

# Influence of rotor structure and process parameters on polyethylene oxide (PEO) nanofibers produced through centrifugal

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## ABSTRACT – REZUMAT

### Influence of rotor structure and process parameters on polyethylene oxide (PEO) nanofibers produced through centrifugal

*The application of new tools and equipment in conventional spinning has increased with the advancements in operations, handling and optimal yarn production. For example, in the centrifugal electrospinning process (CESP), the rotor is assembled for its high-speed production. Therefore, the purpose of this study is to introduce a new rotor design with a triangular groove structure and to investigate its influence on the fast industrial manufacturing of polyethylene oxide (PEO) nanofibers. In addition, electric voltage (45 kV, 55 kV, 65 kV), concentrations of the spinning solution of PEO polymer (6 wt. %, 7 wt. % and 8 wt. %) and the flow rate of the spinning solution were analysed at different levels (45 ml/h, 55 ml/h, 65 ml/h). The subsequent PEO nanofibers were characterized through a scanning electron microscope (FESEM). It was observed that the diameter of PEO nanofibers changed with the variation in voltage, concentration and flow rate. The results revealed the best and uniform fibre diameter dimension at 65 kV, with an 8 wt.% solution concentration and flow rate of 55 ml/h. The outcomes also implied that the proposed triangular groove rotor was an efficient approach for the improvement in the nano fibres with its high uniformity as compared to the conventional structure (rectangular rotor structure).*

**Keywords:** centrifugal electrospinning, polyethylene oxide, nano-fibres, rotor, yarn

### Influența structurii rotorului și a parametrilor de proces asupra nanofibrelor de oxid de polietilenă (PEO) produse prin centrifugare

*Aplicarea de noi instrumente și echipamente în filarea convențională a crescut odată cu progresele în operarea, manipularea și producția optimă de fire. De exemplu, în procesul de electrofilare prin centrifugare (CESP), rotorul este realizat pentru producția la mare viteză. Prin urmare, scopul acestui studiu este de a introduce un nou design de rotor cu structură triunghiulară și de a investiga influența acestuia asupra producției industriale rapide a nanofibrelor de oxid de polietilenă (PEO). În plus, tensiunea electrică (45 kV, 55 kV, 65 kV), concentrațiile soluției de filare de polimer PEO (6 wt. %, 7 wt. % și 8 wt. %) și debitul soluției de filare au fost analizate la diferite niveluri (45 ml/h, 55 ml/h, 65 ml/h). Nanofibrele PEO rezultate au fost caracterizate cu microscopul electronic cu scanare (FESEM). S-a observat că diametrul nanofibrelor PEO s-a modificat odată cu variația de tensiune, concentrație și debit. Rezultatele au evidențiat cea mai uniformă dimensiune a diametrului fibrei la 65 kV, cu o concentrație de soluție de 8 wt.% și un debit de 55 ml/h. Rezultatele au indicat, de asemenea, că rotorul propus cu canelura triunghiulară este eficient pentru îmbunătățirea nanofibrelor prin uniformitatea sa ridicată în comparație cu structura convențională (structura dreptunghiulară a rotorului).*

**Cuvinte-cheie:** electrofilare centrifugală, oxid de polietilenă, nanofibre, rotor, fir

## INTRODUCTION

Tailored fibrous strands are currently in high demand because of their potential applications in a wide variety of fields [1]. These fine fibrous strands from natural or synthetic polymers with diameters extending from tenths of nanometres to a couple of microns [2] presently are produced by techniques such as template synthesis [3], phase separation [4], self-assembly [5], melt blowing [6], and electrospinning [7]. Amongst all techniques, electrospinning (ES) has been considered the most extensive and proficient method for fabricating fine fibrous strands at the

nanoscale [3, 8]. Moreover, electrospinning is believed to be a state-of-the-art fibre and membrane manufacturing technique. The process can produce endless fibrous strands from a huge range of polymer solutions. Fibres produced by the electrospinning technique are considered building blocks for developing tailored membrane structures for a wide range of applications such as energy, biomaterials, filtration, composites and protective textiles etc. [9]. However, the limited production rate of the process hinders acceptability on a commercial scale [10, 11]. Therefore, it is highly needed to figure out some

means to enhance the production rate of the electrospinning process to make it scalable enough to overcome the constraints of fast industrial manufacturing [11].

For this purpose, the needless electrospinning method is believed to be a high potential and feasible option. A thorough literature review suggests that only a very small number of studies have reported the successful fabrication of micro/ nano-fibres using centrifugal electrospinning (CES). Various polymers, such as polyethylene oxide [12–15], polyvinylidene fluoride [16], polymethyl methacrylate [17], and polycaprolactone [18] have been electrospun using CES. Thus, this recently established CES method has gained considerable attention due to its simple working principle and improved rate of production fibres with submicron diameters [19–21]. Additionally, in CES, the rate of fibre production is more ( $\geq 1$  g/min per nozzle) and two nozzles are used [22, 23]. Moreover, the CES has successfully overcome the major obstacles such as (solution conductivity, application of high-voltage electric field, safety and environmental concerns etc.) that the former type of electrospinning (ES) encounters [14, 24–26]. Other studies proposed a model that included solution viscosity, and evaporation rate, and contained a specific charge [27]. Similarly, Gao H. et al. anticipated a touch spinning method for the fabrication of core-sheath nanofibrous piezoelectric yarns with a single filament electroconductive core [28]. Thus, various parameters of the CES process can greatly influence the dimension and diameter of the resultant fibres [29, 30]. These parameters include the concentration of polymer, rotational speed, electric voltage, nozzle size, temperature (for melts), evaporation rate, collector distance, etc. [24–34]. All the parameters are obligatory to configure appropriately before the production of desired dimension fibres [35]. However, amongst these parameters, solution concentration and rotational spinning speed are considered the foremost factors [36]. When the solution concentration is kept lower, the fibres produced have a narrower diameter [37]. Further, there is an elusive relationship between fibre morphology and the rotational speed of the machine [38].

Several studies and research have demonstrated the pros and cons of optimising ultrafine fibres [17, 39–42]. So far, the most associated study describing the influence of rotor structure design in the CES process was reported by Hui Wu et al. for generating highly oriented ceramic nanofibers. It was different from the conventional CEP process as unlike the conventional setup a metallic triangular tip without any solution supply was used as electrospinning sources. The tip was prepared by cutting down a 1-mm-thick aluminium sheet into a 1–5 cm rectangle with a triangular tip; it helped to establish a Tylor cone while electrospinning [43, 44]. Thus, we believe using an electrospinning machine having both centrifugal and electrostatic force with a newly proposed high-speed rotor structure i.e., triangular groove rotor

structure, maybe a possible way forward. The polymer spinning solution was fed to the nozzle with a controlled feeding rate. The centrifugal force throws the spinning solution, electrostatic force draws the polymer jet into fine strands with simultaneous evaporation of the solvent, and resultant fibres are then deposited on the collector. Herein, we report a comparative study and analysis of polyethylene oxide nano-fibres prepared by using a rectangular groove structure rotor and triangular grooved structure rotor via electrospinning. Furthermore, the effect of various process parameters on fibre diameter and its uniformity obtained from both rotor structures was also examined. Compared to fibres produced using a triangular groove structure rotor, fibres produced by a rectangular structure rotor presented better morphology and overall characteristics.

## MATERIALS AND METHODS

### Materials and rotor structures

Polyethylene oxide (PEO) Mw = 100000, was received from Shanghai Macklin Biochemical Co Limited. The metering pump was attained from Shanghai Angel Electronics Co Limited and the DC generator was acquired from the Scholl-run factory of Fudan high school. The solutions for the centrifugal electrospinning experiment were prepared using polyethylene oxide (PEO). PEO powder was dissolved into distilled water in a beaker and stirred with the help of a magnetic stirrer at 80°C. It was done in 4 hours for making spinning solution with the different concentrations i.e., 6 wt. %, 7 wt. %, and 8 wt. %. Figure 1 has shown the two types of rotors i.e., rectangular groove structure (conventional method) and triangular groove structure (proposed method). The diameter and uniformity of PEO nanofibers attained from both rotors were compared and analysed. Table 1 shows the fundamental scheme of producing PEO nanofibers, i.e., four process variables with three levels.

