Synthesis of TiO2 Nanofibers for Solar Cells and Their Analysis Using Statistical Tool-Taguchi Method

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Abstract*—the rapid population growth, global energy crisis, global warming, and diminution of fossil fuels at a shocking rate necessitate the need for renewable, inexpensive, clean, environmentally friendly, and practical alternatives to fossil fuels. The development of nice performance energy storage devices has be there in high demand thanks to the long cyclic functions. Additionally for prime power densities. Nano fibers stay wide used for energy storage devices in the meantime they need high area, flexibility, pore structure with interconnecting pores, light weight features and other altering properties. Electospinning method is flexible and capable method to synthesis the nanofibers. Nanofibers synthesized by electrospinning are characterized to have high surface area and very good porosity. Electrospun Nanofibers of specific composition of TiO_2 have been identified suitable for energy harvesting. This paper is mainly focused on the synthesis of TiO_2 electrospun nanofibers suitable for energy applications which range from sensing, capturing, conversion and storage for multitude of sources which could be wind, solar, photo, mechanical, pyro, magnetic in nature. This paper encapsulates the important considerations for synthesis of TiO_2 nanofibers using electospinning. Additional, the properties of electrospun nanofibers and its applications in solar cells, fuel cells, Nano generators, Lithium batteries and different energy applications. The benefits and drawbacks of electrospinning and a view on the probable future guidelines are also deliberated.

Keywords: **Electospinning, Nanofibers, Synthesis, Energy, $TiO_{2,}$ Energy applications.**

I. INTRODUCTION

The electrospinning method offers a potential sanctioning breakthrough to get rid of the barriers by dramatically reducing fiber diameters, leading to huge enhancements in fiber mechanical properties. There is also significant enhancement of other properties such as filtration, electronic, thermal conductivity of material by using these nanofibers. Electrospinning may be a non-contact drawing method within which a chemical compound solution drop originating on the tip of a spinneret is attracted towards a grounded collector underneath the action of a hydrostatic surface tension and potential difference applied. The electro-static forces basis the driblet to stretch, succeeding into bending unpredictability and flogging of the elongated jet manufacturing fibers of nano-scale diameter with very long lengths. Vaporization of solvents additionally takes place because the nanofibers are deposited onto the grounded collector.[1]

There are more than 150 polymer, composite solutions being tried and tested to synthesize Nanofibers catering to several application ranging from Tissue Engineering, Nano-Sensors, Caring Clothing, Improving Skin Masks, Filter Media and Electro-Magnetic and Photovoltaics'. Considering that Energy is most sought after field for researchers, we decided to venture into Solar Energy Harvesting for which we chose to synthesize TiO2 nanofibers.

II. EXPERIMENTAL SETUP FOR ELECTROSPINNING

Electrospinning could be a easy and versatile method to get ultra-thin fibers from a range of chemical compound, ceramic or composite solutions. The fundamental four components associated with the electrospinning process as seen in the schematic of Figure 1. In the electrospinning method, a solution driblet is served to the spinneret tip at a well-ordered rate employing a programmable dispensing pump. The dispensing pump may be a Model NE -1000 Multi-Phaser equipped by New Era Pump Systems INC., and has the capability of holding a syringe up to 50 mm in diameter. This pump can dispense solution over a wide range for the volume of 0.1 ml per min. to 10 ml per min. The solution driblet at the tip of the spinneret is acted upon by electro-hydrodynamic forces. The electrical forces area unit because of the potential drop applied between the spinneret and therefore the collector plate.[2]

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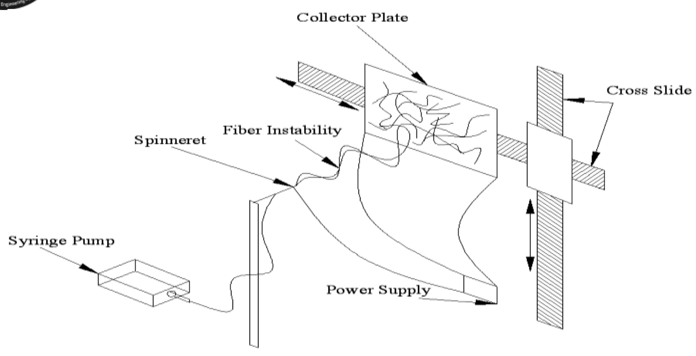

