

Investigation of PAN:Hemp Stems Nanofibers Produced by Electrospinning Method

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Abstract

In this study, the hemp stem (cannabis) nanofibers have been produced employing the electrospinning method by changing parameters including voltage, and deposition time. The nanosized particles of hemp stems have been mechanically produced by ball milling technique. The powder hemp stem obtained by ball-milling have been prepared in polyacrylonitrile (PAN) polymer with N, N-dimethylformamide (DMF) solution. The optical, morphological and chemical bonding properties of the obtained hemp nanofibers have been analysed employing Ultraviolet-Visible-Near infrared (UV-Vis-NIR) spectroscopy, Scanning Electron Microscopy (SEM) and Fourier Transform Infra-Red (FTIR) spectroscopy, respectively. The diameters of hemp stems nanofibers with PAN polymer have been obtained ranging from 30 to few hundreds of nanometre. Absorbance spectrum of PAN: Hemp stems have been plotted covering from UV to infrared region. Energy band gap value has been calculated as 3.5 eV using Tauc-plotting equations. PAN: Hemp stems has absorbed more photons in UV and visible regions than infrared region. In order to determine the thermal endurance of the produced hemp nanofibers, Thermogravimetric Analysis (TGA) has been carried out for temperatures up to 800 °C. TGA measurements has inferred that both PAN and Hemp stem nanofibers continue losing weight gradually after first sharp decrease at around 300 °C and almost completely disintegrate at 800 °C.

Keywords: Cannabis sativa, electrospinning, hemp stem, nanofiber

Elektro Eğirme Yöntemiyle Üretilen PAN:Kenevir Saplı Nanoliflerin İncelenmesi

Öz

Bu çalışmada, voltaj, substrat-igne uzaklığı ve biriktirme süresi gibi parametreler değiştirilerek elektro-eğirme yöntemi kullanılarak kenevir sapı (kenevir) nanolifleri üretilmiştir. Kenevir saplarının nano boyutlu parçacıkları, bilyalı öğütme tekniği ile mekanik olarak üretilmiştir. Bilyalı öğütme ile elde edilen toz kenevir sapı poliakrilonitril (PAN) polimerinde N,N-dimetilformamid (DMF) çözeltisi ile hazırlanmıştır. Elde edilen kenevir nanoliflerinin optik, morfolojik ve kimyasal bağlanma özellikleri sırasıyla Ultraviyole-Görünür-Yakın kızılötesi (UV-Vis-NIR) spektroskopisi, Taramalı Elektron Mikroskopu (SEM) ve Fourier Dönüşümü Kızılötesi (FTIR) spektroskopisi kullanılarak analiz edilmiştir. PAN polimerli kenevir sapı nanoliflerin çapları 30 ila birkaç yüz nanometre arasında değişmektedir. PAN'ın absorpsiyon spektrumu: Kenevir sapları UV'den kızılötesi bölgeye kadar çizildi. Enerji bant aralığı değeri, Tauc-plotting denklemleri kullanılarak 3.5 eV olarak hesaplanmıştır. PAN: Kenevir sapları, UV ve görünür bölgelerde kızılötesi bölgeye göre daha fazla foton emmiştir. Üretilen kenevir nanoliflerinin termal dayanıklılığını belirlemek için 800 °C'ye kadar olan sıcaklıklar için Termogravimetrik Analiz (TGA) yapılmıştır. TGA ölçümleri, hem PAN hem de Kenevir sapı nanoliflerinin 300 °C civarındaki ilk keskin düşüşten sonra kademeli olarak ağırlık kaybetmeye devam ettiğini ve 800 °C'de neredeyse tamamen parçalandığını ortaya çıkardı.

Anahtar Kelimeler: Cannabis sativa, elektrospinning , kenevir sapı, nanolif

INTRODUCTION

Electrospinning method is known as high efficiency technique to fabricate polymer nanomicrofibers. Producing fibers in nano-micro diameters required in this technique, synthetic polymers including polyurethane, poly (methyl methacrylate), polyvinyl alcohol, ethylene oxide (Goroškaitė, 2020) and natural polymers such as hydroxyapatite, chitosan, poly gelatin, polyhydroxybutyrate and etc polymers in recent years as solvent solution (Cassano et al., 2013; Fahimirad et al., 2021; Kalantari, Afifi, Jahangirian and Webster, 2019; Sutka, Gravitis, Kukle, Sutka, and Timusk, 2015). Hemp stem based materials have larger specific application areas from sound absorption (Liao, Zhang and Tang, 2020) to drug delivery applications (Andriotis et al., 2021).

Electrospun nanofiber mat is a good candidate for wound dressing due to its well-connected porous structure and very specific surface area to inhibit external microorganism invasions and expel fluid. Since drug loading by electrospinning is very easy, electrospun nanofibers have demonstrated many advantages as potential drug carriers (Fang, Wang and Lin, 2011). Electrospun nanofibrous membranes are lightweight, large surface area, high porosity (breathable nature), high filtration efficiency, etc., which are also desirable properties in protective clothing and are considered as potential apparel applications. It has been observed that electrospun nanofibers laid in a layer with high porosity but small pore size provide resistance against chemical harmful agents in spray form (Gibson, Schreuder-Gibson and Rivin, 1999).

