www.tubiad.org
ISSN:2148-3736
El-Cezerî Fen ve Mühendislik Dergisi
Cilt: 4, No: 2, 2017 (190-200)

El-Cezerî Journal of Science and Engineering
Vol: 4, No: 2, 2017 (190-200)

ECJSE

Research Paper / Makale

Mechanical Properties of MWCNT Reinforced Polyvinyl Alcohol Nanofiber Mats by Electrospinnig Method

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Received/Geliş: 12.04.2017 **Revised/Düzeltme:** 29.05.2017 **Accepted/Kabul:** 30.05.2017

Abstract: In this study, the production of weight ratios of 1, 3 and 5 % multi-walled carbon nanotube reinforced / unreinforced polyvinyl alcohol (PVA) nanofiber mats was carried out by electrospinning method. Tensile strength, modulus of elasticity and elongation amounts were investigated by tensile tests under static loading with / without reinforced nanofiber mats. % 1 MWCNT reinforced PVA nanofiber mats, $\sigma_{ç}$, E and toughness increased by 50, 88 and 12 %, respectively, when compared with PVA nanofiber mats. Fourier Transform Infrared Spectroscopy (FT-IR) and Differential Scanning Calorimetry (DSC) analyzes were performed on the chemical changes and interactions occurring in the system formed by combining the produced nanofiber mats with PVA and MWCNT. In addition, scanning electron microscope (SEM) images were also investigated to determine the physical properties of reinforced / unreinforced PVA solutions and to monitor the surface porosity of networked polymers at the specification of nanofiber mats.

Keywords: Electro spun, Multi Wall Carbon Nanotubes, Polyvinyl Alcohol, Nanofiber Mats

Elektro-eğirme Yöntemiyle MWCNT Takviyeli Polivinil Alkol Nanoelyaf Keçelerin Mekanik Özellikleri

Özet: Bu çalışmada elektro-eğirme metodu kullanılarak ağırlıkça % 1, 3 ve 5 oranlarında çok cidarlı karbon nanotüp takviyeli/takviyesiz polivinilalkol (PVA) nanoelyaf keçelerin üretimi gerçekleştirilmiştir. Takviyeli/takviyesiz üretilen nanoelyaf keçeler, statik yükleme altında çekme deneyleri yapılarak çekme dayanımı, elastiklik modülü ve uzama miktarları incelenmiştir. % 1 MWCNT takviyeli PVA nanoelyaf keçe, $\sigma_{ç}$, E ve toklukları PVA nanoelyaf keçe ile karşılaştırıldığında sırasıyla % 50, 88 ve 12 artış gözlenmiştir. Üretilen nanoelyaf keçelerin PVA ve MWCNT ile bir araya gelmesi ile oluşan sistemde meydana gelen kimyasal değişimleri ve etkileşimleri Fourier Dönüşümlü Kızılötesi Spektroskopisi (FT-IR) ve Diferansiyel Taramalı Kalorimetre (DSC) analizleri yapılmıştır. Ayrıca nanoelyaf keçelerin özelliklerinin belirlenmesinde takviyeli/takviyesiz PVA çözeltilerinin fiziksel özellikleri ve ağ yapılı polimerlerin yüzey gözenekliliğinin izlenebilmesi amacıyla taramalı elektron mikroskobu (SEM) görüntüleri de incelenmiştir.

Anahtar Kelimeler: Elelktro-eğirme, Çok Cidarlı Karbon Nanotüp, Polivinilalkol, Nanoelyaf Keçeler

1. Introduction

Polymers are essential components of our daily life and one of the basic elements of new materials. Depending on recent studies, polymers have become important materials which facilitate human life through manufacturing and use of different plastic, fibre, elastomer types. Polymers are light, cheap,

How to cite this article
Ekrem, M., "*Mechanical Properties of MWCNT Reinforced Polyvinyl Alcohol Nanofiber Mats by Electrospinnig Method*" El-Cezerî Journal of Science and Engineering, 2017, 4(2); 190-200.

Bu makaleye atıf yapmak için
Ekrem, M., *"Elektro-eğirme Yöntemiyle MWCNT Takviyeli Polivinil Alkol Nanoelyaf Keçelerin Mekanik Özellikleri"* El-Cezerî Fen ve Mühendislik Dergisi 2017, 4(2); 190-200.

easy-processed with sufficient mechanical characteristics, high thermal and electrical characteristics as well as high chemical and corrosion resistance. Due to such superior characteristics, polymers are used in many fields such as aircrafts, sports tools, medicine, chemistry, automotive [1-4].

Poly(vinyl alcohol) (PVA) is a water soluble polyhydroxy polymer and the most manufactured resin all over the world. An excellent chemical resistance, physical characteristics and biological degradability caused wide usage tendency of PVA Furthermore, high inflation characteristics in water and biological fluids and elastic structure enable use of this material in manufacturing of different fields such as contact lenses, skin, artificial cartilage and drug-release systems [5-7].

