

# THE EFFECT OF POLYANILINE AND AMINE FUNCTIONALIZED CARBON NANOTUBES ON THE PROPERTIES OF COMPOSITE NANOFIBER WEB

## POLİANİLİN VE AMİN FONKSİYONEL GRUPLU KARBON NANOTÜPLERİN KOMPOZİT NANOLİF ÖZELLİKLERİNE ETKİSİ

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### ABSTRACT

Conductive polymers such as PANI and conductive nanoparticles such as CNTs have very important effects on the polymer matrix. Although there are many studies carried out for just only PANI filler or carried out for just only CNT filler, there are no studies performed to see the synergistic effects of both PANI and functionalized CNTs on polymer matrix. Thus in this study, for the first time, PANI together with CNTs has been used as a filler for polymer matrix, polyacrylonitrile. It has been seen that the diameters of nanofibers increase due to presence of PANI and CNTs and the effect of PANI on the increase in diameter is higher than that of CNTs. An increase in filler content results in an increase in agglomeration risk which degrades the properties of composite nanofiber web. However, higher breaking strength is obtained for the composite nanofiber with 1% CNT and 3% PANI. Insulator PAN became an antistatic material (static dissipative material) by the presence of CNT and PANI. However, an increase in filler content did not increase the electrical conductivity. This may be due to the agglomeration and void formation around the filler which destroy the network leading to the decrease in conductivity. Presence of PANI and CNT increases the crystallinity of PAN. The crystallinity of composite nanofiber with both of CNT and PANI is higher than that of pure PAN nanofiber, nanofiber with PAN, CNT and nanofiber with PAN, PANI. PANI increases the cyclization temperature and decreases the enthalpy; however CNT has a tendency to increase both the cyclization temperature and the enthalpy. When both of them (CNT and PANI) are present in high amount (3%), then both cyclization temperature and enthalpy decrease.

**Key Words:** Functionalized carbon nanotube, Polyacrylonitrile, Polyaniline, Electrospinning, Nanofiber.

### ÖZET

Polianilin (PANI) gibi iletken polimerlerin ve karbon nanotüp (CNT) gibi iletken nano partiküllerin kompozit ürün üzerinde oldukça önemli etkileri mevcuttur. Şu ana kadar gerçekleştirilen nano polimer kompozit çalışmalarda ya sadece PANI ya da sadece CNT kullanılmıştır. Hem PANI hem de CNT'nin bir arada kullanıldığı, kısaca her iki malzemenin sinerjik etkisini kompozit ürün üzerinde analiz etme ile ilgili henüz bir çalışma literatürde mevcut değildir. Böylece, bu çalışmada ilk kez, PANI ve CNT beraber polimer matris (poliakrilonitril, PAN) içerisinde dağıtılarak kompozit nanolif ağı üretilmiştir. PANI'in, CNT'e kıyasla nanolif çapını daha yüksek oranda artırdığı görülmüştür. Katkı malzeme miktarı ve tipi arttıkça, polimer matris içinde topaklanma riski daha çok artmaktadır. Bununla birlikte, kompozit yapı içinde %1 CNT, %3 PANI ihtiva eden ürün, %100 PAN ve diğer kompozit numunelere kıyasla daha yüksek kopma mukavemetine sahip olmuştur. Normalde yalıtkan bir malzeme olan PAN, bu çalışmada kullanılan katkı malzemeleri ile antistatik malzeme özelliğini kazanmıştır. Bununla birlikte katkı malzeme tipinin değişmesi ve oranlarının artması iletkenliği çok fazla değiştirmemiştir. PANI ve CNT mevcudiyeti ile PAN polimerinin kristalinitesi artmıştır. PANI sebebi ile siklizasyon sıcaklığı artarken, entalpi düşmüştür, CNT sebebi ile hem siklizasyon sıcaklığı hem de entalpi yükselmiştir.

**Anahtar Kelimeler:** Fonksiyonlanmış karbon nanotüp, Poliakrilonitril, Polianilin, Elektrospining, Nanolif.

## 1. INTRODUCTION

Conductive polymers have exhibited developing potential for use in many areas with the early work of MacDiarmid, Shirakawa and Heeger (1). Their potential applications in electromagnetic interference shielding, energy storage devices, radiation detector, membranes and sensors make them ideal subject to investigate. Also, conductive polymers such as polyaniline (PANI), polypyrrole (PPy) and polythiophene are used as conducting fillers in insulating polymer matrix to make them conductive composites (2-7).

