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High Safety Electrospinning Device with Several Variables for Producing Polymeric Nanofibers with Different Properties

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Abstract: A high safety electric spinning device was tested to produce nanofibers from different polymers, and the spinning was tested on the human hand directly without feeling any short circuit. The locally designed device consists of a voltage lifter, an injection pump and a rotating cylindrical collector. The voltage booster is based on converting AC current from 220V to 50KV. The injection pump has four variables: the first variable to control the speed of pumping the solution from the injector, the second variable to control the movement of the injector on a metal rail back and forth, and the third variable to control the speed of rotation of the cylinder accumulator through time, The fourth variable is to push the injector in reverse to refill it again, and the distance of the collector from the injector can be controlled, and it can also be replaced with a flat metal plate. The viscosity of polyacrylonitrile solutions with different concentrations was studied and then spun with the designed device and the radii were measured via a scanning electron microscope, then the effect of the voltage change on the change of the average diameter of the fibers was studied when the concentration was fixed and it was found that with the increase in the applied potential difference the average diameter decreases The resulting nanofibers.

Keywords: Electrospinning, Nanofibers, Polyacrylonitrile, Rheological Properties

1. Introduction

The reason for the spread of PAN fiber production in many parts of the world is due to the following important characteristics: the average molecular mass of the obtained PAN, the rheology of its spinning solutions, the relatively cheap polymerization process, and the ease of the heat treatment system. These qualities enable us to control the required mechanical and structural properties of the produced fibers [1].

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 the medium of DMF showed the occurrence of severe association processes in the solution due to the mutual effects between the dipoles [2].

1.1. Electrospinning Technique

Electrospinning is one of the modern and advanced technologies, and it is considered a scientific revolution in the field of nanofiber production. Despite the discovery of this effective technique in the thirties of the twentieth century, its industrial use was only made in recent years because of its important industrial applications. It has produced continuous nanofibers with diameters of 2nm and even a few micrometers, and that is a wide range of natural and synthetic polymers, which made it enjoy an advanced position at the level of international research. [3]

The electrospinning mechanism relies on electrostatic forces to produce polymeric fibers with nanoscale diameters. These tiny structures are characterized by important surface and structural properties such as surface-to-volume ratio, high porosity, mechanical and optical properties. [4] The technique of electrospinning has begun to gain a very large 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]

1.2. Applications of Nanofibers Generated by Electrospinning

Since the mid-nineties, and to this day, research, publications and scientific articles related to this technology are increasing because of its very wide applications in different fields, and great progress has been made in understanding the process of electrospinning, and then developing in specialized electrospinning techniques, (axial electrospinning and spinning). double electrophoresis), and as a result, it is now possible to produce industrial or laboratory electrospinning machines to produce nanofibers from most synthetic and natural polymers. Figure 1 shows the increasing rate of publication of research related to electrospinning applications from 1990 to 2020 [6-9].

