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# Epoxy K-153A/ Poly(propylene glycol) Diglycidyl Ether Polymer Composites Enhanced Nanosilica and Graphene Oxide

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### ABSTRACT

This article introduced effects of graphene oxide (GO) and nanosilica contents on properties of polymer composites based on K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether with hardener of polyethylene polyamine (PEPA). Viscosity was determined by Brookfield Model RVT-Series 93412. Hardness of material was determined as by TECLOCK- Jisk 6301A and Thermogravimetric analysis (TGA), Energy Dispersive X-ray Spectrometry (EDX) were used to characterize the samples. Results showed that GO content of 2 weight percent (wt%) and nanosilica content of 1.5 wt% were the most suitable for 100 wt% of K-153A epoxy resin/ 10 wt% of Poly(propylene glycol) diglycidyl ether (PPGDE) mixture in manufacturing polymer composites. Surface hardness of polymer composites with 2 wt% of GO was 88 Shore D and was 84 Shore D for polymer composite with 1.5 wt of nanosilica these values were much higher than that of K-153A/ Poly(propylene glycol) diglycidyl ether mixture of 69 Shore D. Besides that thermal resistance and ash remaining of polymer composites also increase in comparison to those of K-153A epoxy resin/ Poly(propylene glycol) diglycidyl ether mixture.

## 1. Introduction

Epoxy resins are widely used for adhesives, polymer composite, coatings etc. because of their excellent properties [1-4]. They are compatible with variety of reinforcement phases so epoxy resins are one of the best choice for polymer composites matrices [5-7]. To overcome the disadvantages as brittleness, long curing time, humidity and UV sensitivity of epoxy resins, researchers have used carbon nanotube, graphene nanoplatelets, nanoclay, nanographene, graphene oxide (GO), nanosilica or modified epoxy by plant oil, rubbers, thermoplastics, etc. to improve properties of epoxy-based polymer composite as [1, 3, 5, 8-12].

As mentioned, to overcome the disadvantages of pure epoxies or modified epoxies, additives or others polymers have been added. In general speaking, viscosity of epoxy resin is high for manufacturing polymer composites, especially, nanocomposites. In practice, active solvents are used to reduce viscosity of epoxies, it means that more additives can be used to improve properties of

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polymer composites or coatings or adhesives, etc based on epoxies but they have been published hardly. This article will present the influence of GO and nanosilica on properties of polymer composite based on K-153A epoxy resin and active solvent of Poly(propylene glycol) diglycidyl ether (PPGDE). In which, K-153A epoxy resin (K- 153A) is made by simultaneously modifying ED-20 epoxy resin with thiokol and oligoether acrylate (MGF-9).

In this investigation, effect of GO and nanosilica contents some properties of polymer composite based on K-153A/ poly(propylene glycol) diglycidyl ether mixture. The study was carried out so as to find the most suitable contents for manufacturing polymer composites. The paper also showed results of Energy Dispersive X-ray Spectrometry and thermal resistance of polymer composites and K-153A/ poly(propylene glycol) diglycidyl ether mixture.

## 2. Materials and Methods

### 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, cPa.s: 6,000-12,000. Polyethylene polyamine (PEPA) supplied by Chimex Ltd (Russia): Molecular weight: 230- 250 g/mol, Third amin group: 5- 9. Poly(propylene glycol) diglycidyl ether supplied by Sigma-Aldrich with liquid form and average Mn of about 380. Nanosilica supplied by Sigma-Aldrich: Fine powder, Purity: 99.8 %, Average size: 12nm, Specific surface area: 175- 225 m<sup>2</sup>/g (according to BET method). 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 ratio of 100 wt% and Poly(propylene glycol) diglycidyl ether 10 wt% were well pre-mixed then above-mentioned mixture and GO of 0- 2,5 wt% were stirred until getting a homogenous mixture. PEPA hardener with 10 wt% part and 100 wt% part of the above mixture was stirred well before pouring into the mold to make polymer composite samples. Polymer composite samples were dried at 80 °C in six hours then were kept seven days at temperature of (25 ± 2) °C with relative humidity of (65 ± 2) % before being tested.

