:

Matrix Materials

Epoxy as a matrix (Nitofill, EPLV with Nitofill EPLV hardener from Fosroc Company) was used to prepare composites. The weight ratio of the epoxy resin to the hardener was 3:1 and gelling time 40 minutes at 35°C, mixed viscosity 1.0 poise at 35C°. The properties of epoxy resin are given in Table (1).

Table 1 Properties of epoxy at 35°C

Property	*Epoxy (EP)*
Density (gm/cm^3)	1.04
Tensile strength (N/mm^2)	26.0
Compressive strength(N/mm^2)	93.0
Flexural strength(N/mm^2)	63.0
Young modulus in Compression (GPa)	10

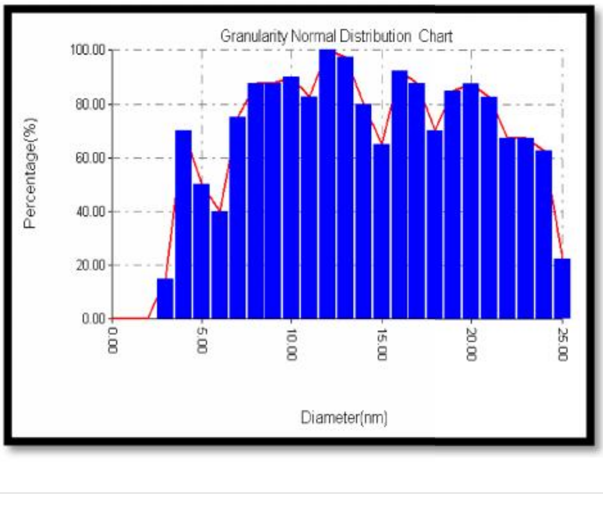
Reinforcing Particles

The reinforcing particles used to improve the mechanical properties of epoxies and FRPs were:

1. SiO_2 nanoparticles (Aerosil-200) from Aerosil pharma (hydrophilic silica) with average diameter of 12nm, a specific surface area of 200±25m2/g. Atomic force microscopy (AFM) was used (CSPM scanning probe microscope) to measured the average particles size of SiO2 nanoparticales, the particles size distribution is shown in Figure (1).
2. SiO_2 microparticles (Mallinckrodt 2847) by Cambridge lab. with mean diameter of 100µm. Some properties of the reinforcing particulate are given in Table (2).

Table 2 properties of the particulate materials properties

Particulate Materials	**Density g/cm3**	**Surface area m2/g**	**Particle Size**	**Purity**
Micro SiO2	2.1	-----	100 µ m	99.99
Nano SiO2	0.05	200± 25	12 nm	99.8



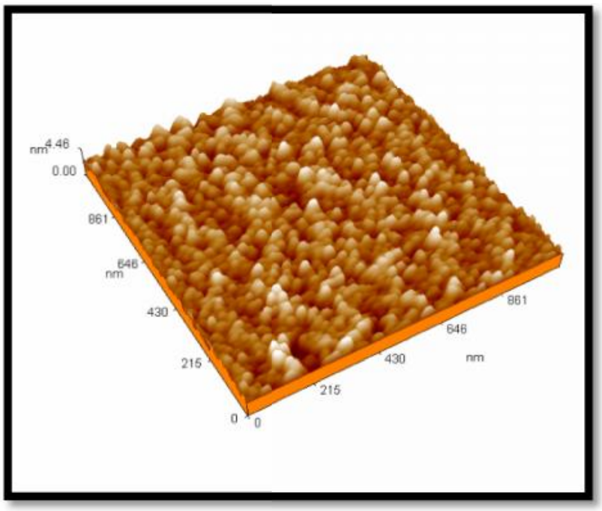


Figure 1 AFM of silica nanoparticles

Preparation Method

Impact test specimens were prepared by hand lay-up technique and mixing process which can be summarized by the following steps:

Preparation of Neat Epoxy

The neat epoxy specimens were prepared by simple direct mixing of epoxy resin with the hardener, epoxy resin and hardener are weighted for suitable mixing ratio by using Electronic balance type (Sartorius BL 210S/Germany) with sensitivity of 10-4g and with rang up to 2Kg, and mixed in container by mechanical stirring for 15 minutes to get good homogeneity between epoxy resin and hardener before casting it as sheets (of dimensions 15x15cm2), by using clean glass mould (cleaned by distilled water to remove the dirt and dust present on the surfaces) were used for casting. The casting was cured at room temperature for 24 hours. Sheets were casted in an oven for 1hour with temperature 50 oC, it was left for 48 hours before pulling out from molds and then left at room temperature for 7 days before processing further and kept then in vacuum chambers.

