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Effect of polymer concentration on electrospinning of hydroxypropyl-β-cyclodextrins/PEO nanofibers

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Abstract Hydroxypropyl-β-cyclodextrin (HP-β-CD) is a β-cyclodextrin (β-CD) derivative which is toxicologically harmless to mammals and other animals. In this study, HP-β-CD is electrospun from an aqueous solution by blending with poly (ethylene oxide) (PEO). The aqueous solutions containing different HP-β-CD/PEO blends (50:50–90:10) with varying concentrations (4–12 wt.%) were electrospun at 1 ml/h feed rate, 12 cm working distance and 7 kV applied voltage. The morphology of the nanofibres was investigated by scanning electron microscope. The average diameter of the nanofibers was measured using ImageJ software. It was found from the results that the uniform nanofibres with an average diameter of 264, 244 and 236 nm were obtained from 8 wt.% solution of 50:50, 60:40 and 70:30 HP-β-CD/PEO blends, respectively. The average diameter of the fibre decreases with increasing HP-β-CD/PEO ratio. However, higher proportion of HP-β-CD (i.e. above 70:30 HP-β-CD/PEO blend) in the spinning solution increases the possibility of creating more beads in the fibres. Although the polymer concentrations have not shown a significant effect on fibre diameter, the 8 wt.% solution of 50:50 HP-β-CD/PEO yielded uniform smooth fibres with the narrowest distribution of the diameters. As the aim of this study is to maximize the HP-β-CD content in the fibre, the blend ratio of 70:30 HP-β-CD/PEO and solution concentration of 8 wt.% were optimized to obtain smooth HP-β-CD/PEO nanofibres.

Keywords: electrospinning; hydroxypropyl-β-cyclodextrin; poly(ethylene oxide); nanofibres.

Introduction

Electrospinning is an effective method for producing nanosized fibres. Many synthetic and natural polymers and their blends have been successfully electrospun to make nanofibres (Huang, Zhang, Kotaki, & Ramakrishna, 2003; Ramakrishna, Fujihara, Teo, Lim, & Ma, 2005). Other methods such as melt blowing, drawing, bicomponent spinning, flash spinning, force spinning, template synthesis, phase separation, selfassembly (Huang et al., 2003; Zhou & Gong, 2008), are not convenient for producing nanoscaled fibres (Ramakrishna et al., 2005). In recent years, electrospinning has found increasing use in different potential applications such as tissue-engineered scaffold, drug delivery, filtration, sensor, protective clothing and wound dressing (Doshi & Reneker, 1995; Dotti et al., 2007; Khil, Cha, Kim, Kim, & Bhattarai, 2003; Lee & Obendorf, 2007; Reneker & Chun, 1996; Srinivasan & Reneker, 1995). The structural properties such as fibre diameter and morphology, and the stability of electrospinning process are dependent on several factors such as solution properties (polymer concentration, solution viscosity, etc.), process parameters (flow rate, applied voltage, working distance, etc.) and ambient conditions (room temperature, humidity, air flow, etc.) (Deitzel, Kleinmeyer, Harris, & Beck Tan, 2001; Doshi & Reneker, 1995; Hekmati, Rashidi, Ghazisaeidi, & Drean, 2013; Zong et al., 2002).

Cyclodextrins (CDs) are water-soluble biocompatible materials, constituted with cyclic oligosaccharides composed of D-glucose units which are joined by α-1,4-glucosidic linkages. These CDs can be easily extracted by enzymatic degradation of maize, rice, corn husk, potatoes, sago and other sources (van de Manakker, Vermonden, van Nostrum, & Hennink, 2009). The most common and popular CDs are αCD, βCD and γCD which have 6, 7 and 8 glucose units respectively, although other varieties exist. The CDs structures are formed with a hydrophobic internal cavity that can accommodate suitably sized, generally low molecular weight lipophilic molecules. It is well tolerated by animal and human body, and shows minor toxicity when dosed orally (Gould & Scott, 2005). These properties have made it a good aqueous carrier for delivering drugs in the biological system (van de Manakker et al., 2009). Moreover, the hydrophobic internal cavities of cyclodextrins can entrap suitably sized low molecular weight volatile organic compounds (VOCs) by forming inclusion complexes. These properties give CDs uniqueness for being used in medicine, and consumer and health care products to control undesirable molecules, gases or smells.

