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Article

Study of the impact of polymeric solution preparation parameters on the production of nanofibers based on PAN and PVA

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Abstract: The search for the development of nanostructured materials has led to the study of the properties of their precursors. For the production of nanofibers by the electrospinning process, it is necessary to determine the rheological parameters of the precursor solutions. Since these properties can be influenced by the processing variables and chemical composition of the polymer, this study aims to elucidate the effect of the addition of vinyl monomers in the formulation of nanofibers based on polyacrylonitrile and to determine the optimal parameters for the production of the precursor polymer solution. The effects of temperature and addition of vinyl monomers were evaluated by rheometry, from the analysis of the variation of the viscosity of the solutions, and by microscopy, the morphology of the nanofibers produced. It was observed that the increase in the temperature used to produce the solutions improves the fibers' properties. Still, there is a relationship between the time of exposure of the polymeric solution to the temperature and the homogeneity of the fibers, which cannot exceed 45 min. The addition of vinyl monomers, to produce PAN-PVA co-polymeric fibers, increases the conductivity and reduces the viscosity of the solutions, resulting in more refined and homogeneous fibers.

Keywords: nanofiber; copolymer; PAN; PVA; electrospinning; rheometry

1. Introduction

The Fourth Industrial Revolution is unlike anything humanity has ever experienced. New technologies allow the fusion of the physical, digital, and biological worlds to create promising solutions to old problems. Materials engineering greatly influences this process since it studies technologies that enable the execution of such fusions. Although there are various nanostructured materials like nanoparticles, nanodots, nanoleaves, nanorods, nanoflowers, etc. [1–3].

At the forefront of the development of nanostructured materials is the production of polymeric nanofibers, which, with their unique characteristics, manufacturing methods, and adjustable properties, have become a much-explored area for application in the most diversified areas such as aeronautics and automobiles [4], sensors [5], tissue engineering [1], catalysis [6], energy devices [7], environment [8], pharmaceutical [9], food [10], filtration [11], distillation [2], etc.

There has been a growing interest in developing nanostructured composites from inserting reinforcement fibers produced by electrospinning techniques. However, the market interest in these fibers goes beyond the already-known structural applications [4,10–12]. PAN and PVA-based copolymer nanofibers are promising for biomedical applications due to the biocompatible and biodegradable character of the monomers [13–16].

Electrospinning can produce these, where nanofibers are obtained from precursor polymer solutions. Electrospinning is one of the most promising methods for producing nanofibers. This versatile technique allows process adaptations that enable the production of fibers with optimal morphological characteristics for the desired application.

Electrospinning is an easy and versatile method to prepare nanofibers (NFs) using natural and synthetic polymers and a high-voltage power supply [17–19]. First reported in the 30's by Formhals [20], electrospinning presented itself as a versatile, multifunctional, and relatively simple method [21] since, for the production of nanofibers, it is only necessary to have a spinneret with a metal needle, a syringe pump, a high voltage power supply, and a grounded collector to mount the electrospinning apparatus [22,23].

The primary mechanism of this technique involves a complicated electrophysical relationship between the polymeric solution and the electrostatic force [24] to produce ultra-thin polymeric fibers with micro to nanometer diameters [25,26]. Electrospinning generates a high-voltage electric field between the injection needle and the collecting apparatus [27]. During the controlled injection process, a drop of polymeric solution is formed in the region of the needle tip [28]. The Taylor cone is formed from this droplet, which is the result of the conical stretching of the polymer droplet with an increase in the surface charge on the polymer droplet over time, increasing the voltage [29]. A polymer jet begins to form shortly after it exceeds the surface charge of the polymer droplet. As the solvent in the polymer jet evaporates, the surface charge of the jet increases, which causes its instability [30]. To compensate for this instability, the polymer jet splits geometrically, initially into two jets and eventually into many jets. This division is caused by the electrostatic force acting on the polymer droplets in constant fragmentation, resulting in the characteristic pattern of nanofibers.

