Epoxy resin K-153A and poly(propylene glycol) diglycidyl ether/graphene oxide polymer composite based for properties enhancement

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ABSTRACT

This article introduces the effects of graphene oxide (GO) content on some properties of a composite based on the blend of epoxy resin K-153A and poly(propylene glycol) diglycidyl ether, the blend is hardened with polyethylene polyamine (PEPA). Epoxy resin K-153A is a product of ED-20 epoxy resin modified with oligomer acrylate of MGF-9 and thiokol. Tensile strength of polymer composite with 1.5 wt% of GO is 86.65 MPa and flexural strength of the polymer composite is 105.56 MPa the values are much higher than that of K-153A/ Poly(propylene glycol) diglycidyl ether blend are 68.42 MPa and 79.56 Mpa, respectively. Thermal oxidation resistance polymer composite also increases in comparison to those of K-153A epoxy resin/ Poly(propylene glycol) diglycidyl ether blend.

Keywords: Polymer composite; K-153A epoxy resin; Poly(propylene glycol) diglycidyl ether; Mechanical properties; Thermal oxidation resistance.

1. INTRODUCTION

Epoxy resins with their excellent properties are used in many high-performance engineering applications such as adhesives, polymer composites, coatings, etc. They are well known for their electrical properties, good mechanical properties, and so on [1-3]. Because they are compatible with a variety of reinforcement materials, epoxy resins are one of the best matrices for polymer composites [4-6]. Although having good performances but, they have some disadvantages such as brittleness, long curing time, humidity and UV sensitivity. Researchers have investigated additives to improve properties of epoxy-based composite as flexural strength, tensile strength, thermal performance with nanoclay, nanographene, graphene oxide (GO), graphene nanoplatelets, carbon nanotubes, nanosilica [7-11].

As mentioned above, many authors have investigated polymer composites based on neat epoxy or polyurethane modified epoxy or plant oil-based epoxy, etc but polymer composites based on modified epoxy and active solvents reinforced by GO have been hardly published. This paper will present the effects of GO on some properties of polymer composite based on K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether (PPGDE) blend matrix. In which, K-153A epoxy resin (K- 153A) is manufactured by modifying ED-20 epoxy resin with thiokol and oligoether acrylate, simultaneously. In comparison with common epoxies, it is more flexible and has a lower viscosity. In this investigation, the influence of poly(propylene glycol) diglycidyl ether on K-153A epoxy resin viscosity and GO content some properties of the composite based on K-153A/ poly(propylene glycol) diglycidyl ether blend, were studied so as to find the best suitable ratio for manufacturing the composite. The article also showed comparison results of FT-IR infrared spectroscopy analysis and thermal oxidation resistance of the composite and K-153A/ poly(propylene glycol) diglycidyl ether blend.

2. EXPERIMENT

2.1. Chemicals

K-153A epoxy resin (Russia): Physical state and appearance: Homogeneous liquid from

light to dark brown. Mass fraction of epoxy groups, %: 15-17.5. Dynamic viscosity at 25 °C: 6,000-12,000 cPa.s.

Polyethylene polyamine (PEPA) was supplied by Chimex Ltd (Russia): Molecular weight: 230-250 g/mol. Third amin group: 5- 9.

Poly(propylene glycol) diglycidyl ether was supplied by Sigma-Aldrich in liquid form with an average Mn of about 380.

Graphene Oxide (GO) was a product of ACS MATERIAL LLC (USA). Appearance form: powder. Colour: brown. Molecular weight: 4.239,48 g/mol. Carbon > 85%. Oxygen > 14.95%.

2.2. Sample preparation

Firstly, K-153A epoxy resin with a ratio of 100 wt% and Poly(propylene glycol) diglycidyl ether 10 wt% were well pre-mixed, then mixed with the above mentioned mixture and GO of 0- 2 wt% until a homogeneous mixture was obtained. A mixture of PEPA hardener with 10 wt% part and 100 wt% part of the above mixture was stirred well before into the mold. Composite samples were dried at 80 ºC in 6 hours [10], then samples were kept 07 days at a temperature of (25 ± 2) ºC with relative humidity of (65 ± 2) % before being tested.

