

Düzce University Journal of Science & Technology

Research Article

Thermo-mechanical behaviours investigation of Nano-Sized Al₂O₃, TiO₂, and Graphene Nanoplatelet Reinforced Epoxy Composites

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ABSTRACT

Composite materials have been frequently used for extensive applications in various industries, thanks to their remarkable properties. These sectors include energy, maritime, motor sports, aviation, space and defense. The materials commonly used in these sectors are fibre reinforced plastic (FRP) composite materials. Epoxy materials are suitable for the production of FRP materials. This study delves into the enhancement of epoxy-based nanocomposites by using graphene nanoplatelets (GNP-5nm), TiO₂ (13nm), and Al₂O₃ (8nm) nanoparticles. These nanoparticles were added at varying mass ratios into a commercial epoxy to investigate their effects on some chemical, thermal and mechanical properties. Meticulous mixing methodologies were used to reduce clumping effects and ensure even distribution during the process. The curing process was carried out in a PLC (Programmable Logic Controller) controlled hot air oven under isothermal conditions under the influence of 100 °C for 30 minutes. Tensile strength, elongation at break, toughness, resilience modulus, elasticity modulus, hardness, FTIR analysis and thermal conductivity properties were characterized to assess the nanoparticle influence on the epoxy matrix. The results showed that there were remarkable improvements in mechanical properties with nanoparticle reinforcement. Especially, 1.25% Al₂O₃ inclusion exhibited a substantial increase of 140.32% in tensile strength and a 7% rise in shore D hardness compared to pure epoxy. This enhancement was attributed to enhanced O-H bonding between 'O' atoms in Al₂O₃ nanoparticles and epoxy polymer chains, enhancing matrix-filler interactions. Additionally, the effect of 1.0% TiO₂ led to plasticity, displaying a 32% rise in elongation at break, signifying improved deformation energy absorption compared to neat epoxy. In thermal conductivity measurements, the highest thermal conductivity was observed in the sample with 1.25% GNP added and it increased by 123.5% compared to neat epoxy. In TiO₂ and Al₂O₃ added samples, an increase of 69% and 47%, respectively, was observed at 1.25% additive rates compared to neat epoxy. According to the results, thanks to the nanoparticle reinforcement added into the epoxy matrix, composite structures can be given new and superior properties specific to the applications.

Keywords: Epoxy resin, Al₂O₃, TiO₂, GNP nanoparticles

Nano Boyutlu Al₂O₃, TiO₂ ve Grafen Nanoplatelet Takviyeli Epoksi Kompozitlerin Termo-mekanik Davranışlarının İncelenmesi