Preparation of Epoxy Nano/Microcomposites

Nanocomposites with different volume fraction (1, 3, 5, 7 and10%) of nano SiO_2 particles and reinforced with two, four and six layers of the two types of E-glass fibers(CM and WR) were prepared by molding method, in this study they chose reinforcing with six layers due to better obtained results than in reinforcement with two and four layers. Since properties of composite depend upon the volume fraction of reinforcement, mass for a given volume fraction of SiO_2 was determined from the volumes of individual components matrix and SiO2 fillers by using:

$$V_f + V_m = 1 \qquad \ldots\ldots\ldots\ldots(3-1)$$

$$V_f = W_f / \left[W_f + W_m \left(\frac{\rho_f}{\rho_m} \right) \right] \qquad \ldots\ldots(3-2)$$

Where,
V f is the volume fraction of SiO2 fillers,
Vm is the volume fraction of epoxy matrix,
Wf & ρ_f is the mass and density of fillers for the prepared composite,

Wm & ρ_m is the mass and density of epoxy matrix for the prepared composite.

The nanocomposites were prepared in more complicated method such that; The nanoparticles were preheated at 120℃ for 2 h in order to eliminate possible absorbed water on their surface,to prepare homogenous mixture of epoxy and the nanoparticles, a volume fraction percent of nanoSiO_2 particles weighted with electronic balance of four digits type (Sartorius H51) then mixed with epoxy resin, the mixture put inside a container, an oil bath was used to heat up the mixture to desired (75℃) temperature so the viscosity of epoxy base is reduced. Proper mechanical stirring (2h) at this stage resulted better dispersion of nanoparticles, then the mixture was cooled to room temperature after that the hardener was added to the formulation being mixed by mechanical stirring (10 minutes). Sheets were left at room temperature for 24hours for curing, and was cast in an oven for 1 hour at 50 oC for post curing, it was left for 48 hours before pulling out from molds and left for 7 days before any test to get better curing conditions and kept then in vacuum chambers.

Impact Test sample cutting

The sheets of the nanocomposites are cutting into specimens, by using a circular iron saw, pluses from the samples were removed by using the iron rasp, the samples were polished by using abrasive emery papers of grade 400.The shape and dimension of the samples cut for impact test according to [ISO-179 TYPE D] shown at Figure (2) and Figure (3).

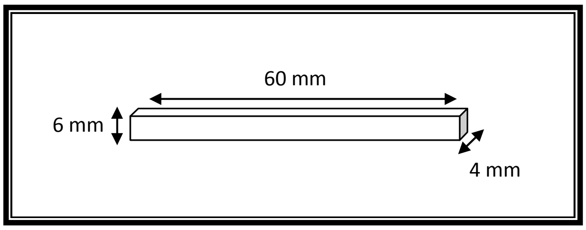
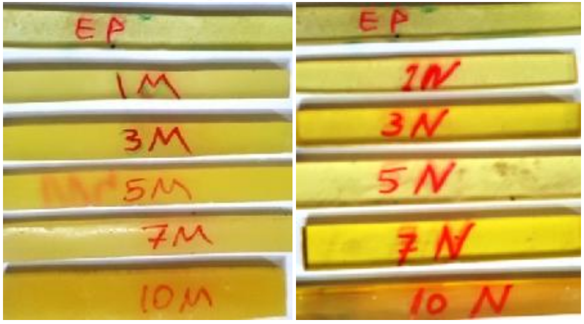


Figure 2 Dimensions of Impact Test Specimens. [11]
[ISO-179 TYPE D]



A B

Fig 3 Final epoxy and epoxy nano/microcomposite specimens prepared for Impact Test
(a) Pure epoxy and EP/SiO2 microcomposites.
(b) Pure epoxy and EP/SiO2 nanocomposites.