For polymer composite reinforced by nanosilica, K-153A epoxy resin with ratio of 100 wt% and Poly(propylene glycol) diglycidyl ether 10 wt% were well pre-mixed then mixture and nanosilica of 0- 2 wt% were stirred until getting a homogenous mixture. 100 wt% part of the above mixture and 10 wt% part PEPA were well stirred before pouring into the mold to make polymer composite samples. Polymer composite samples were dried at 80 °C in six hours then samples were kept seven days at temperature of (25 ± 2) °C with relative humidity of (65 ± 2) % before being tested.

### 2.3. Analysis Methods

Viscosity was determined by Brookfield Model RVT- Series 93412 at (25 ± 0.5) °C. Hardness of material was determined as ISO 7619-1:2010 by TECLOCK- Jisk 6301A. Abrasion resistance was determined as ISO 4649:2017. Thermal resistance was carried out in nitrogen condition by Thermogravimetric analysis (TGA) on NETZSCH TG 209F1 LIBRA machine with temperature raising rate of 10 °C/minute from room temperature to 600 °C. The elemental composition of produced samples was determined on Energy Dispersive X-ray Spectrometry (EDX) analyzer model HORIBA 7593H. The samples were prepared as a plate with an accelerating voltage exposure of 20 kV.

### 3. Results and Discussion

#### 3.1 Effect of GO and nanosilica content on on physical state and viscosity of K-153A/ poly(propylene glycol) diglycidyl ether mixture

Properties and fabrication process of polymer composite are strongly effected by the content and dispersion of reinforcement particles in the matrix. Influence of GO and nanosilica content on viscosity and physical state of mixture 100 wt% K-153A epoxy resin and 10 wt% poly(propylene glycol) diglycidyl ether at  $(25 \pm 0.5) ^\circ\text{C}$  were shown in Table 1 and Table 2.

**Table 1**

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

No.	K-153A/(PPGDE) mixture, Wt%	GO, Wt%	Physical state	Viscosity, cP
1	100	0	Liquidity, transparence	33
2	100	0.5	Liquidity, transparence	61
3	100	1	Liquidity, transparence	185
4	100	1.5	Liquidity, opaque	518
5	100	2	Liquidity, opaque	593
6	100	2.5	Gel	-

**Table 2**

Physical state and viscosity of K-153A/(PPGDE) blend with different nanosilica content

No.	K-153A/(PPGDE) mixture, Wt%	Nanosilica, Wt%	Physical state	Viscosity, cP
1	100	0	Liquidity, transparence	33
2	100	0.5	Liquidity, transparence	100
3	100	1	Liquidity, transparence	245
4	100	1.5	Liquidity, opaque	576
5	100	2	Gel	

Table 1 showed that with GO content up to 2 wt%, mixture remained its viscous liquid state, when GO content reached 2.5 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 mixture. GO content continuously increased, viscosity of mixture increased sharply and when GO content reached 2,5 wt%, mixture became gel, which could not be used for the polymer composite. It could be explained that GO has very high surface energy or huge surface area, dispersing in the mixture, they formed a complex network and linked with each other and with polymer molecules to increase the viscosity and density of the mixture. 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 so as to increase mixture's viscosity. Besides that, GO particles content was high enough, they would themselves agglomerate and caused gelation [7, 13- 15].

Table 2 showed that with nanosilica with a small content (0.5 wt%), viscosity of mixture increased slightly compared to that of the mixture. When nanosilica content increased continuously, viscosity of mixture increased sharply and reached maximum value when nanosilica was about 1.5 wt% with viscosity of 548 cP. Nanosilica content reached 2 wt%, mixture became gel, which could not be used to make polymer composite. This could be explained that there have silanol groups (Si-

OH) on surface of nanosilica, when nanosilica in the mixture high enough, silanol groups would interact with hydroxyl groups in epoxy resin and led to gelation. Besides that, when nanosilica particles in mixture were too high, they would agglomerate themselves and caused gelation [6, 16, 17].

