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THE EFFECT OF AMOUNT OF GRAPHENE FOR THE STUDY OF BARRIER PROPERTIES OF EPOXY-GRAPHENE NANO COMPOSITES

SANGEETA KULKARNI

Department of Chemistry,Govt. First Grade College
Kumareshwar Nagar, Dharwad, Karnataka 580008, INDIA

ABSTRACT
The vapor barrier film of epoxy graphene nanocomposites of 0.75 and1.5wt% was synthesized by dispersion method.This dispersion of graphene mainly attributes to the excellent water barrier property. When epoxy combined with graphene nanoparticles show significantly increased toughness and dampness capacity of epoxy system.This is mainly due to free volume reduction respect to the pristine polymer. The nanocomposites derived from graphene show excellent electrical, thermal and mechanical properties. The excellent electron transport, high surface area and mechanical properties of graphene epoxy nanocomposites has potential applications in food packaging, anticorrosive coating. The synthesized barrier film of epoxy graphene composites were characterized by differential scanning calorimetric techniques (DSC). The barrier properties were investigated by using electrochemical impedance spectroscopy (EIS).

Key words : Epoxy nanocomposites, Barrier properties, Graphene nanoparticles

INTRODUCTION

The development of novel polymer nanocomposites with the unique features such as high aspect ratio, excellent toughness and strength due to the functionalities such as inorganic nanoparticles such as graphene into polymer matrices, such as epoxy system is one of the most promising research fields in various areas of application [1–4]. The loading of graphene has significantly increased electrical and thermal properties and the mechanical properties of nanocomposites.[5-6]. However, with the aim of increasing the barrier properties nanofiller such as graphene used in these materials have strong tendency to agglomerate which would cause in homogeneous dispersion of nanofillers in matrices, and reduction in free volume of thesenanocomposites [7].In this study, the effect of graphene on barrier properties of epoxy system has beenevaluatedby incorporating reduced concentrations of nanoparticles (i.e., 0.75 and 1.5% by weight) into waterborne epoxy resin.Impregnation of epoxy with graphene filter cake without pre-mixing, extraordinary properties of nanocompositesare exceptionally high surface to volume ratio of the nanofiller and/or its exceptionally high aspect ratio.

*** Corresponding Author:**
sangeeta_bk1234[at]yahoo[dot]com

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MATERIALS AND METHODS

Graphene nano particles used in this work are made of few layers with width of less than 2 µM, an average thickness of about 2 nm and a surface area 500 m^2/g. The epoxy system is a commercial water borne resin without corrosion inhibitors. It contains additives such as emulsifying and dispersing agents and surfactants mainly 63% weight of solid $TiO_{2.}$ [8] (Table 1)

Table 1:Specimen names of different epoxy graphene nanocompostes

Plain epoxy system	EPX
Loading with 0.75% wt. of graphene	Epgo/EG 0.75
Loading with containing 1.5% wt of graphene	Epgo/EG 1.5

In order to evaluate the effect of the nano filler on the epoxy matrix properties, a thermal analysis was carried out taking 10 mg of the sample. Three scans (heating, cooling and heating again) were carried out from 30-250 ^{0}C with a heating rate of 10 ^{0}C per minute for each specimen.

The barrier properties of the modified epoxy systemwere investigated by using Electrochemical Impedance Spectroscopy (EIS).A conventional electrochemical cell was used including a saturated calomel reference electrode (SCE), platinum as counter electrode, and the coated aluminium sample as working electrode [9,10]. A frequency response analyzer (FRA), in conjunction with a potentiostat/galvanostat, 1255 and 1286 Solartron respectively, was employed. The electrolyte used was an air saturated 3.5 wt.% NaCl aqueous solution and the area exposed was of about 5 cm^2. Measurements were carried out at open circuit potential (OCP) over a frequency range from 10^5 to 0.02 Hz with an amplitude sinusoidal voltage of 10 mV up to an immersion time of 21 days.[11,12]

RESULTS AND DISCUSSION

The measured water contact angle (see Table 2) of the unfilled epoxy systemis about 66.3 °; when the nanoparticleis dispersed in the epoxy resin, the contact angle increased to 88.1(Epgo/EG0.75sample) and 102.1(Epgo/EG1.5sample).The increase in the contactangle is mainly due to the increase in amount of graphene nanoparticle which in turn increase the hydrophobicity of the epoxy graphene nanocomposites leading to the reduction of sorption of water with the films. (Table 2)

Table 2 : Water Contact angle of epoxy graphene nanocomposites

Sample	Epx	Epgo/EG 0.75	Epgo/EG 1.5
Water contact angle (degree)	66.6	88.1	102.3

When exposed to the3.5%wt of aerated NaCl aqueous solution the epoxy graphene nanocomposites films show increase in the impedance modulus, when compared to no loaded epoxy system, which indicates better barrier properties. Even after one day exposure to the test solution at low frequency is slightly less than 10^7 Ωcm^2.The increase in impedance modulus at low frequency was mainly due to the formation of local anodicarea for prolonged exposure of nanocomposite surface up to 21days.This was mainly attributedto the sorption of water and electrolyte which in turn shift impedance modulus at low frequency.