Fig. 1. Schematic of Electrospinning Setup

The spinneret is unbroken at a +ve potential and therefore the collector plate is sometimes unbroken grounded. A FC Series,120 Watt Controlled High Voltage DC Power deal provided by Glassman High Voltage, Inc., preserved a voltage of 18kV between the spinneret and therefore the collector plate. Because of this applied electrical phenomenon, the answer drop on a tip of the spinneret acquires +ve charge on the surface. The hydro-dynamic forces are because of the physical phenomenon of the liquid resolution. the answer drop is interested in the collector plate and forms a 450 semi-angle at the tip. The fashioned form is termed a "Taylor Cone" [3]. once the consistence of the answer is ample to produce stringiness, there's associate degree elongation of the drop into a jet,

III.Synthesis Procedure For TiO2/PVP Nanofibers

A. Material /Chemical Utilized

Titanium (IV) Isopropoxide (97%,Sigma Aldrich), Polyvinyl pyrrolidone (PVP, MW=1,300,000g/mol, Sigma Aldrich) , Acetic Acid (Sigma Aldrich) , Ethyl Alcohol (Sigma Aldrich).

B. Electrospinning Machine Process Parameters

Flow Rate:1.5ml/hr , Distance:15cm ,Voltage:15Kv ,Syringe Type:2ml , Needle Diameter: 0.2mm. Every solution was enthused on magnetic stirrer for ~1hour, The as-spun nanofibers were left to dry overnight.

C. Instruments Details

The technical specification of Electrospinning machine is as follows: Have high voltage supply up to 50Kv, User Interface for setting up feed rate, drum speed, applied voltage, Provides option for 2ml,5ml syringes for the solution, Comes with rotating drum ,thus producing continuous nanofibers Plastic Enclosed to avoid dust and provide vacuum environment, Can produce nanofibers in micro and nanometer scale.

D. Magnetic Stirrer with Hot Plate

The make of this instrument is Regular Warm Plate APS-421.The other technical specification related to instrument are as follows: Heating Plate Size:300×455mm, Digital Temperature Controller and Indicator, Continuous heating till 370°C, Electric Supply 220/230V AC,50/60Hz.

IV.FLOWCHART FOR SYNTHESIS PROCEDURE OF TIO2/PVP NANOFIBERS

V.CHARACTERIZATION OF TIO2/PVP NANOFIBERS

Here numerous different characterization technique which would recognize various different characteristics of nanofibers such as morphological structure, diameter, chemical composition, mechanical strength .These properties would help us to determine the areas of application were the nanofibers would be well suited. Out all the other technique following are the technique which we utilized to report the properties of nanofibers:

A. Scanning Electron Microscope

B. Tunnelling Electron Microscope

C. Elemental Data Analysis

A.Scanning Electron Microscope:

Working Principle: The Scanning Electron Microscope as the name suggests it uses the electron beam to scan and form the image of the sample. Unlike standard microscopes that uses the optical lightweight to scan and enlarge the sample, SEM makes use of the magnetism fields to enlarge the sample [4,5]. The most advantage of SEM is that it's high depth of sharpness which delivers info about the sample when observed at different distances from scanning level. It provides various information of sample such as Topographical, Morphology, Composition and Crystallographic Structure [6]

Instrument Details: The Scanning Electron Machine images were obtained from FESEM .The make of the machine was FEI, Model: NOVA NANOSEM 450.This machine was operated in High Vacuum Mode.

B.Tunneling Electron Microscope :

Working Principle: In Transmission Electron Microscope a ray of electron is passed through a thin specimen. An image of the sample is obtained once the beam of electron interacts because it permits over the sample and it's enlarged on the fluorescent screen. TEM is employed to check the composition, growth of layers , defects in semiconductors. High resolution is employed to research the standard, form, size and density of important wells, wires and dots.[6] Instrument Details TEM images were obtained from Tunneling Electron Microscope, Made in Netherlands, FEI, Model TECNAI G2 20 –U TWIN, Operating at 200kV, Filament LaB6.

