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Effect of chemical oxidation process on adhesive performance in two component adhesive with nano particle and filament additives

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Abstract: Adhesive, which is defined as the substance that binds the same or different types of materials together, is used in almost all sectors. However, the use of composite materials in the automotive, aerospace and aviation sectors due to their lightness and durability increases the importance of adhesive and adhesive joints. Adding nanostructures to the adhesive is a popular method today to improve the performance of the adhesive. However, homogeneous dispersion of nanoparticles added to the adhesive and increasing the wettability of nanofibers are very important in terms of adhesive performance. In the present study, chemically surface-treated nanoparticles and nanofibers were added to the adhesive to improve the performance of the two-component structural adhesive. In the study, DP460 structural adhesive was used as adhesive, functionalized Multi Walled Carbon Nanotubes (MWCNT-COOH) with COOH and carbon fiber (CF) chemically surface treated with HNO_3 solution were used as nanostructures. In the experimental study, eight different parameters were investigated as the nanostructure was (i) undoped, (ii) 1 wt% MWCNT-COOH added, (iii) 1wt%. untreated CF added, (iv) 0.5 wt% chemically treated CF added, (v) 1 wt% chemically treated CF added, (vi) 2 wt% chemically treated CF added, (vii) 0.5 wt% MWCNT-COOH and 0.5 wt% chemically treated CF added, and (viii) 1 wt% MWCNT-COOH and 1wt% chemically treated CF added. According to the results of the study, adding nanoparticles to the adhesive increases the performance of the adhesive by about 12%, while adding nanofibers increases the performance of the adhesive by about 18%. In addition, increasing the inertness and wettability of nanofibers by chemical treatment, as well as the use of nanoparticles and fibers together, significantly increases the performance of the adhesive. In addition, the obtained results were supported by fourier transform infrared spectroscopy (FT-IR) analysis and scanning electron microscopic (SEM) analysis.

Keywords: Polymer composites, Adhesive, Chemical oxidation, Carbon fiber, Nano particle, Strength.

1. Introduction

The use of composite materials in the automotive, aerospace and aviation sectors due to their lightness and durability increases the importance of adhesive and adhesive joints day by day. Adhesive joints; it offers important advantages in terms of not causing any change in the crystal structure as a result of melting, not forming stress concentrations, performing the joining process under melting temperatures, and also obtaining smoother and cleaner surfaces as in traditional joint methods [1].

There are important factors to improve the performance of adhesive joints. The first of these is to increase the force of attraction that exists between two different materials, which makes these two materials bond together. In order for these forces, also known as adhesion forces, to be strong, the adhesive must penetrate the roughness on the material surface and wet the entire surface. The second important factor is the cohesive force of attraction between the adhesive molecules, which keeps the adhesive together. The greater the covalent bond strengths that allow the polymer molecules to hold together, the greater the performance of the adhesive [2]. When the studies aimed at increasing these covalent bond strengths are examined, the addition of nanostructures to the adhesive comes into consideration. These studies were generally examined under the title of polymeric composites and they were named as "nano composites" because they contain nanostructures. The chemical and mechanical bonding between the nanostructure and the polymer is the main reason for the improvement in the mechanical properties of the polymer and the bond. Studies have shown that carbon nanostructures that do not have surface groups also improve the mechanical properties of the polymer [3].

The nanostructure added to the adhesive to improve the performance of the adhesive was generally carbon nano-

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tube (CNT) and carbon black samples. In a study published by Park et al. [4], the epoxy adhesive used in glass/epoxy composite bonds was modified with carbon black. The results indicate that carbon black reinforcement reduces the thermal expansion coefficient of the adhesive and sufficiently increases the load bearing capacity of the joint. In addition, Sihn et al. [5] concluded that the nanocomposite adhesive obtained by adding CNT to the adhesive increases the thermal conductivity of the adhesive joint. However, in modifying the epoxy adhesive with CNT, the ratio of CNT addition is an important parameter in terms of bond strength. Hsiao et al. [6] conducted a study on this subject and stated that there was a direct correlation between the CNT additive ratio and the bond strength as a result of the study.

Another non-structure added to the adhesive to increase the performance of the adhesive is filament structures. The performance of adhesives with filament structure additives is better than adhesives with nano particle additives. However, the most important disadvantage of adhesives with filament structure additives is the poor possibility of wettability with epoxy due to the chemical inertness of the filament structures added to the adhesive. This affects the performance of the adhesive. Therefore, in order to remove the chemical inertness of the filament structure and thus increase its wettability, the filament structures must be subjected to chemical surface treatments. In a study by Tiwari et al. [7], chemical surface treatment with nitric acid (oxidation with nitric acid) was applied to carbon filaments at different times (15-180 minutes) and the performances of these samples were investigated. According to the results of the study, the load carrying capacity of the filaments under tension decreased by 40% after 180 minutes of oxidation, and the best performance in terms of adhesive performance was obtained with 90 minutes of oxidation with nitric acid. In a study by Lou et al. [8], three different methods (ultrasonic surface cleaning, nitric acid oxidation and combination of ultrasonic and nitric acid oxidation) were applied to treat the surface of carbon filaments added to epoxy. In terms of the performance of the composite, they stated that the most suitable method among these applied methods is the method in which ultrasonic and nitric acid oxidation are applied together.