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The subsequent shift of phase angle at high frequency as function of immersion time is also constituent with further degradation of nanocomposite films. The increase in phase angle of 90°C with minimum (3degrees) is observed at frequency of about 0.44Hz after exposure of one day to test solution.

The decrease in impedance modulus with the time show better stability of loaded epoxy nanocomposites.The lower value of impedance modulus after one day immersion for neat epoxy system accounts for poor barrier properties when compared to graphene loaded epoxy nanocomposites, which shows continuously slightly increased values of impedance modulus up to 21 days.(Fig 1)

The increase in impedance modulus ($8\times 10^7\ \Omega\ cm^2$), value for EG1.5 as function of immersion time show better barrier properties of epoxy graphene nanocomposites. The compact behavior of epoxy graphene nanocomposites was explained on the basis of no full penetration of the electrolyte in the given time interval.

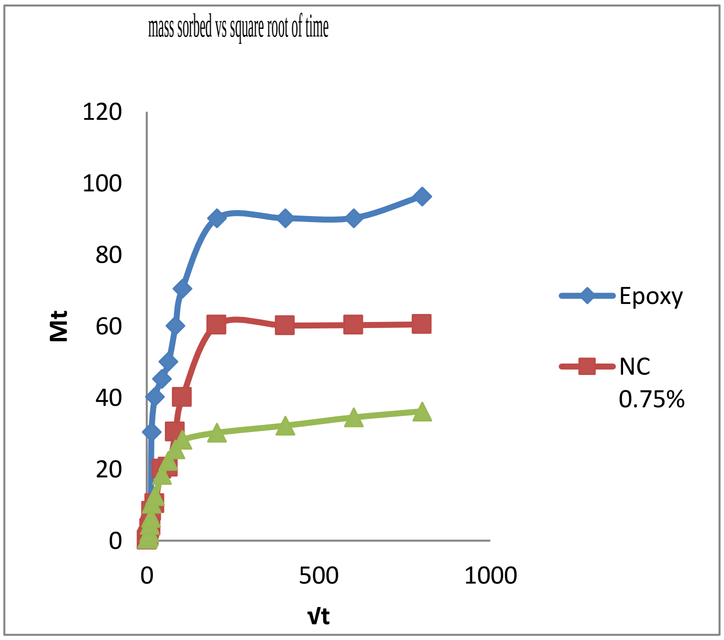

Figure 1 :Mass sorbed vs square root of time

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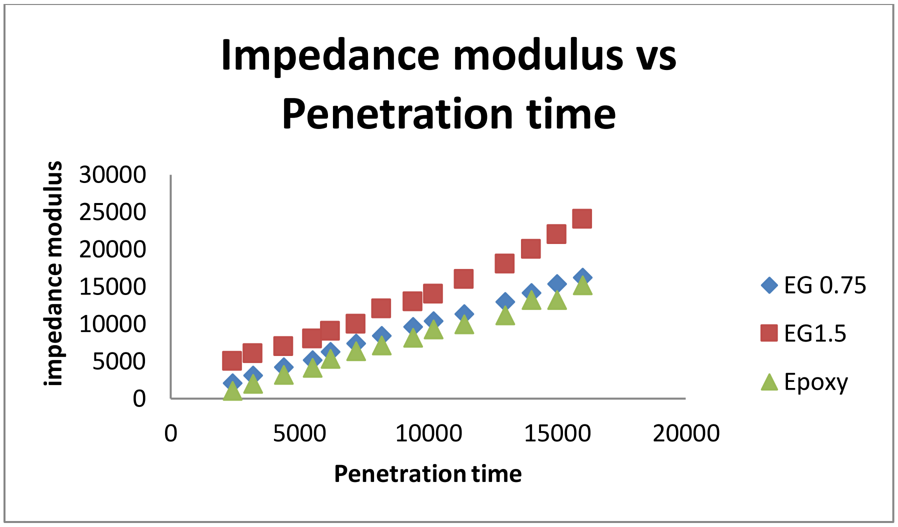

Figure 2 :Impedance modulus vs penetration time

Meaningful impedance related to the effect of graphene on the Barrier properties of the epoxy nanocomposites is evaluated at 0.02 Hz are reported. As can be seen from this figure 2, the filled nanocomposites exhibited impedance modulus greater than the unfilled base epoxy for all exposure times showing the noticeable effect due to the use of graphene. In addition, EG1.5 showed an impedance modulus of one order of magnitude greater than that exhibited by the base epoxy for all exposure times.

