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Research Article

Influence of Salts on Electrospinning of Aqueous and Nonaqueous Polymer Solutions

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A roller electrospinning system was used to produce nanofibres by using different solution systems. Although the process of electrospinning has been known for over half a century, knowledge about spinning behaviour is still lacking. In this work, we investigated the effects of salt for two solution systems on spinning performance, fibre diameter, and web structure. Polyurethane (PU) and polyethylene oxide (PEO) were used as polymer, and tetraethylammonium bromide and lithium chloride were used as salt. Both polymer and salt concentrations had a noteworthy influence on the spinning performance, morphology, and diameter of the nanofibres. Results indicated that adding salt increased the spinnability of PU. Salt created complex bonding with dimethylformamide solvent and PU polymer. Salt added to PEO solution decreased the spinning performance of fibres while creating thin nanofibres, as explained by the leaky dielectric model.

1. Introduction

Polymer nanofibres have attracted increasing attention in previous decades because of their high surface to mass ratio, small pore size, and special characteristics attractive in advanced applications. They have potential application in tissue engineering scaffolds, filters, wound dressings, drug delivery materials, biomimetic materials, electronics, and composite reinforcement, among others [1–6].

Techniques to produce nanofibres have been developed for many years. Electrospinning is one of the versatile methods to produce nanofibres. Various worldwide researchers have started to develop alternative methods to produce nanofibres to improve production rates and quality. The most common methods are melt-blown, phase separation, self-assembly, template synthesis, bicomponent, centrifugal, and drawing methods, among others [7–13].

An effective electrospinning method was recently investigated by Jirsak et al. [14]. The principle of this method is based on free surface spinning. This method involves an electrode rotating roller that is immersed in a solution bath. The role of the roller is to feed the solution to the surface of the roller to

continue spinning. Fibres form between the roller surface and the collector. By changing the spinning parameters, having hundreds of Taylor cones on the surface of the roller at the same time is possible. Therefore, a highly dense nanoweb can be achieved by using this method. In general, the diameter of fibres changes from 50 nm to 800 nm depending on the solution properties and spinning parameters.

This paper aims to evaluate the influence of salt on the spinning performance of both aqueous and nonaqueous solution systems by using the roller electrospinning system. To date, many researchers have studied the salt effect on nanofibre morphology, but only a few have focused on spinning performance. For instance, Cengiz and Jirsak [15] examined the effect of salt on polyurethane nanofibre and spinning performance. They found that adding salt increases the number of Taylor cones on the roller surface, thus increasing spinning performance. By contrast, Dao and Jirsak showed that adding salt to polyvinyl alcohol (PVA) solution decreases the number of jets and spinning performance [16].

To the best of our knowledge, no study has been made to explain the opposite effect of salt on spinning performance with different solution systems. To achieve this aim, we used

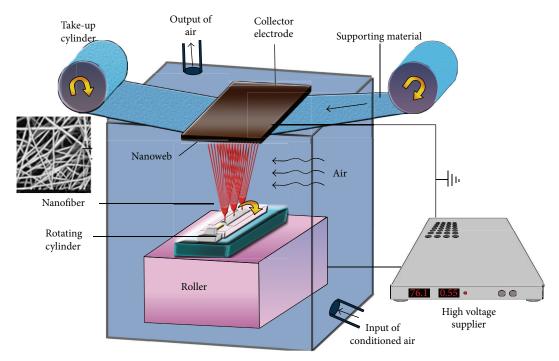


FIGURE 1: Diagram of the roller electrospinning system.

polyurethane (PU) and polyethylene oxide (PEO) polymers with various concentrations of tetraethylammonium bromide (TEAB) and lithium chloride (LiCl) salts. We chose PU in this work for two reasons. First, the PU used in this study is pure and industrially produced. Second, much information on this polymer is available from previous studies, including information on optimum spinning conditions.

In the current work, PEO was used with both TEAB and LiCl salts. In case of PEO, adding salt decreases its spinning performance, similar to PVA solutions. Moreover, PEO is produced in better purity than PVA. One of the aims of this study is to determine the different spinning behaviours of both water-soluble and -insoluble polymers. Using PVA, which is mostly used in electrospinning, is possible. Although PVA has been studied by many researchers, PEO was chosen in this work for its more stable quality and better purity. The possible salt-polymer, salt-solvent, and solvent-polymer relations are explained in the following section.