It is understood from the literature research that the addition of CNT and CF to the adhesive is very important for the performance of the adhesive. However, the rate of addition of CF to the adhesive, the homogeneous distribution of CNT to the epoxy and the removal of chemical inertness of CF affect the adhesive performance. In addition, the effect of using these two nanostructures together on the performance of the adhesive has not been examined. In the presented study, chemically surface-treated nanoparticles and filaments were added to the epoxy part to improve the performance of the two-component (epoxy and hardener) structural adhesive. In the study, DP460 structural adhesive was used as adhesive, functionalized Multi Walled Carbon Nanotubes (MWCNT-COOH) with COOH and carbon filament (CF) chemically surface treated with HNO3 solution were used as nanostructures. In the experimental study, eight different parameters were investigated as the nanostructure was (i) undoped, (ii) 1 wt% MWCNT-COOH added, (iii) 1wt%. untreated CF added, (iv) 0.5 wt% chemically treated CF added, (v) 1 wt% chemically treated CF added, (vi) 2 wt% chemically treated CF added, (vii) 0.5 wt% MWCNT-COOH and 0.5 wt% chemically treated CF added, and (viii) 1 wt% MWCNT-COOH and 1wt% chemically treated CF added. In addition, the experimental results were compared with the results of fourier transform infrared spectroscopy (FT-IR) analysis and scanning electron microscopic (SEM) analysis.

2. Material and method

In the presented study, a two-component (epoxy-hardener) DP460 (manufactured by 3M Company, St. Paul, MN, USA) structural adhesive, which is frequently used in aerospace and automotive fields, was used as the bonding material. The ratio of epoxy hardener in two-component adhesive is 2:1. The curing of this structural adhesive varies depending on temperature and time. After 30 minutes at 24⁰C, the epoxy and hardener react and begin to cure. The adhesive was completely cured by keeping it at 70⁰C for 120 minutes. In addition, nanostructures added into the adhesive are COOH-functionalized Multi Walled Carbon Nanotubes (outside diameter 10 to 20 nm, length 10 to 30 mm, purity 96%, surface area 200 m2/g) and 5 mm long trimmed 3K Carbon Fiber (fiber thickness : 0.22 mm, weight: 200 gr/m2, warp / scarf: 215/215 tex, weaving type: plain, filament thickness: 70µm). These nanostructures were purchased from Graphene Chemical Industry (Turkey). Mechanical properties for the adhesive and nanostructures used in experimental studies are given in Table 1.

Table 1. Material properties of the adhesive and carbon fiber [9].

	DP 460	Carbon Fiber
E (MPa)	$2044^{\pm 47}$	$228000^{\pm 2500}$
ν	0.38	-
σ_t (MPa)	$44.6^{\pm 1.2}$	$3800^{\pm 350}$
ε_t (%)	4.2	1.9

E:Young's modulus; ν: Poisson's ratio; σ_t : Ultimate tensile strength; σ_t : Ultimate tensile strain

Approximately 5 g of adhesive is required to prepare a standard bulk sample of adhesive. Since 3 samples will be produced from each parameter in the study, 15 g of adhesive was taken into account. Considering this situation, for the preparation of Multi Walled Carbon Nanotubes-COOH (MWCNT-COOH) nanostructure additive, 10 grams of epoxy and adhesive amount (15 g) of 1 wt% (0.15 g) of nanostructure was added to an empty beaker with the help of precision balance (Figure 1 a).). Then, acetone was added to the same beaker, approximately twice the amount of epoxy, and mixed in an ultrasonic mixer at a frequency of 30 KHz for 20 minutes

(Figures b and c). The aim here is to increase the distance between nanoparticles and to ensure homogeneous dispersion of the nanostructure in the adhesive. The beaker containing epoxy, MWCNT-COOH and acetone was kept at 50°C and the acetone evaporated. Whether the acetone evaporated completely or not was checked with a precision balance. Then, 5 g of hardener was added to the mixture and mixed by hand for 10 minutes. This method was first applied in a project made within the scope of Tübitak 1001 [10].

In the presented study, two different CFs were used for the Carbon filament (CF) added adhesive. These are chemically surface-treated CF and acetone-washed CF. In the chemical surface treatment of CF, 65% pure HNO_3 solution was used. The carbon filament fabric shown in Figure 2 a was kept in HNO_3 solution at 100^{0}C for 60 minutes and then washed with distilled water. The filament fabric washed with pure water was dried in drying-oven at 120^{0}C for 120 minutes (Figure 2 b). In the preparation of CF washed with acetone, carbon filament fabric was soaked in acetone of 95% purity for 10 minutes. Afterwards, the filaments were separated from the fiber fabric and cut into 5 mm length with scissors and trimmed filaments were obtained (Figure 2 c). The method used in the addition of the MWCNT-COOH nanostructure was applied to the attachment of the filaments to the adhesive.

In the production of bulk samples with or without nanostructure additives, a mold covered with non-stick tape was used to adjust the thickness and dimensions (Figure 3). Prepared adhesive is poured into this mold and flattened in the mold with the help of a gauge. The curing conditions of the adhesive were provided by placing the mold in the hot press. After curing was complete, the adhesive was removed from the mold (Figure 3).

Bulk sheets obtained from adhesives were cut to ISO 2818 [11] sample sizes given in Figure 4 a (Figure 4b) and gauge length was drawn on the sample to obtain more precise displacement data with video extensometer during testing (Figure 4 c).

In the present study, chemically surface-treated filaments and nanoparticles were added to the epoxy part to improve the performance of two-component DP460 structural adhesive. The shear strength of the produced nanostructured composite adhesives was investigated under the tensile test. The parameters used in the experimental study are given in Table 2 and 3 samples were produced from each parameter.