Table 1

PRODUCTION SCHEME OF PEO NANOFIBRES				
Level	Solution concentration (%) (A)	Additional voltage (kV) (B)	Rotor speed (R/min) (C)	Spinning fluid flow rate (ml/h) (D)
1	6	45	4600	45
2	7	50	5500	55
3	8	55	6400	65

### Sample preparation

The dissolved PEO solution was prepared and sucked through the vessel meter. The flow rate (ml/h) of the solution was kept constant with the metering pump. The motor rotated the spinning rotor smoothly. The liquid dropped down under the action of centrifugal force and electric field. The development of the jet was collected on the circular collector and finally, the

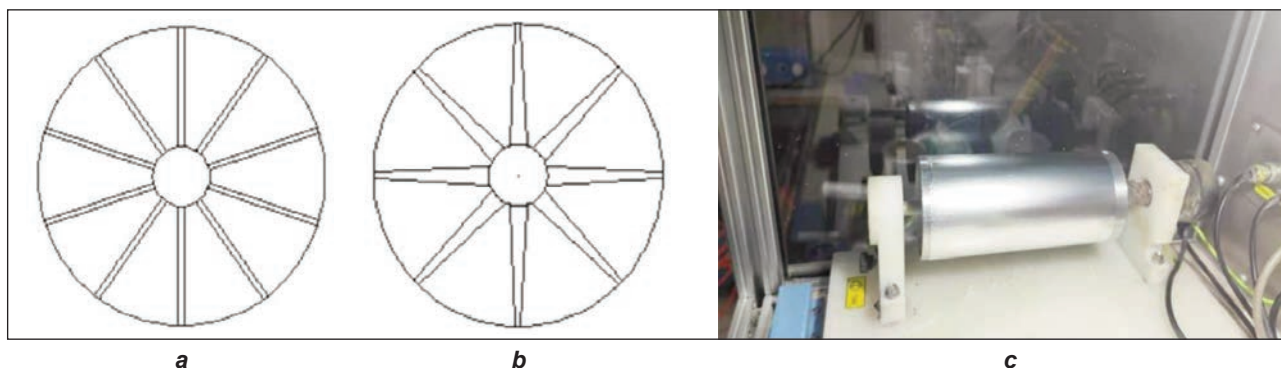


Fig. 1. Two types of rotor structures:  
a – rectangular groove rotor; b – triangular groove rotor; c – rotor adjustment machine

continuous PEO nanofibers were obtained. The receiving distance (between the needle and the circular collector) was fixed at 30 cm. The factors i.e., the spinning solution concentration, rotor speed, liquid flow, and electric voltage, were examined. The fabricated material (samples) was acquired on the aluminium foil. The whole spinning process was completed in 1 hour.

### Working principle

The centrifugal electrospinning system (CES) used was simple and capable of excluding the limitations of the electrospinning (ES) process. All through the CES process, a polymer spinning solution was fed to the nozzle, which was controlled by the metering pump. The polymer solution was dropped down in the centre of the spinning rotor. When the rotational speed of the rotor reached a critical value, the centrifugal force has thrown out the spinning solution from the rotor. The spinning solution received a stretch due to the strong electric field and deposited on the collector forming dried nanofibers (shown in figure 2).

Additionally, the high rotational speed indorsed fast and accessible fibre fabrication, improving the production rate by two to three orders of magnitude. It reduced the production cost in association with the electrospinning process. Otherwise, the centrifugal electrospinning process enables the fabrication of nanofibers from polymer solutions with much higher concentrations than the needle electrospinning process, which also reduces the production cost by using less solvent. PEO is a man-made polyether that

is easily accessible in a range of different molecular weights. Water solubility is the premium property of these polymers. Higher molecular weight PEOs (white and waxy solids) have melting points relative to their molecular weights to an upper limit of about 67°C [45]. While low molecular weight PEOs are colourless and viscous liquids. According to the standard of food and drug administration (FDA), PEO is nontoxic and could be used as excipients in various foods, cosmetics and pharmaceutical formulations [46].

### Characterization

The morphology of nanofibers was characterized using FE-SEM (Scanning electron microscope) instrument. All the samples were coated with gold before the observations. 100 different fibre points of nanofibers were randomly selected from the FESEM images. The diameter of the selected nanofibers was

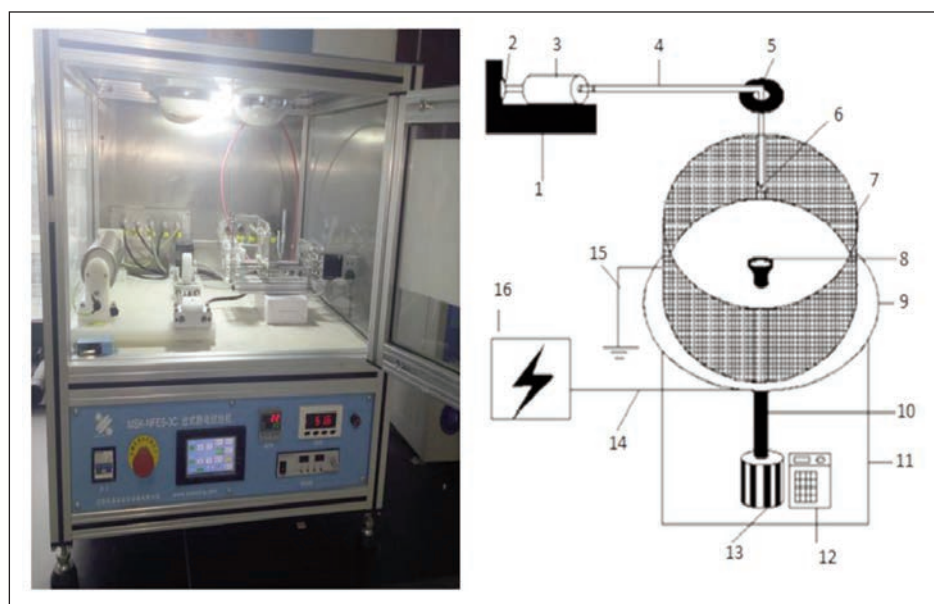


Fig. 2. Structure of centrifugal electrostatic spinning device: 1 – metering pump; 2 – piston; 3 – spinning liquid reservoir; 4 – wire liquid conveying hose; 5 – pipe support plate; 6 – flow plastic tube; 7 – receiving device; 8 – spinning rotor cup placement slot; 9 – insulating support plate; 10 – rotating spindle; 11 – case; 12 – inverter; 13 – drive motor; 14 – positive power cord; 15 – negative power cord; 16 – high voltage electrostatic generator