Hydroxypropyl-β-cyclodextrin (HP-β-CD) is a hydroxyalkyl derivative of β-cyclodextrin, synthesized by reacting β-CD with propylene oxide in alkaline aqueous solution. This biodegradable and biocompatible polymer is commonly used as a drug delivery agent in the pharmaceutical industry. However, due to its ability of capturing VOCs, HP-β-CD can also be used in textile fabrics, air filters, nappies and wound dressing materials. Fabrics containing HP-β-CD can be used in window curtains, bed sheets, under garments, personal feminine products and hospital clothes to absorb unwanted smell. Normally, HP-β-CD is applied on fabrics using additional adhesive or resin which may make the fabrics hard and stiff. However, the application of HP-β-CD by electrospinning does not need any adhesive material except water. Thus, HP-β-CD can perform its function

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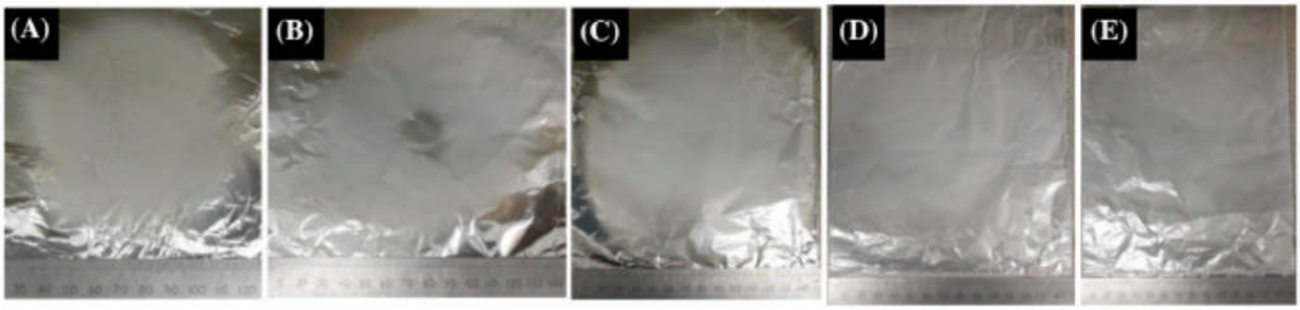

Figure 1. Fibre collection zones produced from 8 wt.% of 50:50 (A); 60:40 (B); 70:30 (C); 80:20 (D); and 90:10 (E) HP-β-CD/PEO blend solutions.

without making the substrate hard or stiff. However, the HP-β-CD is not suitable for electrospinning because of its low molecular weight.

So far, very few studies (Celebioglu & Uyar, 2010; Uyar & Besenbacher, 2009; Uyar et al., 2009) have been carried out to investigate the electrospinning of cyclodextrins and their derivatives. Uyar and Besenbacher (2009) have successfully electrospun the cyclodextrin-functionalized PEO nanofibres and yielded smooth fibres from 4% (w/v) PEO containing 50% w/w cyclodextrins (respect to weight of PEO). Solubility of HP-β-CD is higher than other CDs such as α-CD, β-CD and γ-CD (Szente & Szejtli, 1999), which aids in the mixing with PEO to make a homogeneous solution for electrospinning. In this study, 2-hydroxypropyl-β-cyclodextrin (HP-β-CD) is used as the main polymer and PEO is used as a carrying polymer for electrospinning.

Experimental

Materials

2-hydroxypropyl-β-cyclodextrin (Mw = 1460; brand name: CAVSOL®W7 HP) and poly(ethylene oxide) (PEO)(Mw = 900,000) were purchased from WACKER Chemie AG and Sigma-Aldrich respectively, and used without further modification.

