

Synthesis and characterization of the electrospun fibers prepared from waste polymeric materials

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Abstract

Recently, the demands for the production of many different types of plastic wastes are greatly growing that subsequently, lead to the serious challenges in environmental considerations. Since, these materials are rarely resolved by microorganisms, hence, their recycling to the useful materials is crucial. In the present study, we used a solution electrospinning technique to synthesize nano/microfibers. SEM and FTIR analyses were performed for characterization of the prepared samples. The investigations obviously confirmed the formation of nano/microfibers of polyethylene terephthalate (PET), polystyrene (PS) and polycarbonate (PC) from the related waste plastics. To investigate the effect of various operational parameters of electrospinning method, the effect of the used needle diameter and voltage difference was taken into consideration.

Keyword: Characterization; Electrospinning; Nano/ microfibers; Recycling; Waste polymers.

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INTRODUCTION

The generation of large amounts of the waste plastic materials has been converted to a serious problem for many years. Since these materials are rarely removed by microorganisms, therefore, their recycling to the useful materials is necessary. In recent years, the recycling of plastic materials to beneficial requirements has been significantly attracted many attentions. There are two mechanical and chemical well-known routes for the recycling of plastic wastes. In mechanical methods, the plastic wastes are recycled to the usage materials by chipping into very fine scraps, followed by heating, melting and remolding to new products [1].

Chemical recycling of plastic wastes to the initial monomers consists of depolymerization by hydrolysis, methanolysis, glycolysis and aminolysis. This practical technique has been frequently employed for the recovery of waste plastics to the usage cycle. Chemical treatment of

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waste materials is another aspect which is widely used for the production of technically-important carbon materials [2, 3].

Mechanical recycling of polyethylene terephthalate (PET) blended with the other polymers has been also investigated by many researchers [4, 5] which includes the significant improvements for the regeneration of PET wastes. For chemical recycling of PET waste, the microwave irradiation in the presence of ethylene glycol (EG) and some catalysts such as zinc acetate and sodium sulfate has been supposed in the literature [6, 7].

Brennan *et al.* [8] studied the effect of recycling of Polystyrene (PS) on the properties of ABS. Mechanical and thermal properties of the virgin and recycled polymers have showed that the effect of recycling on the tensile strength and tensile modulus of the blend was negligible in comparison to the virgin and pure components. Various investigations have been reported for

the recycling of polycarbonate (PC) by the use of mechanical treatments [9, 10]. The results confirmed that both tensile strength and tensile modulus were slightly affected by the recycling process.

The chemical recycling of PC waste was reported by Hidaka *et al.* [11]. According to them, the main products of the chemical recycling of PC waste were bisphenol A and carbohydrate carbonates. Recently a simple chemical recycling method of PC waste was suggested by Tsintzou *et al.* [12] based on which the chemical recycling of PC waste was carried out with water in microwave reactor in the presence of NaOH under controlled temperature and pressure conditions.

However, these conventional methods are useful to recover the waste plastics into the usage cycle, but, in recent decades, large amounts of researches have been reported the preparation and characterization of electrospun nanofibers by the use of a wide range of polymer wastes [13-16]. Electrospinning of polymers in the form of solutions or melts results in the fabrication of the nanofiber mats with considerable properties. Large specific surface area and highly porous structure makes the nanofibrous mats beneficial in many applications such as protective clothing, filtration, and biomedical systems [17]. To prepare fibers with the expected properties, the synthesis conditions must be controlled on the basis of the intrinsic properties of the solution (viscosity, polymer concentration, molecular weight of polymer, electrical conductivity, elasticity, and surface tension), the processing conditions (applied voltage, distance from needle to collector, volume feed rate, needle diameter), and finally, the ambient conditions [17].

Many researchers have reported the synthesis of various polymer compounds by the use of the electrospinning process such as polyethylene terephthalate (PET) [17, 18], polystyrene (PS) [19-21], polycarbonate (PC) [22, 23] and so on. In most studies on polymeric nanofibers, an experimental pure grade of the initial precursors as granules was applied. Recently, the environmental considerations have encouraged some researchers to synthesize nano/microfibers from the plastic wastes as precursors [13-16, 24].

Shin & Chase [14] prepared nanofibers from recycle waste expanded polystyrene through electrospinning by the use of d-limonene as a natural solvent. They compared the variations of

fibers diameters, where different types of solvent were applied. Strain *et al.* also synthesized PET nanofibers via electrospinning method [15] using waste bottles, but, their main focus was on the application of the resulted PET as smoke filter. They just investigated the effect of PET concentration on the fibers diameters.

Zander *et al.* [16] studied the formation of nanofibers from pure and mixed waste streams using electrospinning. They basically focused on the fibers morphologies based on the viscosity of initial used solution. Macossay *et al.* and Mo *et al.* carried out an experimental work on some parameters such as needle diameter of the syringe and voltage effect for the synthesis of electrospun nanofibers [25, 26].

