



Physical Properties of Nanofibers of Different Concentrations of Polyacrylonitrile

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Abstract: The specific concentration of the polymeric structure composition of polyacrylonitrile was determined and found to start at (5% W). By weight, where several samples of polyacrylonitrile with different concentrations were prepared in a dimethylformamide solution at a temperature of 298 K, the prepared solutions are spun with a high-safety homemade electrospinning device that contains many variables and in specific parameters such as needle distance from the collector, rate from pumping the solution, the speed of the compound, the stabilization of the voltage and the diameter of the needle. The only variable was concentration, and the average spun fiber diameters of samples prepared from polyacrylonitrile were measured with a microscope. Electronic scanning, where it was found that the diameter of the average nanofibers spun from polyacrylonitrile increases with increasing concentration, then some physical properties of the resulting polyacrylonitrile spun nanofibers were studied according to crystallization. The polyacrylonitrile size, streptococcus resistance spectrum, and loss factor of spun samples were studied, and it was found that the studied samples had uniform crystallinity. The relaxation time of samples prepared from polyacrylonitrile nanofibers at a temperature of 298 K was calculated, and by drawing the relationship between the relaxation time and the concentration of the samples, it was found that the relaxation time increases linearly with increasing concentration.

Keywords: Electrospinning, Nanofibers, Polyacrylonitrile, IS

1. Introduction

Rheological properties are an important tool for the characterization of polymeric solutions. The study of the relationship of the viscosity of PAN with its concentration in a medium of dimethylformamide DMF, which is an organic solvent, showed the occurrence of severe association processes in the solution due to the mutual effects between the dipoles [1]. The reason for the widespread production of PAN polyacrylonitrile fibers is due to the following important characteristics: the average molecular mass of the spun PAN, the distinctive physical and chemical properties of its spinning solutions, the relatively cheap polymerization process, and the ease of the heat treatment system. These qualities enable the control of the mechanical and structural properties required for the resulting fibers [2].

Electrospinning technology is considered a scientific revolution, and it is one of the modern and advanced technologies, and it is considered a scientific revolution in the field of producing polymeric nanofibers [3]. The electrospinning mechanism relies on electrostatic forces in the production of polymeric fibers with nanoscale diameters, where these very small structures have the characteristics of important surface and structural properties, such as surface-to-volume ratio, high porosity, and mechanical and optical properties [4]. The technique of electrospinning has begun to acquire a very great interest by researchers, and this can be attributed to the boom that occurred in the field of nanotechnology and its applications, as the number of universities, institutes, and research centers interested in this

technology exceeded two hundred [5]. Figure 1 shows the increasing rate of publication of research papers related to electrospinning applications from 2001 to 2022 [6-8].

This technique allowed the production of nanofibers with a wide range of applications, including: antibacterial by incorporating biocides into electrospinning solutions and wound dressings [4, 8]. It has also been used in the electrodes of solar cells and nanofibers can be used to purify liquids

from particles with diameters less than a micron by filtering through electrically spun nanofibers [9]. Because of the urgent need to search for new sources of pure water, electrospinning is at the forefront of current research to obtain clean water through the treatment of industrial and wastewater by Nano filtration [10], and nanofibers have also contributed to improving the properties of the electrodes of battery cells [11].

Table 1. Factors affecting electrospinning technology.

	The variables that influence the different factors
Factors affecting the polymeric solution	Viscosity, solution concentration, polymer molecular weight, solvent properties, surface tension, and electrical conductivity
Factors affecting electrospinning technology	Applied voltage, distance between injector head and collector, flow rate
External factors	temperature, relative humidity

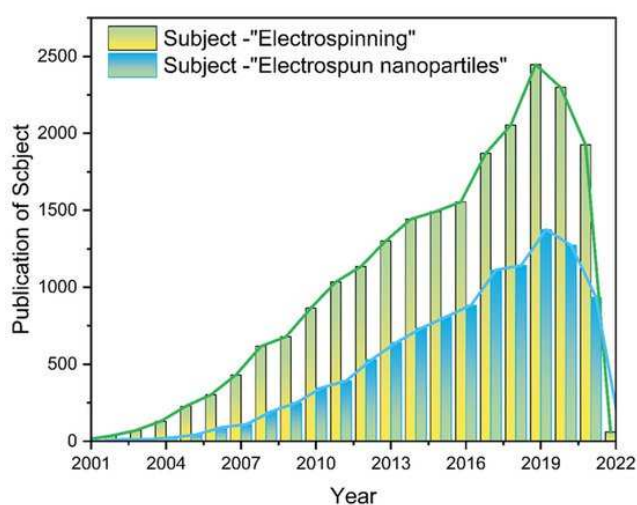


Figure 1. The rate of publication of research on electrospinning.