2. Experimental

2.1. Materials. Polyurethane Larithane LS 1086 (Novotex, Italy), which is an aliphatic elastomer composed of 2000 g/mol linear polycarbonate diol and isophorone diisocyanate and extended by isophorone diamine, was chosen as the second polymer.

Most PUs are block polymers prepared with a dissocyanate, which is a short diol such as 1,4-butanediol or 1,6-hexanediol, a diamine (the chain extender), and a diol, with a molecular weight of 500 to 4000 based on a polyether, polyester, or polycarbonate. Preparation is usually performed in two steps: the reaction of the longer polyol with isocyanate

in the first stage and that with the chain extender in the second stage [17]. PU has excellent damping properties, good mechanical and physical properties even at low temperatures, high combustion resistance, and low thermal conductivity [18]. DMF (Fluka, Switzerland) was used as the solvent.

Water-soluble PEO with molecular weight of 400 kDa was purchased from Scientific Polymers, Inc., USA. Distilled water was used as the solvent. PEO is a water-soluble and non-ionic polymer. PEOs are also commonly studied in electrospinning. They are available in a large range of molecular weights. PEOs can be applied in areas such as textile applications, cosmetics, antifoaming agents, and food industry, among others. PEOs are produced by the polymerization of ethylene oxide, and they have a structural polyether unit of -CH₂-CH₂-O-. They are a good candidate for an electrospinning system because of their high spinnability and water solubility.

Tetraethylammonium bromide was purchased from Fluka (Switzerland) and LiCl from Lach-Ner s.r.o. (Czech Republic). Based on previous works, 6% PEO and 17.5% PU were chosen as the constant polymer concentrations. Various amounts of salt were used according to the molar ratio of salt. The nomenclature of solutions is tabulated as shown in Nomenclature and Symbols of Solutions according to salt content. We used a small amount of LiCl salt content for the PEO solution because the fibre diameter increases with increased amount of salt.

2.2. Methods

2.2.1. Spinning Conditions. The solutions were spun using a spinning device, as shown in Figure 1. All the measured

Sample	Voltage (kV)	Distance (mm)	Roller speed (rpm)	RH (%)	Temperature (°C)	Roller length (mm)	Roller diameter (mm)
6% PEO + salt series	42	150	1	28.5 ± 2	23 ± 1	145	20

TABLE 1: Spinning conditions of PEO solutions in the roller electrospinning system.

TABLE 2: Spinning conditions of PU solutions in the roller electrospinning system.

Sample	Voltage (kV)	Distance (mm)	Roller speed (rpm)	RH (%)	Temperature (°C)	Roller length (mm)	Roller diameter (mm)
17.5% PU + salt series	62	130	1.5	24.5 ± 2	16 ± 1	145	20

results in the figure have an error bar at 95% confidence intervals. The spinning conditions of PEO and PU are shown in Tables 1 and 2.

- (i) Measurement of surface tension: measurement was carried out using a KRÜSS tensiometer at 25°C and LabDesk software by using plate method.
- (ii) Measurement of viscosity: the zero-shear viscosities of solutions were measured by Haake RotoViscol at 23°C.
- (iii) Measurement of conductivity: the conductivities of polymer solutions were measured at 23°C by a Radelkis OK-102/1 conductivity meter.
- (iv) Measurement of jets and spinning area: a Sony Full HD NEX-VG10E Handycam E 18–200 mm lens camera was used in the experiments. By using camera, the number of jets was recorded. Spinning area and number of jets were determined by taking an image from the camera and using NIS-Elements software. 10 images per second were taken. A number of jets were counted by using images.
- (v) Measurement of spinning performance and performance per jet: $10 \times 10 \, \mathrm{cm}^2$ nanofibre webs were prepared and measured on a balance. The calculations were made according to (1)-(2).
- (vi) Measurement of fibre diameter and diameter distribution: images of the microstructure of the nanofibre membrane were taken by scanning electron microscope (SEM; Feico). NIS-Elements software was used to determine the fibre diameter and diameter distribution
- (vii) Measurement of nonfibrous area: using SEM images and NIS-Elements software, nonfibrous areas were calculated.
- 2.2.2. Calculation of Spinning Performance. Spinning performance (SP) can be determined from the mass of nanofibres produced in a 1 m long roller spinning electrode in 1 min. Spinning performance is calculated from an area weight of the produced nanofibre layer as follows:

$$SP = \frac{G * v * L_f}{L_r}, g/min/m,$$
 (1)

where G is the area weight of the nanofibre membrane per area in g/m^2 . ν is the velocity of running of the collected fabric in m/min. L_f is the width of the nanofibre membrane on the collected fabric in m. L_r is the length of the spinning roller in m.

Spinning performance per one Taylor cone (SPC) can be calculated from the known values of spinning performance and an average total number of Taylor cones in the spinning electrode Nc using (2). SPC is an amount of polymer solution transported through one Taylor cone (or a jet):

$$SPC = \frac{SP * L_r * 60}{Nc}, g/h.$$
 (2)

SPC is one of the parameters to be measured in the experiments to determine whether spinning performance is realized through SPC or Nc.

3. Results and Discussions

3.1. Polymer Solution Properties. The basic properties of polymer solutions are given in Figures 2, 3, and 9.

Surface tension of the solutions corresponds to that of the used solvents and is not significantly dependent on the content of salts. Thus, surface tension is not an influencing independent parameter, such as spinning performance and fibre diameter, in the experiments.

Viscosity of the solutions, as a function of share rate, shows considerably different characteristics of both polymers. Effective viscosity of PEO strongly depends on share rate and that of PU shows only moderate dependence. Therefore, the macromolecules of PEO 400 kDa show a high degree of mechanical entanglement and a highly macromolecular characteristic. The strength of PEO jets as a necessary requirement for spinnability is satisfactorily high at a relatively low polymer concentration and corresponding viscosity. However, spinnability of PU requires a high polymer concentration and corresponding viscosity. Viscosity of PU solutions increases with salt content; this is not the case in PEO solutions.

The addition of LiCl to PU solution increases its viscosity. Erokhina et al. explained that this increase in viscosity could be due to the same coordination of lithium cation bonds in the solution with DMF molecules. They concluded that the partial recoordination of the lithium cation from the DMF carbonyl groups to the PU carbonyl groups in the ternary system probably caused the unfolding of macromolecular

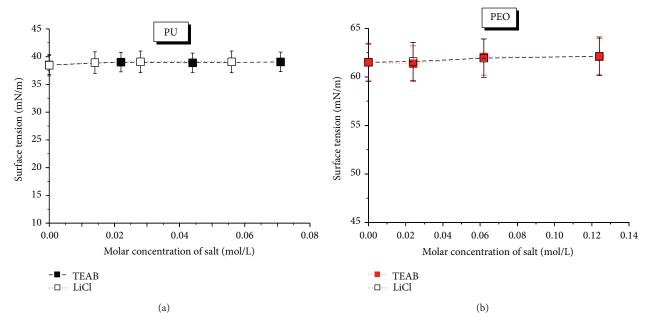


FIGURE 2: Surface tension of polymer solutions.

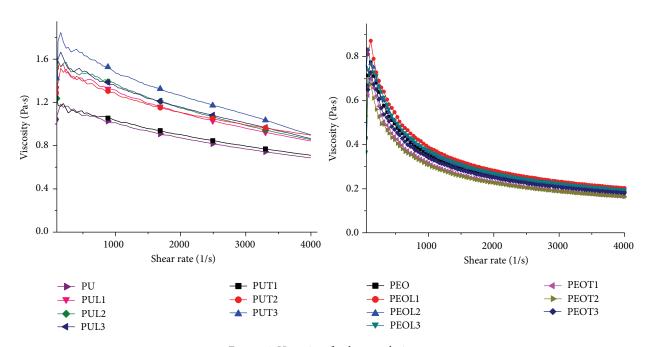


Figure 3: Viscosity of polymer solutions.

coils in PU with the formation of intermolecular crosslinks [20].