Determining the electrospinning parameters and the rheological and electrical properties of the solution allows the control of the shape and morphology of the fibers. The solution behavior of these materials results from the intermolecular interactions of solubilized oligomers and solvent molecules and the physicochemical properties of the components. They are commonly reported as "unstructured" materials, but many polymers have interactions that are responsible for organizing polymer chains in solutions. These interactions can generate the alignment of the chains, the formation of micelles, weak intermolecular bonds between the chain and the solvent, and the formation of clusters, which directly influence the electrospinning process, impacting the physical and morphological characteristics of the fibers obtained [31].

The composition, average molecular weight, and tactics of the polymers strongly influence the behavior of the polymeric precursor solution of electrospun nanofibers. The viscosity, electrical conductivity, and surface tension of polymeric solutions influence the morphology and geometry of the nanofibers produced [32].

To obtain uniform nanofibers, the viscoelasticity of the solution must be minimal, and this parameter is directly proportional to the diameter of the fibers obtained. The low viscoelasticity of polymeric solutions is achieved with the low concentration of chains in the solution; However, this low concentration results in capillary instability in the electrospinning jet, generating an electrostatic scattering causing the deposition of polymeric granules in the middle of the nanofibers [7].

Controlling the solution's surface tension can mitigate bead formation in the process. Taylor cone formation occurs when the applied potential exceeds the surface tension of the polymer solution, and the lower the solution's surface tension values, the lower the probability of bead formation.

The average diameter of the fibers produced depends on the electrical conductivity of the polymeric solution because, with the application of the external electric field, the charges are oriented, allowing the droplet to undergo a greater elongation, resulting in less thick segments and reducing the diameter of the fibers. Other factors influencing nanofibers' cross-geometry and morphology are ambient temperature, humidity, and air composition. The chemical nature of the polymer, combined with the presence of moisture in the electrospinning environment, can alter the diameter of the nanofibers. Moisture can generate porosity in the nanofibers produced in hydrophilic polymers [31].

Some parameters can be controlled and determined to obtain fibers with the desired morphologies. Among them, it is possible to list the rate of evaporation of the solvent, the difference in polarity between the components of the solution, electrical conductivity, surface tension, and viscoelasticity of the polymeric solution, in addition to synthetic characteristics such as average molar mass and degree of polymer entanglement and environmental parameters such as temperature and humidity [4].

This work is the first to compare the morphology of homopolymeric PAN nanofibers and copolymeric PAN-PVA nanofibers obtained using different parameters of solution preparation and electrospinning.

2. Methods

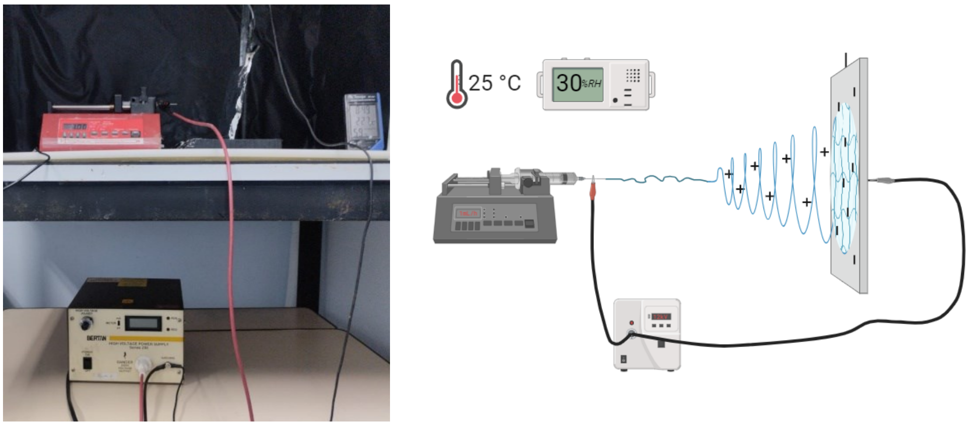
Polymeric solutions with 10% W Polyacrylonitrile (PAN) (Quimlab) and Polyacrylonitrile-co-Vinyl Acetate (PAN-PVA) (Quimlab) in N,N-dimethylformamide (Neon) were prepared by varying temperature and solubilization time, as shown in **Table 1**.

Table 1. Preparation parameters of polymeric solutions.