Although electrospinning is the simplest method for manufacturing polymeric nanofibers, there are some important factors that can have a significant impact on the structure and diameter formation of the generated nanofibers. Table 1 shows the most important factors affecting the electrospinning process: [12, 13].

It is worth noting that the concentration of the polymer is a qualitative characteristic, related to the polymer itself, its molecular mass, and its viscosity [2, 14]. The addition of surface-active colloids strongly affects the structural and mechanical properties of polymeric solutions, because the effectiveness of the mutual effects between the molecules of the polymer itself, is greatly affected by the presence of these materials, and the surface-active colloids also affect the shape and dimensions of the formed nanofibers [15].

Research is accelerating in the last period to study the effect of adding some electrolytes, surface-active colloidal materials on the structural properties of polymer nanofibers prepared by the electrospinning method because of their importance in terms of controlling the diameter of the resulting nanofibers, thus increasing the surface area and increasing their mechanical strength and effectiveness in various applications [16].

2. Materials and Methods

2.1. Materials Used

1. Polyacrylonitrile of known molecular mass is 100,000 g/mol.
2. Dimethylformamide "DMF" solution with purity (GC) (99.5%) from MERCK company.
3. Octadecylamine (C₁₈H₃₇NH₂) ODA from Alfa Aesar.

2.2. Devices Used

1. The German-made falling ball viscosity device from HAAKE company, which complies with ISO 12058.
2. A locally manufactured electric spinning device.
3. X-ray diffraction device (XRD): Where an elevator made of copper was used that gives x-rays of a wavelength $\lambda = 1.5418 \text{ \AA}$, Take measurements between angles $2\theta = (5^\circ - 65^\circ)$.
4. Scanning Electron Microscopy" (SEM), an American-made Tuscan Veca 2 model, in collaboration with the University of Wisconsin in Milwaukee, USA. It has the following specifications: Scanning Electron Microscope Flex SEM 1000 II Accelerating Voltage: 20 kV, Secondary Electron (SE) Image, Magnification: 60,000X, Resolution: 4.0 nm.
5. Impedance Spectroscopy (IS): It is a programmable Gain Phase analyzer from SCHLUMBERGER CORPORATION No. (SI1253) that generates a spectrum of frequencies confined to the 0.01 HZ- (20000) range.

2.3. Method of Work

Solutions of different concentrations of polyacrylonitrile were prepared by dissolving the samples in a dimethylformamide solution by continuous stirring on a magnetic mixer at laboratory temperature, until obtaining transparent polymeric solutions with the addition of ODA at a fixed concentration of ($5 \times 10^{-5} \text{ M}$).

Prepare a solution of octadecylamine ODA at a concentration of $100 \times 10^{-5} \text{ mol/l}$ with a weight of 0.0269g of

ODA, and put it in a volumetric flask with a capacity of 100ml with DMF solution.

3. Practical Results and Discussion

3.1. Viscosity Measurement

The viscosity of the prepared solutions of different concentrations of PAN with a constant concentration of ODA ($5 \times 10^{-5} \text{M}$) was calculated, with the following relationship (1):

$$\eta = (d_1 - d_2) t \quad (1)$$

Table 2 shows the results of measuring the viscosity of the solutions prepared in DMF solution at a temperature of 298K before and after adding a fixed concentration of ODA ($5 \times 10^{-5} \text{M}$), in Table 2.

Table 2. The viscosity of the solutions prepared at a temperature of 298K, before and after adding a fixed concentration of ODA is ($5 \times 10^{-5} \text{M}$).

PAN W%	Viscosity η (mPas)	
	With no addition	With ODA ($5 \times 10^{-5} \text{M}$)
1	1.495	0.886
3	8.831	3.231
5	30.902	14.902
8	109.333	81.333
10	264.968	228.012
12	530.7	468.227
15	1345	1159.5
18	2289	2038

Figure 2 shows the viscosity of the solutions prepared at 298K, before and after adding a fixed concentration ($5 \times 10^{-5} \text{M}$) of ODA.