### 3.2. Effect of GO and nanosilica contents on abrasion and surface hardness of polymer composites

To study effects of GO and nanosilica contents on abrasion resistance and surface hardness of polymer composites based on K-153A/ PPGDE, samples were prepared and cured at 80 °C in six 6 hours with GO contents of 0 - 2 wt% and nanosilica contents of 0 – 1.5 wt%. Samples were kept seven days at temperature of (25± 2) °C and relative humidity within the range of (65 ± 2) % before determining abrasion resistance and surface hardness. Results were shown in Figure 1, Figure 2, Figure 3 and Figure 4.

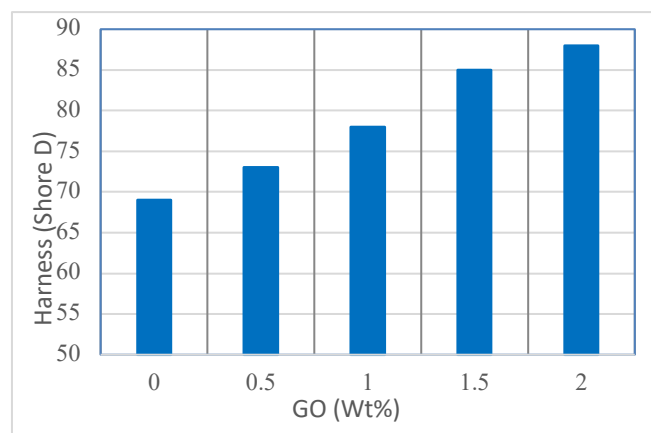


Fig. 1. Effect of GO content on surface hardness of polymer composite

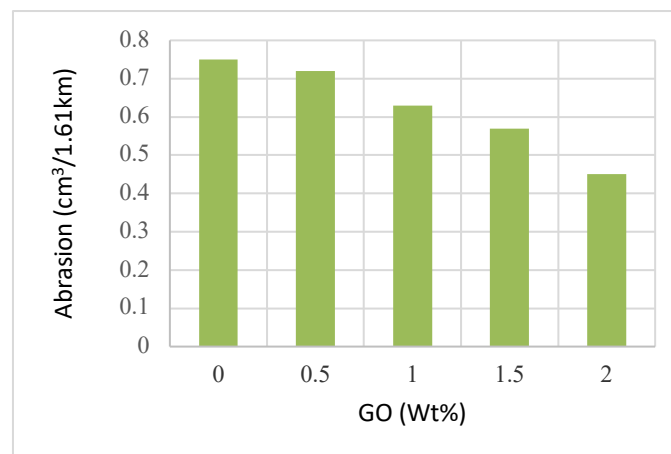


Fig. 2. Effect of GO content on abrasion of polymer composite

Figures 1 and 2 showed that the surface hardness of polymer composite increased in case of GO content increased. GO content reached to 2 wt%, surface hardness of polymer composite reached 88 Shore D (this value was much higher than that of K-153A and PPGDE mixture, 69 Shore D). This can be explained that K-153A/ PPGDE mixture was the matrix phase and GO worked as the reinforced phase and that improved its resistance to deformation the polymer composite. Besides that GO

particles had a higher hardness than that of K-153A/PPGDE mixture, so GO content increased hardness of polymer composite would be increased correspondingly. Abrasion resistance of polymer composite had been improved, when GO content increased, abrasion resistance of material was improved significantly, this meant that the loss of polymer composite decreased together with the content of GO particles [7, 9, 18, 19]. Polymer composite with GO content of 2 wt% was chosen for next study.

