

Parastoo Amiri,
S. Hajir Bahrami

Textile Engineering Department,
Amirkabir University of Technology,
Tehran 15914 – Iran
E-mail: hajirb@aut.ac.ir

Electrospinning of Poly(acrylonitrile-acrylic acid)/ β Cyclodextrin Nanofibers and Study of their Molecular Filtration Characteristics

Abstract

Acrylonitrile acrylic acid copolymer was synthesised using suspension polymerisation and β -cyclodextrin was mixed with this copolymer in different ratios. Then poly(acrylonitrile-acrylic acid)-cyclodextrin (PANAA/-CD) nanowebs were produced using the electrospinning process. The effect of different electrospinning parameters such as polymer concentration, distance, flow rate and voltage on nanofibre properties was studied. The presence of CD molecules in the PANAA solutions did not affect the diameter of nanofibres significantly. The presence of CD molecules in the PANAA/CD nanofibres produced was studied by means of Fourier transform infrared (FTIR) spectroscopy. The molecular filtration capability of the nanowebs produced was studied using phenolphthalein as a model organic molecule, by means of a UV-Vis Spectrophotometer. Results show that the nanofibres produced are able to remove phenolphthalein effectively from the solution. These nanowebs have the potential to be used as a molecular filter and/or nanofilter for filtration/purification/separation purposes.

Key words: electrospinning, cyclodextrin, poly(acrylonitrile-acrylic acid), molecular filtration.

Introduction

Nanofibres have gained much attention due to their high surface area and pronounced micro and nano structural characteristics which enable a myriad of advanced applications. Their enhanced properties make them applicable in many different areas like nanocatalysis, filtration and absorbent material, tissue scaffolds, drug delivery systems, wound healings, protective textiles, storage cells, etc. [1 - 5]. Among the different ways of nanofibre production, electrospinning is commonly used since it is a simple, versatile and cost effective technique. It can be used for almost all soluble or fusible polymers in order to produce fibres with very thin diameters. Electrospinning is applied to many natural, synthetic and bio - polymers, and has also been used to produce metal, ceramic and glass nanofibres, exploiting precursor routes. Moreover functional nanofibres have been produced by incorporating functional elements [6], one of which is cyclodextrin.

Cyclodextrins are cyclic oligosaccharides made of α -(1,4) linked glucopyranoside subunits. Depending on the number of D-glucose units, they are named α (6 units), β (7 units) and γ (8 units) cyclodextrins [7]. Cyclodextrins have a torus shape with a lipophilic central cavity and hydrophilic outer surface. These molecules are famous for their ability to form noncovalent host-guest inclusion complexes (CD-ICs) with organic or inorganic molecules. These inclusion complexes can modify physical or chemical

properties of the guest molecules. This unique property of cyclodextrin makes them useful in various applications such as pharmaceuticals, foods and flavours, cosmetics, personal care and toiletry, agricultural and chemicals industries, textiles, etc. [7]. CDs have been incorporated in different polymers in order to increase their acceptance and retention of additives, such as dyes, fragrances, antibacterial agents, etc. [8]. They have also been used in different polymeric adsorbents and membranes for separation usage [9].

Table 1. Nanofibre code and composition electrospun at feed rate: $0.5 \text{ cm}^3/\text{h}$, applied voltage: 10 kV and tip to collector distance: 20 cm ; *Because of high viscosity of PANAA10/CD50 and PANAA10/CD100, electrospinning of these solutions was not possible.

Sample code	Polymer concentration, g/dl (% w/v)	β -CD % (w/w)
PANAA5	5	0
PANAA6	6	0
PANAA7	7	0
PANAA10	10	0
PANAA5/CD10	5	10
PANAA5/CD25	5	25
PANAA5/CD50	5	50
PANAA5/CD100	5	100
PANAA6/CD10	6	10
PANAA6/CD25	6	25
PANAA6/CD50	6	50
PANAA6/CD100	6	100
PANAA7/CD10	7	10
PANAA7/CD25	7	25
PANAA7/CD50	7	50
PANAA7/CD100	7	100
PANAA10/CD25	10	25

Some researches tried to combine the high surface area of nanofibres with the ability of CDs to form inclusion complexes by incorporating CDs into electrospun nanofibres in order to enhance the properties of these nanowebs [10 - 21]. Non-soluble polyelectrolyte nanowebs were produced by crosslinking poly(acrylic acid) with β -cyclodextrins [10]. Poly(N-vinylpyrrolidone)/ β -cyclodextrin composite nanofibres were produced and the effect of adding β -cyclodextrin molecules on fibrous electrospun PVP was studied [11]. In another study β -cyclodextrin was used as a stabilising and reducing agent for producing gold nanoparticles in PVP nanofibres [12].

In this paper PANAA copolymer was synthesised and β -CD added to this polymer solution, from which nanofibres were produced. Due to the inherent properties of CD and nanofibre's high surface area and its application in filtration, nanofibres were produced from PANAA solutions functionalised with β -CD using different concentrations of PANAA and β -CD. The concentration of PANAA varied from 5 to 10% w/v in N,N-dimethylformamide (DMF) and from 10% to 100% w/w in β -CD. The effect of different parameters of electrospinning on the morphology of nanofibres was studied. The morphology and characteristics of the nanowebs produced were investigated by scanning electron microscopy, Fourier transform infrared and X-ray diffraction. The molecular filtration capability of the nanowebs produced was studied using phenolphthalein as a model organic molecule, by means of a UV-Vis Spectrophotometer.

