

## Effect of Temperature on physical properties of Electrospinning Nanofibers of Polyacrylonitrile

Alia H<sup>1\*</sup>, Noor SA<sup>2</sup>, Omar SAH<sup>3</sup>, Masri MY<sup>4</sup> and Subhi MH<sup>5</sup>

<sup>1</sup>Postgraduate Student (PhD), Department of Chemistry, Faculty of Science, University of Aleppo, Syria

<sup>2</sup>Department of Chemistry, Faculty of Science, University of Aleppo, Aleppo, Syria

<sup>3</sup>Department of basic science, Mechanical Engineering College, University of Aleppo, Aleppo, Syria

<sup>4</sup>Pro, Faculty of Science, University of Aleppo, Syria

<sup>5</sup>Professor at University of South Carolina-Sumter, South Carolina

\*Corresponding Author: Alia H, Postgraduate Student (PhD), Department of Chemistry, Faculty of Science, University of Aleppo, Syria, Email: allyfirst1@hotmail.com

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### Abstract

The electrical conductivity of polyacrylonitrile solutions was measured at different concentrations in the presence and absence of the surfactant octadecylamine (ODA), then the prepared solutions were spun with a locally manufactured electrospinning device at a voltage of 25 KV and a flow of 0.5 ml/h with a distance between the collector and the injector 10 cm, with a rotation speed of the collector 100 cycles/ A minute and using a 5ml syringing with a diameter of 0.5 mm injection needle and from the images of the scanning electron microscope (SEM) the average diameters of the prepared fibers for polyacrylonitrile were measured and found that they ranged between (100-500) nm, using the complex impedance spectrum it was found that the equivalent electrical circuit is a resistance in parallel with a capacitor, then calculated the relaxation time as a function of concentration and found that the relaxation time increases with increasing concentration, at several temperatures and that with increasing temperature the relaxation time decreases for the same sample, Then he calculated the number of dipoles for each sample and at a constant temperature and found that the number of dipoles is not related to concentration, which is a qualitative characteristic.

**Keywords:** Electrospinning, Polyacrylonitrile, Relaxation Time, Number of Dipoles

## Introduction

Chemical fibers are the raw material for many textile industries [1,2], which have different applications in many fields, whether medical, industrial or engineering [3,4].

### Textile Fibers

There are many sources of these fibers according to their type and methods of preparation, and these fibers can be classified into two main groups:

The first group: which includes natural textile fibers, the second group: which includes synthetic chemical fibers and is divided into two types:

1 Renewable fibers, which are made from natural and cellulosic raw materials such as viscose, or from inorganic materials such as glass fibers.

2 Synthetic fibers, which are prepared from raw materials by various chemical methods, and are often polymers such as polyacrylonitrile, polyvinyl alcohol, polyvinyl acetate, and others [5]. Methods for forming these fibers have developed rapidly over the past century, and there are three ways to prepare them, which are the extrusion method, the cold drawing method, and the electrospinning technique from the liquid phase, whether from the polymer solution or from the molten polymer [6-8].

Electrospinning technology is considered a scientific revolution in the field of nanofiber production [9], where nanofibers can be manufactured using electrospinning method from a very large number of polymers. The mats of these fibers have the porosity of the fibers, their flexibility, and the ease of controlling their shape and composition to make them suitable for the application to be obtained [11].

### Electrospinning:

Electrospinning is the simplest method for fabricating nanofiber materials, but there are some important factors that can have a significant impact on the composition and structure of the generated nanofibers. These factors are categorized into:

- (a) Factors affecting the polymeric solution
- (B) Factors affecting electrospinning technology
- (C) External (ambient) factors.
- (d) Factors affecting the polymer solution include: concentration or viscosity, electrical conductivity, molecular weight, and surface tension, while technical factors include: the value of the applied high voltage responsible for generating the electric field, the distance between the injector head and the collector, the shape of the collector, and the flow rate of the polymeric solution. Where Each of the previous factors greatly affects the shape and structure of the resulting fibers, so an appropriate combination of all these factors must be found to obtain the fibers with the appropriate diameter and structure. As for the external factors that affect the electrospinning process, they are the temperature and relative humidity in which the nanofibers spinning process takes place. [12]

The urgent need to search for new sources of pure water has made electrospinning at the forefront of current research for the possibility of obtaining clean water through the treatment of industrial and sewage water by Nano filtration [13]. Characteristics of the resulting cathode [14], nanoparticles. Electrodes for solar cells, fuel cells, and in electronic devices, and nanofibers can also be used to purify liquids from particles with diameters less than a micron by filtering through electrically spun nanofibers [15] and Figure (1) shows the different applications of the fibers.