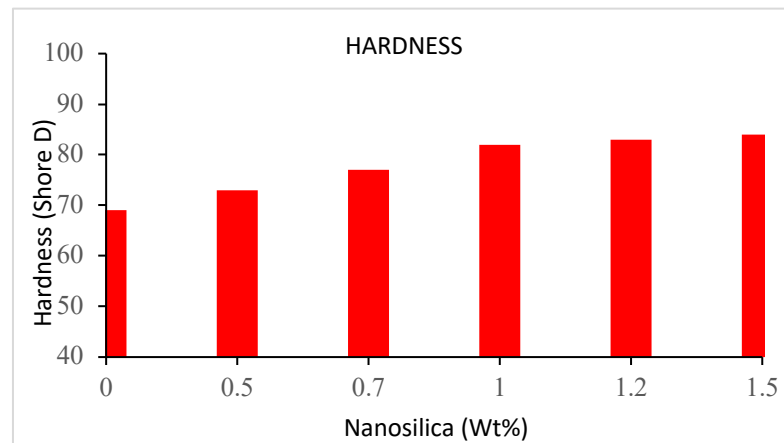


Fig. 3. Effect of nanosilica content on surface hardness of nanocomposite

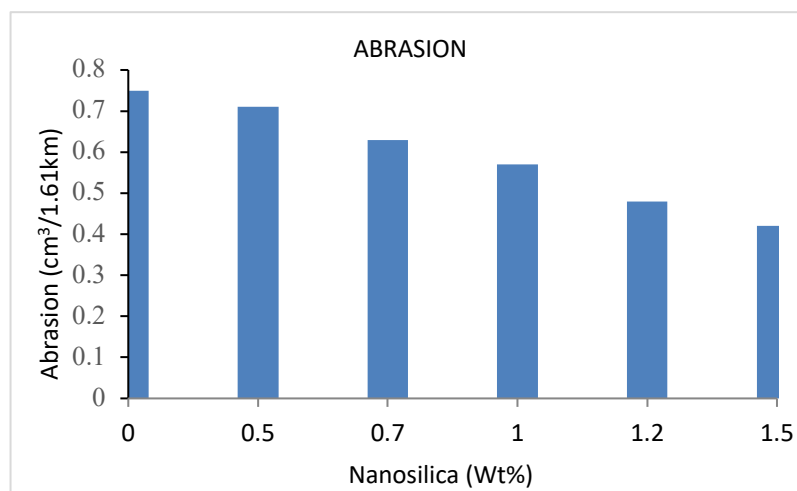


Fig. 4. Effect of nanosilica content on abrasion of nanocomposite

Zeolite is a crystalline aluminosilicate mineral that forms naturally through the interaction of volcanic ash with alkaline water [1-3]. The name “ZEO” is derived from two Greek words: “zeo,” meaning to boil, and “lithos,” meaning stone. Zeolites possess three-dimensional structures arising from oxygen-linked frameworks of  $[\text{SiO}_4]^{4-}$  and  $[\text{AlO}_4]^{5-}$  polyhedral [4,5]. The lattice structure of zeolites carries a net negative charge, enabling them to attract and bind various cations, including sodium, lead, manganese, and zinc. They are porous crystals with the structural formula expressed as  $\text{M}_x/n [(\text{Al}_2\text{O}_3)_x(\text{SiO}_2)_y \cdot n\text{H}_2\text{O}]$ , where M represents metal cations external to the framework and n denotes their valence states. The parameters x and y correspond to the number of aluminum and

silicon tetrahedra, while 'w' indicates the number of water molecules present [6-8]. The formation of zeolite is suggested to be significantly influenced by several parameters, including batch composition, silica-to-alumina ratio, presence of organic templates, water content, aging time, temperature, pH, and crystallization duration [9,10]. Among these factors, crystallization temperature plays a crucial role in the synthesis of zeolites. It can affect various aspects such as growth velocity, the interface reactions between different crystal faces or directions, and the diffusion rate of active crystal particles [11-13].