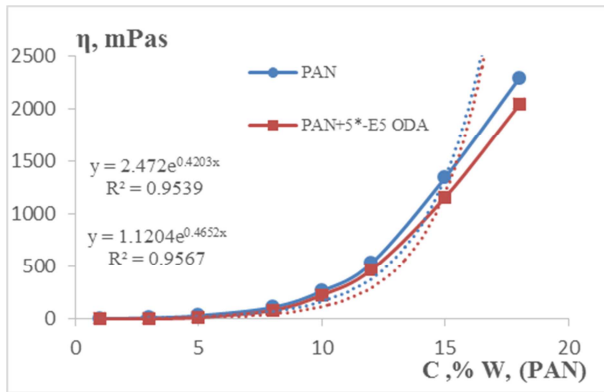


Figure 2. Viscosity of the prepared PAN solutions.

It is observed from Figure 2 that an exponential relationship at high concentrations of $y = A \cdot e^{bx}$ relates the viscosity to the concentration in PAN solutions in the presence and absence of ODA. Where the viscosity increases with concentration linearly and slowly at small concentrations, but it begins to increase significantly starting with the marginal concentration to form the polymeric structure (approximately 5%w), that is, at this concentration, strong bonding bundles of polymeric molecules begin to form due to the presence of dipoles, and hydrogen bonds are formed, which in turn leads to a noticeable increase in

viscosity, and a decrease in viscosity should be observed in general by adding ODA to different concentrations of PAN.

3.2. Electrospinning

The prepared samples were spun with the homemade electrospinning device shown in Figure 3.

The prepared PAN solutions were spun after adding a fixed concentration ($5 \times 10^{-5} \text{M}$) of ODA, and we found the following:

Electrospinning of PAN/DMF solutions of concentrations between (5-18W%) was performed at specific parameters:

Solution flow rate: 0.5ml/h., Voltage lifter: 20 kv. Accumulator rotation speed: 100 cy/min, The distance of the collector from the injector: 10 cm, Diameter of the injector needle: 0.5 mm., Rotary cylindrical assembly.



Figure 3. Homemade electric spinning device.

3.3. A Superficial Study

Using a “Scanning Electron Microscopy”. SEM, an American-made Tuscan Veca 2 model, a surface image of the prepared fibers was taken. Figure 4 shows the SEM electron microscopy images of the samples where the applied voltage is 5 kv and at a magnification of 15000. We conclude that the diameters of the resulting nanofibers range from (100-500) nm and Table 3 shows that the average diameters of the resulting nanofibers increase with increasing concentration.

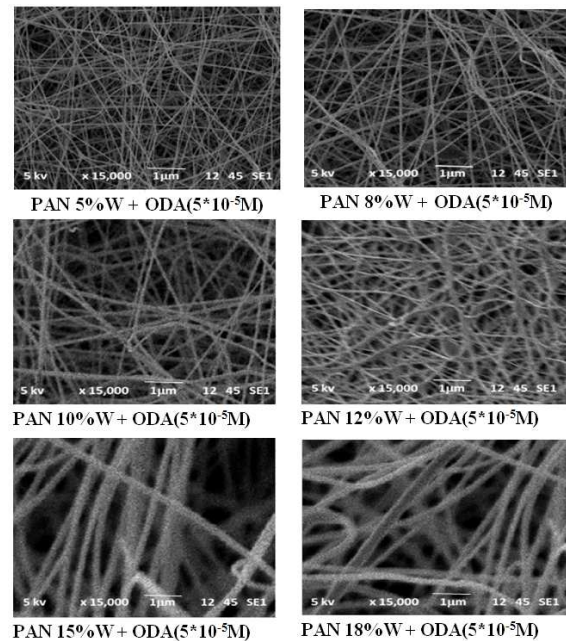


Figure 4. SEM electron microscopy images of spun PAN samples.

Table 3 shows the increase in the average diameters of

nanofibers for the prepared samples with increasing concentration.

Table 3. The average diameter of nanofibers for samples prepared at the concentration.

PAN, C % w+ ODA ($5 \times 10^{-5} \text{M}$)	5	8	10	12	15	18
Av. fiber diameter (nm)	115	155	200	255	340	450

Figure 5 shows the average diameter of the nanofibers of the prepared samples in terms of the concentration, and that the relationship between the average diameters of the nanofibers of the spun samples and the concentration is a linear relationship of the figure $y = 26.093x - 43.219$ due to the increase in the viscosity of the spun polymer solution [17]. As for the concentrations (1,3%w), spinning was not done because the polymeric structure of polyacrylonitrile was not formed, and therefore the crystal structure of polyacrylonitrile begins to form at a concentration of 5% W of its concentration in DMF [18].