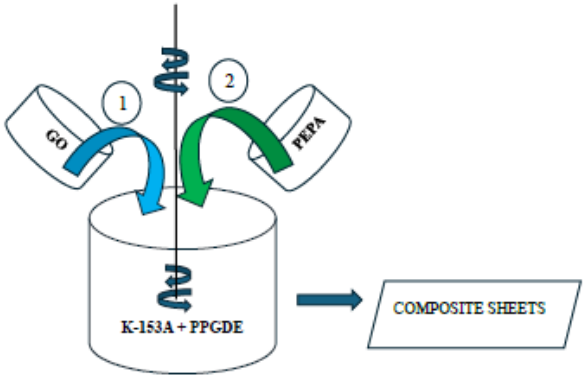


Figure 1. *Preparation process of GO – reinfored K-153A-PPGDE epoxy composite sheets.*

2.3. Analysis Methods

Infrared spectroscopy (FT-IR) was carried out on the Fourier FTIR-8700 series converter. Viscosity of K-153A and mixture of K-153A/ Poly(propylene glycol) diglycidyl ether was determined by Brookfield Model RVT- Series 93412, at (25 ± 0.5) ºC. Flexural strength was determined as ISO 178:2010 on Instron 5582-100 kN device, with a bending speed of 5 mm/minute. Tensile strength was determined as ISO 527-1:2012 on Zwick device with a sample pulling speed of 5 mm/minute, at a temperature of (25 ± 2) ºC, humidity of (70 ± 2) %. Thermal oxidation resistance was carried out by Thermogravimetric analysis (TGA) in air conditions on NETZSCH TG 209F1 LIBRA machine with a temperature raising rate of 10 ºC/minute from room temperature to 600 ºC.

3. RESULTS AND DISCUSSION

3.1. Effect of GO Content on the physical state and viscosity of K-153A/ poly(propylene glycol) diglycidyl ether blend

Properties and fabrication process of polymer composite were strongly affected by the percentage and dispersion of GO particles in the matrix. Polymer composite samples were made from K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether matrix and GO particles. The influence of GO content on the physical state and viscosity of K-153A epoxy resin with a ratio of 100 wt% and Poly(propylene glycol) diglycidyl ether at 25 ºC was shown in Table 1.

Table 1 showed that with GO content up to 1.5 wt%, the mixture remained its viscous liquid state, when GO content reached 2 wt%, mixture became gel. GO content of a small quantity (0.5 wt% and

below), viscosity of mixture slightly increased comparison to that of K-153A/Poly(propylene glycol) diglycidyl ether blend. When GO content increased continuously, the viscosity of the mixture increased sharply and when GO content reached 2 wt%, the mixture became a gel state, which could not be used as a matrix for the polymer composite. It can be explained that GO has very high surface energy or a huge surface area. When dispersed in the blend, they formed a complex network and linked with each other and with polymer molecules, leading to an increase in the viscosity and density of the blend. Besides that oxidized functional groups on the GO surface (such as hydroxyl, carboxyl) could interact chemically and physically (hydrogen bonding, van der Waals) with K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether to increase the viscosity of the mixture. Added to that GO particles were in K-153A/Poly(propylene glycol) diglycidyl ether blend high enough that they would themselves agglomerate and cause gelation [9, 12-14].