Since discovered by Iijima in 1991, carbon nanotubes (CNT) have become major research area due to their unique mechanical, electrical, chemical and thermal properties (7). There are various studies related to composite PAN nanofiber with only CNTs (8-16). However there are very limited studies carried out on PAN composite nanofiber together with PANI (6,7).

Although both CNT and PANI have electrical conductivity properties and also PANI can be processed in solution form rather than nano particle form as in CNT, there is no studies which uses PANI solution containing CNT as filler. Since there are no studies to search the synergistic effect of PANI and functionalized CNT on polymer matrix such as PAN, in this study, for the first time polymer matrix+PANI+CNT composite nanofiber has been studied and substantial contribution to the published literature is provided. The effect of each additive (PANI, CNT) and the amount of additives on various properties of composite polymer nanofiber was analyzed and compared to each other by intensive analyses such as mechanical behavior, electrical conductivity, thermal analysis and morphological examinations with instruments such as tensile tester, electrical conductivity meter, FTIR, DSC, XRD, SEM.

Statistical analyses (ANOVA) were performed for 95% significance level in order to analyze whether the differences between the mean values are significant or not.

## 2. EXPERIMENTAL DETAILS

### Materials and Methods

Polyacrylonitrile (PAN) with a molecular weight of 150.000 g/mol was purchased from Sigma Aldrich. MWCNTs as pristine MWCNT (diameter 60-100 nm, length 5-15  $\mu$ m) was purchased from NTP (China). Polyaniline (PANI) with molecular weight of 65.000 g/mol was purchased from Sigma Aldrich. Dimethyl sulfoxide (DMSO) was used as solvent. Camphor sulfuric acid (CSA) was purchased from Sigma Aldrich.

### Synthesis of CNT-NH<sub>2</sub>

To obtain amine functionalized carbon nanotubes, Zhao *et al.*'s amino functionalization method was used (17). As

prepared MWCNTs-COOH were mixed with NaNO<sub>2</sub> and isophoronediamine. Concentrated H<sub>2</sub>SO<sub>4</sub> and DMF were added. Then the mixture was stirred and heated for 1 h at 60 °C. The mixture was cooled to room temperature, then DMF was added and the mixture was filtered with a PTFE membrane (0.45  $\mu$ m pore size). The solid was sonicated in DMF and filtered again, and the process was repeated until the DMF was colorless after sonication. The sample was then dried at 60 °C overnight under vacuum. CNT-NH<sub>2</sub> was obtained.

### Preparation of the solutions

Campforsulfonic acid (dopant) and polyaniline (equivalent molar ratio PANI: campforsulfonic acid = 1:2) were added to DMSO and mixed with magnetic stirrer at 40 °C, 300 rpm for 2 days. The solution was filtered with Sartorius Stedim filter paper and amine functionalized carbon nanotubes added in the solution. CNT-NH<sub>2</sub>/PANI/DMSO solution was dispersed for 10 min with ultrasonic homogenizer (Bandelin Sonopuls HD 2070, probe type: KE76) and then for 45 min with ultrasonic bath. PAN (7 wt% PAN concentration) was dissolved in the stable suspension of CNT-NH<sub>2</sub>/PANI in DMSO. The concentration of PANI with respect to the weight of PAN was 3 %.

On electrospinning system, the feeding rate of the polymer solutions was 1 mL/h with 15 kV electrospinning voltage and the distance between the needle tip and collector was 10 cm. The list of the samples can be seen in Table 1.

### Instrumentation

Measurements have been carried out at the standard atmospheric condition (20  $\pm$ 2 °C, 65% $\pm$ 5 RH). Tensile tester with a 100N load cell at a crosshead speed of 20 mm/min was used for the determination of breaking strength and breaking elongation of nanofiber webs. The gage length was 15 mm and specimens were cut in 35 mm (length) x 5 mm (width) dimensions for tensile testing. Average has been taken from at least seven samples. The thicknesses of the specimens were measured with a Mitutoyo digital micrometer.

Microtest LCR Meter 6370 (0.01 m $\Omega$ -100 M $\Omega$ ) with two circular probe-four wire system is used for measuring of the electrical resistance of composite nanofibers. Average has been taken from seven and more samples. The thicknesses of the samples were measured with the integrated thickness meter. Volume conductivity of the samples in S/cm was calculated using the formula as indicated in ASTM standards (18,19). Volume resistance values were measured and the conductivities of the composite nanofibers in S/cm were calculated by the use of geometric dimensions of the samples.

