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MWCNT REINFORCED COMMERCIAL EPOXY RESIN COATING FOR ROBUST HYDROPHOBIC COATING

Atharv Themdeo[1], Aniket Kalambe[1], Shilpa Pande[2]

[1]*(Department of Chemical Engineering, Laxminarayan Institute of Technology, RTMNU, Nagpur, India)*

[2]*(Department of Physics, Laxminarayan Institute of Technology, RTMNU, Nagpur, India)*

ABSTRACT: Robust superhydrophobic coating was prepared by reinforcing bisphenol A diglycidyl ether (BADGE) epoxy resin with functionalized multi-walled carbon nanotubes (MWCNT). 0.1, 0.3 and 0.5 % of functionalized MWCNT in epoxy resin were coated on stainless steel. Functionalized MWCNTs improved the interfacial bonding between the nanotubes and epoxy resin. Field Emission Scanning Electron Microscope (FESEM) images confirmed the superior dispersion of MWCNT in the epoxy matrix. The dispersion of functionalized MWCNT reinforced epoxy composite was studied by X-ray diffraction. Coated panel were kept in water for the period of 3 and 7 days for fouling studies. The study of hydrophobicity and changes after keeping panels in water did by using contact angle test. The effect of percentage of functionalized MWCNT in epoxy resin on hydrophobicity of coating was discussed in detail.

KEYWORDS: Contact angle, Epoxy resin, Functionalized multi walled carbon nanotubes, Superhydrophobic surface.

1. INTRODUCTION

Polymer-based superhydrophobic surfaces and coatings have recently attracted a tremendous amount of attention with extraordinary water repellency. The Superhydrophobic surface has been used to describe a surface that exhibits contact angles of greater than 150° [1]. This kind of surfaces have attracted tremendous attention due to their broad applications in scientific and industrial applications, such as self-cleaning [2-5], anti-fouling and anti-scaling [6-8], anti-icing [9, 10], oil-water separation [11, 12], drag reduction [13, 14], medical applications, transparency and anti- reflection [14] andothers.

Epoxy resin is one of the best choices for designing superhydrophobic coating due to its versatility and practical characteristics such as robust physical, chemical, and mechanical properties, safety, heat absorbance, high bonding strength and low costs [15-16]. Good compatibility of epoxy resin with hard as well as soft surfaces gives high versatility for the applications in advanced functional materials [17].

A large number of studies have been done to design superhydrophobic surface by using graphene. The carbon nanotubes in coating gives structural integrity as well as superhydrophobicity even after exposure to extreme thermal stresses and have excellent thermal and electrical properties [18]. MWCNT composite film provides a superhydrophobic coating with high contact angle values and high adhesion force for stainless steel [19]. In the present study hydrophobic nanoparticles (MWCNT) and epoxy resin was used to prepare the superhydrophobic film with high bondingstrength.

2. EXPERIMENTAL SECTION

2.1 Material

Epoxy resin and polyamide (curing agent) were procured from Resin and plastic LMT, Mumbai. Surfactant that was, Triton X 100 were obtained from Central Scientific India, Nagpur.

2.2 Functionalization of MWCNT

200 ml of hydrogen peroxide and 300mg of MWCNT are added to a flat bottom flask fitted with a condenser. The flask is heated on a hot plate with continuous magnetic stirrer at 65^0C. This process was continued for 48 hours. After 48 hours of continuous process MWCNT solution is filtered by filter paper and later it is annealed for 12 hours at 60^0C. 125mg of functionalized and annealed MWCNT powder is added to 25ml of water in a glass beaker and 3 drops of Triton X 100 (non-ionic surfactant) was added to the mixture. This mixture was placed in ice bath and sonicated using probe sonicator for 90 min. After this procedure the functionalized CNT is homogeneously dispersed.

2.3 Superhydrophobic coatingpreparation

Functionalized MWCNT, dispersed in water, was added to epoxy resin in different proportion to make MWCNT dispersed hydrophobic epoxy coating. For the preparation of 0.1% MWCNT containing sample 10g of epoxy resin was dissolved in 3g of tetrahydrofuran and with 0.06g of MWCNT dispersed solution. Polyamine used as curing agent, is added to the mixture as 30% by weight of epoxy resin. This whole mixture is sonicated for 20 minutes. After sonication of this mixture stainless steel plate is dipped and then removed and dried for 3 hours at 100^0C. Thus, the film obtained is 0.1% MWCNT incorporated epoxy film and designated as EC-1. 0.3 % and 0.5 % MWCNT incorporated epoxy films were made in the same way and designated as EC-3 and EC-5respectively.