In addition, electrospun fibers are possible to make some three-dimensional structures as they have a sizable static charge during their deposition. This allows them to become an excellent candidate for use in filtration (Lv et al., 2018), membrane applications (Gao et al., 2020), scaffolds for tissue engineering (Lin, Chen, Qu, Li and Man, 2020), drug delivery and release systems (Kamsani, Haris, Pandey, Taher, and Rullah, 2021), wound healing applications (Liu, Zhou, Gao and Zhai, 2019) etc. Polyvinylpyrrolidone (PVP) (also known as povidone) is a water and other polar solvent soluble, biodegradable and biocompatible polymer. PVP has been used as a binder in many pharmaceutical tablets,

that is why it is usually mixed with other polymers to possess better mechanical stability (Kariduraganavar, Kittur, and Kamble, 2014). Another most used polymer in electrospinning method is poly(vinyl alcohol) (PVA) which has many application areas including filtration of undesirable chemicals, biomedical applications, membranes, drug delivery & release, optics and protective clothing (Kamoun, Loutfy, Hussein and Kenawy, 2021; Khanzada et al., 2020; Mohtaram et al., 2020; Yang, Qin and Wang, 2008). Cannabis sativa L. possess some biological activity because of its chemical structure. The main nonpsychotropic phytocannabinoid that are present in Cannabis sativa L. plant which are Cannabigerol (CBG) and Cannabidiol (CBD) (Andriotis et al., 2021; Deiana, 2017). Novel studies show that CBG has a broad pharmacological profile and can be suggested for cancer treatment, exhibits anti-inflammatory and analgesic specifications and therefore it also can be applied for treatment of skin conditions (Deiana, 2017). Nevertheless, there are no studies that shows CBG potential in electrospinning technique, so all the investigations and tests are important to analyze the possibility of applying Cannabis sativa L. in textile nonwoven forming technology.

There has recently been a rapid transition from macro to microstructures in the globalizing world with the rapid development of technology. Electrospinning technique firstly reported in 1897, that produces thin (nm to μm) polymer fibers by using electrostatic forces. Ordinary setup of electrospinning technique (with syringe type electrode) is used in this technique. A high electric field is applied to a polymer solution or melt which is held together by its surface tension at the tip of a syringe (Xue, Wu, Dai and Xia, 2019).

Electrospinning technique has some advantages of providing ease of application for parametric studies such as adjusting the application voltage, the needle and substrate distance and the flow rate. After applying high voltage, mostly between 1 kV and 30 kV, the pendant drop of polymer solution becomes highly electrified, and the induced charges are evenly distributed over the surface. By using a syringe pump polymer solution is pumped through the needle with a particular feeding rate. Upon increasing the

Research article/Araştırma makalesi
 DOI:10.29132/ijpas.1092339

electrical field strength (increasing applied voltage), repulsive electrical forces prevail the surface tension forces of polymer drop, and the drop become a cone with angle of $49,3^\circ$ (i.e. Taylor cone, the equilibrium shape of a dripping solution at a critical voltage). Then a critical value of applied voltage is reached, a jet from Taylor cone is formed, resulting in the ejection of a charged jet of the solution (Agarwal, Burgard, Greiner and Wendorff, 2016; Hu et al., 2014; Theron, Zussman and Yarin, 2004). Geometry (diameter undulations and branching processes) of fiber can be affected by the processes such as Rayleigh instabilities as well as the electrically driven axi-symmetric instabilities. The jet coming out

of the tip in the beginning for a very short time flows in a straight path and then bending, winding, looping and curling of the jet occurs. Stationary collectors can provide randomly oriented fibers, while aligned fibers can be collected on rotating collectors (Huang, Zhang, Kotaki and Ramakrishna, 2003). The electrospinning technique has several important advantages: easily achieved production of very thin fibers with a large surface area; produced fibers are easy to functionalize; controlled porosity of electrospun material; possibility to create a 3D structure. Electrospun nano-microfibers are flexible to spin into a variety of different shapes and sizes as well as to form a structure of controlled porosity for

