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## 3D-printed nanocomposites filled with untreated and surface-modified PTFE powders treated by a Na-naphthalene-system

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### ABSTRACT

This study focuses on the mechanical properties of DLP/LCD-type 3D-printed nanocomposites comprised of polyester acrylate resin with DPGDA reactive diluent filled with untreated PTFE and surface-modified PTFE powders by the Na-Naphtalenide system. To obtain the nanocomposites, untreated and surface-modified PTFE powders were incorporated into the resin systems at loading ratios ranging from 1% to 6%. The X-ray photoelectron spectroscopy (XPS) data following the Na-naphthalene system treatment demonstrated the existence of functional groups such as OH, carbonyl, and C=C unsaturation groups on the surface of the untreated PTFE powders. The study showed improvements for the nanocomposites obtained through a DLP/LCD type 3D printer up to a certain ratio in terms of tensile strength, Young's modulus, Izod impact resistance, and Shore D hardness values. Evaluating the promising samples, the nanocomposites with surface-modified PTFE powders of 2% and 1% showed increases of 5.1% and 7.6% in ultimate tensile strength and Izod impact resistance compared to the unfilled polyester acrylate sample. On the other hand, the nanocomposite with untreated PTFE powders of 1% only showed increases of 2.4% and 3.2% in ultimate tensile strength and Izod impact resistance. Moreover, Young's modulus showed less decrease for surface-modified PTFE-filled nanocomposites.

## I. INTRODUCTION

Recent years have seen a surge in research into new formulations as the need for high-performance composite materials in additive manufacturing grows [1-3]. With its adjustable qualities and good processing characteristics, polyester acrylate (PEA) has become a potential resin system for 3D printing when combined with dipropyl glycol diacrylate (DPGDA) reactive diluent [4]. Nonetheless, the investigation of particular additives is driven by the need for even greater improvements [5]. In this work, untreated PTFE powders and surface-modified PTFE powders modified with Na-Naphtalenide (m-PTFE) were combined with DPGDA reactive diluent to create an innovative polyester acrylate resin. The loading ratios of these powders ranged from 1% to 6%, making it possible to compare how each one affected the mechanical characteristics of the final 3D-printed composites. Untreated PTFE has been shown to improve impact resistance, wear resistance, and thermal stability when added to polymer matrices because of its unique characteristics, which include a high T<sub>g</sub> thermoplastic structure, a low friction coefficient, and a high thermal decomposition energy of C-F bonds [6,7]. These commercially available PTFE powders were used in our earlier study to modify the Na-Naphtalenide system [7]. This allowed us to obtain significant functional groups on the surface of the pure PTFE powders, including COOH, OH, and C=C [8-10]. These functional groups were identified by XPS analysis. The surface property of these m-PTFE powders was thought to offer improved mechanical properties. This is due to the fact that our earlier research [7] shown significant gains in tensile strength, Young's modulus, and Izod impact resistance of 22%, 32%, and 95% when these treated PTFE powders were added to epoxy resin. The goal was to

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increase the interfacial strength and surface energy between the fillers and matrix because of their functional groups on the PTFE powder's modified surface. Because of their large interfacial surface area, porous fillers, like fume silica particles, have been employed in literature to enhance the mechanical characteristics of UV-curable systems [8-11]. Sadej and Andrzejewska's study [12] served as a literary example. Tensile strength increased by 81% with silica/aluminum oxide hybrid fillers in their study. UV-cured urethane acrylate nanocomposites were created by Lahijania et al. [13] by combining untreated and silane-treated nanosilica fillers into the resin. For the non-treated silica filled samples, they saw an increase in the modulus of elasticity of 55%; however, for the silane-treated silica filled samples, the increase was 88%. While the untreated silica-filled samples showed an increase of 91% in hardness, the silane-treated silica-filled samples showed an increase of 200% in hardness.

In the light of this information, the present study decided to use surface-modified PTFE powders by Na-Naphtalenide system in the concentration range of 0–6% for the PEA/DPGDA resin system in order to achieve enhanced mechanical properties by the usage of a DLP/LCD type 3D printer. These results were also compared to the untreated PTFE powder-filled PEA/DPGDA-based nanocomposites produced using a 3D printer of the DLP/LCD type. This research aims to systematically evaluate the mechanical properties of these novel composite materials, shedding light on the potential advantages and limitations associated with the introduction of untreated PTFE and surface-modified PTFE powders. The outcomes of this study not only contribute to the fundamental understanding of the synergies of untreated PTFE and surface-modified PTFE filling powders and PEA/DPGDA resin systems but also provide valuable guidance for optimizing these composite materials for specific applications in industries such as coatings, adhesives, and advanced materials [14,15]. In this context, undiluted polyester acrylate resin, which has a very high viscosity value, was diluted with DPGDA reactive diluent. The polyester acrylate resin that was used in this study has good flexibility, good reactivity, and good hardness. It exhibits moderate adhesion, good chemical resistance, and excellent abrasion resistance [16]. Moreover, DPGDA, which is the other component of the prepared resin system in this study, is a difunctional reactive diluent. It is generally used for applications that require improved flexibility, adhesion, and good moisture resistance [17,18]. This prepared PEA/DPGDA resin system was filled with commercial untreated PTFE powders with a mean particle size of 24 nm and surface-modified PTFE powders. These PTFE powders were surface-modified by the Na-Naphtalenide system, as mentioned. The mechanical properties of the nanocomposites that were produced from these PTFE powder-filled PEA/DPGDA resin systems, such as their tensile strength, Shore D hardness, Young modulus, tensile strain, and notched-Izod impact resistance, were investigated. In addition, the SEM morphologies of the fracture samples were observed following the tensile test.