ÔΖ

Kompozit malzemeler, dikkate değer özellikleri sayesinde çeşitli endüstri alanlarında geniş uygulama alanlarına sahiptir. Bu sektörler arasında enerji, denizcilik, motor sporları, havacılık, uzay, savunma gibi sektörler yer almaktadır. Bu sektörlerde yaygın olarak kullanılan malzemeler fiber takviyeli plastik (FRP) kompozit malzemelerdir. Epoksi malzemeler FRP malzemelerin üretiminde matris olarak yaygın olarak kullanılmaktadır. Bu çalışma, grafen nanoplateletler (GNP-8nm), TiO₂ (13nm) ve Al₂O₃ (5nm) nanopartikülleri kullanılarak epoksi bazlı nanokompozitlerin geliştirilmesini araştırmaktadır. Çalışmada nanopartiküller, bazı kimyasal, termal ve mekanik özellikler üzerindeki etkilerini araştırmak için ticari bir epoksiye kütlece farklı oranlarında eklenmiştir. İşlem sırasında topaklanma etkilerini azaltmak ve eşit dağılım sağlamak için titiz karıştırma metodolojileri kullanılmıştır. Kürleme işlemi PLC (Programlanabilir Mantıksal Denetleyici) kontrollü sıcak hava fırınında izotermal koşullar altında 100°C sıcaklık etkisi altında 30 dakika süreyle gerçekleştirilmiştir. Epoksi matris üzerindeki nanoparçacık takviyesinin etkisini değerlendirmek için çekme mukavemeti, kopma uzaması, tokluk, esneklik modülü, elastikiyet modülü, sertlik, FTIR analizi ve termal iletkenlik özellikleri karakterize edilmiştir. Bulgularda nanopartikül ilaveleriyle mekanik özelliklerde dikkate değer gelişmelerin olduğu görülmüştür. Özellikle, %1,25 Al₂O₃ katılımı, saf epoksi ile karşılaştırıldığında çekme mukavemetinde %140,32'lik önemli bir artış ve Shore D sertliğinde %7'lik bir artış sergilemiştir. Bu gelişme, Al2O3 nanopartiküllerindeki 'O' atomları ve epoksi polimer zincirleri arasındaki gelişmiş O-H bağlarına atfedilerek matris-dolgu etkileşimlerini güçlendirdi. Ek olarak, %1,0 TiO₂'nin eklenmesi plastisiteve yol açarak kopma uzamasında %32'lik bir artış gösterdi; bu da saf epoksi ile karsılaştırıldığında daha iyi deformasyon enerjisi emilimi anlamına geliyordu. Isıl iletkenlik ölçümlerinde en yüksek ısıl iletkenlik %1,25 GNP ilaveli numunede gözlendi ve saf epoksiye göre %123,5 arttı. TiO₂ ve Al₂O₃ katkılı numunelerde saf epoksiye göre %1,25 katkı oranlarında sırasıyla %69 ve %47 artış gözlenmiştir. Sonuçlara göre epoksi matris içerisine eklenen nanopartikül takviyesi sayesinde kompozit yapılara uygulamalara özel yeni ve üstün özellikler kazandırılabilmektedir. Termal iletkenlik ölçümlerinde ise en yüksek termal iletkenlik %1.25 GNP katkılı numunede gözlenmiştir ve saf epoksiye kıyasla %123.5 artış sağlamıştır. TiO₂ ve Al₂O₃ katkılı numunelerde ise %1.25 katkı oranlarında, saf epoksiye göre sırası ile %69 ve %47 artış gözlenmiştir. Elde edilen sonuçlara göre, epoksi matris içerisine eklenen nanoparçacık takviyesi sayesinde kompozit yapılara uygulamalara özel yeni ve üstün özellikler kazandırılabilmektedir.

Anahtar Kelimeler: Epoksi reçine, Al₂O3, TiO₂, GNP, nanoparticle

Received: 19/01/2024, Revised: 27/03/2024, Accepted: 03/04/2024

I. INTRODUCTION

Composite materials, structures composed of one or more materials merging to form structures with superior properties, are referred to as composite materials. Composite structures consist of two fundamental components: the matrix and the reinforcement phase[1]. One of the most commonly used matrix materials belongs to the thermoset polymer group, which includes epoxies [2], [3]. Epoxy resins are widely employed due to their outstanding mechanical, thermal, and electrical properties. However, cured epoxy materials possess cross-links formed during the curing process between hardener and epoxy groups, making the material more brittle [4].

Various filler materials are utilized to improve the properties and eliminate the brittleness of cured epoxy materials. Filler materials are classified into two groups: fiber reinforcement and particle

reinforcement [5]. Recent studies on epoxy polymers mainly involve applications utilizing particle reinforcements [6]. Better results in enhancing epoxy polymer properties have been achieved by obtaining for nano-sized particles instead of micro-sized ones. The decrease in particle size leads to an increase in the surface area within the mixture, forming better interfacial interactions. Combinations of epoxy-nano fillers have shown good improvements in material properties such as fracture toughness, coefficient of thermal expansion, and resistance to cutting and compression. For the industrial applications, the addition of nano additives to epoxy significantly influences the matrix phase to acquire a more rigid structure. A stiffer matrix distributes applied loads more homogeneously within the structure, enhancing resistance against cracking [7], [8]. The post-curing mechanical properties of epoxy relates to many variables, including the type of nano additives, compatibility with the epoxy-hardener system, the rate of homogeneous distribution within the matrix, interfacial interactions, and curing methods [8-9].