Preparation of HP-β-CD/PEO spinning solution

Predetermined amounts of 2-HP-β-CD and PEO powder were dissolved in distilled water in a sealed glass bottle. Then the sealed bottle was transferred to a magnetic stirrer and the solution was gently stirred at room temperature for 24 h to obtain a homogeneous spinning solution. To investigate the effect of blend ratios and solution concentrations on electrospinning stability and the morphological structure of the electrospun HP-β-CD/PEO fibres 50:50, 60:40, 70:30, 80:20, and 90:10 blends of 8 wt.% and 50:50 blend of 4–12wt.%HP-β-CD/PEO solutions were prepared.

Instrument set-up and electrospinning process

The electrospinning device consists of a pump, a high-voltage power supply, a grounded metal collector and a syringe with a blunt-tip needle. The syringe, filled with a specific amount of spinning solution, was fixed to the pump. The collector was placed opposite of the syringe needle 12 cm from the needle tip. The electrospinning process parameters were adjusted at 1 ml/h feed rate, 12 cm working distance and 7 kV applied voltage. The electrospun fibres were collected onto an aluminium foil. The fibres on the foil were dried for 24 h at room temperature to remove the residual solvents.

Characterization of the electrospun fibres

The morphology and structure of the electrospun nanofibres were observed by a field gun emission scanning electron microscope (SEM) (PHILIPS XL30 FEG-SEM). The electrospun fibre mesh on the aluminium foil was cut to about 0.5 cm×0.5 cm size and attached on a specimen stub using carbon tape. Then these samples were sputter-coated with carbon using Gatan Precision Etching Coating System (model: 682). The images were taken at 2000×, 10,000× and 20,000× magnifications. The SEM operating parameters were set at an accelerating voltage of 6 kV and spot size 3. The fibre diameters were manually measured using the line drawing feature of ImageJ (ImageJ, 2004) software from 50 randomly selected fibres from the 10,000× and 20,000× magnification images. However, the diameters of the nanofibre webs were measured by ImageJ software from the two selected samples, and taken three measurements from each sample.

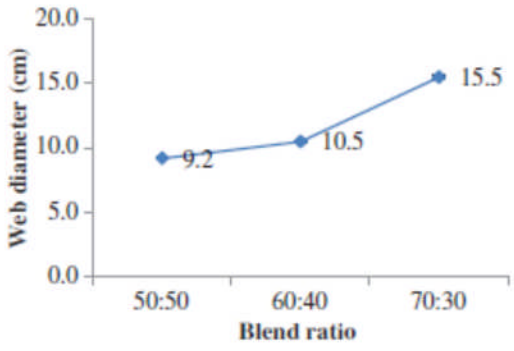

Figure 2. Blend ratio of HP-β-CD/PEO vs. the diameters of the nanofibre webs.