The current study aims to propose a method for recycling of widely-used polymeric wastes including PET, PS and PC which is respectively recycled from the plastic bottles, styrofoams and compact discs (CDs). In this research, we make an attempt to investigate the effect of some technical factors which have significant role on the fibers morphologies. Our focus is on the needle diameter of the used syringe and the applied voltage during electrospinning process.

EXPERIMENTAL

In the present study, the applied polymers were originated from the plastic bottles (PET), styrofoams (PS) and compact discs (PC) to synthesize the nanofiber mats by the use of the electrospinning technique. All the other chemicals were analytical grade (Merck) and used with no further purifications. Briefly, for the fabrication of PET nanofiber mats, water bottles were cut into granules (2×15 mm) and solved in desired mixtures of trifluoroacetic acid (TFA) and dichloromethane (DCM) at the ambient temperature. PET wastes were easily dissolved in the solvents, until a clear solution was formed which usually took around 10 min. Then, the resulted solution was transferred to a suitable syringe and located in the appropriate place of the electrospinning machine (Nanoazma Co.). Electrospinning processes were conducted at different voltages with a flow rate of 4 mlit/h. The nonwoven fibrous mats were collected by a grounded rotating drum (1000 rpm) adjusted at 12 cm far from the needle tip. To investigate the effect of washing and make the fibers bead free, the synthesized mats were treated by ethyl methyl ketone (EMK), such that the fibers were fully

sprayed by EMK for maximum 1 min to eliminate the beads. Then, the resultant mats were dried at 40 °C for 16 h.

PS and PC electrospun fiber mats were fabricated in the same way as PET, except in the case of the used precursors and solvents. In other words, since both styrofoams and compact discs are easily dissolved in DCM, hence, the use of TFA is not required in these cases.

As mentioned previously, numerous parameters have indicated impacts on the characteristics of the electrospun fiber mats. Here, we studied the effect of applied voltage for the PET-containing solutions, then, we made an attempt to produce PS- and PC-containing mats. Moreover, two different types of the syringe were used; i.e. injection syringes with needle diameters of 0.69 and 0.23 mm.

FTIR spectra were collected by ThermoNicolet Avatar 370 infrared spectrometer at room temperature using KBr pellet technique. Morphology and diameter of the nanofibers were analyzed by scanning electron microscopy (SEM). SEM images were obtained using a scanning electron microscope (KYKY-EM3200), operating at 20 kV.

RESULTS AND DISCUSSION

Fig. 1 demonstrates FTIR spectra of the electrospun samples made of the waste polymers. All spectra are associated with the characteristic peaks of the desired polymers and the residual solvents. For instance, in the case of PET spectrum (Fig. 1a), the peaks appeared at 502.16, 1290, 1369.35 and 1576.05 cm^{-1} are assigned to C–Cl stretching vibrations, CH_2 –Cl bending vibrations, C–H bending vibrations and H–O–H bending vibrations [27], respectively, originated from the presence of DCM residues. Similarly, the peak centered at around 1449.43 cm^{-1} points out to the symmetric and asymmetric stretching vibrations of the carbonyl group in TFA residues [28].

In Fig. 1a, the peak appeared at 726.52 cm^{-1} is attributed to C–C and C–H out of plane bending in aromatic ring [29]. The other peaks observed at 872, 1016.1 and 1241.54 cm^{-1} , respectively, stand for parasubstituted benzen ring, C–H in-plane bending and eventually, O=C–C stretching of ester [29].

The peaks appeared at 1260 and 1406.57 cm^{-1} are accordingly associated with the in-of-plane stretching vibration of C–H bonds in the

benzene ring and the stretching vibration of ester group [30]. The peaks centered at 1623, 1671 and 1717.28 cm^{-1} are associated with stretching vibration of carbonyl groups [27-28]. The presence of the peaks at 1338.58 and 2957.93 cm^{-1} are respectively corresponding to C=O stretching and stretching vibration of CH_2 bonds. The peaks observed at 972 and 1096.08 cm^{-1} simultaneously assign to the vibration of C–O group [30].

Fig. 1b shows the FTIR spectrum of PS electrospun fibers. As mentioned above, the peaks centroid at 537.83, 1290, 1368.8 and 1580.93 cm^{-1} are indicative of the presence of the residual solvent within the fabricated PS mats [29]. The peaks at 696.8 and 1597.42 cm^{-1} are respectively arisen from out of phase ring deformation of monosubstituted aromatic rings [31] and aromatic C=C stretching vibration mode [31]. The peak appeared at 1260 cm^{-1} is due to in-of-plane stretching vibration of C–H bonds on the benzene ring and the stretching vibration of ester group [30].