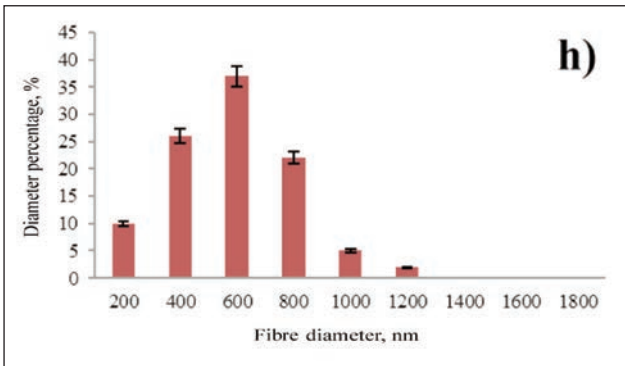
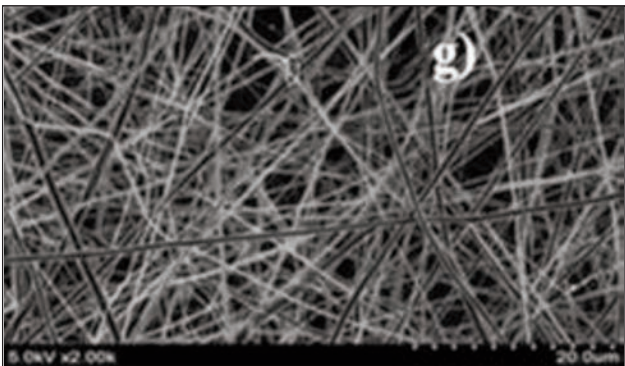
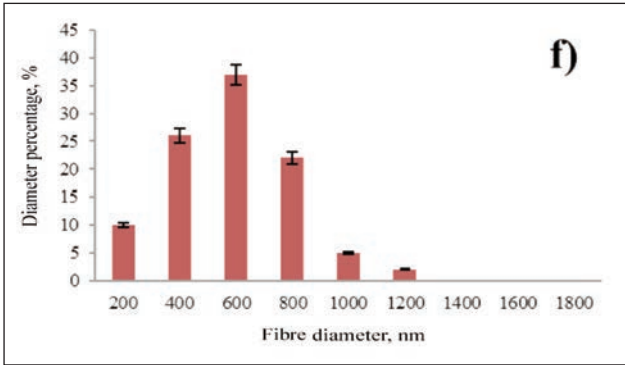
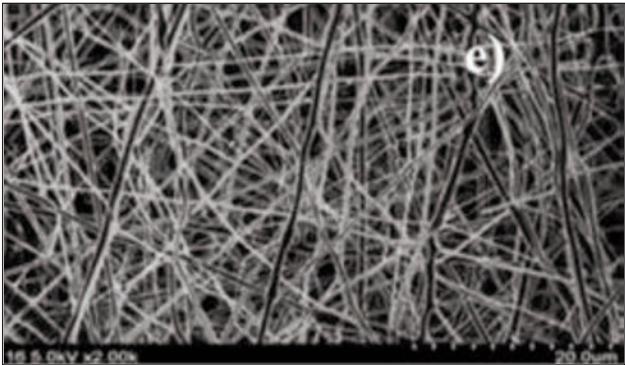
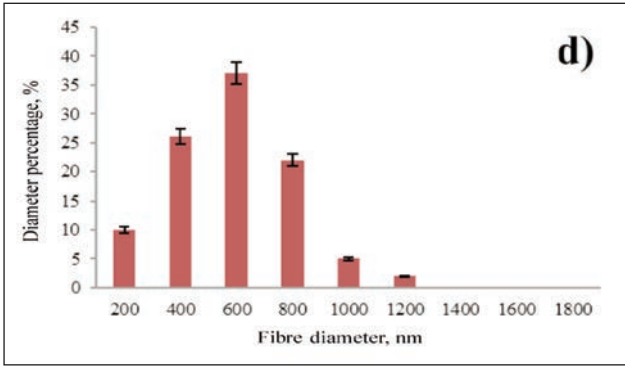
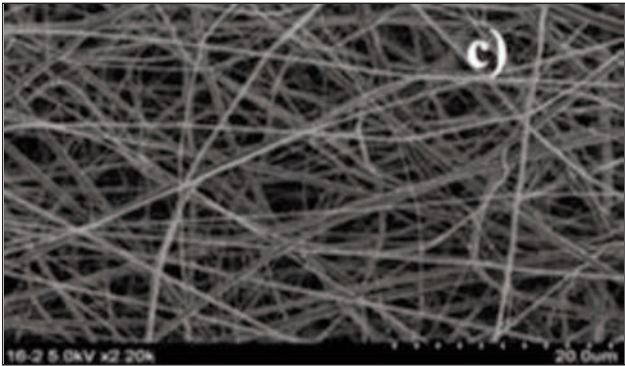
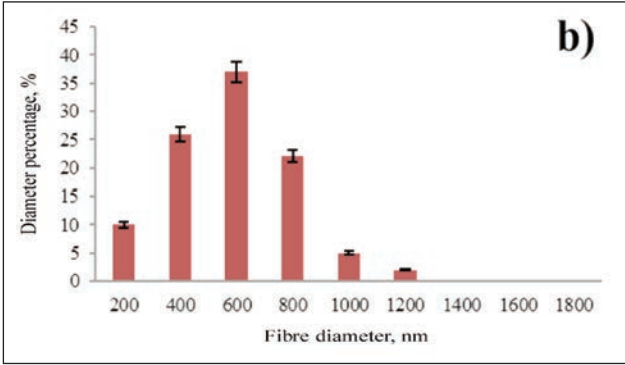
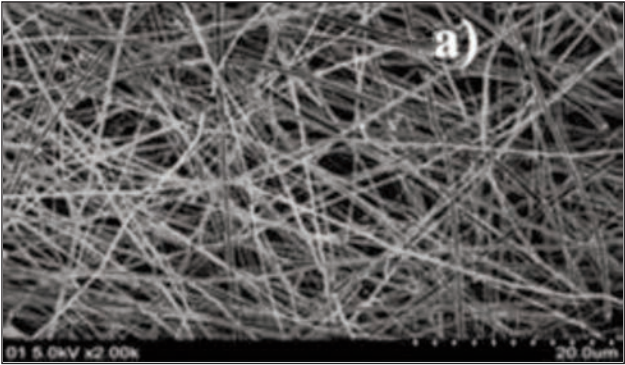
measured through the nano measure software, used for the collection of SEM images data.

RESULTS AND DISCUSSIONS

Influence of spinning solution concentration

At a concentration of 6%, it was found a large number of droplets and a small number of beads in the fibre resulted in non-continuous fibre. With concentration (7%), there was a reduction in the number of droplets and beads which resulted in a long, continuous fibre with an improvement in fibre morphology.

With the solution concentration of 8%, the resultant fibres were developed into a more fine and continuous form. It was found that fibre diameter increased with the increase of solution concentration. The reason is that the higher the solution concentration, the higher the viscosity and the surface tension. Thus, the formed fibres were thicker due to insufficient chain stretch. Figure 3 shows SEM images and corresponding fibre distributions of PEO nanofibers with different solution concentrations, i.e., 6 wt. % to 8 wt. %.



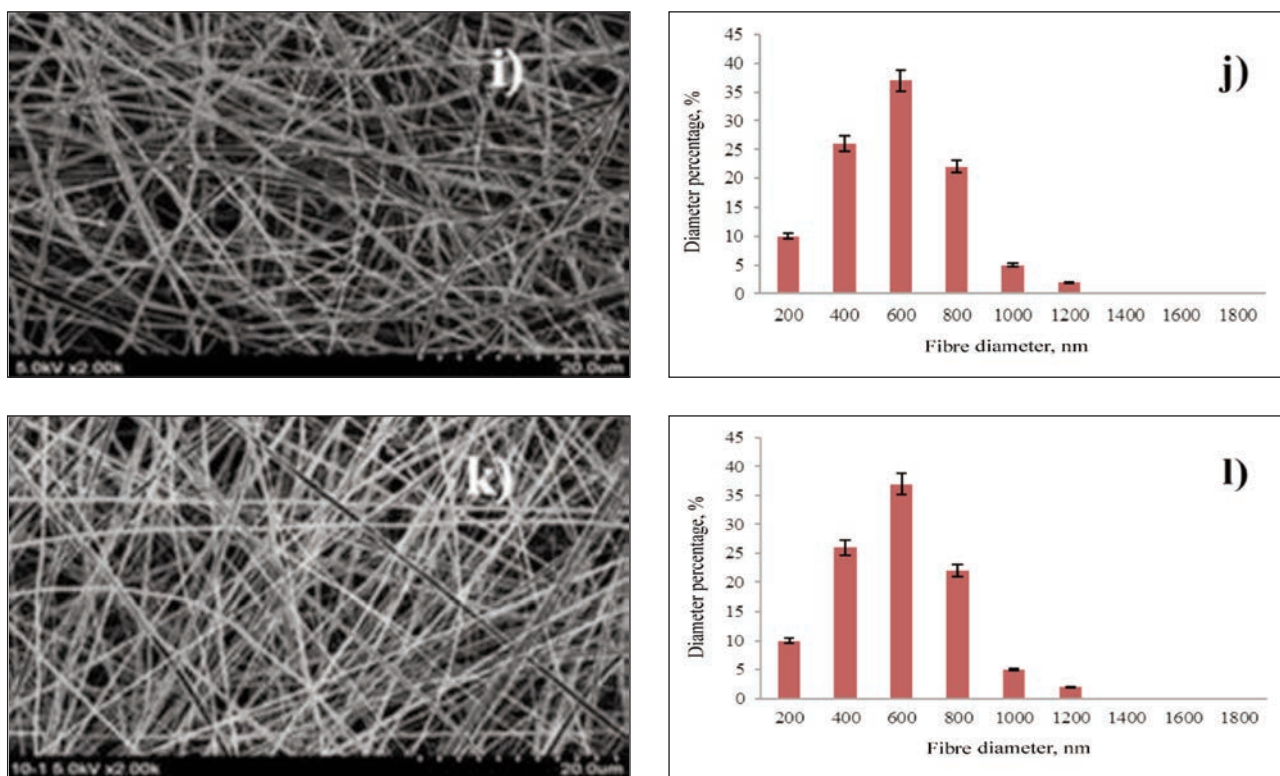


Fig. 3. SEM images and diameter of PEO nanofibers at 4600 rpm centrifugal electro spun with different solution concentrations (6 wt %, 7 wt %, and 8 wt %) using 45 kV electric voltage and 45 ml/h liquid flow rate, through the triangular rotor