Table 1. *Physical state and viscosity of K-153A/(PPGDE) blend with different GO content.*

No.	K-153A/(PPGDE) blend, Wt%	GO, Wt%	Physical state	Viscosity, cP
1	100	0	Liquidity, transparency	31
2	100	0.5	Liquidity, transparency	67
3	100	1	Liquidity, opaque	205
4	100	1.5	Liquidity, opaque	562
5	100	2	Gel	-

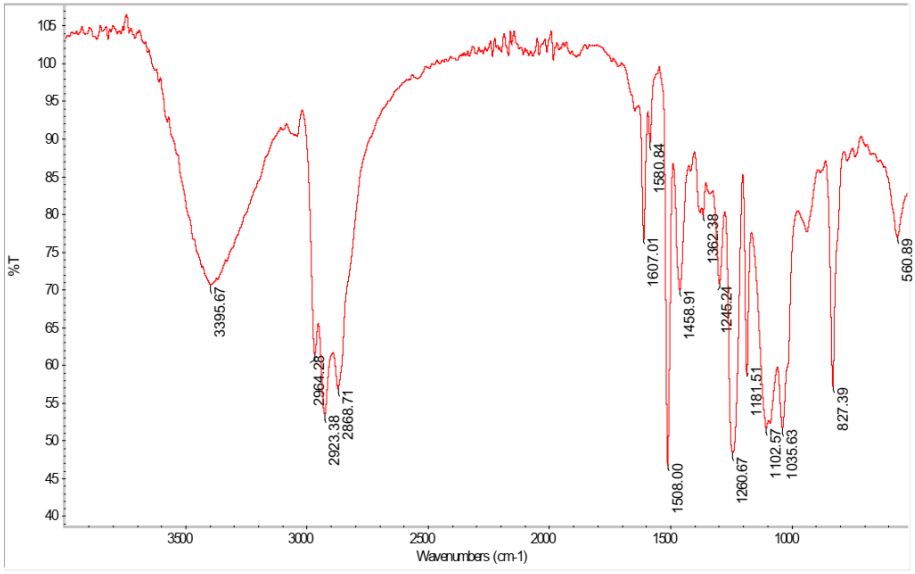
3.2 Fourier-transform infrared spectroscopy (FT-IR) analysis

To determine the presence of GO in the polymer composite, FT-IR was used. A polymer composite sample with GO content of 1.5 wt% was chosen. Results were shown in Figures 2a, 2b and Table 2.

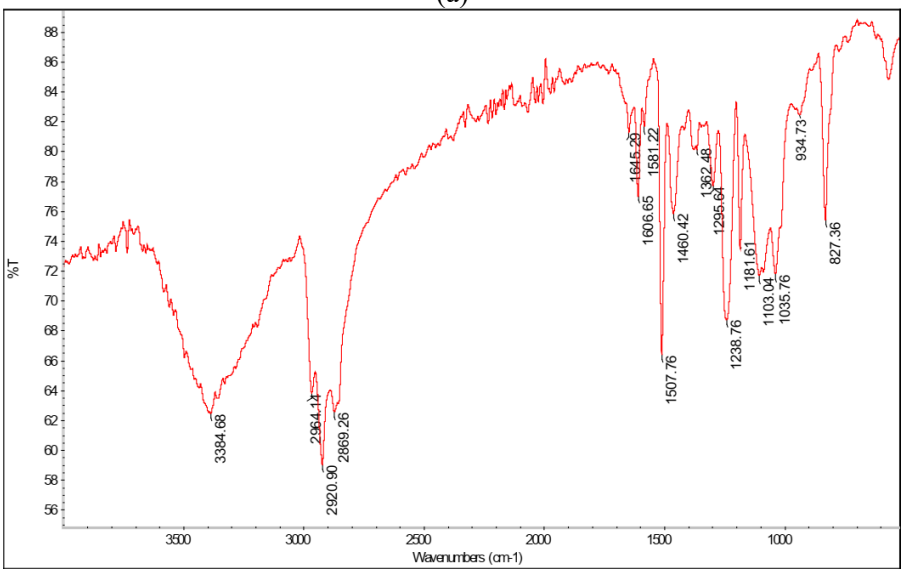
Table 2. *Selected measured FT-IR bands.*