3. CHARACTERIZATION

All three different samples that are 0.1%, 0.3% and 0.5% of MWCNT in epoxy resin are dipped into distilled water for 3 days and for 7 days. Contact angle is calculated for all six samples. This is basically to see the effect of water on prepared coating.The prepared pure epoxy resin and its MWCNT-doped composites were structurally characterized via X-ray diffraction (XRD) uisng Philips powder X-ray diffractometer (Model: PW1710) in the 2θ rangefrom10◦to80◦.TheFouriertransforminfrared(FTIR)spectrawererecordedusingaPerkin-Elmerspectrum

100. FTIR spectrometer in the range of 400– 4000 cm−1 with the KBr pellets of the samples. Then the pure epoxy resin and its MWCNT composites were morphologically analyzed using a scanning electron microscope (SEM) .

4. RESULTS AND DISCUSSION

The nanocomposite film samples have been made with an aim to design hydrophobic coating in a simple way without taking help of speciality polymers. The films are prepared and characterized to understand the effect of various MWCNT content in the film and also the distribution of MWCNT in the film and theirstatus.

4.1 XRD

The XRD pattern in Figure1 of functionalized MWCNTs indicates a sharp diffraction peak at 25^0 in sample EC-3, 27.57^0 in sample EC-5, which reveals the crystalline nature of the f-MWCNTs. This peak is not observed in sample EC-1 because of the very less amount of MWCNT in sample. The XRD pattern of the epoxy matrix figure 1 represents a broad peak at 2θ value of 43.24^0 and 50.57^0 in EC-1, 43.13^0 in EC-3 and 43.13^0 and 50.35^0 in EC-5. Neat epoxy exhibits broad peaks at 2θ values around 43^0 and 50^0 due to the scattering from cross linked network of epoxy, which indicates the amorphous nature of epoxy. Nevertheless, the functionalized MWCNT/epoxy nanocomposites exhibit peaks corresponding to the epoxy and functionalized MWCNT. The intensity of the peak increases with increasing wt% of functionalized MWCNT, and a slight shifting in peak position is also observed.

The simultaneous presence of epoxy and functionalized MWCNT characteristic peaks in the nanocomposite gives the evidence of the formation of epoxy and functionalized MWCNT nanocomposites [20,21].

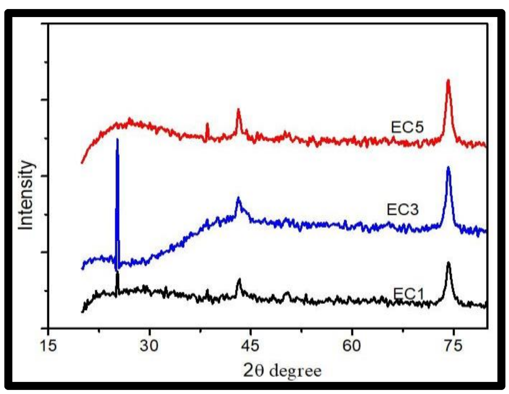

Fig 1: X-ray diffraction of EC-1, EC-3 and EC-5.

4.2 FTIR

The FTIR analysis of the nanocomposite is carried out to realize the nature of interactions between functional groups present in the constituting components, i.e., epoxy matrix and functionalized MWCNT. From the figure 2 FTIR spectrum of functionalized MWCNT and epoxy resin respectively. The generation of carboxylic acid (–COOH) groups is identified on the surface of the oxidized MWCNTs Vibrational modes observed at around 3400 cm^{-1} in all three samples are attributed to –OH stretching which does not exist in unfunctionalized MWCNTs. Peak around 2900cm^{-1} is observed in all three samples are corresponds to the C–H stretching. The presence of the characteristic band (–O–H stretching) in the FTIR spectrum of neat epoxy gives an indication of polar functional groups such as (– O–H) in the structure of neat epoxy. The peaks at 1650 and 1450 cm^{-1}, which are observed in all three samples, correspond to the amides. The peak observed at 1509 cm^{-1} in EC-5 spectra can be assigned to the bending mode of N–H [22, 23]. FTIR was used to monitor the presence of the epoxide group indicated by the presence of an absorption band in the 910-920 cm^{-1} range, related to the contraction of the C-C bond and the stretching of both C-O bonds of the epoxy ring. As can be seen for all samples shown in Fig2.