Materials and methods

Materials and copolymer synthesis

Acrylonitrile (99%) was purchased from Fluka, acrylic acid (99%), N-dimethyl formamide (99.8%), absolute ethanol, buffer (pH11) and phenolphthalein from Merck (Germany), and β -cyclodextrin from Sigma (USA). They were all used without any prior purification. Copolymer of acrylonitril-co-acrylic acid was synthesised by the suspension polymerisation process with a molar ratio of 90 to 10 (AN-AA), based on a previous report [22]. The intrinsic viscosity of the resulting poly(acrylonitrile-co-acrylic acid) measured was 2.33 dl g^{-1} .

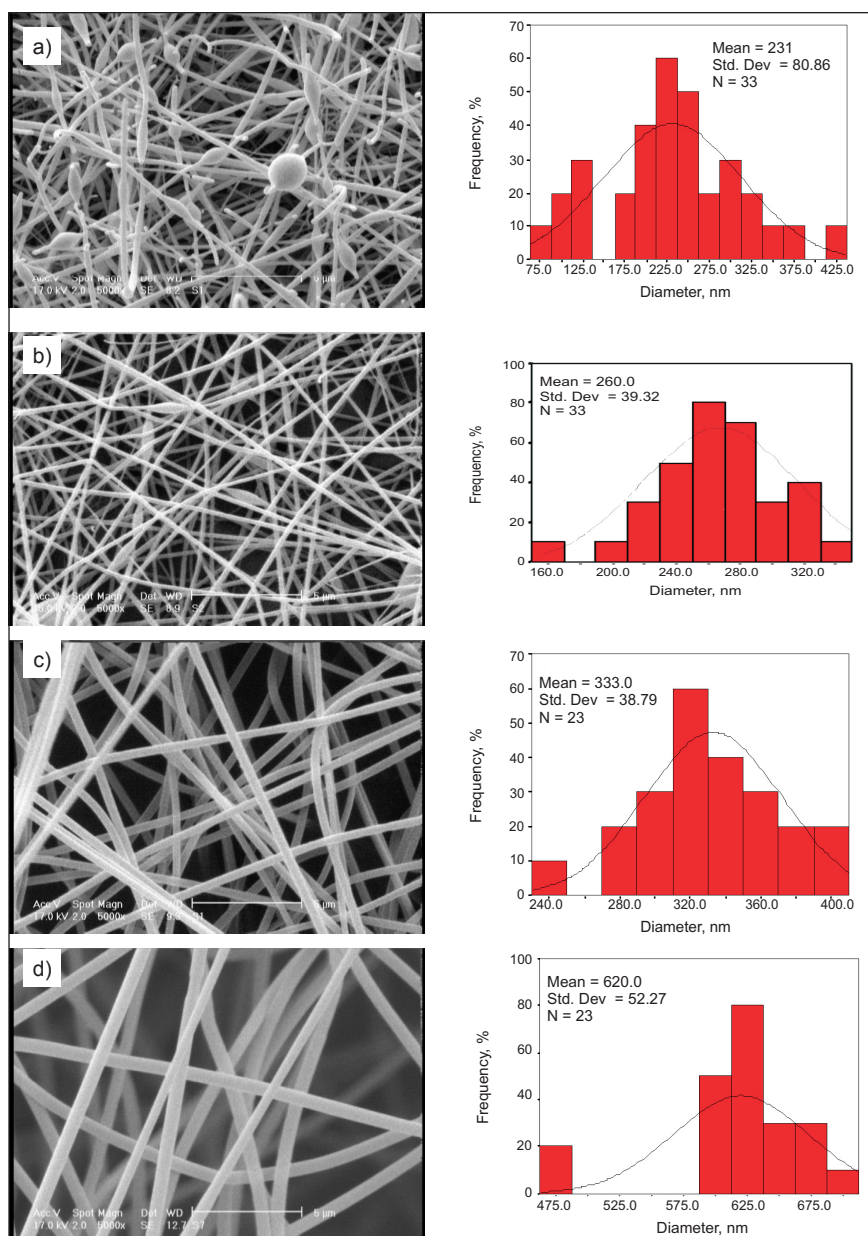


Figure 1. SEM images and fibre diameter distributions of electrospun (a) PANAA5, (b) PANAA6, (c) PANAA7 and (d) PANAA10 nanofibres.

Preparation of solutions and electrospinning

PANAA/ β -CD solutions with different concentrations were prepared by dissolving PANAA and β -CD in DMF at room temperature. PANAA concentration varied from 5 to 10% w/v with respect to DMF, and for the β -CD content it was from 10 to 100% w/w (**Table 1**). Solutions were stirred until they were homogeneous and clear. After air bubbles were removed completely, the solution was placed in a 10 ml plastic syringe with a flat metal needle (gauge 22) on its head. The needle was connected to a power supply and an aluminum foil collector was connected to a grounded counter electrode. Electrospinning was carried

out in a horizontal position at room temperature. The effect of different apparatus parameters on the fibre morphology, like the feed rate, needle tip to collector distance and voltage applied, was studied. It was found that by applying the feed rate 0.5 cm³/h, applied voltage 10 kV and tip to collector distance 20 cm, the most stable webs were obtained from all polymer solutions. Therefore these conditions were employed in order to get nanowebs from different solutions which can be compared. The effect of parameters like the feed rate, needle tip to collector distance and applied voltage on the fibre morphology was investigated for the same solutions.

