Synthesis and Characterization of Polyvinylpyrrolidone(PVP) Nanofibrous Thin Films

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Abstract. In the present work we have synthesised PVP composite nanofiber by using the electrospinning technique. The development, and morphology of the readied nanofibers were analysed by SEM. Arrangement of ultra-fine consistent smooth fibers with diameter across in the scope of 64-116 nm and length up to a few microns was seen in the case of PVP composite nanofibers. Experimental investigation was performed by electrospinning of PVP considering 6grams dissolved in 40ml Di-MethylFormamide(DMF).The process parameters considered were voltage of 20kV, flow rate 0.5ml/hr, needle dimension 21 gauge and tip-to-collector distance 100 mm. It was found that concentration of polymer is a critical factor in controlling the beads formation and fiber diameters although applied voltage and Spinneret to-collector distance also influenced the fiber diameter.

1. Introduction

Nanoscience is associated with the study of material property changes at atomic and molecular scale. Nanotechnology involves the design, characterization, production and application of system and devices with at least one of the structural dimension at nanoscale (less than 100 nm). Last two decades has witnessed an explosion in the advancement of material properties resulting in miniature components, accompanied by challenges in manufacturing. In spite of these challenges, nanoscience and nanotechnology have made unparalleled progress. Different techniques are in use for the development of nanomaterials such as phase separation, drawing, self-assembly, templet synthesis and electrospinning. Among these, electrospinning is relatively simple and expensive but generates polymer fibres in nano dimension consistently. Global market of polymer nanofibers was worth $80.7 million in 2015, and is anticipated to be $101.5 million by end of 2016. By 2020, the total revenue is expected to reach nearly $2.2 billion. The mechanical/chemical sector is anticipated to cover 73.2% of all revenues by end of 2016. Electronics is one of the fastest growing sector with total revenue of $6.4 million in 2015 and is expected to reach $323 million in 2020. Polymer nanofibers finds its potential globally in wide range of fields viz., cosmetic skin mask, life science, tissue building platform, military defensive garments, filter media.Electrospun nanofibers display novel qualities, such as high specific area, flexibility, surface functionalities and superior morphological structures. The conventional mechanical spinning techniques are not capable to produce fibers with diameters lesser than 2 μm. Different varieties of polymers have been synthesised and processed into nanofibers by various techniques such as melt blowing, drawing, template synthesis and phase separation. Nanofibers produced from these process possess higher dimensional orders and is less effective for thin film applications, which demands high specific surface area.[1-6] Electrospinning process has pulled in impressive consideration as a generally financially savvy and straightforward amalgamation strategy for one dimensional nanostructure.The unique nanofibers prepared by electrospinning generally exhibit high surface zone to volume proportions, alongside high porosity, nano measurements and phenomenal mechanical quality[7] Electrospinning is the process of...
fiber generation with the use of electrostatic force to draw charged polymer solutions into fibers of nano dimensions. Electrospinning utilizes the characteristics and properties of both the processing techniques of electro spraying and conventional solution dry spinning of fibers [8-11]. Polyvinylpyrrolidone (PVP) a typical hydrophilic and synthetic polymer has great film arrangement properties which makes it well known for electrospun nanofibers [12-13]. It exhibits the properties of viscosity enhancement, low chemical toxicity and biocompatibility. PVP thin film is widely used as a sensitive layer in Surface Acoustic Wave (SAW) sensors in order to detect the volatile organic compounds [14-15]. The key parameters of electrospinning that has direct relation on the fiber diameters are concentration of the solvent blend (wt.%), solution flow rate and applied electric field strength [16].

In the present study PVP of 6 grams dissolved in 40 ml of dimethylformamide (DMF) viz., was electrospun for the development of thin films. Review of open literature indicates wide investigation carried on electrospinning of PVP with a Mw of 1,30,000. Not very many have written about the electrospinning of PVP with a Mw estimation of 3,60,000. Hence, the introduction examination was gone for electrospinning of high sub-atomic weight PVP and studies the effect of process parameters on the fiber dimensions. Electrospinning technique was identified due to its capability to synthesize fibers in nano dimension and ease of operation.

1.1 Electrospinning process

Electrospinning is a versatile method for generating fibers in nano dimension from materials including polymers, composites and ceramics. Electrospinning system has three basic components viz., high voltage supply, spinneret or capillary tube and metal collector. This process can be administrated by several parameters, characterized widely into solution parameters, process parameters and surrounding parameters.

Polymer solution pumped through an injection system emerges from the nozzle and forms a spherical droplet. Electrostatic force from the high voltage supply overcomes the surface tension of the sphere and stretches the droplet. At a characteristic voltage, the polymer sphere stretches forming a Taylor cone with an apex angle of 98.6°. This process continues and the liquefied polymer forms a long and continuous fiber. As the jet enters the field formed between the nozzle and collector, it stretches and loops. The solvent undergoes evaporation and is separated from the polymer as a result only the polymer is deposited on the collector plate.

