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Electro spun PAN Nanofiber with Optimized Diameter

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ABSTRACT
The recent trends in nanotechnology have led to an improvement in the manufacturing quality of smaller mechanical, electronics, and optical products and devices; all the attention is focused on producing compact, portable, and lightweight equipment and devices to reduce the overall energy consumption. Nanofibers are increasingly being used in a variety of applications. As a result, the researcher focuses on the development of nanofibers. They can be produced by various techniques, but the electrospinning method is the most commonly used in the industry. Polymer solutions can be used to create fibers with diameters, which has a high rate of production and requires a relatively low cost. In this work, an effort is made to produce nanofibers of polyacrylonitrile (PAN) insolvent dimethyl formamide (DMF) that have a minimum diameter. Four important controllable parameters are considered, which can affect the electrospinning process and resultant PAN nanofibers: flow rate, applied voltage, concentration, and distance between the collectors drum and syringe tip. The Taguchi design approach has been used to find the optimized electrospinning parameters that result in the minimum PAN nanofiber diameter. The diameter and surface morphology of PAN nanofibers were analyzed using a scanning electron microscope (SEM). The results were used to find the variance (ANOVA) analysis, which explains the significance of the parameters on the diameter of PAN nanofiber and predicts that flow rate is the most dominating parameter for PAN nanofiber diameter. The optimized set of produces minimum fiber diameter of 58.467 nm.

Keywords: Nanomaterials, Electrospinning, Carbon Nanofibers, optimization, Taguchi Method, and ANNOVA.

1. Introduction

The research on advancement in electronic devices has led to smaller dimensions of components and challenges in the field of manufacturing of parts at a micron level, becoming one of the most popular levels over the last few decades; Nanoscience and nanotechnology have made unprecedented strides in response to these advances and challenges (Singh, Lye, and Miao, 2021) and (Mottaghitalab and Haghi, 2011). Research has been carried out to develop advanced methodologies and techniques for the preparation of nanomaterials and their characterization, since nanomaterials have many commercial and functional applications (Xue, Wu, Dai, and Xia, 2019). Nanofibers are one-dimensional nanomaterials with various desirable properties like low density and a very large specific surface area due to its small diameters; the membranes of nanofiber have large porosity and good pore interconnectivity (Bolarinwa, Onuu, Fasasi, Alayande, Animasahun, Abdulsalami, Fadodun, and Egunjobi, 2017). They have excellent mechanical properties in proportion to their weight and have the ability to produce mesh at the micron-scale. Because of the exceptional properties, characteristics, and features of these nanofibers, they can be used in many commercial and functional applications as a material for advanced electronic devices (Gugulothu, Barhoum, Nerella, Ajmer, and Bechlany, 2018) and (Bansode, Chavan, Tarwadi, Jadhav, and Bansode, 2018).
There are various methods and techniques to produce nanofibers, including template synthesis, temperature-included separation of phase, drawing, electrospinning, and molecular self-assembly. Electrospinning is the most widely used of these methods (Bolarinwa et al., 2017) and (Samadian, Mobasheri, Hasanpour, and Faridi Majidi, 2017). Electrostatic spinning is very simple as well as comprehensive technique used to manufacture nanofibers with diameters upto one micrometer from various materials like polymers, composites, and ceramics. Fibers with diameters of less than two micrometer are unable to be created by conventional mechanical fiber spinning methods. Continuous polymer nanofiber production from polymer solutions is the simplest and most effective method. The Nanofibers have verity of applications in the field of medical science, such as cosmetic skin masks, skin healing, skin cleansing, and skin therapy with medicines, as well as muscle and bone fracture healing. They have variety of applications in the field of life science as carriers for drug delivery, wound dressings and hemostatic devices. Air and water pollution are controlled by nanofiber mats; these mats can be used as liquid, gas, or molecular filtration processes. Nanofibers are used in sensor devices for cells, arteries, and veins. Nanofiber materials have large energy conversion and storage efficiencies than larger-sized materials. Nanofibers have several electrical and optical applications for ultra-small devices and liquid crystal optical shutters. Also,

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nanofibers have found number of applications in solar and fuel cells, anode materials, battery electrolytes, and super-capacitors in the electronic field (Shi, Zhou, Ma, Ma, Bridges, Ma, and Hu, 2015) and (Karaka¸s, 2015).