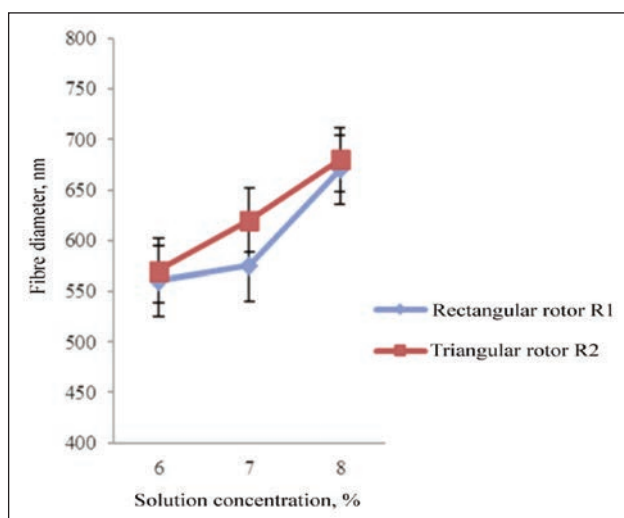


Fig. 4. Influence of solution concentration and fibre diameter

Figure 4 has shown that with the increase in solution concentration the diameter of spun fibres increased. It meant that once the solution concentration reached a high range, for example at 8% or more, the highly-viscous solution produces the fibre with a thicker diameter. It was due to the higher surface tension which resisted the elongation resulting in an insufficient drawing under a certain centrifugal force and electric field strength.

#### Influence of electric field

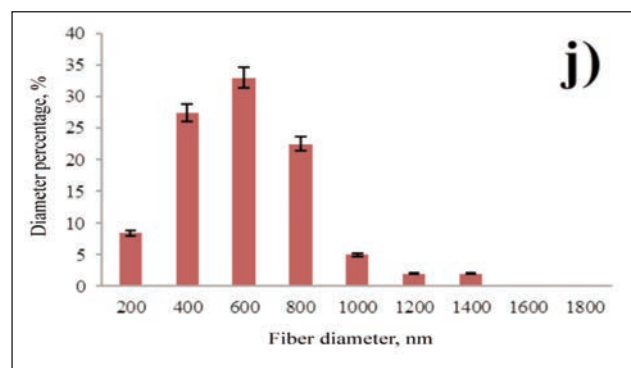
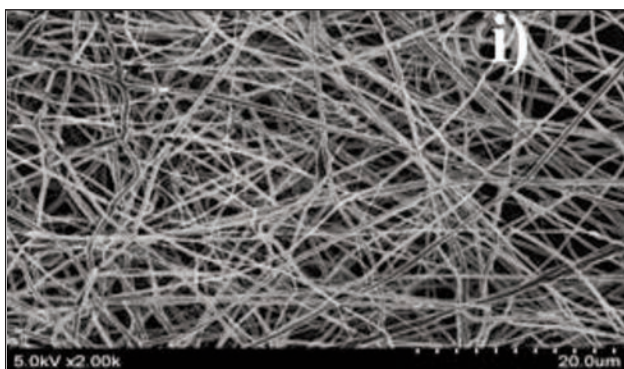
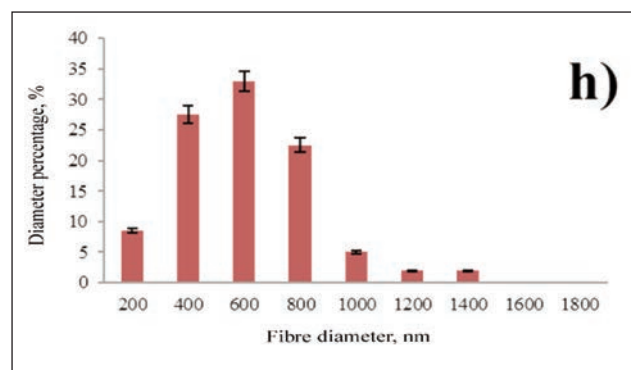
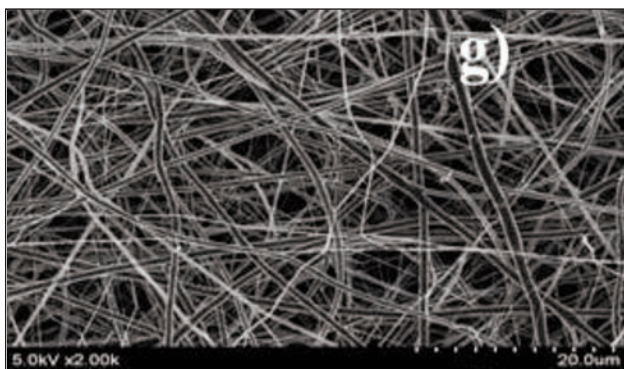
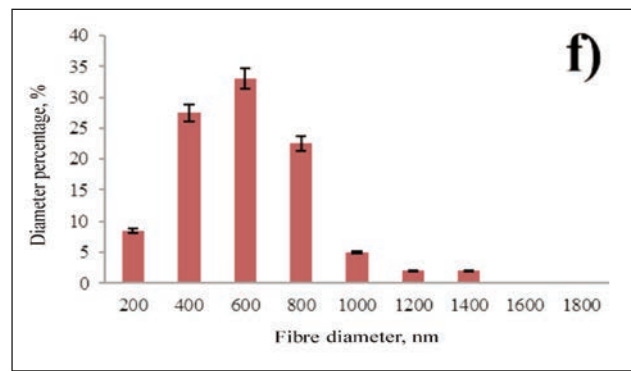
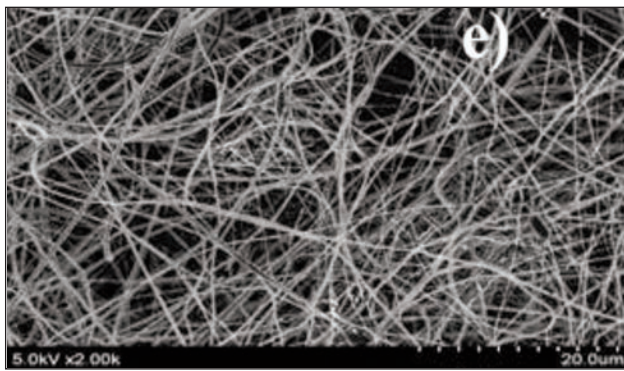
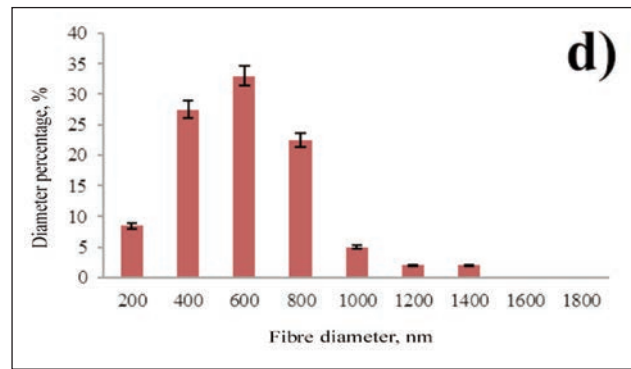
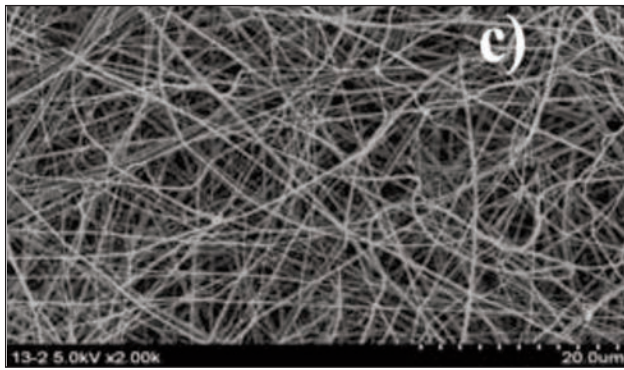
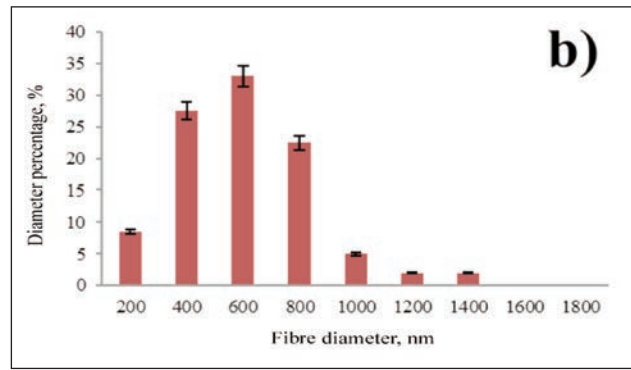
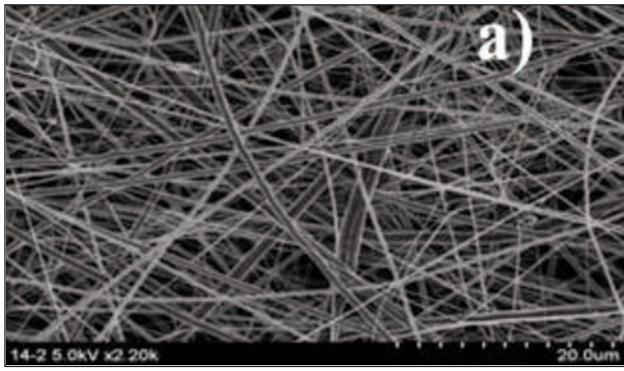
For the effect of the electrostatic force on fibre morphology, PEO nanofibers of varying voltage were