Zeolites are produced on a large scale, often synthesized using costly chemical reagents [3,14]. Due to the high production costs, researchers have investigated more affordable materials as precursors for zeolite synthesis. Previous research has shown that scientists have investigated low-cost and effective alternative materials, including kaolin, rice husk, fly ash, municipal solid waste, industrial sludge, coal ash, clay minerals, and volcanic glasses, as substitutes for chemical reagents in zeolite production [15-19]. The well-known, industrially significant zeolite is ZSM-5, which is known for its ability to catalyse a variety of reactions due to its exceptional physicochemical properties [14]. These features, exceptionally high stability, selectivity, and sensitivity, significantly influence its catalytic reactions. Formerly synthesized in 1971 by Argauer and Landolt [20], ZSM-5 has found widespread applications in industries such as petroleum refining, chemical production, and gas separation. It catalyzes processes such as isomerization, alkylation, and hydrocarbon conversion and can also function as a bifunctional catalyst when combined with metals or acids. Traditional ZSM-5 synthesis often involves the use of chemicals that can potentially pose environment and health risk [21,22]. Therefore, exploring alternative, more sustainable materials to replace these chemicals is crucial for making the production process more cost-effective and environmentally friendly.

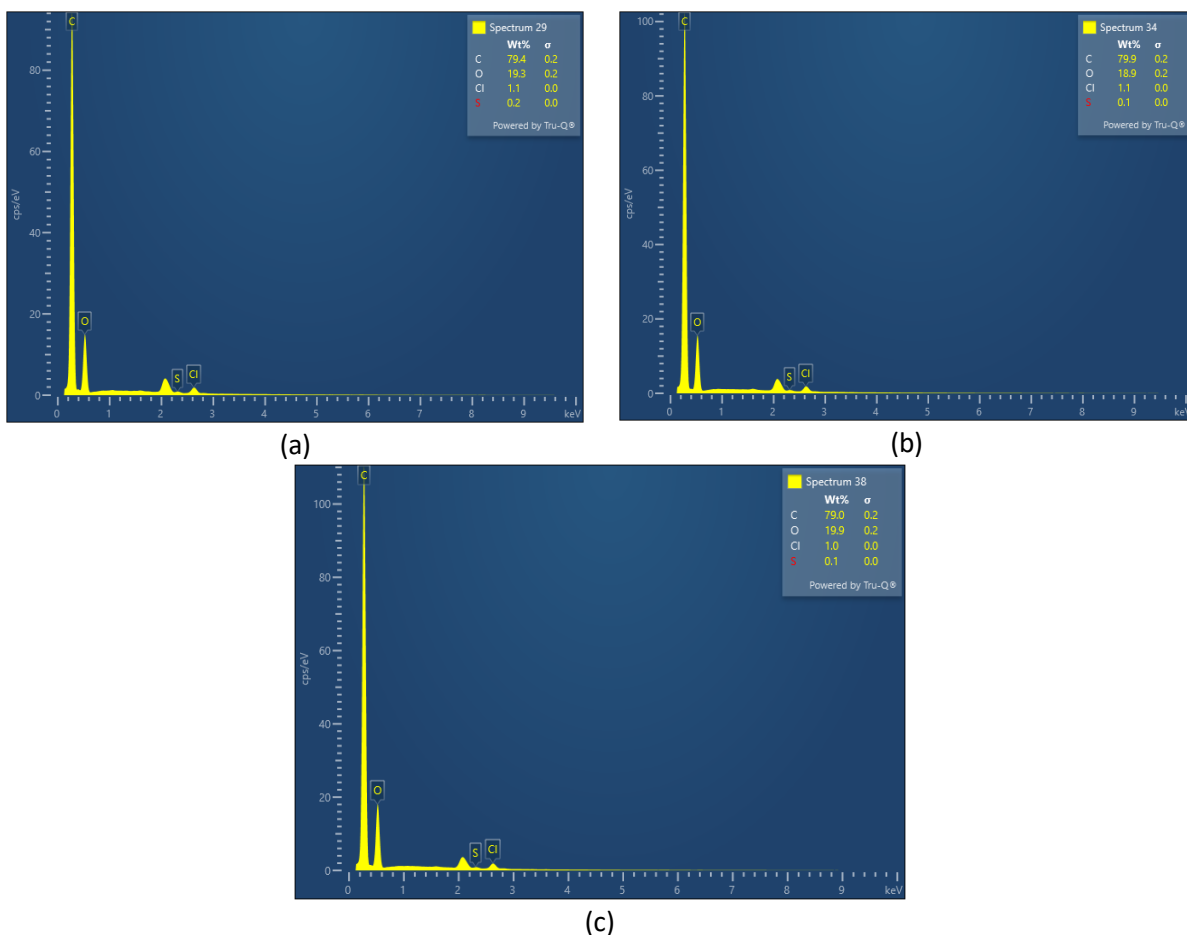
The natural minerals obtained from alternative materials differ in their chemical, processing, and mineralogical compositions, which may significantly affect the final product of zeolites when used as precursors. Rice husk, known for its high silica content and the presence of palygorskite, can be converted into highly siliceous ZSM-5. However, investigations by Jiang et al. [18], and Panpa and Jinawath [17] revealed that the addition of aluminum to reduce the Si/Al ratio had an unfavorable effect on the formation of ZSM-5 from palygorskite. Chareonpanich et al. [23] found that fly ash was a feasible source for zeolite synthesis. However, the synthesis process requires high temperatures exceeding 200°C. Despite this, when using fly ash as a precursor, only low yields are obtained due to the limitations posed by the temperature range. Among these materials, kaolin has been studied as a potential alternative to chemical reagents [5,10]. However, the composition and structure of kaolin vary significantly depending on its geographic origin and formation process. These variations in composition and structure can influence its chemical reactivity during synthesis [24,25]. Kaolin has a layered structure with the chemical composition  $Al_2Si_2O_5(OH)_4$ . Its primary constituent, kaolinite, is characterized by a unique structural arrangement of alternating alumina octahedral sheets and silica tetrahedral sheets. It shares a similar silicon-to-aluminum (Si/Al) ratio with zeolites, making it a potential candidate for zeolite synthesis [24,25]. These layers are interconnected, where the tips of the silica tetrahedrons share a standard layer with adjacent octahedral sheets, as illustrated in Fig. 1.