C. Elemental Data Analysis :

In this process ,the elements are analyzed for their chemical composition sometime isotopes also. Elemental analysis can be quantative or qualitative [6].

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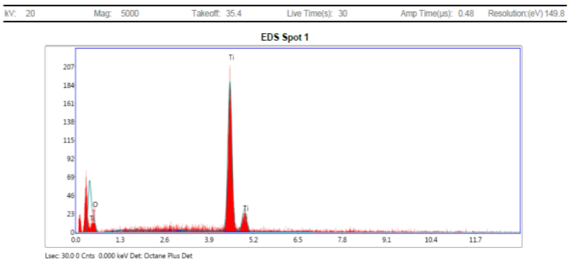

eZAF Smart Quant Results

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
O K	33.22	59.83	12.61	17.40	0.04	1.16	0.93	0.09	1
TiK	66.78	40.17	182.82	3.04	0.61	0.91	1.03	1.01	1

Fig. 2. Elemental Analysis of TiO2/PVP Nanofibers.

VI. Optimization of NANOFIBERS

To synthesize 1-D nanostructures one of the efficient technique is Electro spinning Process. The main advantages of this process is it is relatively simple, cost effective, produces continuous nanofibers. The morphological and structural characteristics such as diameter depends on polymer properties such as polymer concentration, molecular weight, viscosity. The 2nd set of parameters that affect the nanofibers properties are process parameter such as Applied Voltage (k V), Feed Rate (mL/h), and Tip-to-Collector Distance (cm) .These parameters can be optimized and analyzed by Taguchi Method. To finalize the parameters for optimization of nanofibers, two pilot experiment were conducted, depending on these experiments, the parameters were selected for Taguchi Method. Optimization involves a process to reduce set of parameters without interfering some constraints thus the reducing cost, improving quality and productivity, to achieve this an engineering statistical method is used called as Design of Experiment.

VII.DESIGN OF EXPERIMENT

The method of changing all the input parameters at same time and test simultaneously to observe the changes in output is called as Design of Experiment (DoE).This method was invented by British Statistician R.A.Fisher. It is also known as Full Factorial Design of Experiment .This method probes into all possible combination for a particular set of factors. As it involves lot of parameter thus resulting in large no. of experiment .To minimize the no. of experiment to a feasible level, a new method. It was invented to improve the overall quality of produced components and now being applied to engineering [7].

A.Taguchi Method

This method was invented by Genichi Taguchi. It is combination of Design of Experiment with optimized control parameters to find the top result, this method also considers noisy inputs and their effect during experimentation thus resulting the product or process robust. It focuses on obtaining the best level of quality feature with minimum variation .It is particular of the powerful and efficient technique for developing processes that working consistently and optimally on variety of condition. Taguchi methodology involves a careful choice of method parameters and bifurcating them into management and noise factors, It main aims is to spot correct management factors to get the optimal results of the processes. The Taguchi methodology contains 3 major style procedures for getting a sturdy method and result for very best quality. Following are three major design procedures considered in Taguchi Process of Optimization[8,9].

1) System Design: This focuses on development of systems to function under an initial set of optimal condition

2) Parameter Design: It is one the most important part of Taguchi method. Here finalizing the design parameter for the optimum level and achieving maximum robustness. This step is often known as Robustification.

3) Tolerance Design: once the parameter style isn't adequate for reducing the output variation, the last section is taken into account that conjointly referred to as Tolerance design.

There are a unit 3 sorts of signal factors or parameters that influence the output response of process/product. Out of that noise factors area unit troublesome or costly to manage and may cause variation in response, control factors area unit elite to scale back the sensitivity of product‘s response. The Taguchi methodology utilizes the different set of arrays referred to as ORTHOGONAL ARRAY. It’s wont to study the big range of variables/parameter with tiny range of experimental information. This helps to check the result of various parameters on performance of the system.

