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Poly(vinyl alcohol) membranes produced by portable electrospinning device for *in situ* applications

André Luiz dos Santos[1]* and Sérgio Henrique Pezzin[2]

[1]Programa de Pós-graduação em Ciência e Engenharia de Materiais, Universidade do Estado de Santa Catarina – UDESC, Joinville, SC, Brasil

[2]Departamento de Química, Universidade do Estado de Santa Catarina – UDESC, Joinville, SC, Brasil

**andrelusantos11@gmail.com*

Abstract

Portable electrospinning devices have some advantages over conventional benchtop equipment, such as lower cost, better transportability, ease of operation and flexibility in size, shape and deposition surface, especially for *in situ* applications, such as wound dressings, drug release and cosmetics. In this work, poly(vinyl alcohol) membranes were produced from aqueous solutions, with different viscosities and at different electric field values, using a portable electrospinning device prototype. Field-emission scanning electron microscopy was used to characterize the morphology of the membranes. The results showed that the prototype allowed the electrospinning of membranes, presenting fiber diameter of 147±11 nm and deposition width of 4,6±0.3 cm, similar to the values obtained with the benchtop device.

Keywords: *electrospinning, portable devices, PVA, nanofibers, in situ applications.*

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1. Introduction

To bring electrospinning technology closer to end users, especially for *in situ* applications, such as wound dressings, miniaturized, portable and more affordable electrospinning equipment has been researched and presented commercially[1-7]. The possibility for the nanofibrous membrane to be deposited directly on the desired surface, reduces patient care time, prevents infection, eliminates the need for packaging and transporting the membranes, reduces pain and would make it possible to produce membranes in different shapes and sizes, depending on the skin lesion[1,8-10]. In addition to the potential medical applications, especially in outdoor activities, such as disaster rescues and battlefields, where it can be used to deposit a functional dressing directly on wounds, this technology can be also applied in the cosmetic and drug delivery areas, by adding active ingredients or drugs, such as anti-inflammatories and analgesics, to the polymeric solution[11]. Therefore, portable devices are more convenient for hospitals ambulances and possibly for everyday use by the end consumer[8,12-14]. Several authors have reported the development of these portable devices, using common commercial components, such as a miniature high voltage converters, which can supply voltages in the range of 1 to 10 kV. Some are battery operated and current limiting measures are employed for safer operation. These portable devices can be operated with just one hand, some can be placed in the pocket, and are operated at distances of 3 to 5 cm from the deposition surface[10,11,15,16]. Studies related to portable *in situ* electrospinning reported small, lightweight devices capable of electrospin submicron fibers made from various polymers with similar quality to those made on a conventional device presenting good performance and real-time control, demonstrating that these portable electrospinning devices can be an attractive alternative to existing benchtop machines[17].

However, a factor of concern in the electrospinning of polymeric solutions is the use of toxic solvents, not only due to the possibility of residual solvent present in the nanofibers produced, but also regarding to their handling during the spinning process[18]. Due to their cytotoxicity, organic solvents cannot be used for *in situ* electrospinning, as they can compromise wound healing, nor in cellular electrospinning (solutions with the addition of live cells)[5,19]. In this sense, following the concept of "Green Electrospinning", the use of toxic solvents can be avoided by using water-soluble polymers, even though only a small range of polymers are water-soluble[20]. Poly(vinyl alcohol), PVA, is one of these few vinyl polymers that is water-soluble, non-toxic, biocompatible, susceptible to biodegradation in the presence of microorganisms and considered a green polymer[21-24]. It is a synthetic, semi-crystalline polymer, produced from the hydrolysis of poly(vinyl acetate), with a degradation temperature starting at 150 °C[25,26]. It is commercially available in variations regarding its molecular weight and degree of hydrolysis (DH, from 70% to 99%)[23,27,28].