The most efficient method for polymer-based nanofiber mat manufacturing is electro-spinning. Continuous nanofiber mats with varying thickness and diameters from 3 nm to 1 µm are obtained from many types of polymer through this method. Structure and morphology of nanofiber mats may be controlled efficiently by some parameters including polymer type, solution concentration, effect of the solvent, the voltage applied, the distance between the syringe and the collector, solution feeding rate and electrical conductivity. Depending on some characteristics such as highly specific surface area, higher width/length ratio and high porosity, many polymers and inorganic/polymer nanofiber mats were searched for development of PAN, PVA, Nylon-6.6 electro-spinning technology. These nanofiber mats present interesting implementations like filtration, nano-electronic, protective clothing, tissue skeleton, optic sensor etc [6, 8, 9].

Many studies were conducted to investigate manufacturing conditions, molecule weight and effects on solution compound of PVA nanofiber mats produced from PVA water solutions during recent year [10, 11]. However, PVA nanofiber mats manufactured through electro-spinning method were not strong enough for many applications because of poor molecular chain orientation along fiber axis originated from low tension forces during formation of the finer. Furthermore, semi-crystal structure of PVA caused free spinning of PVA nanofiber mats under regular orientation. Many approaches including cross linking, final procedure and blending were utilized during manufacturing of PVA nanofiber mats produced through electro-spinning method improvement of the characteristics (i.e. low mechanical resistance) and functionality (i.e. biomedical implementation). Involvement of nanoparticles into PVA matrix was considered to be one of the most efficient methods for PVA nanofiber mat reinforcement manufactured through electro-spinning method [12].

In the present study, multiple walled carbon nanotubes (MWCNT) were added by 1, 3 and 5% in weight into the polyvinyl alcohol (PVA) solution. From the prepared solution, PVA nanofiber mat manufacturing was performed by 4 ml solution and electro-spinning method via a 0.8 mm injector. Nanofiber mats produced with/without reinforcement were exposed to tensile tests under static loading and tensile strength, elastic modules and toughness were examined and compared with each other. Chemical changes and interactions of nanofiber mats produced were analysed through Fourier Transformational Infra-red Spectroscopy (FT-IR) and Differential Screening Calorimetry. Furthermore, screening electron microscope (SEM) images were also reviewed to be able to monitor surface porosity of reticulated polymers as well as physical characteristics of PVA solutions with/without reinforcement for determination of mat characteristics.

2. Experimental

2.1. Materials

MWCNT was manufactured by NANOCYL company through chemical vapour accumulation method in the present study. Diameter is 5 to 50 nm and the length is 10 to 30 µm. The PVA polymer may be made a solution by adding purified water and a well spinning may be done. Closed formula of PVA is [-CH2CHOH-]n and molecular weight is 124000 g/mole. It was supplied by Sigma Aldirch company. Sodium dodecyl sulphate (SDS) with a closed formula of $CH_3(CH_2)_{11}OSO_3Na$, a general anionic surface active substance purchased from Merck company has an analytical purity with a molecular weight of 288.38 g/mole.

2.2. PVA Nanofiber Mats Manufacturing by Electro-Spinning Method

Solution preparation was started with PVA attainment. As shown in Figure 2.1., 10 g of PVA powder (10% w/w) was added into 90 g of pure water which was heated up to 70°C slowly. This mixture was mixed by a magnetic mixer for 3 hours and complete dissolution of PVA was achieved. The pure water disappeared from totally 100 g of solution was added and mixture was continued. A solution was prepared with a g of surface active substance, sodium dodecyl sulphate (SDS) and 99 g of pure water and mixed in a magnetic mixer for 10 minutes. One gram of 1% SDS solution was added versus 10 g of PVA solution to reduce surface tension. MWCNT by 1%, 3% and 5% in weight was added into PVA-SDS solution per 10 g of PVA. The final solution was mixed in an ice bath for 5 minutes by 15-minute intervals through a probe homogenizator.