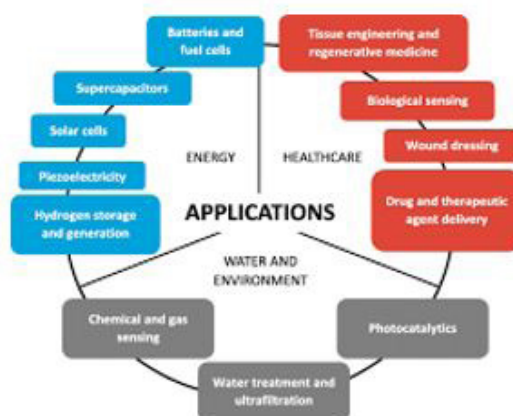


Figure 1: Different applications of nanofibers

### Polymeric solution:

The preparation of the polymeric solution is the most important part in the production of nanofibers by electrospinning method, and the factors that affect this step include: concentration, average molecular weight of the polymer, type of solvent used, surface tension of the solution, electrical conductivity, and viscosity [16].

The concentration of the solution used in electrospinning is usually low so that the electrostatic force can pull the fibers toward the collector. The concentration of the polymeric solution plays a major role in the formation of nanofibers during electrospinning. With the increase in concentration from low to high, the viscosity of the solution increases until a certain level of viscosity is reached, which is not subject to the spinning process, which is a qualitative characteristic related to the type of polymer used [17,18]. Therefore, ion soluble salts or surface-active colloids in small concentrations (surfactant) are added to the polymeric solution to increase its electrical conductivity and decrease its viscosity [19]. It was noted that the polymeric solution with good electrical conductivity allows obtaining fibers with small diameters and the value of these diameters decreases with increasing conductivity. In solutions with low electrical conductivity, the electrostatic force generated by the electric field is insufficient to achieve a large and stable elongation of the fibers, and the movement of these fibers is mostly turbulent. Also, the possibility of knots forming on these fibers, whose diameters are not homogeneous, increases and thus affects the formation of nanofibers. On the other hand, the relatively high conductivity of the polymer solution causes the polymer flow to become unstable, especially when the applied electric field strength increases, which may cause a disturbance in the movement of the fibers and a random distribution of their diameter values [20,21].

### Effect of surfactant-active colloids:

The surfactant colloidal materials are characterized by their ability to reduce the surface tension in the polymeric solutions to be electro spun on the interfaces between the phases. In addition, these materials are characterized by their ability to form aggregates of molecules called micelles [22]. The surface-active colloidal materials strongly affect 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 colloidal materials also affect the shape and dimensions of the nanofibers formed [23,24]. The effect of the surface-active colloidal Octadecylamine ODA on the spun polyphenol nanofibers coated with urea, it increased the rate of urea release up to 30 days, which helped in better plant growth as a result of reducing the diameter of the resulting nanofibers [25]. Surfactant action of octadecylamine ODA on the copolymer of polyvinyl alcohol and polyvinyl acetate PVA-co-PVAc. It was found that there was a decrease in the surface tension of the prepared polymeric solutions and thus a decrease in the radii of the electrically spun nanofibers [26]. Its smoothness and surface quality [27]. Polyacrylonitrile fiber ranks third in terms of production among all synthetic fibers due to its unique properties, such as having outstanding features such as light resistance, chemical stability, non-toxicity and mechanical flexibility. Many different solvents can be formed from a solution of these solvents, namely DMF, DMSO, DMA, chloroacetonitrile, dioxanone, dimethyl phosphite, dimethyl sulfone, beta-butyrolactone, ethylene carbonate, nitric acid and sulfuric acid [28]. Polyacrylonitrile nanofibers have also been used recently in medicine, but to a limited extent as dressings for wounds and an aid in healing and as dressings that prevent germs from reaching live wounds loaded with antibiotics and medical salts [29].