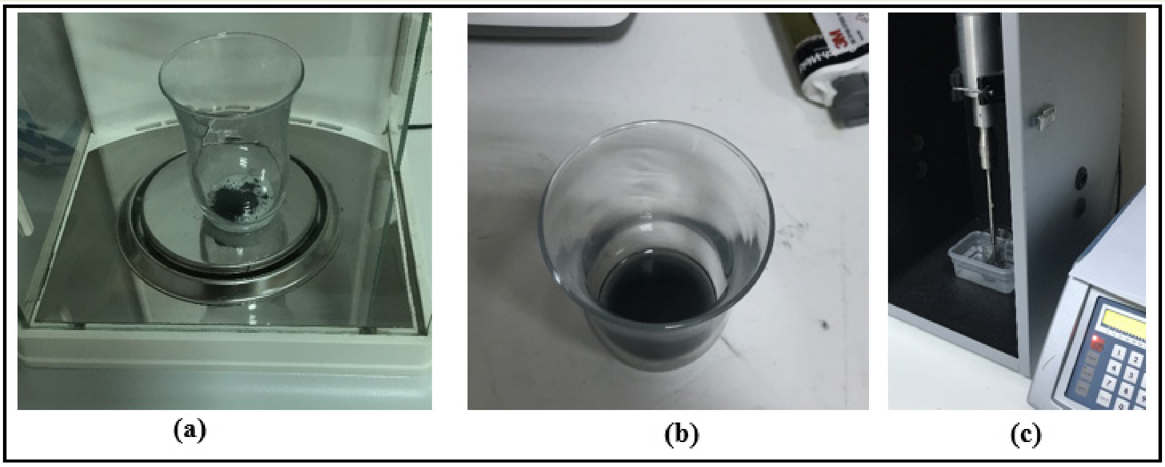


Figure 1. a) Addition of epoxy and MWCNT-COOH, b) Addition of acetone to the mixture, c) Ultrasonic mixer.

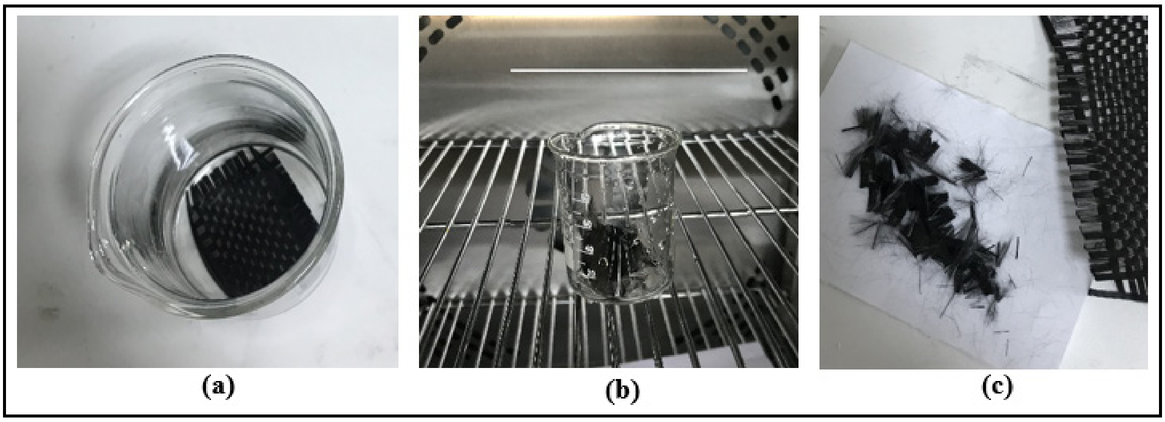


Figure 2. a) Fiber fabric in HNO_3 solution, b) drying of fiber fabric in drying-oven, c) 5 mm long CF.

The tensile test of the adhesive joints was carried out on a computer-controlled Instron-5982 (USA) universal tensile device at a tensile speed of 1 mm/min (Figure 5). The tensile test procedure applied to adhesive joints is given in Figure 5. In addition, tensile tests of a total of 24 bulk samples, three from the configuration given in Table 3, were carried out and the average shear stress and strain of the adhesives were obtained.

Fourier Transform Infrared Spectroscopy (FT-IR) analysis was performed to examine the chemical nature and interaction of nanostructured and undoped adhesives.

Table 2. Parameters used in the experimental study.

Samples	Chemical surface treatment for fiber	nanostructure	wt % nanostructure ratio
A-1	-------	--------	--------
A-2	-------	MWCNT-COOH	1
A-3	-------	CF	1
A-4	HNO_3	CF	0.5
A-5	HNO_3	CF	1
A-6	HNO_3	CF	2
A-7	HNO_3	wt % 0.5 MWCNT-COOH + wt % 0.5 CF	1
A-8	HNO_3	wt % 1 MWCNT-COOH + wt % 1 CF	2

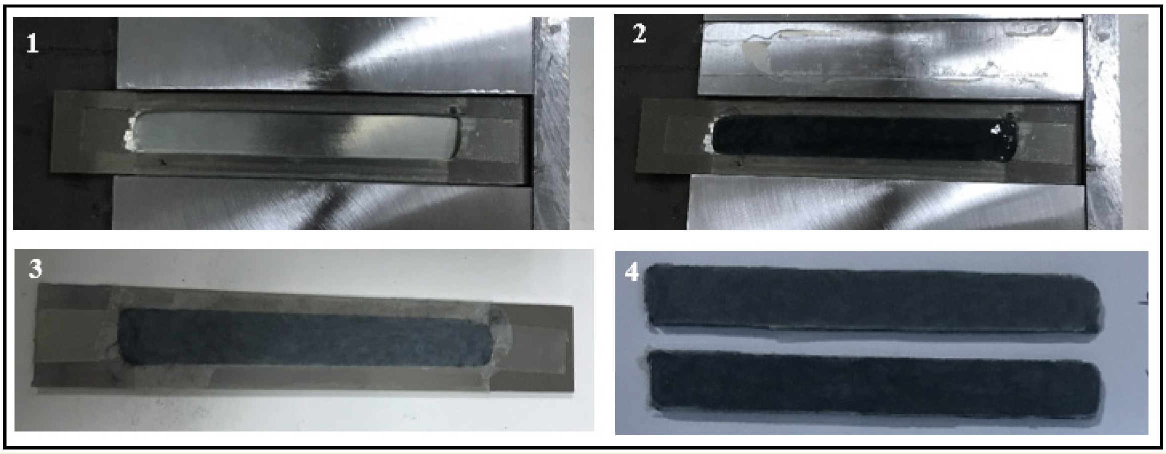


Figure 3. Bulk adhesive production scheme.