2. Proposed Methodology

The aim of this work is to determine the best set of electrospinning factors to minimize the diameter of PAN nanofibers using a single spinneret. The various experiments were carried out to investigate the effects of the electrospinning factors such as distance in the collector drum and the syringe tip, flow rate, voltage, and the concentration on nanofiber diameter at various levels of electrospinning parameters. The Taguchi design technique was used to find the best electrospinning factor for obtaining the minimum nanofiber diameter. The interconnections between the factors are investigated using an experimental design, which is based on the L-9 orthogonal array. SEM machine is used to measure PAN nanofiber and to study its morphology. The signal-to-noise ratio was used to enhance the experimental results, which were then examined using analysis of variance (ANOVA). A confirmation tests was performed at the optimal set to verify the result.

3. Electrospinning Process

One-dimensional nanostructures, wire, rod, tube, fibers, the belt are of current interest because of their variety of applications in mesoscopic physics and nanoscale electronics. One-dimensional nanostructures, in contrast to other nanostructures, can offer significant advantages for studying the electrical, mechanical, and thermal performance of dimensionality. For the potential application of one-dimensional nanostructures into existing microscopic devices, there is need to achieve a novel synthesis route for one-dimensional nanostructure with the nano-scaled diameter as well as microscopic length.

J.F.K. Ghule was the first to patent electrospinning, under the title ”Improved method of an apparatus for the electrically separating relatively volatile liquid component from the components of relatively mixed substances of the composite fluid,” which is a novel simple and exercise technique has been attracting numerous scientist attention for the growth of one-dimensional nanostructures consisting of a tunable diameter, continuous length, aligned direction, and a diversified and customizable composition. The force developed by electric field acting on the solution of polymer melts to create the electro-spun jet, and lastly formed fibers may be obtained by electrified jet stretching for electrostatic repulsion in the surface charge and solvent evaporation.

Electrospinning is a method that uses an electric field to produce polymer fibres from solution of polymer. When a voltage is fed to a polymer solution, droplet of a cone-shaped forms at nozzle’s tip. Electrospinning is a method and apparatus used for manufacturing artificial filaments by applying an electric charge to liquids that include solid materials contained within them. By passing the solutions from an electrical field formed in electrodes in a drop, they are split into a number of threads. The intertwined threads repel each other in the electrical field when spaced apart, thus they are pile up in parallel and are collected in a bundle.

3.1. Electrospinning Setup

Focused Ion Beam (FIB) writing, Electron-beam, lithography, chemical vapour deposition, solution method hydrothermal, electrospinning and other innovative techniques for the formation of one-dimensional nanostructures have all been developed since 1990. Electrospinning is the simplest method for fabricating a one-dimensional nanostructure with solid and hollow interiors, constant length, controllable diameter, and aligned direction, and diverse and controlled composition (Wang, Pan, He, and Cao, 2013) and (Zhuang, Jia, Cheng, Guan, Kang, and Ren, 2013). Electrospinning is a modification of the electrospray technique that involves an electrical charge force applied to a polymer solution, which is different from other methods for the formation of one-dimensional nanostructures. Solidified fiber can be achieved in electrospinning by stalling an electrified jet for the electrostatic repulsion in surface charges and evaporated solvent, as shown in Figure 1. Synthetic nature polymers, polymers and polymer alloys coated with functional nanomaterials could all benefit from this method. Electrospinning’s distinctive features provide distinct properties for a variety of applications (Li, Hao, Yu, and Na, 2008) and (Wan, He, and Yu, 2007).