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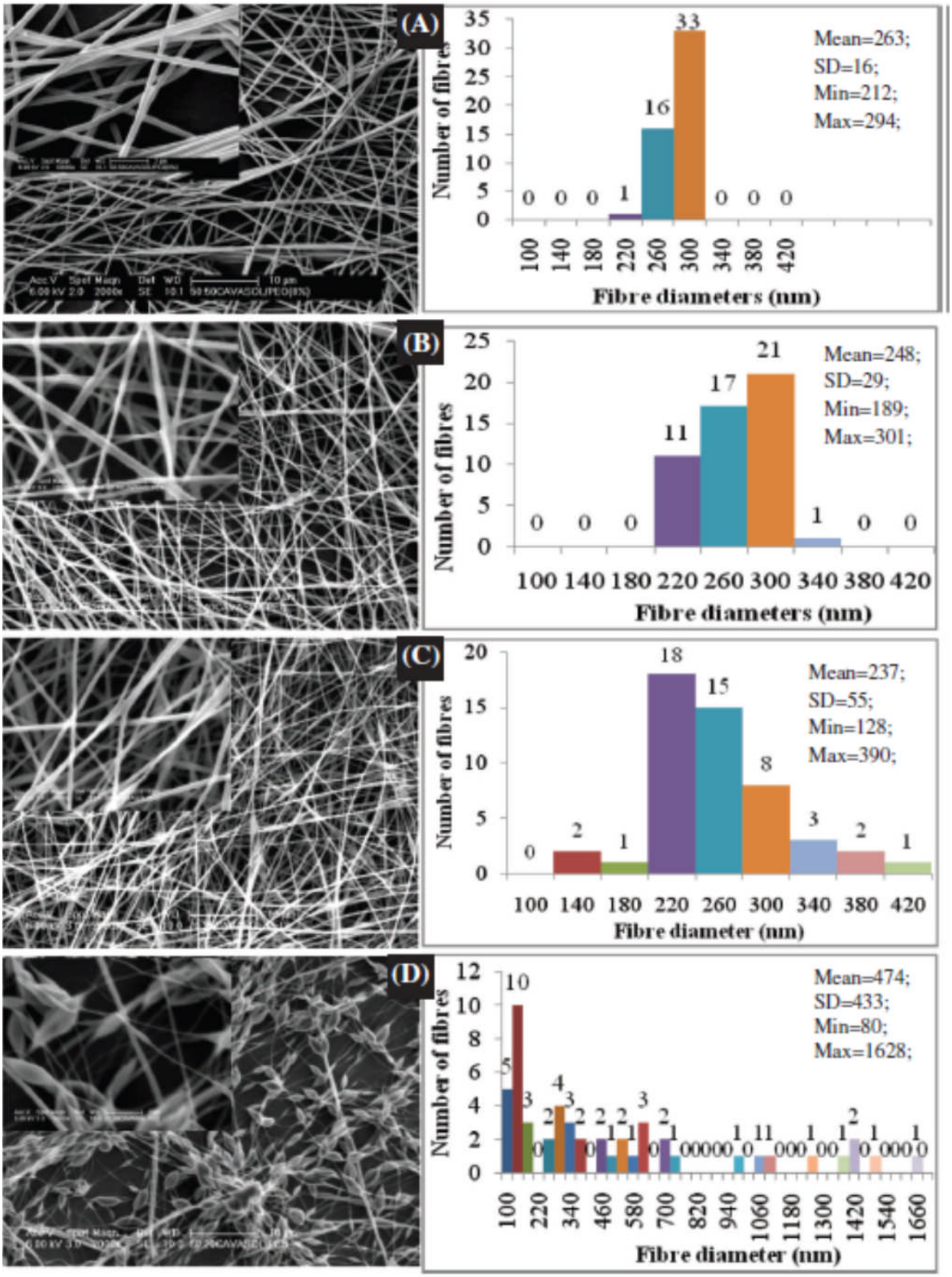

Figure 3. SEM images 2000×(inset 10,000×) and fibre size distribution of 8 wt.% of 50:50 (A), 60:40 (B), 70:30 (C), 80:20 (D) HP-β-CD/PEO electrospun-nanofibres.

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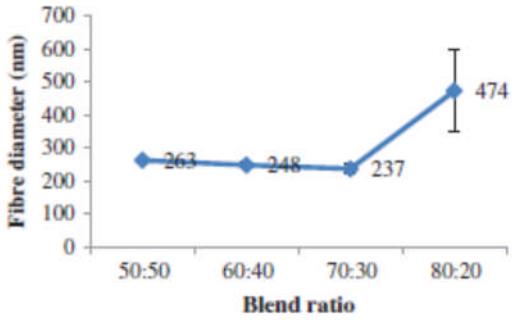

Figure 4. The fibre diameter vs. blend ratio.