Therefore, the aim of this work is to use a lab-made portable device prototype for electrospinning membranes from aqueous PVA solutions, employing different electric field

configurations (voltages of 6 and 8 kV and needle-to-collector distances of 5 and 6 cm) and solution viscosities (for three different ranges of molecular weights and concentrations of 10 and 9% m/m), to evaluate the viability of this device in obtaining membranes and analyze the material and process parameters that influence their morphology (fiber diameter and deposition width). To the best of our knowledge, there is no literature that had reported on the combined use of this specific material, process parameters and electrospinning aparattus, which gives an innovative aspect to this study. It allowed a better understanding of electrospinning parameters, like the deposition width behavior, which is especially relevant for applications that require greater precision and smaller deposition area, such as portable devices.

2. Materials and Methods

2.1 Materials

Polyvinyl alcohol (PVA, Selvol™, Sekisui, China) partially hydrolyzed (88% DH), in three different molecular weight ranges: PVA1 ('Low Mw', 31,000-50,000 g/mol), PVA2 ('Intermediary Mw', 85,000-124,000 g/mol) and PVA3 ('High Mw', 146,000-186,000 g/mol), kindly provided by Vetta Quimica (Brazil), in powder form. The aqueous solutions were prepared with deionized water (Milli-Q) by magnetically stirring the systems at 80 °C for 12 h.

2.2 Electrospinning device

A lab-made prototype of a portable electrospinning apparatus, composed of a siringe support, a collector made of a flat aluminum plate with distance adjustment, and an adjustable miniature high voltage electrical supply (10x10x5.2 cm), which converts 220 V (AC) to 10 kV (DC), is shown in Figure 1a. An Eletrotech Lab EF 2B CRT 0212 (DBM Eletrotech Ltd., Brazil) benchtop electrospinning equipment, consisting of a high voltage supply (0 to 20 kV) QSC2020 (Inergiae, Brazil), a syringe pump (0.6 to 600 ml/h) and an adapted flat aluminum plate collector, Figure 1b, was used for comparison with the results obtained using the portable device. When using both devices, a 10 ml disposable syringe, with a stainless steel needle 0.55x20 mm (24G ¾"), was used as a reservoir for the polymeric solutions. The collector was covered with a 7x7 cm aluminum foil on which all membranes were deposited. All experiments were carried out in a laboratory with controlled temperature and humidity of approximately 22 °C and 45%, respectively. The spinning time was 3 min for all membranes.

2.3 Electrospinning experiments

After preliminary experiments to identify a range of viable process parameter values, a PVA2 polymeric solution at 9% (m/m) was electrospun under the action of four different electric fields (1; 1.2; 1.3 and 1.6 kV/cm), generated by varying voltage (6 and 8 kV) and needle-to-collector distance (5 and 6 cm), using the portable device prototype. In a second experiment, using the bench-top electrospinning device at 8 kV and needle-to-collector distance of 6 cm, four PVA solutions of different viscosities, in two different molecular weight ranges (PVA2 and PVA3) and concentrations (9 and 10% m/m) were electrospun in order to evaluate the effect of these parameters on the morphology of the membranes and also to compare and validate the results obtained with the portable device. All the electrospinning experiments were performed in triplicate.

2.4 Solution viscosity and conductivity

The viscosities of the polymeric solutions were measured using a MVD-5 digital rotational viscometer (Marte, Brazil). The viscosity values were obtained by an average of 6 measurements for each solution, at 12, 30 and 60 RPM, according to the viscosity range of each solution. The intrinsic viscosity [η] was calculated using the Mark Houwink relationship ($[\eta] = 6.51.10^{-4}\ Mw^{0.628}$) for PVA dissolved in water[29]. The conductivities of the polymeric solutions were measured using a NI CVM (Nova Instruments) benchtop conductivity meter at an ambient temperature of approximately 24 °C.

