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Original Article

STUDY OF THE FEASIBILITY OF DEVELOPING A DRESSING USING POLYVINYLPYRROLIDONE, ETHANOL, AND *ALOE VERA* BY ELECTROSPINNING

ESTUDO DA VIABILIDADE DO DESENVOLVIMENTO DE UM CURATIVO COM POLIVINILPIRROLIDONA, ETANOL E ALOE VERA POR ELETROFIAÇÃO

1	Carlos Henrique Vieira	carlos_vieira_1995@hotmail.com	
2	Marcelo Castanheira da Silva	marcelo.silva@ufac.br	
3	George Chaves da Silva Valadares	george.valadares@ufac.br	
4	Braúlio Soares Archanjo	bsarchanjo@inmetro.gov.br	
5	William Ferreira Alves	william.alves@ufac.br	
6	Bruce Willys da Silva Moreira	bwillys998@gmail.com	
7	Rogério de Freitas Lacerda	rogerio.lacerda@ufac.br	
8	Anselmo Fortunato Ruiz Rodriguez	anselmo.rodriguez@ufac.br	
9	Tiago de Jesus Santos	tiago.jesus@ufac.br	
10	Carlos Henrique Moreira Lima	carlos.lima@ufac.br	
11	Lourivaldo de Oliveira Santos	lourivaldooliveira04@gmail.com	

1 Lourival Pinho State High School
2,3,6,7,9,10 Center for Biological and Natural Sciences, Federal University of Acre
4 Materials Metrology Division, National Institute of Metrology, Quality and Technology (Inmetro)
5 Nanobiotechnology Laboratory, Center for Biological and Natural Sciences, Federal University of Acre
8 Biotechnology and Biodiversity Postgraduate Program, Federal University of Acre
11 Teacher José Rodrigues Leite Hign School

ABSTRACT

The work aimed to develop a dressing for wound treatment using the polymer polyvinylpyrrolidone, ethanol, and crushed leaves of the medicinal plant Aloe vera, produced by the electrospinning method. Six samples were produced and observed by scanning electron microscopy (SEM), as well as by infrared spectroscopy (FTIR). The percentage distribution of fiber diameters was analyzed, and the increasing of *Aloe vera* leaves mass in the solution tends to favor this distribution and increase the fiber diameter. The infrared spectra indicated the presence of the amine, phenol, alkene, alkylamine, and alkane. There was a similarity in the infrared line shapes for all the samples, except for sample 3, which indicated the presence of an aryl-alkyl ether group. The presence of the phenol functional group can assist in the prevention and treatment of skin diseases.

KEYWORDS: *Aloe vera*. Electrospinning. Polyvinylpyrrolidone.

RESUMO

O trabalho teve como objetivo desenvolver um curativo para tratamento de feridas utilizando o polímero polivinilpirrolidona, etanol e folhas trituradas da planta medicinal Aloe vera, produzida pelo método de eletrofiação. Seis amostras foram produzidas e observadas por microscopia eletrônica de varredura (MEV) e por espectroscopia no infravermelho (FTIR). A distribuição percentual dos diâmetros das fibras foi analisada, e o aumento da massa das folhas de *Aloe vera* na solução tende a favorecer essa distribuição e aumentar o diâmetro da fibra. Os espectros no infravermelho indicaram a presença de amina, fenol, alceno, alquilamina e alcano. Houve similaridade nas formas das linhas do infravermelho para todas as amostras, exceto para a amostra 3, que indicou a presença de um grupo aril-alquil éter. A presença do grupo funcional fenol pode auxiliar na prevenção e tratamento de doenças de pele.

PALAVRAS-CHAVE: *Aloe vera*. Eletrofiação. Polivinilpirrolidona.

INTRODUCTION

Aloe vera is recognized for its biological properties and health benefits and is considered one of the most studied herbs in the natural products category, as it has remarkable regenerative properties for the skin and other soft tissues. *Aloe vera* contains more than 75 biologically active compounds such as polysaccharides, vitamins, enzymes, amino acids, and minerals [1-2].