Table 1. List of the samples produced.

Samples	PAN	CNT-NH <sub>2</sub>	PANI
PAN-DMSO	present	-	-
(3%)PANI/PAN-DMSO	present	-	3%
(1%)CNT-NH <sub>2</sub> /PAN-DMSO	present	1%	-
(3%)CNT-NH <sub>2</sub> /PAN-DMSO	present	3%	-
(1%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	present	1%	3%
(3%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	present	3%	3%

$$\gamma_v = t / (A \times R_v) \quad (1)$$

where:

$\gamma_v$  = electrical conductivity, S/cm

$R_v$  = volume resistance,  $\Omega$ ,

A = area of the electrodes,  $\text{cm}^2$ , and

t = distance between the electrodes, cm.

The morphology and structure of the CNT/PAN nanofibers coated with gold were investigated by SEM Carl Zeiss EVO MA10. The SEM tests were applied at 5 kV voltage. The diameters of the nanofibers were measured on SEM photomicrographs and analyzed using Image J Software. At least 50 measurements were done to obtain average fiber diameter.

Wide-angle X-ray diffraction traces were obtained using a Bruker<sup>®</sup> AXS D8 Advance X-ray diffractometer system using nickel filtered  $\text{CuK}\alpha$  radiation ( $\lambda$ , 0.15406 nm) and voltage and current settings of 40 kV and 40 mA, respectively. Counting was carried out at 10 steps per degree. The observed equatorial X-ray scattering data was collected in reflection mode in the  $5-40^\circ$   $2\theta$  range. X-ray data-curve fitting developed by Hindeleh et al. (20) has been applied. Apparent X-ray crystallinity is based on the ratio of the integrated intensity under the resolved peaks to the integrated intensity of the total scatter under the experimental trace (21).

The FT-IR spectra of composite nanofibers were recorded using Nicolet IS10 FT-IR spectrometer. A minimum of 16 scans were averaged with a signal resolution of  $4 \text{ cm}^{-1}$  within the  $400-4000 \text{ cm}^{-1}$  range. ATR method was used to collect the IR.

DSC Q10 (temperature range between  $20-350^\circ\text{C}$ ) was used for thermal analysis at a heating rate of  $20^\circ\text{C}/\text{min}$ , under nitrogen atmosphere.

High standard deviations can be experienced owing to uncontrolled parameters in electrospinning process. Statistical analyses were performed using SPSS software. All the data were expressed as the mean  $\pm$  SD (standard deviation) and coefficient of variation (CV %). Variation analyses were done with ANOVA test in the 95% confidence interval to see whether the differences between the average values were important or not.

### 3. RESULTS AND DISCUSSION

#### Analysis of morphological properties of the nanofiber web

There are several mechanisms that can affect the final diameter of nanofiber. Conductive filler particles have an influence on the conductivity of the solution and this may

promote two phenomena having opposite effects on fiber formation and diameter (9). They may either increase flow rate, which can lead to larger fibers (22) or increase net charge density, which suppresses the Rayleigh instability and enhances the whipping instability, leading to the formation of bead-free and smaller fiber diameter (23, 24). An increase in filler content will also result in an increase in diameter due to agglomeration and the increase in the material. Thus, the diameter of the nanofibers depends on the phenomenon which is dominant.

The diameters of composite nanofibers obtained from SEM observations can be seen in Table 2. Pure PAN nanofiber's average diameter is 515 nm. The diameter increased because of the presence of CNT and PANI filler. The effect of PANI on the increase in diameter is higher than that of CNT.

According to statistical analysis (F tests, 95% confidence interval), it has been seen that differences between the diameter of PAN-DMSO nanofiber and 3%PANI/PAN composite nanofiber and also 3%CNT-NH<sub>2</sub>/3%PANI/PAN composite nanofiber are significant at 0.05 significance level. However, the difference between PAN-DMSO nanofiber and PAN with 1% and 3% CNT-NH<sub>2</sub> is not significant at 0.05 significance level.