2.5 Morphological characterization

A field emission gun – scanning electron microscope (FEG-SEM) JSM-6710F (JEOL, Japan) was used to analyze the morphology of the membranes, which were covered with gold for 90 s at 40 mA, using a Denton Vacuum (U.S.A.) Desk V equipment. The fiber diameters were estimated by

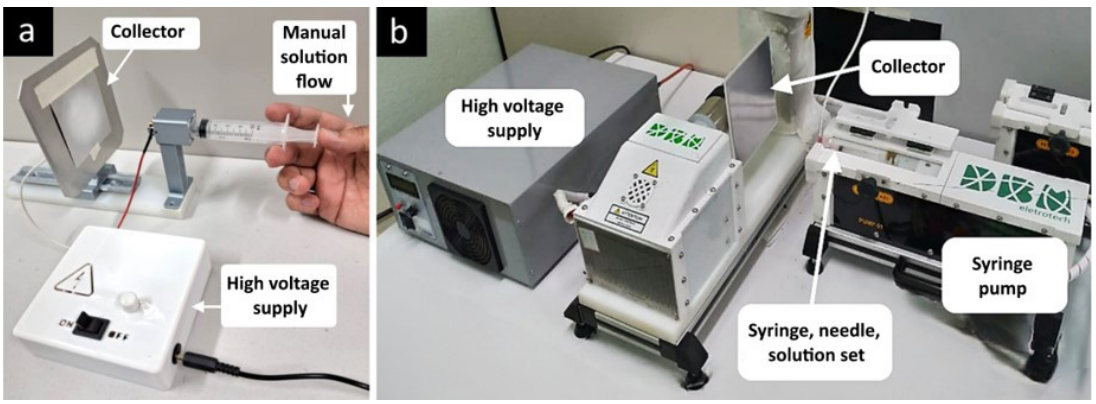

Figure 1. (a) Portable/miniature electrospinning system, (b) Benchtop electrospinning system.

manually measuring the diameter of 20 fibers homogeneously distributed in the micrographs at 15,000X magnification, using the ImageJ® software. The percentage pore area on the surface of the membranes was measured by methodology described by Wijayanti et al.[30], with the aid of the ImageJ® software. For the determination of deposition widths, the diameters of the electrospun membranes deposited on the collector surface were photographed and measured, following the methodology proposed by Chui et al.[31].

2.6 Statistical analysis

The statistical software MINITAB17 was used to process the fiber diameter values measured in each membrane, making it possible to obtain an average diameter value, with standard deviation and graphical representation through a histogram of its diameter distribution. Pearson's P correlation coefficient ($-1 \leq P \leq 1$) was calculated to statistically analyze the direction and strength of the relationship between the variables (for instance, electric field and fiber diameter). Values of between 0 and 0.19 indicate a very weak correlation, from 0.2 to 0.39 a weak correlation, 0.4 to 0.69 a moderate correlation, 0.7 to 0.89 a strong correlation and 0.9 to 1.0 indicate a very strong correlation.

3. Results and Discussions

3.1 Solution viscosities and conductivities

The viscosity and conductivity values of polymeric solutions, composed of PVA with different molecular weights and concentrations, are presented in Table 1. It is observed that the viscosity of the PVA solution increases with the molecular weight and concentration, which can be attributed to the greater entanglement between the polymer chains and intermolecular interactions between hydroxyl groups, respectively[32].

The properties of nanofibrous membranes are significantly affected by the physicochemical properties of the polymeric solutions, such as rheological behavior. When analyzing PVA membranes, Morais et al.[32] observed that there is a direct relationship between the viscosity of the formulation and the entanglement of the chains in the electrospun nanofibers, as there was an increase in viscosity between polymeric solutions with 7 and 8% (m/m) PVA in water. The concentration of the solution plays an important role in stabilizing the fibrous structure of electrospun nanofibers and it was observed that a fibrous structure could not be stabilized for $[\eta]C < 4$, where $[\eta]$ is the intrinsic viscosity and C is the concentration. For the formation of stable nanofibers, the $[\eta]C$ values must be between 5 and 12 ($5<[\eta]C< 12$)[27].

The electrical conductivity can also limit the electrospinning process. The formation of defect-free fibers will not occur at very low conductivity values, where charge migration to the surface of electrospinning formulations is almost completely avoided, and also at excessively high conductivity values[33], i.e. there is an ideal range of conductivity values in which electrospinning is viable, generating micro- or nano-metric fibers. The fiber diameter tends to decrease, and the diameter distribution to increase, with electrical conductivity[32]. Conductivity values varying from 277.6 to 313.6 µS/cm for 7 - 8% (m/m) aqueous PVA (Mw 85,500 g/mol – 89.5% DH) solutions, close to the values shown in Table 1, has been recenty reported[32].