Characterisation Techniques

Charpy Impact Test

This instrument consists mainly of pendulum and energy gauge. Charpy impact test consists of standard test piece that would be broken with one flow of a swinging hammer. The test piece is supported at both its ends in a way that the hammer strikes it at the middle.

The testing method of this instrument includes lifting of the pendulum to its maximum height and fixing it firmly. The specimen is fixed in its pertaining place, and then the energy gauge is initialized (on zero position), after that, the pendulum is freed whereas its potential energy would be changed to kinetic energy. Some of this kinetic energy is utilized to fracture the specimen, while the energy gauge reads the value of fracture energy (Uc) for the sample under test. Impact strength (I.S) is calculated by applying the relationship:[12]

I.S =Uc/A(KJ/m^2**)**
Where
Uc: is the fracture energy (Joule) which is determined from charpy impact test instrument.
A: is the cross-sectional area of the specimen.

RESULTS

Impact Strength of Composites

The results of Impact strengths of EP/SiO_2 microcomposites in Table (3) showed that Impact strength has a random behaviour with increasing volume fraction of SiO_2 microoparticles of EP/SiO_2 microcomposites.

The results show a decrease of Impact strengths with increasing the SiO_2 microparticles volume fraction at 1% vol. of SiO_2 microparticles, this behaviour is due to existence SiO_2 microparticles in epoxy matrix causing an increase of the immobility of epoxy chains, then the Impact strength increases with increasing volume fraction of SiO_2 microparticles until the Impact strength reach to maximum values (6.74 KJ/m^2) at 3% vol. of SiO_2 microparticles.

Where the impact strength values decrease with increasing the volume fraction of SiO_2 microparticles. This behaviour in microcomposites is due to low or missing interfacial strength (adhesion), where microparticles have adhesion with epoxy chains less than the adhesion of SiO_2 nanoparticles with epoxy chains due to the characteristic of microparticles surface (low porosity) where almost the interactions of epoxy with the surface of microparticles are very week.

Also microparticles size increase the space distance between epoxy chains which leads to increasing of bad bonding between epoxy chains, these reasons lead to decrease of flexural strength of EP/SiO_2 microcomposites, figure (4) shows the variation of Impact strengths of EP/SiO_2 microcomposites vs. SiO_2 microparticles volume fraction.

Table 3 Impact Strength of epoxy and EP/ SiO_2 micrcomposites

Sample	**Impact Strength (I.S), KJ/m^2**
EP	3.30
EP/ 1% microSiO_2	2.38
EP/ 3% microSiO_2	6.74
EP/ 5% microSiO_2	6.73
EP/ 7% microSiO_2	3.91
EP/ 10% microSiO_2	2.97

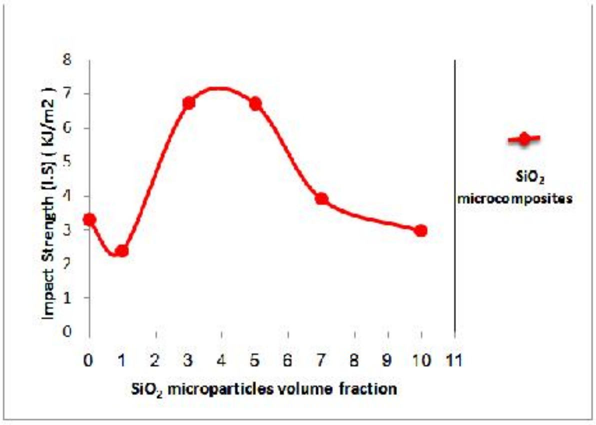


Fig. 4 *Charpy impact strength vs. the volume fraction of epoxy and EP/SiO_2 microcomposites.*

The results of Impact strengths of EP/SiO_2 nanocomposites in Table (4) showed that Impact strength has a random behaviour with increasing volume fraction of SiO_2 nanoparticles of EP/SiO_2 nanocomposites. It was noticed the Impact strength reach to maximum values (20.11 KJ/m^2) at 3% vol. of SiO_2 nanoparticles, then decrease with increasing volume fraction of SiO_2 nanoparticles. Increases Impact strength due to Increase of crack deflections (creating more than one crack propagation direction) which means crack propagation resistance and increase of chains supporting resulting from nanoparticles existence.