Sample code		Temperature (°C)	Prepare Time (min)
PAN	I	50	60
	II	80	15
	III	80	30
	IV	80	45
	V	80	60
PAN/PVA	I	50	60
	II	80	15
	III	80	30
	IV	80	45
	V	80	60

The solubilization of the polymeric mass is carried out in the previously heated solvent under magnetic agitation. To verify the influence of the solution's homogenization time on the morphological characteristics of the nanofibers, the stirring time of the solutions prepared at 80 °C was varied, as shown in **Table 1**. Stabilization of polymeric solutions is carried out at rest under refrigeration (4–5 °C) overnight [33].

Electrospinning was performed in an environment with a temperature of 25 °C and air humidity below 30%. The setup consisted of a high-voltage power supply, an electrically conductive spinneret (hypodermic needle), a syringe pump, and a conductive collector plate. A schematic of the setup was created at biorender.com and shown in **Figure 1**.



(a) **(b)**

Figure 1. (a) Picture of the experimental equipment under working conditions; **(b)** Schematic of the assembly of the experimental apparatus for electrospinning.

The nanofibers were deposited on a stationary copper plate covered in aluminum foil. The needle selected for extruding the polymer solution had an inner diameter of 0.2 mm. The electrospinning parameters were constant throughout the experiment, having a needle tip-to-collector distance of 10 cm, applied voltage of 12 kV, and solution flow rate of 1.0 mL/h [34].

The polymeric solutions studied were analyzed using a rotational test on a rheometer (Thermo Scientific HAAKE MARS rheometer). Viscosity was determined from shear stress versus strain rate curves at 25 °C, using a cylindrical rotor; 100 complete cycles with 300 s were applied. The electrical conductivity of the solutions was determined using a conductivity meter (Láctea, model mCA 150, Brazil), with measurements performed at room temperature (25 ± 2 °C). The viscosity and conductivity determinations of the samples were performed in triplicate, and the means were compared by Tukey’s test for a significance level of 5%, using analysis of variance (ANOVA) in the Origin 8.5 software. The nanofibers’ morphology was evaluated by scanning electron microscopy (SEM) in a Zeiss microscope model EVO MA10.

3. Results and discussions

Rheological analyses performed on polymeric solutions indicated the Newtonian behavior of the polymer/solvent system, expressed for Pearson correlation coefficient (*R*) in **Figure 2**. Therefore, considering the effect of the power phenomena described by Ostwald de Waale, the rheological parameters need not be analyzed [35].

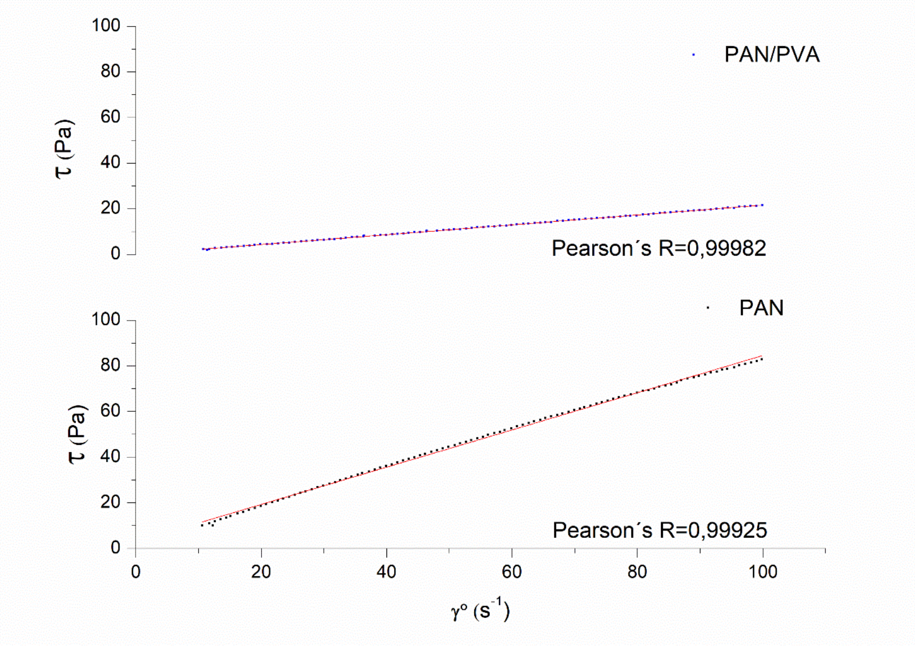


Figure 2. Rheological results of the behavior of the fluid studied.

