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Morphological and Electrical Characterization of Polyacrylonitrile Nanofibers Synthesized Using Electrospinning Method for Electrical Application

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***Abstract*—**Electrospinning is the most widely utilized method to create nanofibers because of the direct setup, the capacity to mass-deliver consistent nanofibers from different polymers, and the ability to produce ultrathin fibers with controllable diameters. Smooth and much arranged ultrafine Polyacrylonitrile (PAN) nanofibers with diameters going from submicron to nanometer were delivered utilizing Electrospinning technique. PAN powder was used as a precursor to prepare the solution utilized as a part of this process. At the point when the electrostatic repulsion contradicted surface tension, a charged stream of polymer solution was shot out from the head of the spinneret and along these lines ultrathin nonwoven fibers were created. The effect of electrospinning parameter such as applied voltage, feed rate, concentration of polymer solution and tip to collector distance on the morphology of electrospun PAN nanofibers were investigated. The nanofibers were heat treated for carbonization to examine the changes in properties and composition to make for electrical application. Scanning Electron Microscopy (SEM) was performed before and after carbonization to study electrical conductivity and morphological characterization. The SEM images have shown the uniform fiber diameter and no beads formation. The average diameter of the PAN fiber observed 365nm and 280nm for flat plat and rotating drum collector respectively. The four probe strategy was utilized to inspect the electrical conductivity of the nanofibers and the electrical conductivity is significantly improved with increase in oxidation temperature exposed.

***Keywords*—**Electrospinning, polyacrylonitrile carbon nanofibres, heat treatment, electrical conductivity.

I. Introduction

ELECTROSPINING, otherwise called electrostatic fiber spinning, is a modern and proficient strategy to create nonstop nanofibers that ranges from submicron diameters across down to nanometer diameters. It was first presented by Formhals in 1934 [1]. The utilization of this strategy ranges from labs upto industries on a vast scale. Numerous sorts of polymers have been effectively electrospun into nanofibers in recent years generally in dissolvable solution and some in melt form [6]. PAN is used as the antecedent for 90% of carbon fiber creation as it has high carbon yield [2]-[4]. PAN possesses extraordinary properties, for example, low thickness, thermal stability, high quality and modulus of elasticity. Utilizing high potential electric field, this procedure can draw fine fibers from the polymer solution and does not require the utilization of coagulation science or high temperatures. A high voltage DC electric supply is connected between the end of the syringe and a collector. The electrospun filaments are generally saved on the collectors as nonwoven nanofibers mats. Nature of electrospun fibers for the most part rely on solution, process and ambient parameters [9]. Controlling these parameters, the desired property or usefulness can be accomplished, offering greater adaptability in surface functionalities.

Oxidative stabilization is must to prevent melting or fusion of the fibers. The main objective of adjustment is to amplify the final carbon yield in the subsequent stage of carbonization. Carbon nanofibers for composite applications are fabricated from precursor polymer nanofibers [5]. The huge surface region (which is 103 times of that of a microfiber) accessible on nanofibers prompts the enhanced properties in different applications, for instance, defensive apparel, tissue building and filtration advancement to help complex nanomaterial [6], [7]. However electrospinning presently has a few limitations, for example, surprising expense of PAN powder and making exceptionally aligned nanofibers [8]-[14]. A few difficulties, for example, to get nanofibers with consistent diameter across, to enhance the productivity of nanofibers and to gather consistent single nanofibers have not yet been accomplished totally.

The literature review demonstrated that there is a need to optimize the parameters to create smooth nanofibers with no bead development. The present examination emphasizes on getting ready smooth highly aligned nanofibers by controlling the procedure parameters utilizing E-spin software of Electrospinning machine ES-2. Subsequently, the fibers were carbonized to deliver carbon fibers from PAN nanofibers.

II. Experimental Techniques

A. Materials

The polymer solution having concentration 5% utilized as a part of Electrospinning was comprised of PAN powder (molecular weight- 150000) along with N, N Dimethyl Formamide (DMF Solvent) which were acquired from E-Spin Nanotech Pvt. Ltd., Kanpur, India. Magnetic stirrer was used for making the solution by steady mixing at 60°C for 3 hours and afterward resulting cooling to room temperature [15].