PU and LiCl salts have secondary bonds similar to the H-bridges between PU and LiCl ions (Figure 4). Intermolecular interactions are positively influenced by polar groups. Beside this, LiCl makes the functional groups of PU more polar.

The interactions between dimethylformamide (DMF) and TEAB [19] or between PU and salts [15, 21] are shown in Figures 5 and 6.

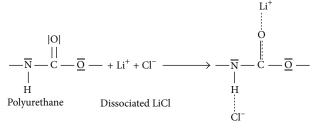


FIGURE 4: Chemical interaction between PU and LiCl.

Fry studied the interactions between polar organic solvents and salts [19]. The electrostatic interaction between the dipolar solvent and the individual ions of the salt is greater than the attraction of the ions of the salt for each other in the lattice. Salts dissolve in polar solvents, and this phenomenon is called as general solvation. Fry [19] found that, aside from general solvation, small or highly charged metal cations, such as Li⁺ or Mg⁺², in water or other electron pair donor solvents could also attract a shell of tightly bound solvent molecules. This phenomenon, known as inner-sphere or specific solvation, provides added stability to the positive charge in the cation through its interaction with the negative end of the solvent dipoles. General solvation mainly depends on the dielectric constant (ε) of the solvent regardless of its chemical structure. Conversely, specific solvation depends on the chemical structures of both solute and solvent. Fry conducted a computational study demonstrating that smaller tetraalkylammonium ions (Me₄N⁺ and Et₄N⁺) are surrounded by a strong solvation shell in the strong donor DMF solvent. The four solvent molecules are distributed symmetrically around the tetrahedral cation, and no remaining space is sterically allotted for a fifth solvent molecule. The tetrahedral arrangement of solvent molecules is the same as the structure of $Et_4N^+/(H_2O)_4$, as established by molecular dynamics, and is similar to that of the Li(THF)₄⁺ion, as established by X-ray crystallography [19].

Rastogi [22] studied the ion-dipole interaction energy of alkali metal cations (e.g., Li⁺), anions (e.g., Cl⁻), and symmetrical tetraalkylammonium ions in DMF and other solvents. He showed that the ion-dipole interaction energy decreases in increasing order of Li⁺ > Cl⁻ > Et₄N⁺ in DMF solvent. Moreover, the ion-dipole interaction energy of ions is generally higher than the dipolar interaction energy of solvents that cause secondary solvation in large ions (Cl⁻, Br⁻) and longrange polarization in small ions (Li⁺).

In the case of PEO-water solutions, the addition of salt only affects conductivity and permittivity. Viscosity of solutions does not change when salt is added. Salt and polymer macromolecules do not seem to have a significant interaction.

The values of solution conductivities of LiCl and TEAB salts in the same molar concentration in DMF are illustrated in Figure 7. Dash lines indicate the connection points.

The conductivity of LiCl and TEAB in the same molar concentration in water is illustrated in Figure 8. Dotted lines indicate the connection points.

The conductivity of the solutions of both TEAB and LiCl in water and DMF is generally high, and all the values are surprisingly close to each other. TEAB shows the same conductivity in water as LiCl does despite its evidently larger ions. The values of conductivity in DMF are surprisingly close to those in water, thus indicating the high degree of dissociation of salt in DMF. Conversely, the conductivities of polymer solutions containing salt differ from each other to some extent. PEO solutions show higher conductivity than PU solutions because of their lower viscosity and corresponding greater movability of ions in a direct electric field. PU solutions containing LiCl are more conductive than those

with TEAB because their ions are more movable in highly viscous liquid.

According to Karmakar and Ghosh, in PEO-lithium salt-based solid polymer, the macromolecule coils around Li⁺ ions and the O-atom in PEO chain provide a coordination site for Li⁺ ions through the Lewis acid-base interaction. Li⁺ ions jump from one coordination site to another within the amorphous phase. Moreover, the chain mobility of the polymer host, which plays an important role in ion transport, makes the ion transport mechanism in polymer electrolytes complex [23].