Among nano materials compatible with epoxy, graphene-based particles, Al_2O_3 (alumina), and TiO_2 stand out for their superior properties. Graphene and its derivatives, possessing two-dimensional carbon content, have been widely used in numerous applications due to their excellent mechanical, electrical, and thermal properties in enhancing the strength of composite materials [10]. Epoxy materials are generally known as polymers with brittle properties. With the use of nano particle reinforcements, this amount of brittleness is reduced and a certain amount of toughness can be provided to the material. In this sense, there are many studies on the use of nanoparticle reinforcement in different types, sizes and contribution rates into the epoxy matrix. Studies have been reported that the addition of alumina to epoxy enhances properties such as tensile-fracture strength, wear resistance, and thermal conductivity. Nanoparticles reinforced within the epoxy matrix provide improvements depending on the area of use. The ability of materials to eliminate instantaneous thermal loads to which they are exposed and to quickly distribute these thermal loads also plays an important role in the mechanical strength of the material [11-12]. TiO₂ stands out in recent research due to its high values in hardness, fracture, corrosion resistance, low cost, and additional attributes like bacterial resistance [13].

In a study by Aradhana et al. obtained by mass variable ratios of graphene ranging from 0% to 1.0%, 0.5% additive ratio showed approximately as 31% increase in tensile strength compared to pure epoxy [14]. Zandiatashbar et al. used graphene additives at the ratios of by mass 0.1-0.3-0.5 then observed a significant enhancement in material mechanical properties [15]. Mirmohseni and Zavareh used TiO₂ nanoparticles to increase tensile strength without reducing impact resistance while observing increases of 64% and 168% in tensile and impact resistance, respectively [16]. Kaushal Kumar et al. incorporated TiO₂ in sizes of 30-40 nm at the ratios of by mass 5-10-15% as noting increase of 22.38% in tensile strength and 37.5% in mechanical strength at a 10% additive ratio [17]. Hamid Rıza Salehi observed a 7.8% increase in tensile strength by employing TiO₂ at ratios of by mass 0.25-0.5 and 1% [18]. Fahamsyah et al. used of by mass 0-10% Al₂O₃ then obtained decrease in tensile, fracture, and hardness values with increasing additive ratios [19].

The purpose of this current study is to investigate the thermal and mechanical properties of a commercially used epoxy resin by reinforcing nanoparticles at certain rates by mass. TiO₂ (13nm), Al2O3 (5nm), GNP (8nm) nanoparticles were added at a rate of 0.75-1.0-1.25 percent by mass into the epoxy system consisting of Epikote MGS L285 epoxy and $100/40 \pm 2gr$ MGS LH285 hardener provided for this purpose, and they were cured at 100 degrees. After the curing processes, tensile test, hardness test, FTIR (Fourier Transform Infrared Spectroscopy) analysis and thermal conductivity measurements of the samples were carried out. Besides, SEM (Scanning Electron Microscope) images were analysed both the nanoparticles before curing and the samples prepared by mixing them with epoxy after curing.

II. MATERIALS AND METHODS

In this study, a commercial Epikote MGS L285 epoxy and $100/40 \pm 2$ gr MGS LH285 hardener were used. The properties of the epoxy system are given in Table 1.

	EPIKOTETM MGS L285	MGS LH285
Equivalent weight (g/g)	155-170	-
Density (g/cm ³)	1.18-1.23	94-0.97
Amine value (mgr KOH/gr)	-	480-550

Table 1. The properties of the epoxy system.

In addition to the epoxy matrix structure, nano-sized Al_2O_3 , TiO_2 and GNP powders were added in certain proportions. The mass ratios of the particles selected and used in the study were decided by considering the studies given in the relevant introduction. These ratios are given in the Table 2 based on weight fraction.

Table 2. The properties of the epoxy system

	Al ₂ O ₃	GNP	TiO ₂
Particle reinforcement rates (% by weight)	0.75	0.75	0.75
	1	1	1
	1.25	1.25	1.25

A. PREPERATION OF THE SPECIMENS

The mixtures were prepared by subjecting them to mechanical mixing for 15 minutes and magnetic mixing for 5 minutes at room temperature in order to ensure homogeneous distribution of nanoparticles into the resin. In the next stage, the mixtures were kept in an ultrasonic bath for 15 minutes to remove air bubbles and were mixed with the hardener at a stoichiometric ratio (100: 40 ± 1) at room temperature. Schematic view of the mixing stages of the components are given Figure 1.



Figure 1. The Schematical view of preparation of the mixtures.