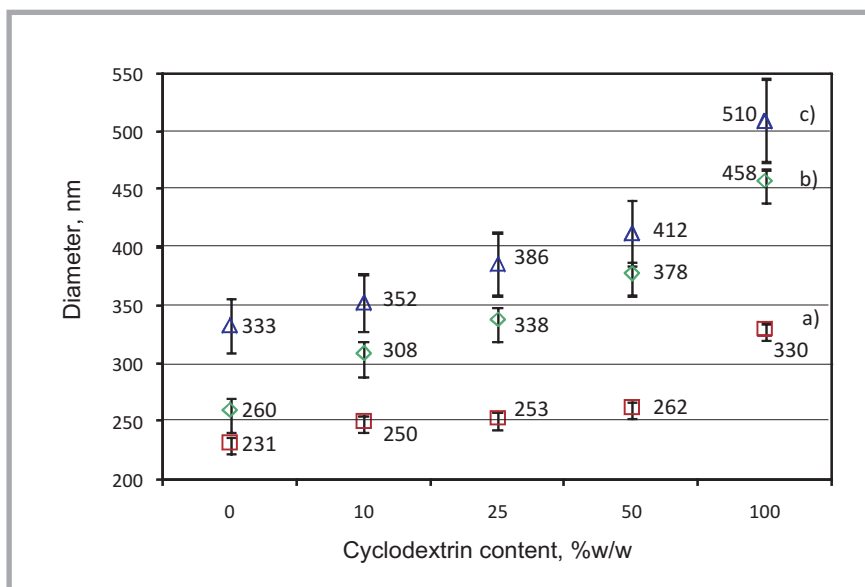


Figure 2. Change in fibre diameter vs cyclodextrin content for acrylic copolymer solution with 5% (a), 6% (b) and 7% (c) concentration.

Research and methods

After coating the surfaces of nanofibres with gold by means of a sputter coater-SCDOOS (BAL-TEC Co.), the morphologies of nanofibres were inspected by means of a scanning electron microscope (Philips-XL30). The presence of CD molecules in nanofibres was studied by means of Fourier transform infrared (FTIR). FTIR spectroscopy was performed using a Nicolet Nexus 670 FTIR spectrometer. The samples were prepared by blending with potassium bromide

(KBr), and pellets were made under high pressure. The spectra were recorded in a spectral range of 500 – 4000 cm^{-1} . XRD X-ray diffraction patterns were measured using Philips Analytical X-Ray with Cu $K\alpha$ radiation in a 2θ range of $5^\circ - 35^\circ$. (XRD) was used to investigate the formation of inclusion complexes.

The potential of electrospun nanocomposite fibres to remove organic molecules was studied using the phenolphthalein (PHP) test method. PHP is often used as a model molecule for studying

the inclusion complex formation of β -cyclodextrin. The phenolphthalein test is based on changes in the absorbance of PHP in the solution. The absorbance of PHP molecules was measured using a UV-vis spectrophotometer in a wavelength range of 400 – 700 nm. PHP test solution was prepared by adding buffer (pH 11) drop wise to 2×10^{-4} PHP solution in absolute ethanol to adjust the pH above 11. 16 mg of each nanoweb were floated in 5 cm^3 of this solution separately in screw cap vials. The pH of the solutions was measured before and after UV-Vis experiments by means of a pH meter. The pH remained unchanged during the experiments.

Discussions and results

Concentration of PANAA and β -cyclodextrin

The concentration of copolymer in the solutions varied from 5 to 10% (w/v) with respect to DMF. **Figure 1** shows SEM images and fibre diameter distributions of PANAA fibres electrospun from solutions containing different amounts of PANAA. Polymer solutions with both 5 and 6 wt% polymer concentration produced beaded nanofibres. At low polymer concentration, the capillary breakup of the jet during electrospinning by surface tension leads to beaded nanofibre formation. It is also observed that a mixture of short length fibres and droplets is produced for 5 wt% polymer solution. However, uniform and continuous bead-free fibres with a smooth surface were produced when the polymer concentration increased to 7 wt% & 10 wt%. Moreover the average fibre diameter increased from 231 to 620 nm by increasing the PANAA concentration from 5 to 10% (w/v). Large variations are seen in both the SEM image and histogram (**Figure 1**) for PANAA5 and PANAA6. In contrast to the higher concentration of PANAA, much smaller variations are observed in both the SEM image and diameter histogram. Because of more chain entanglements at a higher polymer concentration, there is greater resistance from the solution to stretching; thus by increasing the solution concentration, the average diameter of fibres increases.

β -CD was added to PANAA5, PANAA6 and PANAA7 from 10 to 100% (w/w, with respect to PANAA). It was observed that by adding β -CD to PANAA5 solution up to 50% w/w, the diameter of fibres did not change significantly (**Fig-**

