International Journal of Advance Research in Science and Engineering
Volume No. 11, Issue No. 09, September 2022
www.ijarse.com
IJARSE
ISSN 2319 - 8354

Optimization and characterization of PVA nanofibers by electrospinning techniques

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ABSTRACT

Nanofibers are the most intriguing materials in the scientific world due to their numerous uses in diverse sectors. Their uses commence with the production of energy, continue with the resolution of issues affecting the environment, and continue with the medical community, among many other applications. Electrospinning has gained popularity in recent years because it can produce polymer fibers with Nano to micrometer-sized diameters using polymer solutions or melts. Compared to other conventional method, it is a simple, cost effective method for producing a wide range of porous structures. Electrospinning was the method that was employed in the synthesis of poly vinyl alcohol nanofiber membranes. We have experimented at electrospun polyvinyl alcohol (PVA) under a number of different parameters. The parameters of the electrospinning process, including applied voltage, solution concentration, polymer concentration, rotational speed of the collecting drum, collecting distance and flow rate were optimized in order to achieve the smallest possible fibre diameter. Scanning electron microscopy (SEM) was used to examine the optimization of the electrospun nanofibers.

Keywords: *Electrospinning, Nanofibers, Polyvinyl alcohol(PVA), Nanofiber membrane.*

I INTRODUCTION

Biomedical and pharmaceutical advancements have recently made substantial progress due to the development of adaptable biomaterials that can be tailored to fulfil even the most stringent criteria. The application of wound dressings, in particular, is critical in both wound healing and infection prevention. The various materials used in wound dressing can be categorized into several types, including foams, gauze, transparent films, alginates, composites, hydrocolloids, and hydrogel. When it comes to wound healing, each dressing has its own set of advantages and disadvantages [1].

Doctors and nurses face a challenging problem in selecting the optimum wound dressing for the right wound scenario at the right time. Polymeric films and foam wound dressings, for example, are popular because they may imitate the physiologic state of different tissues [2].

In the development and manufacturing of wood dressing and oral drug delivery products, vinyl polymers such as polyvinyl acetate (PVAc), polyvinyl alcohol (PVA), and polyvinyl pyrrolidone (PVP) are utilized in a significant manner [3]. Polyvinyl alcohol (PVA) is one of these types of polymers that is utilized in the synthesis of Nano fibrous structures. Because of its significant mechanical properties, non-toxicity, biocompatibility, and non-carcinogenicity, it is widely used in a variety of biomedical applications [4]. This biodegradable synthetic polymer, which is soluble in water that can be easily gelled using a variety of different cross-linking agents. Because of these properties, PVA has a wide range of applications in the medical, cosmetic, food, pharmaceutical, and packaging industries [5].

Nanomaterials are projected to have greatly better qualities due to their small size, which is virtually as small as their atomic and molecular sizes. In comparison to microfibers, all polymer nanofibers have a significant surface area-to-volume ratio, high porosity, appreciable mechanical strength, and functionalization flexibility [6]. Drawing, electrospinning, self-assembly, template synthesis, and thermal-induced phase separation are among of the processes used to create nanofibers [7]. Electrospinning is one of these techniques used here. Electrospinning has several advantages over conventional nanomaterial manufacturing technologies. It is a one-of-a-kind, flexible, easy, and cost-effective technology for controlling the size of nanostructures with high aspect ratios and producing continuous nanofibers from synthetic and natural polymers. Because of the vast range of uses for biomaterials, the topic of nanofibers has sparked a great deal of interest in biotechnology and medicine, and it has grown rapidly in recent years [8].

In this study, dissolved PVA was electrospun under various conditions. For the optimization of nanofibers, various electrospinning parameters such as applied voltage, collecting drum rotational speed, needle tip to collector distance, polymer solution concentration and flow rate are investigated. For this optimization, scanning electron microscopy was used. The effects of applied voltage, solution concentration, and feeding rate on the morphology of electrospun PVA nanofibers were also examined.