fabricated at constant operational conditions of rotational speed, liquid flow rate, and solution concentration. Figure 5 (R1) and (R2) show the SEM images and corresponding fibre diameter distributions of PEO nanofibers under different electric voltages, i.e., from 45 kV to 55 kV. Moreover, at a very high voltage, the formation of the fibres was fine but uneven (not uniform).

When the applied voltage was lower than 45 kV, the fibre has a relatively larger diameter. There were also many beads or small droplets on the surface of the fibre that could not form continuous fibres. Thus, with less voltage, an electric field produced was not enough to overcome the surface tension of the small droplet and make it difficult to form the fibre jet. The small droplets were normally not drawn and the discharged fibres were not sufficiently stretched. Therefore, they become uneven. The small droplets on the surface of the fibre disappeared. The beading was hardly seen. However, there were some thick segments, the fibre diameter was still coarse. With the increase of the voltage (55 kV), the beads completely disappeared and the droplets turned into the fibre surface liquid. The resultant fibres were thin and uniform; however, there were still obvious thick joints even with the appropriate applied voltage and the electric field.

When the voltage was raised to 65 kV, the fibre-forming effect was relatively good. It completely overcome the surface tension of the droplets and made them fully stretched. The dimensions were thin with non-uniformity in the thickness. There was also a certain







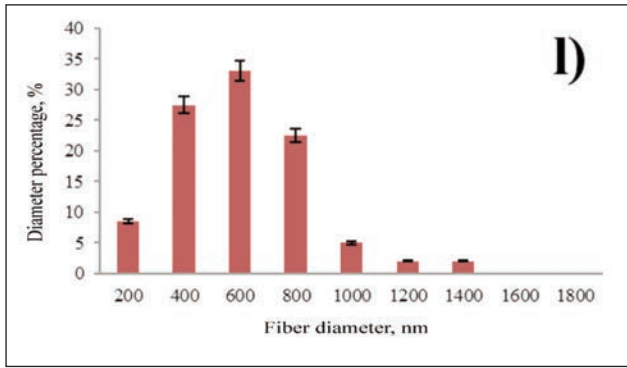
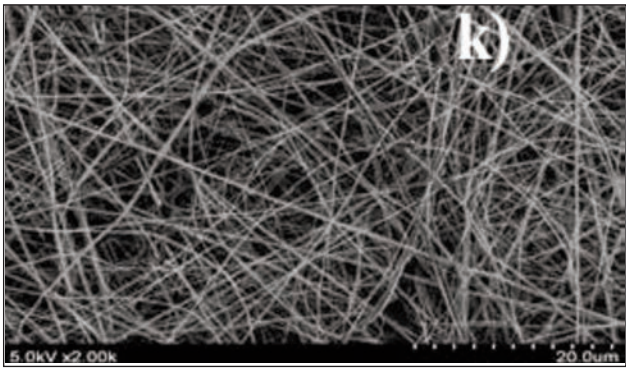


Fig. 5. SEM images and diameter of PEO nanofibers at 4600 rpm centrifugal electro spun with different electric voltage (45 kV, 55 kV, 65 kV) using 6% solution concentration and 45 ml/h liquid flow rate: *a* to *f* – PEO nanofibers through rectangular rotor structure (R1); *g* to *l* – PEO nanofibers through triangular rotor structure (R2)

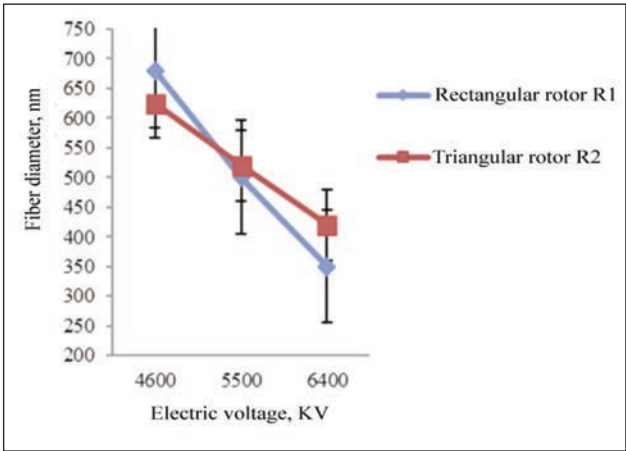


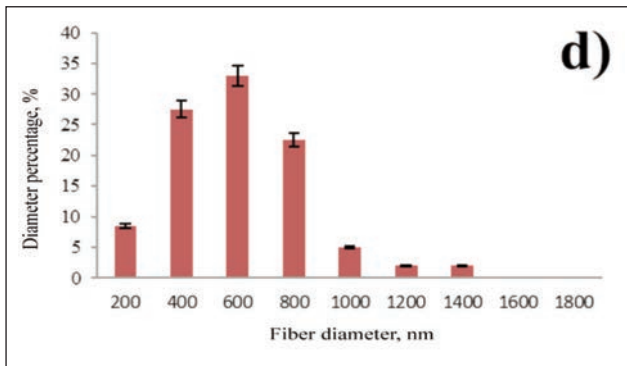
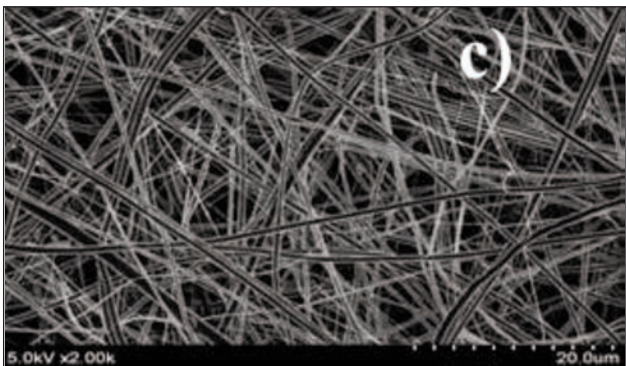
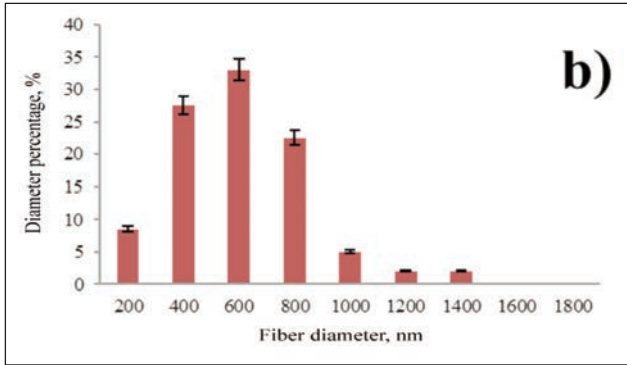
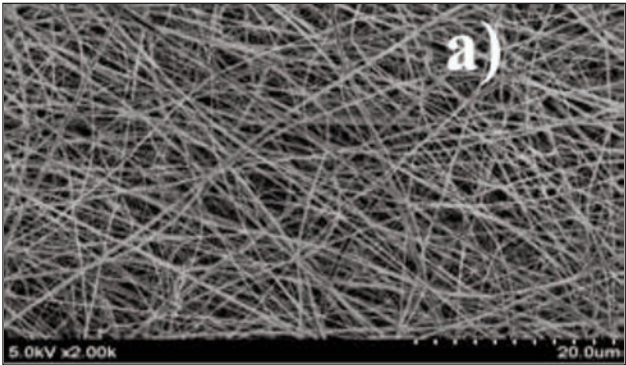
Fig. 6. Influence of electric voltage and fibre diameter

