Effect of dispersing agents on the electrical and mechanical performance of GNPs filled epoxy nanocomposite

Wong Wee Chun[1], Teh Pei Leng[1*], Yeoh Cheow Keat[1], Osman, Azlin Fazlina[1], Lim Bee Ying[1], Mohamad Syahmie Mohamad Rasidi[1]

[1]Faculty of Chemical Engineering & Technology, Universiti Malaysia Perlis, Kompleks Pusat Pengajian Jejawi 2, Taman Muhibbah, Jejawi, 02600 Arau, Perlis, Malaysia

ABSTRACT

In this work, graphene nanoplatelets (GNPs) filled epoxy nanocomposites with the addition of different dispersing agents were fabricated using a method combines mechanical mixing and tip sonication. The loading of GNPs used is 0.8 vol% determined previously as the amount required to achieve the percolation threshold to conduct electricity. Three dispersing agents were used in this work: Sodium dodecyl sulphate (SDS), ethanol and Phenyl glycidyl ether (PGE), with loadings varying from 2 vol% to 10 vol%. The incorporation of dispersing agent enhanced the electrical bulk conductivity of GNPs filled nanocomposites. The mechanical performance (flexural properties and fracture toughness) of the nanocomposite were evaluated and compared. The optimum loading of SDS to obtain the highest flexural strength and fracture toughness is 2 vol%, where further increases will deteriorate the performance of nanocomposites. On the other hand, the optimum loading of ethanol and PGE are 4 vol%. The fracture toughness of GNPs filled nanocomposites improved with the addition of 2 vol% SDS and deteriorated with increasing loadings of SDS up to 10 vol%. By incorporating 4 vol% of ethanol, the optimum fracture toughness of the nanocomposite is achieved. Fracture toughness is then dropped with further increases in ethanol. The addition of PGE caused deterioration in fracture toughness of GNPs filled epoxy nanocomposite.

1. INTRODUCTION

Global market and demand for semiconductor are blooming in these recent years. This is due to the increasing applications of electronic devices, especially in military, automotive, telecommunication, aerospace, medical, etc. Chips shortage during the covid season expressed the importance of the semiconductor industry to the world. Nowadays, the semiconductor industry is closely linked to human's daily life and relies heavily on electronic devices. The emergence of nanotechnology leads humankind to a new stage of technology revolution. Rise of small devices with greater properties using nanotechnology is important in various fields, including electronics, biomedicals, sensors, and energy storage. Nanotechnology refers to the engineering of matter at a very small, molecular scale. Nanotechnology involved the use of nanoparticles usually ranging from 1nm to 100 nm in size [1]. The incorporation of the nano-

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scale fillers into a matrix can be done using mechanical mixing, injet printing and solvent casting techniques [2-3] to form a nanocomposite where its properties may strengthen by the nanofillers [4].

Graphene was discovered by Andre Geim and Konstantin Novoselov using a ground-breaking experiment [5] and it became one of the popular "super" materials ever since its discovery. Graphene has gain attentions of research around the world due to its fascinating properties, such as exceptional thermal conductivity (5000 W m^{-1} K^{-1}), high electron mobility at room temperature (250,000 cm^2/Vs), high Young's modulus of 1 TPa and very large surface area (2630 m^2/g) exhibits by graphene which making it as promising fillers that in various applications [6-10].

Electrically conductive adhesives (ECAs) are emerging as the alternative solution to replace traditional solders. ECAs is more suitable to apply on temperature sensitive components where it only requires low temperature compared to traditional soldering. Besides, ECAs usually exhibit greater fatigue resistance compared to solder joints. It is widely used in electronic devices such as mobile phones, touch screens, flexible PCBs, RFID tagging and RFI shielding. They are two main components of ECAs: a) Polymer system and b) conductive filler. A few types of polymers are used in the fabrication of ECAs, such as epoxy, acrylate, urethane, and silicone which are insulators. Conventional conductive fillers incorporated into the insulated polymer matrix that alter its electrical properties are silver, copper, nickel, and graphite. Several studies stated that incorporating carbonaceous filler greatly enhanced the electrical, mechanical properties of the epoxy system. There are several studies on using carbon nanofillers in epoxy system stated that the incorporation of carbonaceous filler greatly enhanced the electrical and mechanical properties of the system [11-13]. However, the performance of the nanocomposite is strongly related to the dispersion state of the nanofillers, which then affects the interaction between fillers and the polymer matrix. Strong interaction will result in good electrical and mechanical properties [14].

Dispersing agent is a type of chemical that is usually used to improve the dispersion of fillers throughout the polymer matrix. Many dispersing agents are used to enhance carbonaceous fillers' dispersion in different polymer systems such as ethanol, Dichlorobenzene, 1-propanol, acetone, THF, and DMF [15-23]. In addition, it should be taken note that residual solvents that are used as dispersing agents which do not completely evaporate will affect the performance of the polymer system [24-25].