II EXPERIMENTAL

2.1 Experimental method

The electrospinning instrument (Holmarc Optomechatronics Pvt Ltd, Cochin, India- 683104) consists of a high voltage power supply [Zeonics Systech, India (upto 30 kV)] that provides a static voltage of 22 kV. The polymer solution was held in a BD Discardit II, 10 ml 21gauge syringe (0.8mmx40mm, 14.5 cm) and was positioned horizontally on a syringe pump [HOSPLF-04] against a 0.11 m diameter mandrel rotator [HO-MR-01]. After the solution was loaded into the syringe, the needle was connected to the positive electrode. The solution was electrospun vertically onto the negative collector. The polymer solution was drawn into a bundle of aligned fibers on the grounded rotating target after being subjected to high voltage.

2.2 Preparation of PVA solution

Polyvinyl alcohol (PVA) has a molecular weight of 85000-1,24,000 g/mol. PVA crystal was dissolved in distilled water to prepare PVA solution with varying concentrations of 2 wt%, 5 wt% ,10wt% and 20 wt%. Prior to electrospinning, the mixture was swirled under steady magnetic stirring for 24 hours at a temperature of 500C to achieve a homogenous solution. A 5ml syringe filled with PVA solution was positioned horizontally and the concentrations of 2 wt %, 5 wt %, 10 wt %, and 20 wt % were varied. An optimization of the electrospinning settings was carried out in order to accomplish the goals of attaining the appropriate fibre diameter and porosity as well as uniform and bead-free fibers. Experiments were carried out for optimization by varying different parameters, which resulted in distinct experimental runs.

2.2 Characterizations

The structural characterizations of PVA nanofibers were done using Scanning Electron Microscopy (SEM Jeol 6390LA/ OXFORD XMX N, Japan).

III RESULTS AND DISCUSSION

3.1 Optimization of parameters of PVA nanofibers

Different variables/parameters that influence the electrospinning of polymer solutions are broadly classified into two categories: solution parameters and process parameters. We can produce electrospun fibres with the desired morphologies and diameters by properly controlling those parameters that have an effect on the morphologies of the fibres. [9]. The solution parameters include the polymer's molecular weight, solution concentration, viscosity, solvent properties, and so on. The viscosity of a polymer solution is determined by its molecular weight, which influences other properties such as surface tension, conductivity, and so on [10].

3.2 Solution parameters

3.2.1 Concentration of solution

Polymer solution concentrations play an important role in fibre formation during the electrospinning process. Fig.1 depicts the morphologies of polymer fibers with varying concentrations. The morphology of the fibre changed as the concentration of the polymer solution was gradually increased from 2.5wt percent to 20wt percent while other parameters remained constant. The aforementioned result implies that the size and morphologies of the as-prepared nanofiber are strongly dependent on the concentration of the polymer solution. Polymeric microfibers are obtained at a low concentration (2wt%), and a mixture of polymeric micro and nanofibers is obtained at a concentration of 5wt%. At a concentration of (10% w/v), smooth fiber is formed and at a concentration of 20wt%, mixture of beads and fibers is obtained which are shown in fig.1(c) and (d). Viscosity of the solution can also be varied by changing the concentration of the solution. Electro spraying was produced in solutions with a concentration close to the critical entanglement concentration (2 wt%), whereas fibres were observed in solutions with a concentration above the critical entanglement concentration [10].