degree of adhesion between the fibres. This was due to the increase of the voltage that caused the acceleration to the surface of the droplet for transforma-

tion. Too much-drawing causes irregularity since it seldom provides enough time for the solvent to be volatile and fibre quickly received at the receiving plate. Figure 6 has shown that the diameter decreases with the increase of electric voltage with both rotor structures. It was observed that the increase in electric voltage decreases the diameter. Furthermore, with the increase in electric field strength, the dropped liquid has a greater surface charge density. Thus, it is subjected to a larger electrostatic repulsion. At the same time, the higher electric field strength accelerated the dropped liquid. These two factors have generated a greater drawing force and resulted in a strong drawing under a certain centrifugal force and electric field strength.

#### Influence of rotational speed of the rotor

Figure 7 (R1) and (R2) has shown the SEM images and corresponding fibre diameter distributions of PEO nanofibers with different rotational speeds i.e.,



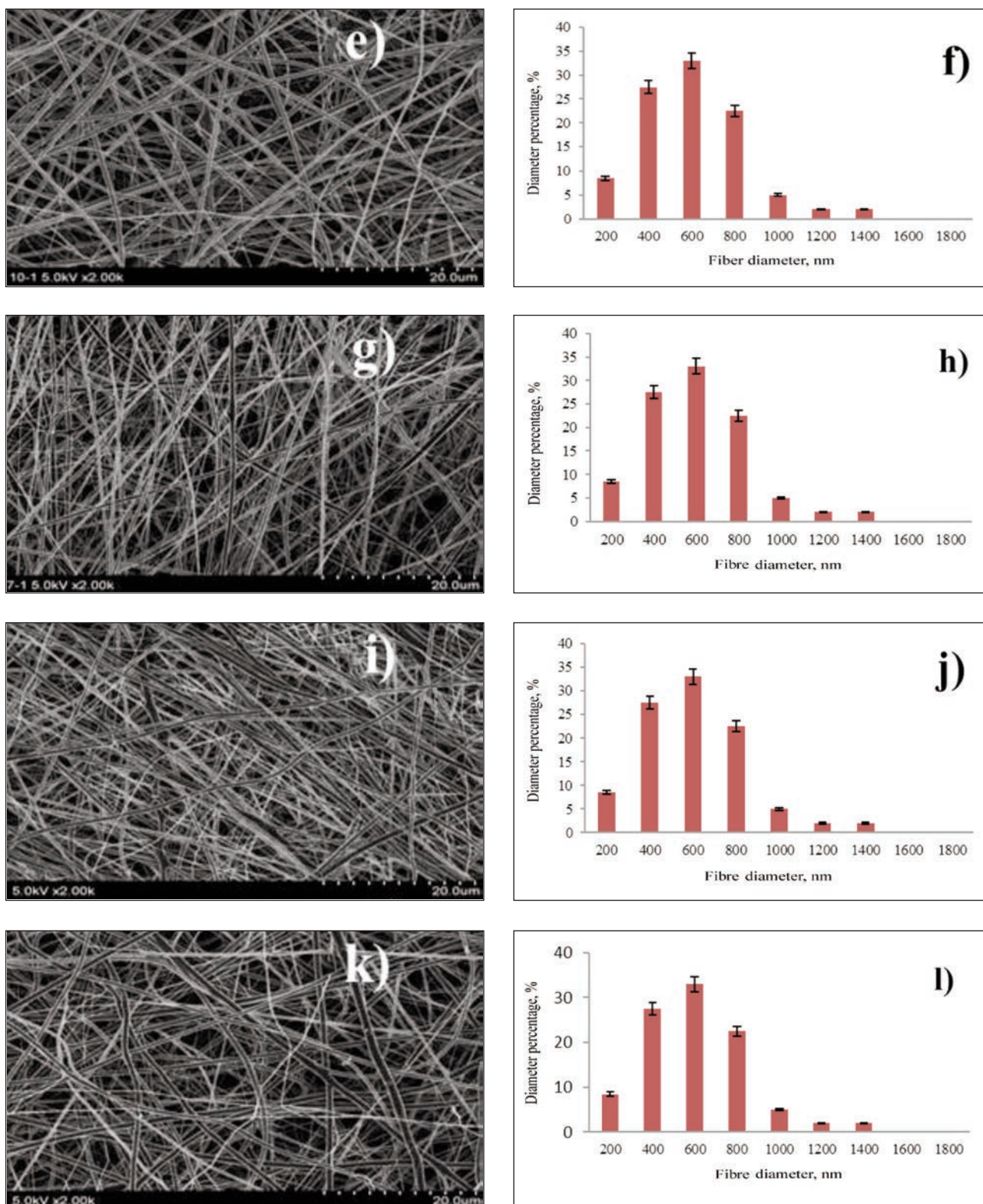


Fig. 7. SEM images and diameter of PEO nanofibers centrifugal electro spun at different rotational speeds (4600 rpm, 5500 rpm and 6400 rpm) with 45 ml/h using 6% solution concentration and 45kV electric voltage: a to f – PEO nanofibers through rectangular rotor structure (R1); g to l – PEO nanofibers through triangular rotor structure (R2)

from 4600 rpm to 6400 rpm. However, a very higher rotational speed caused the formation of more beads, non-uniformity and weak fibres. When the rotor speed was lower than 4600 rpm, the fibre fineness was improved to some extent, however, it was not uniform, and several fibres were thicker. With the increase in rotor speed i.e., increased to 5500 rpm, the fibre was thinner and more uniform. Thus, fibre

has the best effect. When the rotor speed was increased further, the fibre was finer but uneven. This was because the spinning rotor rotates at a slow speed and the small droplets received a little force while throwing/dropping out in the spinning rotor. The number of droplets dropped was smaller. The droplet area was large, and the electrical energy required generated the jet, increasing fibre diameter. When