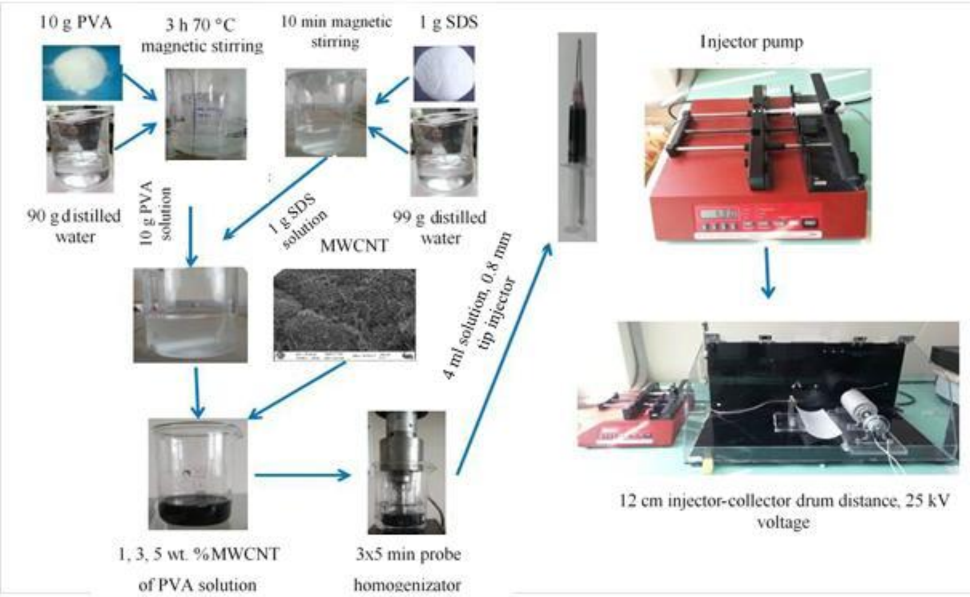


Figure 2.1 Schematic presentation of PVA nanofiber mats manufacturing procedure by electro-spinning method [13]

The samples exposed to spinning procedure were put into the electro-spinning mechanism prepared as rotating drum at horizontal axis. The syringe pump was selected with a 1.30 ml/h rate; syringe-collector distance was selected as 12 cm. High tension power source was adjusted at 25 kV and the experiment was started. The experiment was carried out in a laboratory environment at a room temperature of 26°C. Fiber samples obtained were dried in a drying-oven at 60°C for 1 hour.

Thickness of PVA nanofiber mats manufactured through electro-spinning method was measured by SEM and micrometre and shown in Figure 2.2. First, PVA nanofiber mats were cut by 20x20 mm in size. Thickness of the cover glass was measured as 1.07 mm by a micrometre. Then, nanofiber mats are put between two cover glasses and measured as 0.13 mm (Figure 2.2). Mean thickness was measured as 135µm by SEM (Figure 2.2.b).

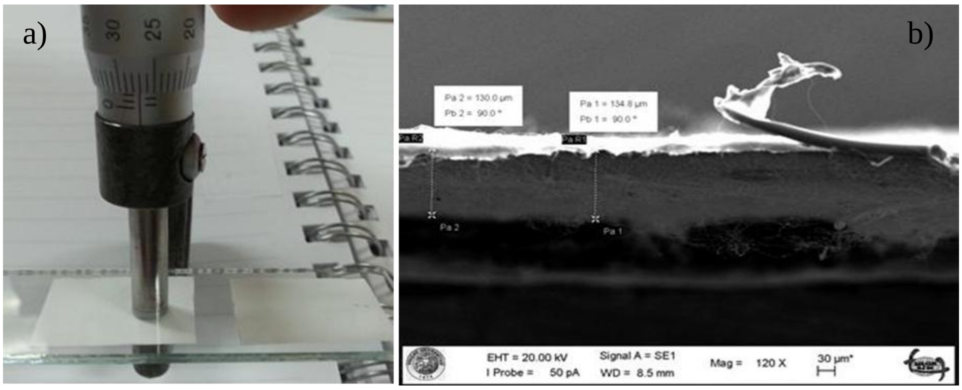


Figure 2.2. Thickness measurement of nanofiber mats

2.3. Tensile Tests of Nanofiber Mats

The PVA nanofiber mats manufactured through electro-spinning method were cut sensitively in 200x20 mm dimension with the separator fabric according to ASTM D 882-02 standards (Figure 2.3). After cutting, the separator fabric was peeled off from nanofiber mats. A double-sided polyurethane foam band with a thickness of 1.6 mm was stuck on both sides to avoid breakage and shedding of pulling jaws. Moreover, four samples from each sample were prepared.

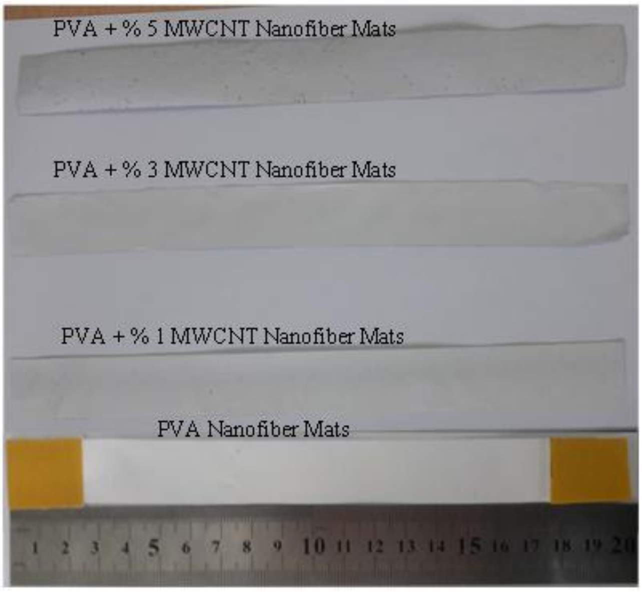


Figure 2.3 Dimensions of PVA nanofiber mats (in mm)

Tensile tests were performed by Shimadzu AGS-X hydraulic desktop test device at room temperature. Load cell with a precision of 10 ±0.05 kN was used for load measurements. As seen in Figure 2.4, connection of PVA nanofiber mats to the pulling test device was shown. Device jaws were adjusted at 15 mm/min during tensile procedure.