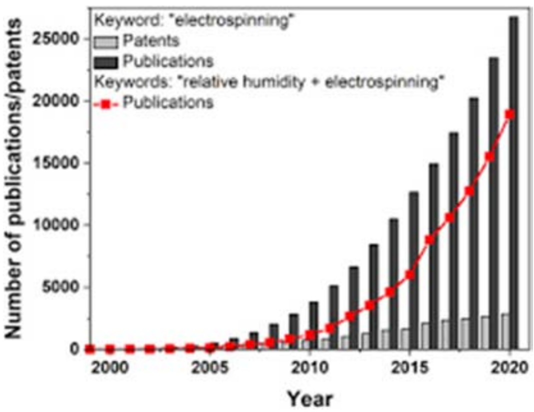

Figure 1. *The rate of publication of research on electrospinning.*

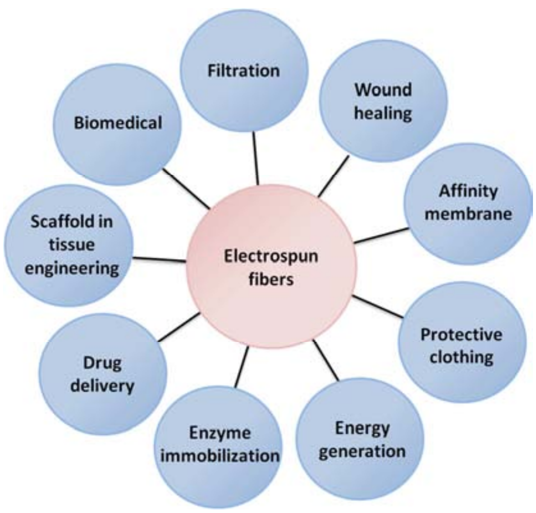

Figure 2. *Various applications of nanofibers.*

This technique allowed the production of antibacterial nanofibers by incorporating biocides into electrospinning solutions, or adding them to nanofibers after electrospinning [10]. And fuel cells and in electronic devices, nanofibers can be used to purify liquids from particles with diameters less than a micron by filtering through electrically spun nanofibers [11]. 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 [12], and nanofibers have also contributed to improving the properties of the electrodes of battery cells [13], and Figure 2 shows various applications of nanofibers.

1.3. Electrospinning Mechanism

The electrospinning mechanism relies on the use of a high potential difference to generate a strong electric field between the polymer solution injector tip (connected to the positive electrode) and the collector (grounded or grounded to the negative electrode). Where the electrostatic force produced by the field after a certain limit can overcome the surface tension forces of the viscous polymeric solution, and it pulls a thread from this solution towards the collector very quickly, which leads to the elongation of this polymeric thread, and a significant decrease in its diameter so that it turns into a nanofiber before it arrives to the complex. [4, 14].

1.4. Electrospinning Device

The electrospinning apparatus consists of three main sections:

1) Voltage booster: which gives the polymeric solution a very high positive charge ranging from 5-50 kV, depending on the type and concentration of the polymer used, and this high charge is able to fragment the solution and turn it into nanofibers before it reaches the complex, due to the evaporation of the solution. And the great speed in the exit of the fibers.
2) Injector assembly: It is the chamber in which the polymeric solution is placed, and it is often a syringe made of glass or plastic, at the end of which is a needle or capillary tube of a specified diameter (connected to the positive electrode of the voltage lifter) from which the charged solution comes out, and at a specified and constant flow rate It is controlled by the injection pump.
3) Grounded collector: on which nanofibers are collected and connected to the negative pole of the high voltage lifter, and it has two types: either in the form of a flat metal plate covered with aluminum or tin foil to facilitate the process of removing the fibers, and the fibers resulting from this type of collector are randomly distributed, or the collector is Earthed in the form of a rotating cylinder. Through which it is possible to control the pull of the fibers, and in this way we get fibers with a high orientation by stretching the fiber axis and the resulting fiber diameters are smaller than what they are in the planar complex [15-17]. Figure 3 shows an illustrative diagram of the two types of electrospinning devices (the cylindrical collector and the collector with the planar plate).

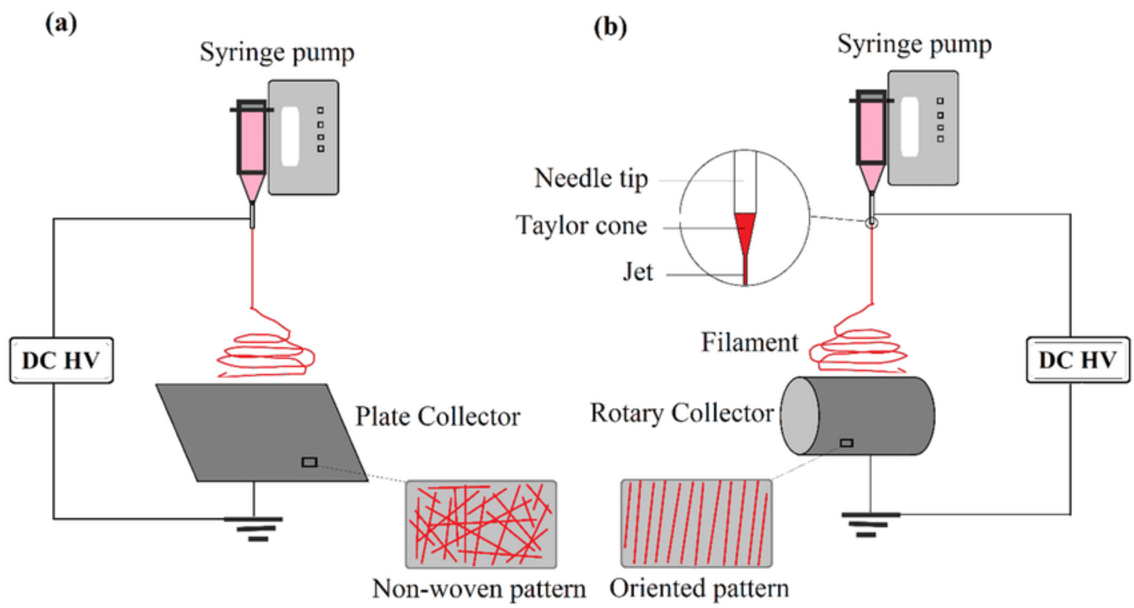

Figure 3. *Diagram of the two types of electrospinning.*

1.5. Droplet Extrusion Process and Taylor Cone Formation [18]

The polymer solution is prepared in a certain concentration, and the solution is placed in the injector. The needle of the injector is usually connected to the positive electrode and the collector is connected to the negative electrode or grounded.