#### IR analysis

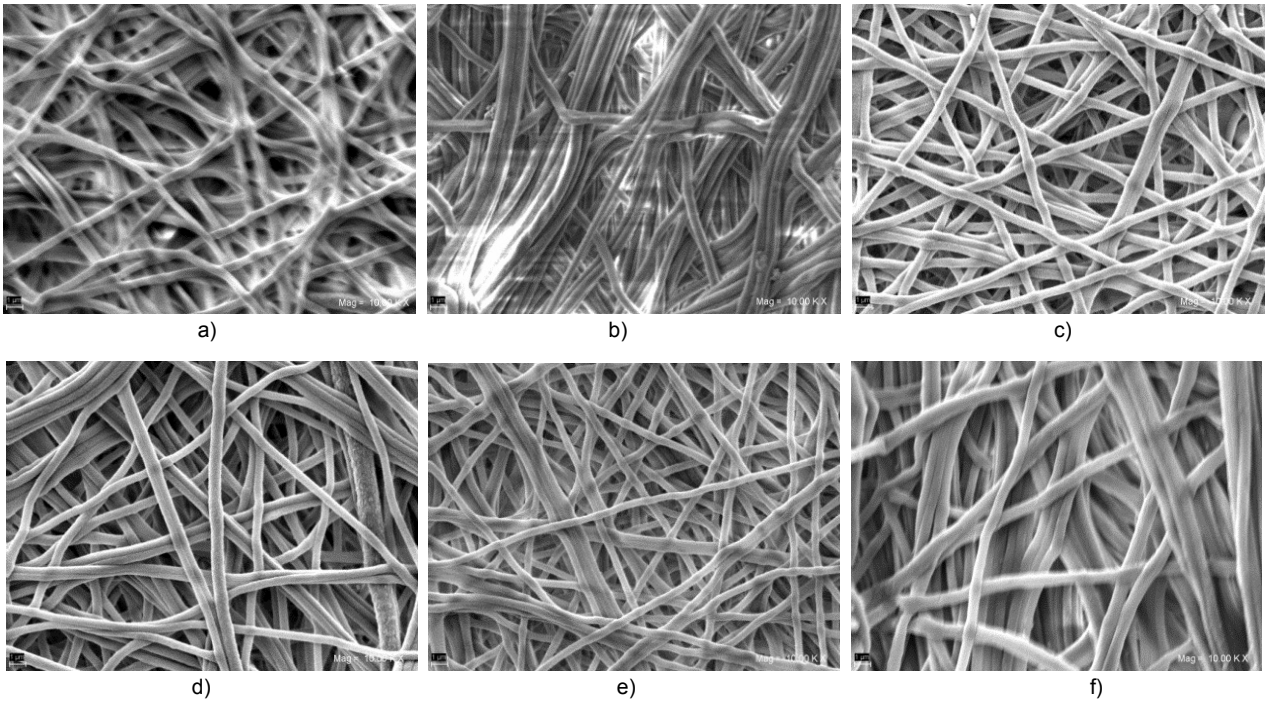
Infrared spectrum of composite nanofibers has been carried out to observe atomical vibrations. Figure 2 shows the IR spectra of PAN-DMSO, PAN/CNT-NH<sub>2</sub>-DMSO and PAN/PANI/CNT-NH<sub>2</sub>-DMSO nanofibers, respectively.

In Figure 2.a PAN-DMSO spectrum can be seen. Peaks at  $1450$  and  $750 \text{ cm}^{-1}$  are due to the  $-\text{CH}_2$  vibrations. The characteristic peak of  $-\text{CN}$ , from PAN is at  $2200 \text{ cm}^{-1}$ . All other samples contain polyacrylonitrile have the same peaks. At Figure 2.b, the peak at  $1600 \text{ cm}^{-1}$  can be assigned to C=C stretching of carbon nanotube structure and C=O stretching of amide ( $-\text{NH}-\text{C}=\text{O}$ ) structure (25).