Figures 3 and 4 showed that nanosilica content increased the surface hardness of polymer composite increased. Nanosilica content reached 1.5 wt%, surface hardness of polymer composite reached 84 Shore D (this value was much higher than that of mixed K-153A and PPGDE without nanosilica particles, 69 Shore D). As explained above K-153A/PPGDE mixture worked as matrix and nanosilica worked as the reinforcement phase and that improved its resistance to deformation the polymer composite. Besides that, nanosilica particles had higher hardness than that of K-153A/ PPGDE mixture, so nanosilica content increased hardness of polymer composite would be increased correspondingly. Abrasion resistance of polymer composite had been improved, when nanosilica

content increased, abrasion resistance of material significantly improved, this meant that the loss of polymer composite decreased together with the content of nanosilica particles [6, 10, 20, 21]. This phenomenon was also observed by [8], epoxy resin was the matrix and nanosilica worked as the reinforcement phase and it improved its resistance to deformation loaded by external forces to nanocomposite. Besides that nanosilica particles are much higher hardness than epoxy resin, when nanosilica content increased the nanocomposite's hardness would be increased.

### 3.3 EDX analysis of polymer composites

To study influence of GO and nanosilica particles to chemical composition of polymer composites observation via EDX was conducted. EDX of the fractured surface of K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether (PPGDE) mixture, polymer composite with GO content of 2 wt% and polymer composite with nanosilica content of 1.5 wt% were observed. The observation images were depicted as in the following Figure 5.