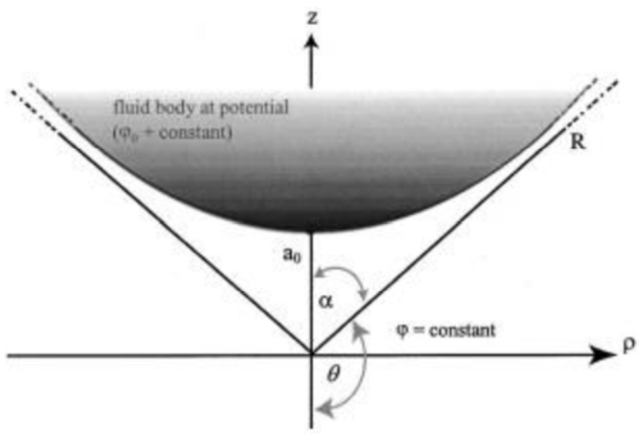

Figure 4. *Taylor Cone.*

1.5.1. Taylor Cone Formation

When high voltage is applied to the polymeric solution, the shape of the drop on the tip of the needle is almost spherical. Geoffrey Taylor in 1966 found that when a potential difference is applied between the needle and the collector, it leads to the accumulation of charges on the head of the formed droplet and it stretches at an angle of 49.3, and what is formed is It is called Taylor Cone, as shown in Figure 4. The tangential electrical stress causes the polymer solution to move, which creates a hydrodynamic pressure on the surface of the droplet, and the interactions between electrical and hydrodynamic stresses cause the drop to deform. It was found experimentally that for each type of polymeric solution, a certain potential difference must be applied [19].

What determines the shape of the drop (jet) is the strength of the electrostatic field. Taylor showed that there is a limit value of voltage V_c at which the drop turns from a spherical shape to a conical shape as shown in Figure 5.

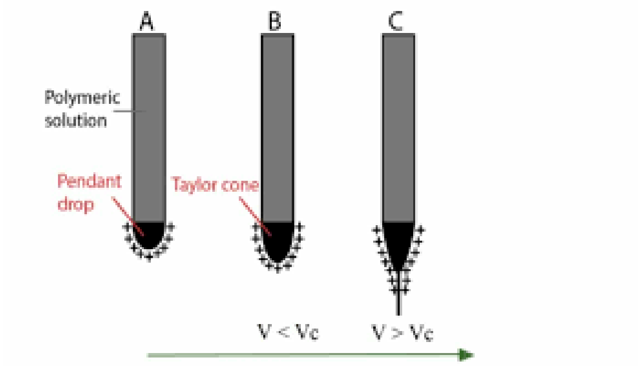

Figure 5. *Drop pop.*

1.5.2. Factors Affecting Electrospinning Technology

Electrospinning is the simplest method for manufacturing nanofibers, 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:

1) Factors affecting the polymeric solution.
2) Factors affecting the technique of electrospinning.
3) External (surrounding) factors.

The electrospinning process cannot be successful if the previous factors are not precisely and specifically controlled and that any change in one of the conditions changes the course of the electrospinning process, and thus changes the shape, structure, and diameter of the resulting nanofibers.

Table 1. *Shows the most important factors affecting the electrospinning process [20].*

factors	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

It is worth noting that the polymer concentration is a qualitative characteristic, related to the polymer itself, its molecular mass, and its viscosity [2, 21].

The importance and goal of our research stems from the design and development of a safe and economical electric spinning device, which can be worked on from a very close distance, and without an insulator, and that the designed device works with less than one ampere, and can be used for spinning fibers for natural and industrial polymers, including spinning polyacrylonitrile fibers due to its industrial importance.