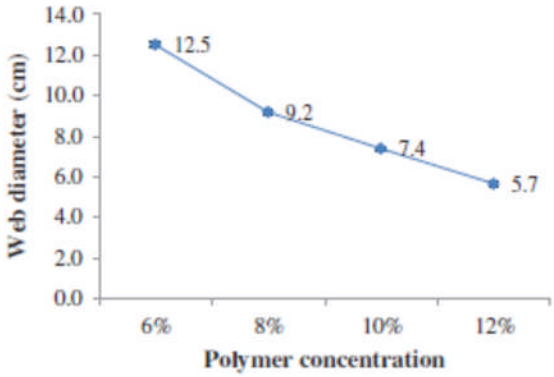

Figure 6. concentration vs. diameter of the nanofiber webs.

Results and discussion

Effect of HP-β-CD/PEO blend ratio

Effect of HP-β-CD/PEO blend ratio on electrospinning process

In general, the HP-β-CD is not electrospinnable due to its low molecular weight; thus, PEO is used as a carrying agent to make HP-β-CD nanofibres. In this study, a series of HP-β-CD/PEO solutions with varying blend ratios, ranging from 50:50–90:10 have been electrospun at 1 ml/h feed rate, 7 kV applied voltage and 12 cm working distance to investigate the effect of blend ratios on electrospinning. Figure 1 shows the effect of different blend ratios of HP-β-CD/PEO with a 8 wt.% polymer concentration on the fibre collection zone (nanofibre web). It was observed that the 50:50, 60:40 and 70:30 polymer blends produced controlled fibre jets that were deposited on the collector making a circular pattern. This indicates the stability of the electrospinning process. However, the 80:20 and 90:10 polymer blends dispersed the fibres over a large area of the collector, even beyond the collector without making a clear circular fibre collection zone. This may have happened due to higher content of HP-β-CD at higher solution concentration, in which a molecular aggregation is occurred. This may lead to an incomplete stretching of solution jet during electrospinning, which was seen in the previous study (Uyar, Kingshott, & Besenbacher, 2008). A similar effect was also observed in the solution of 50:50 blend of 4 wt.% polymer concentration. This may have happened due to insufficient amount of polymer in the solution, which results in sufficient viscosity and molecular chain entanglement. In both of these cases, an electrospraying HP-β-CD/PEO may have taken place instead of electrospinning. Moreover, the presence of low-molecular weight HP-β-CD in the solution reduces the viscosity, which influences the electrified solution jet to spread over a large area. However, as shown in Figure 2, the diameter of the fibre webs increases with increasing of HP-β-CD content in the solution. The results revealed that the diameters of the nanofibre webs were found to be 9.2, 10.5 and 15.5 cm for the solutions of 50:50, 60:40 and 70:30 blend ratios, respectively.

Effect of blend ratio on fibre morphology

Previous studies (Desai & Kit, 2008; Kim et al., 2005; Uyar & Besenbacher, 2009) have shown the effect of mass ratio on fibre diameter and morphologies of the electrospun fibres. Figure 3 presents the SEM images and the corresponding fibre size distribution of the electrospun nanofibres obtained from the 8 wt.% of 50:50, 60:40, 70:30 and 80:20 HP-β-CD/PEO blend solutions. The results revealed that the uniform fibres with an average diameter of 263, 248, 237 and 474 nm were yielded from 50:50, 60:40, 70:30 and 80:20 blend solutions, respectively. As Figure 4 shows, there is a clear trend of decreasing the diameter of the fibres and widening the size distribution with increasing blend ratios. The 50:50 and 60:40 blends produce smooth fibres and the 70:30 blend produces smooth fibres compromising some spindle-like beads. However, the 80:20 blend produces beaded fibres and formed pearl-on-string structure. The ANOVA test found F (F = 13.217) is greater than F crit (F crit = 2.650), which indicates that the data series of diameters obtained from different blend ratios are not equal and they have significant difference.