The peaks centered at 903.98 and 1066.37 cm^{-1} are assigned to C–H out-of-plane bending and C–H in-plane bending, respectively [21]. The observed peaks at 749.1 and 1025.32 cm^{-1} are attributed to the existence of the phenyl group, which respectively confirm the presence of phenyl group [32] and C-phenyl bonds [21]. The observed peaks at 1447.99 and 1489.37 cm^{-1} are ascribed to the stretching vibration of the C–C bonds in the benzene ring of PS [33]. The appearance of the bands at 1715.78 and 1936.72 cm^{-1} refers to the aromatic overtones [34]. Also, the bands at 2845.68 and 2914 cm^{-1} are as the proofs of asymmetric and symmetric stretching vibrations of CH_2 [33]. As expected, the peaks appeared at 3021.98, 3056.78 and 3080.59 cm^{-1} note to the aromatic C–H stretching vibration [31].

Fig. 1c exhibits the FTIR transmittance peaks related to the PC sample. Similar to the aforementioned FTIR results of PET and PS, the peaks appeared at 551.48, 1290.81, 1361.92 and 1585.02 cm^{-1} are respectively attributed to C–Cl stretching vibrations, CH_2 –Cl bending vibrations, C–H bending vibrations and the presence of molecularly adsorbed water (H–O–H bending vibrations) [29]. The other peaks in Fig. 1c are indexed to the presence of the bonds related to PC. According to the literature [31], the peaks centered at about 760.2, 1012.14 and 1157.24 cm^{-1} are pointed out to mono-substituted

ring, symmetric stretching of carbonate group (O-C-O) deformation and asymmetric stretching of carbonate group (O-C-O), respectively. The observed peaks at 825.93 and 882.71 cm^{-1} note to C-H out-of-plane bending [31]. The presence of the peaks centered at 1185.77 and 1217.69 cm^{-1} are indicative of the asymmetric stretching of carbonate group (O-C-O). The presented peak at 1260 cm^{-1} is attributed to in-of-plane stretching

vibration of C-H bonds on the benzene ring and the stretching vibration of ester group [30]. Furthermore, the peaks observed at 1077.8, 1405.31, 1498.32 and 1763.31 cm^{-1} , respectively, stand for the vibration of $-\text{CH}_3$ group, distortion of $-\text{CH}_3$ group, stretching vibration of C=C and C=O stretching. Eventually, one peak centroid at 2966.17 cm^{-1} assigns to C-H asymmetrical and symmetrical flexing vibration of alkyl ($-\text{CH}_2$) [35].