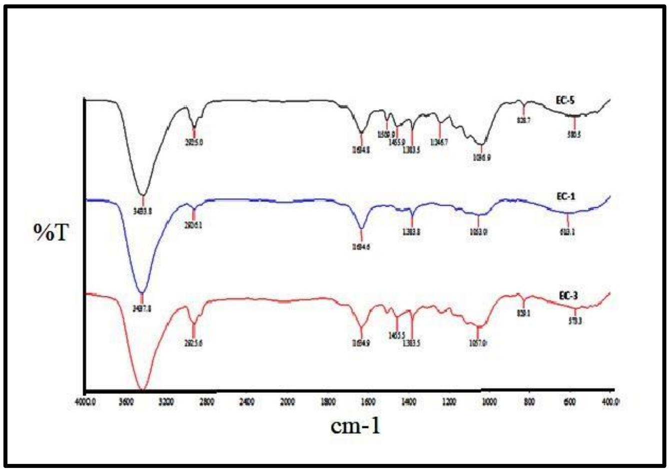

Fig 2 : FTIR spectra of MWCNT/epoxy nanocomposites (EC-1, EC-3 and EC-5)

4.3 SEM

Figure 3,This peak has almost disappeared after curing, which is an evidence of a high degree of cross linking in the nanocomposites and in the neat epoxy. Similar results may be found in the literature [24]. The chemical modification of MWCNTs improves the effective interfacial interaction between chemically functionalized MWCNTs and the epoxy matrix. From the FTIR spectra of epoxy and MWCNT nanocomposites, the presence of hydrogen bonded hydroxyl groups at the interface of functionalized nanotubes and epoxy matrix is noticed. Beak broadening must be there.

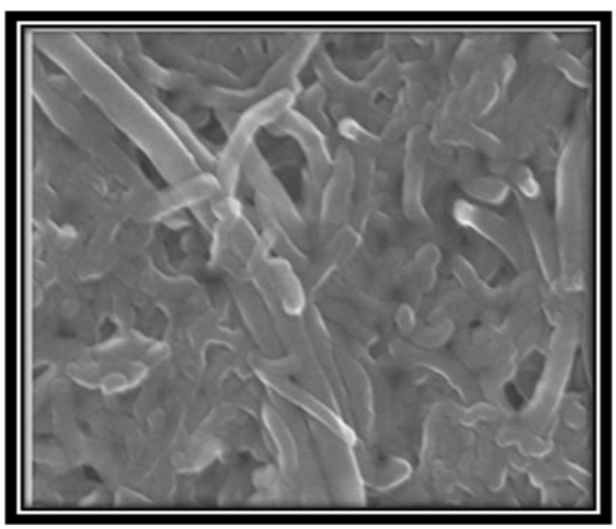

Fig 3: Scanning electron microscopy of Functionalized MWCNT.

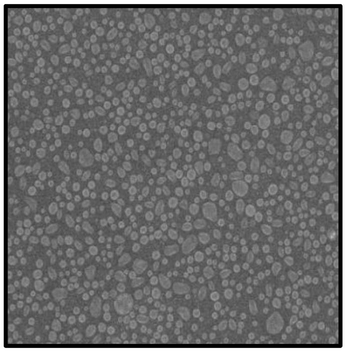
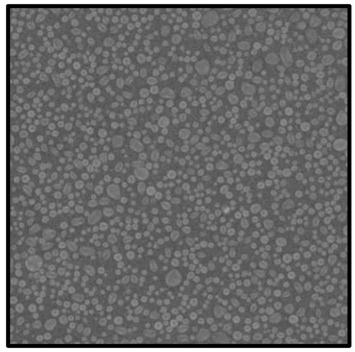
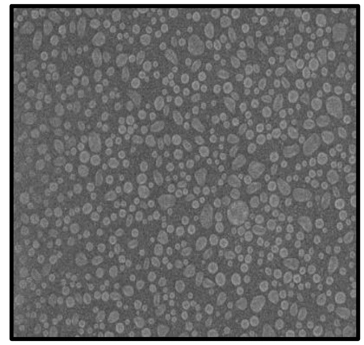

Fig 4,5,6: Scanning electron micrograph of Epoxy resin/MWCNT nanocomposite

The SEM image of MWCNTs shown in Figure 3 clearly showed that MWCNTs were not gathered as crowd together because this MWCNT functionalized. This proves that the method of Functionalization and method of dispersion are accurate. Selection of surfactant that is Triton X 100 also proved to be correct. The SEM image of coating on stainless steel shown in Figure 4, 5and 6 indicates that MWCNT is dispersed properly and film formed uniformly.