The polyvinylpyrrolidone (PVP) biopolymer has high solubility in polar solvents, low toxicity, high hydrophilicity, and good physiological adaptation, it is also part of the so-called biomaterials, that are synthetic materials that can be used to replace parts of a living being or in contact with tissue [3]. This polymer can be electrospun and has a variety of applications in medicine [4-5].

The electrospinning technique [6-7] consists of using a syringe containing a polymeric solution, in which its needle is connected to the positive terminal of a source of direct current and the conductor collector, as well as the negative terminal of the source, are grounded. The injection is performed by controlling the polymeric solution present in the body of the syringe through a syringe pump. The application of high voltage and low current generates an electric field between the needle and the collector, while, simultaneously, the pump injects the fluid contained in the syringe, which also becomes electrically charged.

The surface tension is overcome when the amount of charge in the solution becomes high enough, forming a Taylor Cone at the tip of the needle and consequently the jet will be attracted to the collector due to the applied voltage. During the flight of the jet, the solvent evaporates and there is a reduction in the diameter of the beams [6,8].

Electrospinning allows obtaining small diameter fibers (from micrometers to nanometers) capable of covering a large surface area, allowing the constituent molecules of the plants to spread more effectively than traditional dressings, considerably reducing wound healing time. Electrospun fibers have been used in several fields such as: filtration, biomedical, nanocatalysis, pharmaceutical, medical care, biotechnology, among others [7-8].

The objective of this work was to investigate the potential development of a bandage to treat wounds or burns in animals or humans, using a polyvinylpyrrolidone solution dissolved in 92.8º ethanol and *Aloe vera* leaf extract. This research seeks to

investigate an alternative method for the treatment of wounds based on the use of medicinal plants.

MATERIAL AND METHODS

Aloe vera was collected in June 2015 in the Tucumã neighborhood in Rio Branco, Acre, Brazil. The leaves, with the best visual appearance, were selected, washed in running tap water and then in distilled water. The leaves were cut, and their gel discarded, then, they were dried in an oven at 45ºC for 48 hours. Finally, they were crushed in a blender.

Five polymeric solutions were prepared using polyvinylpyrrolidone (PVP) polymer (mol wt. 360,000) manufactured by Sigma-Aldrich, hydrated ethanol 92.8% INPM and adding different mass values of the crushed leaves of the plant.

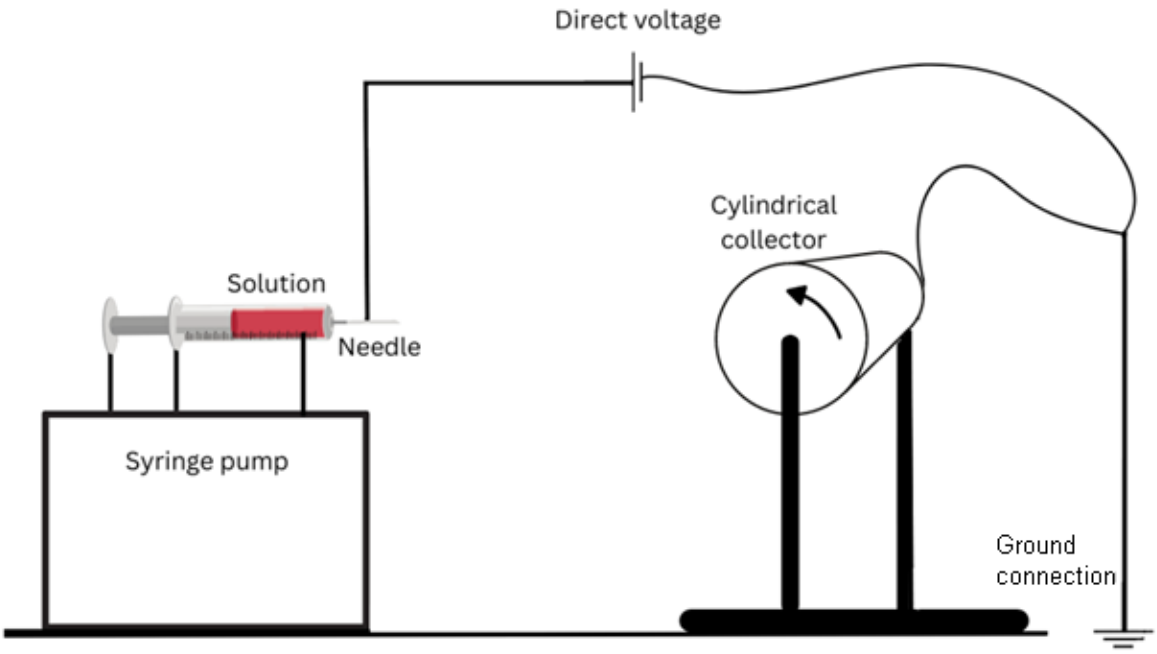
Each polymeric solution generated six types of samples, after being electrospiked, according to Table 1. Sample 6 is the reference (standard), that is, it did not contain leaf mass. Initially, only the masses of Aloe Vera leaves were diluted in 10 mL of ethanol, then placed on a magnetic stirrer for 24 hours. Then an amount of 0.40 g of PVP was added to each solution and placed again on a magnetic stirrer for 24 hours. Each of the resulting solutions was inserted into 20 mL plastic syringes to start the electrospinning process.