2. EXPERIMENTAL DETAILS

2.1. Materials

Graphene nanoplatelets (GNPs) in granular form with a thickness of 11-15 nm were purchased from SkySpring Nanomaterial, Inc. Diglycidyl ether of bisphenol-A (DGEB-A) epoxy resin with the density of 1.16 g/cm^3, and epoxy hardener were both supplied by Euro Chemo Pharma Sdn. Bhd. Sodium dodecyl sulfate (SDS) with a molecular weight of 288.38 g/mol, supplied by Sigma Aldrich (M) Sdn Bhd while 95% concentrated ethanol and phenyl glycidyl ether (PGE) with a molar mass of 150.17 g/mol were supplied by HmbG GmbH and Merck Millipore Sdn Bhd respectively.

2.2.   Sample preparation

A control nanocomposite is the epoxy nanocomposite filled with 0.8 vol% of GNPs, which is determined as the minimum loading of GNPs to achieve the percolation threshold for electrical conductivity. In this work, there are three different dispersing agents: Ethanol (ET), Sodium dodecyl sulfate (SDS), and phenyl glycidyl ether (PGE) with various loadings (2, 4, 6, 8 & 10 vol%) were used in the preparation of epoxy/GNPs nanocomposites to determine the effect of dispersing agent on the final properties of GNPs filled epoxy nanocomposites.

As for the fabrication of GNPs filled epoxy nanocomposites with the addition of ethanol, 0.8 vol% GNPs were added into solvents. The mixture was then added into epoxy resin followed by tip sonication with 30% amplitude for 30 minutes. Afterwards, the mixture was placed into an oven with a temperature of 100 $^{\circ}$C and leave it overnight for solvents evaporation. On the next day, the mixture was taken out and proceed with the addition of hardener. The mixture was then casted on mould, followed by vacuum degassing for 30 minutes and a curing process in the oven at which the temperature was set to 100 $^{\circ}$C for 1 hr. Lastly, cured epoxy/ethanol-GNPs nanocomposite were cut into pieces for the following testing and characterizations. This specimen was abbreviated as ET-GNPs filled nanocomposite. To prepare SDS-GNPs filled nanocomposite, the SDS was first dissolved in distilled water with a 10 vol% ratio to distilled water. Then the pre-weigh 0.8 vol% GNPs was added into the solution and proceeded with same steps and conditions as ET-GNPs filled nanocomposite. In addition, this work used 2 vol% to 10 vol% of PGE as dispersing agent. 0.8 vol% of GNPs were added into PGE, mixed with the epoxy resin, followed by tip sonication with 30% amplitude for 30 minutes. Then the mixture is proceeded with the addition of hardener, casting, vacuum degassing, and oven curing process. Evaporation of PGE is not conducted to investigate the reaction between PGE and epoxy resin.

2.3.   Testing and characterizations

Electrical bulk resistance measurements were conducted using a multimeter at room temperature in direct current mode. In order to measure the electrical bulk resistance, the specimens were drilled into a circular shape with a diameter 20mm with silver coating on both sides. At least five specimens were tested to minimize errors in value measurements. Three-point flexure bending tests were conducted according to ASTM D790 procedure A. This test was conducted using the universal tensile machine model 5569. The crosshead speed was set to 2.38 mm/min with 50 KN applied loads. At least five specimens were tested, and the average value was reported whereas the dimension of the specimen is 60 mm x 12.7 mm x 3 mm. Fracture toughness measurements were tested using universal tensile machine model 5569 according to ASTM E399-78. Single-edge notch was made on the specimens by sawing and sharpening with a razor blade. The single-edge notched specimens were then subjected to static tensile loading with a crosshead speed of 1 mm/min. In this study, graphene nanoplatelets' morphology and dispersion state in epoxy matrices were observed using a scanning electron microscope JEOL JSM-6460LA. Samples were mounted on aluminum stubs and allowed to undergo sputtering coating by using JEOL JFC-1600 Auto Fine Coater. A thin palladium layer of 20 nm was sputter coated on the surface of the samples to avoid electrostatic charge during measurement.