![Electrospinning Process](image-url)

**Figure 1:** Electrospinning Process
2. Materials and Method

2.1 Polyvinylpyrrolidone (PVP) of average molecular weight (Mw) of 3,60,000 was supplied from Sigma Aldrich Bangalore, solvent Di-MethylFormamide (DMF) from Ace Rasayana, Bangalore.

2.2 Dimethylformamide: Dimethylformamide is a natural compound with the equation (CH3)2NC(O)H. Ordinarily curtailed as DMF (in spite of the fact that this acronym is now and again utilized for dimethylfuran, or dimethyl fumarate), this drab fluid is miscible with water and the majority of organic liquids. DMF is a typical dissolvable for substance responses. Dimethylformamide is scentless have a fishy odor because of polluting influence of dimethylamine. As its name shows, it is a subordinate of formamide, the amide of formic corrosive.

DMF is a polar (hydrophilic) aprotic dissolvable with a high breaking point. It encourages responses that take after polar instruments, for example, SN2 responses. DMF is a typical dissolvable utilized as a part of electrospinning process.

2.3 Electrospinning of pure PVP
With a specific end goal to think about the impact of electrospinning parameters on PVP fixation in DMF, PVP solutions of 6grams, of molecular weight 3,60,000 was dissolved in 40ml of Dimethylformamide (DMF). Electrospinning of the prepared solutions was completed by loading of
each of the solutions in a 10 ml syringe. The needle tip was made flat by grinding the stainless steel
gauge 22 of 1.5 cm long. A thin aluminium foil was wrapped around the hard copper plate to serve as
stationary collector plate.

3. Characterization and measurement

The fibers were obtained from PVP solution at an electrical voltage of 20kV, at room temperature. By
using Scanning Electron Microscope(SEM) VEGA3 LM, the formation and morphology of the
electrospun PVP fibers were determined. A completely PC controlled SEM with traditional tungsten
warmed cathode planned both for high vacuum and additionally for low vacuum operations. The fiber
diameters have been measured by utilizing the image processing software, Image J (Image Pro-
Express, Version 5.0.1.26, Media Cybernetics Inc).

4. Results and Discussions

4.1 Result & Discussions

SEM pictures representing the morphological appearance of the electrospun fibers from PVP
solution in DMF is appeared in Figures 5a-h

4.2 Effect of Molecular weight

In the greater part of the literature identified with the electrospinning of PVP, low molecular weight of
1,30,000 is very common. Only a few have reported the utilization of high molecular weight PVP of
3,60,000. SEM images represent the morphological appearance of the spun fibers from PVP solution
in dimethylformamide. Quantitative outcomes as far as normal measurement of the spun fibers are
outlined in Figure h-d. High molecular weight was critical for the development of uniform fibers. Well
aligned and ultrathin fibers with average diameter in the scope of 64nm to 116nm were effectively delivered.

4.3 Alignment of the Electro spun PVP Fibers

The trademark highlight of electro spun fibers that has gotten much interest recently is the likelihood of aligning fibers. For the preparation of PVP thin films, a stationary copper collector plate was used to align the electro spun fibers. At high electrical potential of 20 kV, a uniform lateral speed of the polymer jet between the spinneret and the collector screen takes place and this finally executed in the form of well aligned electrospun fibers. It was found that with increasing applied potential the bead formation become less probable. The high charge density on the electrospinning jet leads to the broad extending, which is in charge of such little small fiber measurements. The chosen distance is sufficiently extensive to permit the adequate drying of the fibers when they achieve the collector plate. The well-defined electrospinning conditions enable us to create continuous fibers of PVP.

4.4 XRD Plots of PVP Nanofibrous Thin Films

![XRD Plots of PVP Nanofibrous Thin Films](image)

Figure 6 XRD Plots of PVP Nanofibrous

X-ray diffraction peaks are created by constructive interference of a monochromatic beam of X-rays diffracted at specific angles from each arrangement of cross section planes in a specimen. The pinnacle forces are controlled by the dissemination of particles inside the lattice. Here the pinnacles shows that PVP is in formless structure.

5. Conclusion

High quality PVP fibers with uniform structures and smooth surfaces were produced by using basic electrospinning setup. The fiber diameter varied between 64nm to 116nm. High molecular weight was significant for the formation of uniform fibers. Well aligned and ultrathin fibers which are having an average fiber diameter in the range of 64nm to 116nm were successfully produced. Minimum fiber diameter obtained was 64 nm and the maximum fiber diameter obtained was 116nm. poly(vinylpyrrolidone) (PVP) nanofibrous thinfilms are used in sensor applications.

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