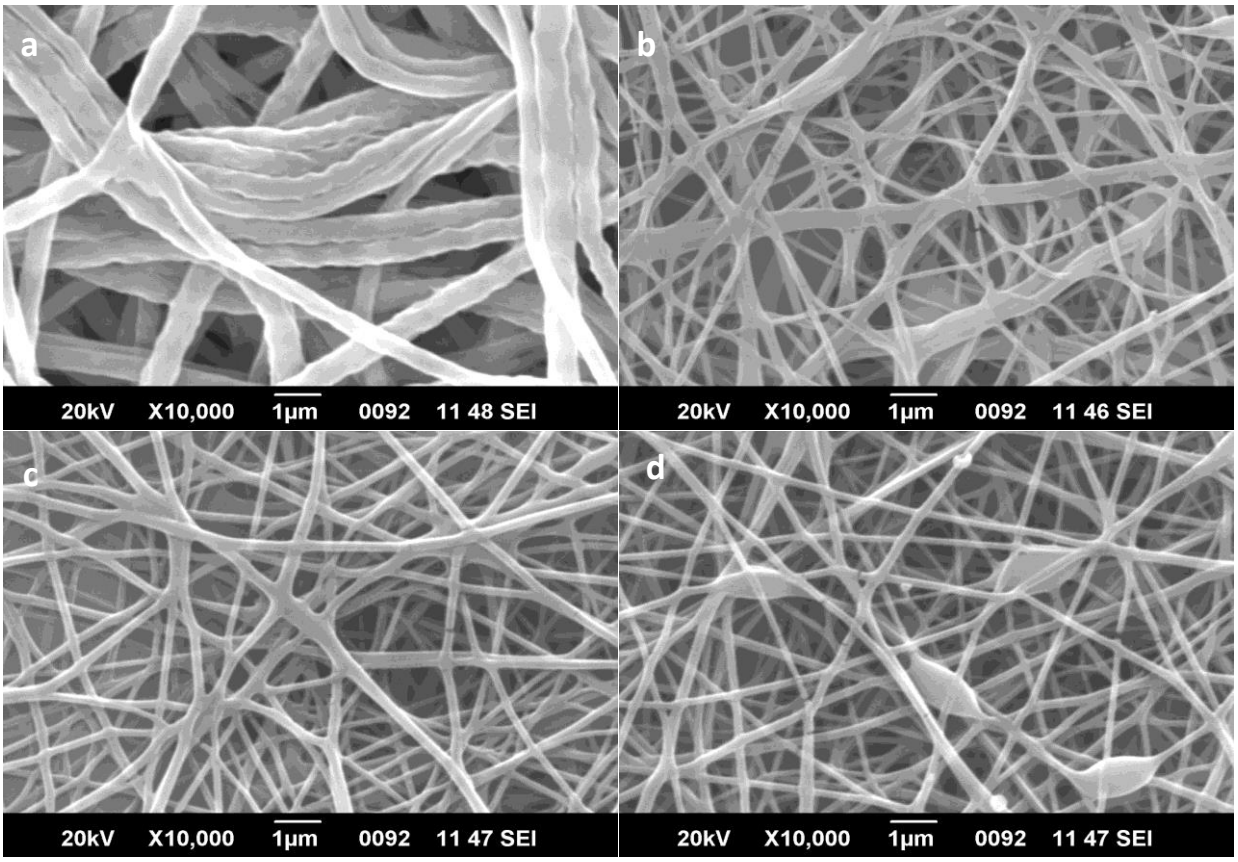

Fig.1 SEM images of fibers prepared with varying concentrations of polymer solution. (a) 2% w/v (b) 5% w/v (c) 10% w/v and (d) 20% w/v.

3.2.2 Molecular weight of the polymer

The polymer's molecular weight has a significant impact on the morphologies of electrospun fibers. In theory, molecular weight is a measure of the entanglement of polymer chains in solutions, which is expressed as solution viscosity. Electrospinning can produce a variety of structures such as beads, beaded fibres, and fibres depending on the rheological characteristics of the solution, which is influenced by molecular weight [11]. A. Koski, K. Yim, S. Shivkumar, and colleagues demonstrated that stable nanofibrous structures can be formed when $[\eta]C>5$, where $[\eta]$ is the intrinsic viscosity and C is the concentration [12]. This work makes use of the molecular weight obtained under these conditions. Surface tension, which is affected by the solvent composition of the solution, is another important factor in electrospinning. As previously stated, water was used as the solvent in this case. The surface tension changes as the concentration is adjusted.

3.2.2 Effect of electrospinning time

Fig.2 depicts the fibre morphologies as the electrospinning time is varied. The gap between the fibres narrows and the density of the fibres increases as the time increases from 15 minutes to 1 hour.