The electrospinning setup has clearly shown in the figure 1. This setup contains the three measure components; the first component is the syringe pump with a plastic or metallic syringe, the second high voltage DC or AC power supply, the third one is a fixed collector plate or rotating cylinder. In the setup, the syringe is used to inject polymer solution at a controlled rate by a syringe pump. As voltage is applied through high power AC or DC power supply; the induced charges are spread over the surface of polarized solution on syringe’s tip. The charged polymer is transported toward the collector under the influence of the strong electric fields. The significance of

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electrostatic force in this application is to enhance the utilization of normal mechanical forces, such as hydrostatic or pneumatic, to generate a jet and reduce the size of the fiber, therefore the name Electro Hydrodynamic Jetting. Here the collector plate could be of good electrical conductivity to neutralize the charge carried out by the polymer nanofiber.

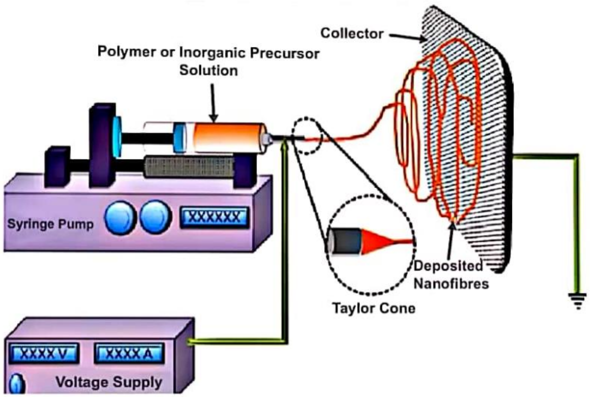

Figure 1: Electrospinning PAN schematic diagram with basic electrospinning setup

Electrospinning provides for significantly thinner fibers than conventional methods. Now, how the electrospun fibers are thinner down in electrospinning? A capillary device is used to pass a liquid polymer solution through. The droplet is then exposed to voltage, which decreases the surface tension and allows a very thin layer to be dragged out. The applied voltage is extremely high. The typical range is 8 to 25 kilovolts. The applied voltage seems to have an inverse correlation with the fiber diameter. Electrostatic forces and Gravitational forces are then push the fiber towards a collection plate. The fibers form a mesh network when they are randomly arranged on the collecting plate.

4. Experimental Design

4.1. Taguchi Optimization Technique

Taguchi Optimization methodology is a statistical tool that is commonly used in experimentaldesign to reduce noise factor variance and determine optimal factorss by Signal-to-Noise (S/ N) proportions graphs. The objective of current research to obtain the appropriate factors for obtaining the smallest possible PAN fibre diameter. The Taguchi technique involves three types of quality attributes: larger is better, nominal is better, and smaller is better. In this work, the effect of electrospinning parameters on fibre shape as defined by fibre diameter is investigated. Taguchi analysis is performed using smaller quality characteristics since the PAN fibre diameter should be reduced to a minimum (Mohite and Jadhav, 2015) and (Chaudhary and Ahuja, 2014).

4.2. Selection of Operating Parameters of Electrospinning

There are various parameters which may affect on diameter nanofiber in the electrospinning process; properties of the solution via viscosity, conductivity, and the surface tension, also applied voltage, concentration of solution, flow rate, the distance in the syringe tip and collection drum. Also, atmospheric parameters via humidity, temperature, and velocity of air in the chamber. The experimental design was used to investigate the effect of these electrospinning factors on the diameter of the fiber of PAN solution.

The experimental controlled parameters and its levels were selected by some preliminary experimentation using the one factor-at-a-time technique. It is observed in the preliminary investigations, the fibers are found to be collectible only in a specific range of the parameters and it is very essential to decide the range of each process parameter. The levels of electrospinning parameters mentioned in Table I were selected for experimentation as per the Taguchi optimization technique.