Collins et al. [24] showed that, in the absence of an electric field, charged structures capable of supporting current could be produced by the general equilibrium as follows:

Neutral molecule
$$\underset{k_2}{\overset{k_1}{\rightleftharpoons}}$$
 ion pair $\underset{k_f}{\overset{k_d}{\rightleftharpoons}}$ free ions (3)

The neutral molecule and the ion pair are not capable of supporting current, and the rate constants k_1 and k_2 are generally not known and are not important to the treatment of the problem of conduction in liquids. This step that produces free ions from ion pairs is critical to understanding the development of conduction in liquids. The rate constant k_d is related to the dissociation of the ion pair into the charged ions, and the rate constant k_f is related to the removal of free ions through the recombination into ion pairs. Moreover, with the application of a voltage with a positive polarity to the electrode that supports the solution, the mechanism of the charge carrier generation is called field enhanced dissociation. Negative charges are immobilized in the electrode, leaving mobile positive charges to respond to the electrostatic stresses imposed by the electric field. The unconstrained surface of the fluid enables multiple spinning sites to develop, as shown in Figure 10 [24].

In the case of PEO in water solution, the dissociation of the ion pair into the charged ions of the water molecules under electric field is expressed as follows:

$$2H_2O \longleftrightarrow H_3O^+ + OH^-$$
 (4)

This creates a high number of ions. Negatively charged ions are immobilized in the positively charged spinning electrode, whereas positive charges move towards the collector electrode. Adding salt increases the conductivity of solution over the value required for the leaky dielectric model and leads to the decreased number of Taylor cones. In the case of PU solution, the molecules of DMF solvent do not dissociate. Therefore, field enhanced dissociation is also not present.

PEOs in water solution show extremely high spinning performance because of their high polarity and hygroscopicity. The PEO chains are used as a hygroscopic part of detergents because of these properties. Their high polarity, especially in water solutions, is characterized by a high value of the dielectric constant, $\varepsilon = 39$ [25, 26].

Other basic properties of the solutions were not measured. However, a number of differences between the two solutions may exist that may cause their different behaviours in the electrospinning process. For instance, the kind and

FIGURE 5: Computed structure of the tetramethylammonium ion and lithium ion complexed to four N,N-dimethylformamide molecules [19].

FIGURE 6: Chemical interaction between LiCl and PU.

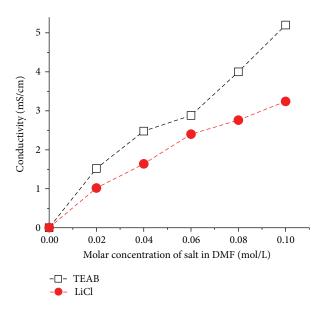


FIGURE 7: Conductivity of TEAB and LiCl solutions in DMF. Dash lines indicate the connection of points.

concentration of polar groups in polymers, solvents, and polymer-solvent-salt systems are responsible for the interactions of the component solutions with the electric field. The characteristic and content of polar groups influence the dielectric constant of materials. Water, DMF, and PEO show high values of permittivity (80, 38, and 39, resp.) [25, 26]. The permittivity of PU is low (5–7), which may be the reason for its poor spinnability. Spinnability of PU considerably increases with the addition of salt [15]. This increase may be

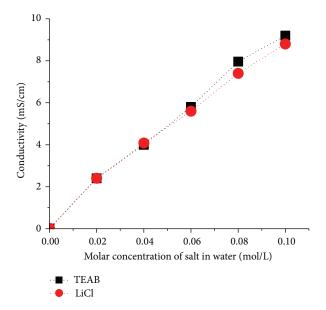


FIGURE 8: Dependence of water solution conductivity on the concentrations of LiCl and TEAB. Dash lines indicate the connection points.

caused by the interactions between DMF and TEAB [19] or between PU and salt.

3.2. Number of Jets. In electrospinning, PU and PEO show important differences in their behaviour, such as the number of jets on the spinning roller, as shown in Figure 11.

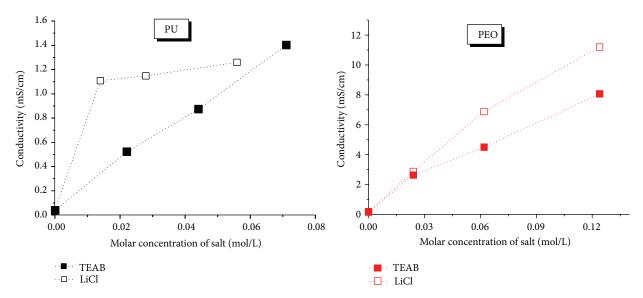


FIGURE 9: Conductivities of polymer solutions with salt content (dotted lines indicate the connection of points).