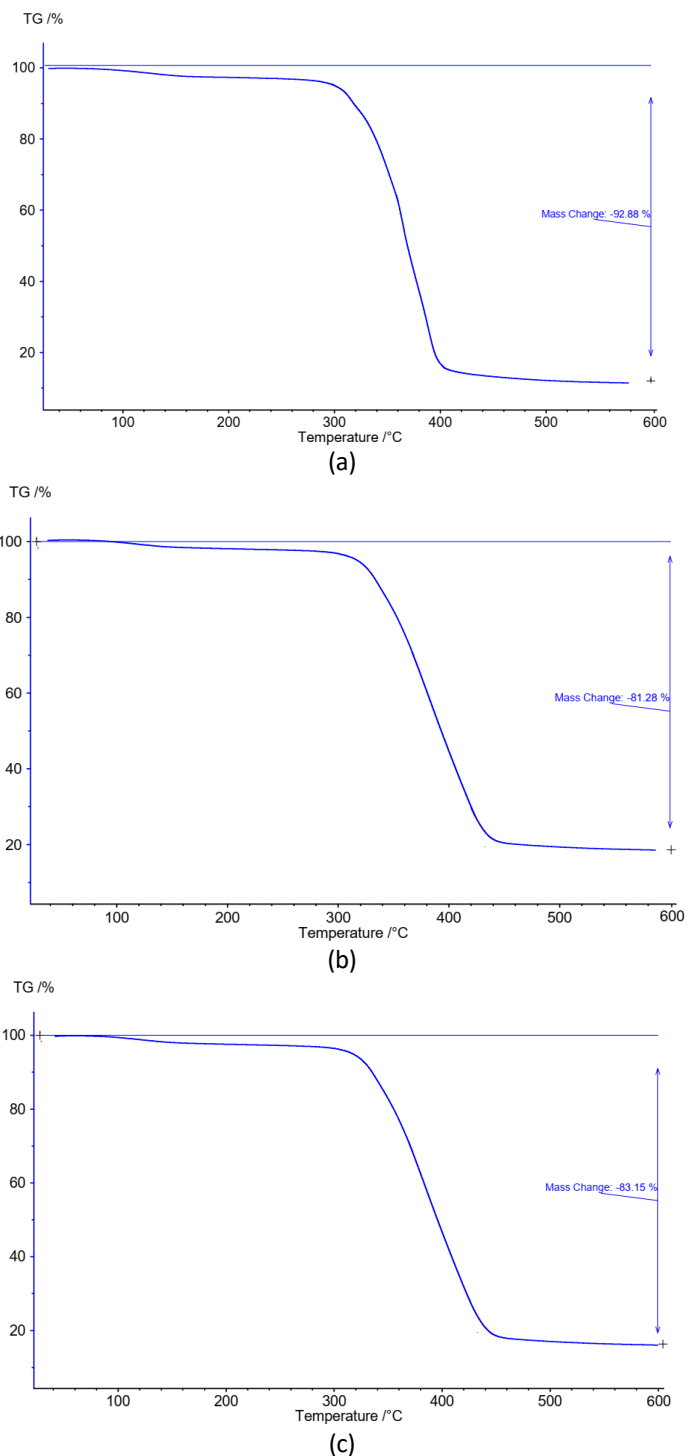


**Fig. 3.** EDX of (a) K-153A and (PPGDE) mixture (b) polymer composite with 2 wt% GO  
(c) polymer composite with 1.5 wt% nanosilica

It was shown that in Figure 5b the GO particles has appeared so the percentage of C was increased, meanwhile, on Figure 5c the nanosilica particles appeared so the percentage of O was increased. Results explained for the presence of GO and nanosilica particles in the polymer composites. These results also confirmed that the presence of GO and nanosilica particles had significantly improved the mechanical properties of produced polymer composites [9, 18, 22, 23].

### 3.4 Thermal resistance of polymer composite and nanocomposite

To study the thermal resistance of K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether (PPGDE) mixture, polymer composite with GO content of 2 wt% and polymer composite with nanosilica content of 1.5 wt%, Thermogravimetric analysis (TGA) was used. The TGA analysis was carried out in the nitrogen atmosphere with a heating rate of 10 °C/min. from room temperature to 600 °C. TGA results were shown as in the following in Table 3 and Figure 6.