3. RESULTS AND DISCUSSION

3.1.   Electrical bulk conductivity

Figure 1 shows the electrical bulk conductivity of control 0.8 vol% GNPs filled epoxy nanocomposite and nanocomposites with the addition of different dispersing agents. It can be observed that the electrical bulk conductivity of nanocomposites increased with increasing loadings of dispersing agents. By adding 2 vol% of SDS, the electrical bulk conductivity increased significantly from 0.00219 $\Omega^{-1}cm^{-1}$ to 0.00712 $\Omega^{-1}cm^{-1}$. Electrical bulk conductivity of GNPs filled epoxy nanocomposite decreased along with further increases in the loading of SDS from 4 to 10 vol%. On the other hand, the addition of ethanol and PGE up to 4 vol% slightly increased the electrical bulk conductivity of nanocomposites. A similar trend was observed where electrical bulk conductivity deteriorated with further increases in ethanol and PGE loading. SDS-GNPs filled nanocomposites also exhibit higher bulk conductivity compared to other nanocomposites. SDS as anionic surfactant greatly inhibits the re-agglomeration of the GNPs. More random distributed GNPs throughout the epoxy matrix led to more formation of conducting paths which promotes the transportation of the electrons within the composite. However, excessive dispersing agents affect the electrical bulk conductivity of GNPs filled epoxy nanocomposites where re-agglomeration of GNPs occurred which disrupt the transportation of electrons throughout the epoxy matrices. All the values for electrical bulk conductivity of GNPs filled epoxy nanocomposites with and without the addition of dispersing agent were shown in Table 1.
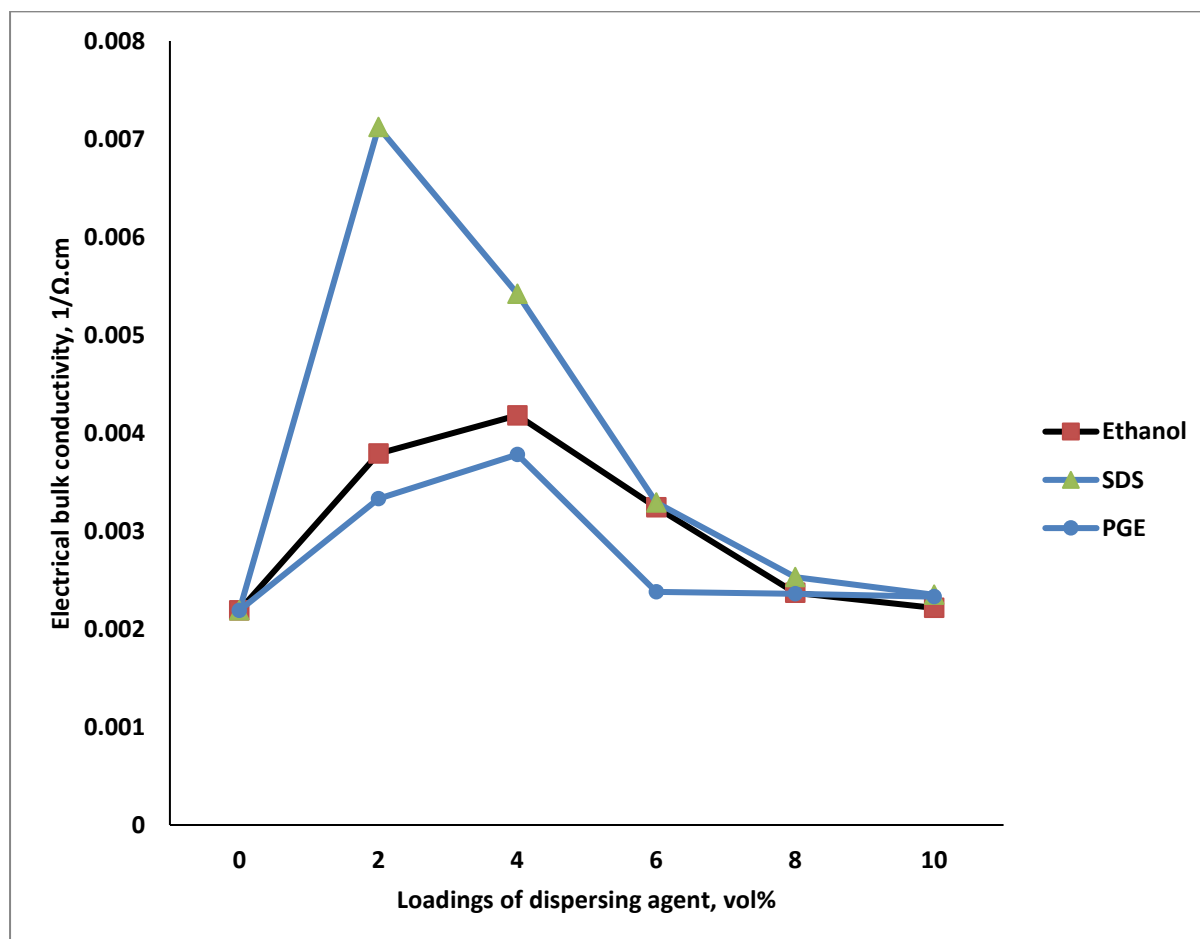

Figure 1: Electrical bulk conductivity of GNPs filled epoxy nanocomposites with the addition of different dispersing agents and loadings.

Table 1: Values of electrical bulk conductivity of GNPs filled epoxy nanocomposites with the addition of different dispersing agents and loadings.