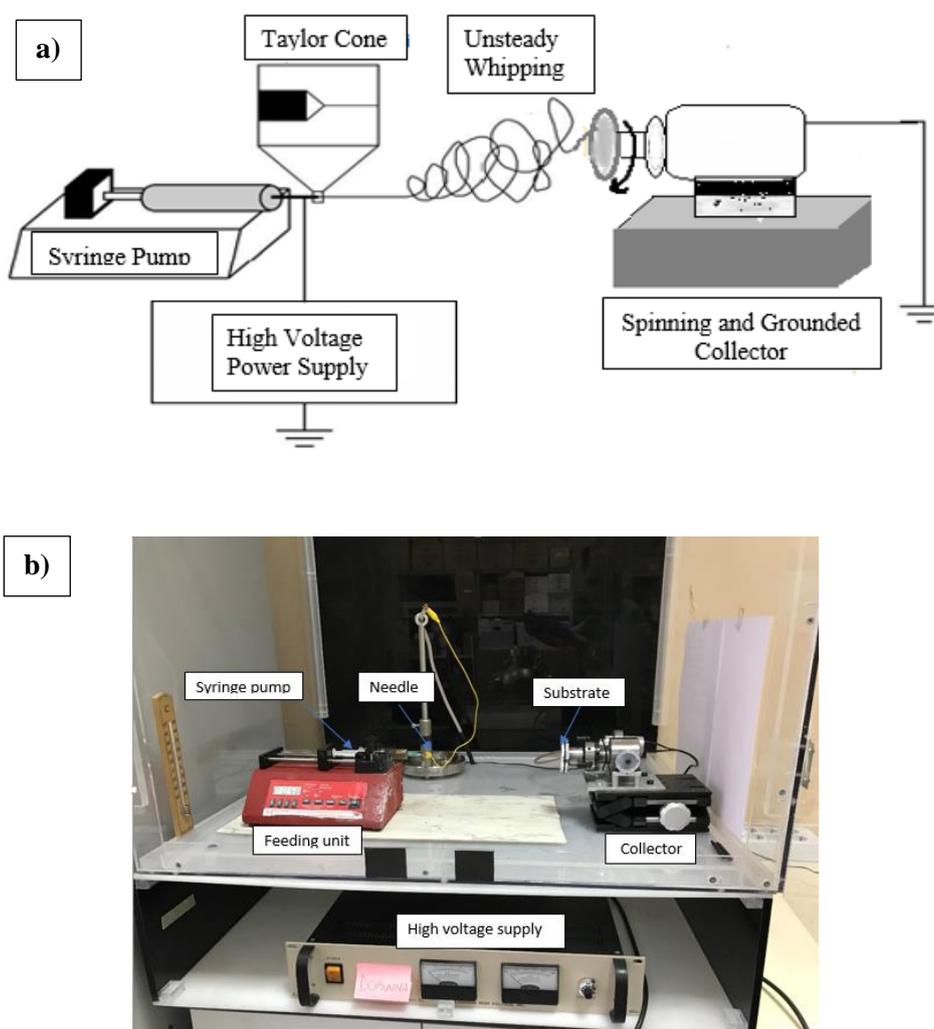


Figure 1. a) Schematic diagram of electrospinning system, b) Experimental setup of the Electrospinning system

each specific application (Stepanyan et al., 2016; Zaarour, Zhu and Jin, 2020). Moreover,

electrospinning technique allows to functionalize the nano-microfibers during preparation. It can be done

by incorporating viruses, bacteria, enzymes, drugs, catalysts, metal nanoparticles, nanotubes and nanowires (Agarwal et al., 2016; Goroškaitė, 2020). The research was done to analyse and understand the possibility of incorporating hemp extract into the polymer solution and create a bioactive material that could be used for biomedical purposes.

In this study, the hemp stems (cannabis) nanofibers were obtained by employing the electrospinning method, which is one of the most effective and practical methods in the production of polymer-based nanofibers, compared to the traditional nanofiber production methods. Initially, the nanosized particles of hemp stems were mechanically produced by ball milling technique. The obtained Hemp stem's powder were prepared in polyacrylonitrile (PAN) polymer solution. The nanofibers of hemp stems with PAN polymer were produced by utilising electrospinning method. Nanofibers ranging from 30 to few hundred nanometres were produced. The optical, morphological and chemical bonding properties of the obtained Hemp nanofibers were analysed by employing Ultraviolet-Visible-Near infrared (UV-Vis-NIR) spectroscopy, Scanning Electron Microscopy (SEM) and Fourier Transform Infra-Red (FTIR) spectroscopy, respectively. In order to determine the thermal endurance of the produced Hemp nanofibers, Thermogravimetric Analysis (TGA) was carried out at temperatures starting from 20 to up to 800 °C.

MATERIAL AND METHODS

For the synthesis of nanofibers in the study, PAN (polyacrylonitrile, C_2H_3NO) polymer, DMF (N,N-dimethylformamide, C_3H_7NO) and hemp stems ($C_{21}H_{30}O_2$) were employed (Hemp stems were obtained commercially from 3A Girisim ve Tarim Egitim Danismalik Sanayi Ticaret Limited Sirketi). The nanofibers were produced using an electrospinning device as shown in Figure 1. The electrospinning device used in the production of nanofibers consists of 3 main parts. These are the feeding unit (syringe pump), red device on the lefthand side in Figure 1, where the prepared solution is placed, the collector (rotating aluminium plate) and the high voltage power supply. A new era syringe pump was utilized to deliver the polymer solution to the collector in a controlled manner, and a Glassman

High Voltage Series EL high voltage power supply was employed to provide the high voltage that is required to produce nanofibers.