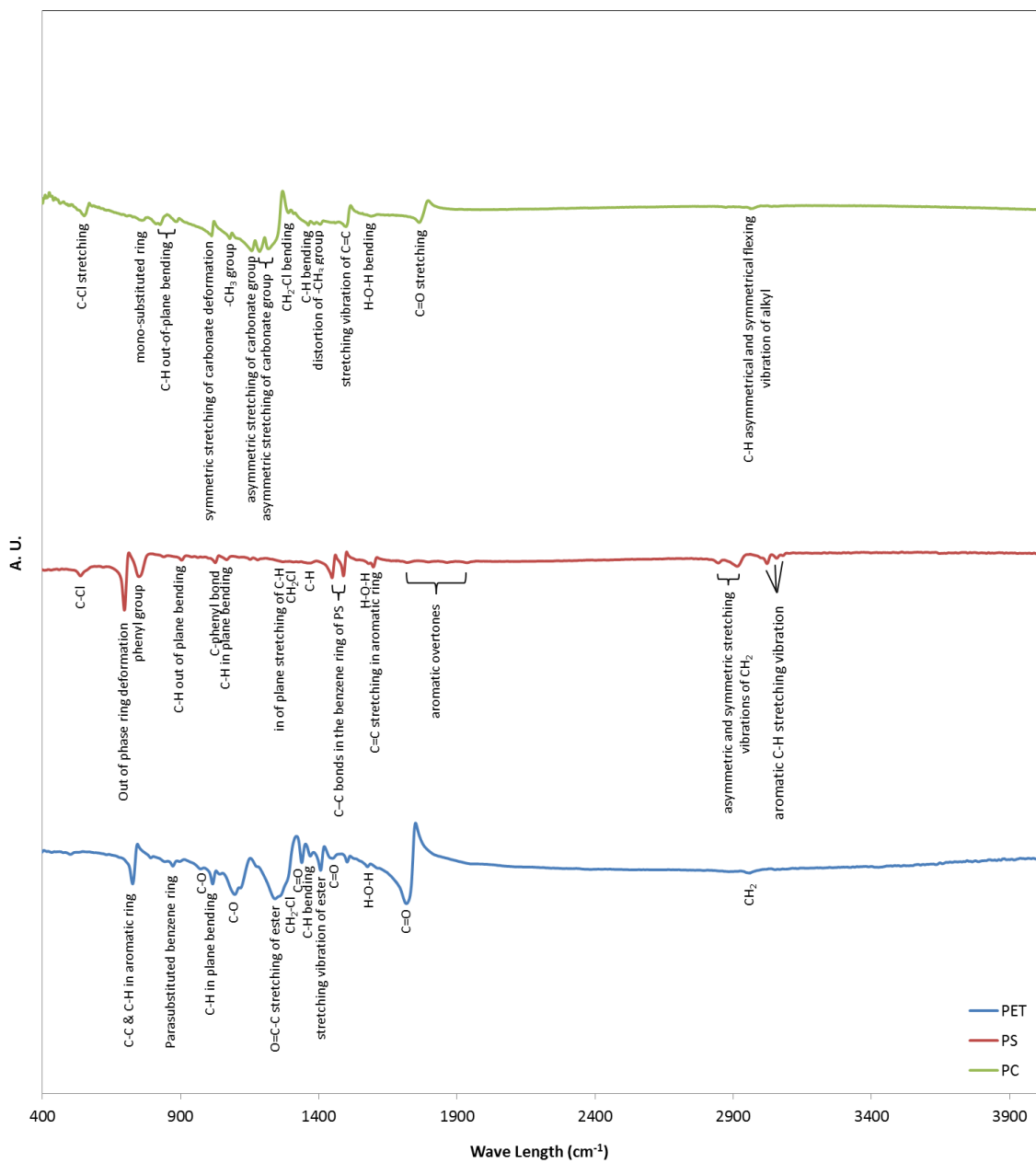


Fig. 1: FTIR spectra of electrospun nanofibers of PET, PS and PC made of the waste materials.

Fig. 2 presents SEM images and the corresponding fiber diameter histograms of the as-prepared (without any subsequent treatments) PET electrospun mats, such that for the synthesis of these fibers various voltages have been applied. The used syringe has a needle diameter of 0.69 mm. The histograms in Fig. 2 demonstrate that the sample prepared at 20 KV has a narrower distribution of fibers diameters in comparison with

the ones synthesized at 15 and 25 KV, however, the fibers diameters exhibit a decreasing propensity with the increase of the applied voltage. In the other word, the increase of the electrospinning voltage causes increase of the electrostatic stress on the jet [26, 36]. According to Fig. 2, one may observe the presence of the beads within the nanofibers which is in good accordance with literature [16, 17].

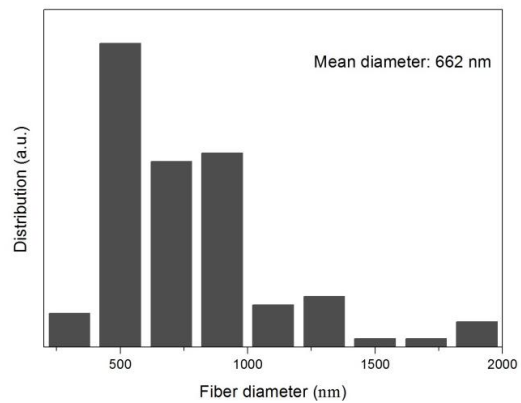
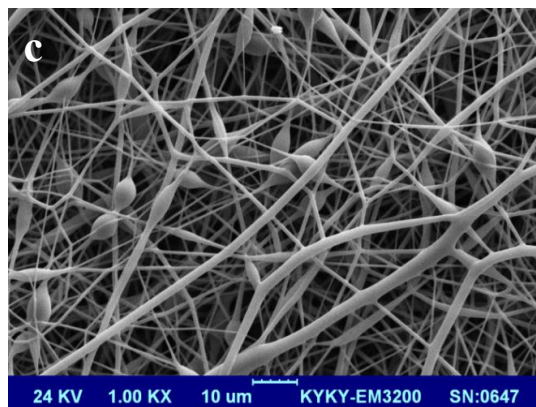
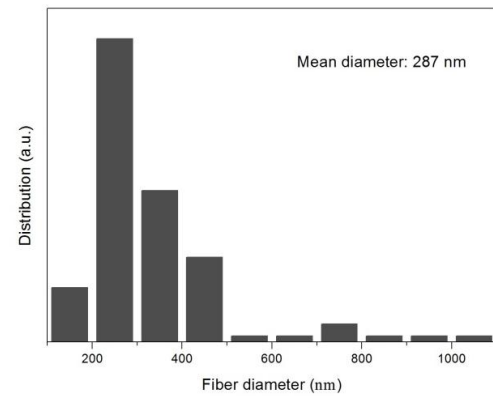
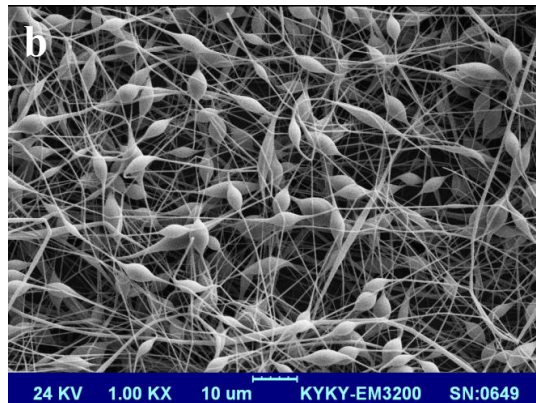
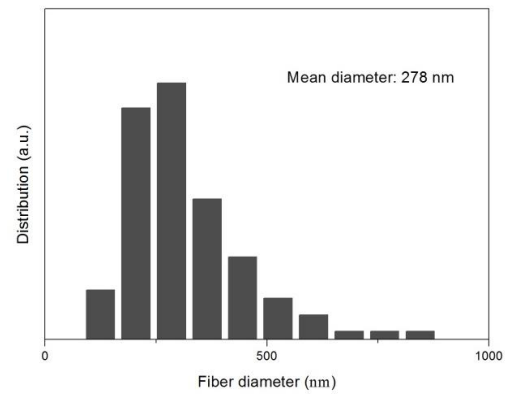
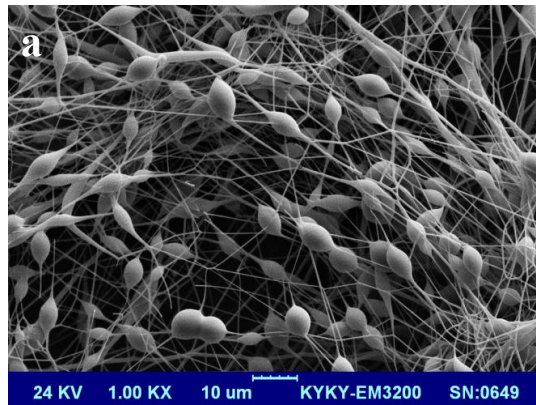