## II. EXPERIMENTAL METHOD

### 2.1 Materials

The main commercial resin was EBECRYL® 884, an undiluted polyester acrylate resin. Excellent hardness, reactivity, and flexibility characterize it. It has also outstanding abrasion resistance, strong chemical resistance, and moderate stickiness. In plastics, EBECRYL® 884 is utilized. Allnex was the supplier of this primary resin. Reactive diluent, dipropylene glycol diacrylate (DPGDA), was acquired from BASF. Poly (tetrafluoroethylene) (PTFE) homopolymer powder, of which Teflon™ PTFE 7B is its trade name, was acquired from GLEMCO. 24

$\mu\text{m}$  is its mean particle size (ASTM D4894). The resins were cured by using a Phrozen sonic mini resin DLP/LCD type 3D printer. The photoinitiator utilized was bis(2,4,6-trimethylbenzoyl)-phenylphosphine oxide (BAPO) (IRGACURE® 819).

### *2.2 Surface Modification and Functionalization of PTFE Powders*

By adding metallic Na to a round-bottom flask fitted with a magnetic stirrer, naphthalene flakes were dissolved in one liter of THF at room temperature. For a while, this solution was stirred. This procedure led to the preparation of a bath for the PTFE modification. After adding the PTFE particles, this bath was mechanically stirred for five minutes while exposed to the atmosphere. Following the stirring procedure, the powders were rinsed and cleaned three times using distilled water and acetone. Following the washing procedure, the powders with surface modifications were vacuum dried for 24 hours at 80 °C. It was noted that the functionalized and surface-modified PTFE powders had a light brown color. The X-ray photoelectron spectroscopy (XPS) results of untreated PTFE and the surface-modified PTFE powders are given respectively in Figure 1 and 2.

### *2.3 Preparation of PTFE and surface-modified PTFE powders filled polyester acrylate/DPGDA resin and curing by DLP/LCD type 3D printer*

Commercial polyester acrylate (PEA) was used as the main resin. These resins were diluted by DPGDA. DPGDA decreased the viscosity of the main resins and increased the crosslinking density. Besides that, it was thought that the unique flexible structure of DPGDA would gain improved mechanical properties after the crosslinking reactions in the resin tank of the 3D printer. Each resin system was adjusted to 1/1 by weight with DPGDA. This resin system was mechanically stirred in a beaker for 10 minutes to achieve homogeneity. As filling materials, untreated commercial PTFE and surface-modified PTFE powders were used in the resin system. Surface modification of PTFE powders was carried out by the Na-naphthalenide system in our previous study. The presence of functional groups such as OH, carbonyl groups, and C=C unsaturation points on the surface of the PTFE powders was confirmed by X-ray photoelectron spectroscopy in our previous study [7], as shown in Figure 2. The concentration range of 0–6% was used to introduce these untreated PTFE and surface-modified PTFE powders into the resin systems. PEA and DPGDA were stirred at equal weight. After obtaining homogeneity, PTFE powders were added to the beaker. These powders in the resin system were stirred both mechanically and ultrasonically simultaneously for 45 minutes in a beaker. Therefore, the powders are uniformly distributed in the PEA/DPGDA resin system. Then BAPO photoinitiator at a weight concentration of 5% was added to the filled PEA/DPGDA resin system and stirred again mechanically and ultrasonically for 45 minutes. After obtaining a clear and uniform resin system, the 3D printer's resin tank was filled with this prepared resin system, and the nanocomposite products were generated by the curing of the digital light process in the 3D printer.