No.	Typical spectrum	Wavenumbers (cm-1)
1	νOH	3395.87
2		3395.67
3		3384.68
4	Nch (Aliphatic hydrogens)	2964.28
5		2923.38
6		2964.14
7		2920.90
8		2869.28
9		2869.43
10		2868.71
11	ν (C=C) of alkene	1607.01
12		1606.65
13		1581.22
14		1580.84
15	νC-OH	1238.65
16	ν_aC-O-C (asymmetry)	1181.61
17		1181.51
18		1103.04

19		1102.57
20	C=O	1052.88
21	ν_a C-O-C (symmetry)	1035.76
22		1035.63
23	νC=C-H (out of plane)	934.73



(a)



(b)

Figure 2. *FTIR of cured K-153A/ PPGDE blend* (*a*)*, polymer composite* (*b*).

Figure 2a showed that peaks 3395.87 cm^{-1}, 3395.67 cm^{-1} and 3384.68 cm^{-1} of OH group in oxiran group in epoxy resin; peaks 2964.28 cm^{-1}, 2964.14 cm^{-1}, 2929.90 cm^{-1}, 2869.43 cm^{-1}, 2869.43 cm^{-1} and 2868.71 of CH group of aliphatic hydrogens; peaks 1607.01 cm^{-1}, 1606.65 cm^{-1}, 1581.22 cm^{-1}, and 1580.84 cm^{-1} of C=C of alkene;… There were some new peaks in Figure 2b, in which, peak 1645.29 cm^{-1} as C=C vibration of graphite; peak 934.73 cm^{-1} represented for vibration of C=C-H (out of plane), and peak 1238.6 cm^{-1} was typical for the

vibration of the C-OH group of GO, which proved in the captured sample FT-IR spectrum with GO [10, 15, 16]. Figure 2b also showed that there were some slight shifts, such as 3395.67 or 3395.87 shifted to 3384.68; 1102.57 shifted to 1103.04, etc. This occurred because the electron density was affected by the surrounding environment, there was no breaking of bonds or new bond formation. In other words, oxygen-containing groups on the surface of GO did not participate in chemical reactions to open epoxy rings, they simply created physical interactions (van der Waals forces or hydrogen bonds) only [8, 17].

3.3. Effect of GO content on tensile strength and flexural strength of polymer composite

To study the effect of GO content on tensile strength, flexural strength of the polymer composite, samples with GO content of 0- 2 wt% were prepared and cured at 80 °C in 6 hours. Samples were kept 07 days at a temperature of (25 ± 2) °C with relative humidity of (65 ± 2) % before testing tensile strength, flexural strength. Results were shown in Figure 3.

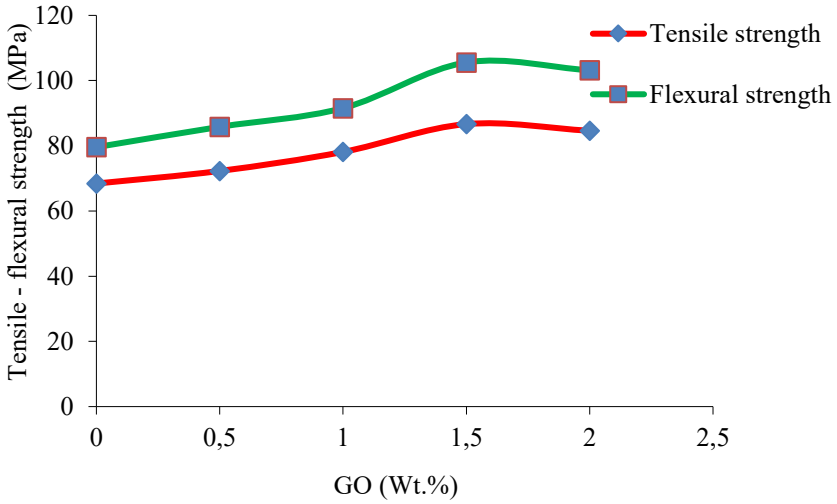


Figure 3. *Effects of GO content on tensile strength and flexural strength of polymer composite.*