2. Experimental

2.1. Materials and Devices Used

1. Synthetic fibers of Polyacrylonitrile with a known molecular mass of 100,000 g/mol [22].
2. Dimethylformamide "DMF" purity (GC) (99.5%) from MERCK.
3. Locally designed and manufactured electric spinning device.
4. Scanning Electron Microscopy" SEM, American-made "Tuscan Veca 2" model.
5. German-made ball drop viscosity device from HAAKE company, which complies with ISO 12058, and this device is equipped with a water shirt to keep the temperature with an accuracy of ± 0.03°C, and it measures with this accuracy up to 80°C. This device enables us to measure the viscosity within the range (0.3-75000 mPas) depending on the ball used.

2.2. Procedure

An electrospinning device was developed and designed based on electrostatic forces, and it is a very safe device. This device consists of three main sections:

1) Voltage riser, which is an electrical transformer that contains two variables, the first to feed the injection pump and the rotary cylinder, and the second to raise the voltage to the required level: The voltage riser works to convert alternating current from 220 volts to 50 kV and this voltage lifter is designed from 15 electronic circuits to raise The voltage, then, is an economical device, and can operate with less than one ampere.
2) The injection pump, which is the second piece of the device, which contains several keys to control the required variables, which are:
   a) A large rotating screw to control the voltage with a digital panel showing the amount of voltage given.
   b) A holder for two syringes that can be used together or just one.
   c) A metal rail allows the injectors to be moved horizontally to spin at a greater distance.
   d) Control switch (upper) at the pumping speed, and the pumping speed can be controlled between (0.5-5) ml/h.
   e) The control switch (middle) moves the injector on a metal rail back and forth, at a rate of speed ranging from (0.5-10) mm/sec, to weave the fibers on a larger area than the rotating cylindrical collector, and thus control the number of layers of fibers (thickness) woven.
   f) Control knob (lower) for the speed of rotation of the rotating collector through time to obtain a certain thickness of the fabric produced on demand. It also includes two independent syringes so that two different polymers can be spun simultaneously on the same compound.
3) Rotating cylindrical collector: The distance of this collector from the syringe can be controlled by a distance of (5-25) cm, and the speed of its rotation can be controlled between (0-500) cy/min, and we can replace the collector with a flat metal plate.

Polyacrylonitrile solutions were prepared with different concentrations and spun with the designed electrospinning device and we used the rotary cylindrical collector, and the work was according to the following variables:

a) Solution flow rate: 0.5ml/h.
b) Voltage lifter: 20 kv.
c) Rotation speed of collector: 100 cy/min.
d) The distance of the collector from the injector: 10 cm.
e) Diameter of the injector needle: 0.5 mm.

Figure 6 shows the designed electrospinning device with its three main parts:

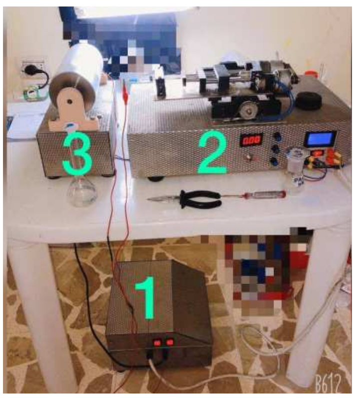

Figure 6. *Electrospinning device with its three main parts (1- high voltage supply) (2- Pump and main control) (3- Collector drum).*

3. Results and Discussion

3.1. Safety Test of the Designed Device

Figure 7. *Safety test of the electrospinning device.*

We conducted a high safety test for the electric spinning device designed by placing the hand directly and at a very close distance of 5 cm instead of the collector, which gave the desired result in terms of safety test. Figure 7 shows the high safety test directly on the hand:

3.2. Determination of Viscosity of Prepared Polyacrylonitrile Solutions

The viscosity of PAN solutions was determined at 298K. We calculated the viscosity of the solutions from the following relationship:

$$\eta = k\,(d_1 - d_2)\,t$$

where k: device constant for the sphere, d_1 density of the sphere, d_2 density of the solution, t: time of fall of the sphere.

The results are shown in table 2.

Table 2. *Viscosity of PAN solutions at 298K.*

PAN (W%)	1	3	5	8	1	12	15
Viscosity η, (mPas)	1.495	8.831	30.902	109.333	264.968	530.70	1345

Figure 8 shows the relationship of viscosity to the concentration of polyacrylonitrile solutions.