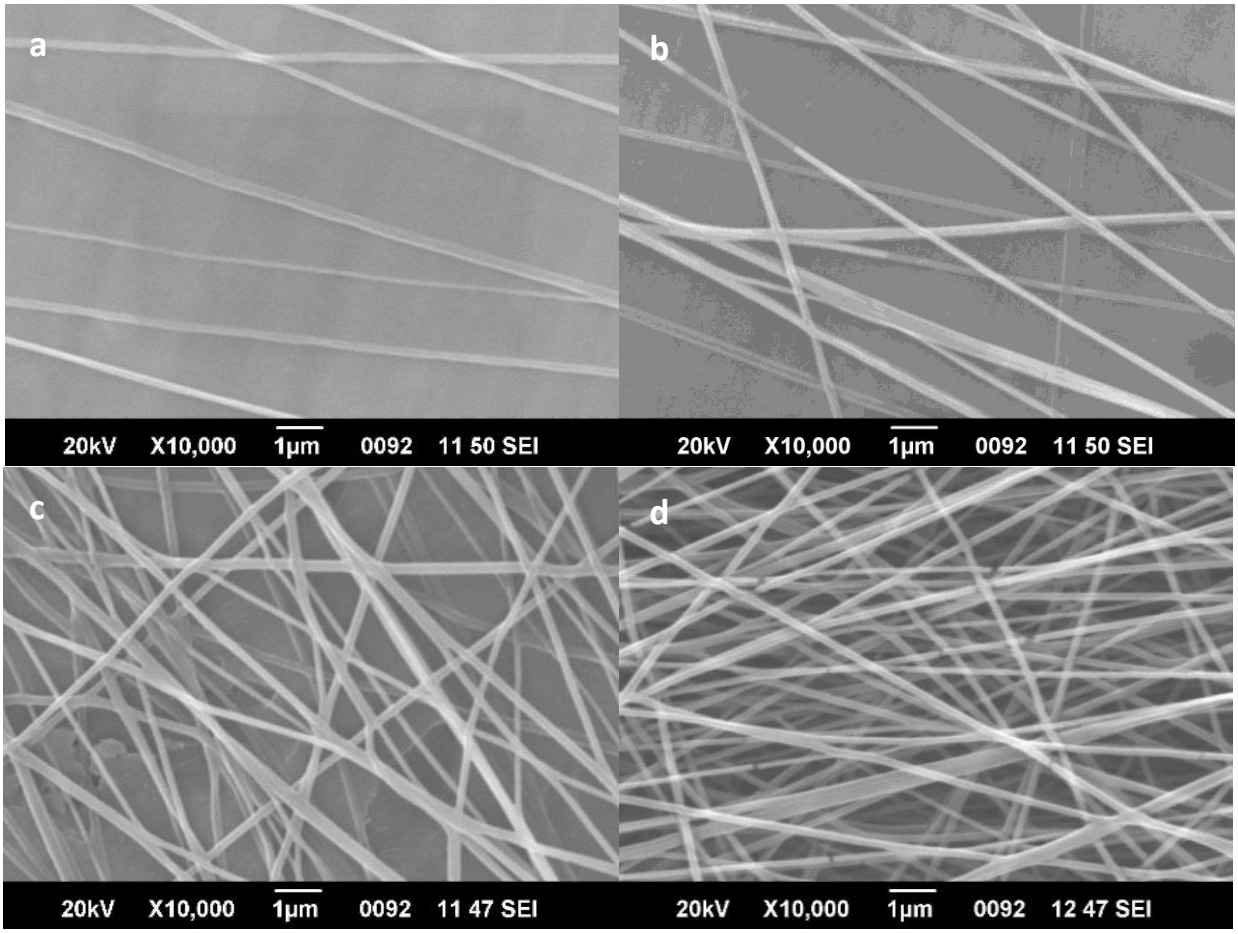

Fig.2 SEM images of fibers with varying time of electrospinning (a) 10min (b) 20min (c) 30min and (d) 1hour.