The behaviour of decreasing Impact strength due to nanoparticles agglomerations lead to increase the space distance (free volume space) between epoxy chains, so, increasing of free volume space which leads epoxy chains to bear lesser-forces. In general all the results obtained after the addition of SiO_2 nanoparticles to epoxy were higher than that of epoxy specifically at low volume fraction of SiO_2 nanoparticles. The increase of volume fraction of SiO_2 nanoparticles leads to increase of constraint of epoxy chains where chains deflections and mobility will be reduced, so all the results of Impact strength were higher than that of epoxy, figure (5) show the variation of Impact strengths of EP/SiO_2 nanocomposites vs. SiO_2 nanoparticles volume fraction.

Table 4 Impact Strength of epoxy and EP/ SiO_2 nanocomposites

Sample	**Impact Strength (I.S), KJ/m^2**
EP	3.30
EP/ 1% nanoSiO_2	2.97
EP/ 3% nanoSiO_2	20.11
EP/ 5% nanoSiO_2	5.26
EP/ 7% microSiO_2	10.72
EP/ 10% nanoSiO_2	5.25

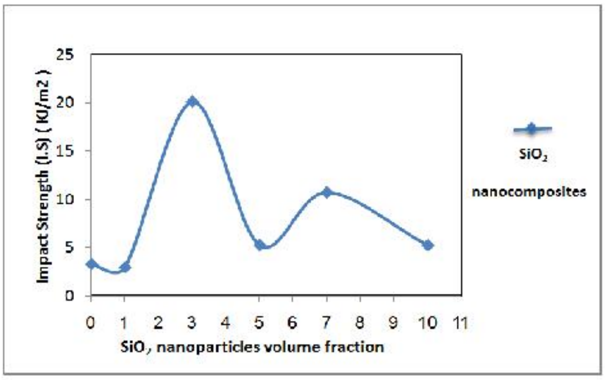


Fig. 5 Charpy impact strength vs. the volume fraction of epoxy and EP/SiO_2 nanocomposites**.**

The results of Impact strength of EP/SiO_2 nanocomposites in Figure (6) show the variation of Impact strengths of EP/SiO_2 nanocomposites vs. SiO_2 nanoparticles volume fraction compared with the Impact strengths of EP/SiO_2 microcomposites. The results of Impact strength have most probably high values at low volume fraction of SiO_2 nanoparticles, specifically at 3% vol. At higher addition of SiO_2 nanoparticles, Impact strength of EP/SiO_2 nanocomposites decrease with increasing volume fraction of SiO_2 nanoparticles but the results of Impact strengths still higher than that of epoxy.

In comparison the composites with silica nanoparticles exhibits higher mechanical properties than the composites containing micro size silica particles, The results indicated that the Impact strength stress of the micro composites specimens is lower than the nanocomposites specimens at low volume fraction. However, the Impact strengths is generally better for nanocomposites than for micorcomposites.

The Charpy Impact test for specimens of EP/SiO_2 micro/nanocomposites with 1, 3, 5, 7and 10% volume fraction showed that all specimens with SiO_2 micro/nanoparticles suffer brittle fracture during the test course as shown in Figures (7) and (8).

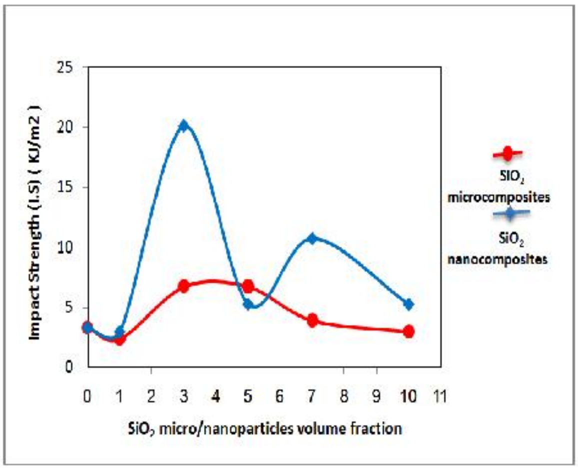


Fig 6 Charpy impact strength vs. the volume fraction of epoxy and EP/SiO_2 nanocomposites and microcomposites.