### 2.4 Characterization of Test Specimens

For every weight percentage, three samples were generated. For the results, the mean of these three values was employed. Based on the average outcomes, analyses were carried out. X-ray photoelectron spectroscopy (XPS) was used to measure the binding energies of atoms in order to describe the bonds on the surface of surface-modified PTFE particles. Young's modulus, ultimate tensile strength, and elongation at break were measured using standard tensile tests in accordance with ASTM D638 to describe the mechanical properties of the samples. A deformation speed of 5 mm/min was used for the tensile testing. Zwick Z010 equipment was used for the tensile test, which was conducted at room temperature. Notched samples were used to assess the IZOD impact resistance according to ASTM D 256. With a hammer of 5.4J and a striking rate of 3.96 m/s, Zwick B5113.30 equipment was used for this test. A Carl Zeiss Ultra Plus SEM (Scanning Electron Microscope) instrument operating at an acceleration voltage of 20 kV was used to study the fracture surface morphologies following the tensile test. Prior to SEM investigations, the specimens were coated with 2-4 nm of Au/Pd using a Quorum Q150R device in an ion beam sputtering system.

## III. RESULTS AND DISCUSSIONS

In this study, PTFE powders were exposed to surface modification treatments utilizing a Na-naphthalenide system. The resulting surface-modified powders were then mixed at ratios ranging from 0 weight percent to 6 weight percent with the PEA/DPGDA resin system. It was anticipated that PTFE would be included as a reinforcement component in the PEA/DPGDA resin system. These surface-modified PTFE powders' strong interactions with the PEA/DPGDA resin system would therefore make them more effective and matrix-compatible. Functional groups must be present on the surface of the surface-modified PTFE particles in order to generate strong interactions between the PEA/DPGDA matrix and the powders. XPS characterization provided the identity of these functional groups.

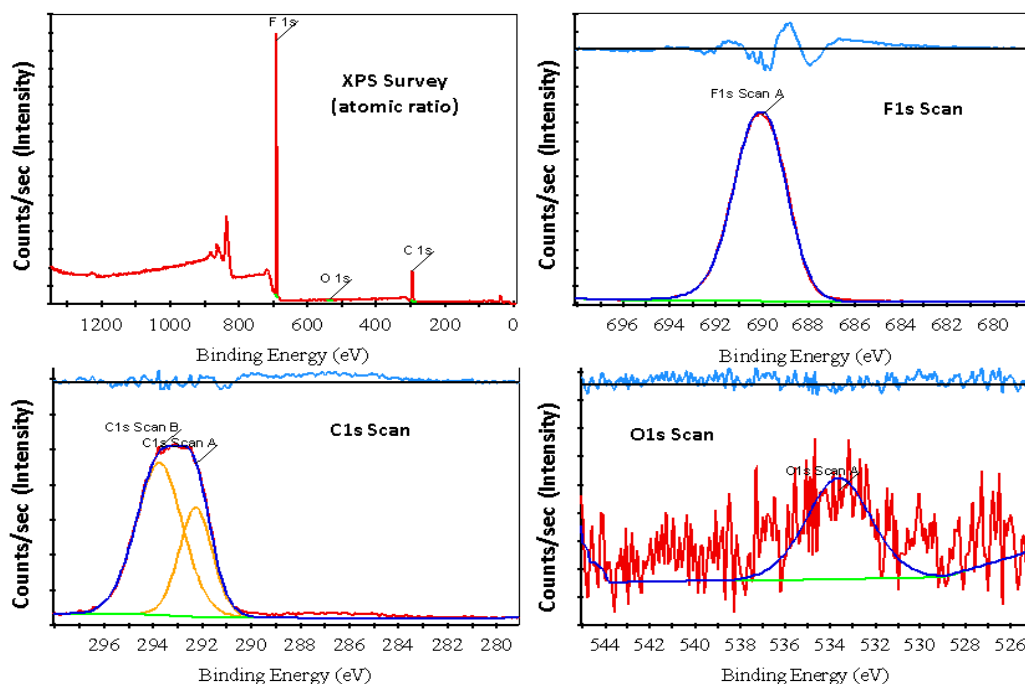


Figure 1. XPS examination of the surface of untreated PTFE powder

Considering the surface examinations of the untreated and Na-naphthalenide-etched PTFE powders as a whole, the XPS analysis substantially contributed to the determination of the functional groups on the polymer surface. These XPS peaks are shown in Figure 1 and 2. The C1s main peaks of the untreated PTFE were at 292.26 eV and 293.75 eV, which successively corresponded to  $-CF_2-$  and  $-CF_3-$  bonds. On the contrary, the surface-modified PTFE powders exhibited two major C1s peaks at 284.66 eV and 285.25 eV successively corresponding to sp<sup>2</sup> ( $-C=C-$ ) and sp<sup>3</sup> ( $-C-C-$ ) carbons. It was thought that the lower C1s peaks at 287.55 eV and 289.59 eV could be related to  $-C-CF_x-$  bonds and carbonyl groups. The F1s peak of the untreated PTFE powders was single and had a much higher intensity compared to that of the surface-modified PTFE powders. This peak was at 689.97 eV, which corresponded to  $-CF_2-$  bonds. The surface-modified PTFE powders also exhibited a small peak that corresponded to  $-CF-$  bonds besides  $-CF_2-$  bonds. This peak was at 686 eV [5-7,8].