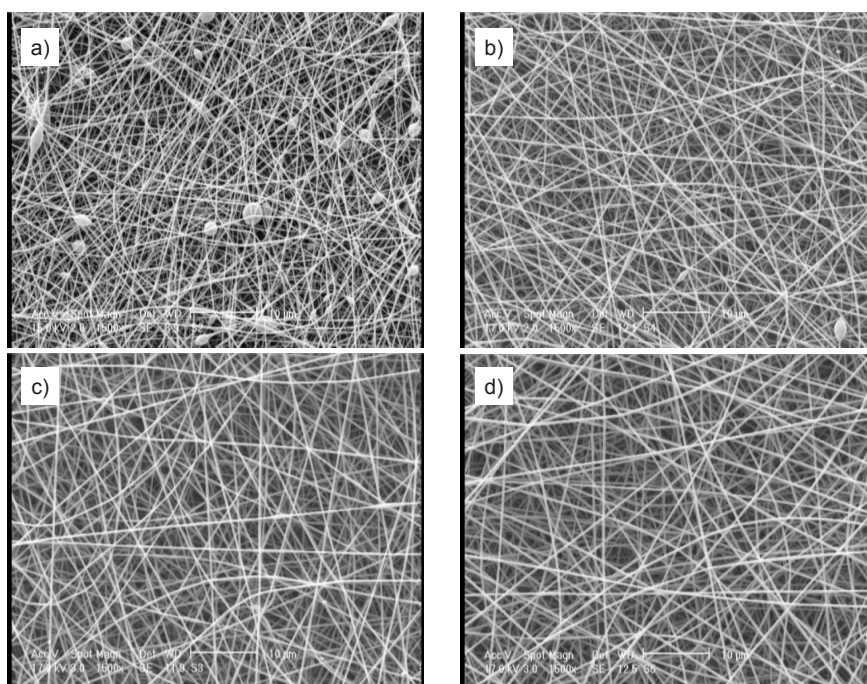


Figure 3. SEM images of electrospun fibres of (a) PANAA6, (b) PANAA6/CD10, (c) PANAA6/CD25, (d) PANAA6/CD50.

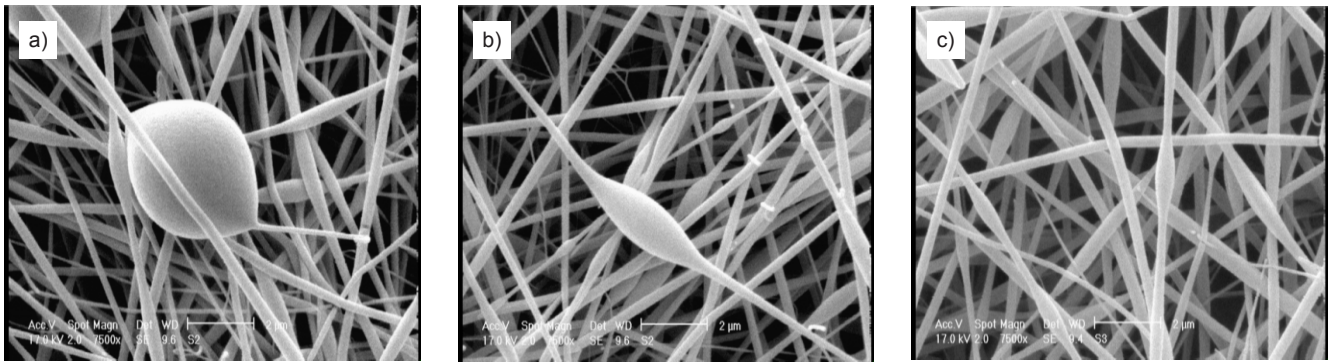


Figure 4. SEM images of electrospun fibres of (a) PANAA5/CD10, (b) PANAA5/CD25, (c) PANAA5/CD50.

ure 2.a). But by increasing the content of β -CD above 50% w/w, the diameters of nanofibres produced increased significantly, which could be due to the hindrance of polymer chain mobility in the solution and change in the hydrodynamic volume caused by the addition of cyclodextrin at higher concentration. The effect of β -CD concentration of 6 and 7% in the copolymer on the nanofibre's mean diameter is more significant (Figure 6.b and Figure 6.c). The average diameter of fibres shows a higher increase by increasing the β -CD concentration from 10 to 100% w/w.

It was observed that adding β -CD to the solution changed the morphology of electrospun fibres and leads to the formation of continuous nanofibres with significantly fewer beads. For example, when a copolymer solution with 6% copolymer concentration was electrospun, a nanofibrous web with beads was obtained. However, the addition of 10% w/w% β -CD to the solution (PANAA6/ β -CD) leads to bead-free nanofibre formation (Figure 3), which means the addition of cyclodextrin enhanced the spinning process by lowering the chain entanglement or controlling the rheological behaviour of the solution during electrospinning. The same phenomenon was observed for a solution with 5% concentration. Beads became more elongated by increasing the amount of β -CD in the solution with 5% concentration. Moreover the shape of beads changed from a sphere to a spindle by increasing the amount of cyclodextrin from 10 to 50% w/w (Figure 4). An electrical field applied to the solution jet could have a better effect by the launching the jet, elongation of the straight segment and development of whipping instability, resulting in the production of fibres with a uniform diameter.

In order to see if the majority of cyclodextrin molecules are located on the surface of nanofibres, we tried to remove the cyclodextrin added by washing it with warm water. Interestingly SEM images showed nanometric furrows which had appeared on the surface of fibres (Figure 5). The presence of these nanofurrows on the surface of nanofibres may indicate that CD molecules present on the surface of nanofibres were removed by washing and that they did not have any chemical bonding with the copolymer or, in other words, did not form any inclusion complex. For PANAA nanofibres which do not contain CD molecules, no such furrows were observed after washing.

The voltage applied was increased from 10 kV to 20 kV while the tip to collector

distance and feed rate were kept constant at 20 cm and 0.5 cm³/h, respectively. It is observed that the fibre diameters and number of fibres increase with increasing the applied voltage. During electrospinning, when the voltage is applied, jet initiation takes place instantaneously. The coulombic repulsion of surface charges on the jet has an axial component that elongates the jet in its passage towards the collector. As the jet thins, the surface area per unit mass of jet material increases while the surface charges per unit area decrease [23]. Solvent evaporation continually increases the surface charge per unit area, driving the increase in surface area through extension. With increasing the applied voltage, electrostatic forces are increased, thus splitting of the jet increases and the rate of solvent evaporation also increases, which results