### Materials used

- 1 Polyacrylonitrile, with a known molecular mass of 100,000 g/mol, contains the following monomers: acrylonitrile  $\text{CH}_2=\text{CH}-\text{C}\equiv\text{N}$  by 85%w by weight, acrylonitrile  $\text{CH}_2=\text{CH}-\text{CO}-\text{NH}$  and vinyl acetate  $\text{CH}_2=\text{CH}-\text{O}-\text{CO}-\text{CH}_3$ .
- 2 Dimethylformamide "DMF" solution with purity (GC) (99.5%) from MERCK company
- 3 Octadecylamine ( $\text{C}_{18}\text{H}_{37}\text{NH}_2$ ) ODA from Alfa Aesar
- 4 Hydrochloric acid 37-38% purity from MERCK. company

### Devices used:

- 1 The electrical conductivity device WTW-Inolab-Level-1, made in France, with an accuracy of  $\pm 0.01\%$ , and equipped with a conductivity measuring cell consisting of two platinum plates in the form of a disc coated from the inside with a constant KCell= (0.478) platinum sponge powder, and equipped with a temperature sensor.
- 2 A locally manufactured electric spinning device [30]
- 3 Scanning Electron Microscopy," SEM, American-made

Tuscan Veca 2 model, in cooperation 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

### Streptococcus Impedance Device 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. It is used to draw the complex impedance spectrum of the studied sample through which a set of parameters can be measured and inferred, as well as To find out the equivalent circuit.

### Preparation of solutions for spinning

1. Solutions with 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 the ODA surface active colloid at a fixed concentration (at a concentration of  $100 \times 10^{-5}$ ). mol/l, the electrical conductivity of the prepared solutions was studied before and after the addition of ODA surfactant.

2 A solution of octadecylamine ODA was prepared at a concentration of  $100 \times 10^{-5}$  mol/l, with a weight of 0.0269 g of ODA, and put it in a volumetric flask with a capacity of 100ml, then 1ml of HCl was added, and the volume was supplemented with DMF solution, and the solution was placed on a magnetic stirrer for 3 hours. At 50°C until complete dissolution of octadecylamine.

3 The prepared solutions were taken and measured with a homemade electric spinning device.

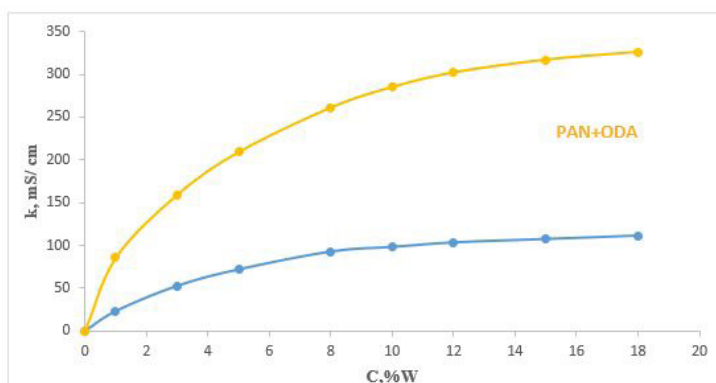
### Practical results and discussion:

#### 3-4-1- Measurement of electrical conductivity:

The electrical conductivity of the prepared solutions was measured before and after adding the ODA surfactant, and Table (1) shows the values of the specific conductivity of the prepared solutions at a temperature of 303 K.

PAN C,%W	k, mS/ cm	
	without ODA	+ODA
1	23	85.5
3	52.5	159
5	71.6	209
8	91.4	261
10	97.8	285
12	103	302
15	107	317
18	111	326

**Table 1:** values of specific electrical conductivity before and after adding octadecylamine to the prepared solutions, at a temperature of 303 K



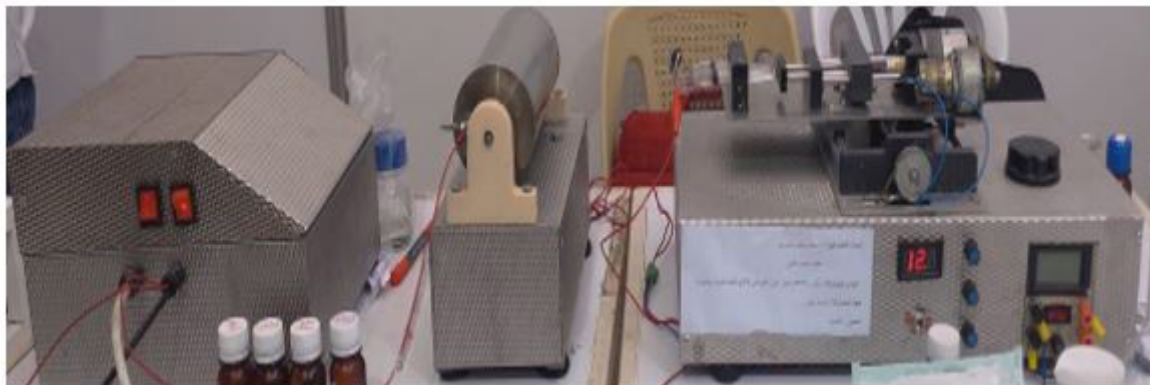
**Figure 2:** Shows the relationship of the specific electrical conductivity to the concentrations of the prepared PAN solutions in the presence and absence of ODA, at a temperature of 303 K