Table 1 - Samples produced by varying the masses of crushed *Aloe vera* leaves.

Sample	1	2	3	4	5	6
Mass of *Aloe vera* leaves (mg)	5.0	10.0	15.0	20.0	25.0	0

The electrospinning equipment (Figure 1) included a syringe pump (NE-1600 Six Channel Programmable Syringe Pump), a DC voltage source (HIPOT CC MOD EH5005C – Electrotest) and a mechanical stirrer (model 713 – Digilab).

Figure 1 - Basic schematic diagram of an electrospinning apparatus. Source: the authors.



The syringe, containing the sample 1 solution, was attached to the syringe pump whose pumping speed was set to 1.0 mL/h. The cylindrical metallic collector, with a radius of 2.0 cm, was covered with an aluminum foil and worked as substrate for the samples produced.

During the electrospinning process, humidity and temperature were maintained at around 40% and 23.0°C, respectively. A continuous voltage 40.0 kV was applied between the syringe needle and the grounded cylindrical collector, whose rotation was fixed at 3,000 revolutions per minute (rpm). The distance from the tip of the syringe needle to the end of the collector was 14 cm. At the end of the process, 4.0 mL of the polymeric solution were injected into the syringe, and the average film production time was around four hours. The other samples were electrospun in the same way as described.

An important study to characterize the quality of polymeric fibers, produced by the Electrospinning technique, is the analysis of the distribution of their diameters. The fiber diameter distribution in electrospinning is crucial for controlling mechanical properties, porosity, surface area, drug delivery, electrostatic properties, and optical effects of the resulting nanofibers. In this way, it could direct the electrospun fibers to certain applications [9].

The films obtained were sent to the National Institute of Metrology, Quality and Technology (INMETRO) in Xerém, Duque de Caxias, RJ, Brazil, to make measurements with the scanning electron microscope (SEM) Helios NanoLab 650.

RESULTS AND DISCUSSIONS

Figure 2 shows the photograph of sample 2 taken after electrospinning, which is similar to the images of the other samples. The predominant white color is due to the use of the PVP polymer.