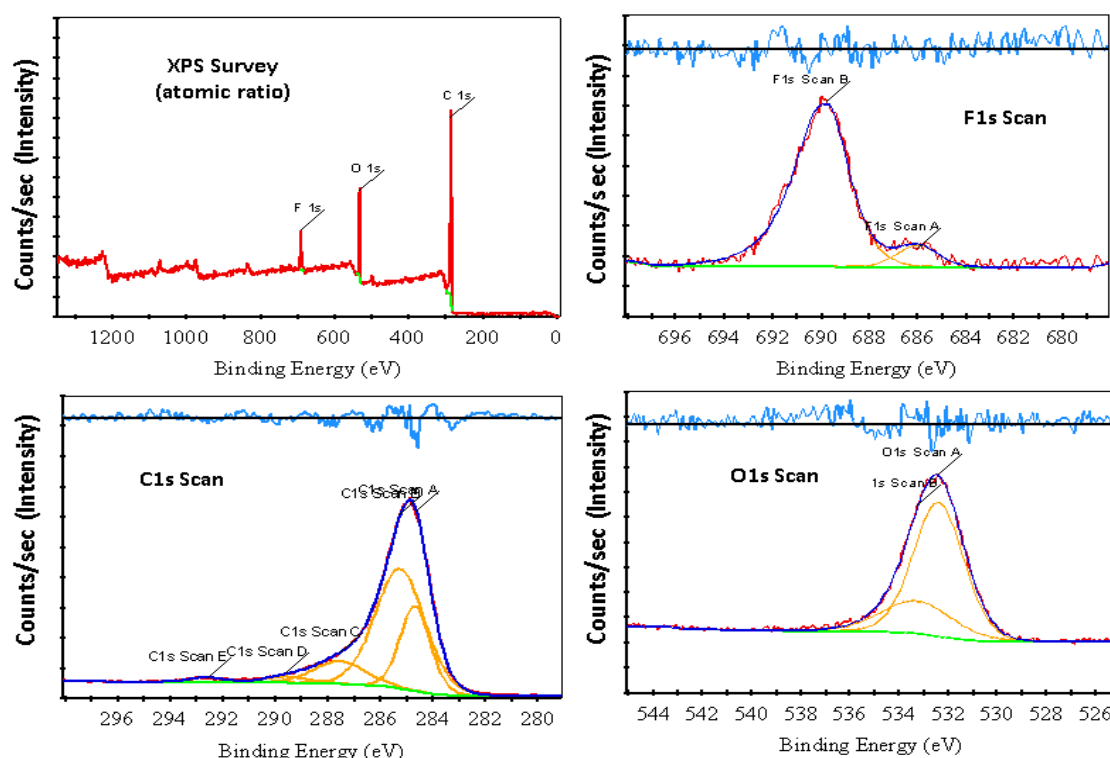


Figure 2. XPS examination of the surface of surface-modified PTFE powder

The O1s peak intensity of the untreated PTFE powders was negligibly low. It was thought that this could be due to contamination. The surface-modified PTFE powders exhibited two O1s peaks that had much higher intensities. It was thought that these peaks belonged to C-O and C=O groups that formed after the Na-naphthalenide system treatment. Moreover, the XPS curves that are given in Figures 1 and 2 provide the opportunity to compare the atomic ratios of F, C, and O between the untreated and surface-modified PTFE powders. The XPS results of the untreated PTFE powders showed fluorine and carbon atoms. Fluorine atoms had a much higher ratio than carbon, as expected. On the other hand, the XPS results of the surface-modified PTFE powders showed a much higher carbon ratio, and their fluorine ratio was substantially lower. Moreover, oxygen atoms became prominent and had a high ratio in the surface-modified PTFE powders. The order of the atomic ratios of the examined elements was as follows:  $C > O_2 > F$  [7-9,10].