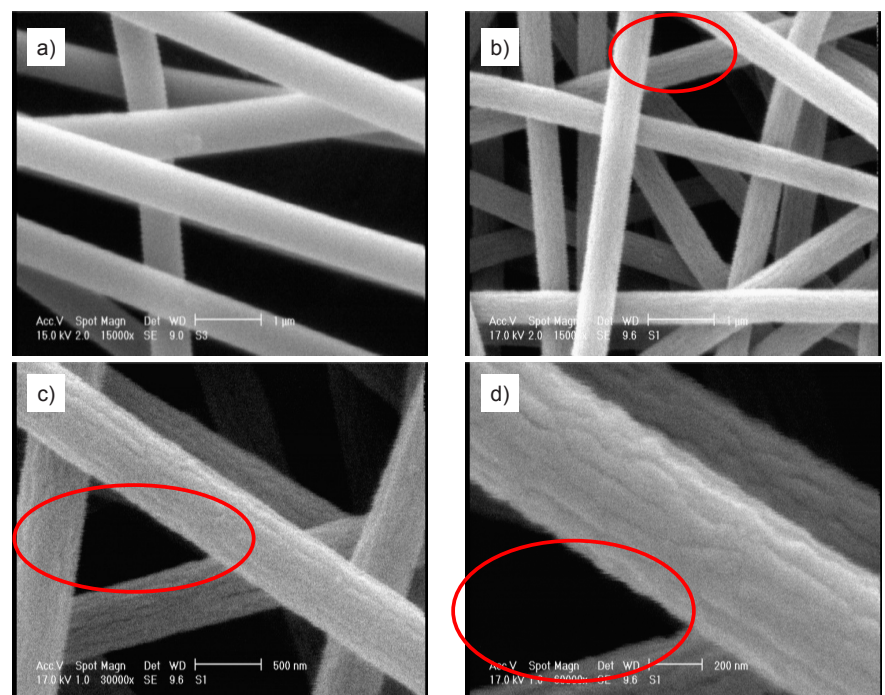


Figure 5. SEM images of electrospun PANAA7/CD100 fibres (a) before and (b, c and d) after washing by warm water at different magnifications (appearance of nanometric furrows on the surface).

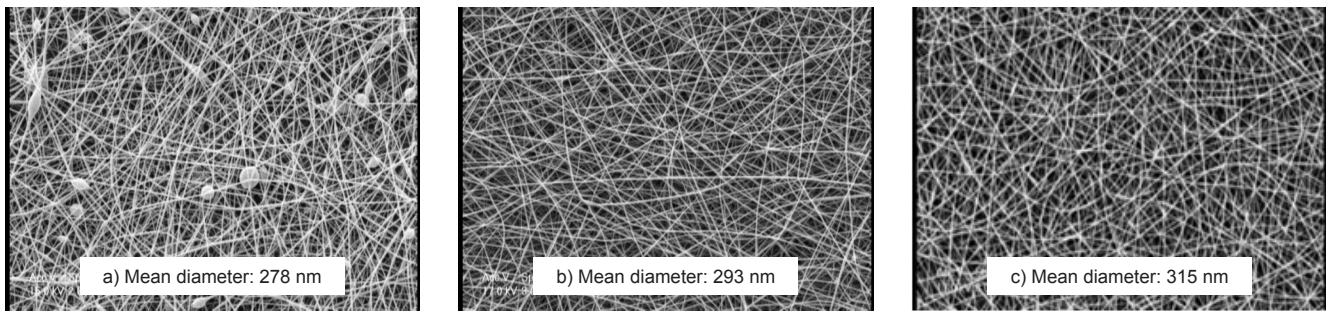


Figure 6. SEM images of PANAA7 electrospun fibres as a function of applied voltage, at a fixed distance from the tip to the collector plate of 20 cm and feed rate of 0.5 cm³/h. (a) 10 kV; (b) 15 kV; (c) 20 kV.

in fibres of smaller diameter. Increasing the applied voltage in this range reduces the number of beads and leads to uniform and smooth nanofibres. In other words, higher voltage induces more charges on the surface of the polymer solution and makes it fully stretched (**Figure 6**).

Applied voltage

Figure 7 shows that by increasing the applied voltage from 10 kV to 20 kV, the diameter of PANAA/CD fibres increases significantly. Regardless of polymer solution concentration this trend is observed. Another point is that when the concentration of the polymer solution is increased, the fibre diameter also increases. The spinning process is directly related to the solution surface tension and its electrical properties. With increasing the solution concentration the surface tension of the solution increases. When the viscosity is high enough, it may discourage second-

ary jets from breaking off from the main one, which contributes to the increased fibre diameter.

Feed rate & tip to collector distance

The effect of the feed rate on fibre diameter was examined by increasing the solution feed rates from 0.5 to 1.5 cm³/h for PANAA10/CD25 while keeping the applied voltage and tip to collector distance constant. The diameters of PANAA10/CD25 nanofibres increase from 749 to 842 by increasing the flow rate of the solution from 0.5 to 1.5 cm³/h (**Figure 8**). A smaller feed rate gives more time to the solvent to evaporate and fibres to stretch, which leads to the formation of more uniform nanofibres with smaller diameters. On the other hand, at a higher feed rate, due to the greater volume of solution drawn from the needle tip, the jet will take a longer time to dry. As a result, solvents in the fibres deposited may not have enough time to evaporate given the

same flight time. Residual solvents may cause the fibres to fuse together where they make contact, forming webs [23]. The amount of solution available for electrospinning will be determined by the feed rate. For a given voltage, there is a corresponding feed rate if a stable Taylor cone is to be maintained. When the feed rate is increased, there is a greater volume of solution that is drawn away from the needle tip; therefore, there is a corresponding increase in the fibre diameter or bead size