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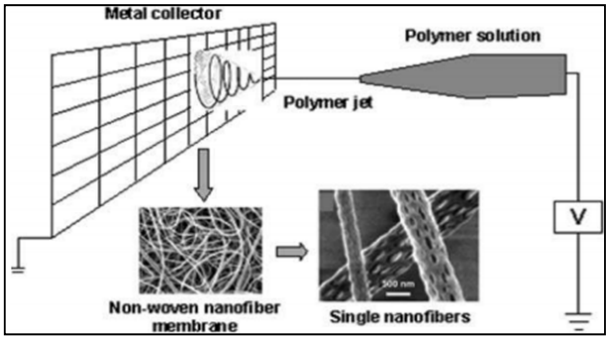


Fig. 1 Schematic diagram for the electrospinning process [7]

B. Electrospinning Setup

Electrospinning Super ES-2 machine fundamentally comprises of a high voltage control supply; two syringe pumps and a collector which is grounded as shown in Fig 1. Other apparatus incorporates a voltage controller box, a chamber heater, a dehumidifier and an exhaust which is all PC controlled. The polymer solution is gathered in the syringe. The syringe pump is used to get a continuous solution feed rate which can be controlled. The machine consists of various types of collectors as per requirements such as Flat Plate, Rotating Drum, Disc, etc. Electrospinning process uses a high voltage to draw the polymer jet of solution. The syringe pump is utilized to get a nonstop solution feed rate which can be controlled. The machine comprises of different kinds of collectors according to prerequisites, for example, Flat Plate, Rotating Drum, Disk, and so forth. Electrospinning process uses a high voltage to draw the polymer stream of solution from the syringe. The solution globule which is held at the tip of the spinneret because of its surface tension is strongly restricted by electrostatic force. In this manner, the surface of the liquid globule advances toward getting to be charged. At the point when the intensity of the electric field builds, the liquid droplet at the tip of the needle is broadened and a Taylor cone is framed from the syringe. At the point when the connected voltage comes to a critical value, a stream of fluid ejects from the head of the Taylor cone. Because of the bending instability, stretching and thinning of fibers occur which comes about into the development of smooth uniform nanofibers [10], [11]. The solvent gets vanished while going towards the collector and only long and thin fibers are gathered on the target as irregular nonwoven mat [2], [3]. Fig. 2 shows the E-spin Nanotech Electrospinning setup used for experimentation.

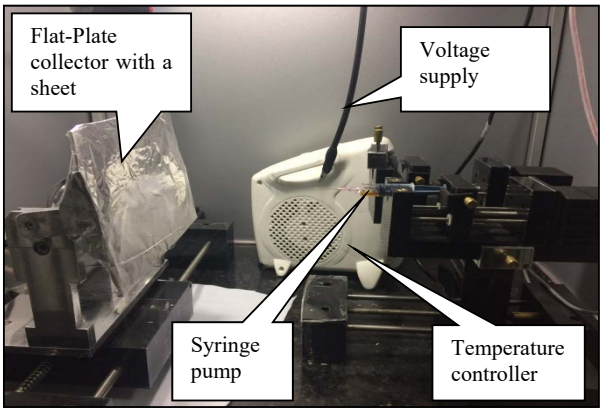


Fig. 2 E-spin nanotech Electrospinning setup used for experiment

C. Optimization of Processing Parameters

The fibers can be optimized by altering the composition of the solution and the design of the electrospinning process, with changing the arrangement of electrospinning set up the morphology of the fibers being delivered is as shown in Table I. The main aim of optimization of processing parameters is to enhance the morphological and electrical properties pan nanofibers. The concentration of the solution and applied voltage play vital role in morphological properties of fibers and distribution of fiber [17]. The nature of the flow can be regulated by varying the separation and the voltage until the point that a stable flow is noticeable. If the droplet of polymer solution at the head of the syringe is slanting toward the collector yet is not confining a flow, then the voltage can be increased. The flow of nanofibers from the spinneret is spreading all over rapidly on the collector. This can be controlled either by decrease the voltage or increase of the distance between the syringe tip and the collector. If the jet continues shaking use a higher polymer concentration or include a solvent with a slower drying [16]. Fig. 3 shows electrospun PAN fibers using flat plate collector on E-Spin Nanotech setup. Average diameter is determined by SEM analysis is shown in Table II.