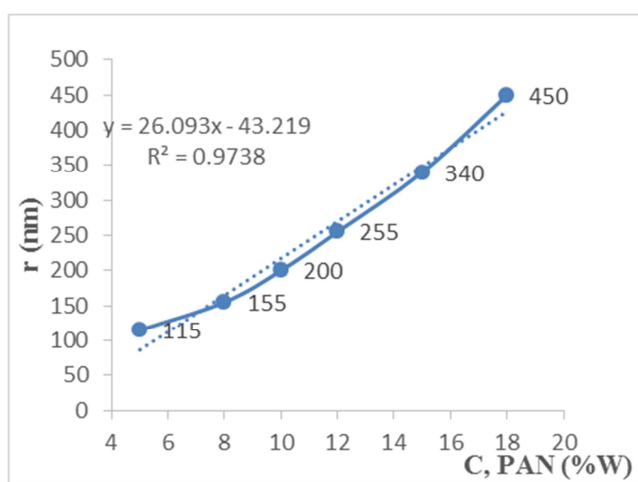


Figure 5. The average diameter of the nanofibers of the prepared samples as a function of concentration.

3.4. X-ray Diffraction Spectrum (XRD)

The X-ray diffraction spectrum (XRD) was studied using an anode-made copper X-ray spectrometer within an angular range of (5° - 65°), for samples spun at room temperature $T = 303 \text{ K}$ in order to know the structural properties of the prepared sample. Figure 6 shows the X-ray diffraction spectrum of the samples spun.

The crystal structure was determined based on XRD studies. In all samples, the characteristic peaks of polyacrylonitrile were in the presence of ODA at peak $2\theta_{max} \sim 17$, which was characteristic of the hexagonal structure of PAN [19], and because of the strong interaction between the polymer chains and the surfactant ODA that hinders their movement, thus limiting the process of increasing the crystallization size.

3.5. Determining the Average Size of the PAN Crystals

The average size of the PAN crystals in the presence of ODA was determined from the XRD spectrum shown in Figure 6, from the mid-width of the X-ray diffraction peak intensity, in the hexagonal plane of PAN [20] and Figure 6 shows the X-ray

diffraction of the prepared samples, where a peak is observed at $2\theta_{max} \sim 17$ of the hexagonal plane of the PAN.

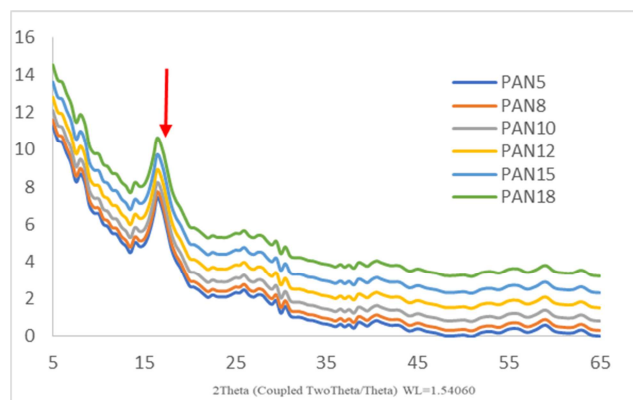


Figure 6. XRD diagrams of the prepared samples.

Using the following Debye-Scherrer (2) relationship [19, 21]:

$$\beta = \frac{\kappa \cdot \lambda}{L_{hkl} \cdot \cos \theta_{max}} \quad (2)$$

where: L_{hkl} average size of crystallization size β mid-intensity width of the plane-corresponding diffraction peak in one radian, κ : constant 0.89 in the case of crystal particles, $\lambda = 1.5401 \text{ \AA}$ X-ray wavelength, θ_{max} plane-corresponding diffraction angle peak (maximum peak).

Thus, L_{hkl} is given by the following relationship (3):

$$L_{hkl} = \frac{\kappa \cdot \lambda}{\beta \cdot \cos \theta_{max}} \quad (3)$$

Where: $\beta(\text{Rad}) = 0.026$ as per the XRD plots of Figure 6.

The distance R between the polymeric chains of PAN polyacrylonitrile and the surfactant octadecylamine ODA was calculated, depending on the peak of the crystalline peak from the following relationship (4) [21]:

$$R = \frac{5 \cdot \lambda}{8 \cdot \sin \theta_{max}} \quad (4)$$

From the Bragg relationship, the distance d between the polymeric chains within polyacrylonitrile (the spacing) was determined from the relationship (5):

$$n\lambda = 2d \sin \theta \quad (5)$$

where n is an integer of linear deviation.