It is observed from Figure (2) that a logarithmic relationship relates the conductivity to the concentration, where the specific conductivity increases in PAN solutions with the increase in concentration, but at large concentrations of PAN the conductivity becomes almost constant, while in PAN/ODA solutions the conductivity increases much more than it is in PAN solutions and that Because of the mutual effects between ODA

and PAN molecules, because adding ODA weakens the mutual effects between the PAN molecules [31-34].

### Electrospinning

The prepared samples were spun with the homemade electrospinning device shown in Fig. (3).



**Figure 3:** Homemade electrospinning device

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

At a concentration of less than 5%w of PAN/DMF solutions, spinning was not completed, due to the incomplete crystalline structure of the polyacrylonitrile and the low viscosity of the solution, so the droplet emanating from the injector did not form a Taylor cone.

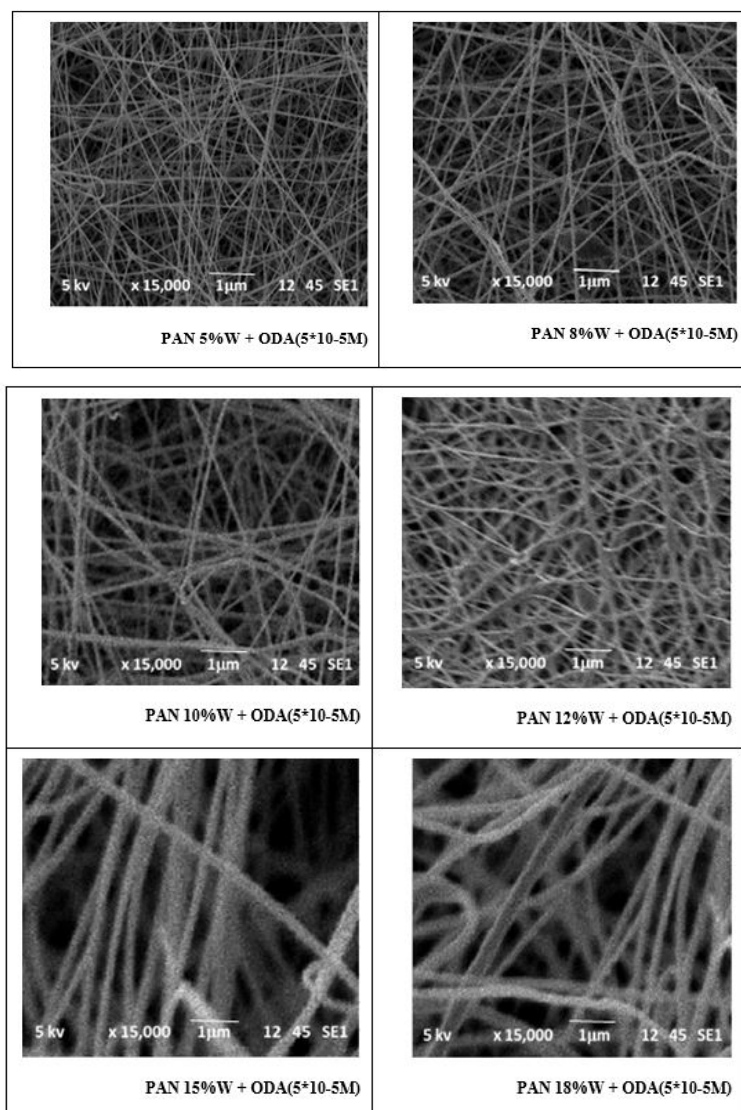
At concentrations greater than 18%w of the PAN/DMF solutions the viscosity is so high that the drop does not emerge from the injector to form a Taylor cone.

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

- Solution flow rate: 0.5ml/h.
- Voltage lifter: 25 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

### 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 (2) shows that The average diameters of the resulting nanofibers increase with increasing concentration.