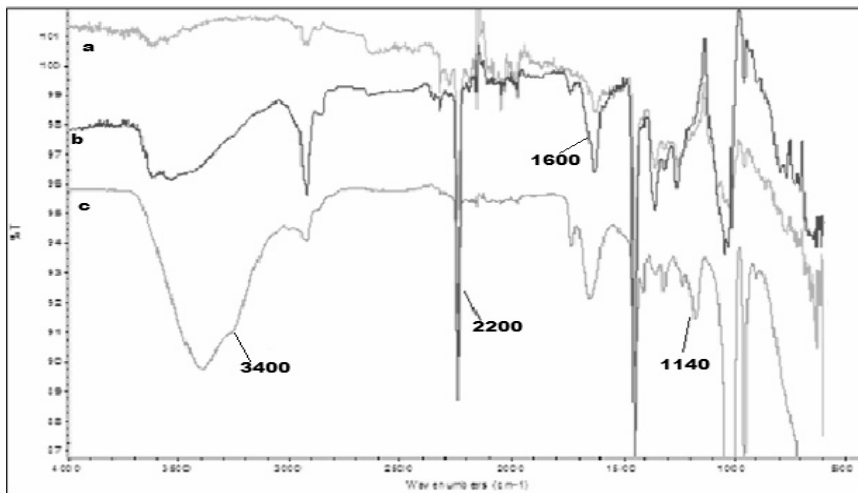
Peak at  $1490 \text{ cm}^{-1}$  is attributed to polyaniline's benzenoid (N-B-N) vibrations. The stretchings at  $1140$  ve  $1302 \text{ cm}^{-1}$  can be attributed to C-N and N=Q=N quinoid vibrations, respectively (26). The increased peak in the  $3200-3500 \text{ cm}^{-1}$  region can be attributed to the effect of PANI. The peak at around  $3390 \text{ cm}^{-1}$  is due to the free imin ( $-\text{NH}$ ) groups of PANI while the peak at around  $3280 \text{ cm}^{-1}$  is due to the aromatic C-H stretching (17).

Table 2. Diameters of the composite nanofibers.

Samples	Diameter (nm)	CV (%)
PAN-DMSO	515 $\pm$ 72.1	14
(3%)PANI/PAN-DMSO	575 $\pm$ 86.2	15
(1%)CNT-NH <sub>2</sub> /PAN-DMSO	536 $\pm$ 91.1	17
(3%)CNT-NH <sub>2</sub> /PAN-DMSO	531 $\pm$ 74.3	14
(1%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	556 $\pm$ 77.8	14
(3%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	719 $\pm$ 122.2	17



**Figure 1.** SEM images of a-PAN-DMSO, b-(3%)PANI/PAN-DMSO, c-(1%)CNT-NH<sub>2</sub>/PAN-DMSO, d-(3%)CNT-NH<sub>2</sub>/PAN-DMSO, e-(%1)CNT-NH<sub>2</sub>/(3%)PANI/PAN-DMSO, f-(%3)CNT-NH<sub>2</sub>/(3%)PANI/PAN-DMSO composite nanofibers.



**Figure 2.** FTIR spectra of a)PAN/DMSO b)PAN/CNT-NH<sub>2</sub>/DMSO c)PAN/PANI/CNT-NH<sub>2</sub>/DMSO composite nanofibers.

### Analysis of Mechanical Properties

Mechanical properties of nanofibers can be seen in Table 3. Increased nanofiller may result in an increase of agglomeration leading to the degradation of mechanical and other properties of composite products (27). However, higher breaking strength was obtained for composite nanofiber with 1% CNT and 3% PANI. However, when ANOVA analysis (F test) was carried out, it has been seen that there is no statistically significant strength differences between the samples except for (3%)CNT-NH<sub>2</sub>/(3%)PANI/PAN-DMSO.

### Electrical conductivity of composite nanofibers

Polyacrylonitrile is an insulator material with  $10^{-12}$  S/cm conductivity value (23). The addition of conductive filler such as CNT and conductive polymer such as PANI resulted in an increase of electrical conductivity of composite nanofiber.

Thus composite material became an antistatic material with a conductivity of  $10^{-8}$  S/cm (28).

As seen from Table 4, an increase in conductive filler did not increase the conductivity. This may be due to the agglomeration and void formation around the filler which destroy the network leading to the decrease in conductivity (27).

From the statistical analysis, differences between 3% CNT-NH<sub>2</sub>/PANI/PAN/DMSO and others were found to be significant at 0.05 significance level.

### Analysis of X-ray Diffraction Results

As seen from Table 5, crystallinity of PAN increased with the addition of PANI and CNT. The crystallinity of composite nanofibers with both CNT and PANI is higher than that of pure PAN, PAN with CNT and PAN with PANI.