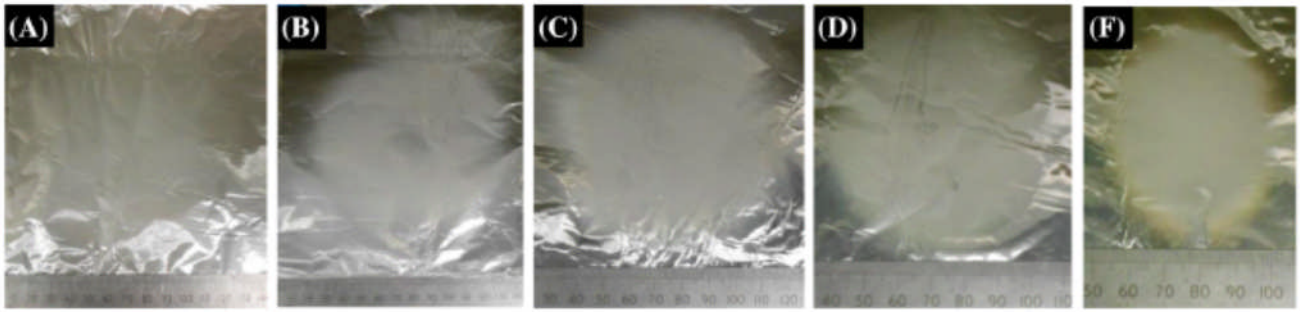

Figure 5. Nanofibre webs produced from 50:50 HP-β-CD/PEO of 4 wt.% (A); 6 wt.% (B); 8 wt.% (C); 10 wt.% (D); and 12 wt.% (E) solution concentration.

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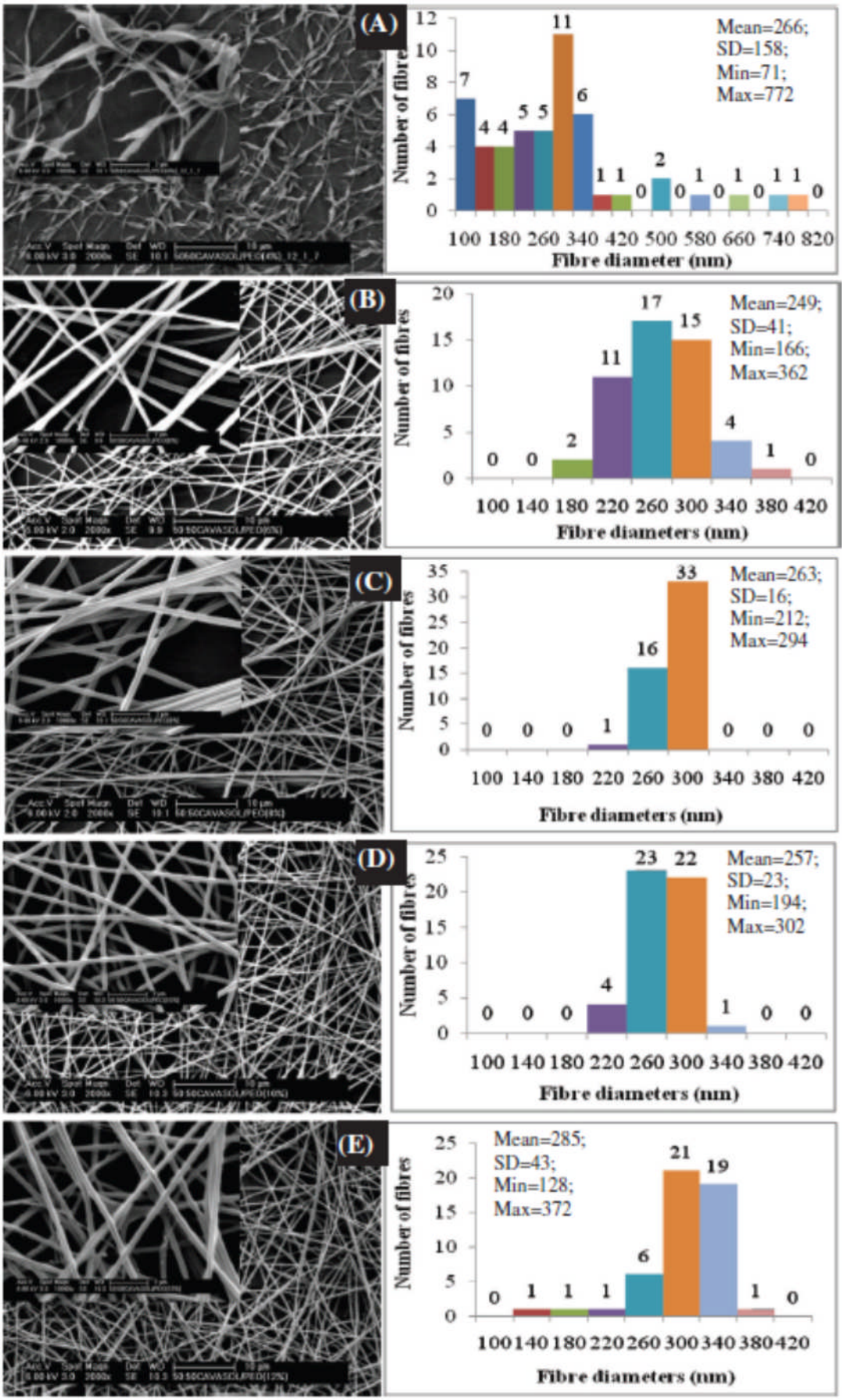