Polymer-solvent interactions are of great importance in the electrospinning process and, consequently, in the diameter and morphology of the resulting fibers. The initial step of the electrospinning process involves preparing the polymer solution by dissolving the solid precursors in a solvent. Ideally, stable solutions should be obtained which do not undergo significant changes in viscosity over time [31]. Polymer solubilization is a slow process that involves two steps; obtaining an accurate solution is preceded by the gel swelling step. In this first stage, the solid tends to swell when it meets the solvent, which penetrates the polymeric mass's interior through the diffusion of the molecules. As the diffusional process of the solvent continues, the swelling gel disintegrates, with the consequent formation of a true solution [36].

Solubility is related to the cohesive energy of polymers and solvents. Cohesive energy is required to remove a molecule from its environment and from its neighbors. Thus, cohesive energy is associated with evaporation for solvents, while cohesive energy is associated with solubilization for polymeric solids. It is a measure of the level of intensity of secondary forces [36].

Among the basic rules of polymer solubilization is the importance of chemical and structural similarity between the polymer and the solvent and the directly proportional relationship between the solubility and the preparation temperature of the solution for a given polymer/solvent pair [37,38].

One way to quantify this energy is through generalized solubilization parameters (δ), which are calculated from the relative contributions to dispersion forces, hydrogen bonds, and dipole-dipole interactions. For solubilization to occur, the modulus difference between the solubility parameter of the polymer and the solvent must be as slight as possible, i.e., they must have chemical and structural similarities. **Table 2** shows the values of the theoretical solubilization parameters for the homopolymers and solvents studied in this work [36].

Table 2. Values of the theoretical solubilization parameters for materials used.

Compound	δ (g·cm^{-3})
DMF	12.14
PAN	15.42
PVA	9.43

In materials science, a general rule of mixtures is a weighted mean used to predict various properties of a material. It provides a theoretical approximation of final properties. Thus, with the addition of vinyl monomers in the formulation of the PAN/PVA copolymer, the solubilization parameters are expected to be reduced, approaching the value presented by the solvent.

Figure 3 shows the lower viscosity of the copolymeric solution when compared to the homopolymer solution, which is explained by the higher affinity between the solvent and the solute that reduced the viscosity of the prepared PAN/PVA solutions, regardless of the preparation conditions.

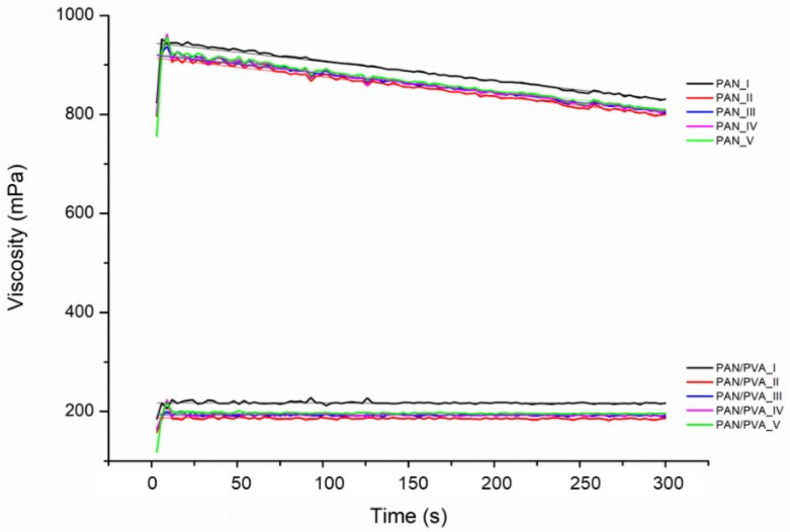
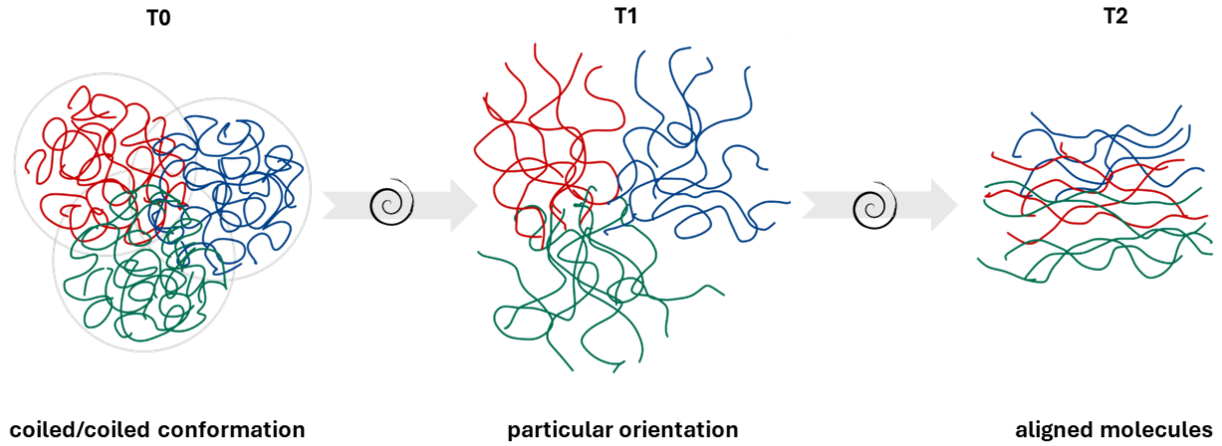