**Table 3.** Mechanical properties of composite nanofibers.

Sample	Tensile Strength		Tensile Strain		E-modulus	
	(N/m <sup>2</sup> )		(%)		(N/mm <sup>2</sup> )	
	Mean	cv%	Mean	cv%	Mean	CV%
PAN-DMSO	8.64±3.1	35.6	9.0±2.7	30.5	10.6±36.5	36.3
(3%)PANI/PAN-DMSO	8.61±1.1	13.0	73.4±5.2	7.1	59.2±17.1	29.0
(1%)CNT-NH <sub>2</sub> /PAN-DMSO	8.99±2.0	21.9	9.7±2.7	28.3	38.2±17.8	30.6
(3%)CNT-NH <sub>2</sub> /PAN-DMSO	9.35±1.2	13.0	14.0±2.4	17.1	95.7±36.0	37.6
(1%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	10.85±3.1	28.3	14.2±5.1	36.1	87.3±33.1	38.1
(3%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	5.76±1.7	30.0	65.4±11.5	17.6	41.8±14.0	33.6

**Table 4.** Electrical conductivity of nanofibers.

Sample	Conductivity S/cm	CV (%)
(3%)PANI/PAN-DMSO	3.08*10 <sup>-8</sup> ±8.1*10 <sup>-9</sup>	26.60
(1%)CNT-NH <sub>2</sub> /PAN-DMSO	2.80*10 <sup>-8</sup> ±3.9*10 <sup>-9</sup>	14.00
(3%)CNT-NH <sub>2</sub> /PAN-DMSO	2.10*10 <sup>-8</sup> ±7.8*10 <sup>-9</sup>	37.19
(1%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	3.07*10 <sup>-8</sup> ±1.1*10 <sup>-8</sup>	36.12
(3%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	3.89*10 <sup>-9</sup> ±1.2*10 <sup>-9</sup>	32.50

**Table 5.** XRD results of composite nanofibers.

Sample	Degree of Order (%)	PAN (100) (°2θ)	PAN Disordered (°2θ)	PAN (110) (°2θ)	CNT (002)	PANI Peak-1 (°2θ)	PANI Peak-2 (°2θ)	PANI Peak-3 (°2θ)
PAN-DMSO	11.3	16.70	26.60	29.20	-	-	-	-
(3%)PANI/PAN-DMSO	23.8	16.97	26.66	29.3	-	15.1 (broad)	20.0 (broad)	24.0 (broad)
(1%)CNT-NH <sub>2</sub> /PAN-DMSO	21.5	17.04	-	29.2	26.5	-	-	-
(1%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	25.5	16.97	-	29.2	26.5	15.1 (broad)	20.0 (broad)	24.0 (broad)

## Thermal Analysis

DSC results of composite nanofibers are presented in Table 6. Addition of polyaniline into PAN resulted in an increase in cyclization temperature and decrease in enthalpy while CNT had a tendency to increase both the cyclization temperature and the enthalpy. This may be explained by the decrease of molecular movement. However, (3%)CNT-NH<sub>2</sub>/(3%)PANI/PAN-DMSO nanofiber showed lower values which might have been due to an increase in agglomerations and voids with the increase in filler.

**Table 6.** Cyclization temperatures and enthalpy values of nanofibers.

Samples	T <sub>c</sub> (°C)	ΔH (J/g)
PAN-DMSO	303.40	437.7
(3%)PANI/PAN-DMSO	330.62	293.2
(1%)CNT-NH <sub>2</sub> /PAN-DMSO	318.84	411.8
(3%)CNT-NH <sub>2</sub> /PAN-DMSO	323.61	468.0
(1%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	323.25	516.2
(3%)CNT-NH <sub>2</sub> /(3%)PANI/PAN-DMSO	304.32	349.7

## 4. CONCLUSION

Following results have been concluded;

- The diameter of nanofiber increases because of presence of CNT and PANI filler. However, the effect of PANI on the increase in diameter is higher than that of CNT.
- Increased nanofiller may result in an increase in agglomeration leading to the degradation of mechanical

and other properties of composite product. However, higher breaking strength was obtained for composite nanofiber with 1% CNT and 3% PANI. The improvement in the breaking strength compared to pure PAN nanofiber was around 25%.

- Insulator PAN became antistatic material with the use of CNT and PANI. An increase in conductive filler did not increase the conductivity. This may be due to the

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agglomeration and void around the filler which destroy the network leading to the decrease in conductivity.

- Crystallinity of PAN increases with presence of PANI and CNT. The crystallinity of composite nanofiber with both of CNT and PANI is higher than that of pure PAN nanofiber, nanofiber with CNT and nanofiber with PANI.
- Addition of polyaniline into PAN results in an increase in cyclization temperature and decrease in enthalpy while CNT has a tendency to increase both the cyclization

temperature and the enthalpy. PAN with 3% CNT and 3% PANI has the lowest cyclization temperature and enthalpy.

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