Figure 3 showed that when GO content increased to 1.5 wt%, tensile strength and flexural strength of polymer composite were significantly improved in comparison to that of K-153A/ PPGDE blend without GO. Maximum tensile strength of the polymer composite was 86.65 MPa when GO content was 1.5 wt%, compared to 68.42 MPa of K-153A/ PPGDE blend. Meanwhile, the flexural strength of the polymer composite increased to a maximum value of 105.56 MPa when GO content reached to 1.5 wt%, compared to the value of 79.58 MPa of K-153A/ PPGDE blend. The increase in of tensile strength and flexural strength of the polymer composite could be explained by the fact that GO particles were well dispersed in K-153A/ PPGDE matrix uniformly up to 1.5 wt%. The close interaction between GO surface and K-153A/ PPGDE matrix led to stress transfer between the two phases. Besides that GO particles acted as agents to prevent cracks from propagating when the destruction of the polymer composite occurred. In addition to that GO particles were much harder than K-153A/ PPGDE blend, so when the polymer composite was loaded, deformation between the two phases would be delayed, and more energy absorption so tensile strength, flexural strength of the polymer composite increased. But when GO content increased to over 1.5 wt.%, GO particles tended to agglomerate into larger particles to release surface energy. The bond between particles was a hydrogen bond, which was relatively weak and could be broken easily, so the tensile and flexural strength of the polymer composite would decrease, respectively [2, 10, 17]. Similar to this investigation, A. Loeffen *et al* [11] reported similar behavior in tensile and modulus properties of pure bio-epoxy and bio-epoxy/GO composites reinforced with GO, the tensile strength increased from 60 ± 1.4 MPa of pure bio-epoxy to 74 ± 2.6 MPa for bio-epoxy/GO, and the modulus from 2.6 ± 0.11 GPa to 3.5 ± 0.19 GPa, correspondingly.

3.4. Thermal oxidation resistance of K-153A/ PPGDE blend and polymer composite

To investigate thermal oxidation resistance of cured K-153A/ PPGDE blend and polymer composite, Thermogravimetric analysis (TGA) was used. Samples were cured K-153A/ PPGDE blend and polymer composite with GO of 1.5 wt%. Results are shown in Table 3 and Figures 4a, 4b.

Figures 4a, 4b and Table 3 showed that the thermal oxidation resistance of the polymer composite was much higher than that K-153A/ PPGDE blend. Results also showed that the ash content of the polymer composite was up to 17.21 %, it was much higher than that K-153A/ PPGDE blend of 8.85 %. Figure 4a showed that K-153A/ PPGDE blend lost its weight of 5 % at the temprature of 298.5 ^{o}C, meanwhile, the value was 308.5 ^{o}C for the polymer composite. It meant that GO had improved the thermal oxidation resistance of the polymer composite. It may be explained that in high temperature condition with the presence of oxygen, polymer chains were cut. At that time, the oxidation of organic substances also occurred. Oxygen had enhanced the formation of free radicals, causing the degradation of polymer chains sharply, which formed a lower molecular polymer formula, meanwhile, GO particles prevented the penetration of heat and oxygen into compounds containing oxygen, so the polymer composite with GO had higher heat resistance than K-153A/ PPGDE blend. In addition, thermal decomposition of GO would coke to form a stable structure like ceramic in polymer composite, it worked as a fence to protect the material from heat [15, 18-20]. This result was in agreement with previous reports [7]. It showed that GO had improved the thermal stability of epoxy resin. The degradation of neat epoxy resin was more different from GO/EPs. There was a little growth in the onset degradation temperature of modified epoxy, meanwhile, the Tmax (the temperature at which the resin loses 50% weight percent) of GO/EPs increased by 9 to 14 ◦C compared with that of neat epoxy. The presence of high specific surface GO acted as a barrier to prevent oxygen from contacting epoxy chains so as to improve the thermal stability of GO/EPs.