Figure 2.4 Connection of the samples to the pulling test device

The load applied to each sample, P and replacement, δ values were recorded instantly and tensile strength of the samples were calculated with the following equation;

$$\sigma_i = \frac{P_i}{A} \tag{2.1}$$

$$\sigma_{max} = \frac{P_{max}}{A} \tag{2.2}$$

In Equation 2.1, σ_i represents the tension appeared at force i (MPa), P_i represents the load applied (N), A represents average section area (mm^2) and in equation 2.2., P_{max} represents the maximum load that the sample can carry (N) and σ_{max} represents the maximum tension appeared on the sample (MPa).

Elasticity module;

$$E_f = \frac{\Delta\sigma}{\Delta\varepsilon} \tag{2.3}$$

Equation 2.3 used to calculate tensile elasticity module where E_f represents tensile elasticity module (MPa), $\Delta\sigma$ represents the difference between two tension point (MPa) and $\Delta\varepsilon$ represents the difference between two selected transformation quantity (normally $\Delta\varepsilon = 0.002$ is used and the difference on tension points between these values determines the magnitude of the elasticity module).

Data obtained by use of the equations above were made numerical and data were compared with each other by converting into tables.

2.4. Characterization of PVA Nanofiber Mats

SEM images of PVA nanofiber mats were obtained by ZEISS Evo LS 10 device at 20kV voltage. Glass transition temperature, specific heat capacity, thermal stability and melting temperature were examined with differential screening calorimeter (DSC, Perkin Elmer Instruments) under 20 ml/min nitrogen flow conditions. Test temperature was tested at 20°C/min heating temperature from 25°C to 400°C.

3. Conclusions and Discussion

3.1. Determination of Physical Characteristics of PVA Solutions

Electrical conductivity, temperature and total particle quantity (ppt) of the solutions were measured by Hanna-Combo tester device under the temperatures set out in Table 3.1.

Table 3.1 Physical characteristics of liquid solutions

Solution	Ppt	Conductivity (mS/cm)	Temperature (°C)
PVA	0.72	1.45	23.2
PVA / 1% MWCNT	0.75	1.50	23.7
PVA / 3 % MWCNT	0.78	1.56	23.9
PVA / 5 % MWCNT	0.81	1.62	23.1

As seen in Table 3.1, electrical conductivity increases by increase of MWCNT added into the solvent. Since electrical conductivity of MWCNT is 106-107 S/m, conductivity of the solvent increases depending on the particle quantity by increase of MWCNT. Total particle quantity in the solution also increased. Increase of total particle quantity increased depending on increase of MWCNT quantity. Furthermore, nanofiber mats changes from white to grey colour by MWCNT increase as seen in Figure 2.3.

3.2. DSC Analyses of PVA Nanofiber Mats

DCS curves of PVA nanofiber mats with/without MWCNT reinforcement by 1, 3 and 5% in weight are shown in Figure 3.1. After manufacturing of PVA nanofiber mats and PVA nanofiber mats reinforced by MWCNT by 1, 3 and 5% in weight through electro-spinning method, the water was evoparated by drying in a vacuum oven at 60°C for 1 hour. DSC curves demonstrated no reaction until melting temperature. No crystallization was observed during DSC heating study according to DSC curves. Therefore, endothermic melting was referred to melting of the crystals appeared during electro-spinning [14].

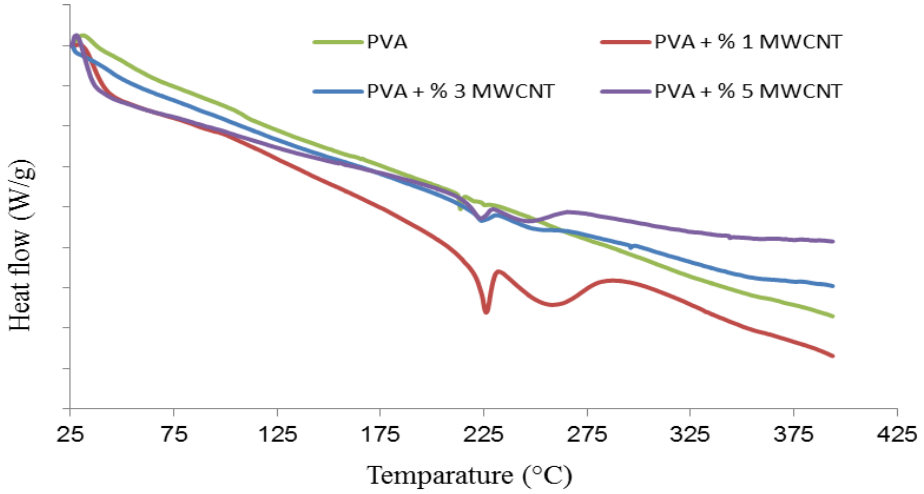


Figure 3.1 DSC curves of PVA nanofiber mats with/without MWCNT reinforcement by 1, 3, and 5% wt.