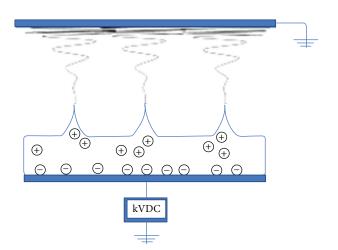


FIGURE 10: Multiple fluid jets using the plane-plane electrode configuration without capillaries.

First, the number of jets is considerably larger in PU than in PEO by adding salt. Two attempts have been made to explain this difference.

- (1) The theory of shielding effect of conducting lightning rods was considered [27]. According to this theory, the electric field is screened out in conical space with a tip at the end of the conductor and a top angle of about $45^{\circ}-60^{\circ}$ [28–34].
- (2) Lukáš et al. [35–37] calculated the distance between jets by calculating the inter-jet distance called the critical wavelength. This parameter enables the estimation of the relative productivity of the electrospinning process.

Second, the number of PU cones increases with the salt content of the solution. By contrast, the number of PEO cones

decreases with the increase in salt concentration. These effects are difficult to explain as salt plays multiple roles.

- (1) Salt (TEAB more than LiCl) creates complex structures (Figure 6) with PU, which leads to changes in the macromolecule-macromolecule and macromolecule-solvent interactions. Consequently, viscosity, related entanglement number, and stronger jets increase. The following are the effects of salt on a PU solution.
 - (i) Stronger jets result in longer average life of jets [20].
 - (ii) The jets are shorter because of higher content of ions and greater viscosity [38], and the number of jets increases.

These effects of salt do not occur in a PEO solution as salt does not create complex structures with PEO.

(2) Salt increases the conductivity of polymer solutions. Increase in conductivity changes the characteristic of the polymer solution from a semiconductor to a conductor. Therefore, the solution loses the characteristic of a leaky model, which leads to the loss of ability to create Taylor cones. The leaky dielectric model was first proposed by Melcher and Taylor [39]. According to Bahattacharjee and Rutledge [40], "a leaky dielectric differs from a perfect conductor or a dielectric material in that free charges accumulate on the surface of the material in the presence of an external electric field and modify the local field. Under these conditions, two components of the electrical field develop, one tangential to the interface and another normal to it. The presence of a tangential component on the surface prevents the interface from being in an equilibrium condition and provokes it to deform. By contrast, the electrical stress in perfect dielectrics and conductors is always perpendicular to the interface.

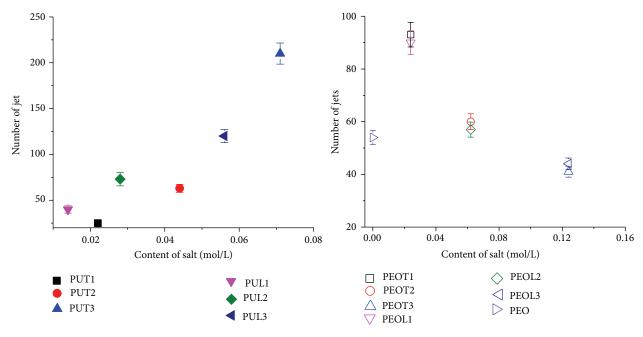


FIGURE 11: Number of jets on a roller.

Electromechanical coupling occurs at the fluid-fluid interface alone; forces resulting from charges in the bulk are negligibly small. As the fluid accelerates, the tangential component of the electrical stress is largely balanced by the viscous, or viscoelastic, response of the fluid. Therefore, both the constitutive behaviour and the electrical properties of the fluid determine the condition of the process. If changes in the conductivity resulting from salt addition are large enough to alter the behaviour of the fluid from that of a leaky dielectric to that resembling a conductor, then the tangential component of the electrical stress that accelerates the fluid is likely to diminish and the flow process to be stopped. Through this limit, the electrical stress is balanced only by the alteration of the shape of the interface and surface tension only" [40].