Table 1: Experimental parameters with levels

Parameters	Name of the Parameters	Levels		
		L-1	L-2	L-3
A	Distance (cm)	10	15	20
B	Flow Rate (ml/hr)	0.2	0.6	1.0
C	Voltage (KV)	16	18	20
D	Concentration (%)	8	10	12

4.3. Experimental Work

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The solution for the electrospinning was prepared by mixing the solution in 10 ml solvent dimethyl formamide (DMF) and PAN using a magnetic stirrer of heating plate type with room temperature. The three sets of solutions were prepared for experimentation with the concentration of 8%, 10% and 12%. The solution was mixed for 1 to 2 hours to have proper mixing of the solution. Before the PAN was dissolved in DMF, the solution was translucent, but after mixing, the colour changed to light yellow. The nanofibers were manufactured for two hours and nanofibers films were collected on aluminum foil. The polymer solution prepared were filled in a syringe of 2 ml for each of the experimental runs. While the three governing parameters were varied the other parameters i.e rotating speed of the drum kept constant with 500 rpm. In this research work, Taguchi experimental design methodology is used with L9 orthogonal array.

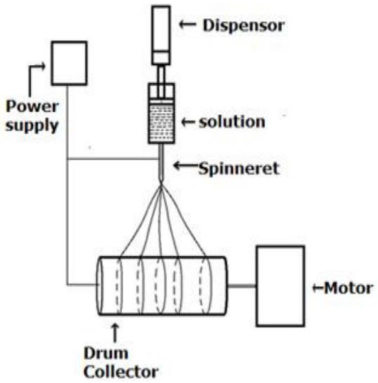

Figure 2: Experimental Setup of Electrospinning
(Zhou, Lai, Zhang, Qian, Hou, Reneker, and Fong, 2009)

Fiber Diameter measurement and S/N ratio: The fiber diameter was analyzed with a Scanning Electron Microscope (SEM). The value fiber diameter values and Signal-to-noise ratio (S/N ratio) optimization process. Under various noise conditions, the S/N ratio analyzes how the output varies in proportion to the nominal or target value. It was observed in each case applying the condition of 'lower is better' as the determining factor for nanofiber diameter.

Smaller is best, $\frac{S}{N} = -10\log\left[\frac{1}{n}\left(\sum y_i^2\right)\right]$; Where, y is observed data and n is the number of observations.

5. Result and Analysis of Experiments

The set of experiments were carried out with various PAN concentrations and flow rates with 0.25 MPa fixed air pressure. Each experiment was simply performed three times to obtain S/N values. The experimental results for PAN nanofiber diameter are given in Table 2.

Table 2: Experimental values of PAN Nanofiber Diameter and S/N Ratio

Expt. No.	Input Parameters				Output Parameters				
	Distance (cm)	Flow Rate (ml/hr)	Voltage (KV)	Concentration (%)	Fiber Diameter(nm) (Response)				S/N Ratio (dB)
	A	B	C	D	R1	R2	R3	Mean(μ)	
1	10	0.2	16	8	392.1	372.4	358.1	374.2	-51.4681
2	10	0.6	18	10	509.9	473.1	480.7	487.9	-53.7712
3	10	1	20	12	1189	1131	1064	1128	-61.0551
4	15	0.2	18	12	1009	988	1057	1018	-60.1584
5	15	0.6	20	8	312.3	401.2	351.5	355	-51.0499
6	15	1	16	10	613.6	753.4	699.4	688.8	-56.7921
7	20	0.2	20	10	547.7	649.5	673.6	623.6	-55.9312
8	20	0.6	16	12	418.2	472.3	389.3	426.6	-52.6286
9	20	1	18	8	1258.2	1314.4	1243.4	1272	-62.0923

5.1. Characterization of Nanofibers

Figure 3 shows characteristic morphologies of PAN nanofiber bundles captured using a scanning electron microscope. The ultrafine PAN fibres were spun with many beads at a concentration of 6% and a flow rate of 0.2 ml/hr. The morphology was changed from beaded fibre to uniform fibre structure, when the concentration increases from 8% to 12% and the flow rate increases from 0.6 to 1 ml/hr. When the concentration

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increases to 14 %, some amount of fibres were formed with large droplets. The fibre diameters of the PAN precursor nano-fibers in the as-electro spun bundle were approximately 58.467 nm. The overall orientation of nanofibers in the bundle was far from perfect, even though the majority of PAN nanofibers were aligned in the rotating direction.