Figure 3. Viscosity behavior over shear time.

Viscoelasticity is a time-dependent phenomenon that occurs in polymer systems. When it comes to dilute solutions, such as those studied in this work, it is possible to verify that at rest, the solute molecules assume a coiled/coiled conformation and random configuration, a condition of equilibrium with lower energy. Once the solution is sheared, these molecules move away from equilibrium. This effect occurs even if, initially, each molecule assumes a particular orientation under the stress; with the maintenance of the shear effort, the molecules will align with the neighboring molecules, reducing the randomness of the distribution and giving rise to viscoelastic behavior [39].

Figure 4 schematically shows the behavior of the polymeric molecules under agitation, which directly influences the viscosity of the studied solutions as a function of time. The analytical results obtained by rheometry indicate that the solutions prepared with the PAN/PVA copolymer presented the lowest viscosities and lowest time dependencies. In addition to the generalized solubilization parameters (δ)

optimized, this comportment can be elucidated too due to the greater chemical and structural similarity between the polymer/solvent pair presented by the copolymer that with the mixture, even if in a low proportion, inserts to the polymeric structure double bonds with oxygen to the polymeric molecule, resembling it even more closely with the n,n-dimethylformamide molecule, **Figure 5**.



NOTE:
* Consider homopolymer. The different colors aim to facilitate visualization of the alignment stages under shear.
* shear time : T2>T1>T0
* ◎ : solution shear

Figure 4. Illustrative scheme of the behavior of molecules in solution when subjected to shearing over time.

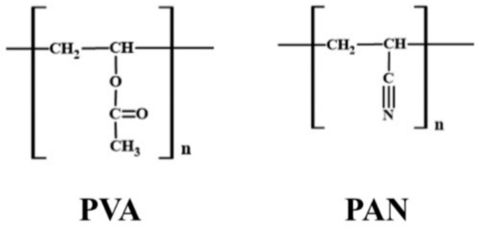


Figure 5. The structural formula of mere PVA and PAN.

In the results presented in **Figures 6** and **7**, it is observed that the increase in the temperature of the preparation of the solutions implies a reduction in the viscosity found. In both cases studied, homopolymeric (PAN_I and PAN_V) or copolymeric (PAN/PVA_I and PAN/PVA_V) solutions, there was an approximate 10% drop in the measured viscosity, **Figure 8**, this occurs since for a polymer/solvent pair the solubility is increased with the increase in temperature, providing greater mobility between the chains.