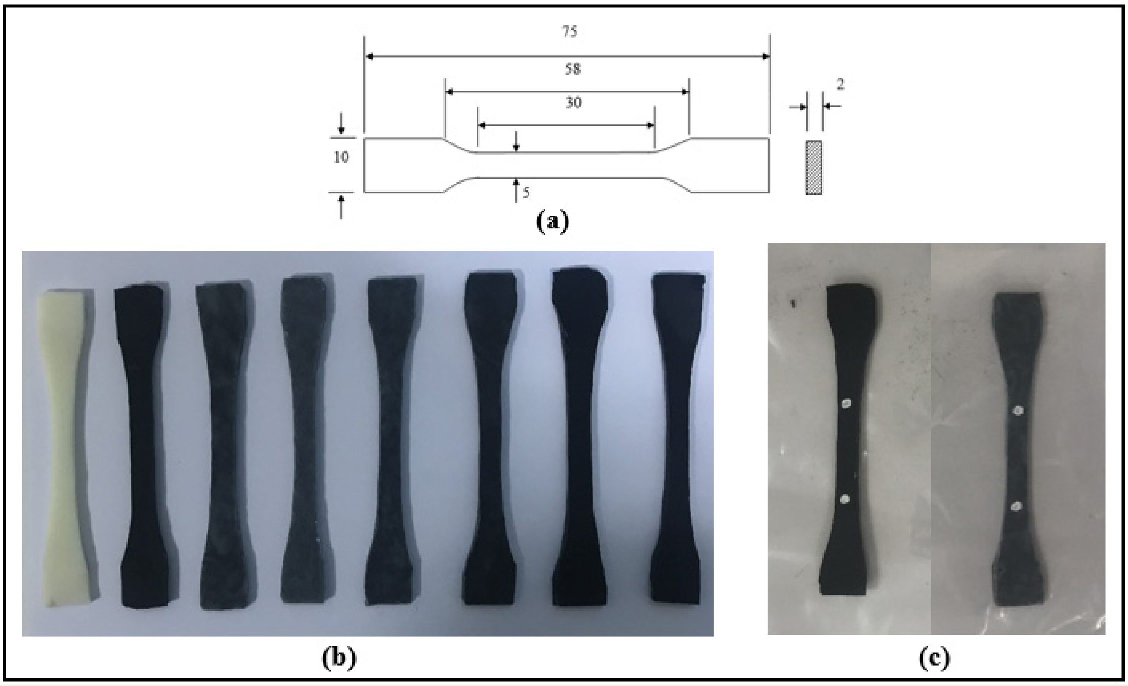


Figure 4. a) Standard bulk sample sizes, b) produced bulk sample samples, c) gauge length marking on the sample.

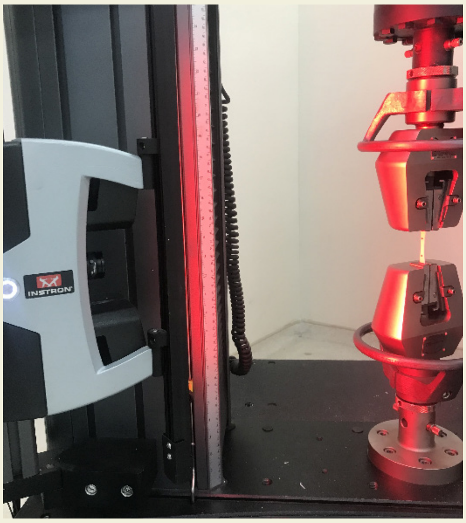


Figure 5. Tensile test application.

For FT-IR analysis of DP460 structural adhesives with no nanostructure additives, MWCNT-COOH and CF additives, an area of 5 mm^2 was taken from the produced bulk samples and placed in the reflectance unit of the FT-IR device. Spectra were made using Shimadzu IRTracer-100 model FT-IR/ATR instrument in the wavenumber range of 4000 to 400 cm^{-1}.

In addition, the surface morphology and the presence of nanostructure of carbon filaments oxidized with nitric acid were observed with a scanning electron microscope (SEM FEI-Quanta 250).

3. Results and discussion

In order to obtain force-displacement data of adhesives with and without nanostructure additives, the samples were loaded until rupture occurred. The force data obtained from the device were converted to stress by dividing the area (thickness x width) from the measuring length region of each sample prior to testing. The aim here is to see the effect of the nanostructure additive on the adhesive independent of the area. First of all, force-elongation curves were obtained as a result of the experiments. Then, true stress-true strain (s_t-e_t) curves for adhesives were obtained using the formulas given in equation (1) (Figure 6) [10, 12].

$$\varepsilon = \frac{\Delta l}{l_0} \quad \sigma = \frac{P}{A} \quad \varepsilon_t = \ln(1+\varepsilon) \quad \sigma_t = \sigma(1+\varepsilon) \qquad (1)$$

When the curves given in Figure 6 are examined, adding 1wt% of MWCNT-COOH into the medium hardness DP460 adhesive more than doubles the deformability of the adhesive. In addition, considering that the area under the stress-strain curve gives the energy absorbed by the adhesive [13], the energy absorbed by the 1 wt% MWCNT-COOH added adhesive (A-2) increased by approximately 335% compared to the adhesive without nanostructure additive (A-1). However, adding CF to the adhesive (A-3, A-4, A-5 and A-6) reduces the deformability of the adhesive somewhat and hardens the adhesive.