Figure 9 demonstrates the effect of changing the distance between the needle and the collector (15 to 30 cm) while keeping the applied voltage and feed rate constant for PANAA10/CD25 nanofibres. It was observed that the fibre diameter decreased by increasing this distance. It is reported that decreasing the working distance has the same effect as increasing the applied voltage

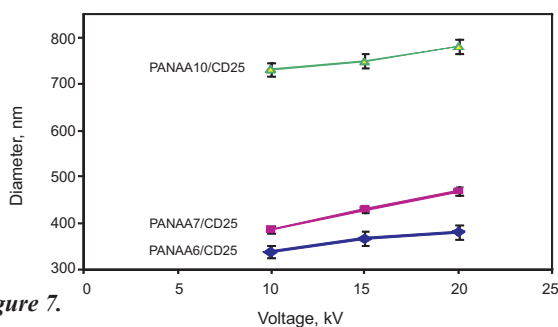


Figure 7.

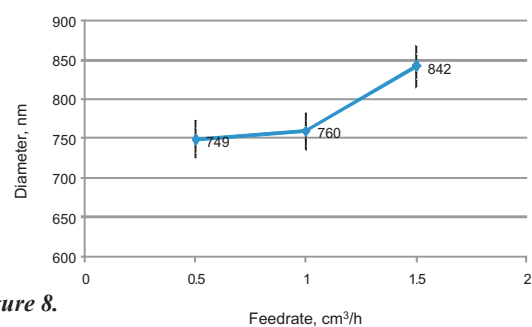


Figure 8.

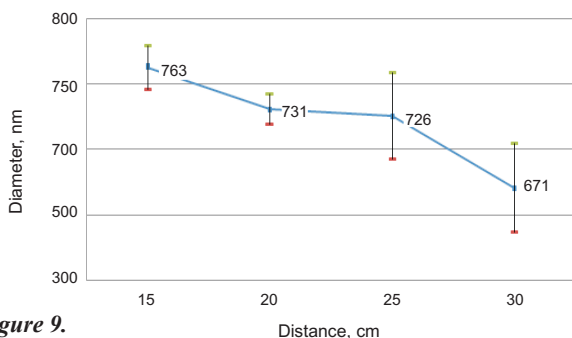


Figure 9.

Figure 7. Fibre diameter vs applied voltage for (a) PANAA6/CD25, (b) PANAA7/CD25 and (c) PANAA10/CD25 solutions.

Figure 8. Change in fibre diameter vs. feed rate for PANAA10/CD25.

Figure 9. Change in fibre diameter vs distance for PANAA10/CD25.

[20 - 25]. When the distance between the tip and the collector is varied from 15 to 30 cm it will have a direct effect on both the flight time and electric field strength. For individual nanofibres to form and be collected on the surface of the collector, the electrospinning jet must have enough time to evaporate its solvents. When the distance between the tip and the collector is reduced the jet will have a shorter distance to travel before it reaches the collector plate. Moreover the electric field strength will also increase at the same time and this will increase the acceleration of the jet to the collector. As a result, there may not be enough time for the DMF to evaporate when it hits the collector [24].

FTIR and XRD characterisation

FTIR spectra were used to confirm the presence of cyclodextrin molecules in PANAA/CD electrospun nanofibres. **Figure 10** shows FT-IR spectra recorded in the spectral range of 500 – 4000 cm^{-1} of pure β -Cyclodextrin powder, PANAA10 and PANAA10/-CD25. Characteristic absorption bands at 1029, 1081 and 1158 cm^{-1} were assigned to β -CD molecules, derived from coupled C–C/C–O stretching vibrations and the antisymmetric stretching vibration of C–O–C [18]. The absorption peak at 2240 cm^{-1} corresponded to C \equiv N stretching vibration, and the one at 1729 cm^{-1} is assigned to C=O stretching vibration. The peak at around 2929 cm^{-1} is derived from the stretching vibration of the methylene (–CH₂–) group. The peak at 1450 cm^{-1} is ascribed to the bending vibration of methylene (–CH₂–). The broad band at around 3222 cm^{-1} in PANAA is a characteristic peak of the hydroxyl group (–OH), which is more intense in the case of PANAA/CD because of hydroxyl groups which exist in CD

XRD analysis was performed in order to investigate the crystalline structure of PANAA and CD molecules in the electrospun PANAA nanofibres and to provide further evidence of the presence of CD molecules within the PANAA/CD nanofibres. **Figure 11** shows the XRD pattern of pure β -cyclodextrin, PANAA7/CD25 and PANAA7/CD100. PANAA is a semi crystalline polymer with a broad halo diffraction pattern which shows two distinct peaks at $2\theta = 17^\circ, 29.5^\circ$ [25]. On the other hand a high crystalline pattern is shown for β -cyclodextrin, with distinct peaks at $2\theta = 7.27^\circ, 11.03^\circ, 13.29^\circ$,

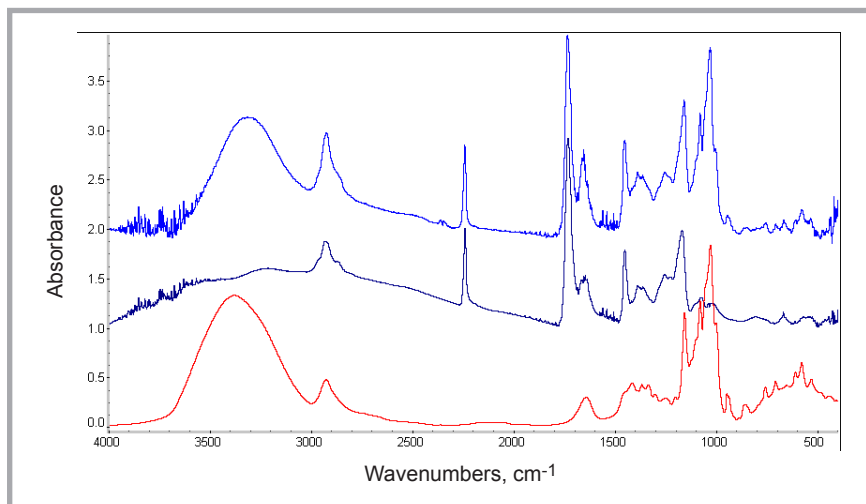


Figure 10. FT-IR spectra of (a) pure β -cyclodextrin, (b) PANAA10 and (c) PANAA10/CD25 electrospun nanofibres.