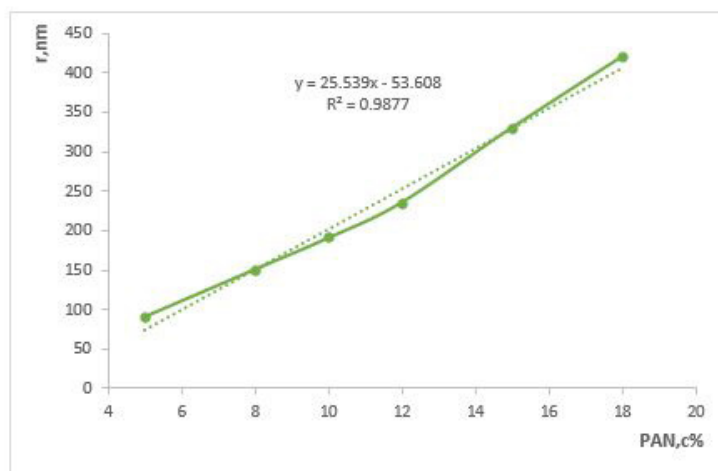
**Figure 4:** SEM electron microscopy images of spun PAN samples

Table 2 shows the increase in the average diameters of nanofibers for the prepared samples with increasing concentration.

<b>PAN,C % w+ ODA(5*10-5M)</b>	5	8	10	12	15	18
<b>Av. fiber diameter(nm)</b>	90	150	190	235	330	420

**Table 2:** The average diameter of nanofibers for samples prepared at the concentration

The following figure (5) shows the relationship of the average nanofiber diameter of the prepared samples to the concentration function.



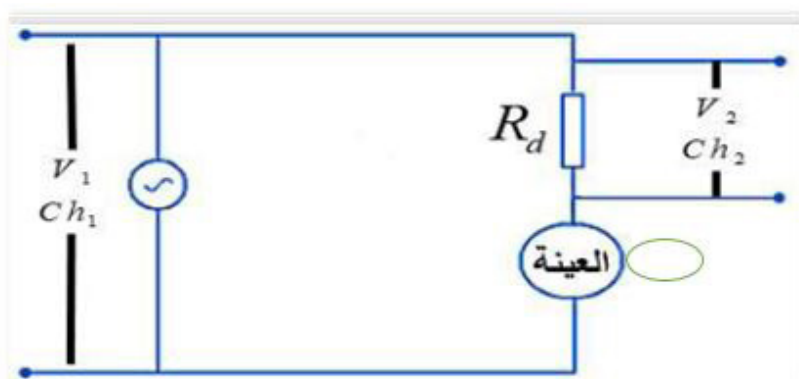
**Figure 5:** The average diameter of the nanofibers of the prepared samples as a function of concentration.

Figure (5) shows the average diameter of the nanofibers of the prepared samples in terms of 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  $y = 25.539x - 53.608$  due to the increase in the viscosity of the spun polymeric solution [30]. As for the concentrations ((1,3%w), spinning was not done because the polymeric structure of polyacrylonitrile was not formed. Therefore, the crystal structure of polyacrylonitrile begins to form at a concentration of 5% W of its concentration in DMF [27,35].

### AC (Septal Impedance IS) measurements

One of the most important methods for characterizing the electrical properties of materials and electrodes is Impedance

Spectroscopy (IS). To use this technique to study and characterize the behavior of films in polar solutions, in biosensors, in composite nanofibers, in double-layer capacitors, in organic photo-detectors, and in the analysis of photovoltaic systems that rely on different technologies using IS, such as silicon, dye and organic solar cells [36]. Where some coefficients affecting the electrical behavior of the material are calculated depending on the complex impedance spectrum, And that is by applying an alternating current so that the studied sample placed between the condenser caps is connected to a gain phase analyzer device with detection resistance  $R_d = 320 \Omega$ , and an alternating signal of variable frequency is applied to the material, with a constant voltage  $V_1$  as shown in Figure (6)



**Figure 6:** Schematic diagram of the method of connecting the sample to measure its complex impedance

The nodal impedance is expressed by the following relationship:

$$Z(\omega) = R(\omega) + jX(\omega) \quad (1)$$

Where

$$R(\omega) = R_d \frac{a - (a^2 - b^2)}{a^2 + b^2} \quad (2)$$

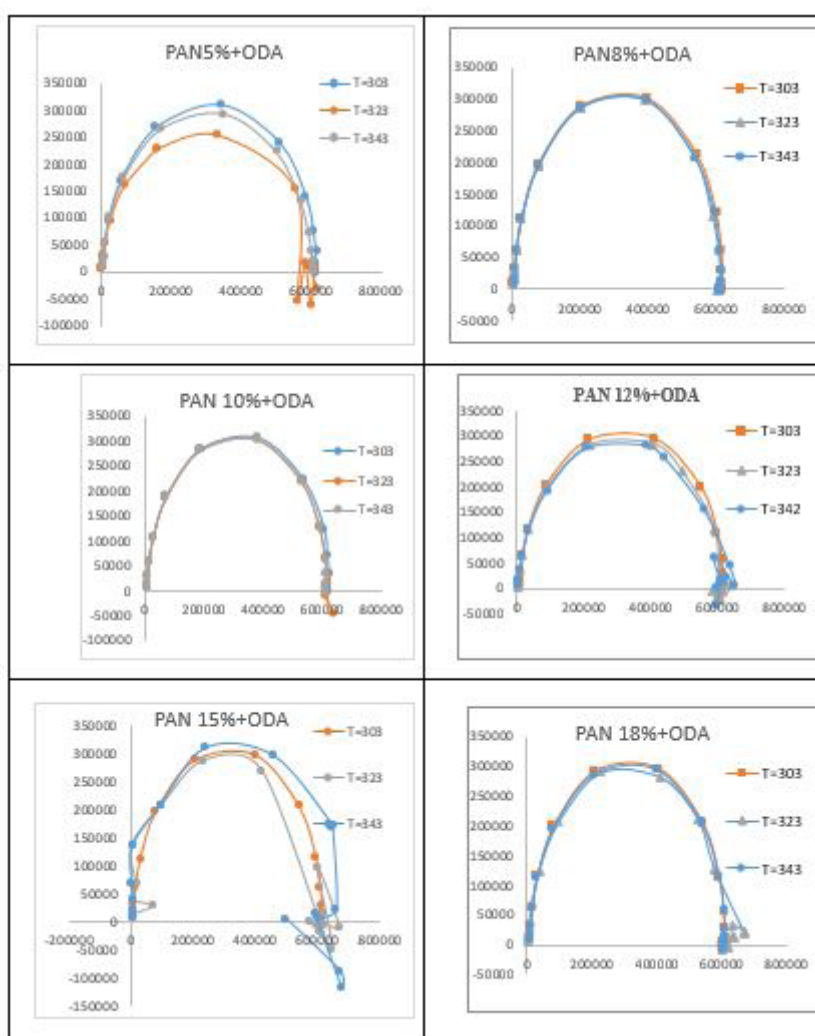
$$X(\omega) = \frac{b R_b}{a^2 + b^2} \quad (3)$$

a, b represent variables obtained from the spectrometer, and by drawing the impedance spectrum, we get an arc of a cir-

cle, where it is achieved, at the top of the arc of the circle  $1 = \omega \tau_p$  in addition to the possibility of calculating the relaxation time  $\tau_p$ , the loss factor D, and the circuit equivalent [37].

Studying the effect of the temperature change of the PAN samples at each concentration and constant voltage  $V=4$  Volt 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 three temperatures  $T = (303, 323, 342)$  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 after adding a fixed concentration ( $5 \times 10^{-5}M$ ) of ODA at temperatures  $T = (303, 323, 342)$  K and constant voltage  $V=4$  Volt



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 [38]

### Relaxation time $\tau_p$

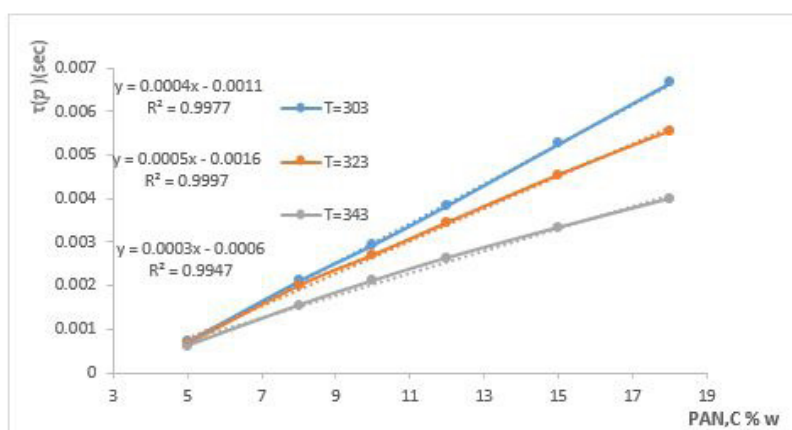
The relaxation time  $\tau_p$  was calculated for the samples prepared from PAN and after adding a fixed concentration

( $5 \times 10^{-5} M$ ) of ODA, at temperatures  $T = (303, 323, 342)$  K and a constant voltage  $V = 4$  Volt as shown in the following table) 3) Using the following relationship (4):

By taking the value of the frequency corresponding to the highest value of  $R(\omega)$