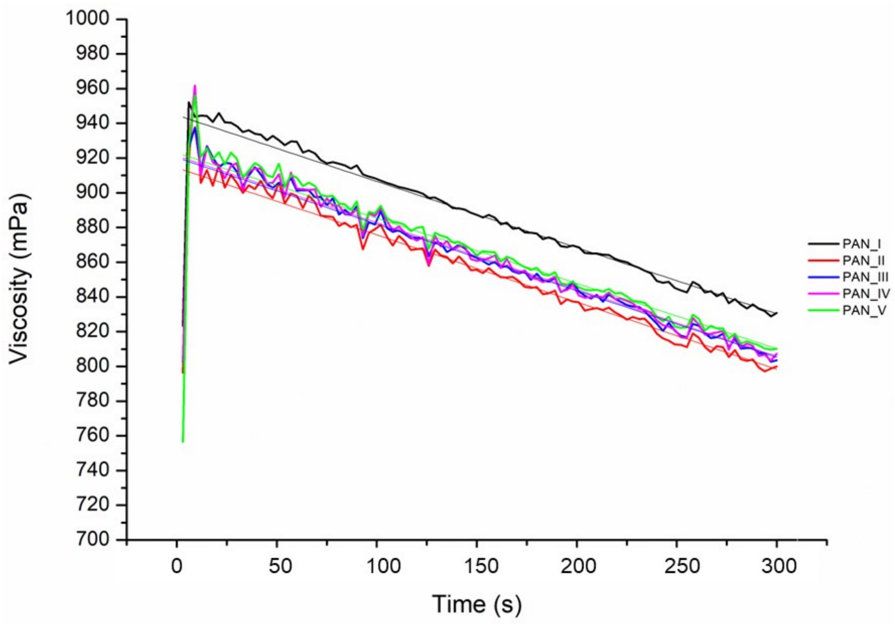


Figure 6. Rheological behavior of PAN solutions at different preparation temperatures.

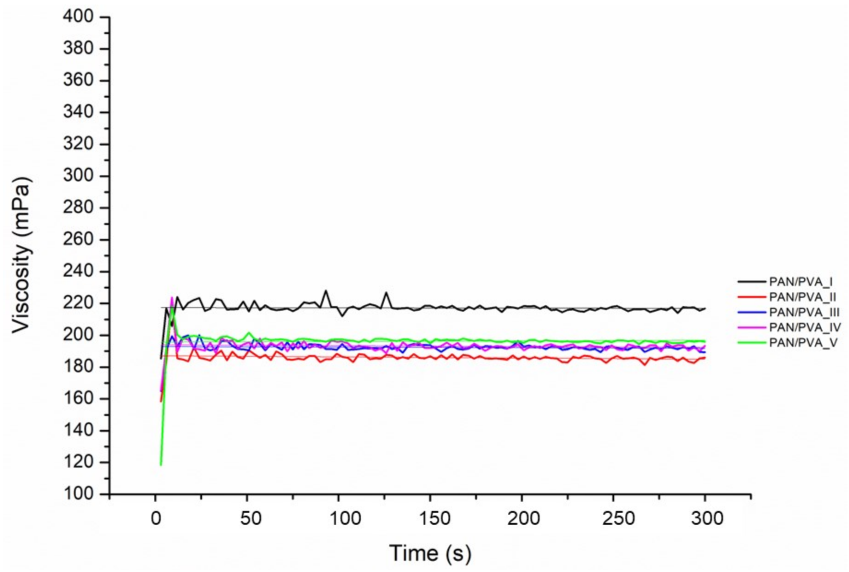


Figure 7. Rheological behavior of PAN/PVA solutions at different preparation temperatures.

When comparing the preparation times of the solutions at 80 ℃, **Figure 8**, it is observed that, in both cases, as the preparation time is increased, the viscosity increases, possibly because the second stage of polymer solubilization has been reached, where homogeneity is achieved in the distribution of the polymeric chains in the organic solution.