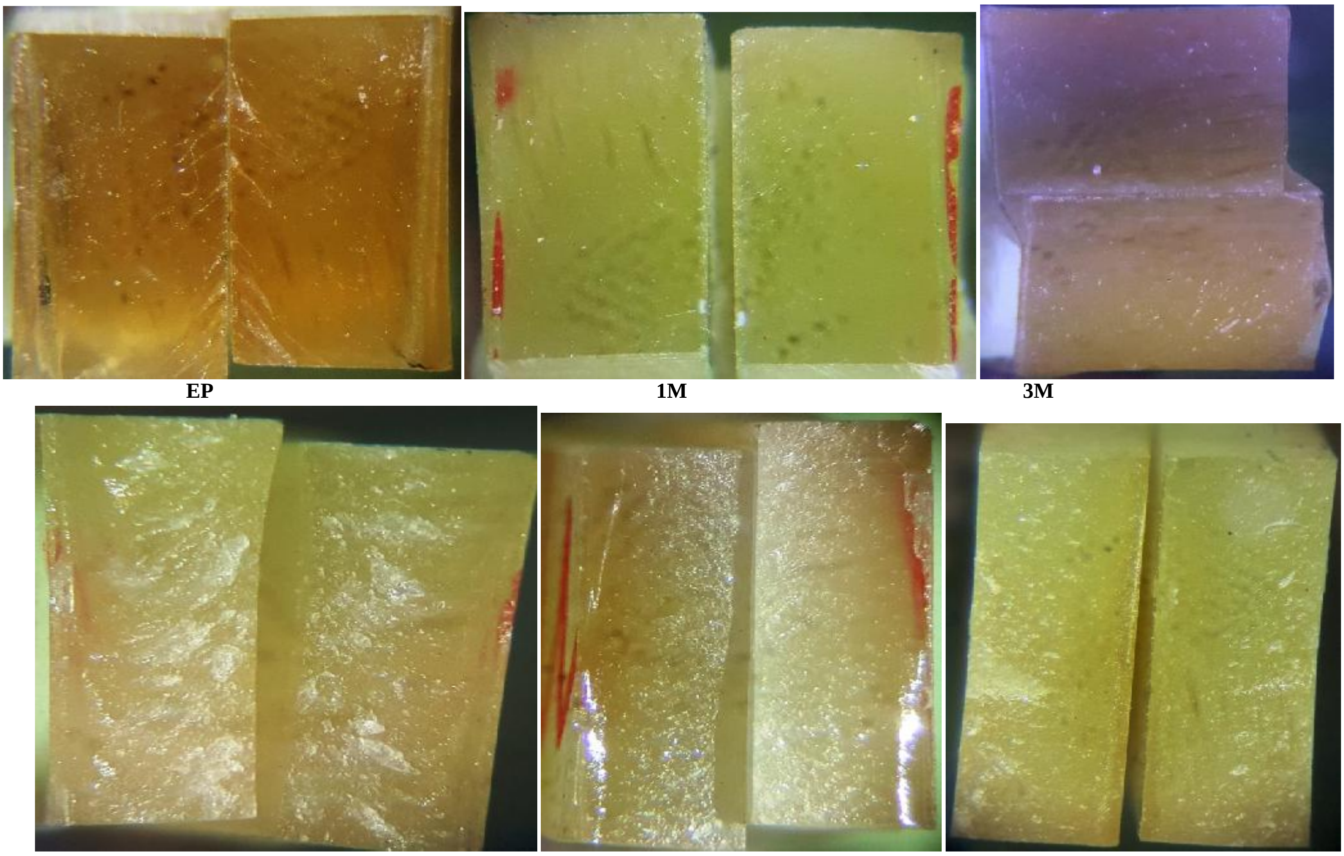


Figure 7 Photographs under optical microscope under 40 X magnification for specimens of EP and EP/ SiO_2 microcomposites with (1, 3, 5, 7and 10%) vol . fraction after test.