B.Orthogonal Arrays

Now that array, the columns are equally orthogonal, which implies for any pair of columns, all combos of factors level happens and that they occur associate equal variety of times. There are several customary orthogonal arrays obtainable, every of them is needed for a selected variety of freelance design variables and levels for any pair of columns, all combos of things level happens and that they occur associate equal number of times. There are several customary orthogonal arrays obtainable, every of them is needed for a selected variety of freelance style variables and levels. To choice an suitable orthogonal array for conducting experiment, the degree of freedom is to be computed. This is explained below:

Degrees Of Freedom: 1 for Mean Value, and 8= (2×4), two each for the remaining factors.

Total Degrees of Freedom: 9 the following diagram shows the furthermost appropriate orthogonal array for L9 experimentation [8, 9].

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TABLE I: *ORTHOGONAL ARRAY (L_9)*

L9(3^4)Orthogonal Array					
	Independent Variables				Performance Parameter Value
Exp . No	Variable1	Variable 2	Variable 3	Variable 4	
1	1	1	1	1	p1
2	1	2	2	2	p2
3	1	3	3	3	p3
4	2	1	2	3	p4
5	2	2	3	1	p5
6	2	3	1	2	p6
7	3	1	3	2	p7
8	3	2	1	3	p8
9	3	3	2	1	p9

C.Robust Design of System:

The main aim of the robust design remains to find the controllable process factors setting for which noise and variation has negligible effect on the product‗s or processes functional characteristics. It should be determined that main objective isn't to search out the parameter settings for uncontrollable noise variables, however the manageable style variables. The influence of noise on system will be found victimization the ratio:

Signal to noise ratio= S/N wherever S is that the variance of the performance parameter for every experiment N is that the total range of experiment.

This magnitude relation can verify the useful variation because of noise .Thus victimization this result, it'd be attainable to predict that management parameter settings can build the method insensitive to noise. There are three main standard S/N ratio generally applicable:

1) Larger the better: Thi1) Larger the better: this can be applicable for characteristics that are fascinating whose value ought to as large as possible.

n=10 Log10 [mean 1) Larger the better: this can be applicable for characteristics that square measure fascinating whose value ought to as giant as attainable.

n=10 Log10 [mean of sum squares of reciprocal of measured data]

2) Nominal the better: this can be chosen once such value is most desired.

n= 10Log10 [Square of Mean/ Variance]

3) Smaller the best: this can be choosen for all undesirable characteristics like ―defects‖, etc. that the perfect worth is zero.

of sum squares of reciprocal of measured data]

N= -10 Log 10[mean of sum of squares]

D.Eight Steps in Taguchi Methodology

The parametric design of Taguchi Method involves the following eight steps:

Step 1: Determine the Quality Characteristics to be enhanced

The first stage in Taguchi Method is to define the superiority characteristics to be optimized. This parameter whose deviation would have critical effect on product quality. It is the output or response to be considered

Step 2: Identify the noise factors and Test Condition

The main objective of this step is to recognize the noise factors that could have -ve impact on system performance and quality. These factors are those parameters that are uncontrollable they include variation in ecofriendly operating conditions, the components used is outdated etc.

Step 3: Identifying the Control Parameters and their Alternate Levels

This step involves identifying the control parameters which were concluded to have significant effects on quality characteristics. These are those factors that can be set and maintained .The test values or levels for each parameter must be chosen at this point.

Step 4: Selection of Orthogonal Array and Defining Data Analysis Procedure

This step involves identifying suitable orthogonal arrays for controlling noise and other parameters. After selecting the array, a process to simulate the difference in the quality characteristics due to noise need to be defined .The most common approach is Monte-Carlo simulation.

Step 5: Conducting the Matrix Experiment

This step objective is conducting matrix experiment and recording the results. Only taguchi method shall be used in any condition where there is controllable method.

Step 6:Analyzing the Data And Defining the Optimal Levels

After conducting the experiment, the optimal test parameters within experimental design must be determined. To analayze the result .Taguchi Method uses the statistical measure of performance called as Signal-to -Noise Ratio.

Step 7: Examination of Data

In this step, the experimental data is an analyzed with different methods such as ANOVA which is also called as Full Factorial Design.