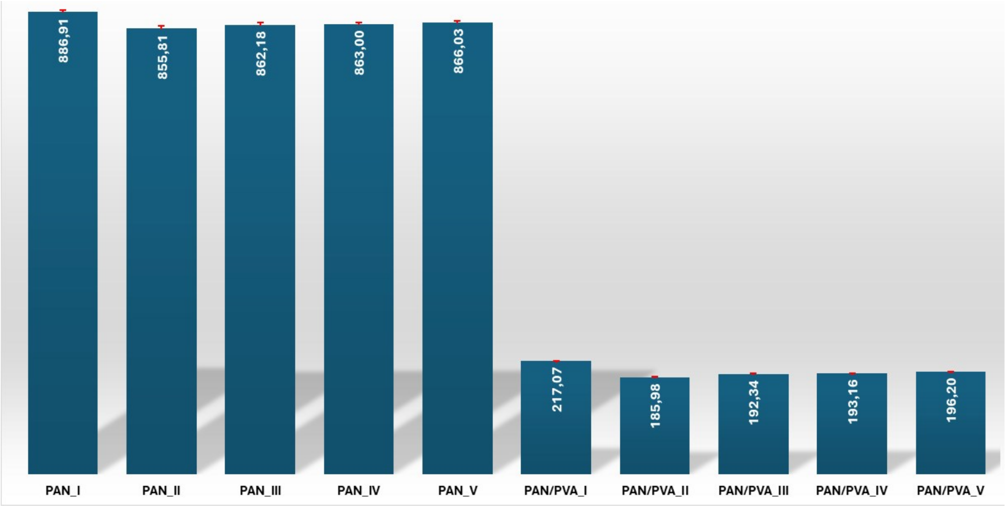


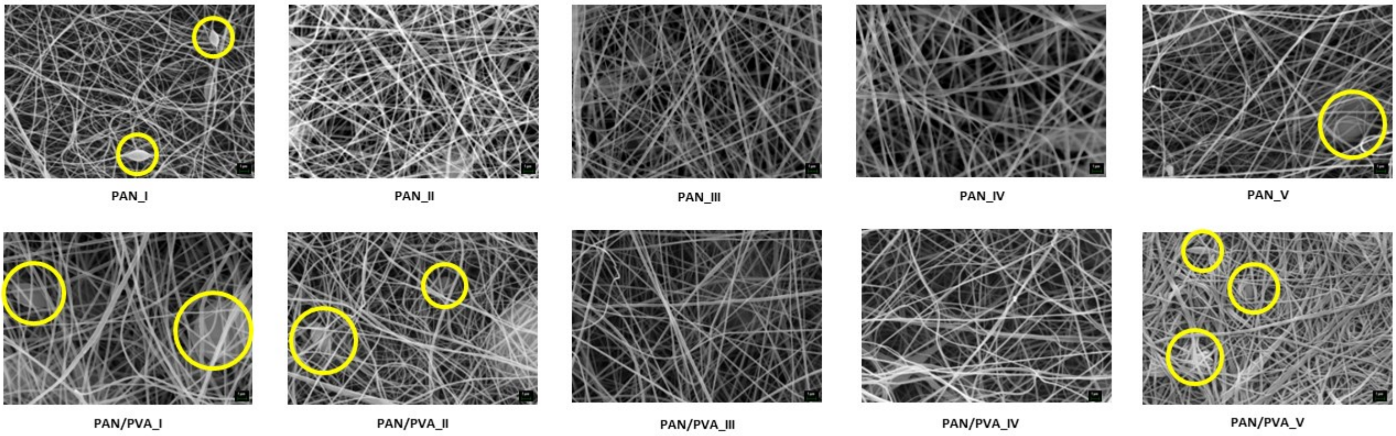
Figure 8. Comparative of the average viscosity of the solutions studied.

The viscosity of the polymeric solution influences the morphology of the electrospun nanofibers since the fibers' homogeneity is obtained from forming a continuous jet without rupture from the tip of the capillary to the collector [38]. A lower viscosity generally favors the formation of finer and more homogeneous fibers. However, if the viscosity is too low, the entanglement of the polymer chains is insufficient for the formation or maintenance of the jet since the interactions between the polymer chains are too weak to overcome the Rayleigh instability. In this case, there will be a greater tendency to spray, producing fibers with defects in the form of beads, which are polymeric agglomerates. As the concentration increases, the jet will no longer break, and the shape of the beads will change from spherical to spindle-like, eventually forming uniform fibers due to increased chain entanglement and viscosity. If the viscosity is too high, it becomes difficult to eject the solution from the spinneret, and polymeric clusters may form in the collector, resembling beads [32].