Fig. 2: SEM images and the associated histograms of pristine PET electrospun mats synthesized at a) 15 KV, b) 20 KV and c) 25 KV without any treatments.

According to Veleirinho *et al.* [17], the increase of PET concentration leads to an enhancement in fiber uniformity and an obvious decrease of beaded fibers which has been frequently confirmed by the other authors. Also, they mentioned that DCM has a high surface tension which causes the formation of beads during electrospinning. Therefore, the formation mechanism of the beaded fibers is attributed to the high surface tension of the primary solvent (i.e. DCM).

As shown in Fig. 2, it seems that the number of beads is dependent on the applied voltages. Based on our observations, the number of beaded nanofibers decreases more intensified at the voltage difference of 25 KV than fewer ones. To the best of our knowledge, various parameters such as surface tension, viscosity of the solution and net charge density of the jet are introduced as the main effective factors on the formation of the beaded nanofibers [16, 17]. Therefore, it appears that the increase of the net charge density of the jet improves its capillary instability and then, decreases the formation of beads on the fibers. That is why the increase of the applied voltage results in a considerable decrease of bead formation within the polymeric fibers.

In most cases, adding a salt to the jet components is preferred to reduce the formation of beads. Zong *et al.* [37] found that the addition of 1wt% of a salt makes the final fibers bead free. They justified that the addition of salts induces more electric charges to the polymer jet during the electrospinning. Zander and co-workers [16] used an ammonium salt (Tributylammoniumchloride (TBAC)) to prevent the formation of beaded fibers through a decrease of viscosity and an improvement of charge density on the surface of the jet. As known, the increasing of the initial concentration of polymeric solution is along with the increase of fibers diameters, then, washing the fibers with an appropriate solvent is a more preferable method. Herein, to prevent the existence of impurities in the fibers, we used a washing agent after the formation of fibers via electrospinning.

Therefore, to fully remove the beads, the prepared fibers were sprayed with EMK followed by drying in oven to exit residuals. The reason for the use of EMK is attributed to its low vapor pressure, such that it is quickly evaporated from the fibers surface; i.e. about 1 min. Also, since the beads have small sizes, the penetration of EMK

into the structure of the beads is very fast and makes the fibers bead free.

The SEM photographs of the washed samples together with their fiber diameter histograms are exhibited in Fig. 3. Compared to the corresponding nanofibers in Fig. 2, no beads are observed within the fibers after the washing process. Moreover, the fibers diameters decrease, when the applied voltage increases. For instance, the mean fibers diameters of around 384.9 nm at 15 KV decreases to the values of 179.5 and 175.9 nm, at 20 and 25 KV, respectively. According to Fig. 3, one may find a narrower fiber diameter distribution for the sample prepared at 20 KV than those synthesized at 15 and 25 KV. These findings are accordingly in good consistent with the results of the other researchers [14, 16]. They observed a significant reduction in the fibers diameters in addition to the elimination of beads.

Fig. 4 exhibits the PET fibers washed with ethyl methyl ketone, when a thinner syringe with a needle diameter of 0.23 mm was used. The images in Fig. 4 demonstrate a similar trend with the corresponding ones in Fig. 3, such that the mean fibers diameters increase at the increased voltages. However, the interesting result is that the mean fibers diameters are decreased, where a syringe with a smaller needle is used. Also, a narrower fiber diameter distribution for the samples prepared with the thinner syringe is found. Macossay *et al.* [25] reported that the needle diameter has no clear effect on the fibers diameters, but, this result is contrary to the results of the other researchers, as received from reports which agrees well with our findings [26, 38].