Table 3. *Thermal oxidation resistance of K-153A/ PPGDE blend and /polymer composite.*

Samples	Weight loss (%)		
	300 ^{o}C	400 ^{o}C	600 ^{o}C
K-153A/PPGDE	5.25	76.34	8.85
Polymer composite	3.96	57.16	17.21

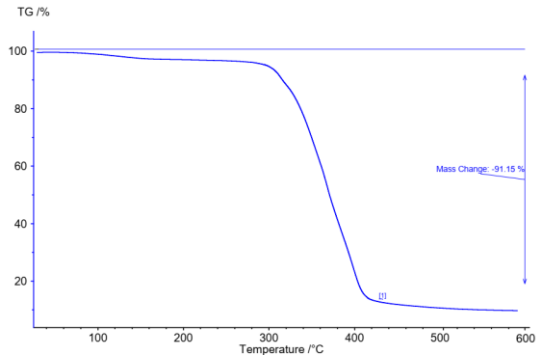


Figure 4a. *TGA of K-153A/ PPGDE blend.*

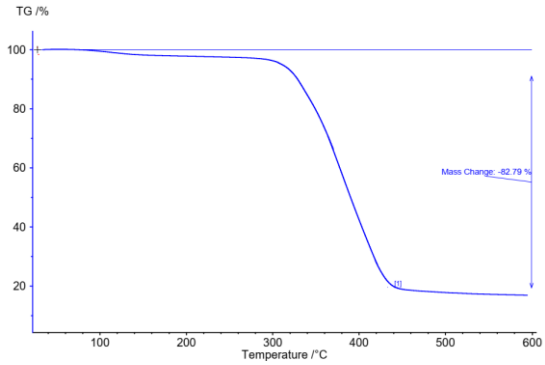


Figure 4b. *TGA of polymer composite.*

4. CONCLUSIONS

Graphene oxide particles strongly affect the physical state and viscosity of K-153A/Poly(propylene glycol) diglycidyl ether blend. The polymer composite's tensile strength was 86.65 MPa and flexural strength was 105.56 MPa with GO of 1.5 wt%, those significantly

increased compared to these of K-153A/ PPGDE blend, which were 68.42 MPa and 79.59 MPa, respectively. Besides that, the thermal oxidation resistance of the polymer composite has been improved with GO and the ash content of the polymer composite was 17.21 %, meanwhile, the value of K-153A/ PPGDE blend was 8.85 %. Considering the properties of polymer composite, GO content of 1.5 wt% was most suitable for making polymer composite based on K-153A epoxy resin with ratio of 100 wt% and Poly(propylene glycol) diglycidyl ether 10 wt%.

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TÓM TẮT

Cải thiện tính chất của poymer composite trên cơ sở epoxy K153A và poly(propylene glycol) diglycidyl ether bằng graphene oxide

Bài báo này giới thiệu ảnh hưởng của hàm lượng oxit graphene (GO) đến một số tính chất của vật liệu polymer composite dựa trên hỗn hợp nhựa epoxy K-153A và poly(propylene glycol) diglycidyl ether, hỗn hợp này được đóng rắn bằng polyetylen polyamine (PEPA). Nhựa epoxy K-153A là sản phẩm của nhựa epoxy ED-20 được biến tính bằng oligomer acrylate mã hiệu MGF-9 và thiokol. Độ bền kéo của vật liệu polymer composite với 1,5% khối lượng GO là 86,65 MPa và độ bền uốn của vật liệu composite polymer là 105,56 MPa, các giá trị này cao hơn nhiều so với hỗn hợp K-153A/poly(propylene glycol) diglycidyl ether có độ bền uốn lần lượt là 68,42 MPa và 79,56 MPa. Khả năng chống oxy hóa nhiệt của vật liệu polymer composite cũng tăng lên so với hỗn hợp nhựa epoxy K-153A/poly(propylene glycol) diglycidyl ether.

Từ khóa: Vật liệu composite polymer; Nhựa epoxy K-153A; Poly(propylene glycol) diglycidyl ether; Tính chất cơ học; Khả năng chống oxy hóa nhiệt.