In this study, the power supply can apply DC power up to a maximum of 50 kV. An Aluminium plate that has a diameter of 5 cm was used as a collector (substrate). The reason for using Aluminium plate is that as high voltage applied between the syringe pump's needle and collector plate at a certain distance, an enormous electric field (approx. 75000 V/m) is produced and thus, a conductor was needed to conduct it. For nanofiber synthesis, 6% PAN (polyacrylonitrile) solution in DMF (N,N-dimethylformamide) was prepared. The prepared solution was mixed in the homogenizer device for 2 hours and left for one night in a container. After PAN polymer solution prepared in the first step was drawn into a plastic syringe with a needle diameter of 800 μ m, the syringe was placed in the feeding unit. Then, it was transferred to the syringe pump for nanofiber production using electrospinning system. The produced nanofibers were deposited in Aluminium plate by electrospinning method. The deposition times duration were change from 1 to 15 minutes. The high voltage supply applied high voltage between 15 to 20 kV. The nanofibers with various diameters and thickness were produced. The optical properties of the produced nanofibers were measured by UV-Vis spectroscopy. The surface morphology and cross-section of hemp nanofibers was characterised using SEM images. The chemically active bonds of the produced hemp nanofibers were measures by utilising FTIR spectroscopy. Moreover, thermal properties of the produced hemp nanofibers were characterised by TGA measurements.

RESULTS AND DISCUSSION

The optical properties of obtained hemp nanofibers were characterized by employing UV-Vis-NIR spectroscopy. Figure 2a demonstrates UV-Vis absorbance spectrum of produced nanofibers for 6 % PAN and Hemp stem with deposition time of 10 minutes and 20 cm substrate needle separation with applied voltage of 20 kV. The most of absorption takes place in UV wavelength region while absorption decreases gradually in longer wavelengths. Energy band gap value of PAN:Hemp stems nanofibers on glass substrate was calculated using absorbance data as 3.5 eV applying Tauc

Research article/Araştırma makalesi
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equations and plotting shown in figure 2 b (Gezgin, Houimi, Gündoğdu, Mercimek and Kılıç, 2021).

Figure 3(a-b) demonstrates SEM images of produced nanofibers for 6% PAN and Hemp stem with deposition time of 1 minute, 20 cm distance between syringe and substrate a 10 kV applied high voltage. Hemp nanofibers between 125 to 180 nm were produced. Figure 3(c-d) illustrates SEM images

of produced nanofibers for 6% PAN and Hemp stem with deposition time of 5 minutes at distance of 20 cm and voltage of 15 kV. The thicknesses of hemp nanofibers procedure ranging from 142 to 193 nm. The SEM images indicate that as the deposition time increases, thickness and density of nanofibers rise notably.

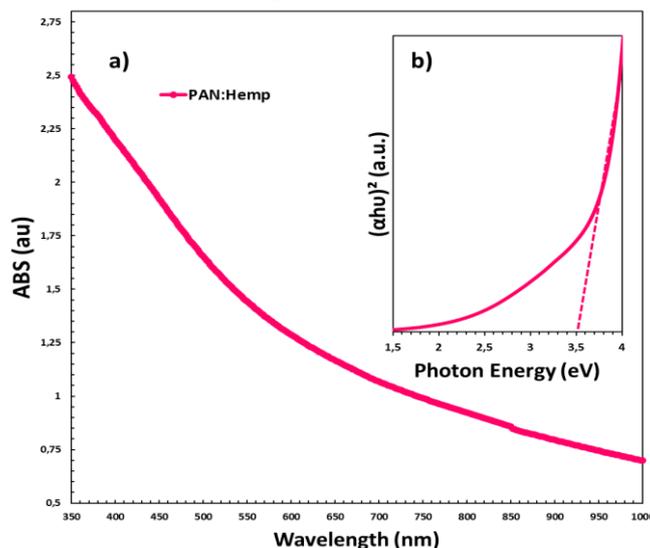


Figure 2. 6 % PAN:Hemp stem with deposition time of 10 minutes and 20 cm distance as well as 20 kV applied voltage a) UV-Vis absorbance spectrum and b) Energy bandgap spectrum of produced nanofiber