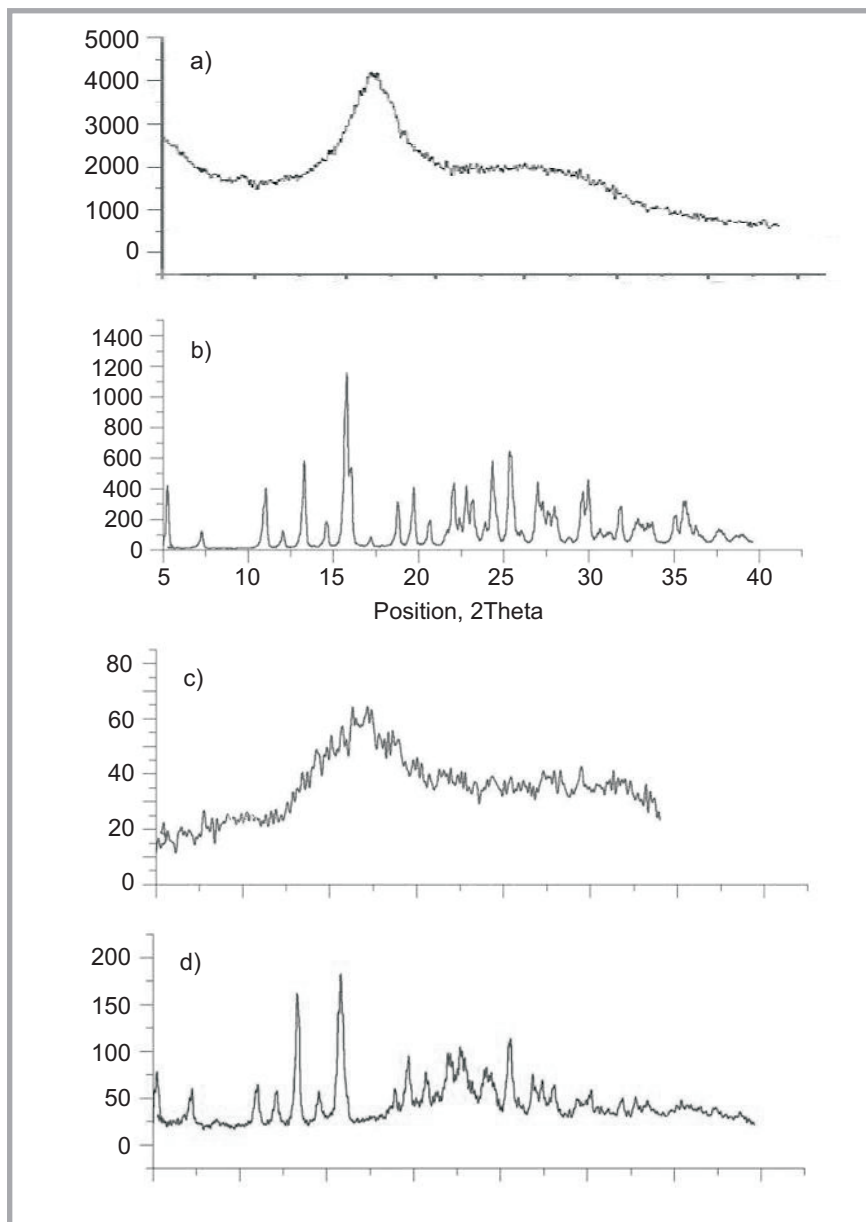


Figure 11. X-Ray diffraction pattern of a) PANAA, b) β -cyclodextrin, c) PANAA7/CD25, d) PANAA7/CD100.

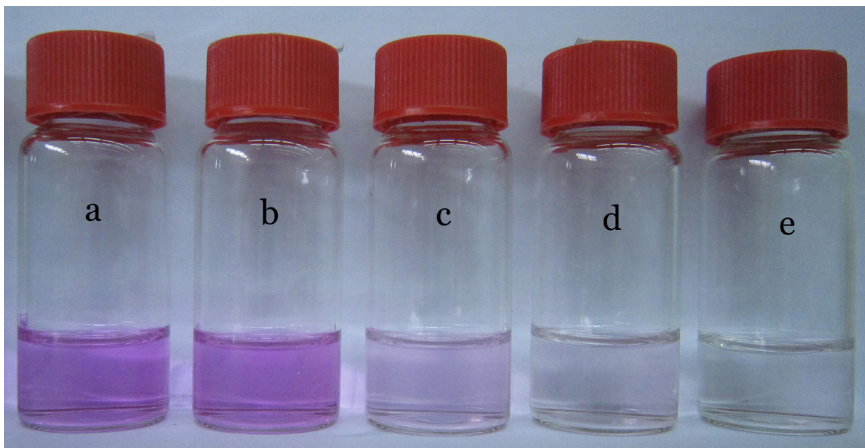


Figure 12. Change in colour of phenolphthalein solutions after one hour floatation of (a) without nanoweb, (b) PANAA10, (c) PANAA10/CD25, (d) PANAA7/CD50 (e) PANAA7/CD100.