3.3 Processes parameters

3.3.1 Flow rate

The flow rate of the polymer solution is another important factor in controlling the morphology of electrospun nanofibers. We can slightly change the morphological structure of the fibre by adjusting the flow rate. Fig.3 shows that for a flow rate of 0.8ml/hour, a microfiber with a width of 1-2m is formed. When the flow rate is increased slightly to 1ml/hour, a micro and nanofiber mixture is formed. Lower flow rates are preferable because the polymer solution has more time to polarize. At a flow rate of 1.2 ml/hour, smooth fibres are obtained. As the flow rate exceeds this value, thicker diameter bead fibres can be seen. The solution jet is accelerated to the capillary tip at a rate greater than the rate at which the solution is removed from the tip by electric forces as soon as the flow rate exceeds a critical value. This change in mass balance causes the formation of an unstable jet, which results in fibers with large beads [13].

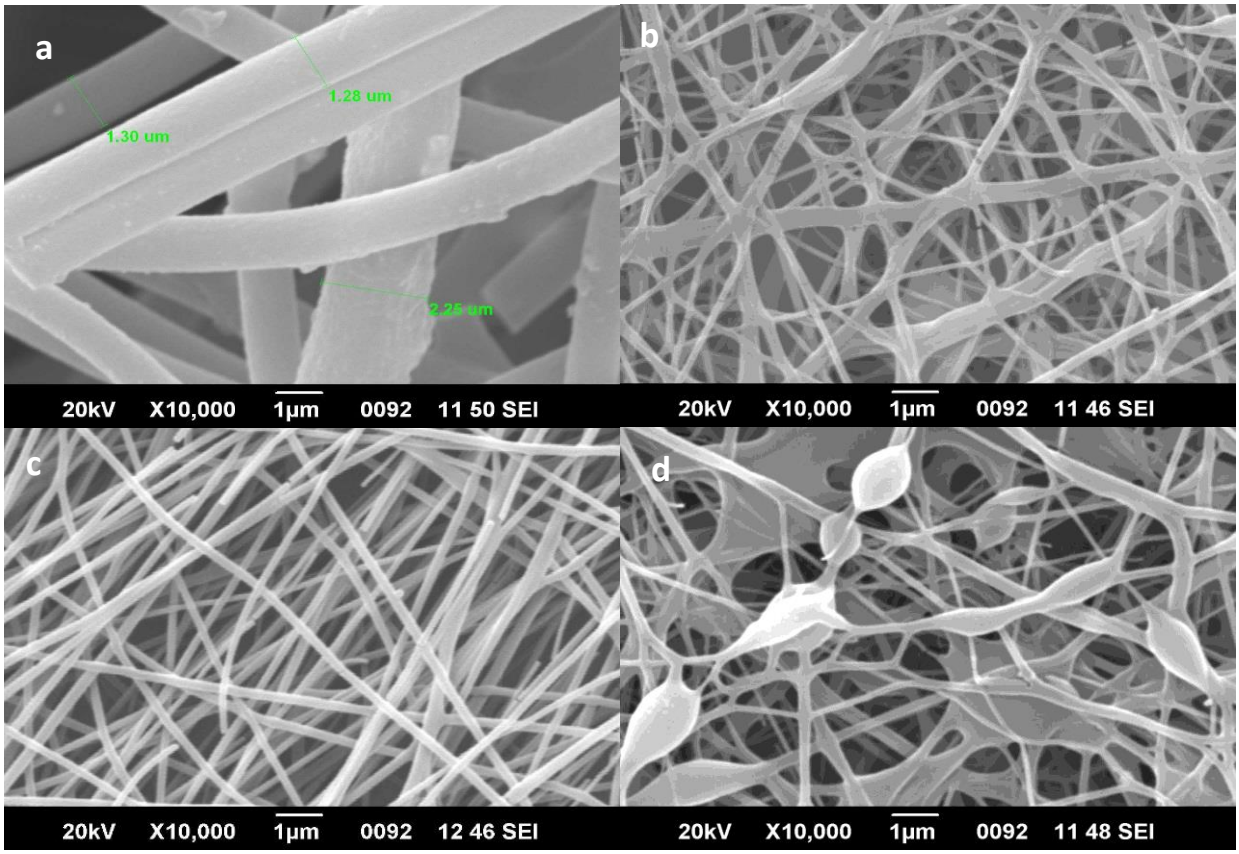

Fig.3 SEM images of fibers with varying flow rates (a) 0.8ml/hour (b) 1ml/hour (c) 1.2ml/hour and (d) 1.5ml/hour.