3.2 Portable electrospinning experiments

Preliminary studies using the portable device prototype were carried out to identify a range of viable process and material parameter values. It was observed that for PVA2 solutions at 10% (m/m), voltages up to 8 kV and needle-to-collector distance of 6 cm, only one Taylor cone is formed at the tip of the needle, projecting a single visible jet (in the stable stage), which allowed the deposition of a circular-shaped membrane with greater deposition precision, Figure 2a. At voltages above 8 kV, the formation of multiple jets was observed.

For solutions of PVA1 and PVA3 at 10% (m/m), the formation of fibers was not possible at these conditions (8 kV and 6 cm) and, thus, they were discarded for further experiments. In the case of PVA1, it was observed the projection of solution drops on the collector, forming a beaded structure, Figure 2b, which characterizes an electrospray process instead of electrospinning[1]. This behavior can be explained by the very low viscosity of the solution[19] and high conductivity[34], leading to greater instability. PVA3 solutions presented difficulty in forming the Taylor cone, due to the higher viscosity and an insufficient needle-to-collector distance to form the zone of instability and thinning of the jet, not allowing proper evaporation of the solvent until its deposition on the collecting surface. This fact caused the accumulation of the polymeric solution in the collector, Figure 2c. In all cases, it was observed dripping of solutions on the support table, as well as projection of some drops onto the collecting surface, which can be justified by the low volatility of the solvent (water), decreasing with the increase in the molecular weight of PVA.

Figure 3 presents the FEG-SEM images of the four membranes electrospun from a 9% (m/m) PVA2 solution using the portable device, with the variation of electric field values (1; 1.2; 1.3 and 1.6 kV/cm), by a combination of two electrical voltages (6 and 8 kV) and two needle-to-collector

Table 1. Viscosity and conductivity values of polymeric solutions.

Weight average molecular weight (Mw) g/mol	Solution concentration (C%) (m/m)	Solution viscosity (mPa.s)	Intrinsic viscosity [n]	[n]C	Solution conductivity (µS/cm)
PVA1	10	21.6 ± 0.1	0.5	5.0	637.4
PVA2	9	185.7 ± 2.2	0.9	8.1	247.9
PVA2	10	372.6 ± 5.1	0.9	9.0	268.7
PVA3	10	1131.5 ± 6.9	1.2	12.0	419.7

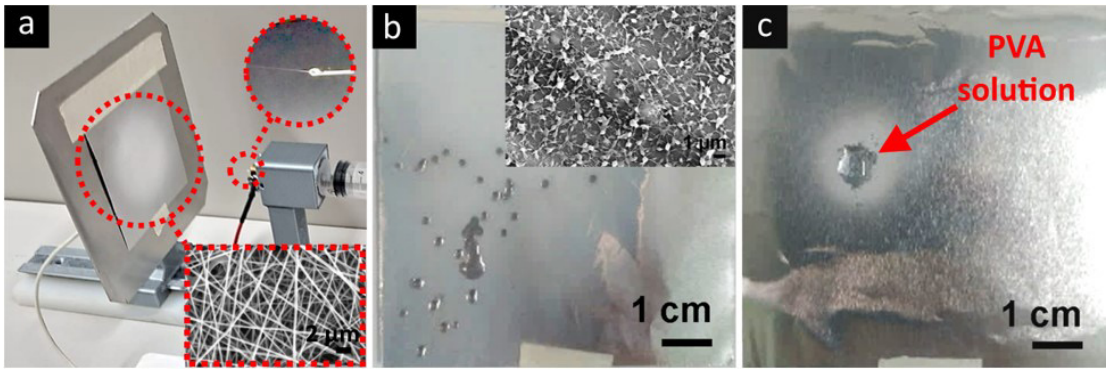

Figure 2. (a) PVA2 at 10% (m/m) membrane in the collecting surface and the polimeric jet at the tip of the needle (insert), (b) electrospun membrane from PVA1 at 10% (m/m) and (c) electrospun membrane from PVA3 at 10% (m/m).