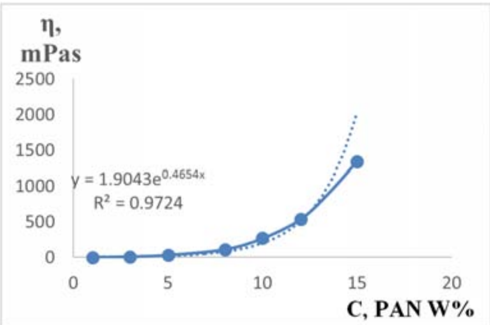

Figure 8. *Relationship of viscosity to the concentration of polyacrylonitrile solutions.*

We find from Figure 8 an exponential relationship whose general form is y=1.9043e0.465x, where the value of R2=0.9724 relates viscosity to concentration. We also find that the viscosity increases with concentration in a linear and slow manner 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 as a result of the presence of dipoles, and they form hydrogen bonds, which in turn leads to a noticeable increase in viscosity [2, 22].

The prepared PAN solutions were electrospun, and we found the following:

a) At a concentration of less than 5%w of the 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.
b) At concentrations greater than 15%w, the viscosity was so high that the drop did not emerge from the injector to form a Taylor cone.
c) The PAN solutions were electrospun to concentrations between 5-15% W, and the resulting nanofibers had a diameter between 200-400nm. The following figure 9 shows the SEM images of the spun samples:
   1) The 5%W PAN sample had an average fiber diameter about of 200nm.
   2) The 8%W PAN sample had an average fiber diameter about of 260nm.
   3) The PAN sample 10% W had an average fiber diameter about of 280nm.
   4) PAN 12% W sample had an average fiber diameter about of 320nm.
   5) The PAN 5%W sample had an average fiber diameter about of 400nm.

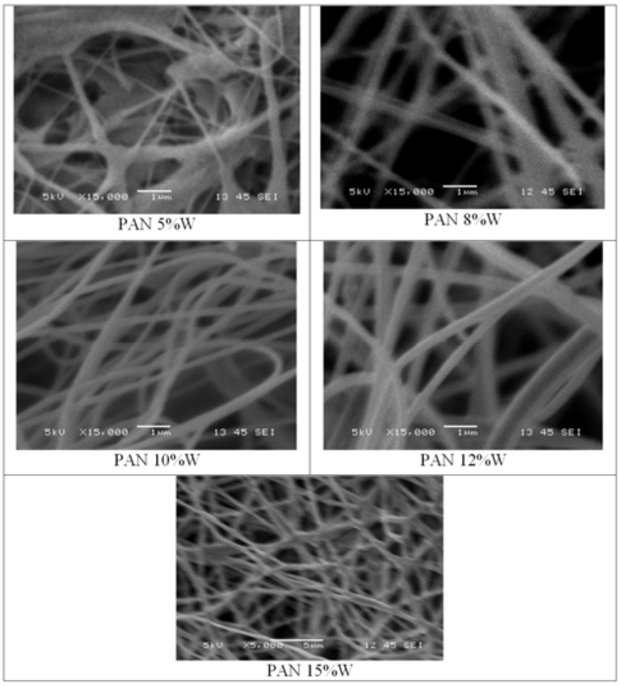

Figure 9. *SEM images of PAN samples at different concentration.*

We show in Figure 10 the relationship of the average diameter of nanofibers to the concentration of PAN% W.

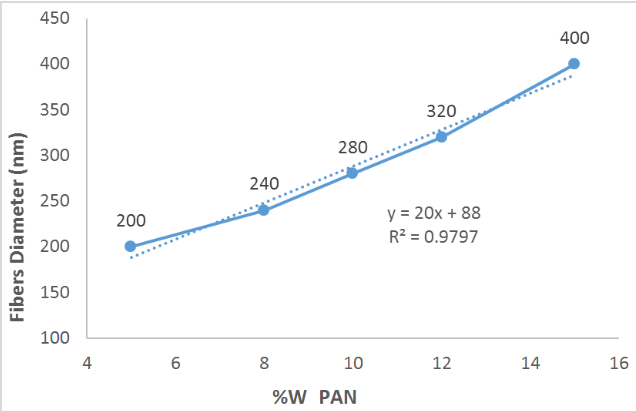

Figure 10. *Relationship of the average diameter of nanofibers to the concentration of PAN.*

We find from Figure 10 that the average diameter of the polyacrylonitrile nanofibers increased by increasing the concentration with a linear relationship, the general shape of which is y = 20 x +88, and where the value of R2 = 0.9797, due to the increase in the viscosity of the spun polymeric solution [21]. As for the concentrations ((1,3%w), spinning was not done because the polymeric structure of polyacrylonitrile was not formed.