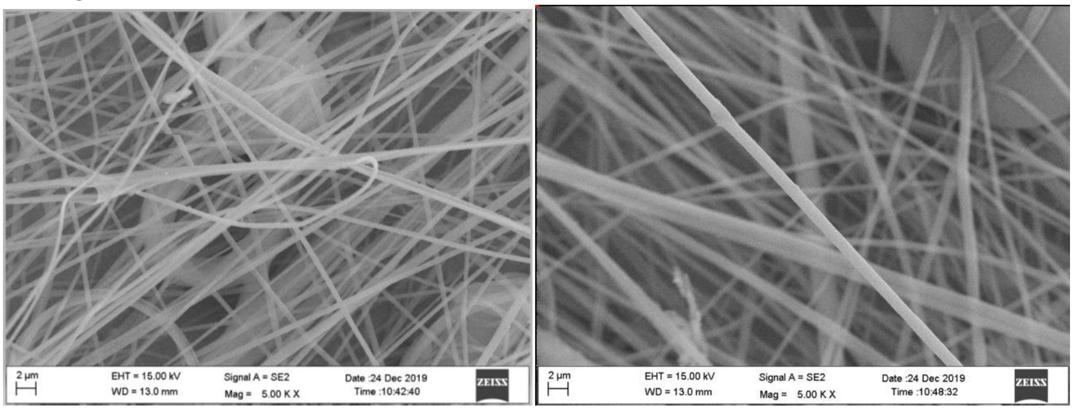

Figure 3: SEM images of PAN nanofiber

5.2. Taguchi Optimization Results

A. Taguchi Analysis:

Minitab statistical software was used to design all of the experiments, graphs, and analyses. The Taguchi analysis using Minitab statistical software gives the ranking of parameters based on the response, fiber diameter. Table 3 shows the mean of each response characteristic for all levels of each parameters. It produces rankings of input parameters based on delta statistics, which indicate the degree of influence in relation to one another. Also, it decided the most influencing and dominating parameter on the fiber diameter.

Table 3: Means of responses and ranking of parameters

Level No.	Distance	Flow Rate	Voltage	Concentration
1	663.4	671.9	496.5	667.1
2	687.3	423.2	926.0	600.1
3	774.1	1029.6	702.2	857.5
Delta	110.7	606.4	429.4	257.4
Rank	**4**	**1**	**2**	**3**

The delta statistic is the difference between the highest and lowest average for each parameter. Minitab statistical software assigns rankings based on delta values; rank 1 refers to the greatest delta value, rank 2 to the second-highest, and so on. The relative significance of each parameter to the response is indicated by these rankings. It is observed that, the flow rate affects more on fiber diameter, followed by voltage, concentration, and distance, in that order.

The Taguchi optimization result gives the main effects plots for mean and S/N ratio. It also provides interaction plots with different combination of input parameters, as well as the optimum combination of input parameters. This graphical representation of interaction plots makes it simple to analyze the effect of each parameter on response.

Graphical representation for Means and S/N Ratio

The main effect plots indicate the influence of various levels of factors parameters on the fiber diameter. It is obvious that the fiber diameter varies with the levels of the input parameters.