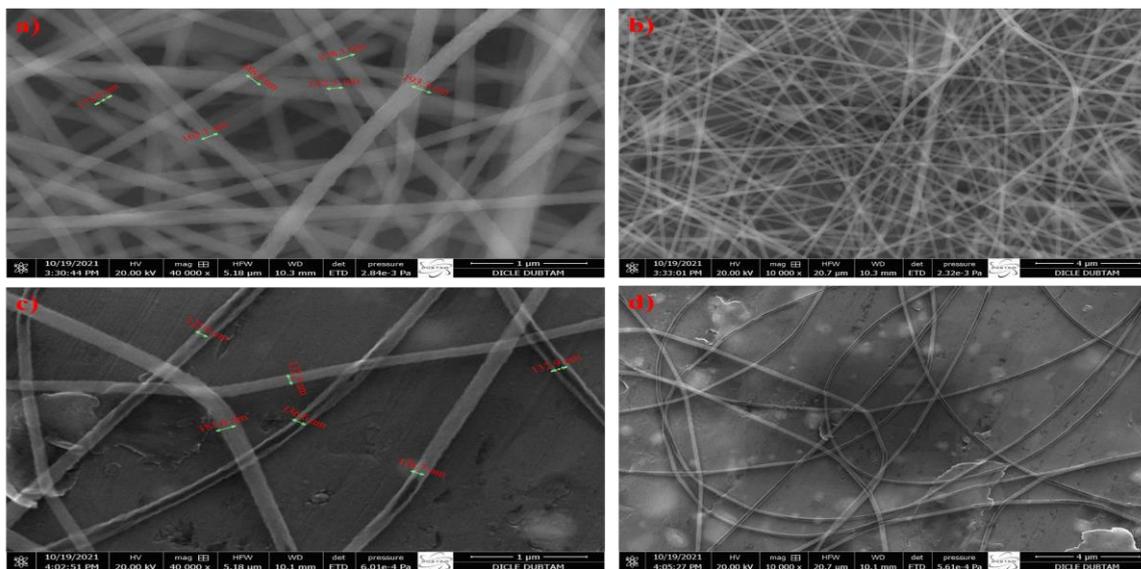


Figure 3. (a-b)SEM image of produced nanofibers for 6% PAN and Hemp stem with deposition time of 1 minutes and 20 cm, 10 kV. (c-d)SEM image of produced nanofibers for 6% PAN and Hemp stem with deposition time of 5 minutes and 20 cm, 15 kV.

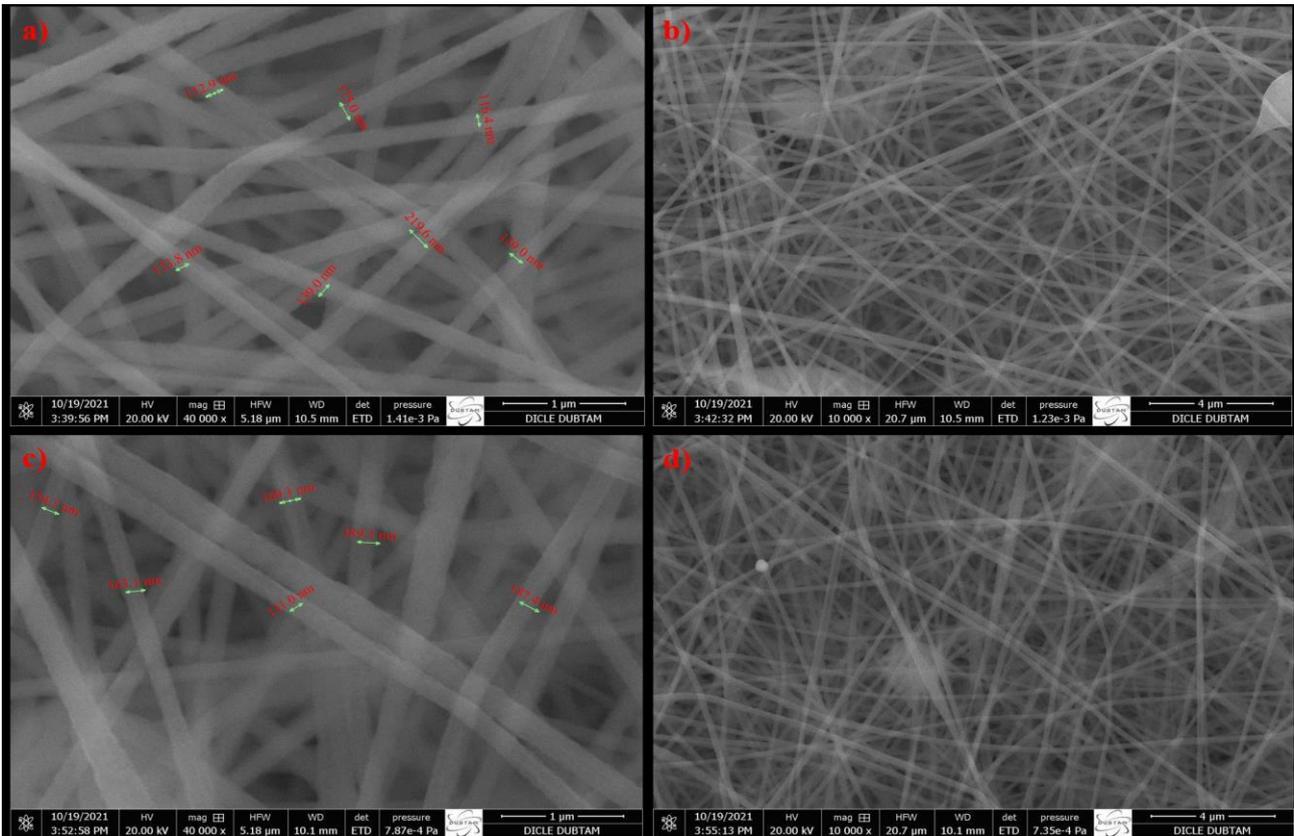


Figure 4. (a-b)SEM image of produced nanofibers for 6% PAN and Hemp stem with deposition time of 10 minutes and 20 cm, 15 kV. (c-d) SEM image of produced nanofibers for 6% PAN and Hemp stem with deposition time of 15 minutes and 20 cm, 15 kV.

Figure 4(a-b) shows SEM images of produced nanofibers for 6% PAN and Hemp stem with deposition time of 10 minutes at distance of 20 cm and voltage 15 kV. The obtained nanofibers are differing at thickness ranging from 116 to 220 nm. The images infer that the density per unit area as well as the average thickness of nanofibers rise as the deposition time upsurges. Figure 4(c-d) demonstrates SEM images of produced nanofibers for 6% PAN and Hemp stem with deposition time of 15 minutes at distance of 20 cm and voltage 15 kV. The obtained nanofibers are differing at thickness ranging from approx. 130 to 250 nm. The correlation between the thickness, density per unit volume and deposition time of nanofibers confirms the previous measurements. Figure 5(a-b) illustrates SEM images

of produced nanofibers for 6 % PAN only with deposition time of 10 minutes and 20 cm, 10 kV. The thickness of the produced PAN nanofibers are ranging between 150 to 210 nm. The density per unit volume agrees with the previous measurements for hemp stem.