The low fracture toughness property of UV- or light-curable acrylated resin systems is a very significant restriction for engineering applications in industrial sectors. The desired mechanical properties of these resin systems also need to be increased to higher levels for some engineering applications such as gears, cams, wheels, impellers, brakes, clutches, conveyors, transmission belts, bushes, and bearings. Various fillers, such as oxides, nitrides, carbides, solid lubricants, and short fibers, have been used in these systems to overcome these limitations [19-21]. In addition to these fillers, elastomers and soft segments, such as liquid rubbers, have also been used. These conventional fillers have provided improvements in certain properties [7,22]. For instance, while the strength or modulus values of the manufactured composites increase, their impact resistance values substantially decrease or do not show remarkable improvement, or vice versa. Furthermore, currently, more efficient functional fillers and reinforcement materials are available for applications to enhance overall mechanical properties. For this reason, it was decided to use surface-modified PTFE powders treated with a Na-naphthalenide system as the filling material. This was because poly (tetrafluoroethylene) has excellent heat resistance and wear resistance due to the C-F bonding dissociation energy and the low friction coefficient of fluorine atoms. Besides, PTFE has excellent thermo-mechanical properties above 150 °C [23,24]. However, PTFE has very low surface tension and poor compatibility with different matrices. To increase its compatibility and achieve strong bonding with the matrix, surface modification and functionalization processes are required [15,25]. This way, it will be feasible to obtain more favorable engineering properties such as mechanical strength, wear resistance, and heat resistance with surface-modified and functionalized PTFE particles. On the other hand, it should be kept in mind that PTFE also has high density besides its low surface tension. Hence, incorporating PTFE without surface modification and functionalization into a main resin or solution via mechanical stirring is very difficult. This is because these particles tend to collapse into the matrix resin [26,27]. Therefore, the surface modification of PTFE powders is becoming popular in attempts to obtain PTFE-filled homogeneous resins easily and achieve an easier process. The surface modification of PTFE has been carried out via various methods, such as chemical treatment, plasma treatment, irradiation treatment, corona discharge, flame treatment, and ozone treatment. Clearly, these methods have their own advantages and disadvantages. For example, plasma treatment and irradiation methods usually require expensive and complex equipment, while chemical methods have potential problems in terms of environmental pollution. Among them, the sodium naphthalene solution method has more merits, such as a simple process, low costs, and a good activation effect; thus, this method is widely used [26]. In addition to these, Benderly et al. [15] reported that a Na-naphthalene solution can be stored and intermittently used without an exhaust device or inert atmosphere, and the activated sample does not need high-temperature treatment. This is an important advantage in terms of practicality. A Na-naphthalenide system is also very effective in promoting the adhesion of fluoropolymers. Mechanical properties were investigated with tensile, Izod impact, and Shore D hardness tests for all filled nanocomposites. The stress-strain curves of the nanocomposites are given in Figure 3. Moreover, the obtained tensile, impact, and hardness test values are shown in Table 1. In the examinations of the samples, the increased PTFE and surface-modified PTFE powder content increased the maximum tensile strength, Izod impact resistance, and Shore D hardness values up to certain ratios. However, the Young's modulus values decreased with increasing the content of PTFE and surface-modified PTFE powders. The comparative trend of these mechanical values for each sample is given in Figure 4. Ultimate tensile strength, elongation at ultimate tensile strength, Izod impact resistance, and Shore D hardness increased up to a certain filler ratio for both filled nanocomposites. While this value was 1% for PTFE-

filled nanocomposites, 2% was the maximum ratio for surface-modified PTFE powder-filled nanocomposites. However, Young’s modulus values showed decreases for both PTFE and surface-modified PTFE powder-filled nanocomposites. The only thing to note is that surface-modified PTFE powder-filled nanocomposites exhibited higher Young’s modulus values compared to the PTFE powder-filled nanocomposites. This could be derived from the characteristic structure of PTFE. Surface modification of PTFE did not affect the rigidity. Evaluating all mechanical results, PEA-DPGDA-1% m-PTFE, PEA-DPGDA-2% m-PTFE, and PEA-DPGDA-1% PTFE samples have come forward in terms of improved mechanical properties. The ultimate tensile strength value of PEA-DPGDA-1% m-PTFE was 11.28 MPa and showed an increase of 5.1%. Moreover, Izod impact resistance was 9.6 kJ/m<sup>2</sup> and showed an increase of 5.4%. PEA-DPGDA-2% m-PTFE showed increases of 3.5% and 7.6% in ultimate tensile strength and Izod impact resistance, respectively. However, the Young’s modulus of PEA-DPGDA-1% m-PTFE and PEA-DPGDA-2% m-PTFE showed decreases of 6.5% and 20.7%, respectively. On the other hand, the ultimate tensile strength and Izod impact resistance of PEA-DPGDA-1% PTFE showed increases of 2.4% and 3.2%, respectively. However, the Young’s modulus showed a decrease of 14.7%. The decrease in Young’s modulus was higher than PEA-DPGDA-1% m-PTFE. Besides them, the ultimate tensile strength and Izod impact resistance values of PEA-DPGDA-1% m-PTFE showed higher increases than PEA-DPGDA-1% PTFE compared to PEA-DPGDA. Moreover, PEA-DPGDA-2% m-PTFE presented the highest Izod impact resistance, and its ultimate tensile strength value was still higher than PEA-DPGDA-1% PTFE and PEA-DPGDA. Considering the nanocomposites without surface modification, except PEA-DPGDA-1% PTFE, the mechanical values of all nanocomposites were lower than the reference sample, PEA-DPGDA