Figure 7. SEM images 2000×(inset 10,000×) and fibre size distribution of electrospun-nanofibres from 50:50 HP-β-CD/PEO of 4 wt.% (A); 6 wt.% (B); 8 wt.% (C); 10 wt.% (D); 12 wt.% (E) solution concentrations.

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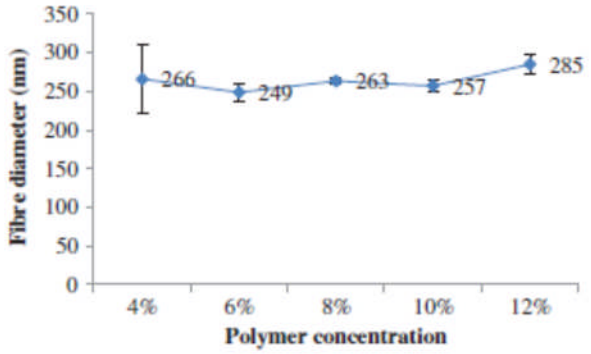

Figure 8. Solution concentration vs. fibre diameter.

These results reveal that the higher content of low molecular weight HP-β-CD in the solution of higher blend ratios cause lower viscosity. Lower viscosities impede polymer chain entanglement and reduce viscoelastic force. Thus, the electrified-solution jet cannot counterbalance the high Coulombic stretching force during electrospinning, resulting in fibre breaks, beads and instability of the process (Zhang & Hsieh, 2008). Thus, beads and pearl-on-string structures of fibres were produced from the 80:20 HP-β-CD/PEO blend solution. The findings are consistent with previous studies (Kriegel, Arrechi, Kit, McClements, & Weiss, 2008) that low molecular weight reduces the molecular chain entanglement which results in beads or droplets.