The strain of the polymeric network (crystalline) ϵ was calculated from the following relationship (6):

$$\beta = 4 \epsilon \tan \theta \quad (6)$$

The data on the crystal structure of the spun samples of pure polyacrylonitrile [20] were collected and compared with polyacrylonitrile by adding ODA in the following table 4:

Table 4. Shows the crystallization volume decrease for PAN by adding ODA.

sample	Diffraction Peak (2)	L (nm)	d-Spacing (Å)	R (Å)	ϵ
PAN [25]	16.85	5.7	3.95	6.58	0.00092
PAN+ ODA	17	5.27	3.907	6.511	0.00099

The results in Table 4 show a decrease in the crystallinity of polyacrylonitrile samples by adding octadecylamine compared with pure PAN, and consequently a decrease in the distance between the polymeric chains d.

3.6. AC Measurements (Septal Impedance IS)

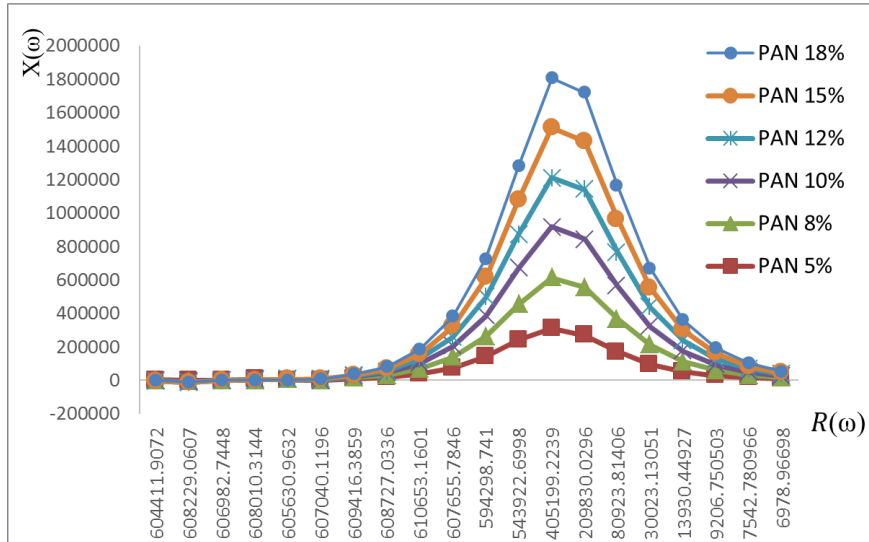
One of the most important methods for characterizing the electrical properties of materials is Impedance Spectroscopy (IS). To use this technique to study and characterize the behavior of films in composite nanofibers [22]. Where some coefficients affecting the electrical behavior of the material are calculated based on the nodal impedance spectrum, by applying an alternating current so that the studied sample placed between the condenser bulbs is connected to a gain

phase analyzer with detection resistance $R_d = 320 \Omega$, and an alternating signal of variable frequency is applied to the material. The Septal impedance is expressed by the following relationship (7): [23]

$$Z(\omega) = R(\omega) + j X(\omega) \quad (7)$$

Studying the effect of changing the concentration of the PAN samples at a constant temperature $T=303K$ and a constant voltage $V=4V$ on the nodal impedance spectrum IS:

The nodal impedance spectra were taken for the samples prepared from PAN and after adding a fixed concentration ($5 \times 10^{-5}M$) of ODA, at a constant temperature $T=303 K$ and a constant voltage $V=4$ Volt as shown in the following Figure 7:

**Figure 7.** The sum of the nodal impedance spectra of the six samples prepared from PAN and after adding a fixed concentration ($5 \times 10^{-5}M$).

It is noticed from Figure 7 that the previous spectra consist of a regular and almost identical circular arc for all samples, which indicates that the material has regular crystals and that all dipoles have the same relaxation time for each of the studied samples [24].

3.7. Relaxation Time τ_p

The relaxation time τ_p was calculated for samples prepared from PAN and after adding a fixed concentration ($5 \times 10^{-5}M$) of ODA, at a constant temperature $T=303 K$ and a constant voltage $V=4$ Volt as shown in the following table 5 using the following relationship (8):

$$\tau_p = \frac{1}{f} \quad (8)$$

By taking the value of the frequency corresponding to the highest value of (ω) .