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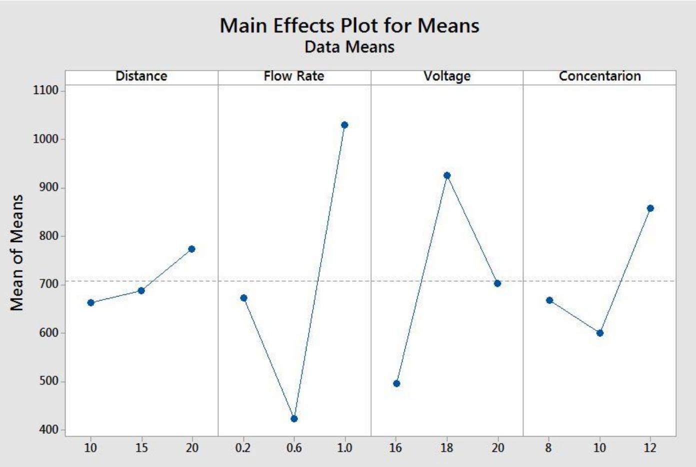

Figure 4: Main Effects Plots for Means

Figure 4 shows that the diameter of the fiber increases as the distance between the collector and the tip increases. But, diameter of the fiber decreases as flow rate and solution concentration increase to some extent and then it is increases with further increase in the flow rate and concentration. The diameter of the fiber increases as voltage increases to some extent and then it decreases as voltage increase further.

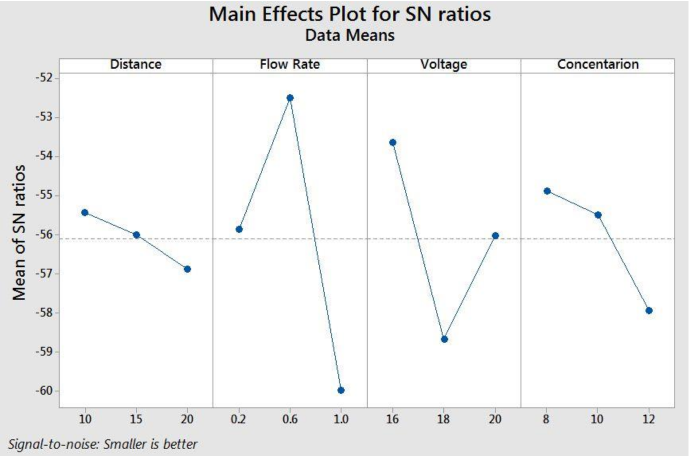

Figure 5: Main Effects Plot for S/N Ratio

The figure 5 shows the plots of S/N ratio values for fiber diameter. It is found that the level-I of distance, voltage, and concentration are -55.4315dB, -53.6296dB, and -54.8701dB respectively; and level-II of flow rate is -52.4832dB, which all are the optimum conditions in terms of S/N ratio. The flow rate has the major impact on the S/N ratio of all of these parameters. Thus, the maximum response value in the graphical representations of main effect plot of S/N ratio and the minimum response values in the main effect plot of means, which assist in the decide optimal set to achieve minimum diameter of the fiber.

The distance between collector and tip of 10 cm, flow rate of 0.6 ml/hr, voltage of 16 KV, and solution concentration of 10% is the optimal set, which gives the minimum fiber diameter.

Effect of Each Parameter

There are various parameters of electrospining, which affects on fiber diameter. The applied voltage is important parameter. It is observed that, as applied voltage increases, the PAN fiber diameter become larger. The optimal voltage was 16 kV and then further increasing the voltage would increase the electrospun PAN fiber diameter for some extent. Therefore, is stated that the voltage applied influences the diameter of fiber, but the amount of influence changes as polymeric solution concentration changes and it depends on the distance between the collector and the tip. If distance in tip and collector decreases, the electrical field increases; and as electrical field increases, the fibers expand and may develop structural abnormalities. Also, the polymer solution flow rate within the syringe is also important factor. As flow rate of solution increases, the fiber diameters decreases. [18, 19]

Plots for the interaction among the parameter

Figure 6 shows the interaction among all the input electrospining parameters. It is observed that there is no strong relation between electrospining parameters.