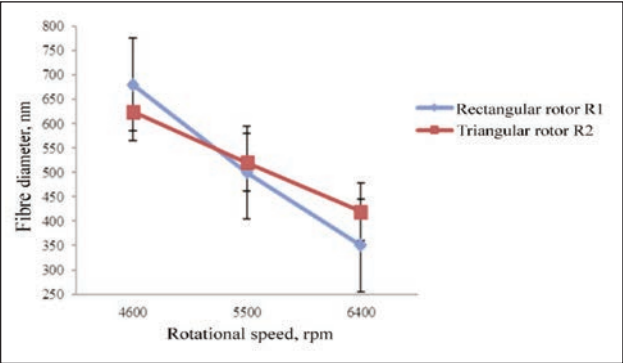


Fig. 8. Influence of rotational speed and fibre diameter

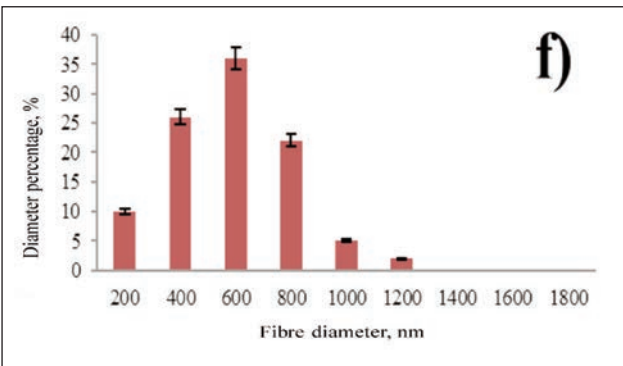
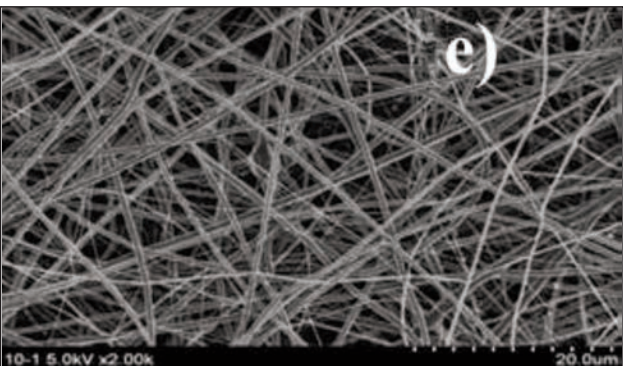
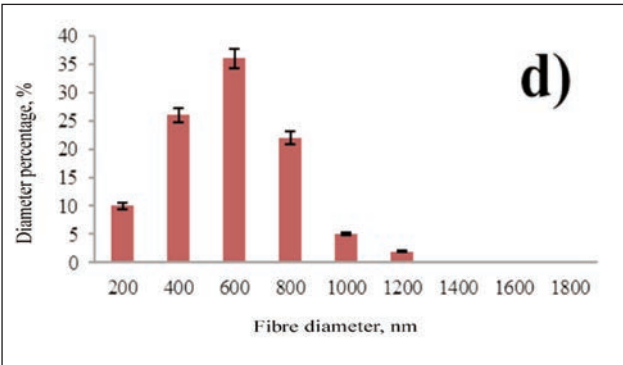
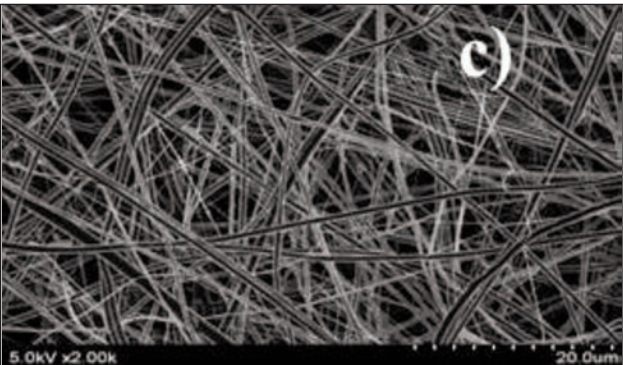
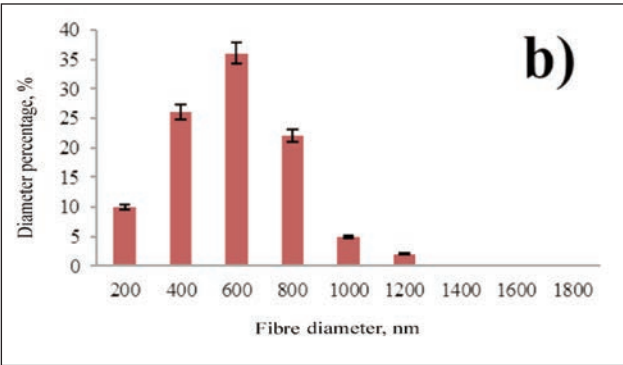
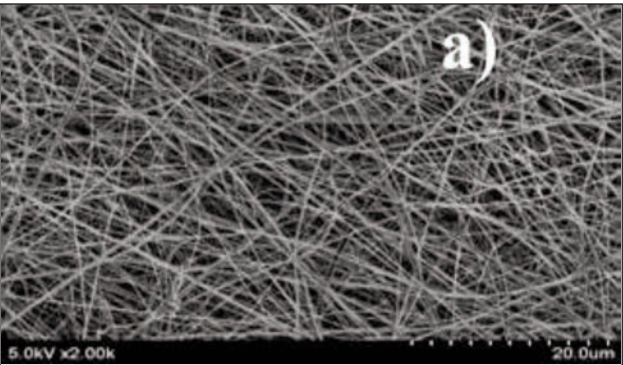
the spinning rotor rotated at a high speed, the centrifugal force of the dropped droplets (small) was greater. Moreover, the falling of several small droplets on the spinning rotor resulted in a small droplet surface area. The diameter of the fibre decreased with the increase in the rotational speed of the spinning rotor. When the rotational speed was high enough i.e., reached a critical value, the fibre began to crack, and then the jet became bead-like and the diameter of the

fibre increased. Rotational speed has a critical role in the determination of the liquid dropped in the drawing process. Figure 8 has shown that the diameter of spun fibres decreases with the increase in rotational speed. Thus, with the increase in rotational speed, the centrifugal force increased and the diameter of spun fibres decreased.

#### Influence of liquid flow rate on fibre morphology

Figure 9 (R1) and (R2) has shown the SEM images and corresponding fibre diameter distributions of PEO nanofibers. It was perceived that very higher flow rates of spinning liquid resulted in the formation of more beads and irregularity in the fibres. It was observed that with the increase in liquid flow rate, the diameter of the fibre increased. This was due to the falling of bigger size drop and faster flow rate of spinning. Moreover, it leads to an insufficient drawing under a certain centrifugal force and electric field strength. It was also noticed that the very slow flow rate of spinning liquid, made the falling of smaller droplets on the spinning rotor.

Additionally, the high-speed spinning rotor has not thrown out small liquid drops continuously. An