Loadings of dispersing agent, vol%	Electrical bulk conductivity, 1/Ω.cm		
	Ethanol	**SDS**	**PGE**
0		0.00219	
2	0.00379	0.00712	0.00333
4	0.00418	0.00542	0.00378
6	0.00324	0.00329	0.00238
8	0.00237	0.00253	0.00236
10	0.00221	0.00235	0.00233

3.2.   Flexural properties

Flexural strength of epoxy/GNPs nanocomposites upon the incorporation of different dispersants: SDS, ethanol and phenyl glycidyl ether were shown in Figure 2. It can be observed that the flexural strength of the nanocomposites enhanced with the addition of 2 vol% of SDS. The flexural strength of the nanocomposite was increased from 62.33 to 69.83 MPa with a total of 12% increment in value compared to the control nanocomposite. By incorporating 2 vol% of SDS, optimum flexural strength was achieved on GNPs filled epoxy nanocomposite. The dispersion of GNPs was improved within the presence of surfactant, SDS, which led to enhanced flexural strength on the GNPs filled nanocomposite. The strong π-π interactions between the SDS and GNPs introduces a steric repulsion force to overcome the Van der Waals forces between the GNPs, which leads to inhibit the agglomeration of GNPs [26]. Thus, the incorporation of SDS help to achieve better dispersion of GNPs into epoxy resin. Dispersion is the most important factor for the enhancement of the flexural properties of the composites; the flexural strength of the composite materials is enhanced with homogeneous dispersion of the GNPs which helps to transfer applied load from the epoxy matrix to the GNPs reinforcement [27].

The flexural strength of the nanocomposites was reduced from 65.95 to 52.85 MPa with the incorporation of 4 to 10 vol% of SDS. GNPs are easier to agglomerate after the saturation of surfactant throughout the polymer matrix. [28]. The void fraction increases at the higher concentration of SDS which causes poor dispersion of GNPs and eventually deteriorate the flexural strength of GNPs filled epoxy nanocomposite. By incorporating ethanol into GNP filled epoxy system, the flexural strength of the nanocomposite was initially increased from 62.33 MPa to 64.23 and 66.71 MPa with loadings of 2 and 4 vol% respectively. Optimum enhancement in flexural strength was recorded by incorporating 4 vol% of ethanol. Further increases in ethanol loading led to a reduction up to 20% in the flexural strength of nanocomposite. Besides that, the flexural strength of nanocomposites was increased by incorporating 4 vol% of PGE. Both the optimum flexural strength of nanocomposites incorporated with ethanol and PGE occurred upon the addition of 4 vol% loading. This indicated that both ethanol and PGE with optimum content of 4 vol% have the greatest effect on the flexural strength of GNPs filled nanocomposite contributed by good dispersion of GNPs. An excess number of dispersing agent

greater than 4 vol% lead to some degree of re-agglomeration of GNPs which then affect the dispersion of GNPs.

The flexural modulus of GNP filled nanocomposite incorporated with different dispersing agents is shown in Figure 3. Flexural modulus refers to the measurement of the stiffness of a material. Usually, high modulus materials become stiffer and less flexible. The data shows noticeable increases in the flexural modulus of the nanocomposite with 2 vol% of SDS. However, with further addition of SDS, weakening effect on the flexural modulus of nanocomposites were detected where there are 18.5% losses in modulus by incorporating 10 vol% of SDS. Decreasing the flexural modulus is due to the excess amount of SDS forming clusters into the composites instead of dispersing the GNPs [29]. The clusters formed in the composites during the dispersion of GNPs decreased the modulus of the composites. SDS act as a surfactant in the GNPs filled nanocomposite which helps in the dispersion of GNPs within the epoxy matrix. The hydrophilic heads of surfactants interact physically with GNPs through Van der Waals forces and the hydrophobic tails interact with the epoxy matrix. The stronger the interactions, the better will be the surfactant action as an intermediate to help to better the dispersion of GNPs in the epoxy matrix. A similar trend was observed in the flexural modulus of nanocomposite with ethanol where the flexural modulus of nanocomposite initially increased with the addition of 2 vol% of ethanol. Further increase in ethanol loading leads to a reduction in the nanocomposite's flexural modulus.