When the data given in Figure 7 are examined in terms of stress, adding 1wt% of MWCNT-COOH to the adhesive (A-2) increases the maximum stress by 17%, while adding 1wt% of CF without chemical surface treatment (A-3) increases the maximum stress by 21%. . However, increasing the oxidative groups on the filament surface (A-5) by chemical surface treatment of the CFs added to the adhesive increases the maximum stress by approximately 41%. In short, oxidation of the surface of CFs with HNO$_3$ solution increases the maximum stress of the adhesive by 20%. To explain the reason for this, the SEM images taken from the untreated (A-3) and chemically surface-treated (A-7) adhesive given in Figure 8 need to be examined. When these images were examined, the oxidative groups on the surface of the filaments treated with HNO$_3$ solution increased. These oxidative groups reduce the surface energy between epoxy and filament, resulting in better dispersion of epoxy and filament, as well as improving adhesive attraction forces. This situation, which prevents

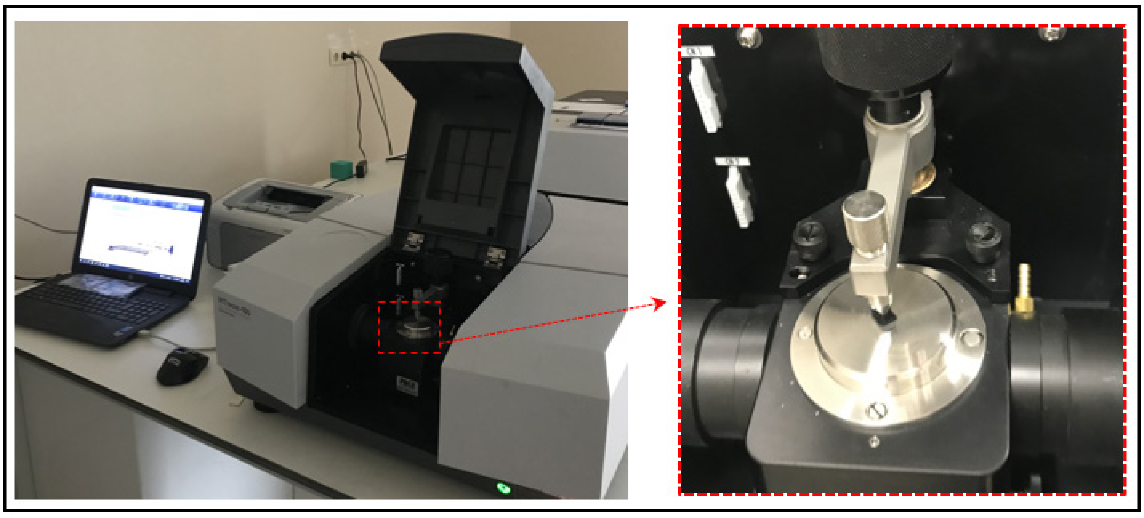
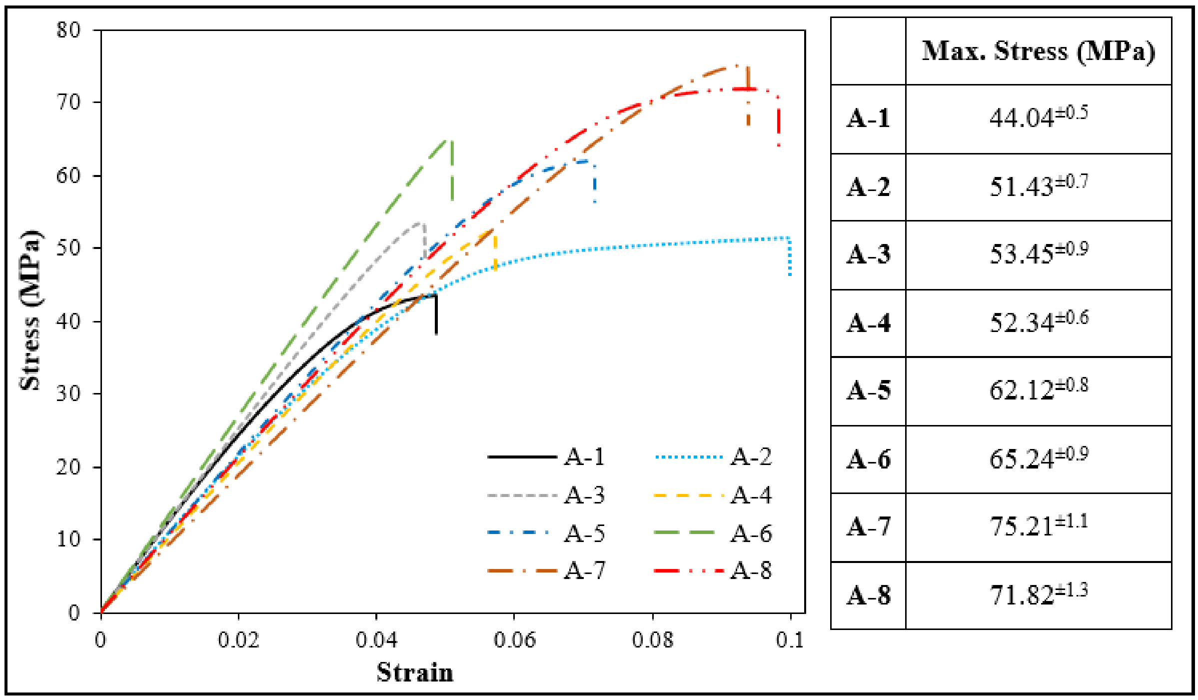


Figure 6. Shimadzu IRTracer-100 model FT-IR/ATR device used in FT-IR analysis

the filaments from being easily stripped from the epoxy, increases the performance of the adhesive.