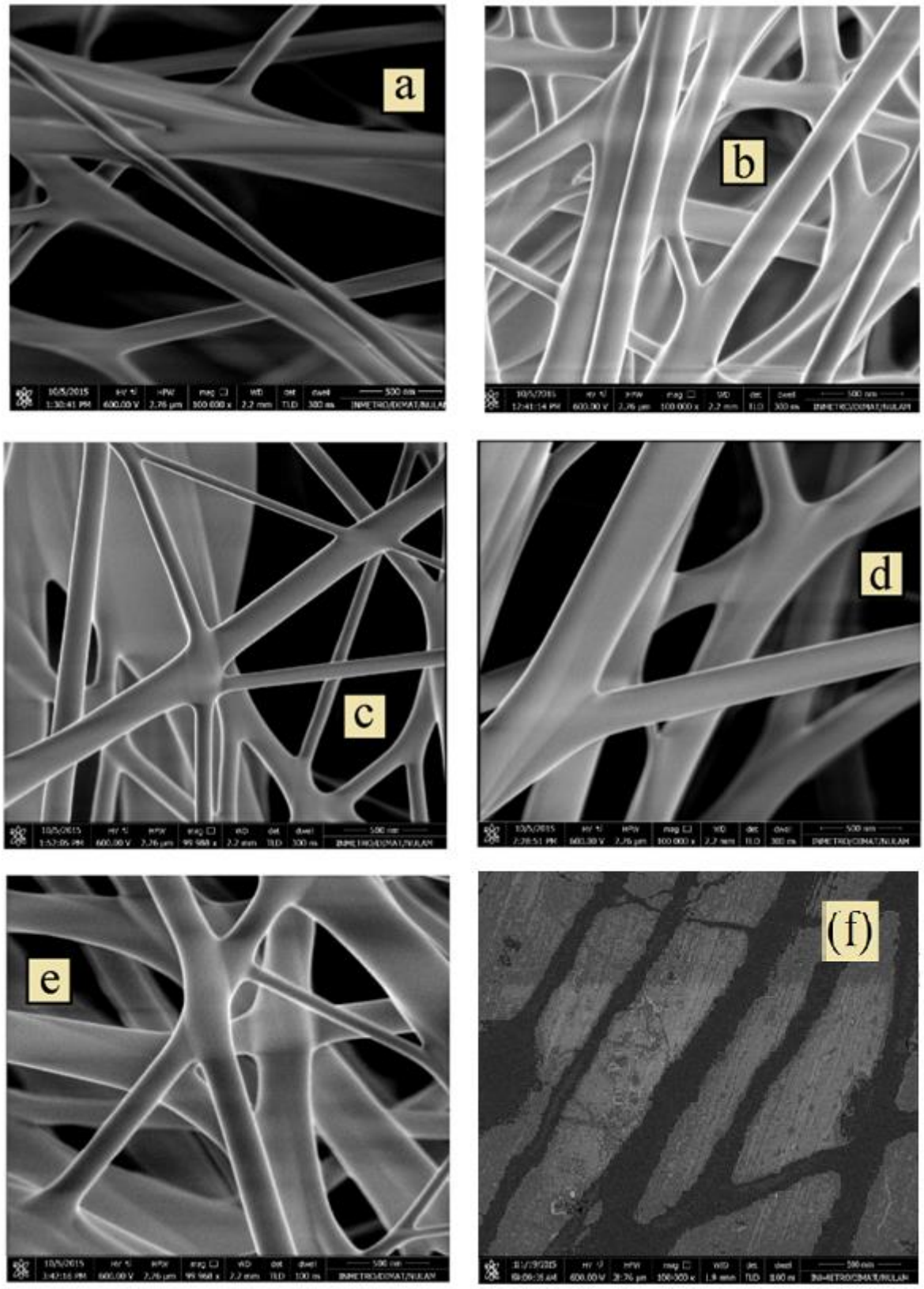
Figure 2 - Photo of sample 2 after undergoing the electrospinning process.

Table 2 displays the parameters used for imaging the six samples by scanning electron microscopy. There was minimal variation in the parameters employed, except for the dwell time, which was reduced to one-third for samples 5 and 6 compared to the others. Additionally, the working distance for sample 6 was decreased by 0.3 mm in comparison to the other samples (this fact occurred due to system vibration and was only observed at the end, as the reduction was minimal in the working distance). Figure 3 presents the microscopy images obtained from all the samples.

Table 2 - Parameters used in microscopy image generation.