3.3.1 Applied Voltage

Another critical parameter in the electrospinning process is the applied voltage. By comparing the different images in fig.4, it is possible to conclude that the diameter of the fibre decreases as the applied voltage increases. The size distribution in fig.4 is not uniform for all applied voltages.

At a critical voltage, the introduction of current from a high-voltage power supply into a solution via a metallic needle causes the deformation of a spherical droplet into a Taylor cone and the formation of ultrafine nanofibers. Depending on the polymer system, this critical voltage varies. As the applied voltage increases, nanofibers with smaller diameters form, which can be attributed to polymer solution stretching and charge repulsion in the polymer jet [14]. When the applied voltage is increased beyond the critical value while the flow rate remains constant, beads or beaded nanofibers form. When the applied voltage is increased, the formation of beads or beaded nanofibers is attributed to a decrease in the size of the Taylor cone and an increase in the jet velocity.

3.3.1 Effect of collector to tip distance

In determining the morphology of electrospun nanofibers, the distance between the metallic needle tip and the collector is critical. The flow rate was set at 1.2 ml/h in this experiment, and the distance between the tip of the needle and the

collector was varied from 10 cm to 21 cm. The SEM images in Fig. 5 were used to evaluate the fibre morphology. This working distance influences the jet path and the amount of time it takes to reach the collector. If the distance between the collector and the fibre is too short, say 10cm, the fibre will not have enough time to solidify before reaching it, resulting in fused fibre morphology. The evaporation and thinning of the jet is less favorable as the distance between the nozzle and the collector increases, resulting in thick fibres with significantly increased diameter. Smooth nanofiber is obtained at an optimal distance of 15cm. The diameter of the nanofiber decreases as the working distance increases. A bead-free, smooth nanofiber is obtained at the optimal distance. It is possible that going beyond the optimal distance will result in a lower yield. This is primarily due to material loss to the surrounding environment as a result of turbulent droplet flight, as well as significantly reduced field strength [Ding et al 2010, Bosworth 2012]. If the working distance is too long, say 21cm, it can cause jet instability, resulting in the jet being drawn bent rather than straight. Bending will result in bead morphology.