B. EXPERIMENTAL TEST SETUP

In experimental studies, mixtures were first prepared as described in the sample preparation phase. The prepared mixtures were poured into glass molds with a 4 mm thick cavity. Then, they were kept in the oven (UTEST/UTD-1305) for 30 minutes to cure at 100 °C. After the curing process epoxy plates were kept in the molds for potential spring back or distortions at room temperature. Then the completely cured rectangular shaped plates were removed from the mold. Later on, plates were cut with a CNC router according to the dimensions of the test type to be applied.

In order to determine the certain mechanical and thermal properties of the samples, tensile test (TS EN ISO 527-1), hardness test (Shore D ASTM D2240), FTIR (chemical wavelength guide) analysis and thermal conductivity tests (ASTM D7984) were carried out.

In addition, SEM imaging was performed to determine the nanoparticle sizes. The gold coating was applied to the surface for better images since the structure is polymer. The SEM imaging was set and captured images powder particles are given in Figure 2.



Figure 2. The view of SEM imaging set and captured images.

III. RESULTS AND DISCUSSION

In the study, the thermo-mechanical properties of nanoparticles were incorporated into the epoxy matrix were examined. The results are given under separate headings in this section.

A. TENSILE TEST RESULTS

The tensile test results obtained are presented in the following graphs and tables. When the graphs given in Figure 3 and Figure 4 are evaluated, tensile strength of neat epoxy was measured higher in Al_2O_3 and TiO_2 nanoparticles types at 0.75% and 1.0% contribution rates. In addition, the highest tensile strength values were measured in Al_2O_3 . In the combination of epoxy matrix and TiO_2 , polymer chain covalent bonding occurs with Van der Waals forces, thanks to the Ti molecules in the structure. These bonds provide a stronger structure [20].

They were also observed and presented in FTIR analysis results. On the other hand, although an increase in the tensile strength, breaking strength and load carrying capacity of the composite structure was observed with the TiO_2 additive compared to pure epoxy, it was as 3% lower compared to Al_2O_3 listed in Table 3.



Figure 3. Stress-strain graph according to nanoparticle type at 0.75% percentage by mass.



Figure 4. Stress-strain graph according to nanoparticle type at 1.0 % percentage by mass

Contrary to Al_2O_3 and TiO_2 additives decrease in tensile strength, then maximum load values have been reached for GNP additives. Similar situations have been recorded in previous studies in this field [21], [22]. This situation can arise from the weak and brittle H-C bonds formed between the "H" atoms in the polymer chains and the "C" atoms in the GNP nanoparticle [23].

When the stress-strain curve in Figure 5 is evaluated, the highest tensile strength value was reached in the samples modified with Al_2O_3 nanoparticles at 1.25%. It is due to the O atoms present in the Al_2O_3 nanoparticles form strong hydrogen bonds with the polymer chains of the epoxy matrix [24]. By

increasing the amount of Al_2O_3 in the epoxy and ensuring its homogeneous distribution, the distance between the polymer chains decreases and atomic movements are minimized. In this way, matrix-filler interaction increases and stronger bonds are formed [25].



Figure 5. Stress-strain graph according to nanoparticle type at 1.25 % percentage by mass

Mechanical properties of the specimens such as tensile strength, elongation at break, toughness, modulus of elasticity, modulus of elasticity are given in Table 3.

Particle reinforcement rates (% by weight)	Specimen	Max. Force (N)	Tensile Stress (MPa)	Breaking Stress (MPa)	Breaking Elongation (mm)	Elongation (mm/mm)
Neat	Epoxy	862.70	24.65	19.97	5.86	0.0732
	Al ₂ O ₃	2247.30	54.39	53.99	5.82	0.0728
0.75	TiO ₂	1807.56	45.16	46.53	6.12	0.0954
	GNP	985.30	26.63	25.66	3.61	0.0453
	Al_2O_3	2346.46	56.82	45.99	9.75	0.1225
1.0	TiO ₂	1812.41	48.35	40.25	16.40	0.2051
	GNP	523.00	13.99	13.98	1.46	0.0181
	Al ₂ O ₃	2446.87	59.24	59.07	9.45	0.1182
1.25	TiO ₂	1874.82	50.16	44.82	14.01	0.1751
	GNP	638.00	17.05	17.01	2.78	0.0351

Table 3. Mechanical properties of the specimens

B. HARDNESS TEST

Figure 6 shows the effect of nanoparticle type on hardness according to additive ratios. It can be clearly seen that the hardness values increased with the increase in the amount of nanoparticle additives in the hardness analysis. This can be attributed to the increase in the intermolecular interactions due to the increase in mixture concentration.