We also studied the effect of changing the applied voltage on the average diameter of the resulting nanofibers on the PAN 10%W sample, and we found that by increasing the applied voltage, the diameter of the resulting nanofibers decreases. Also, the spinning process did not take place at a voltage less than 10 KV for the studied sample, due to the structural and mechanical properties of polyacrylonitrile at a concentration of 10% W [23].

Below are the SEM images of the spun samples:

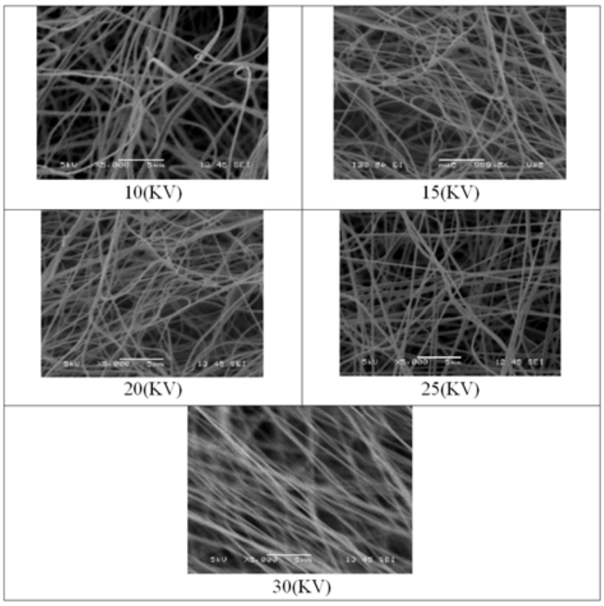

Figure 11. *SEM images of PAN10%W samples at different voltages.*

Table 3. *Relationship of the average fiber diameter to the applied voltage.*

Voltage (KV)	**10**	**15**	**20**	**25**	**30**
Average of fiber diameter (nm)	340	315	280	245	200

Table 3 shows the average nanofiber diameter values resulting from the change in the applied voltage value of the PAN10%W sample.

We show in Figure 12 the relationship of the average diameter of the resulting nanofibers for the PAN10%W samples when the value of the applied voltage changes.

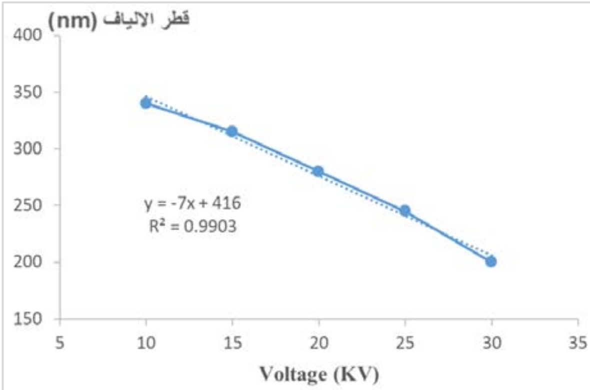

Figure 12. *Relationship of the diameter of the formed polymer fibers to the applied voltage.*

We find from Figure 12 a decrease in the diameter of nanofibers for PAN10%W samples with an increase in the applied voltage with a linear relationship whose general shape is y=-7x+416, where R^2 = 0.9903, with the stability of other variables. The solution is further fragmented the moment it leaves the injector, and thus the diameter of the resulting fibers decreases [24].

4. Conclusions

1. The designed electric spinning device contains a voltage lifter that depends on converting alternating current from 220 volts to 50 kV. 15 stage lift.
2. The designed device has a very high safety, as it is possible to spin on the human hand without any feeling of electric circuit and at a very close distance of 5 cm.
3. The designed device is economical, as it contains two injectors that work together to save time and effort and the possibility of developing it for more than two injectors with the same electrical capacity, and the quality of the resulting fibers, and as a result of its design it can work on one amp.
4. The device contains many variables that contribute to the diversity of the diameter and porosity of the resulting nanofibers according to the application to be worked for.
5. The limiting concentration for polymer structure formation of the used polyacrylonitrile was determined.
6. We found that one variable, the spun polymer concentration, played a large role in the average diameter of the resulting fibers.

7. We found that at concentrations less than 5%W and greater than 15%W the spinning process was not carried out, and this is due to the viscosity of PAN used, which is a qualitative property of the polymer used.
8. We found that the voltage change has a clear effect on the average fiber diameter if other variables are held constant.

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