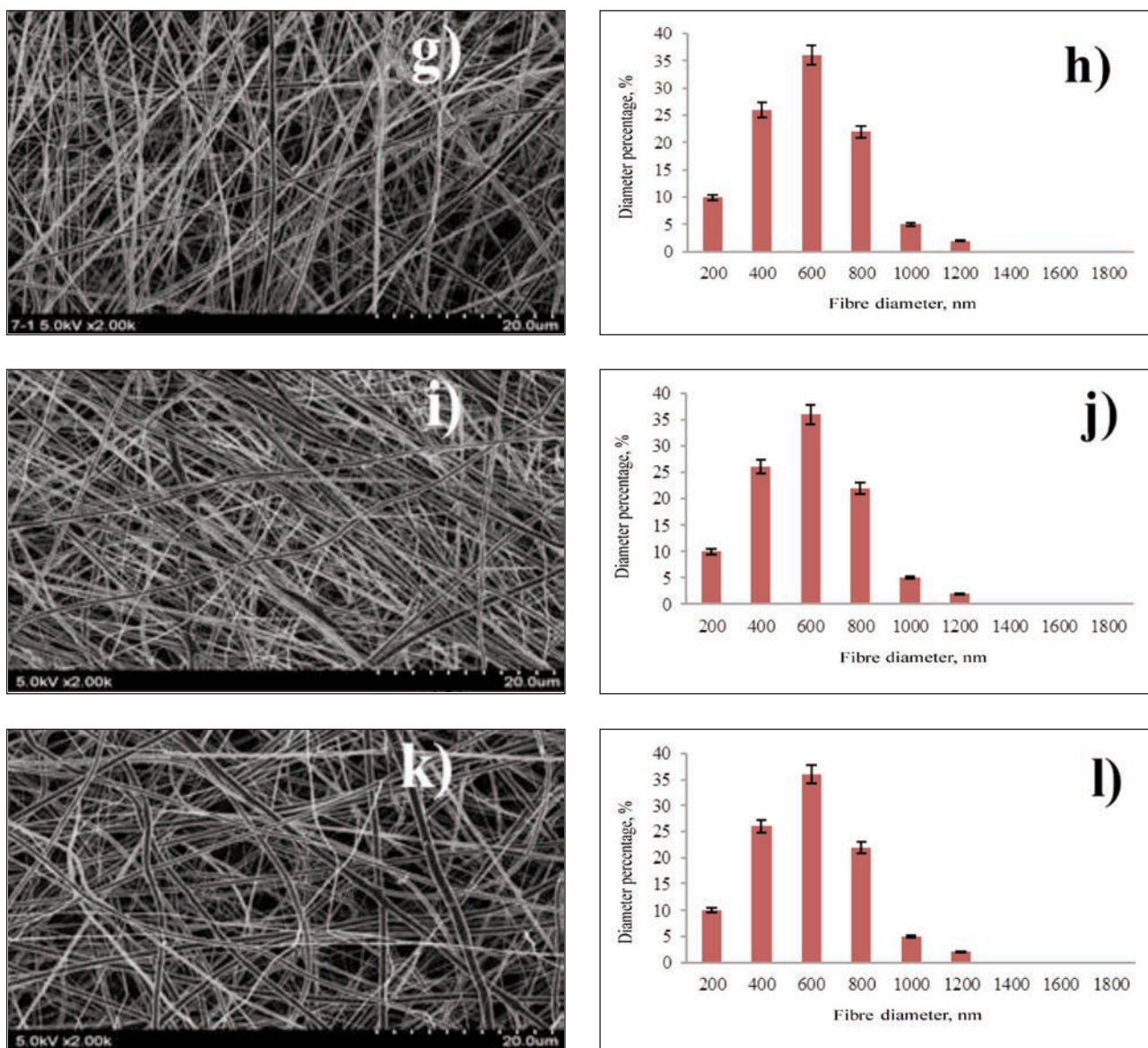


Fig. 9. SEM images and fibre diameter distribution of PEO nano-fibres at 4600 rpm centrifugal electro spun with different liquid flow rates (45 ml/h, 55 ml/h and 65 ml/h) using 6% solution concentration 45 kV electric voltage: a to f – PEO nanofibers through rectangular rotor structure (R1); g to l – PEO nanofibers through triangular rotor structure (R2)

increase (appropriate range) in the rate flow of spinning liquid has filled the groove of the spinning rotor and therefore continuous nanofibers were achieved. In figure 10, it was observed that with the increase in liquid flow rate, the diameter of the fibre also increased. This was due to the falling of bigger size

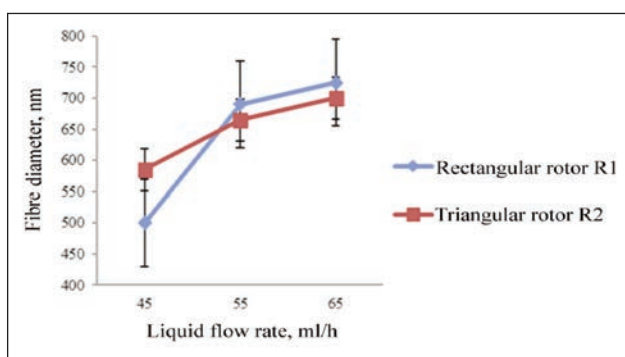


Fig. 10. Influence of liquid flow rate and fibre diameter

drop and its faster flow rate of spinning. Moreover, it led to an insufficient drawing under a certain centrifugal force and electric field strength.

#### Influence of the spinning rotor structure

Figure 11 has shown the SEM images and distribution of fibres prepared from both the rotors i.e., conventional rotor (rectangular rotor structure R1) and newly proposed rotor structure (triangular rotor structure R2). These SEM figures demonstrated that the diameters of PEO nano-fibres spun with triangular groove structure rotor (R2) have finer and more uniform results than the conventional i.e., rectangular groove structure rotor (R1) which has some irregular places in the fibres (deficiency in uniformity and fineness). Thus, the overall result indicated that fibres obtained through a triangular grooved rotor were more even and uniform while the fibres gained through a rectangular grooved structure rotor were less effective.



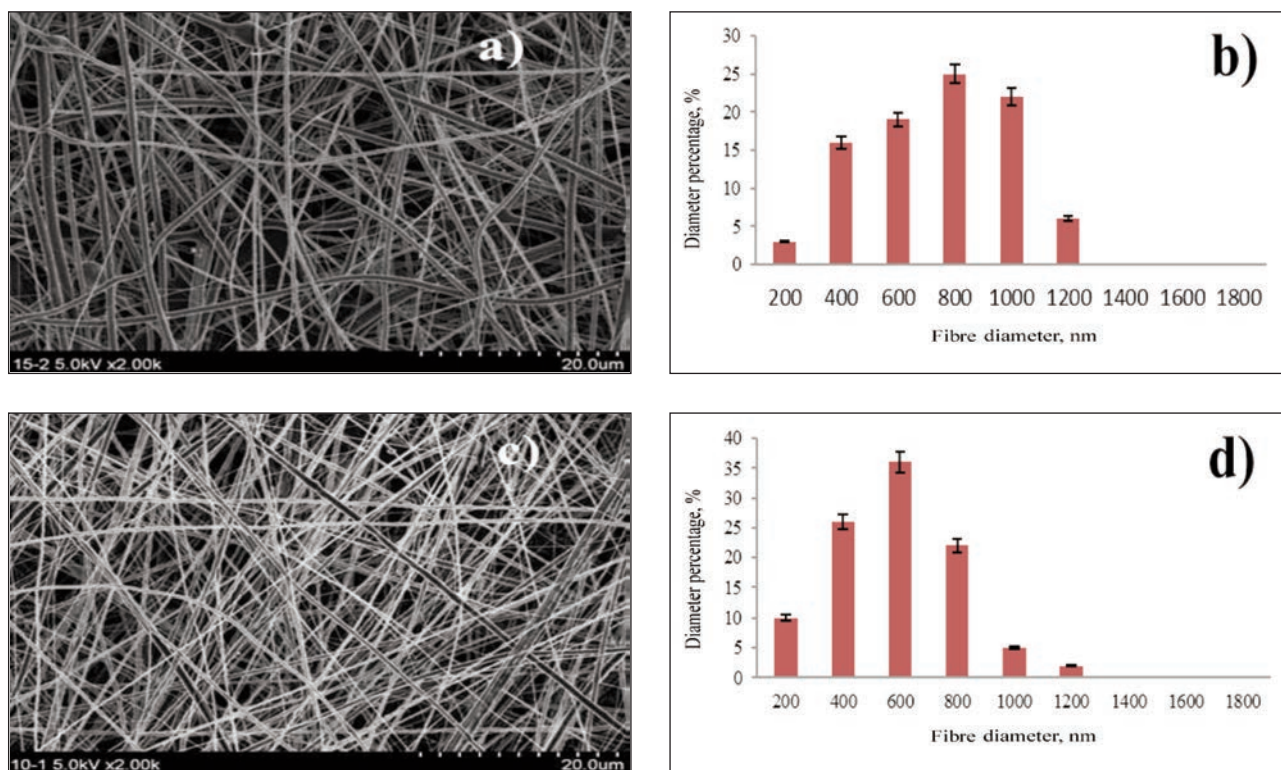


Fig. 11. Comparison of SEM images and effect of two rotor structures: *a* and *b* – effect of rectangular rotor structure (R1); *c* and *d* – effect of triangular rotor structure (R2)

## CONCLUSIONS

In this paper, PEO nanofibers production was investigated and improved through a triangular grooved rotor structure. This is a novel step to develop an adept system for the centrifugal electrospinning (CES) process. The CES technology has low cost and large-scale production of nanofibers. The triangular grooved rotor structure was an effective model in the centrifugal electrospinning process for continuous, fast and high-speed PEO nanofibers. The highest quality of PEO fibres was obtained at 8% solution concentration where the electric voltage of 55 kV,

spinning rotor speed of 5500 rpm and the flow rate of 55 ml/h. The diameter of the produced PEO nanofibers with triangular grooved rotor structure was more even and uniform as compared to the rectangular rotor structure. Hence it is a good choice for more technological advancements in the production of low-cost and large-scale production of nanofibers.

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