Figure 8 presents the relationship of relaxation time with the concentration of the studied samples at a constant temperature $T = 303 K$ and a constant voltage $V = 4$ Volt.

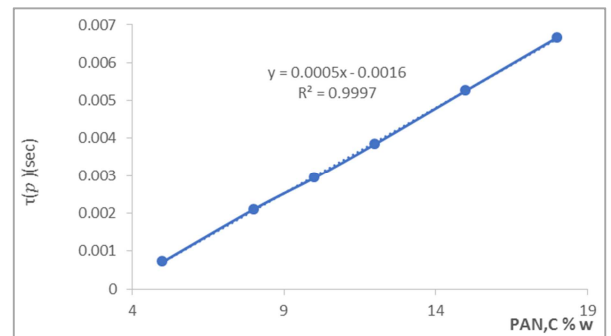
**Figure 8.** Relationship of the relaxation time to the concentration of the studied samples at a constant temperature $T = 303 K$ and a constant voltage $V = 4$ Volt.

Table 5. Calculation of the relaxation time τ_p for samples prepared from PAN and after adding a constant concentration ($5 \times 10^{-5} M$) of ODA, at a constant temperature $T=303 K$ and a constant voltage $V=4$ Volt.

PAN, C% w+ ODA ($5 \times 10^{-5} M$)	5	8	10	12	15	18
$T_p(\text{sec})$	0.000714	0.002105	0.002941	0.003846	0.005263	0.006667

Figure 8 shows the relationship of relaxation time τ_p for the prepared samples in terms of concentration, and it is with a linear relationship of the form: $y = 0.0005x - 0.0016$. Which shows that increasing the concentration of prepared samples increases the relaxation time τ_p .

3.8. Loss Factor

The loss factor was calculated according to the relationship (9), depending on the nodal impedance spectrum [25].

$$D = (\omega) / X(\omega) \quad (9)$$

The following spectrums in Figure 9 show the relationship of the loss factor spectrum to the frequency dependency of the studied samples at a constant temperature $T=303 K$ and a constant voltage $V=4$ Volt.

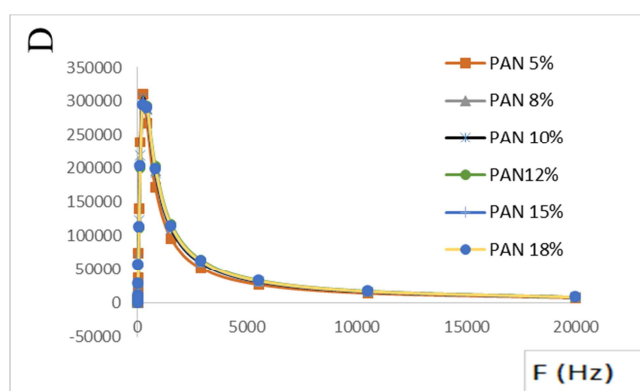


Figure 9. The sum of the loss factor spectra with frequency dependence of the studied samples at a constant temperature $T = 303 K$ and a constant voltage $V = 4$ Volt.

It is noticed from Figure 9 that the values of the loss factor increase at low frequencies until it reaches a maximum value of 222.9 HZ, after which its values begin to decrease, and this decrease is slightly at high frequencies, and this explains that when a changing electric field is applied to the insulator in the case of high frequencies, the polar molecules do not keep pace with the direction of the applied electric field and their frequency becomes different from the frequency of the applied field and a phase difference arises between the polarization and the field voltage when it is $\tau_0 \ll 1/\omega$. Then, the total change of polarization occurs, so the energy loss factor due to the relaxation of the dipoles is zero, and only the contribution of the loss due to the electronic relaxation appears. For electronic relaxation it decreases, while the losses due to relaxation of the dipoles take small values [26, 27].

4. Conclusion

Polyacrylonitrile samples were prepared with different

concentrations and studied for their viscosity. At concentrations less than 5% by weight, Taylor cone did not form for droplet extrusion and therefore the samples did not spin. Also, for concentrations greater than 18% W, it did not spin due to the very high viscosity at certain parameters, by calculating the average fiber diameter for each sample using a SEM device. In fact, it was observed that the average radius of the nanofibers of the samples increased with increasing concentration. Using XRD spectroscopy, according to the average crystallization size of PAN by adding ODA, compared to pure PAN, a significant decrease in the average crystallization size was found after adding the surface active material. Relaxation time τ_p by increasing concentration.

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