One of the main objectives of the presented study is to examine how the performance of the adhesive changes with the addition of both MWCNT-COOH and CF nanostructures to the adhesive. Therefore, the performance of the adhesive (A-7) formed by adding 0.5% by weight of chemically treated CF and 0.5% by weight of MWCNT-COOH to the adhesive is 46% higher than the performance of the adhesive (A-2) formed by adding only 1% by weight of MWCNT-COOH to the adhesive. At



	Max. Stress (MPa)
A-1	$44.04^{\pm0.5}$
A-2	$51.43^{\pm0.7}$
A-3	$53.45^{\pm0.9}$
A-4	$52.34^{\pm0.6}$
A-5	$62.12^{\pm0.8}$
A-6	$65.24^{\pm0.9}$
A-7	$75.21^{\pm1.1}$
A-8	$71.82^{\pm1.3}$

Figure 7. True stress-true strain curves obtained from nanostructure doped DP460 adhesive.

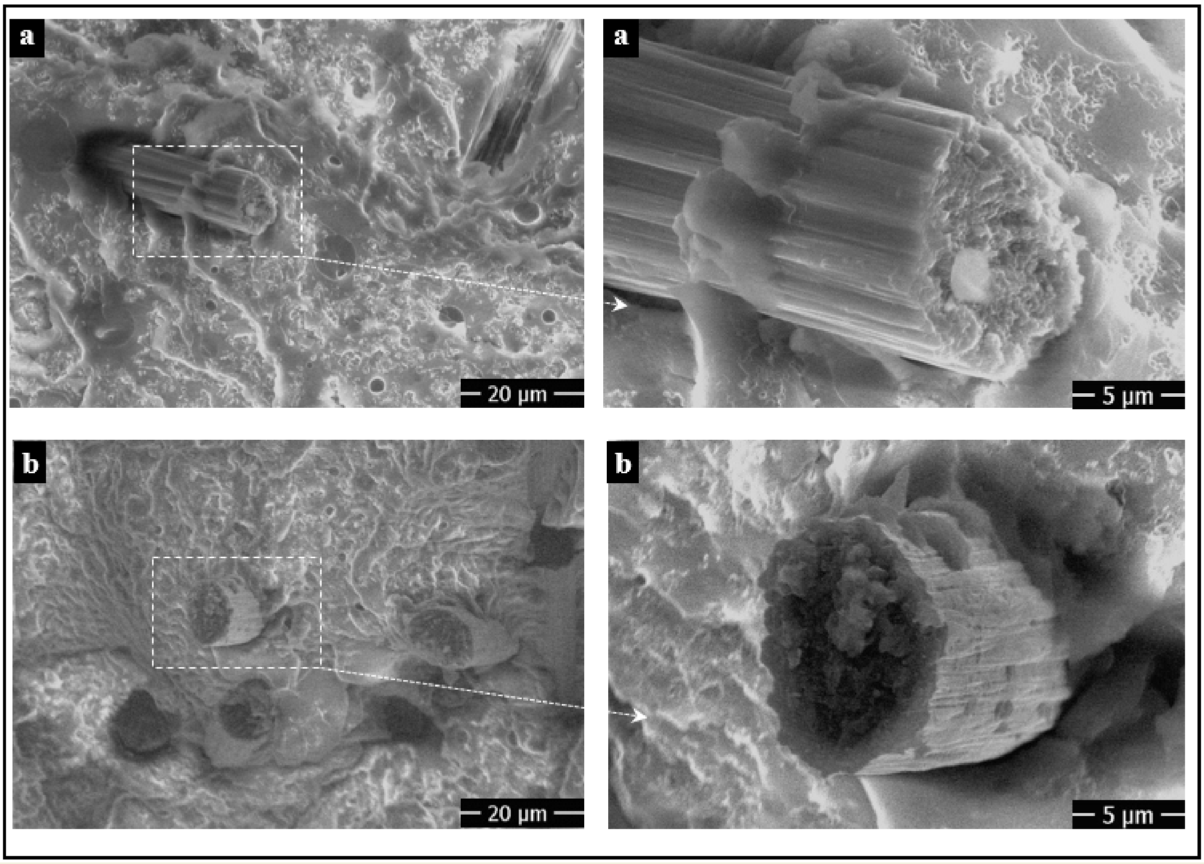


Figure 8. SEM images of untreated and HNO3-treated filaments, a) A-3 adhesive type, b) A-7 adhesive type.

the same time, it is 21% higher than the performance of the adhesive (A-5) created by adding only 1% by weight of chemically treated CF to the adhesive (Figure 7). One of the important results obtained here is that the use of chemically treated CF is more suitable than the use of MWCNT-COOH nanostructure in terms of the performance of the adhesive, and most importantly, the use of MWCNT-COOH and CF nanostructures together without changing the total nanostructure ratio in the adhesive significantly improves the performance of the adhesive. In addition, as the nanostructure ratio in the adhesive increases, the rate of increase in the maximum stress of the adhesive reduces. That is, the maximum stress obtained by adding 0.5 wt% CF to the adhesive by weight is 52.34 MPa, while the maximum stress is 62.12 MPa when the CF ratio is increased by 1 wt%, and the maximum stress is 65.24 MPa when the CF ratio is increased by 2 wt% (Figure 7).