PAN, C % w+ ODA			5	8	10	12	15	18
T, K	303	$\tau_p$ (sec)	0.000714	0.002105	0.002941	0.003846	0.005263	0.006667
	323		0.000667	0.000667	0.000667	0.000667	0.000667	0.000667
	343		0.000606	0.000606	0.000606	0.000606	0.000606	0.000606

**Table 3:** Calculation of the relaxation time  $\tau_p$  for samples prepared from PAN and after adding a fixed concentration ( $5 \times 10^{-5} M$ ) of ODA, at temperatures  $T = (303, 323, 342)$  K and a constant voltage  $V = 4$  Volt



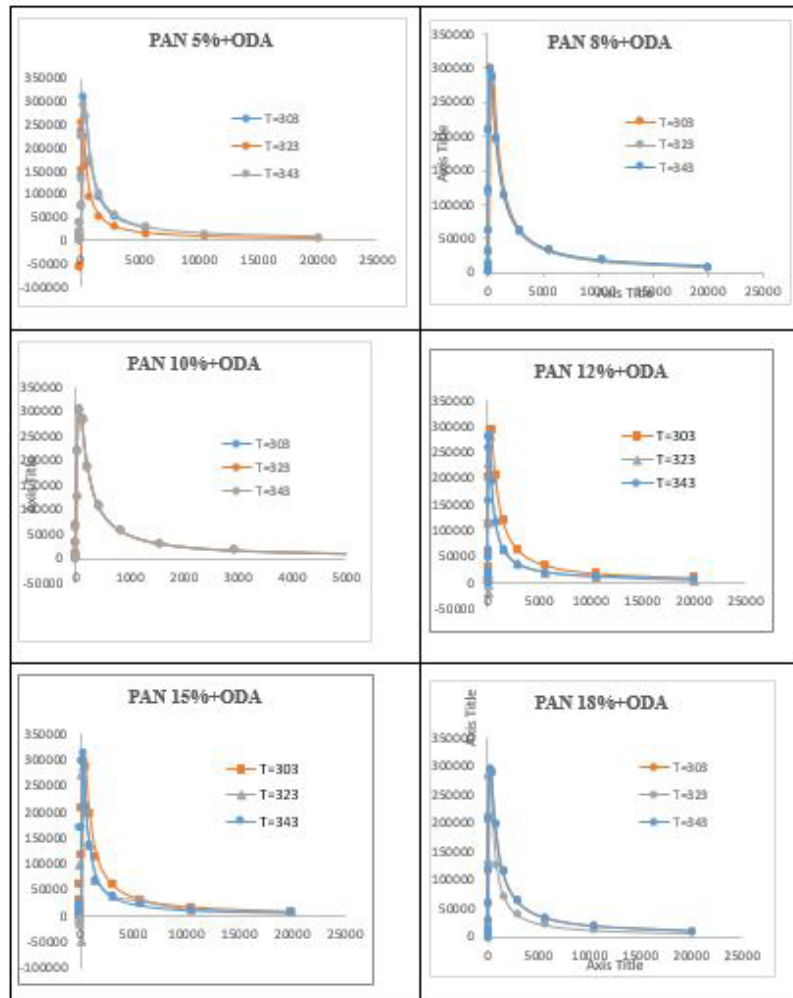
**Figure 8:** Relationship of the relaxation time with the concentration of the studied samples at temperatures  $T = (303, 323, 342)$  K and a constant voltage  $V = 4$  Volt

Figure (8) shows the relaxation time relationship  $\tau_p$  for the prepared samples in terms of concentration, and it has a linear relationship of the form:  $y = A \times e^{b\chi}$ . Which shows that increasing the concentration of the prepared samples increases the relaxation time  $\tau_p$ , and the table shows a decrease in the relaxation time with increasing temperature.

### Loss Factor

The loss factor was calculated according to the relationship (4), depending on the nodal impedance spectrum [39].

$$D = R(\omega) / X(\omega) \quad (4)$$



**Figure 9:** Relationship of the relaxation time with the concentration of the studied samples at temperatures  $T = (303, 323, 342)$  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 they reach a maximum value, 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 It does not keep pace with the direction of the applied electric field and its 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  $\tau_0 \ll \frac{1}{\omega}$ . 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. As electronic relaxation decreases, as for the losses due to the relaxation of the dipoles, it takes small values [40-42].