Figure 5(c) shows SEM image of produced nanofibers' cross-section for 6% PAN: Hemp stem with deposition time of 1 minute and 20 cm with 20 kV. Since the nanofibers are produced on an aluminum foil which has a thickness of approx. 10 μm , the thickness of the produced hemp nanofibers are around few micrometers for deposition of 1 minute.

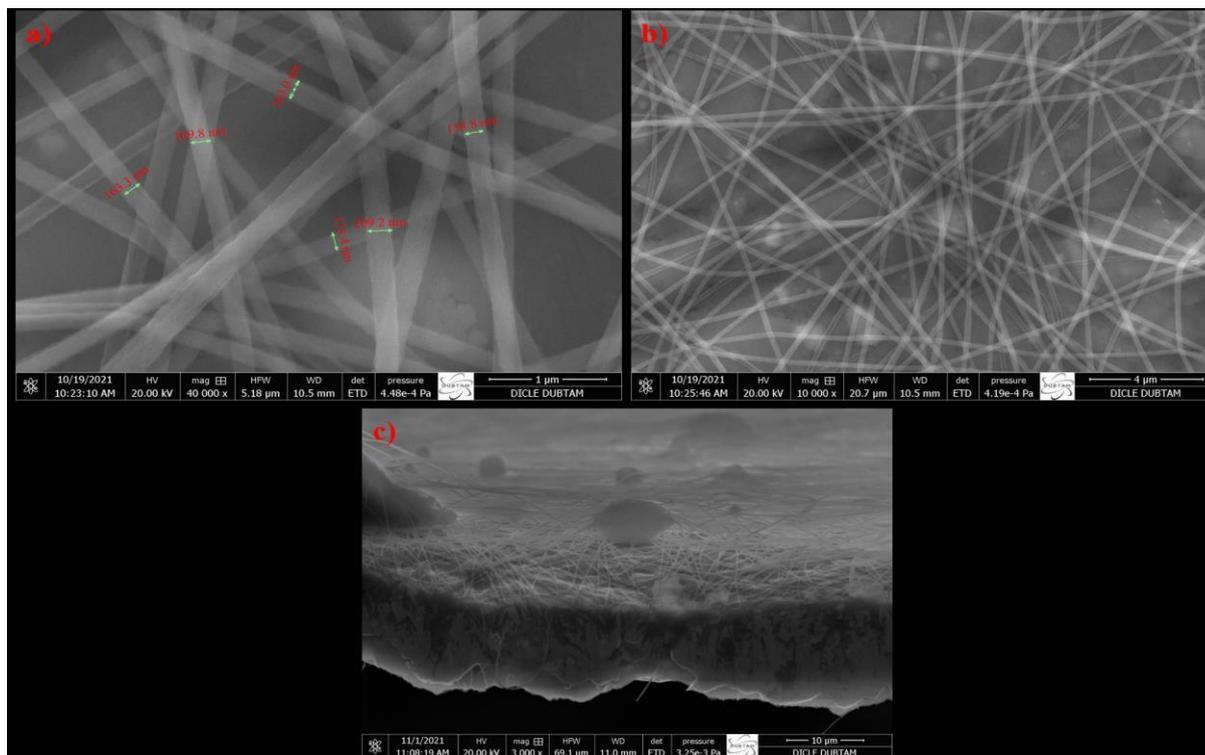


Figure 5. (a-b)SEM image of produced nanofibers for 6 % PAN only with deposition time of 10 minutes and 20 cm, 10 kV. a) 40000 times magnification and b) 10000 times magnification (c) SEM image of produced nanofibers' cross-section for 6% PAN:Hemp stem with deposition time of 1 minute and 20 cm, 20 kV at 3000 times magnification.

Figure 6(a-b) reveal FTIR spectrum of produced nanofibers for 6% PAN and Hemp stem with deposition time of 15 minutes at a distance of 20 cm and voltage of 20 kV. The vibrational modes of produced nanofibers of PAN polymer and hemp stem agrees with previous studies and are given in table 1 (Mohammed et al., 2020).

C-O antisymmetric and asymmetric axial stretching around $1300-1100\text{ cm}^{-1}$ and $1150-1000\text{ cm}^{-1}$ respectively. The stretching vibrations and the bending vibrations of $(\text{CH})_n$ ($n=1,2,3$) appear at $3000-2900\text{ cm}^{-1}$ and $1475-722\text{ cm}^{-1}$ respectively.