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VIII. Pilot Experiment

A. Pilot Experiment 1

1) Chemical Utilized: 1.5ml of Titanium Isopropoxide, 0.675g of Polyvinylpyrrolidone, 3mL of Acetic Acid, 3mL and 7.5mL of Ethanol

2) Electrospinning Parameters: Applied Voltage:20kV, Distance: 15cm, Flow Rate:1ml/hr

3) Other Parameters: The solution has mixed on Magnetic Stirrer for 2-3hours

4) Result Obtained: It was observed there is beads formation in nanofibers. Nanofibers were obtained with very thin diameter.

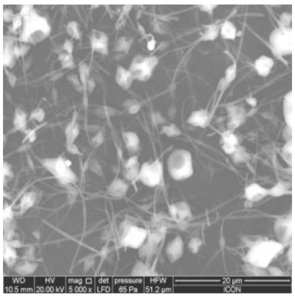

Fig. 3. SEM Images exp. No.1

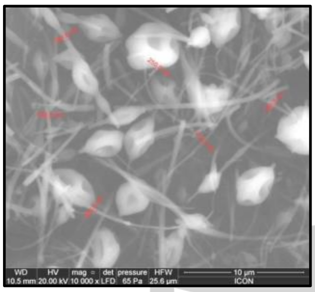

Fig. 4.SEM Images exp. No.1 without Diameter

A.Pilot Experiment 2

1) Chemical Utilized: 0.75ml of Titanium Isopropoxide (T 0.45g of Polyvinylpyrrolidone (PVP), 1.5mL of Acetic Acid 10mL of Ethanol

2) Electrospinning Parameters: Applied Voltage:15 kV, Distance: 15cm, Flow Rate:1.5mL/h

3) Other Parameters: The solution was mixed on Magnetic Stirrer for ~1hours and Was kept overnight before keeping for electrospinning.

4) Result Obtained: It was observed there is lesser beads formation in nanofibers. Nanofibers were obtained with average thickness of diameter.

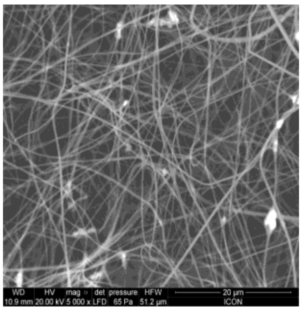

Fig. 5. SEM Images exp. No.2

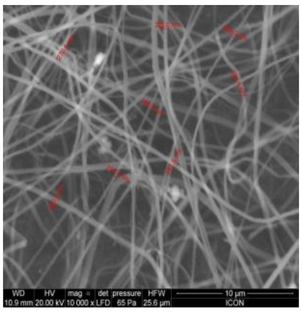

Fig. 6.SEM Images exp. No.2 without Diameter

XI.OPTIMIZATION BY TAGUCHI METHOD

Taguchi Method is the statistical method that emphases on finding the parameters producing the best levels of quality characteristics with lowest difference. Taguchi uses Orthogonal Arrays (OA) that are used to perform a set of experiments. Results of these experiments are then used to analyse the data and predict the quality of materials. . In this experiment, four input parameters which prominently influence throughput of electrospinning process were chosen based on literature survey. It is concluded that diameter of nanofibers synthesized by electrospinning is dependent on these factors[10], thus best approach was to use three levels of each parameter for evaluation, using Taguchi approach. After conducting the pilot experiment for selection of parameters. The L9 orthogonal array shown in Table 5.1 is selected for the experiments which accommodate four levels and three parameters.

TABLE III : Parameters for L9Taguchi Orthogonal Array

Factors	Coded Form	Level		
		1	2	3
Concentration[gm]	A	0.4	0.45	0.5
Flow Rate[ml/hr]	B	1	1.25	1.5
Distance[cm]	C	16	18	20
Applied Voltage[kV]	D	15	17	18

To confirm with the results the iterations of experiment was performed. Table: I is the orthogonal matrix for experiment iteration. Fig. 7 and Fig. 8 represents the SEM images of the experiment performed.