For better comparison, a complete analysis of PET samples is shown in Fig. 5. As seen, for both the unwashed (15 KV, 20 KV and 25 KV) and washed (15 KV-W, 20 KV-W and 25 KV-W) samples, the mean fibers diameters increase with the enhancement of the applied voltage values, but, based on the results, it is obvious that diameter distribution of the fibers is narrower for the sample formed at 20 KV. As mentioned, the use of different syringes for synthesis of the fibers results in the formation of the fibers with various diameter distributions. As observed in Fig. 5, the mean diameters and distribution of the fibers synthesized by the use of the syringe with needle diameter of 0.23 mm (15 KV-W-Ins, 20 KV-W-Ins and 25 KV-W-Ins) drastically decrease, when compared to the fibers prepared by the syringe with needle diameter of 0.69 mm.

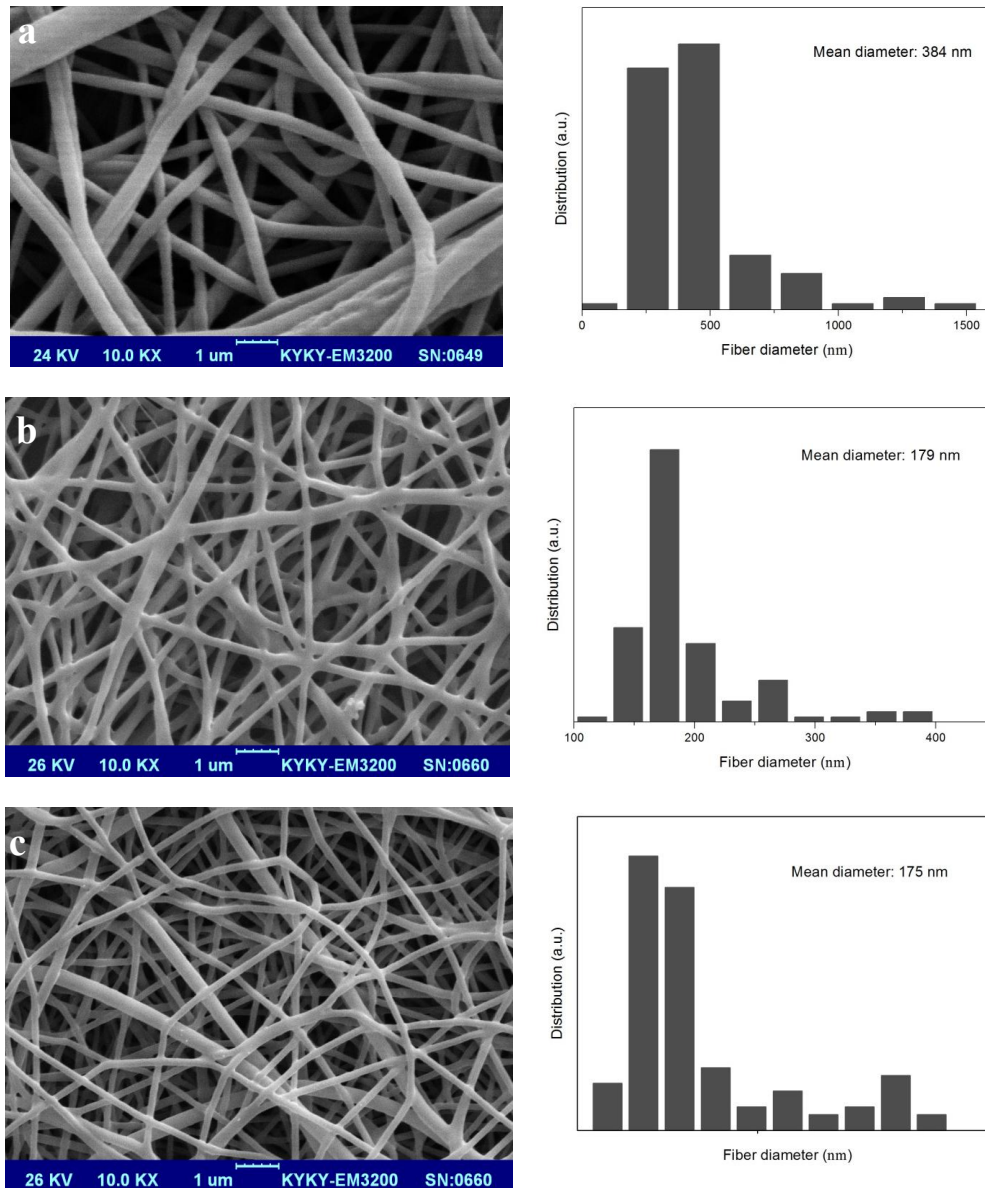


Fig. 3: SEM images and the associated histograms of PET electrospun mats synthesized at a) 15 KV, b) 20 KV and c) 25 KV after washing with EMK.