Table 1. Absorbance bands interval of PAN:Hemp stems

Absorption bands (cm^{-1})	Functional group	Absorption intensity
3011-2920	C-H Stretching vibration	Strong
2873-2852	CH_2 Asymmetric and symmetric vibration	Strong
1464-1435	CH_2 Scissoring vibration	Middling
1377-1237	CH_2 and CH_3 deformation	Middling
1195-1044	C-O-C symmetric stretching vibration	Middling
1044-991	C-O-C Anti-symmetric stretching vibration	Weak

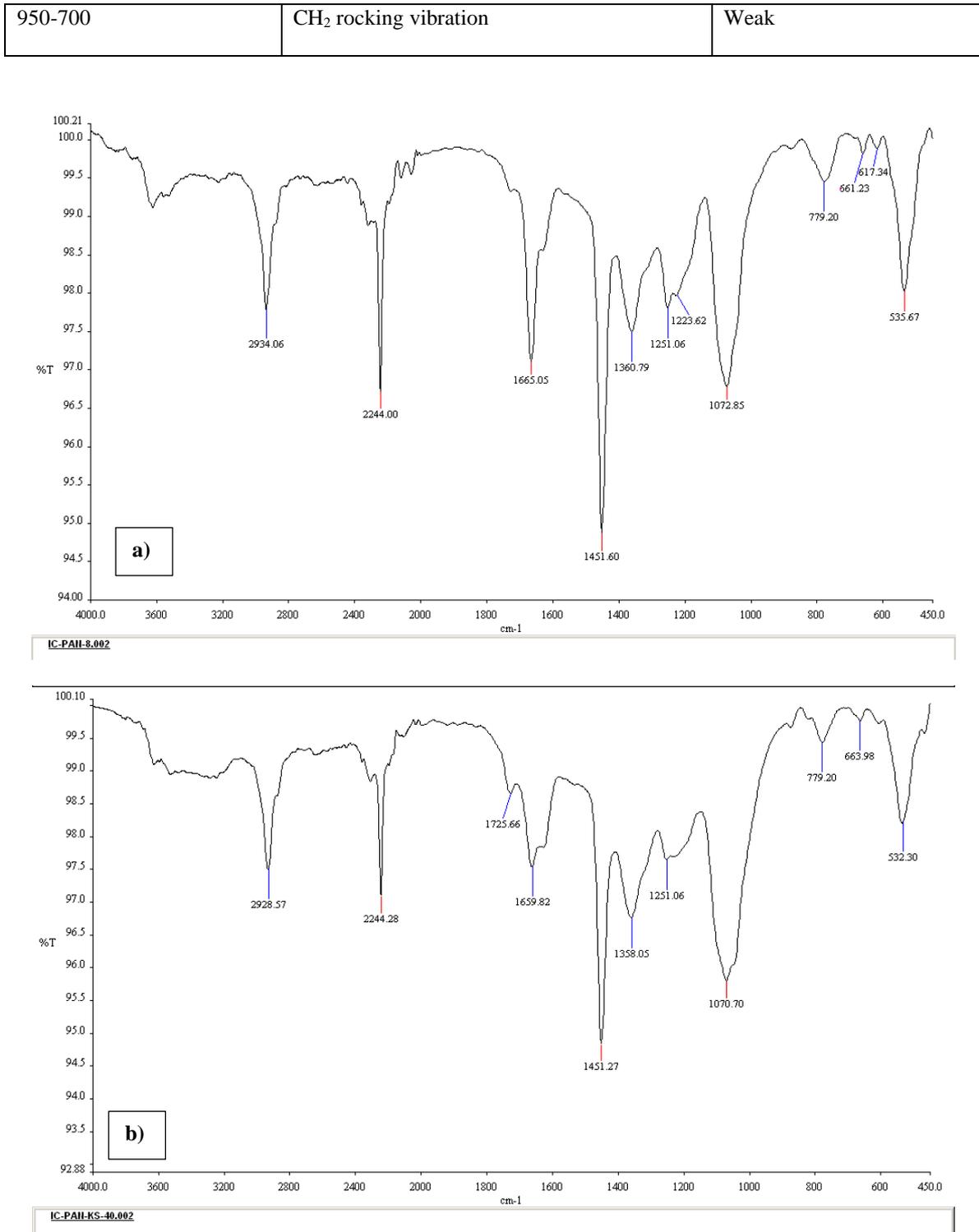


Figure 6. a) FTIR spectrum of produced nanofibers for 6% PAN with deposition time of 15 minute and 20 cm, 20 kV
b) FTIR spectrum of produced nanofibers for 6% PAN and Hemp stem with deposition time of 15 minute and 20 cm, 20 kV.