Glass transition temperature (T_g), specific heat values (ΔC_p), thermal stability and melting temperature (T_m) of nanofiber mats were listed in Table 3.2. When PVA nanofiber mat reinforced by 1% MWCNT in weight was compared with PVA nanofiber mats and T_m, the temperature increased from 215°C to 225°C. However, a decrease was observed in this increase after the ratio of 1% and this is caused by agglomeration in PVA matrix of MWCNT [15]. PVA nanofiber mats have an endothermic peak at 205 to 225°C corresponding to T_m [16].

Table 3.2. T_g, □C_p, thermal stability and T_m of PVA and MWCNT reinforced PVA nanofiber mats

Nanofiber mats	T_g (°C)	ΔC_p (J/g °C)	Thermal stability (°C)	T_m (°C)
PVA	77	0,066	205	215
PVA / 1% MWCNT	85	0,895	215	225
PVA / 3 % MWCNT	80	0,446	212	225
PVA / 5 % MWCNT	74	0,296	210	223

A significant increase was observed in ΔC_p, T_g, thermal stability and Tm in PVAV nanofiber mats reinforced by 1% MWCNT and such values have become 0.895 J/g ºC, 85 ºC, 215 ºC and 225 ºC, respectively. The ΔC_p value for PVA nanofiber mats was 0.066 J/g ºC, such value increased by approximately 14 folds and became 0.895 J/g °C in 1 % MWCNT + PVA nanofibers. Such value decreased by increase of MWCNT quantity. T_g, Tm and thermal stability temperatures also decreased depending on this.

3.3. Mechanical Properties of Pva Nanofiber Mats

The typical tension-unit transformation graph obtained as a result of static tensile experiments of PVA nanofiber mats with/without MWCNT reinforcement was presented in Figure 3.2. As seen in the figure, tensile strength of PVA nanofiber is 3.91 MPa. PVA nanofiber mats reinforced by 1% MWCNT presented an increase of 50% by 5.87 MPa. Moreover, unit transformation of PVA nanofiber mat is 0.58 mm/mm whereas unit transformation of PVA nanofiber mats reinforced by 1% MWCNT decreased 29.3% by 0.41 mm/mm. However, a decrease was detected in tensile strength and ductility by MWCNT reinforcement.

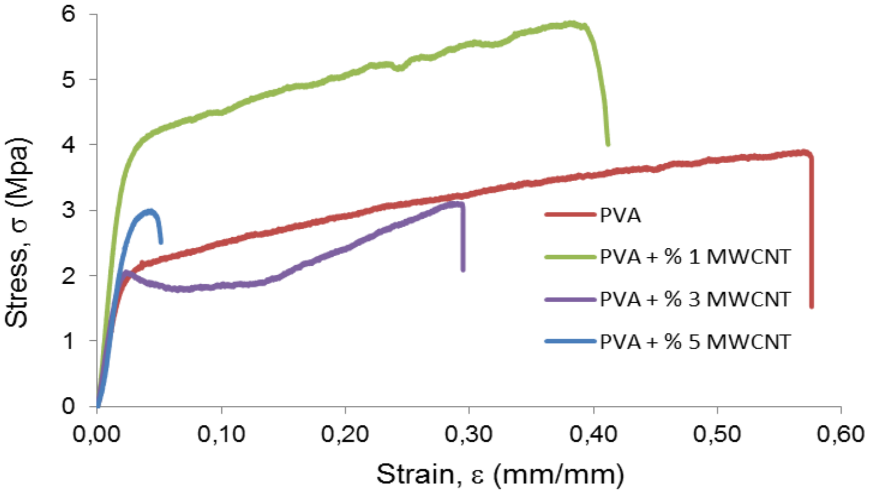


Figure 3.2 Stress-Strain graph for PVA nanofiber mats with/without MWCNT reinforcement

Maximum loads (P_{max}), elasticity modules (E), tensile strengths ($\sigma_ç$), strain (ε) and toughness of PVA nanofiber mats with/without MWCNT reinforcement were presented in Table 3.3. % 1 MWCNT takviyeli PVA nanoelyaf keçenin P_{max}, elasticity module and toughness of PVAV nanofiber mat reinforced by 1 % MWCNT were 15.86 N, 0.203 GPa and 344.15 MJ/m3, respectively and showed an increase by 50 %, 88 % and 12 % , respectively when compared with PVA nanofiber mats. However, the decrease appeared by increase in MWCNT ratios and presented a brittle breakage. The reason for the decrease after 1% MWCNT reinforcement is agglomeration of MWCNT and reduction of diameters of the nanofiber mats due to MWCNT.