### Calculating the number of permanent dipole moment

Spun samples were taken of different concentrations of PAN and by adding a fixed concentration of ODA, so that the dimensions of the studied sample are from the dimensions of the condenser, and it was placed between two circular condenser caps of known dimensions and connected to an aluminum electrode so that the cap was circular in shape and its diameter was equal to the diameter of the sample. The other for the sample is another electrode of aluminum with a diameter equal to the diameter of the sample so that a good Ohmic contact is achieved [32].  $5 \times 10^{-5} \text{M}$ ) from ODA, and the following table (4) shows some physical and electrical properties of PAN, where the area  $A$  and the capacitance of the capacitor  $c$  were calculated from the following relationships:

Physical parameter value	Physical/Electrical parameter
100	molar mass M (Kg/mol)
42.66	Capacitance C (Farad)
13.5×10 <sup>-3</sup>	disc radius r (m)
0.1×10 <sup>-3</sup>	disc thickness d (m)
2.337×10 <sup>3</sup>	disc weight w (Kg)
5.914×10 <sup>-4</sup>	disk space A (m <sup>2</sup> )
2.337×10 <sup>3</sup>	disk density d (Kg/m <sup>3</sup> )
8.776×10 <sup>-10</sup>	Dielectric constant K

**Table 4:** Some values of physical and electrical coefficients for PAN at constant temperature T=303 K

Capacitor Capacity C [42,43]:

$$C = \frac{\epsilon A}{d} \quad (5)$$

Number of instantaneous dipoles D (Debye):

$$D = \frac{\sqrt{\frac{9k_B T P \epsilon_0}{N_A}}}{3.3356 \times 10^{-19}} \quad (6)$$

molar polarity P.

$$A = \pi \cdot r^2 \quad (7)$$

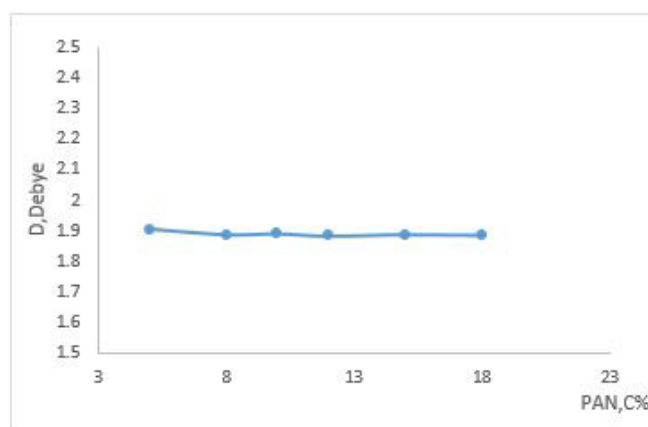
At a fixed frequency f=10520 HZ according to , P and D for each of the samples studied at a temperature of 303K, the calculated values are shown in Table (5).

PAN, C%w+ ODA(5×10 <sup>-5</sup> M)	5	8	10	12	15	18
ε	18.101	18.334	17.822	18.658	18.35	20.707
P (m <sup>3</sup> / mol)	71854	71999	71677	72194	72008	73300
D (Debye)	1.8844	1.8863	1.8821	1.8888	1.8864	1.9033

**Table 5:** Calculated values of ε, P and D for each of the samples studied at a temperature of 303 K

And by drawing the relationship of the number of dipoles in terms of the concentration of samples prepared at a constant temperature of 303 K, as in Figure (10) we conclude that the

number of dipoles is constant for all samples and therefore the number of dipoles is a qualitative characteristic and has nothing to do with the concentration of the substance.



**Figure 10:** RShows the relationship of the number of dipoles to the concentration of samples prepared at a constant temperature of 303 K

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## Conclusion

Samples of polyacrylonitrile were prepared with concentrations of %W (1-18) and their electrical conductivity was studied before and after the addition of ODA. %W did not spin due to lack of crystal structure formation and therefore Taylor cone did not form for droplet extrusion, and for concentrations greater than 18%W it did not spin due to very high viscosity at specific parameters, and by calculating the average diameter of the fibers for each sample using SEM device, an increase in mean equilibrium was observed. The diameters of the nanofibers of the samples were increased by increasing the concentration. By using XRD spectroscopy, according to the average crystallization size of the PAN samples by adding ODA, compared with the pure PAN, a noticeable decrease was found in the average crystallization size after adding the surface active substance. Increasing the relaxation time  $\tau_{(p)}$  by increasing the concentration and it was found that the increasing relaxation time  $\tau_{(p)}$  with increasing temperature, the number of dipoles is constant for all samples and therefore it is qualitative and has nothing to do with the concentration of the substance.

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