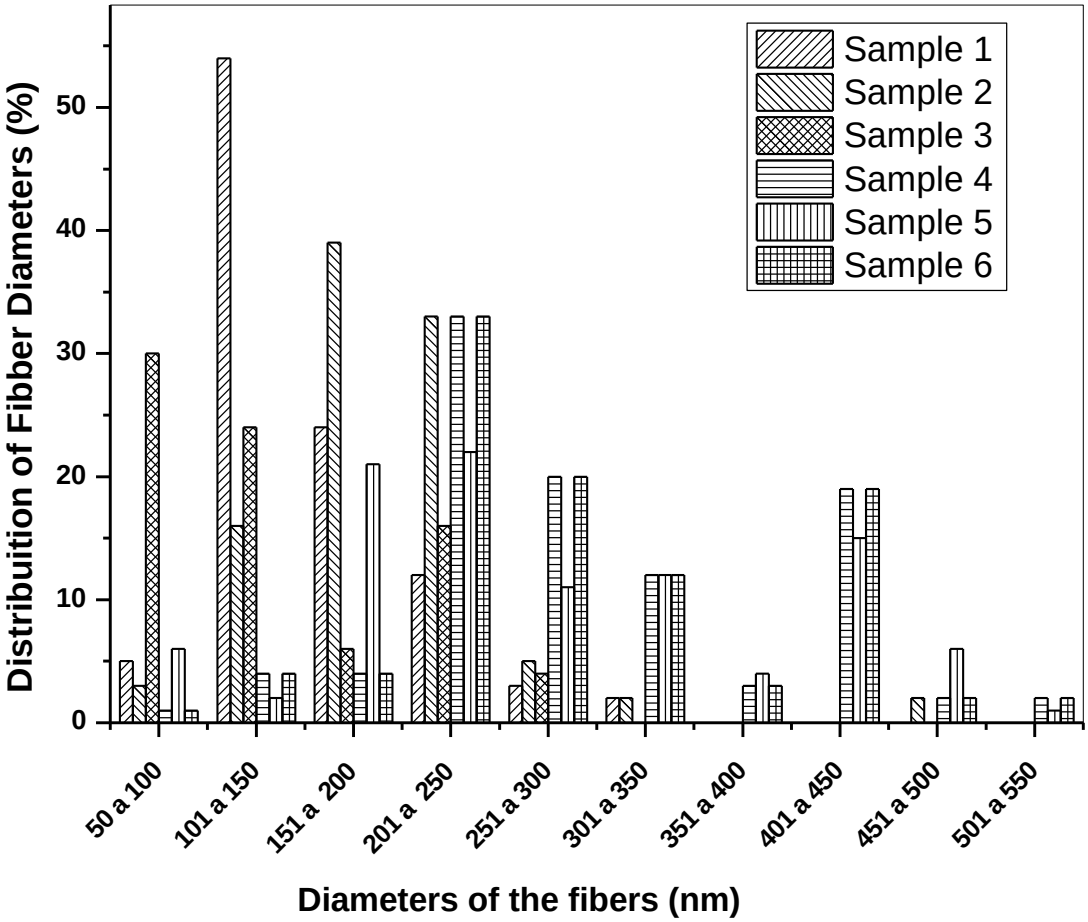
Sample	HV = accelerating voltage (V)	HFW = horizontal field width (μm)	mag = machine magnification	WD = working distance (nm)	det TLD = through the lens detector	dwell (ns)
1	600.00	2.76	100 000 x	2.2	det TLD	300
2	600.00	2.76	100 000 x	2.2	det TLD	300
3	600.00	2.76	99 988 x	2.2	det TLD	300
4	600.00	2.76	100 000 x	2.2	det TLD	300
5	600.00	2.76	99 988 x	2.2	det TLD	100
6	600.00	2.76	100 000 x	1.9	det TLD	100

Figure 3 - Microscopy images of the samples: (a) 1, (b) 2, (c) 3, (d) 4, (e) 5, and (f) 6.



To investigate the distribution of fiber diameters, present in the films, a random measurement of the thickness (or diameter) of 100 fibers was performed using the ImageJ software [10]. The results can be seen in Figure 4.

Figure 4 - Percentage distribution of fiber diameters as a function of fiber diameters in the samples 1, 2, 3, 4, 5, and 6.