In Figure 9, the fracture surface SEM morphology of the adhesive with no filament additives, 1 wt% MWCNT-COOH added and 1% wt CF added adhesive is given. When these images are examined, it is seen that the peaks and valleys in the fracture surface morphology of the pure adhesive are minimal and close to smooth (Figure 9 a). However, when 1% by weight of MWCNT-COOH is added to the adhesive, the adhesive becomes ductile and the fracture surface morphology becomes rough (Fig. 9 b). The reason for this may be the increase of capillary cracks that cause damage to the adhesive due to the homogeneously dispersed and epoxy coated MWCNT-COOH nanostructures of 10 to 30 mm in length. In addition, when the fracture surface morphology of the 1% by weight CF-added adhesive given in Figure 9c is examined, it is seen that the peaks and valleys are sharp where the filament are located, and some filament are stripped from the epoxy. This situation shows the importance of applying HNO_3 oxidation to CFs.

Figure 10 shows the FTIR spectra of pure DP460 structural adhesive, adhesive containing acidic functional group-multi-walled carbon nanotube (MWCNT-COOH), adhesives containing the untreated and acid-treated carbon filament (CF), and composites containing these components in various compositions. From this figure, the peaks around 3440 cm^{-1} in the spectrum of the pure adhesive bind to the hydroxyl group (-OH), while the peaks at 2916, 2848 and 2933 cm^{-1} can be attributed to the asymmetrical and symmetrical C-H stretching, the asymmetrical stretching C-H of $-CH_3$ group and the asymmetrical C-H stretch of $-CH_2$ group, respectively.

Aromatic C-C stretch vibrations are likely represented by peaks observed at 1609, 1560, 1508, and 1449 cm^{-1}. Additionally, peaks at 1228, 1301, 1122, and 1033 cm^{-1} may signify asymmetric –CH2 deformation, asymmetric aromatic C-O stretch, asymmetric aliphatic C-O stretch, and symmetric aromatic C-O stretch, respectively. Furthermore, peaks at 960, 893, and 889 cm^{-1} could be attributed to epoxide ring vibrations, while the peak at 792 cm^{-1} may indicate out-of-plane deformation of aromatic -CH.

In the spectrum of acid-functionalized MWCNT depicted in Figure 10, several peaks are discernible, includ-

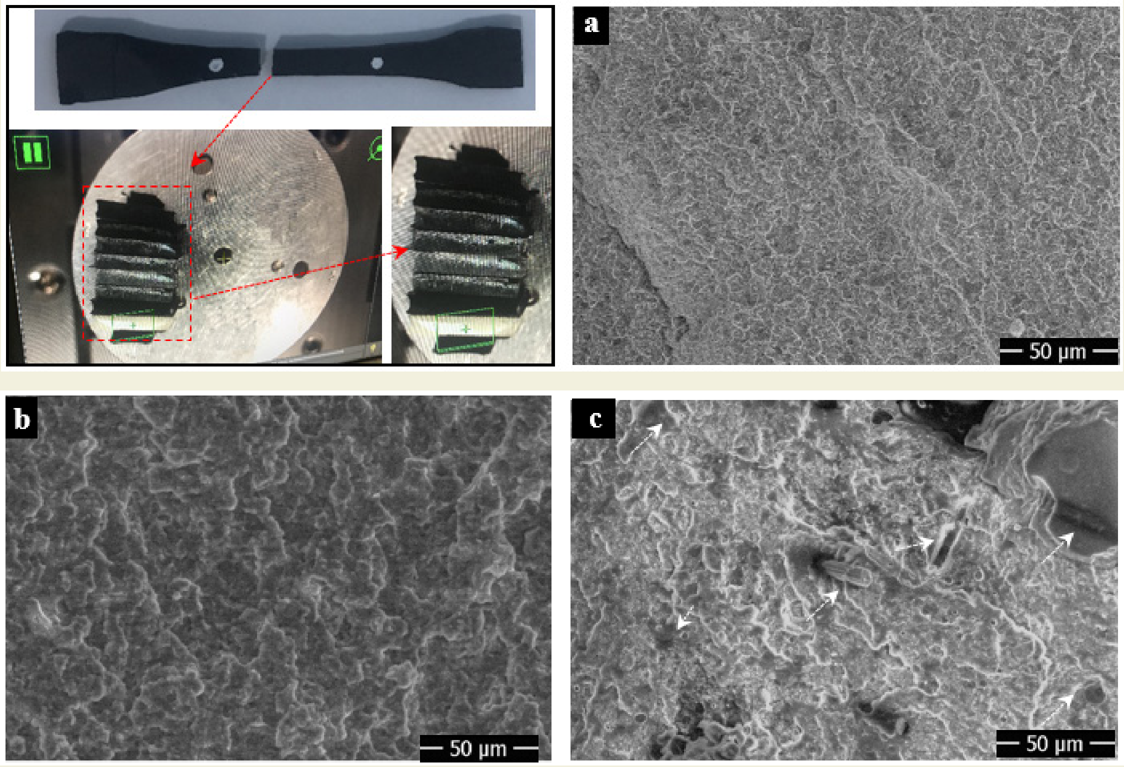


Figure 9. Surface SEM images of the adhesive bulk samples broken after the tensile test, a) A-1 adhesive type, b) A-2 adhesive type, c) A-3 adhesive type.