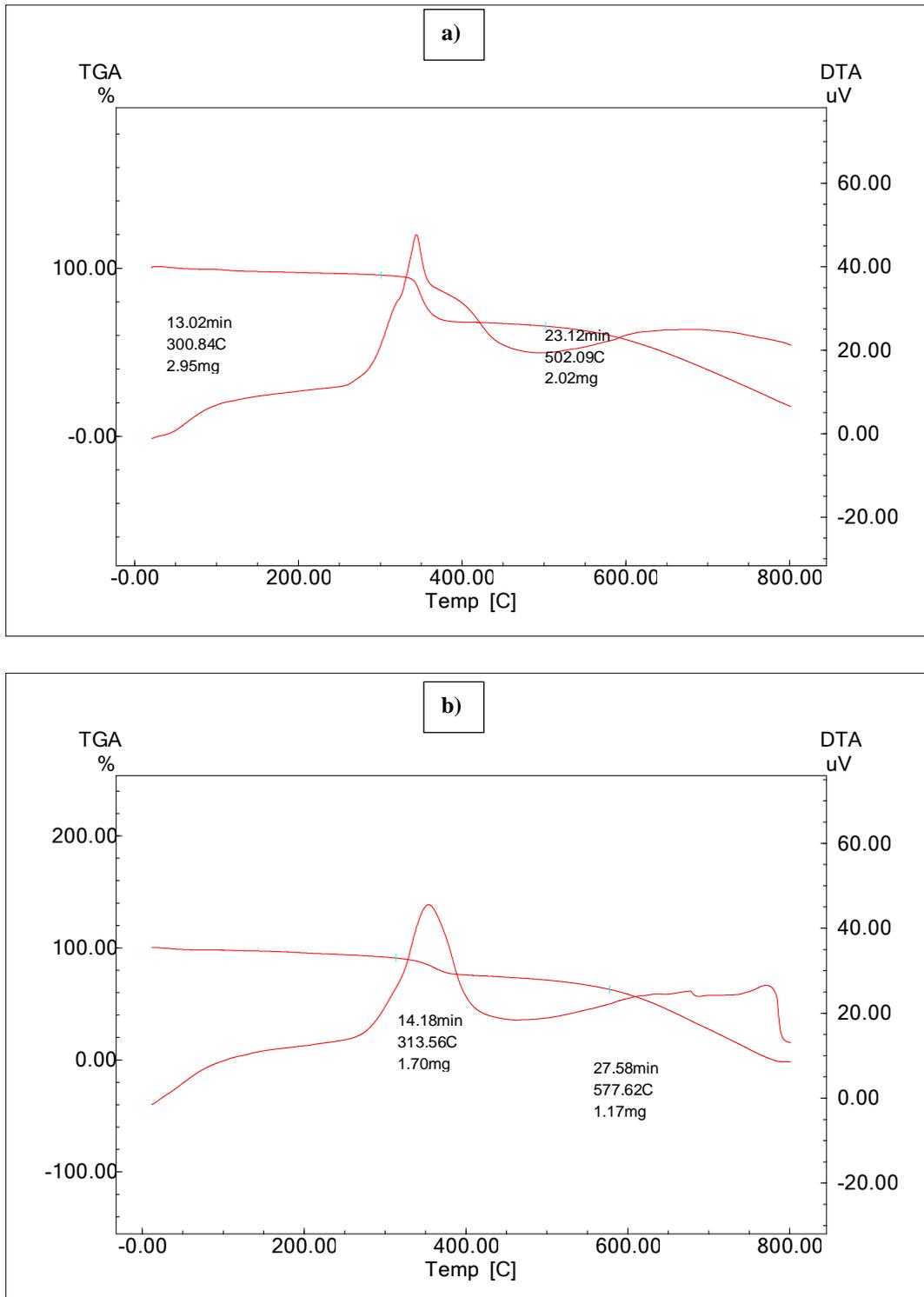


Figure 7. a) TGA plot of 6 % PAN with deposition time of 15 minutes and 20 cm, 20 kV b) TGA plot of 6 % PAN and Hemp stem with deposition time of 15 minutes and 20 cm, 15 kV

Research article/Araştırma makalesi
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Figure 7(a-b) demonstrates TGA plot of 6 % PAN:hemp stem with deposition time of 15 minutes, respectively. Figure 7a shows a 40 % weight loss at 300 °C for PAN nanofibers while figure 7b demonstrates a 20 % weight loss at 314 °C for Hemp stem nanofibers. Both PAN and Hemp stem nanofibers continue losing weight gradually after first sharp decrease and almost completely disintegrate at 800 °C. As a result, it can be said that the hemp stem nanofibers are more enduring compared to PAN polymer nanofibers at high temperatures.

CONCLUSION

The fibers that are reduced from hemp stems to nanosized dimensions were obtained by employing the electrospinning method. Initially, the nanosized particles of Hemp stems were mechanically produced by ball milling technique. SEM images revealed that the thickness of hemp nanofibers increased when deposition times raised. From cross-section measurements, since the nanofibers are produced on an aluminum foil which has a thickness of approx. 10 µm, the thickness of the produced hemp nanofibers are around few micrometers for deposition of 1 minute. The nanofibers diameters of PAN: Hemp Stems are produced ranging from 30 to 250 nm. FTIR spectrum revealed the vibrational modes of produced nanofibers of PAN polymer and hemp stem agrees with previous studies. TGA measurements inferred that both PAN and Hemp stem nanofibers continue losing weight gradually after first sharp decrease at around 300 °C and almost completely disintegrate at 800 °C. As result, it can be said that the hemp stem nanofibers are more enduring compared to PAN polymer nanofibers at high temperatures. The produced PAN: hemp stems nanofibers can be applied to medical usage for future studies.

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CONFLICT OF INTEREST

The Authors report no conflict of interest relevant to this article

RESEARCH AND PUBLICATION ETHICS STATEMENT

The authors declare that this study complies with research and publication ethics.

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