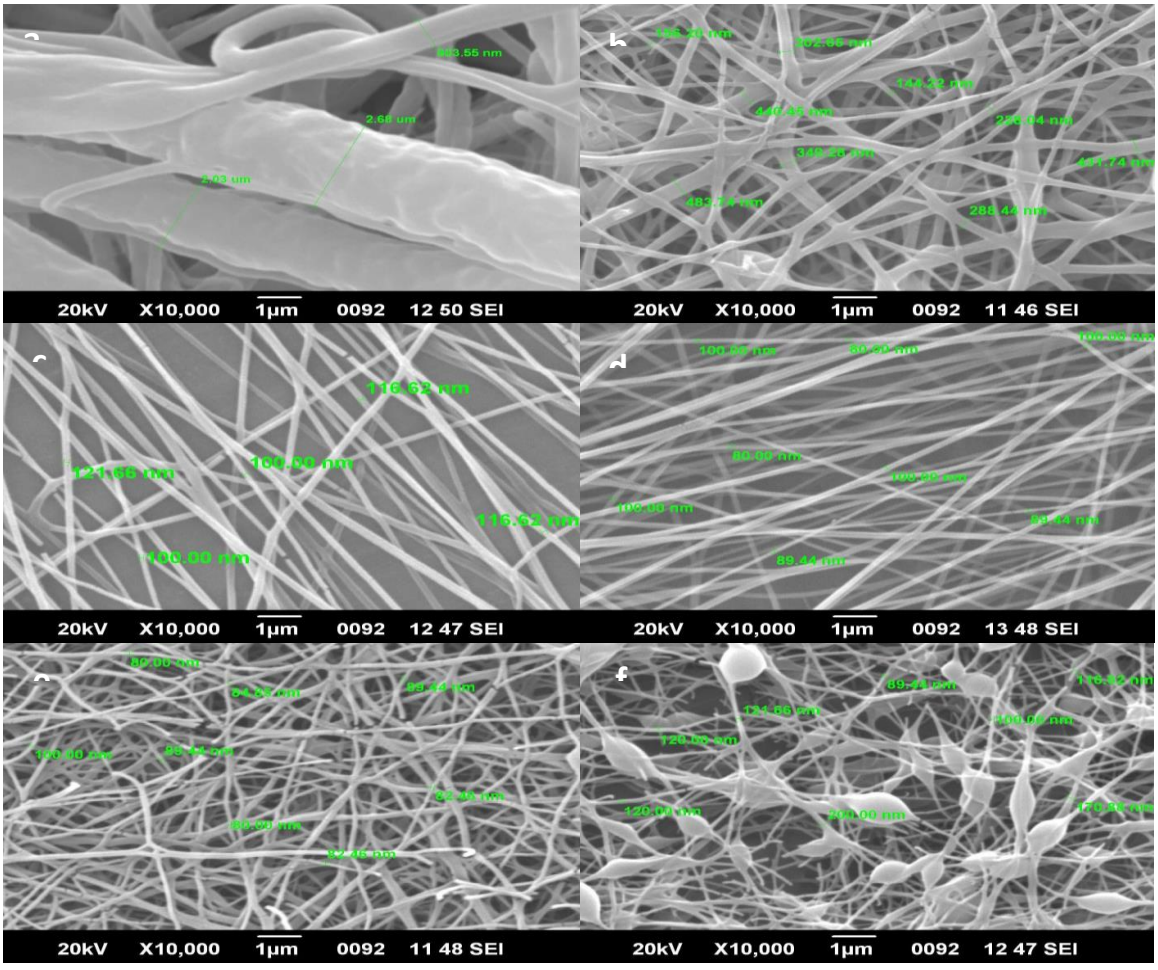

Fig.4 SEM images of fibers with varying applied voltages (a) 13KV (b) 14KV (c) 15KV (d) 16KV (e) 18KV and (f) 21KV.