A.SEM Images of Matrix Experiment Performed:

After performing each of the trials in experiment matrix, the obtained fiber mat was then analyzed with the help of Scanning Electron Microscope, Make:FEI, MODEL Name: NOVA NANOSEM 450.The electrospun nanofibers were observed with different magnification to have a better look at the diameter and morphology . SEM Images are shown below

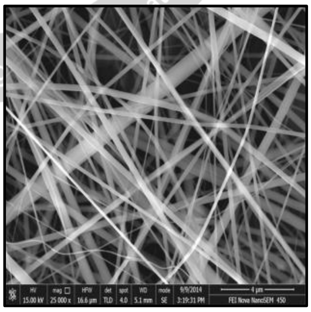

Fig. 7. SEM Images OF T1

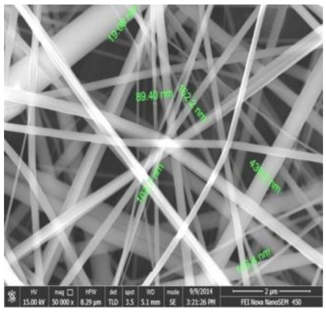

Fig. 8.SEM Images of T1 without diameter

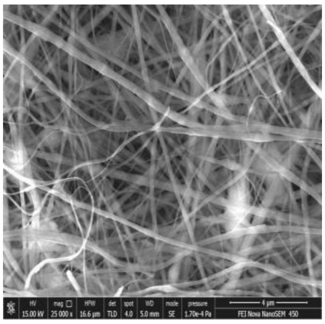

Fig. 9. SEM Images OF T2

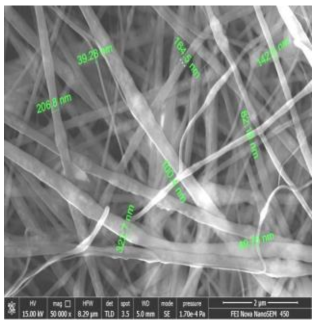

Fig. 10. SEM Images of T2 without diameter

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X.ANALYSIS OF VARIANCE (ANOVA)

To determine vital factors and optimum combination of things, AN Analysis of Variance (ANOVA) was utilised so as to supply a live of confidence by determinate and analyzing the info variance .In ANOVA, the sum of squares of every factor, the entire variation and also the proportion contribution were calculated. By conducting the ANOVA the importance of the regression equation is calculated. F- Test analysis is done to know the importance between response diameter and other parameters.

TABLE IV: Analysis of Variance

Sr. No.	Factors	Sum of Squares	Degrees of Freedom	Variance of Mean Square	F0	% Contribution
1	A	87919.29	2	45959.652	156.70	63.65
2	B	54581.76	2	72290.76	21.06	48.34
3	C	2337.49	2	5778.89	5.23	4.07
4	D	6439.52	2	32194.925	14.9	3.2
	ERROR	32146.41	9	4568.53		
	Total	1969395.012				

XI.RESULTS & DISCUSSION

1. Polymer Concentration: As there is increase in concentration from 0.40 to 0.50gm there is increase in diameter. At the initial level the diameter was reduced due to other factors affecting and dominating the effect of concentration. But as the concentration increases there is slight decrease in diameter which is negligible.
2. Flow Rate: As flow rate increases from 1.0 to 1.5ml/hr, diameter slightly reduces due to other factors affecting at that state of point. But as it increases, the time required for elongation is reduced which thus results in increase of diameter. The variation is less for effect of flow rate. Synthesis of TiO2/PVP Nanofibers by Electrospinning Process & Potential Applications Mean of Means
3. Distance between needle & drum: As the distance increases, diameter reduces due to more time for elongation of jet as shown in fig. 9.
4. Voltage: As the applied voltage is increased from 15kV to 18kV, diameter reduces. But as the voltage is increased there is slight increase in diameter of Nanofibers. The variation in result is negligible. Fig.11 shows the interaction between flow rate, distance, and concentration. Fig 12 shows the interaction between the all the parameter.