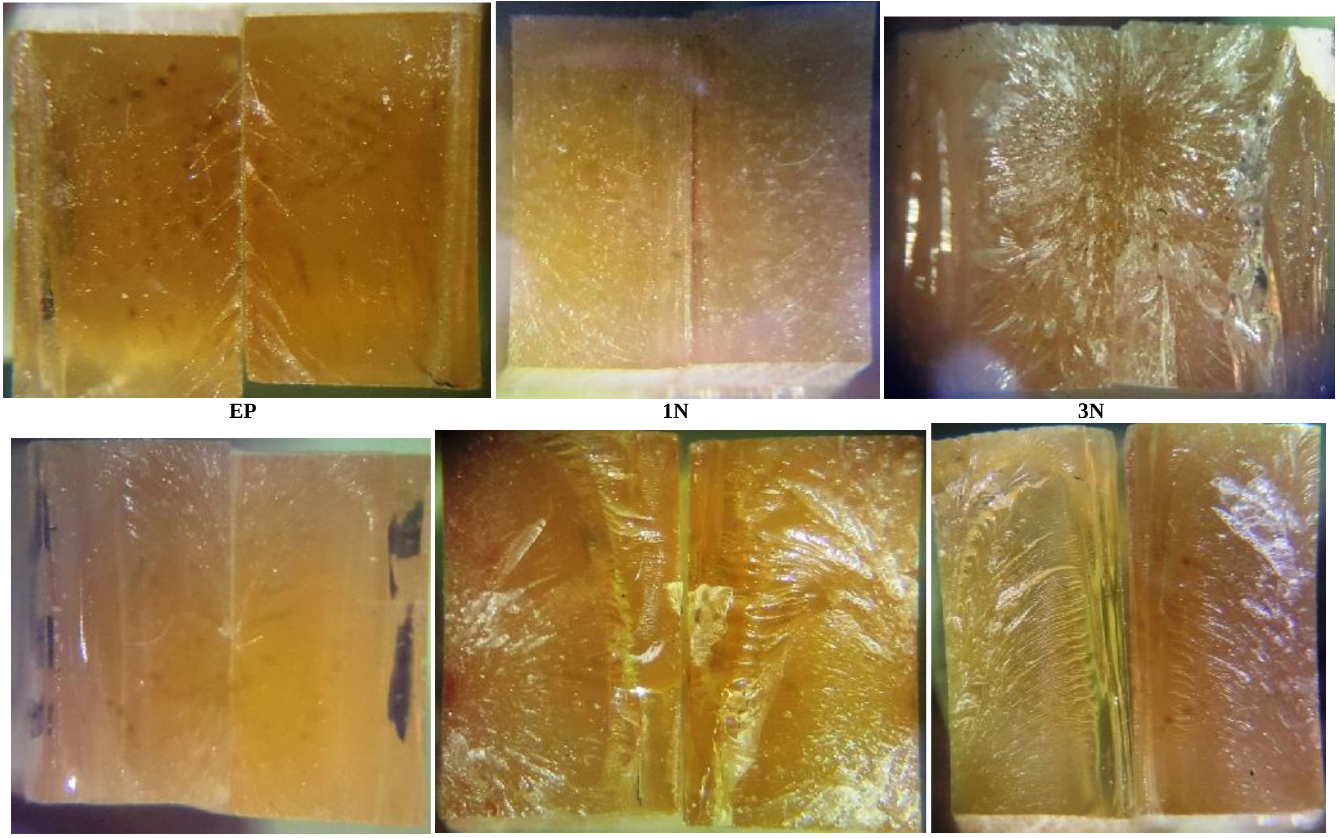


Figure 8 Photographs under optical microscope under 40 X magnification for specimens of EP and EP/ SiO_2 nanocomposites with (1, 3, 5, 7and 10%) vol . fraction after test

CONCLUSION

1. SiO_2 nanoparticles improve Impact strength of epoxy matrix in EP/SiO_2 nanocomposites.
2. Impact strength of EP/SiO_2 microcomposites decrease with increasing volume fraction of SiO_2 microparticles at high concentration (> 3% vol. fraction).
3. Impact strength of EP/ SiO_2 nanocomposites increase with increasing volume fraction of SiO_2 nanoparticles in the epoxy matrix of EP/ SiO_2 nanocomposites.

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