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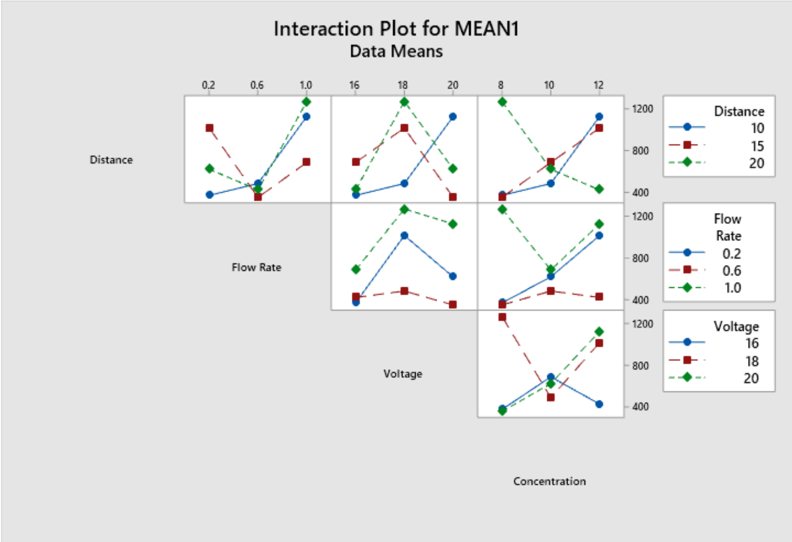

Figure 6: Interaction Plot for Mean

B. Analysis of Variance (ANOVA)

The decomposition of variance, also known as Analysis of Variance, can be used to determine the relative significance of various factors. Analysis of variance is used to make inferences about means rather than variances. The primary principle behind analysis of variance is to classify the variability in the data into two categories: that which can be described by group membership and that which cannot.

Table 4: ANOVA result Table

Sources	D.F.	Adj.SS	Adj.MS	F Value	P Value
Regression	4	328134	82034	0.52	0.730
Distance	1	18382	18382	0.12	0.751
Flow Rate	1	191888	191888	1.21	0.333
Voltage	1	63448	63448	0.40	0.561
Concentration	1	54416	54416	0.34	0.589
Error	4	633615	158404		
Total	8	961749			

The importance of factors is determined by the ANOVA findings. The F-ratio is used to determine the significance of the parameters. It includes the P-value, which is defined as the rate of significance of the operational parameters on response. A low P-value (usually 0.05) signifies that the null hypothesis is strongly confirmed. Table 4 shows the ANOVA results by minitab software.

5.3. Confirmatory Experiments

For confirmation of predicted results by the Taguchi optimization approach, experiment is performed with optimized parameters. The experiment for optimum combination of parameter distance as 10 cm, flow rate as 0.6 ml/hr, voltage as 16 KV and concentration as 10% produced mean fiber diameter of 58.467nm.

6. Conclusion

The polymer solutions were observed to be able to electrospun within a range of parameters, which varies depending on the polymer solution. As a result, it is essential to determine the range of parameters by which fibers can be conveniently collected on the collector by electrospinning. It is found that, the concentrated solution flow rate is the most influencing factor on fiber diameter of PAN followed by voltage while concentration and distance has relatively weak effect on fiber diameter. The percentage contributions of flow rate and voltage on fiber diameter are 66.7% and 43.9% evaluated by ANOVA Method. The fiber diameter depends on flow rate of concentrated solution. It is recommended that the flow rate should be 0.6 ml/hr for minimum fiber diameter for this experimental condition. The PAN fiber diameter increases, as distance between the collector and the spinneret increases; hence distance should not be more than 10 cm, however, if the distance exceeds intensity of the voltage, the fibres begin to scatter and become uncollectable on the collector. The optimal set of electrospining parameters are distance as 10 cm, flow rate as 0.6 ml/hr, voltage as 16 KV and concentration as 10% produces minimum fiber diameter of 58.467 nm.

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