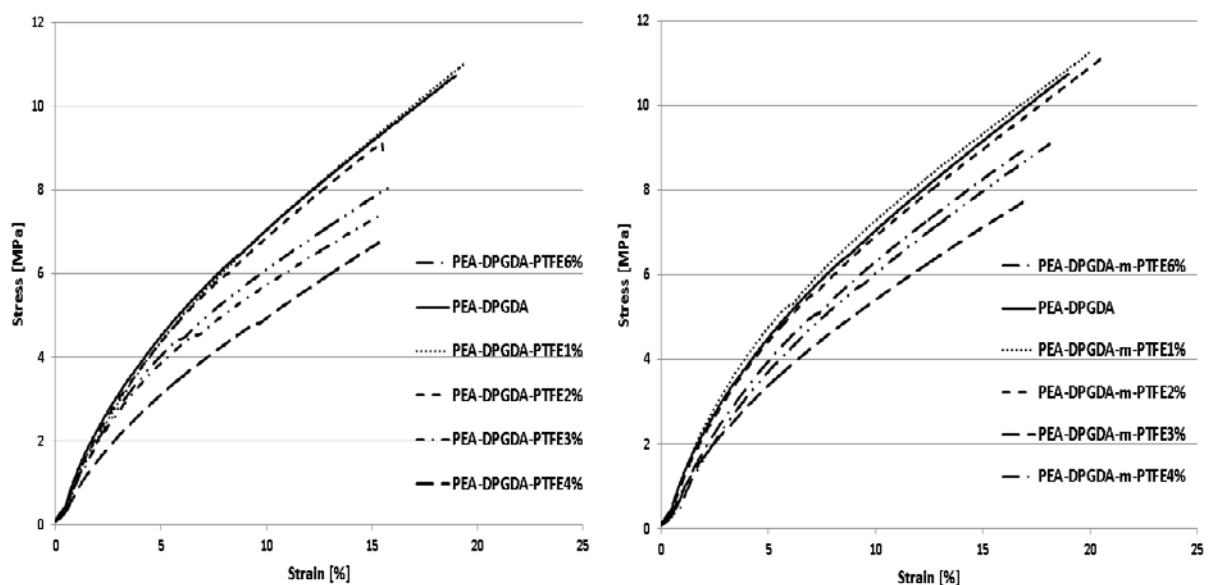
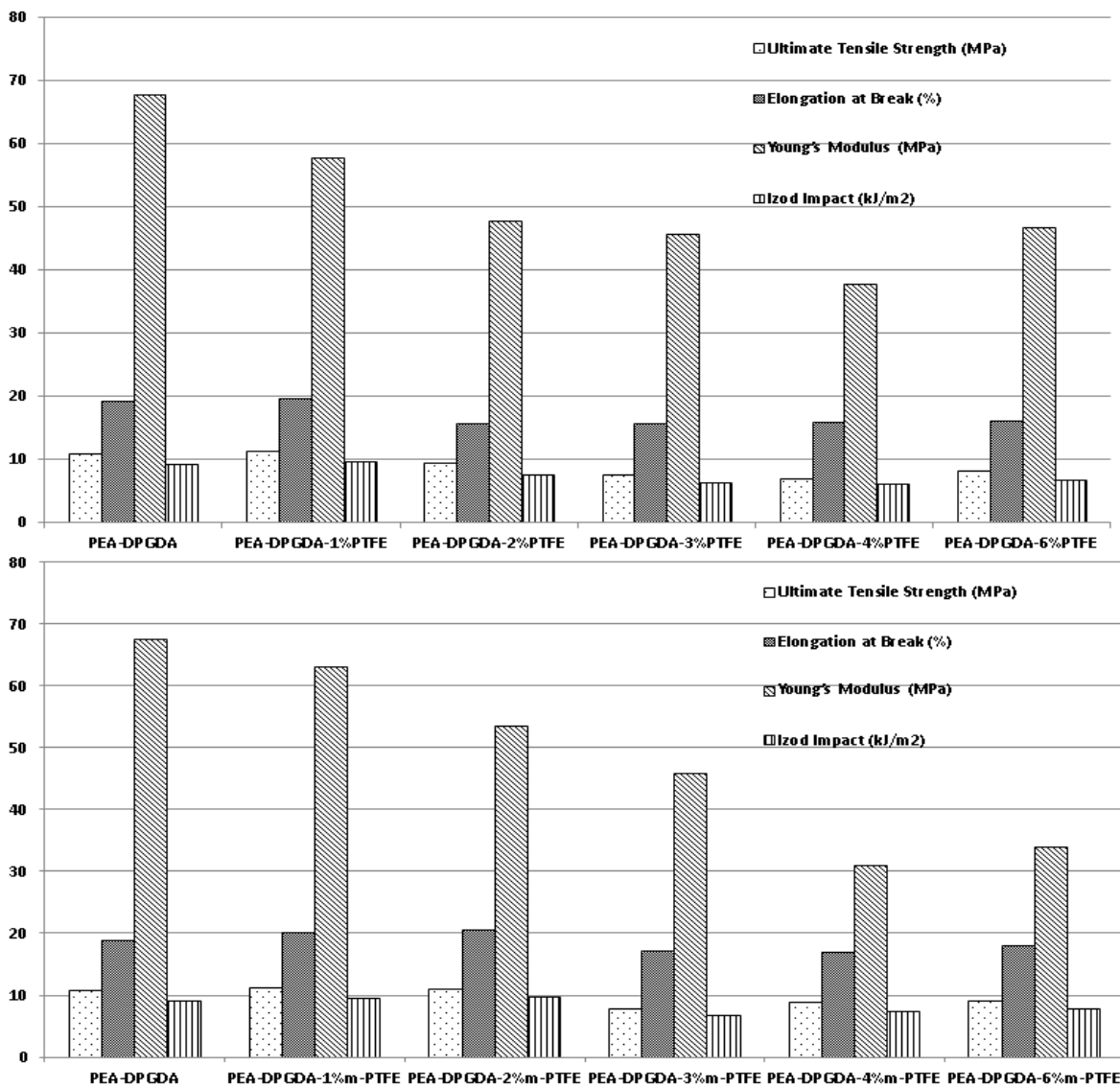