Effect of solution concentration

Effect of solution concentration on electrospinning process

Stable fibre jets and controlled fibre deposition are the primary requirements for successful electrospinning. Appropriate polymer concentration provides the desired viscosity for producing continuous uniform fibres. Electrospinning of 4–12 wt.% of 50:50 HP-β-CD/PEO was carried out at constant electrospinning conditions to investigate the effect of polymer concentration on process stability and fibre morphology. Figure 5 shows the nanofibre webs on the collector obtained from 4–12 wt.% solution concentrations. It was observed that the 6–12 wt.% solutions produced stable fibre jets, and formed clear circular-shaped fibre webs. Conversely, the 4 wt.% solution produced uncontrolled fibre jet which was deposited by the fibres over a large area without forming a clear fibre collection zone. Figure 6 shows the relative changes in the diameter of the nanofibre webs with respect to the polymer concentrations. The diameter of the nanofibre webs were found to be 12.5, 9.2, 7.4 and 5.7 cm for 6, 8, 10 and 12 wt.% concentrations, respectively. These results indicate that the lower polymer concentrations create larger webs. This appears to be happened due to lower viscosity and lower molecular chain entanglement of the HP-β-CD/PEO, which indicates the electrospraying of the solution instead of electrospinning. The electrospraying principle is similar to electrospinning where an overcharged conical droplet breaks up into smaller droplets due to high electrostatic force. In electrospinning, the polymer jet ejects from the tip of the Taylor cone of a mature droplet whereas in electrospraying, the droplet breaks up into micro-droplets before getting mature.

Effect of solution concentration on fibre morphology

Fibre morphology of HP-β-CD/PEO electrospun nanofibre was greatly influenced by polymer concentration. Several studies (Chowdhury & Stylios, 2010; Deitzel et al., 2001; Maeda, Hagiwara, Yoshida, Hasebe, & Hotta, 2014; Theron, Zussman, & Yarin, 2004) have shown the effects of polymer concentration on the diameter and the morphology of the electrospun nanofibres. Higher polymer concentrations are more likely to produce uniform fibres with higher diameters. Higher viscosity, which can be achieved by increasing polymer concentration, is essential for yielding continuous fibres. It promotes polymer chain entanglement and increases viscoelastic force, which resist the Coulombic stretching force during electrospinning. As a result, a continuous fibre jet travels towards the collector without breaking up.

However, very high or very low concentrations inhibit fibre formation. Figure 7 shows the morphology and fibre size distribution of electrospun nanofibers obtained from 50:50 HP-β-CD/PEO of 4–12 wt.% polymer concentrations. The 4 wt.% solution produced beads and spindle-like fibres. This may have happened due to inadequate viscosity and material content essential for molecular chain entanglement of polymer in the thin solution. The results, shown in Figures 7 and 8, found that the solutions of 4, 6, 8, 10 and 12 wt.% produced smooth fibres with an average diameter of 266, 249, 263, 257 and 285 nm, respectively. The ANOVA test found $F = 1.525$ and F crit = 2.408 indicating the insignificant differences between the mean diameters of the fibres obtained from different HP-β-CD/PEO concentrations. However, the solution of 8 wt.% concentration produced the most uniform fibres with narrowest distribution of the fibre diameter, and the 12 wt.% solution produced some irregular and branched fibres.

Conclusions

This study has provided a new approach for the electrospinning of HP-β-CD. The aim of this study was to optimize the HP-β-CD content in the HP-β-CD/PEO blend solution and the solution concentration to obtain smooth nanofibres. Hence, the study has investigated the effects of blend ratio and solution concentration on the electrospinning stability, fibre diameter and morphology of the electrospun HP-β-CD/PEO nanofibres. At a specific electrospinning condition, the lower polymer concentration produced beaded fibres. Uniform fibres with good morphology were obtained from 6 to 10 wt.% polymer concentrations yielding 249–263 nm mean diameter. This study reveals that the mass ratio has a significant influence on the diameter and morphology of the fibres compared to polymer concentration. The fibre diameter decreases and morphology deteriorates with increasing mass ratios of HP-β-CD/PEO. The solutions

with up to 70:30 HP-β-CD/PEO mass ratio produce uniform fibres. However, the results found that the solution concentrations and blend ratios have opposite effects on the electrospinning process stability and the size of the nanofibre webs. Higher polymer concentration produced smaller fibre webs and higher HP-β-CD/PEO ratio produced larger webs.

Due to the ability of absorbing suitably sized target molecules, the HP-β-CD nanofibres can be used in controlled drug delivery, biomedical applications, malodour absorbing wound dressing, filtration and other personal and health care products.

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