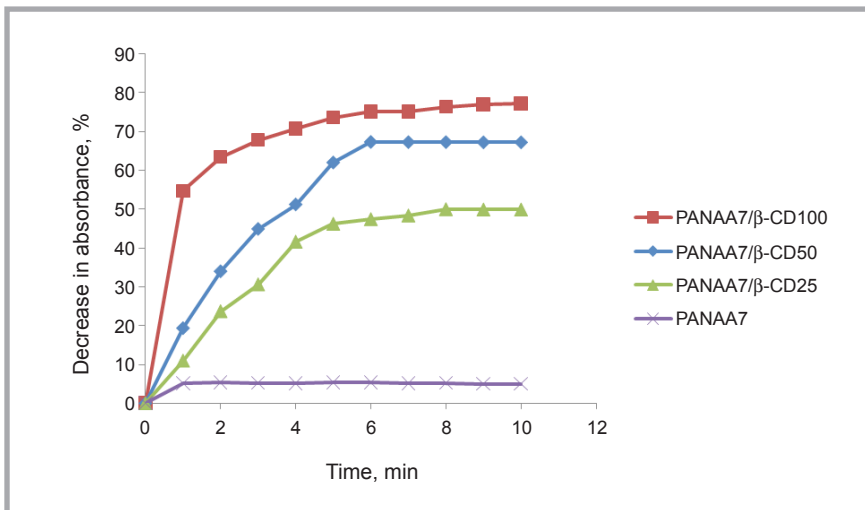


Figure 13. Decrease in absorbance in % of PHP at 553 nm for different nanoweb as a function of time.

15.79°, 19.71°, 22.05°, 24.25°, 25.37°, 26.97°, 29.97°, 31.85°, 35.59° and 37.65°. These peaks appear on the broad halo pattern of PANAA (**Figure 11.b** see page 19) by adding β-CD to PANAA solution, and they become more notable by increasing the content of β-CD PANAA. These results indicate that CD molecules are present in electrospun nanofibres. By increasing β-CD molecules, the XRD pattern does not show any distinct peak for channel-type β-CD crystals. Therefore CD and PANAA did not form any inclusion complexes.

Phenolphthalein test

Different nanoweb were placed in screw tap vials filled with 5 cm³ phenolphthalein (PHP) test solution, and their ability to remove PHP molecules from the solution was studied. **Figure 12** shows the change in colour of the test solutions after an hour. The PANAA nanoweb has

the least potential for removing PHP molecules from the solution. UV-Vis Spectra of PHP test solutions shows absorption maxima at 553 nm, assigned to the purple colour of phenolphthalein. The decrease in absorbance at 553 nm derives from the reduction of purple colour in the solution. It is observed that the absorbance at 553 nm decreases by increasing the content of β-CD in nanoweb.

The absorbance of PHP test solutions at 553 nm was measured as a function of time. Due to the possible instability of PHP control solution, the absorbance of the control solution was measured at the start (C_i) and end of the experiments (C_f) for more accurate results. The percent decrease in absorbance is calculated with respect to 5 cm³ of the PHP control solution [26]:

$$\begin{aligned} \text{Decrease in absorbance \%} &= \\ &= 100 \times (C_n - T_n) / C_n \end{aligned} \quad (1)$$

where: C_n is the absorbance of the control solution and T_n is that of the test solution. Index “n” shows the number of readings. The absorbance of the control solution for each time is calculated from the following equation:

$$C_n = C_i - n\Delta C \quad (2)$$

where: ΔC is the total changes in absorbance of the control solution, which is calculated as follows (assuming all the samples are measured in an equal amount of time):

$$\Delta C = (C_i - C_f) / (N - 1) \quad (3)$$

where, N is the total number of readings. These calculations have been made and the results shown in **Figure 13**. It indicates that the percent decrease in absorbance in 553 nm is much higher for nanoweb which contain cyclodextrin molecules. Therefore nanoweb of PANAA/CD have the ability to remove PHP molecules through β-CDs which are on the surface of these nanoweb, as well as to form inclusion complexes with PHP molecules.

Conclusions

β-cyclodextrin was incorporated into Poly(acrylonitrile/acrylic acid) matrix with the aim of improving the filtration ability of PANAA nanoweb. Uniform nanofibres were produced and the effects of different electrospinning parameters and solution parameters on fibre morphology were studied. FT-IR spectra confirmed the presence of β-cyclodextrin molecules in electrospun nanofibres. XRD patterns provided further evidence of the presence of CDs in the PANAA matrix and indicated that some CD crystal aggregates occurred by increasing the content of β-CD.

The phenolphthalein test showed that fabricated nanoweb of PANAA/CD have the ability to form an inclusion complex with PHP molecules through β-CDs on the surface of these nanoweb. PHP was chosen as a model for organic molecules, and these findings reveal that the incorporation of CD molecules in PANAA nanofibres enhances their filtration ability and may make them applicable for other potential applications.

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The highest category for institutions at which the majority of our authors is affiliated

We are pleased to inform you that in the latest parametric assessment conducted by the Ministry of Science and Higher Education in Poland the following Institutions from Lodz Region working in the textile area received a parametric category “A”:

- Faculty of Material Technologies and Textiles Design of Lodz University of Technology
- Textile Research Institute
- Central Institute for Labour Protection - National Research Institute

We wish them further successful activities and grand achievements in the scientific field related to textiles, fibres and fibrous materials.

The Editors
of *Fibres & Textiles*
in Eastern Europe