To examine our technique for the production of different polymeric fibers and extend this method, we synthesized and characterized PS and PC samples resulted from Styrofoam and CDs, respectively. Fig. 6 illustrates the SEM photographs of PS and PC fibers and their related histograms. Based on Fig. 6, it is clear that the PS sample is composed of the smaller fibers than those of synthesized PC, such that the fibers diameters of PC are found to be in the range of 49-462 nm with mean fiber diameter of around 213 nm, while,

the PS fibers show a fiber diameter distribution between 32-663 nm with mean fiber diameter of about 164 nm. Although, the mean fiber diameter of PC sample is larger than that of PS fibers, but one may find a broader distribution of diameters for the PS fibers than the PC ones.

Fig. 7 summarizes the essential data on the fibers dimeters distribution of PET, PS and PC matrices, when the same syringe (needle diameter of 0.23 mm) and voltage difference (20 KV) are used.

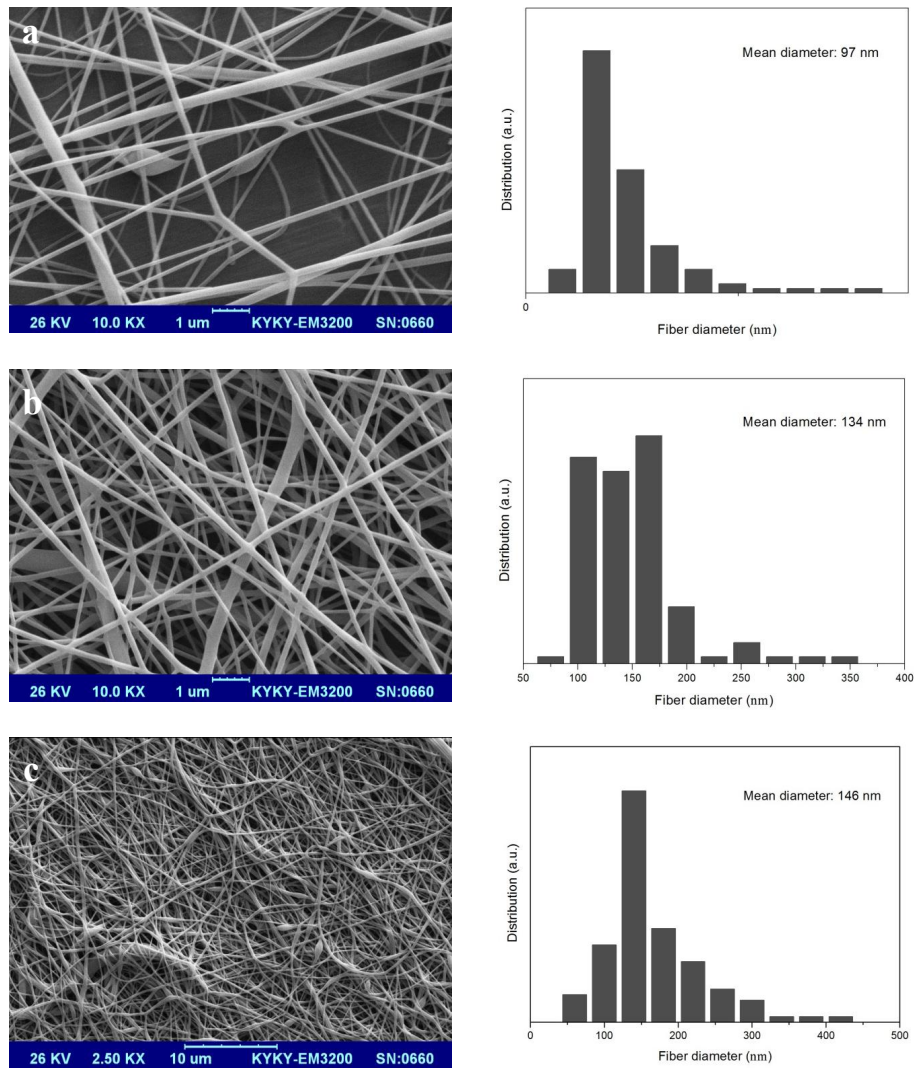


Fig. 4: SEM images and the associated histograms of PET electrospun mats synthesized by a syringe with needle diameter of 0.23 at a) 15 KV, b) 20 KV and c) 25 KV and the washed with EMK.

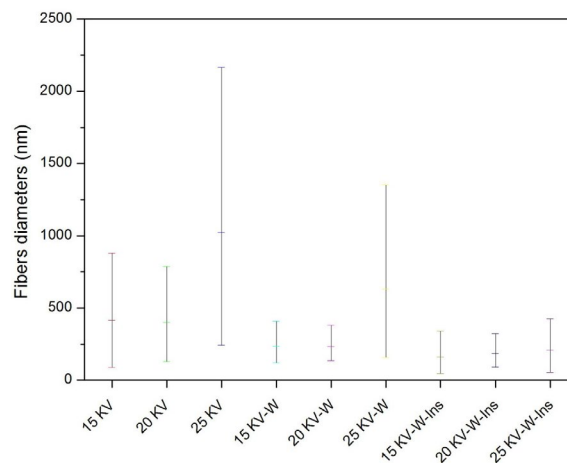


Fig. 5: The effect of type of various syringes on the fibers diameters distribution range for the unwashed and washed PET at different voltages.