4.4 Contact Angle

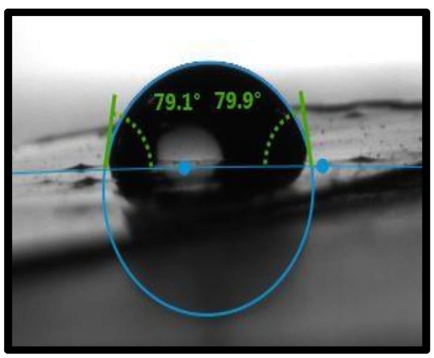

Fig 7**:** Contact angle measurement of 0.1% MWCNT in Epoxy resin(EC-1).

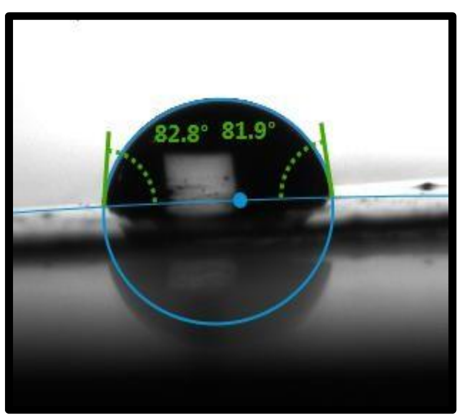

Fig 8**:** Contact angle measurement of 0.3% MWCNT in Epoxy resin(EC-3).

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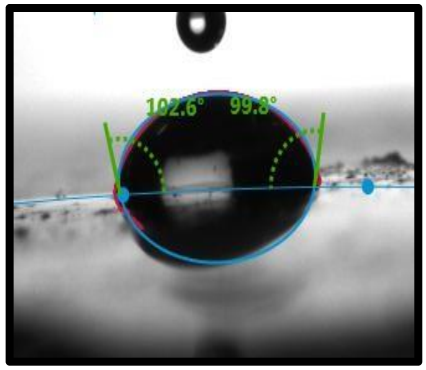

Fig 9: Contact angle measurement of 0.5%MWCNT in Epoxy resin (EC-5).

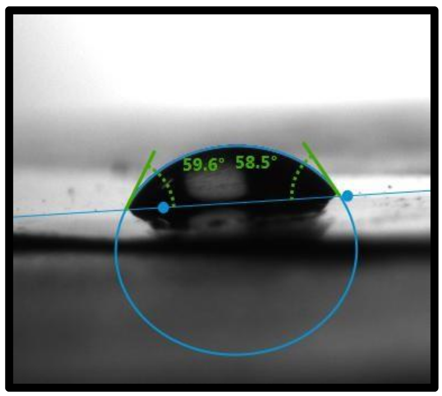

Fig 10: Contact angle measurement of pure Epoxy resin (EC).

As shown in figure 10 contact angle of pure epoxy resin film are 59^0. Figure 7, 8 and 9 shows the contact angles 79^0, 82^0, 102^0 containing 0.1%, 0.3% and 0.5% of MWCNT in epoxy resin respectively. Above results shows that contact angle increases with increasing %MWCNT in epoxyresin.

5. CONCLUSION

FTIR results proved that, Epoxy and MWCNT nanocomposites, the presence of hydrogen bonded hydroxyl groups at the interface of functionalized nanotubes and epoxy matrix is noticed. That indicates, Epoxy resin is properly cured and MWCNT is also equally distributed in epoxy resin. SEM image of the pure MWCNT proves that it is functionalized and dispersed as perrequirement.

XRD results depicts that the simultaneous presence of epoxy and functionalized MWCNT characteristic peaks in the nanocomposite gives the evidence of the formation of epoxy and functionalized MWCNT nanocomposites. A result of contact angle proves that contact angle increases with increasing %MWCNT in epoxy resin. If we increase
%MWCNT in epoxy resin more that did in this project it can make the coating superhydrophobic.

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