The mass concentration of the added plant material in the films resulted in significant differences in the diameter distribution, as observed in Table 3. Sample 4 maintained the pattern of the reference (sample 6), while samples 2 and 5 showed similarity in the range of 201 to 250 nm when compared to the reference as well. The two latter films exhibited a higher percentage of diameter distribution in the range of 151 to 250 nm, although there was a difference in values between them. Films 1 and 3 presented slightly different fiber diameter ranges, 101 to 200 nm and 50 to 150 nm, respectively, but they shared a common range of 101 to 150 nm. Film 1 has diameters

ranging from 50 to 350 nm, while film 3 ranged from 50 to 300 nm, while the other films recorded diameters of up to 550 nm. The increase in crushed *Aloe vera* mass in the polymer solution tends to contribute to a more uniform distribution of fiber thickness and an increase in fiber diameter.

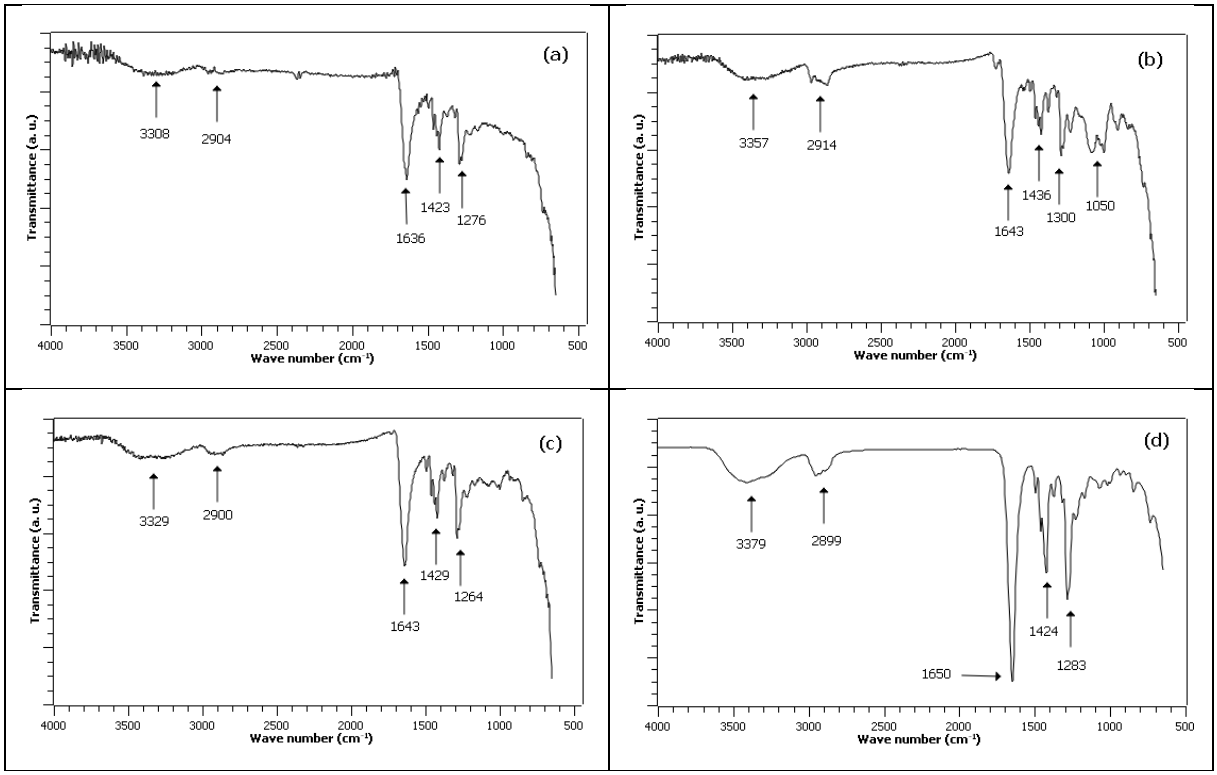
Table 3 – Range and percentage of diameter distribution in the samples.