The water diffusion coefficients through epoxy nanocomposites are calculated from the initial slope of the curve of mass sorbed vs square root of time these water diffusion coefficients of EG 1.5wt%of graphene to the epoxy system are lower than those exhibited by the EG0.75.

The increased performances of epoxy graphene nanocomposites to the barrier effect were mainly due to graphene which induces decreasedwaterdiffusivity.The effect of amount graphene increasesthe pathway length of water through whichother nanoparticles used as a filler.

After the first 4 days of immersion the nanocomposite,show a slight increase, day by day, to the end of immersion time, reaching a steady value, indicating a different water absorption process.The observed behavior of the nano filled epoxy can be attributed to a diffusion mechanism of water adsorption and transport through the nanocomposites.

CONCLUSION

In this work, the addition of nanoparticles to epoxy system led to a slight increase of the hydrophobic character of thenanocomposites. The collected EIS data showed that loaded coatings exhibit improved barrier propertieswhich was attributed diffusion mechanism of water transport through the filled epoxynanocomposites..The water diffusion coefficients of EG 1.5wt%of graphene to the epoxy system are lower than those exhibited by the EG0.75 which can be attributed to a diffusion mechanism of water adsorption and transport through the nanocomposites.

REFERENCES

1. S. Kumar, S. Raj, S. Jain, and K. Chatterjee, “Multifunctional biodegradable polymer nanocomposite incorporating graphene-silver hybrid for biomedical applications,” *Materials & Design*, vol. 108, pp. 319–332, 2016.

ISSN 2397-5547

2. R. B. Ladani, S. Wu, A. J. Kinloch et al., "Multifunctional properties of epoxy nanocomposites reinforced by aligned nanoscale carbon," *Materials & Design*, vol. 94, pp. 554–564, 2016.

3. C. Wang, Y. Lan, W. Yu, X. Li, Y. Qian, and H. Liu, "Preparation of amino-functionalized graphene oxide/polyimide composite films with improved mechanical, thermal and hydrophobic properties," *Applied Surface Science*, vol. 362, pp. 11–19, 2016.

4. S. Zhao, H. Chang, S. Chen, J. Cui, and Y. Yan, "High-performance and multifunctional epoxy composites filled with epoxide-functionalized graphene," *European Polymer Journal*, vol. 84, pp. 300–312, 2016.

5. H. Kim, Y. Miura, and C. W. Macosko, "Graphene/polyurethane nanocomposites for improved gas barrier and electrical conductivity," *Chemistry of Materials*, vol. 22, no. 11, pp. 3441–3450, 2010.

6. S. H. Song, K. H. Park, B. H. Kim et al., "Enhanced thermal conductivity of epoxy-graphene composites by using non-oxidized graphene flakes with non-covalent functionalization," *Advanced Materials*, vol. 25, no. 5, pp. 732–737, 2013.B. Tang, G. Hu, H. Gao, and L. Hai, "Application of graphene as filler to improve thermal transport property of epoxy resin for thermal interface materials," *International Journal of Heat and Mass Transfer*, vol. 85, pp. 420–429, 2015.

7. T. Monetta, A. Acquesta, V. Maresca et al., "Characterization of aluminum alloys environmentally friendly surface treatments for aircraft and aerospace industry," *Surface and Interface Analysis*, vol. 45, no. 10, pp. 1522–1529, 2013.

8. Y. Wang, J. Yu, W. Dai et al., "Enhanced thermal and electrical properties of epoxy composites reinforced with graphene nanoplatelets," *Polymer Composites*, vol. 36, no. 3, pp. 556–565, 2015.

9. A. S. Wajid, H. S. T. Ahmed, S. Das, F. Irin, A. F. Jankowski, and M. J. Green, "High-performance pristine graphene/epoxy composites with enhanced mechanical and electrical properties," *Macromolecular Materials and Engineering*, vol. 298, no. 3, pp. 339–347, 2013.

10. F. Wang, L. T. Drzal, Y. Qin, and Z. Huang, "Mechanical properties and thermal conductivity of graphene nanoplatelet/epoxy composites," *Journal of Materials Science*, vol. 50, no. 3, pp. 1082–1093, 2015.

11. B. Zhang, R. Asmatulu, S. A. Soltani, L. N. Le, and S. S. A. Kumar, "Mechanical and thermal properties of hierarchical composites enhanced by pristine graphene and graphene oxide nanoinclusions," *Journal of Applied Polymer Science*, vol. 131, no. 19, Article ID 40826, 2014.K.-C. Chang, M.-H. Hsu, H.-I. L

12. T. Monetta, A. Acquesta, and F. Bellucci, "Graphene/epoxy coating as multifunctional material for aircraft structures," *Aerospace*, vol. 2, no. 3, pp. 423–434, 2015.

ISSN 2397-5547