The optimum flexural modulus of the nanocomposite that was obtained is 2838.48 MPa with the loading of 2 vol% ethanol. However, the flexural modulus of the nanocomposites was reduced to 2589.54 MPa where approximately 8.77% reduction occurred with 10 vol% of ethanol incorporated. Meanwhile, the addition of 4 vol% PGE indicates a greater flexural modulus to the nanocomposite compared to other dispersing agents. The flexural modulus of nanocomposite increased to 3018.89 and 3092.82 MPa with 2 and 4 vol% of PGE loadings respectively. However, the flexural modulus of the nanocomposite was greatly decreased with a further increased in PGE loadings. Excess amount of the dispersing agent added leads to deterioration of the flexural strength and flexural modulus of GNPs filled nanocomposite. This is mainly due to the formation of pores induced by the dispersing agents. In addition, a plasticizing effect caused by the residual solvents contributes to the lower modulus as the loading of dispersing agents increased. All three dispersing agents: ethanol and phenyl glycidyl ether in the liquid form originally; SDS was dissolved in liquid medium prior to being added into the polymer matrix. Solvent tends to act as a plasticizer in the composite and the flexural modulus of the composites decreases when an excess of the solvent is added to the epoxy resin.
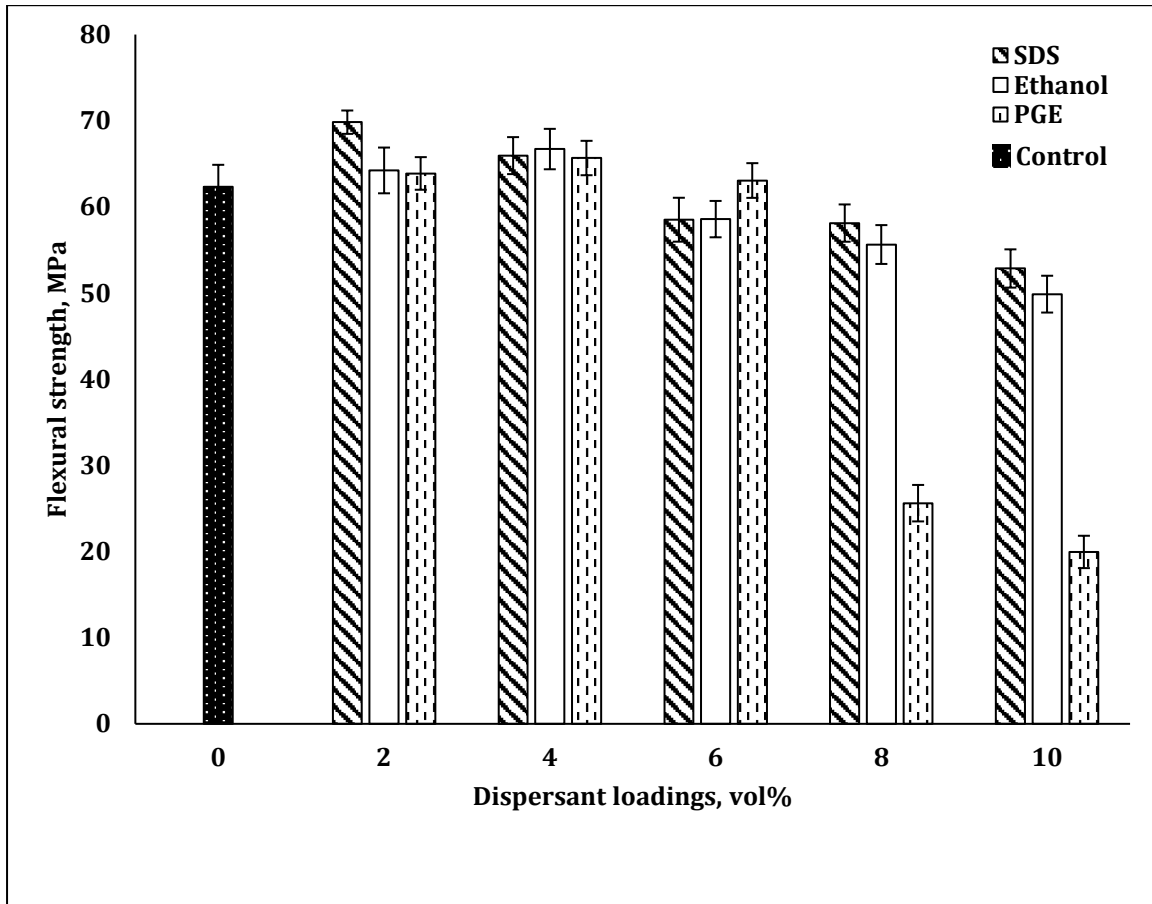
Figure 2: Flexural strength of GNPs filled epoxy nanocomposites with the addition of different dispersing agents and loadings.
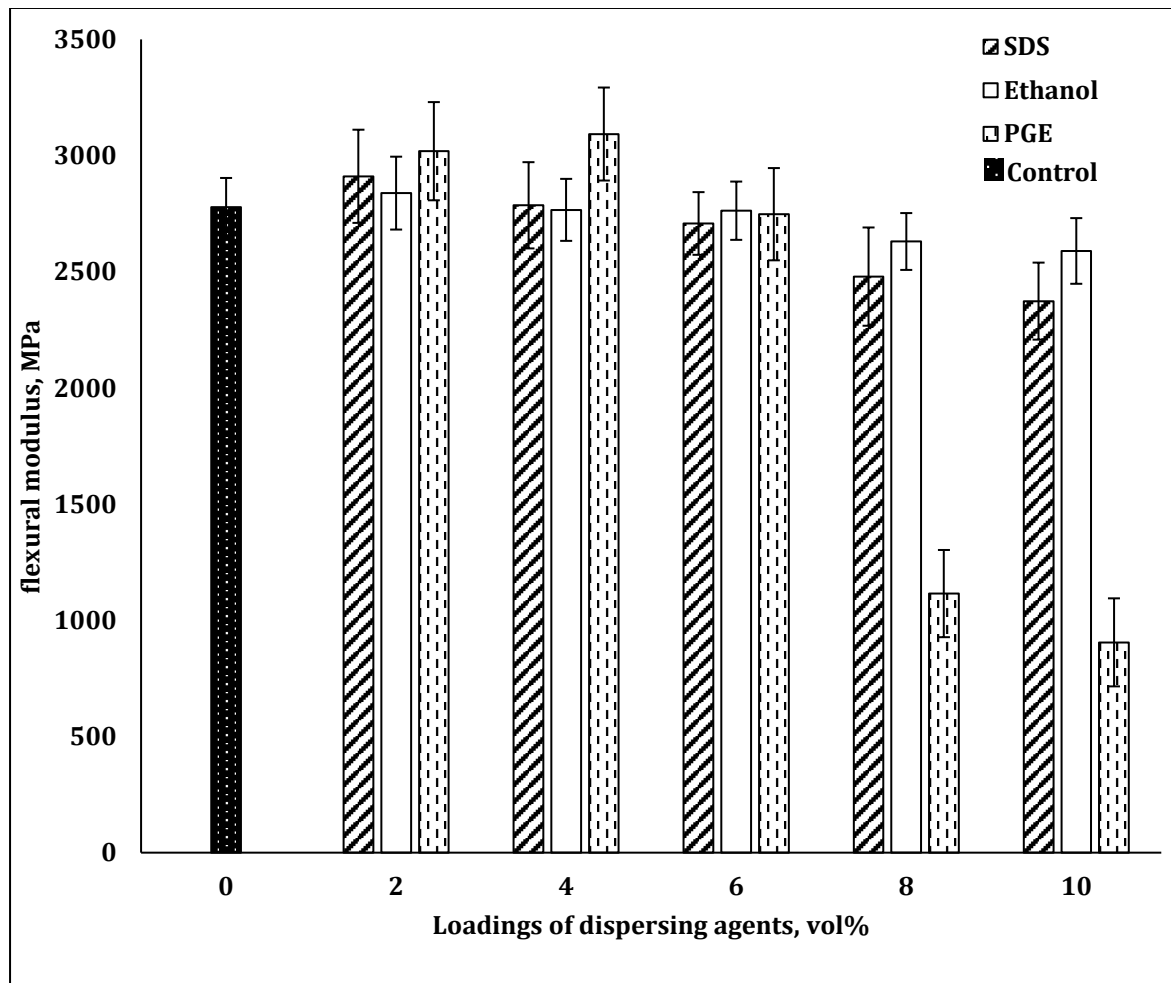
Figure 3: Flexural modulus of GNPs filled epoxy nanocomposites with the addition of different dispersing agents and loadings.