Figure 6. Hardness test results of the specimens.

C. THERMAL CONDUCTIVITY MEASUREMENTS

In the literature study, it was observed that nanoparticles added into epoxy increased the thermal conductivity of the structure, GNP contribution was more effective in improving the thermal conductivity of neat epoxy, followed by TiO_2 and Al_2O_3 , respectively [26-28].

As seen in Figure 7, the thermal conductivity value increased with increasing contribution rate, regardless of the nanoparticle type. The highest thermal conductivity was observed in the 1.25% GNP added sample and a 123.5% increase was achieved compared to pure epoxy. This increase value obtained is 3.5 times higher than the literature[29, 30]. 1.25 % of TiO₂ and Al₂O₃ added samples, an increase of 69% and 47% respectively compared to pure epoxy. 37% and 24% higher results were obtained for TiO₂ and Al₂O₃ respectively according to related studies [31, 32].



Figure 7. Thermal conductivity values of specimens.

D. SEM IMAGES

Figure 8 shows the SEM and EDS analysis results of Al_2O_3 , TiO_2 and GNP added samples. In the SEM images obtained, globular formations were observed in Al_2O_3 , plates in GNP and spheroid formations in TiO₂. It was observed that the data obtained was compatible with the images given in the literature [33]. Pore formation in the Al_2O_3 and TiO₂ doped structure is clearly seen in the SEM images. This detected porous formation is shown in the literature as evidence that an effective matrix/filler interaction occurs and that the nanoparticle distribution in the resin is homogeneous. [34]. Accordingly, under the specified curing conditions, the TiO₂ additive showed a more homogeneous bond with the epoxy and, accordingly, it is thought to have better mechanical strength due to formations with filled pores improve the mechanical properties of the structures [35].

The addition of Al_2O_3 into epoxy, the formation of filled pores was observed in small numbers and in certain regions. Smooth surfaces in SEM images can be sign of the the weakness of the interaction between filler/matrix. [36]. It has been determined that GNPs added to epoxy remain in the form of plates within the structure and are less dispersed in the epoxy as stated in the literature [37].

Figure 8 shows the EDS results for three nanoparticles. Looking at these results, C and Cl peaks were observed in pure epoxy as well as in all nano-filling materials, while trace amounts of O, Au and Pd were detected in their structures. In addition to these results, a high amount of Al was detected in Al_2O_3 and a high amount of Ti was detected in TiO_2 . While the lowest O rate was observed in TiO_2 , O was measured at similar rates in GNP and pure epoxy.



Figure 8. SEM and EDS analysis results of Al₂O₃, TiO₂ ve GNP added samples a) Al₂O₃ b) GNP c) TiO₂ d) Neat epoxy.

E. FTIR ANALYSIS

Data's regarding the peaks detected in the FTIR spectrum are shown in Table 4. When these absorption bands are evaluated, vibrations due to intense C-H and N-H bonding are observed in the samples with 0.75% and 1.0% GNP additive, while vibrations due to C-H bonding in the 1.25% GNP additive are noteworthy. As the GNP contribution increased, voltage vibrations due to C-H bonding increased.

Specimen	Wavelength (cm ⁻¹)	Vibration type
%0.75	1080.732 1241.328	C-C / C-O / C-N
	2901.114	C-H
	3165.923	О-Н / С-Н
	3244.988	O-H
	3394.306	N-H
	3476.437	11-11
	1080.580	C-C / C-O / C-N
	1241.828	
	2884.317	C-H
	3066.970	0-11
%10	3170.098	0-H / C-H
/01.0	3242.415	
	3264.092	O-H / N-H
	3398.928	
	3456.571	N-H
	3473.324	
% 1.25	1241.672	C-C / C-O / C-N
	2898.23	C-H
	3397.752	N-H

Table 4. Data on the peak points of GNP-doped samples detected in the FTIR spectrum.

The peaks detected in the FTIR spectrum are shown in Table 5. When these absorption bands are evaluated voltage vibrations due to intense O-H bonding are noteworthy at all doping ratios. While N-H bonds accompanied the tension vibrations due to O-H bonding in the structure with 0.75% Al₂O₃ contribution, tension vibrations due to C-H bonding were observed at 1.0% and 1.25% contribution rates.