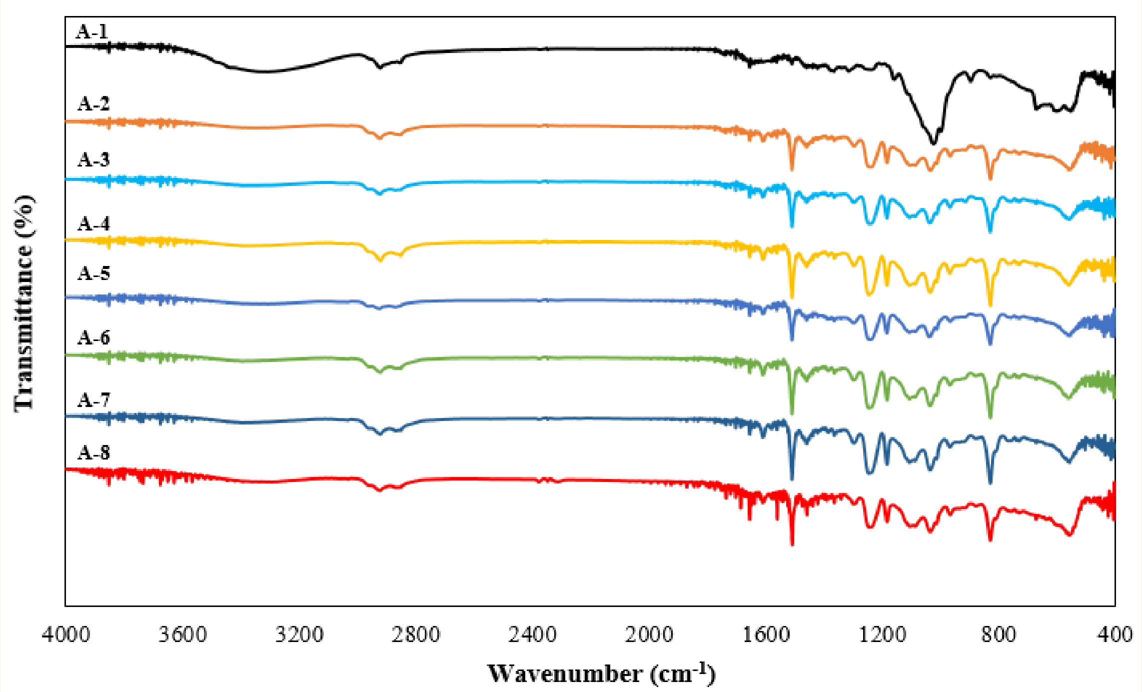


Figure 10. FT-IR spectra of DP460 structural adhesives with no nanostructure additives, MWCNT-COOH and CF additives.

ing those at 1560, 2933, 2848, and 3446 cm^{-1}. The peak around 3440 cm^{-1} is likely due to the presence of the hydroxyl group (-OH), while peaks at 2933 and 2848 cm^{-1} suggest asymmetric and symmetrical CH stretching. Moreover, peaks at 1560, 1722, and 1228 cm^{-1} may correspond to carboxylic acid group stretching modes, serving as clear indicators of the presence of -COOH groups on the nanotubes' surface.

From the spectra of the untreated and acid-treated carbon filament (CF) samples in Figure 10, it can be said that the peak at 3453 cm^{-1} corresponds to the hydroxyl (-OH) groups, the peaks at 2934 and 2849 cm^{-1} correspond to the methylene group, the low intense peak at 1772 cm^{-1} corresponds to the C=O vibrations and the peaks between 1600-1700 cm^{-1} correspond to the C=C vibration. The appearance of peaks indicating -CO and -COOH groups in the spectrum of acid-treated CF can clearly indicate the formation of oxy groups on the CF surface. On the other hand, in all spectra, the peaks at 901 cm^{-1} can be attributed to the stretching vibrations of the epoxy groups, while the peaks at 830 cm^{-1} and the peaks at 3368 and 1560 cm^{-1} can be attributed to C–O stretching vibrations and N-H vibrations, respectively.

In the spectra of the adhesive samples to which MWCNTCOOH and acid-treated CF were added in various proportions, the intense peak indicating aromatic ring -C=C- vibrations at 1506 cm^{-1}, corresponding to the subtituated -C=C- vibrations at 827 cm^{-1} severe peak and intense -C-O stretching vibration peak at 1236 cm^{-1} appeared. The intensity of these peaks especially increased with the increase of the acid-treated CF ratio. The intensity of these peaks especially increased with the increase of the acid-treated CF ratio. This may possibly have led to a decrease in the interfacial energy of CF and epoxy, thus increasing the effectiveness of their adhesive interaction. It can also be argued that the lower interfacial energy of nano- and micro-additives with both lyophilic and lyophobic parts creates a better mixing regime with the epoxy matrix. The decrease in the intensity of the peak, which indicates the epoxy group of the pure adhesive, can also be attributed to the adhesive interactions between this group and the oxy groups.

4. Conclusions

In order to improve the performance of structural adhesives, the appropriate ratio of MWCNT-COOH and CF nanostructures to the adhesive was applied together and chemical surface treatment was applied to ensure the adhesion of the CFs to the epoxy. The removal of the oxide layer on the surface of the CFs with the HNO3 solution and the increase of the oxidative groups on the filament surface increase the performance of the adhesive by approximately 48%. In addition, different ratios of CF were added to the adhesive, and the most suitable ratio was obtained as 1% by weight.

It was concluded that adding 1% by weight of MWCNT-COOH nanostructure to the adhesive significantly increased the strain of the adhesive, but adding CF to the adhesive reduced the strain of the adhesive. Considering this situation, the addition of 1 wt% MWCNT-COOH and CF nanostructures by total weight to the adhesive both balanced the deformation of the adhesive and increased the performance of the adhesive by 71%. In addition, these experimental results were supported by SEM surface morphology images and FT-IR analyzes taken from the nanostructured and undoped adhesive.

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