Table 3.3 P_{max}, E, $□_ç$, □ and toughness of PVA nanofiber mats with/without MWCNT reinforcement

Nanofiber mat	P_{max} (N)	E (GPa)	$\sigma_ç$ (MPa)	ε(mm/mm)	Toughness (MJ/m^3)
PVA	10.55	0,108	3.91	0.58	307.37
PVA / 1% MWCNT	15.86	0,203	5.87	0.41	344.15
PVA / 3 % MWCNT	8.41	0,135	3.11	0.29	106.48
PVA / 5 % MWCNT	8.10	0,123	3.00	0.05	29.95

Figure 3.3 shows the images of PVA nanofiber mats with/without MWCNT reinforcement after the breakage. The PVA nanofiber mat and PVA nanofiber mats reinforced by 1% MWCNT presented a ductile behaviour and fracture occurred. However, PVA nanofiber mats reinforced by 3 % MWCNT and PVA nanofiber mats reinforced by 5 % MWCNT were broken fragilely. Short fiber formation and fracture initiation resulted with brittle fracture by effect of pilling in nanofiber mats due to increase in MWCNT ratios.

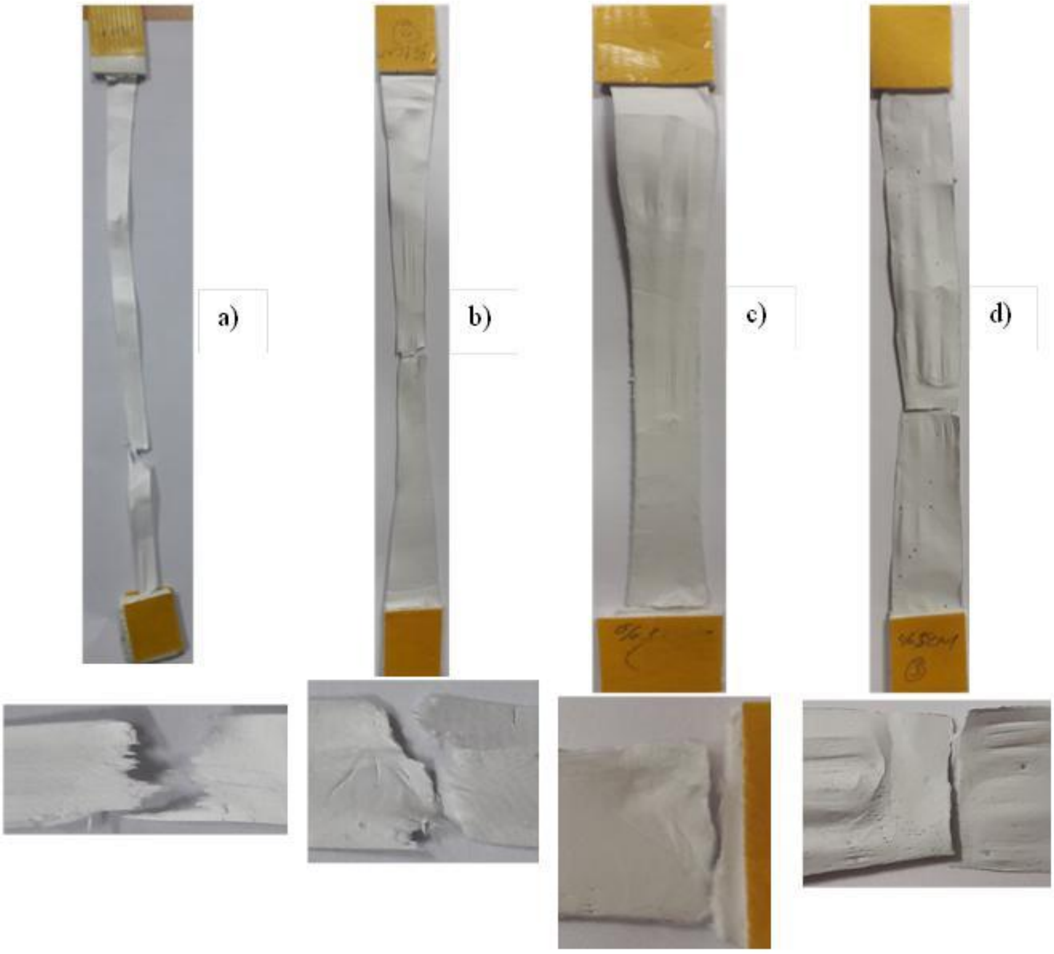
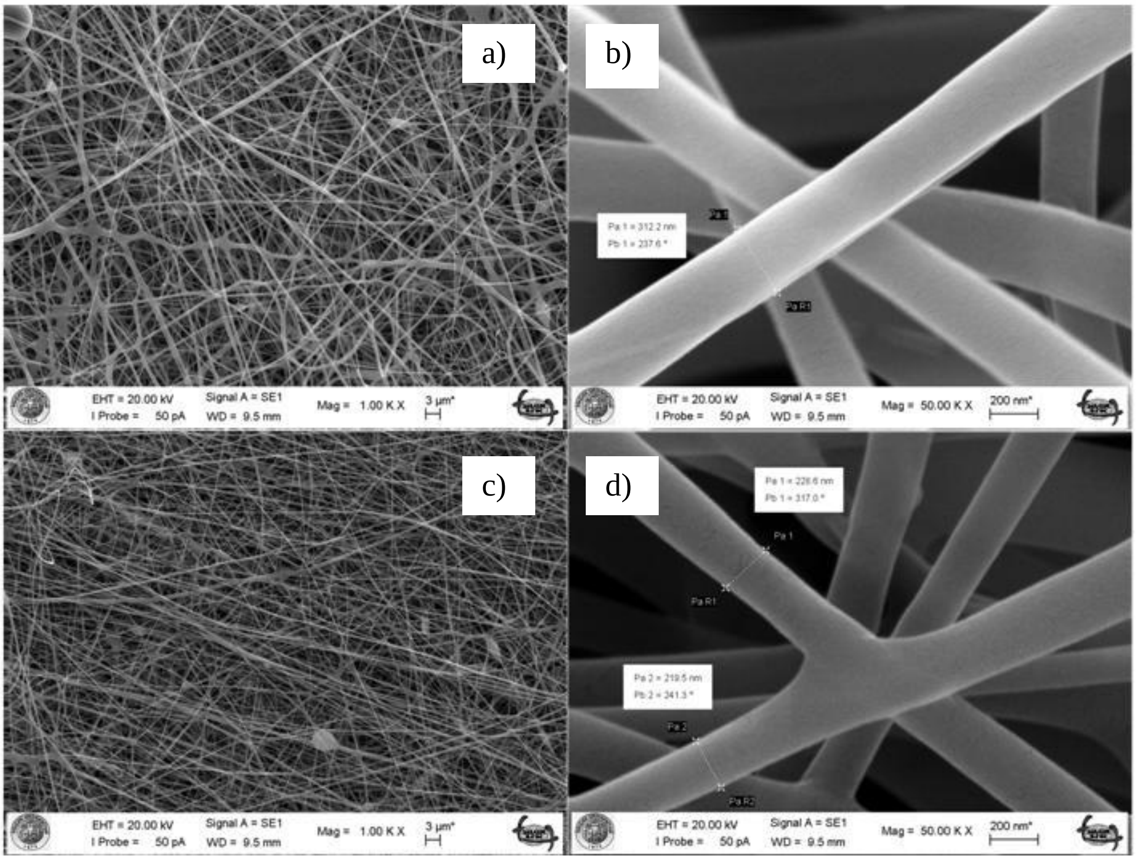