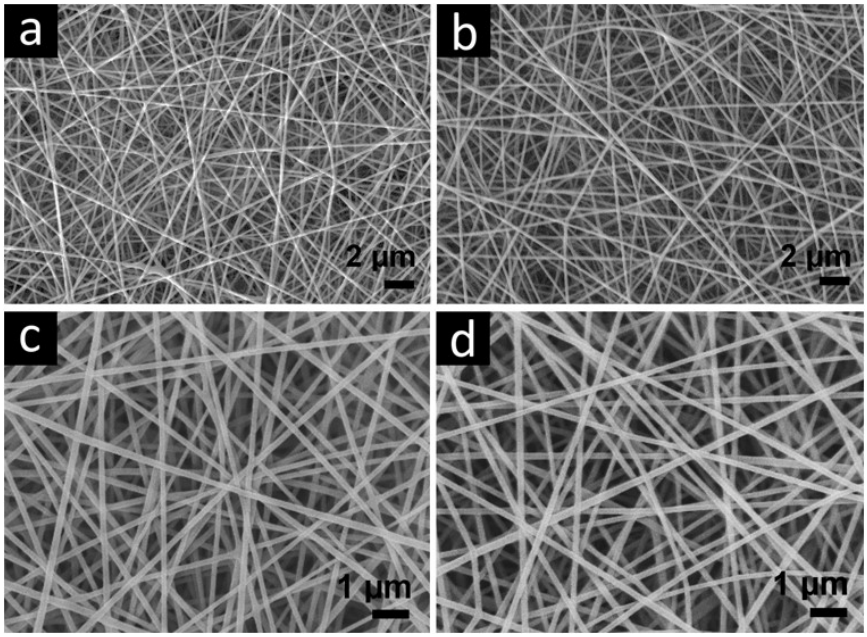

Figure 3. FEG-SEM images of PVA2 membranes produced from 9% (m/m) solutions under different electric field values: (a) 1 kV/cm , (b) 1.2 kV/cm, (c) 1.3 kV/cm, and (d) 1.6 kV/cm.

distances (5 and 6 cm). Table 2 presents the values of average fiber diameter and deposition width (membrane diameter) of the PVA2 membranes.

It is observed the formation of membranes with fibers in the nanometric scale, randomly oriented and without the presence of defects. There was a decrease in the average diameter values of the fibers with the increase in the electric field value, which is characteristic of a strong negative correlation between these two parameters, confirmed by a Pearson correlation coefficient P=-0.844. This behavior has been also reported for the electrospinning of 2.5% PEO aqueous solutions[35]. It is interesting to mention that the value of the electric field, determined by dividing the applied electrical voltage by the distance between the surface of the drop of solution pending at the tip of the needle and the deposition collecting surface, is generally about 1 kV/cm for conventional polymer electrospinning[36]

Table 2. Electric field, fiber diameter and deposition width values of electrospun PVA2 membranes produced from 9% (m/m) solutions.

Electric field (kV/cm)	Fiber diameter (nm)	Deposition width (cm)
1.0	181.9 ± 13.0	7.4 ± 0.2
1.2	150.1 ± 13.6	6.5 ± 0.4
1.3	147.0 ± 11.4	4.6 ± 0.3
1.6	142.1 ± 11.1	3.9 ± 0.2

In summary, surface charges on the fluid surface increase with increasing applied electric field, and the elongation of the

electrospun jet increases with increasing jet surface charges and time of flight[35]. Furthermore, it is observed that the fiber diameters do not vary so significantly by changing the applied electric field as by changing the material parameters, such as conductivity and viscosity.

Figure 4 shows the photographs of electrospun PVA2 membranes deposited on the aluminum collecting surfaces.