Specimen	Wavelength (cm ⁻¹)	Vibration type	
-	1244.497	C-C / C-O / C-N	
	1609.875	C=C / C=O / C=N	
	2958.748	С-Н	
%0.75	3114.407		
	3128.589	0-п/С-п	
	3290.354	N II	
	3450.731	IN-П	
%1.0	1241.459	C-C / C-O / C-N	
	1605.150	C=C / C=O / C=N	
	2892.803	С-Н	
	3142.648	O-H / C-H	
	3245.914	О-Н	
% 1.25	1241.887	C-C / C-O / C-N	
	1606.584	C=C / C=O / C=N	
	2908.987	С-Н	
	3111.109	O-H / C-H	
	3283.234	N-H	

Table 5. Data on the peak points of Al_2O_3 -doped samples detected in the FTIR spectrum.

When the bond structures in Table 5 are evaluated, the cross-bonding model expected to occur between Al_2O_3 nanoparticles and epoxy-hardener system is expected to be as in Figure 9.



Figure 9. Cross-link expansion model in Al_2O_3 +epoxy combination.

When these absorption bands are evaluated, at all doping ratios, tension vibrations due to C-H bonding within the structure, which increases in parallel with the doping ratio are significant. In addition, voltage peaks due to low amounts of O-H bonding were observed in addition to C-H bonds at 1.0% and 1.25% contribution ratios, and an N-H peak was observed in addition to O-H and C-H peaks at 1.25% contribution ratios.

Specimen	Wavelength (cm ⁻¹)	Vibration type	
	1244.497	C-C / C-O / C-N	
	1609.875	C=C / C=O / C=N	
	2958.748	C-H	
%0.75	3114.407		
	3128.589	0-н/С-н	
	3290.354	NL	
	3450.731	IN-H	
%1.0	1241.459	C-C / C-O / C-N	
	1605.150	C=C / C=O / C=N	
	2892.803	C-H	
	3142.648	O-H / C-H	
	3245.914	O-H / N-H	
% 1.25	1241.887	C-C / C-O / C-N	
	1606.584	C=C / C=O / C=N	
	2908.987	C-H	
	3111.109	O-H / C-H	
	3283.234	N-H	

Table 6. Data regarding the peak points of TiO_2 doped samples detected in the FTIR spectrum.

When the bond structures in Table 6 are evaluated, the cross-bonding model expected to occur between Al_2O_3 nanoparticles and epoxy-hardener system is expected to be as in Figure 10.



*Figure 10. Cross-link expansion model in TiO*₂+*epoxy combination.*

IV. CONCLUSION

In this study, the effect of nanoparticle additives on the mechanical properties and curing kinetics of epoxy-based polymer composites was examined together and a new perspective was gained for studies in this field. Below are some highlights and conclusions obtained from the results and suggestions to further improve this study.

With the addition of 1.25% Al₂O₃, the tensile stress and breaking stress of epoxy increased by 140.3% and 195.8%, respectively. The main reason for this increase is due to the strong O-H bonds formed between alumina nanoparticles and the epoxy polymer matrix. On the other hand, the highest elongation at break was measured in the 1% TiO₂ added sample, which showed a 179.9% increase compared to pure epoxy. It is the main reason for the observed increase in TiO₂ contribution is that TiO₂ molecules trigger the cross-linking between the epoxy matrix and polymer chains through Van der Waals interactions.

GNP additive reduced the epoxy tensile strength by 35% due to the formation of H-C bonds between the polymer chains through the interaction between the hydrogen atom of the epoxy and the carbon atoms of GNP. This situation made the epoxy material brittle and its elongation at break decreased.

Elasticity modulus increased for all particle types and additive ratios. The highest modulus value was reached with the addition of 0.75% TiO₂, which increased by 151% compared to pure epoxy. In the hardness measurements, hardness values increased compared to pure epoxy for all particle types and additive ratios. Hardness values with Al₂O₃, TiO2 and GNP additives increased by 7%, 6% and 3.8%, respectively, compared to neat epoxy. The highest thermal conductivity was observed in the 1.25% GNP added sample and a 123.5% increase was achieved compared to neat epoxy.

<u>ACKNOWLEDGEMENTS</u>: This work is supported by Düzce University Scientific Project (Project no: 2019.06.05.1025).

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