Apparently, the effect of salt according to (2) works against that in (1). In the case of PU, the effects described in (1) predominate those in (2). In the case of PEO, only the effects according to (2) apply.

The differences between PU and PEO behaviours are also based on different polymer characteristics.

- (1) PEO 400 kDa has a molecular weight high enough to create strong jets even at a low concentration and corresponding viscosity. This is not the case in PU as PU needs an increase in entanglement level using salt.
- (2) PEO contains strong polar groups to obtain strong interactions with an electric field. This condition is expressed by a high value of dielectric constant. Again, this is not the case in PU as PU needs an increase in polarity by creating complexes with salt.

3.3. Spinning Performance and Spinning Performance per One Cone. Spinning performance and spinning performance per jet were measured and calculated for both solution systems. In case of PU without salt, spinning was not observed. However, in the case of PEO without salt, spinnability was high and only the polymer solution was transported to the surface of the collector without forming fibres. A possible consequence of this behaviour is the electrical conductivity variation.

PU polymer shows good spinnability when salt is added to it. Two kinds of salts (TEAB and LiCl) are used as additives; both increase the conductivity and viscosity of solutions. Cengiz and Jirsak [15] observed that TEAB increases the viscosity of PU solutions, which means more extensive interactions among macromolecules. A polymer network becomes more solid. It leads to higher spinning performance of solution [21].

PEO solution without salt is transported from the spinning electrode to the collector in the electrospinning device, but no fibres are formed; only the polymer solution moves towards the collector. Hundreds of jets can be observed. The addition of salt to the solution decreases its spinning performance, and nanofibres are formed.

In principle, the spinning performance (Figure 12) shows the same tendencies as the number of jets. Nevertheless, spinning performance per jet (Figure 13) is not an independent quantity. The amount of polymer solution flowing through one Taylor cone depends on the viscosity of solution, the thickness of a solution layer, and the drawing force of an electric field, which are dependent on the dielectric properties of the polymer or polymer solution.

3.4. Fibre Diameter and Nonfibrous Area. Quality of the produced nanofibres and nanofibre layers was tested in the

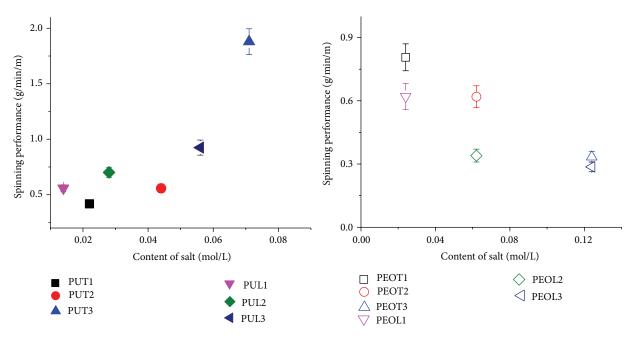


FIGURE 12: Spinning performance of PU and PEO nanofibres in various salt contents.

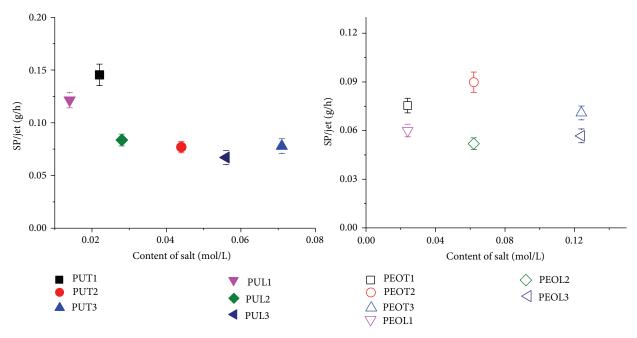


FIGURE 13: Spinning performance per jet of PU and PEO nanofibres in various salt contents.

experiments in terms of fibre diameter (Figure 14) and nonfibrous area (Figure 15). In the PU electrospinning, the highest salt content leads to an increase in viscosity and slightly changes fibre diameters. High salt content also leads to a low quality of PU nanofibre layers. By contrast, PEO nanofibre diameter and quality of nanofibre layers do not significantly depend on salt content above a certain limit.