Considering its approximately circular shape, it is possible to observe a decrease in diameter with the increase of the electric field intensity, as shown in Table 2, characterizing a decrease in the area of the membrane deposited on the collecting surface, which is also characteristic of a very strong negative correlation, according to a Pearson correlation coefficient P= -0.934. In the same way that the unstable region of whipping depends on the intensity of the applied electric field, influencing the stretching of the fiber, in this region, due to cone formation and the jet path, a variation in the formation of the deposition area or spreading diameter is observed, which can be measured by the diameter of the membrane collected on the deposition surface. Therefore, the controllability of nanofiber deposition is also affected by instabilities caused at this stage, with the length of the straight jet in the first stable stage increasing with the increase in the applied electric field and, consequently, the whipping time decreases with increasing applied electric field[35]. To obtain a more precise and controllable fiber deposition, an electrospinning technique with modified electric fields, using a metal cone attached to the capillary and/or an electrode positioned in the jet path has been proposed[37].

3.3 Benchtop electrospinning experiments

In order to validate the results obtained in the previous experiment using the portable miniature electrospinning device, electrospun PVA membrane were also obtained by using a conventional benchtop electrospinning equipment. Figure 5 presents the photograph and the FEG-SEM image of the membrane electrospun from PVA2 at 9% m/m, using the same electric field (1.3 kV/cm, at 8 kV and 6 cm) of the previous portable electrospinning experiment. In this experiment, however, a 1.2 ml/h flow rate was employed. It was observed the formation of a membrane with fibers in the nanometric scale, randomly oriented, with an average diameter of 134.2±10 nm and deposition width of 6.3±1.5 cm.

3.4 Electrospinning devices comparison

In Table 3, the values of average fiber diameter and deposition width are presented for PVA2 membranes electrospun from a 9% (m/m) solution by the portable and the benchtop electrospinning devices. Although it shows a diference of 1.7 cm for the depositon width, it can be observed that there was no significant variation in the mean

Table 3. Comparative analysis of fiber diameter and deposition width results between portable and benchtop devices.

Spinning device	Fiber diameter (nm)	Deposition Width (cm)
Portable	147 ± 11	4.6 ± 0.3
Benchtop	134 ± 10	6.3 ± 1.5

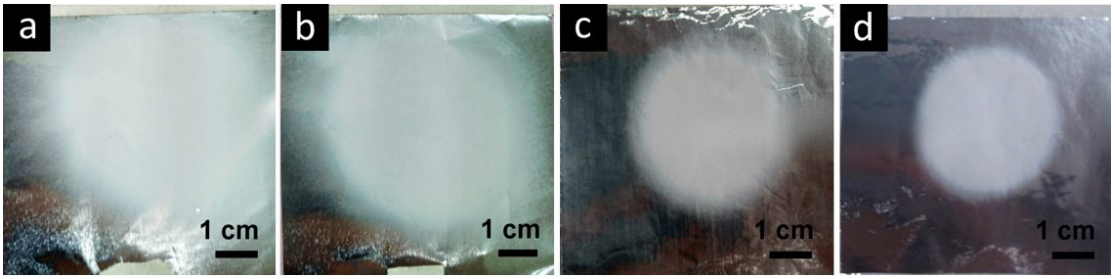

Figure 4. Photographs of PVA2 membranes produced from 9% (m/m) solutions under different electric field values: (a) 1 kV/cm , (b) 1.2 kV/cm, (c) 1.3 kV/cm and (d) 1.6 kV/cm.