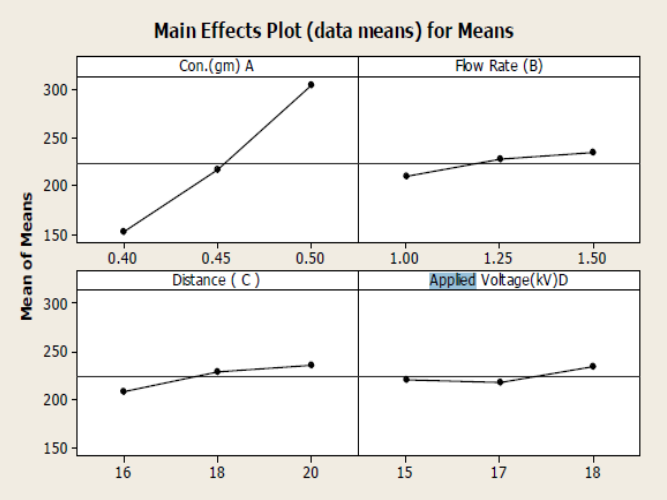

Fig. 11.Main Effects Plot for Means

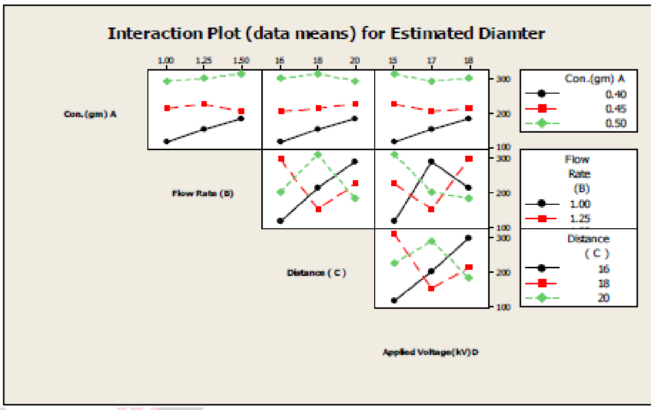

Fig. 12 Interaction Plot for Various Parameters

In Taguchi Method after conducting the experiment, the optimal test parameters need to be determined. To examine the outcomes, the Taguchi technique uses a applied mathematics live of performance known as as S/N that has origin from electrical management theory.

Signal to Noise(S/N) Ratio The S/N ratio developed by Dr.Taguchi is a performance control measure to select control levels that best cope with noise. It takes both mean and the variability into consideration. In other words, the S/N ratio is the ratio of mean (signal) to standard deviation (noise).The unit of S/N ratio is decibel (dB). The S/N equation depends on the criterion for the quality characteristics to be optimized. While there are many different possible S/N ratio, three of them are most commonly used:

1. Larger is better
2. Nominal is better
3. Smaller is better

In our experiment, we have chosen Smaller is better S/N ratio for finding out optimized Nanofibers diameters. Following is the formula: S/N ratio= -10log10[MSD] MSD=Mean square deviation.

Confirmation Test :

The regression equation is
E=-712+1523(A)+51.7(B) + 6.89(C)+ 3.71(D)

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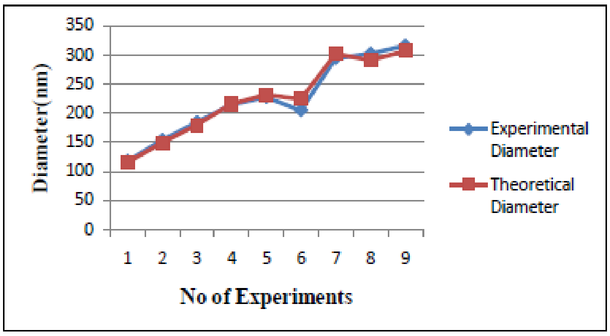

Fig. 13 Comparison of Experimental and theoretical diameter of TiO2/PVP Nanofibers

Values for R-Square and R-Square adjusted: The Fig 13 shows the Comparing the experimental and theoretical values of the mean error it was noted that it is 0.50% less, which is negligible and the Rsquare and Rsq-adj values are near about 90% , thus it can be concluded that the regression model developed is showing consistency in the values of diameter with the experimental diameter obtained.

X. Conclusion

It was found that firstly the concentration has the maximum effect on fiber diameter. and secondly the flow rate .Taguchi Method was used to obtain optimized Nanofibers. Using Four parameters and three levels 117nm was the optimized Nanofibers electrospun with distance 16cm, 1ml/hr feed rate,15 kV applied voltage and concentration 0.4gm.Polymer concentration was found to affect the diameter of as-spun Nanofibers. Model developed by regression method showing the consistency in experimental value of diameter.

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