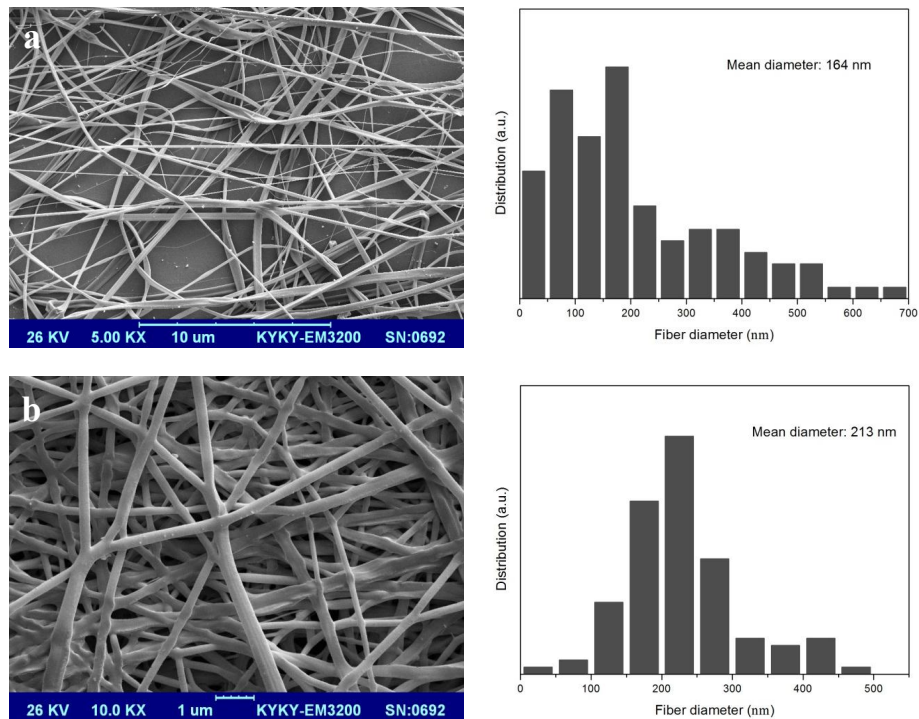


Fig. 6: SEM images of PS (a) and PC (b) electrospun mats synthesized at 20 KV after washing with ethyl methyl ketone.

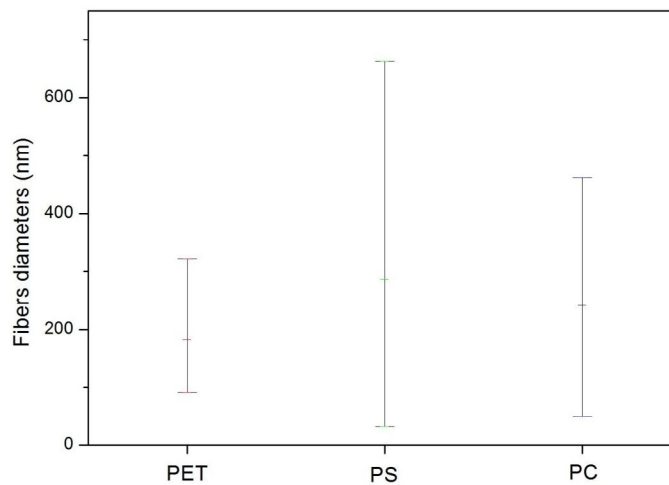


Fig. 7: The comparison of mean fibers diameters and the associated distribution curves of PET, PC and PS electrospun mats synthesized at 20 KV by the use of a syringe with needle diameter of 0.23 mm.

The results reveal that the broadest diameter distribution and also, the highest mean fibers diameters are attributed to the PS sample. Meanwhile, among the samples, the lowest value of the fibers diameters is associated with the PET with the narrowest fiber diameter distribution.

Zander and co-workers [16] also compared the structural morphology and the fibers diameters of

PET, PS and PC. On the basis of their results, PS sample possesses the narrower fiber diameter distribution compared to PET and PC which is in contrast to our observations. This may be attributed to the use of an ammonium salt as anti-bead component which makes some changes in the apparent characteristics of the final polymers.

CONCLUSIONS

The mat-like fibers of PET, PS and PC were formed from the waste plastic materials by the electrospinning technique. FTIR analysis showed the formation of the expected fibers with minimum impurities. A comprehensive study was performed on the SEM photographs of the synthesized fibers to investigate the effect of the amount of applied voltage and the use of different syringes on the mean fibers diameters. According to the results, the increase of the applied voltage led to the increase of the mean fibers diameters; however, the fibers diameters showed the narrowest distribution, when the fibers were prepared at the voltage difference of 20 KV. In addition, the decrease of the needle diameter of the used syringe was along with the formation of PET fibers with lower mean fibers diameters and narrower distribution of the fibers diameters. Recycling of the other waste plastics such as PS and PC also showed different distribution of fibers diameters, such that the broadest fibers diameters distribution was observed for the PS sample.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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