3.3. Fracture toughness

As shown in Figure 4, the K_{IC} value of the control nanocomposite which was prepared previously without the addition of dispersing agent is 3.11 MPa.m$^{1/2}$. The K_{IC} value of nanocomposites increased to 3.354 and 3.137 MPa.m$^{1/2}$ upon the addition of 2 and 4 vol% of SDS as a surfactant, respectively. Further increasing of SDS loading noticeably decrease the fracture toughness of nanocomposites. By using ethanol as dispersing agent, the fracture toughness of nanocomposites increased initially with 2 and 4 vol% loadings. The optimum K_{IC} value of nanocomposite was obtained with the addition of 4 vol% of ethanol, which is 3.25 MPa.m$^{1/2}$. Above 6 vol%, fracture toughness of nanocomposite started to reduce with further increases of ethanol loadings. In contrast, the addition of PGE as dispersing agent shows no improvement towards the fracture toughness of GNPs filled epoxy nanocomposites. The fracture toughness of the nanocomposites was significantly reduced with the addition of PGE. With the addition of 2 vol% of PGE, the K_{IC} value of nanocomposite started to decrease from 3.109 to 2.993 MPa.m$^{1/2}$. Further increases in loading of PGE led to a greater reduction in K_{IC} value, where the lowest K_{IC} value obtained is 1.859 MPa.m$^{1/2}$ with 10 vol% of PGE addition.