Figure 3. The average tensile stress-strain curves of the nanocomposites

**Table 1.** The mechanical values of the nanocomposites

Samples	Ultimate Tensile Strength (MPa)	Elongation at Ultimate Tensile Strength (%)	Young's Modulus (MPa)	Izod Impact (kJ/m <sup>2</sup> )	Shore D Hardness
PEA-DPGDA	10.73	18.99	67.58	9.1	55
PEA-DPGDA-1%PTFE	10.99	19.33	57.59	9.4	57
PEA-DPGDA-2%PTFE	9.13	15.48	47.52	7.3	55
PEA-DPGDA-3%PTFE	7.36	15.39	45.39	6.1	54
PEA-DPGDA-4%PTFE	6.78	15.60	37.67	5.9	51
PEA-DPGDA-6%PTFE	8.06	15.87	46.45	6.5	52
PEA-DPGDA-1% m-PTFE	11.28	20.09	63.13	9.6	56
PEA-DPGDA-2% m-PTFE	11.11	20.55	53.56	9.8	56
PEA-DPGDA-3% m-PTFE	7.79	17.19	45.93	6.7	55
PEA-DPGDA-4% m-PTFE	8.98	17.04	30.98	7.5	53
PEA-DPGDA-6% m-PTFE	9.07	18.10	33.98	7.8	53