4. Conclusion

The main results of the experiments are as follows.

(i) Salt may influence the entanglement number and polarity of macromolecules when creating complex bonds with them. It also increases the conductivity of

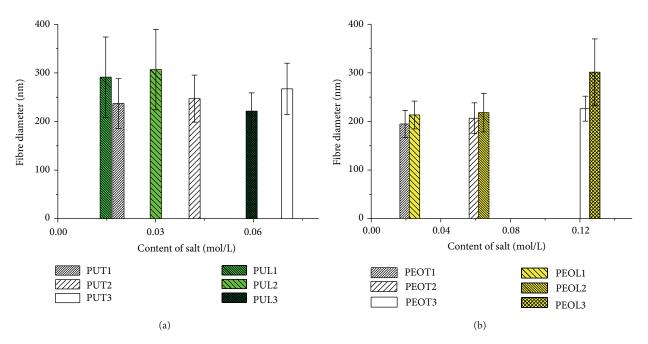


FIGURE 14: Fibre diameter versus salt content (a) PU with TEAB and LiCl salts, (b) PEO with TEAB and LiCl salts.

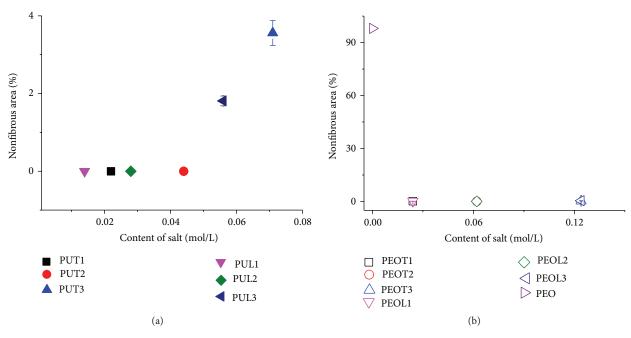


FIGURE 15: Nonfibrous area versus salt content (a) PU, (b) PEO nanofibres.

solutions that may cross the limit suggested in the leaky dielectric model.

- (ii) PEO at 400 kDa, with its high polarity and high entanglement number (strength of jets), shows high spinning performance. This performance is reduced by the increase in conductivity.
- (iii) In the case of PU, salt creates complex bonds with the polymer and increases the low polarity and entanglement number, consequently increasing the spinning

performance. Further addition of salt may lead to reduced spinning performance. However, it cannot be proved because of the extreme increase in solution viscosity.

Future Works

The results of this work should be considered as initial findings on defining the parameters of needleless electrospinning, introducing new parameters and developing methods

to measure these new parameters for both aqueous and nonaqueous solution systems. In the future, the following topics should be focused on.

- (i) Not enough studies have been conducted on the permittivity effect on electrospinning. Theoretical studies should present a full explanation and complete description of the electrospinning process involving the effects of permittivity on dependent parameters, such as length of jet, distance between jets, current on a jet, spinning performance, fibre diameter, lifetime of jets, and spinning area.
- (ii) Studies should be made on dependent and independent parameters for both solution systems.
- (iii) A full understanding of the relation between independent and dependent parameters should be presented.

Nomenclature and Symbols of Solutions

PEO Solutions in Water, Polymer Concentration 6 wt.%

PEO-D: 0 concentration of salts
PEOT1-□: 0.024 mol/L TEAB
PEOT2-O: 0.062 mol/L TEAB
PEOT3-Δ: 0.124 mol/L TEAB
PEOL1-∇: 0.024 mol/L LiCl
PEOL2-Φ: 0.062 mol/L
PEOL3-Φ: 0.124 mol/L.

PU Solutions in DMF, Polymer Concentration 17.5 wt.%

PU-▶: 0 concentration of salts
PUT1-■: 0.022 mol/L TEAB
PUT2-●: 0.044 mol/L TEAB
PUT3-▲: 0.071 mol/L TEAB
PUL1-▼: 0.014 mol/L LiCl
PUL2-◆: 0.028 mol/L LiCl
PUL3-◀: 0.056 mol/L LiCl.

Conflict of Interests

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

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