Figure 3.3 Images after rapture under static load; a) PVA nanofiber mat, b) PVA nanofiber mats reinforced by 1 % MWCNT c) PVA nanofiber mats reinforced by 3 % MWCNT and d) PVA nanofiber mats reinforced by 5 % MWCNT

3.4. Morphology of PVA Nanofiber Mats

SEM images of nanofiber mats at 1000 X and 50,000 X magnification were presented in Figure 3.4. Furthermore, SEM images were pasted into Autocad program and the smallest and largest 10 diameters of each nanofiber mat were measured and average was calculated. Diameter measurements of PVA nanofiber mats were measured between 222 and 403 nm; diameter measure of PVA+1% MWCNT nanofiber mats was measured between 154 and 335 nm; diameter measure of PVA+3% MWCNT nanofiber mats was measured between 117 and 265 nm and diameter measure of PVA+5% MWCNT nanofiber mats was measured between 95 and 192 nm (Figures 3..b, d, f, h).

If electrical conductivity of the solution is increased in electro-spinning process, loads of the solution would also increase. Thus, proper fibres and fibres with smaller diameter may be obtained. Diameter measure of PVA nanofiber mats was compared with diameter measure of PVA+5% MWCNT nanofiber mat and it was measured as 110 nm with a decrease by 64.7%. When such image is examined, diameters of nanofiber mats decrease by increase of KNT added into the PVA solution due to MWCNT conductivity and thin fibres appear. However, by increase of KNT quantity, agglomeration and viscosity increase, pumping of the solution from the syringe becomes difficult and cause agglomeration (Figures 3.., a, c, e, g). Interruptions appear in fibre lengths [17].