The polymer concentration effect is quite interesting. It can be noted that the viscoelastic properties of concentrated solutions say those in the range of 15%–20%, are quite typical for viscoelastic solutions. Thus, in the solutions studied, with a concentration of 10%w, the storage module is independent of the frequency, with no implications in the results due to variations in the injection rates during the electrospinning process [40].

On the other hand, the change in the formulation, with the addition of vinyl monomers, resulted in a change in the conductivity of the solutions studied, which influences the morphology of the fibers produced. As the liquid's electrical conductivity increases, the operational flow rates decrease, and the jet becomes thinner and shorter. The higher electrical conductivity of the solution allows the droplet to undergo greater elongation with the application of the electric field, resulting in fibers with a smaller average diameter [41]. The jet is short and the at high conductivities, resulting in refined fibers.

This behavior was observed in the fibers produced in this study since the electrical conductivity measured for the homopolymer was 30.74 µS/cm, and for the copolymer, 52.29 µS/cm. The most refined fibers were those obtained from the electrospinning of copolymer solutions. **Figure 9** shows the micrographs of the fibers obtained from the solutions and preparation conditions studied.



PAN_I PAN_II PAN_III PAN_IV PAN_V

PAN/PVA_I PAN/PVA_II PAN/PVA_III PAN/PVA_IV PAN/PVA_V

Figure 9. Micrographs of the nanofibers were obtained by electrospinning the solutions studied.

It is observed that the fibers obtained from solutions prepared at 50 °C presented nanofibers with bead formation because of the higher viscosities of these solutions. In addition, the copolymeric nanofibers showed irregularity in their diameters, presenting a wide distribution of fiber diameter.

Since the reduction of the viscosity of the solutions was observed with the increase of the temperature of preparation of the solutions, the systematic study of different preparation times showed that this factor also influences the viscosity of the solutions and, consequently, the morphology of the fibers. It was noted that with the increase in exposure to temperature and shear, there was an increase in the viscosities presented, so it is expected that the fibers obtained from the solutions prepared for shorter times will present a more homogeneous morphology and free of beads. This was true for the homopolymer fibers since it is observed that the diameter of the fibers tends to increase with the increase of the solution preparation time, culminating in the formation of beads in the samples obtained after 60 min of heating and shearing (**Figure 9** PAN_II, PAN_III, PAN_IV, and PAN_V). The formation of beads in this situation may be related to the higher viscosity value. This behavior was not observed in the copolymer nanofibers since even in the lowest exposure time (**Figure 9** PAN/PVA_II), which presented the lowest viscosity, the presence of beads intertwined with the nanofibers was noted; this can be explained by greater difficulty in achieving the stability of the solution due to the different radical groups present in the copolymer. In this case, the nanofibers produced with the solutions prepared for 30 and 45 min (**Figure 9** PAN/PVA_III, PAN/PVA_IV) presented the best morphologies, while due to the higher viscosity, the nanofibers obtained from the solutions prepared for 60 min (**Figure 9** PAN/PVA_V) showed thickening of the diameters and the presence of beads intertwined with the fibers.

4. Conclusions

Based on the above, the addition of vinyl monomers in the formulation of the polymeric solution directly influenced the morphology of the fibers since, with this addition, the viscosity was considerably reduced, and the electrical conductivity of the solution increased, resulting in thinner and more homogeneous fibers. The increase in the preparation temperature by 30 °C in the preparation of the solutions resulted in a reduction of approximately 10% in the final viscosity, implying the production of more homogeneous fibers without forming beads. The exposure time at a temperature of 80 °C showed that times longer than 45 min can lead to the formation of beads, and it is appropriate to apply times between 30 and 45 min of agitation under heating to make the optimal solution for the electrospinning of nanofibers based on PAN.

Author contributions: Conceptualization, methodology, LFPG, AB and EPG; validation, formal analysis, LFPG; investigation, resources, AB; data curation, LFPG and AB; writing—original draft preparation, writing—review and editing, supervision, project administration, EPG. All authors have read and agreed to the published version of the manuscript.

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