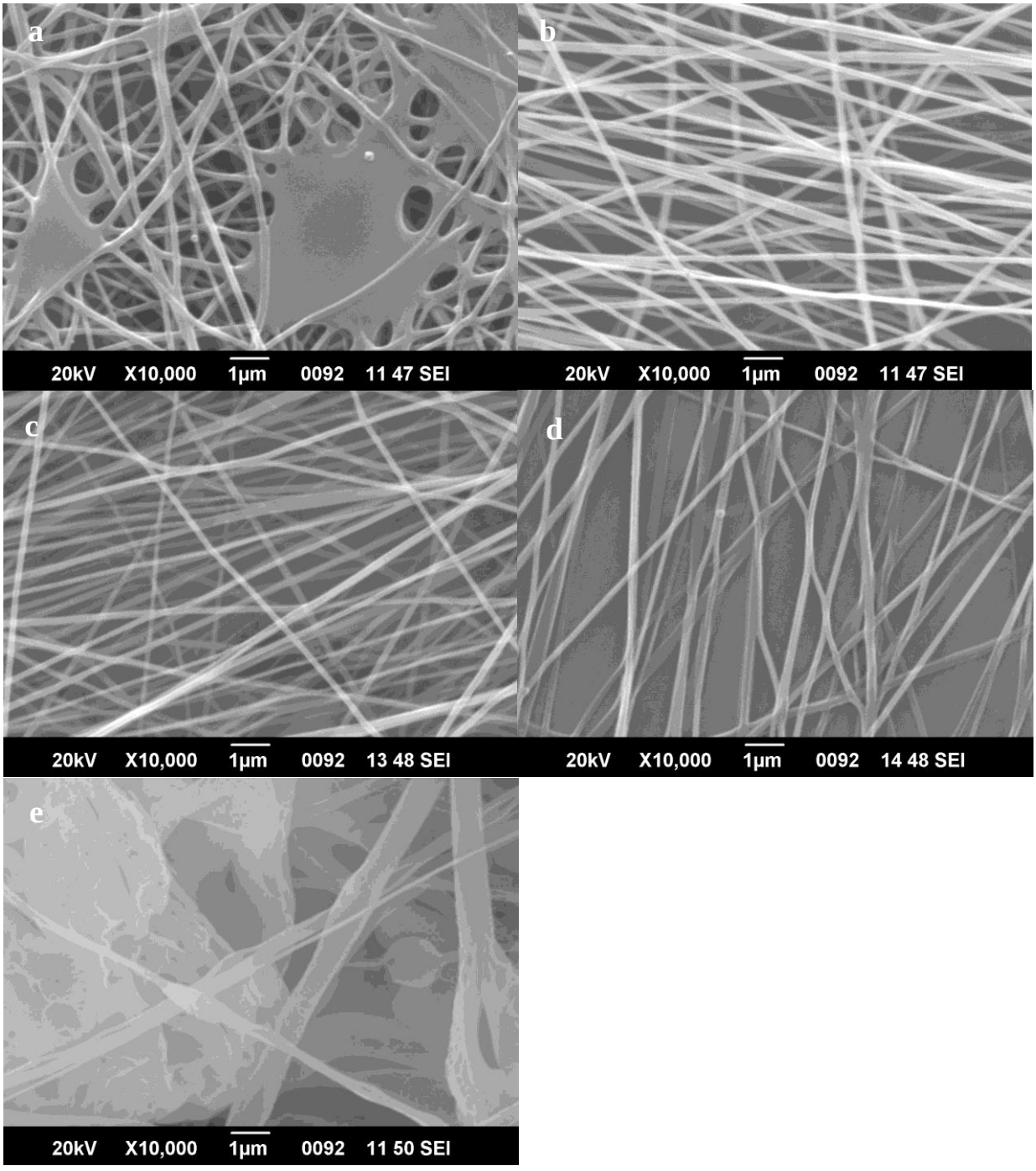

Fig.5 SEM images of fibers with varying distance between tip of the needle and collector (a) 10cm (b) 12cm (c) 15cm (d) 17cm and (e) 21cm.

The optimum conditions for synthesizing PVA nanofiber for this work are 15KV, 15cm, 1.2ml/hour, 1hour deposition time, and a concentration of 10%wt.

IV CONCLUSIONS

Electro spinning has been regarded as the most promising method for producing continuous nanofibers on a wide scale, with fibre diameters ranging from nanometers to micrometers. Electro spinning is a technique of drawing a polymer fluid into fine filaments using a strong electrical field. In this work, nanofibers of PVA were successfully produced by electrospinning technique. The optimization of PVA nanofibers was investigated. The optimum parameters for obtaining smaller and more uniform PVA nanofibers were determined to be 18kV applied voltage, flow rate of 1.2 ml/hr, concentration of 10 wt% and a needle tip to rotor collector distance of 15 cm. The optimized nanofiber showed high porosity and small diameter. For biomedical applications, further research is necessary to load the drug into this optimized nanofiber that could be the future scope of this research.

V ACKNOWLEDGEMENT

The authors acknowledge 'Sophisticated Test and Instrumentation Centre (STIC)' and 'Sophisticated Analytical Instrumentation Facility (SAIF), Cochin', for providing analytical facilities and also grateful to Dr. U.S Sajeev, Govt. College, Nattakam for providing Electrospinning facility for this work.

Conflicts of Interest

The authors declare that there is no conflict of interest in the publication of this article.

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