The optimum fracture toughness of GNPs filled nanocomposites was achieved with the loading of 2 vol% SDS. By incorporating SDS into GNPs filled nanocomposite, better dispersion of GNPs can be achieved at low amount of surfactant. This inhibits the re-agglomeration of GNPs and leads to strong interfacial adhesion between the GNPs and polymer matrix. Thus, greater fracture energy is able to transfer throughout the polymer matrix which slow down the crack propagation process. However, an excess amount of SDS led to poor dispersion of GNPs which caused a negative impact on the fracture toughness of the GNPs filled nanocomposites. However, the fracture toughness of GNPs filled nanocomposite with the addition of ethanol is relatively more stable. With the addition of 4 vol% ethanol led to greater fracture toughness on the nanocomposite compared to the other two dispersing agents. Nevertheless, a reduction in fracture toughness is still observed with increased loading of ethanol. Dispersing agent loading is critical to the mechanical performance of GNPs filled epoxy nanocomposite, especially the fracture toughness. Excessive dispersing agents may cause porosity and weaken the interaction between filler particles and polymer matrix simultaneously becoming stress concentrators when stress is applied to the nanocomposite. The SEM images of nanocomposites filled with 8 vol% of dispersing agents in comparison with control nanocomposite are shown in Figure 5. It can observe that there are some agglomerates were formed with high loading of dispersing agents. Last but not least, the addition of PGE did not enhance the fracture toughness of the GNPs filled nanocomposites. This indicated that PGE is not appropriate to use as a dispersant on epoxy resin in terms of fracture toughness enhancement. According to the investigation, PGE which remained in the nanocomposite acted as the stress concentrator where it was unable to inhibit and/or slow down the crack propagation on the nanocomposite. Thus, lesser fracture energy is required for the crack to propagate, which led to low fracture toughness obtained.
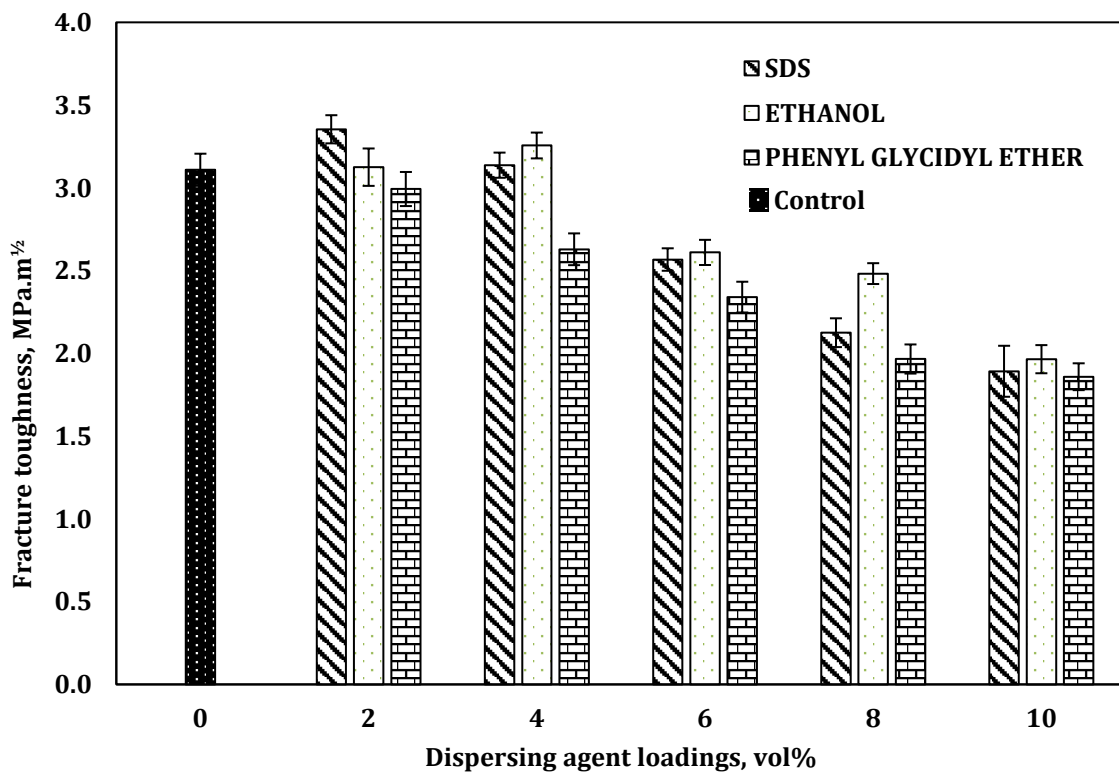