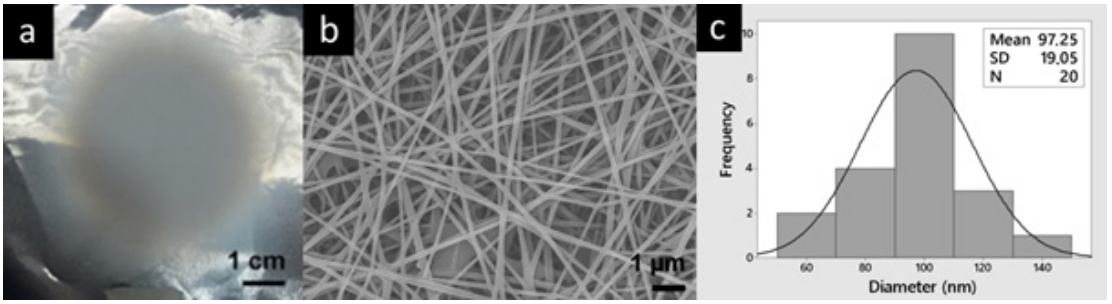

Figure 5. (a) Photograph of the membrane, (b) FEG-SEM image of the membrane (PVA2 at 9% m/m) electrospun under 1.3 kV/cm electric field (fist replica) and (c) fiber diameter distribution histogram (SD = standard deviation; N = number of measurements).

fiber diameter obtained between the two devices, using the same material and process parameters, suggesting that the electrospinning performance and quality of the electrospun membranes produced by the portable device are similar to those obtained using the commercial benchtop equipment.

Another relevant difference between both devices was the use of an uncontrolled/uniform flow rate, as a pressure was mannualy applied to the piston of the syringe by the operator, varying according to the formation or decrease of the drop of solution at the tip of the needle in the portable electrospinning device. This was due to the fact that lower viscosity values were employed, together with the use of water, which presents a relatively low volatility. The formation of a drop of polymeric solution at the tip of the needle and its control during the spinning process was thus easier, as no greater variation in fiber diameter was observed, due to the balance between the rates at which the solution is dispensed/extracted from the capillary[38].

Membranes composed of fibers with random orientation morphology, presenting fiber diameters in the range of 110 to 520 nm, correspond to the ECM of human skin and a porosity range of 63 to 71% facilitates cell adhesion and proliferation[39]. A similar study reported the electrospinning with a portable device at 10 kV of a 8% PVA (Mw 186,000 g/mol) solution with the addition of stem cells and it was observed that the cells were evenly distributed on the filaments of the membrane and that the low voltage of the handheld device did not affect cell viability[19].

4. Conclusions

It is concluded that the developed prototype of a portable electrospinning device is suitable for the electrospinning of aqueous PVA solutions into membranes composed of randomly oriented fibers with nanometric diameter, similar to those obtained by laboratory benchtop equipment. Additionally, this study enabled a better understanding of the effects of process and material parameters on the fiber morphology, deposition width and surface pore area of the membranes. It was observed that the use of lower voltages provides more stable electrospinning, with the formation of only one Taylor cone and polymer jet, resulting in greater deposition precision, which is advantageous for both *in situ* deposition applications and for cell electrospinning, which requires precisely lower values of high electrical voltage.

The use of a biocompatible polymer and a non-toxic solvent provided both a lower environmental impact and greater suitability for medical applications, thus being viable for potential applications such as wounds dresings or for transdermal administration systems. The use of water as a solvent prevented the clogging effect, since no solidification of the solution at the tip of the needle was observed and made possible the manual control of the solution flow rate, which contribute for the elimination of a flow control system and reduction in the cost of an eventual commercial portable device.

5. Author's Contribution

• **Conceptualization** – André Luiz dos Santos; Sérgio Henrique Pezzin.

• **Data curation** – André Luiz dos Santos.

• **Formal analysis** – André Luiz dos Santos.

• **Funding acquisition** - Sérgio Henrique Pezzin.

• **Investigation** – André Luiz dos Santos.

• **Methodology** – André Luiz dos Santos; Sérgio Henrique Pezzin.

• **Project administration** – André Luiz dos Santos; Sérgio Henrique Pezzin.

• **Resources** – André Luiz dos Santos; Sérgio Henrique Pezzin.

• **Software** – NA.

• **Supervision** – André Luiz dos Santos; Sérgio Henrique Pezzin.

• **Validation** – André Luiz dos Santos.

• **Visualization** – André Luiz dos Santos.

• **Writing – original draft** – André Luiz dos Santos.

• **Writing – review & editing** – André Luiz dos Santos; Sérgio Henrique Pezzin.

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