**Fig. 6.** TGA of K-153A and (PPGDE) mixture (b) polymer composite with 2 wt% GO  
(c) polymer composite with 1.5 wt% nanosilica

**Table 3**  
Effect of nanosilica on thermal resistance of coating

Samples	Weight loss (%)		
	300 °C	400 °C	600 °C
K-153A/ PPGDE mixture	5.36	82.42	92.88
Polymer composite with 2 wt% GO	3.25	52.16	81.28
Polymer composite with 1.5 wt% nanosilica	3.66	53.78	83.15

The results presented in Figure 6 and Table 3 shown that in comparison with K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether (PPGDE) mixture, the thermal resistance of polymer composite with 2 wt% GO and Polymer composite with 1.5 wt% nanosilica had increased remarkably. K-153A epoxy resin and Poly(propylene glycol) diglycidyl ether (PPGDE) mixture has ash content of 7.12 %, meanwhile, with 2 wt% of GO, ash content of polymer composite was 18.72 and with 1.5 wt% of nanosilica this value was 16.85. The values of polymer composites were much higher than those of the mixture. Thus, GO and nanosilica had totally improved thermal resistance of produced polymer composite [19-21]. This can be explained that nanosilica particles had efficiently worked as a “fence” to protect polymer composite’s structure from heat and acted as an agent to prevent the decomposition of polymer chain leading to the higher thermal resistance. In addition, under the thermal decomposition of nanosilica would be coked to form a stable structure like ceramic. The role of nanosilica to enhance thermal resistance of polymer composite had been mentioned before [8, 20, 23, 24]. For the data in reference [8] ash content of K-153 was 5.19 % that was lower than that of nanocomposite with 1.2 wt % of nanosilica, up to 13.91 %. For nanocomposite, nanosilica was coked to form ceramic structure.

GO had improved thermal resistance of polymer composite. It may be explained that in high temperature conditions, polymer’s chains were to form the lower molecular polymer formula. Meanwhile, GO particles prevented penetration of heat into polymer’s chains so polymer composite with GO had a higher heat resistance than K-153A/ PPGDE mixture. Similar to nanosilica, thermal decomposition of GO would be coked to form a stable structure like ceramic in polymer composite, it worked as a fence to protect material from heat [7, 17, 23, 25]. This result was expressed in previous investigation [6], in which, GO thermal stability of epoxy resin had been improved. The degradation of pure epoxy was much higher than that of GO/EPs. Onset degradation temperature of modified epoxy was slightly grown, and the Tmax (the temperature at which the resin losses 50 % weight percent) of GO/EPs increased about 9 to 14 °C in comparison with that of pure epoxy. High specific surface GO acted as a barrier to prevent oxygen from impacting with polymer’s chains so thermal stability of GO/EPs was enhanced.

#### 4. Conclusions

Graphene oxide particles strongly influence to physical state and viscosity of K-153A/Poly(propylene glycol) diglycidyl ether mixture. GO improved surface hardness and abrasion resistance of polymer composite, in which surface hardness increased from 69 Shore D to 88 Shore D (about 28 %) and abrasion resistance reduced from 0.75 cm<sup>3</sup>/ 1.61 km to 0.45 cm<sup>3</sup>/ 1.61 km (about 67 %) when GO content increased to 2 wt%. Besides that, at 400 °C thermal resistance of polymer composite has been improved about 160 % with GO, weight loss of polymer composite was 52.16 and of 82.42 for K-153A/ PPGDE mixture and ash content has increased about 260 %, that value of polymer composite was 18.72 %, meanwhile, 7.12 % was for K-153A/ PPGDE mixture. GO content of 2 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%.

Nanosilica has many effects to physical state and viscosity of K-153A epoxy resin and poly(propylene glycol) diglycidyl ether mixture. Nanosilica enhanced surface hardness of nanocomposite that value increased from 69 Shore D to 84 Shore D (about 22 %), meanwhile, abrasion resistance of polymer composite reduced from 0.75 cm<sup>3</sup>/ 1.61 km to 0.42 cm<sup>3</sup>/ 1.61 km (about 80 %) when nanosilica content reached to 1.5 wt%. In addition to that thermal resistance of polymer composite has been improved about 155 % with nanosilica at 400 °C, weight loss of polymer composite was 53.78 and of 82.42 for K-153A/ PPGDE mixture, ash content has increased about 240 %. Ash content of polymer composite was 16.85 %, and that value of K-153A/ PPGDE mixture was 7.12 %. Considering other properties of polymer composite, nanosilica content of 1.5 wt% is suitable for manufacturing 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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