Sample	Range (nm)	Percentage (%)
1	101 to 200	78.0
2	151 to 250	72.0
3	50 to 150	67.5
4	201 to 300 and 401 to 450	72.0
5	151 to 250	43.0
6	201 to 300 and 401 to 450	72.0

The infrared spectrum is characteristic of a molecule, but certain groups of atoms produce bands that occur at approximately the same frequency, regardless of the molecular structure. The presence of these bands from specific groups of atoms is crucial for identifying structural information in polymeric films, as in the samples studied in this work.

The infrared spectroscopy measurements of samples 1, 2, 3, and 6 were conducted in partnership with the Federal Police of Rio Branco, Acre. The equipment was the Smart Multi-Bounce HATR Accessory. Figure 5 displays the infrared spectra of these samples.

Figure 5 - Infrared spectra of the samples: (a) 1, (b) 2, (c) 3, and (d) 6.



The infrared spectroscopy graphs showed similarities in the line shapes of all films. The functional analysis of the compound revealed several vibrational characteristics. In the range of 1636 to 1650 cm^{-1}, a strong stretching of the C=O functional group was observed, indicating its presence. In the ranges of 3308 to 3379 cm^{-1} and 2899 to 2914 cm^{-1}, medium stretches of N-H were identified, suggesting the presence of primary and/or secondary amine. Between 1264 and 1300 cm^{-1}, a strong stretching of the C-O functional group was observed, along with a strong stretching of O-H in the range of 3650 to 3100 cm^{-1}, indicating the presence of a phenol [11].

In the range of 1636 to 1650 cm^{-1}, a strong to medium stretching of C=C was observed, indicating the presence of an alkene. At 1050 cm^{-1}, a strong to medium stretching of C-N was identified, indicating the presence of an alkylamine. In the range of 2899 to 2914 cm^{-1}, a medium to strong stretching of C-H was observed, suggesting the presence of an alkane, along with the medium asymmetric deformation of the CH3 group around 1450 cm^{-1}. A peak at 1050 cm-1 was only evidenced in sample 3, indicating a strong stretching of C-O, indicative of an aryl-alkyl ether [11].

In the electrospinning study by Uslu and Aytmur [5], an aqueous solution containing poly(vinyl alcohol)/poly(vinylpyrrolidone) iodine/poly(ethylene glycol) with

(hydroxypropyl)methyl cellulose (HPMC) and *Aloe vera* was used. They identified a broad peak at 1096 cm^{-1}, indicating the stretching vibration of the C-O group, and an absorption peak at 1657 cm^{-1}, characteristic of the C=O group. Despite involving the addition of a different polymer and HPMC in the solutions, there is a similarity with this work in the identification of the absorption bands.

CONCLUSION

The objective of this study was to characterize samples made with polyvinylpyrrolidone polymer, ethanol, and Aloe vera leaves crushed by the electrospinning method, opening the possibility of making a wound dressing. Six samples were produced and analyzed using scanning electron microscopy and infrared spectroscopy.

The objective of this study was to develop a wound dressing using polyvinylpyrrolidone as a base polymer, ethanol, and crushed *Aloe vera* leaves through the electrospinning method.

The analysis of the percentage distribution of fiber diameters revealed that an increase in the amount of Aloe vera leaves in the solution resulted in a more favorable fiber distribution and an increase in fiber diameter. A favorable fiber distribution is important to ensure uniform wound coverage. The increase in fiber diameter can contribute to the absorption capacity of exudate, which is part of the wound healing process as it helps maintain a moist environment and promotes tissue regeneration [12]. These results can help in controlling fiber diameters, which are essential for the properties and effectiveness of the wound dressing.

The infrared spectra showed the presence of functional groups in all samples, such as primary and/or secondary amine, phenol, alkene, alkylamine, and alkane; however, the aryl-alkyl ether group was only evidenced in sample 3. Phenols can help in the prevention and treatment of skin diseases, such as the early effects of aging, skin diseases, and severe skin damage in the form of lesions [13].

The microscopic and spectroscopic analyses performed provided valuable information regarding fiber distribution and the presence of functional groups, contributing to the understanding of the composition and properties of the films.

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