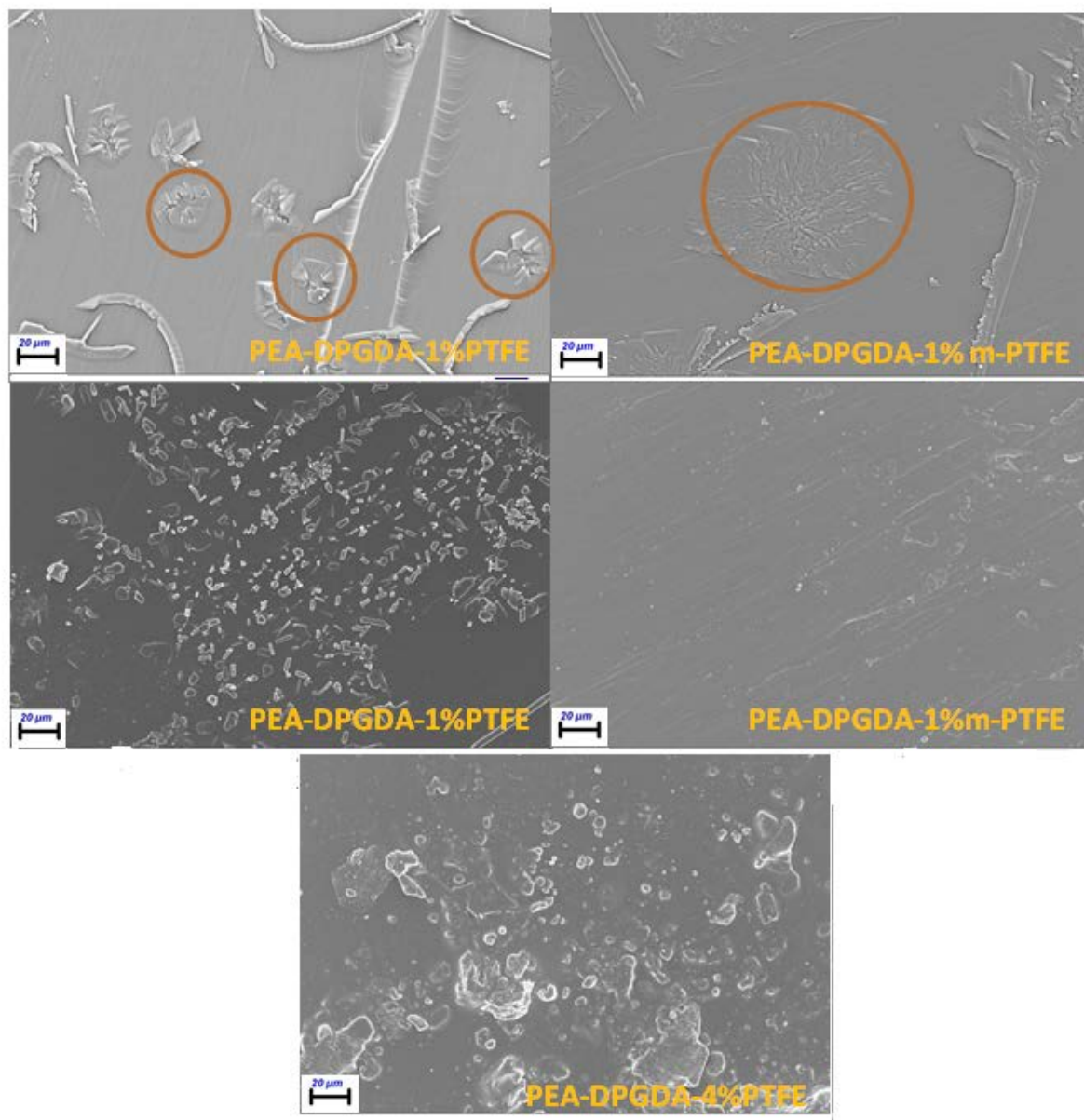


**Figure 4.** Comparative display of mechanical property trend of each sample

Figure 5 presents the fracture surface morphologies of the prominent filled nanocomposite samples (PEA-DPGDA-1%PTFE and PEA-DPGDA-1%m-PTFE), and the nanocomposite sample that showed the highest



decline in mechanical properties (PEA-DPGDA-4%PTFE). White particle agglomerates were attributed to the neat PTFE and surface-modified PTFE powders in the views of the filled nanocomposite samples. The PEA-DPGDA-1%PTFE and PEA-DPGDA-1%m-PTFE sample morphologies shown in Figure 5 appeared to point to a brittle fracture surface. Agglomeration was much less for PEA-DPGDA-1%m-PTFE, and these surface-modified PTFE particles also distributed more homogeneously [28, 29].



**Figure 5.** SEM morphologies of the prominent PTFE and surface-modified PTFE powders filled nanocomposites

Strong interfacial bonding's between the matrix and the surface-modified PTFE powders were also observed in this view, which is represented by the partially formed nodular networks of the surface-modified PTFE particles in the circular shape of Figure 5. However, as seen by the circular form in Figure 5, the PEA-DPGDA-1%PTFE sample exhibited tiny regional nodular hills that belonged to PTFE particles. While these hills had a minor

impact on the mechanical properties, the surface-modified PTFE particles' nodular networks had a more favorable effect [30,31].

#### IV. CONCLUSIONS

In this study, it was aimed at enhancing the mechanical properties of the PEA/DPGDA resin system (polyester acrylate resin containing DPGDA reactive diluent) with the filling of PTFE powders and surface-modified PTFE powders. This surface modification was carried out by treating the PTFE powder surface with a Na-naphthalene system. The pure PTFE and surface-modified PTFE powders were incorporated into the PEA/DPGDA resin system at various ratios of 1, 2, 3, 4, and 6%. The manufacturing of these nanocomposites was carried out by a DLP/LCD-type 3D printer. A Na-naphthalene system was used to modify the surfaces of the PTFE powders, and functional groups such as OH groups, carbonyl groups, and C=C unsaturation points that had been confirmed by XPS in our previous study were formed on the surfaces of the PTFE powders. According to the results of this study, the PEA/DPGDA samples into which PTFE powders whose surfaces were modified using a Na-naphthalene system were incorporated exhibited improved mechanical results. PEA-DPGDA-1% m-PTFE (surface-modified PTFE of 1% filled nanocomposite) and PEA-DPGDA-2% m-PTFE (surface-modified PTFE of 2% filled nanocomposite) showed the most improved properties compared to the PEA/DPGDA reference sample. The ultimate tensile values of PEA-DPGDA-1% PTFE and PEA-DPGDA-2% PTFE were 5.1% and 3.5% higher, respectively, compared to those of PEA/DPGDA. Moreover, the Izod impact resistance values of these samples showed increases at rates of 5.4% and 7.6%, respectively. However, Young's modulus values of PEA-DPGDA-1% PTFE and PEA-DPGDA-2% PTFE showed decreases at rates of 6.5% and 20.7%, respectively. The mechanical properties of the nanocomposites filled with surface-modified PTFE and the pure PTFE powders that have a higher filler ratio than 2% became poorer compared to the PEA/DPGDA sample. This situation could be derived from the additive manufacturing process in this study.

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