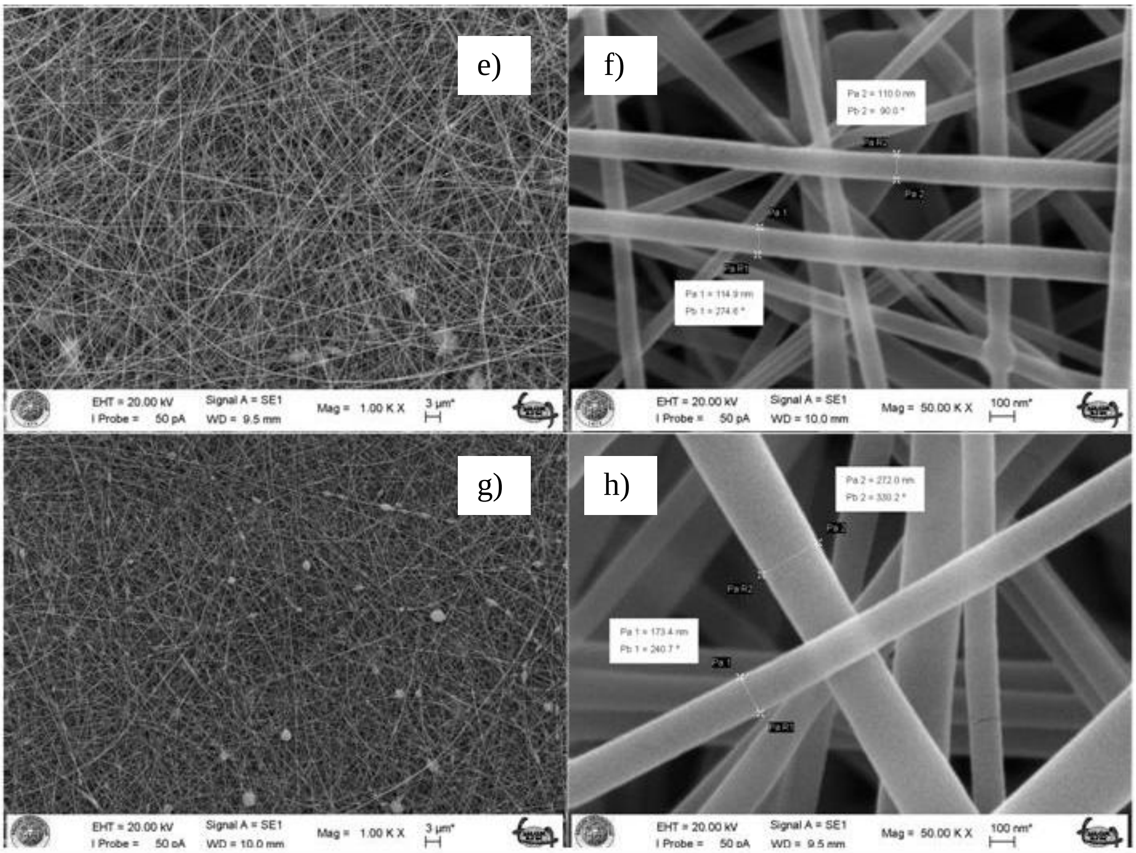


Figure 3.4. SEM images of PVA nanofiber mats; a) 1K X and b) 50 KX PVA, c) 1 KX and d) 50 KX PVA+% 1 MWCNT, e) 1 KX and f) 50 KX PVA+% 3 MWCNT, g) 1 KX and h) 50 KX PVA+% 5 MWCNT

4. Conclusions

Elasticity modules, strain, tensile strengths and toughness of PVA nanofiber mats with/without MWCNT reinforcement manufactured by electro-spinning method were analysed in the present study. Furthermore, characterizations of nanofiber mats as well as physical features of the solution through addition of MWCNT by 1, 3, and 5 % in weight into PVA solution were examined via SEM and DSC. As a result:

1- When PVA nanofiber mat reinforced by 1 % MWCNT, $\sigma_ç$ and E was compared with PVA nanofiber mat, increases by 50 % and 88 % were detected, respectively. A decrease appeared after 1 %. This indicated the best leakage threshold in PVA solution.
2- As a result of DSC analyses, nanofiber mats demonstrated an endothermic reaction. A significant increase was observed in ΔC_p, T_g, thermal stability and Tm of PVA nanofiber mats reinforced by 1% MWCNT and resulted as 0.895 J/g ºC, 85 ºC, 215 ºC and 225 ºC, respectively. A decrease was observed in these values after 1 % ratio and this outcome was referred to agglomeration by increase of MWCNT in PVA matrix.
3- When SEM morphology was examined, diameters of nanofibers decreased by increase of MWCNT ratio. Furthermore, no pilling was observed and a long fibre manufacturing was performed through reinforcement of 1% MWCNT. Pilling and shorter lengths of mats were detected by increase of MWCNT ratio. It was also understood that nanofiber mats reinforced by 1% MWCNT have ideal diameters.

5. Acknowledgments

The financial support for this work has been received from Necmettin Erbakan University, Scientific Research Projects Administration Grant No. 161219006.

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