Figure 4: Fracture toughness of GNPs filled epoxy nanocomposites with the addition of different dispersing agents and loadings.
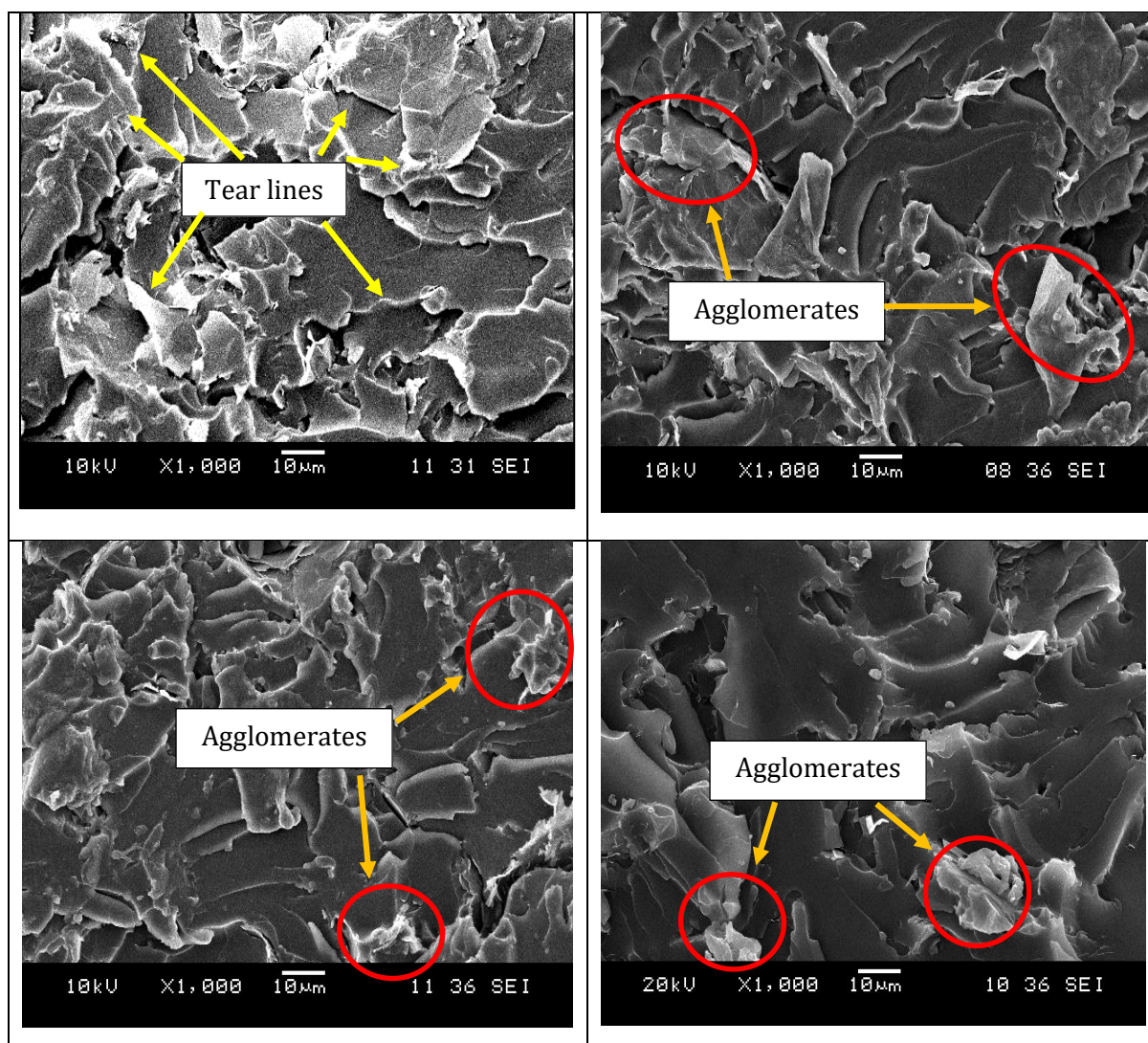
Figure 5: SEM images of: a) Control nanocomposite, b) 8 vol% SDS-GNPs filled nanocomposite, c) 8 vol% Ethanol-GNPs filled nanocomposite, d) 8 vol% PGE-GNPs filled nanocomposite.

4. CONCLUSION

In conclusion, the addition of 2 vol% of SDS improved the electrical bulk conductivity of nanocomposite from 0.00219 $\Omega^{-1}cm^{-1}$ to 0.00712 $\Omega^{-1}cm^{-1}$. Besides that, the incorporation of 2 vol% SDS caused 12% increment in flexural strength and 8% in fracture toughness of nanocomposite. Further increases in SDS deteriorated the performance of GNPs filled epoxy nanocomposite. Excessive SDS that are distributed throughout the polymer matrix disrupt the transportation of electrons and forms pores that become stress concentrators. Hence, an optimum amount of SDS added into GNPs filled epoxy nanocomposite should be controlled within 2 vol%. In contrast, the optimum loading of ethanol and PGE as dispersing agent in GNPs filled epoxy nanocomposite is 4 vol%. The addition of ethanol and PGE beyond 4 vol% deteriorates the electrical and mechanical performance of the nanocomposites. To conclude, sodium dodecyl sulphate (SDS) is the most effective dispersing agent that is used to enhance the performance of GNPs filled epoxy nanocomposite compared to ethanol and phenyl glycidyl ether (PGE).

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