TABLE I
PARAMETERS OF ELECTROSPINNING FOR SYNTHESIS OF PAN NANOFIBERS

Collector	Distance between needle and collector (cm)	Applied Voltage (kV)	Processing duration (hours)	Syringe Capacity (ml)	Flow Rate (ml/hr)	Relative Humidity (%)	RPM of collector
Flat Plate	15	12	2	2	1	54	---
Rotating Drum	15	13	2	2	1	56	500

(a) (b)

Fig. 3 PAN fibers using (a) Flat plate collector and (b) Drum Plate Collector

TABLE II
AVERAGE DIAMETER OF PAN FIBERS FROM SEM

Collector type	Voltage (kV)	Fiber Diameter (nm)
Flat Plate	12	459 ± 30
Rotating Drum	13	351 ± 35
Flat Plate with Carbonization	12	365 ± 30
Rotating Drum with Carbonization	13	280± 35

D.Heat Treatment

The fibers were fully dried before experiencing heat treatment. The PAN nanofiber web was expelled physically from the aluminum foil put on the collector. First step is to stabilize the nanofibers in presence of oxygen. Muffle Furnace was utilized to perform stabilization. Fig. 4 shows the stabilized PAN fibers of both the collectors. Oxidative stabilization assumes a vital part in the carbonization.

Properties, for example, mechanical and chemical stability are enhanced by heat treatment. Carbonization was performed utilizing the technique for Chemical Vapor Deposition in a nitrogen climate at a purging rate of 80 ml/min [21], [22]. Temperature was expanded progressively at a rate of 7°C/min upto 700°C and a dwell time of 1 hour. Carbon fibers were removed after cooling down to room temperature [13]. Fig. 5 shows the carbonized PAN fibers after heat treatment.

(a) (b)

Fig. 4 Stabilized PAN fibers at 700 0 C for (a) Flat plate collector (b) Drum plate collector

E. Morphological Characterization

The network of nanofibers was inspected utilizing a Philips XL-30 Scanning Electron Microscope (SEM).A small sample of nanofibers and carbon fibers was adhered onto SEM plate with a conductive tape. The nature of fibers created was analyzed from photograph sampling using SEM imaging. The average diameter of each example was figured from SEM pictures with high amplification of 10 randomly chosen fibers [20].

(a) (b)

Fig. 5 Images of Carbonized PAN fibers (a) Flat plate collector (b) Drum plate collector

F. Electrical Conductivity of Carbon Fibers

The electrical conductivity of the CNFs was calculated using a four point probe method. Proper contact between the yarns and probes was guaranteed. This system includes bringing four similarly separated probes in contact with the material of obscure resistance. The two external probes are utilized as current source and inward two probes are utilized for estimating the subsequent voltage fall over the specimen. By using the four-point probe technique as shown in Fig. 6, the semiconductor sheet resistance can be evaluated as shown in (1) [18];

$$R = \rho \frac{L}{A} \quad (1)$$

where ρ is the resistivity of the conductor in ohmmeter. At a constant temperature, the resistance, R of a conductor is proportional to its length L and inversely proportional to its cross sectional A. The electrical conductivity of the sample was evaluated by the condition [18], [19] shown in (2);

$$R = \frac{I * F(\frac{t}{s})}{V * 2\pi s} \quad (2)$$

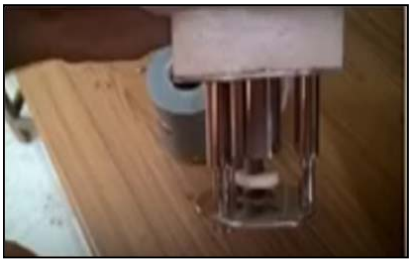
where t and s are respectively the thickness of the mat and distance between two consecutive probes. The thickness t of this mat was estimated carefully with a digital micrometer having a resolution of 1μm.The electrical conductivity of carbonized nanofibers mats was found increasing with raise in the temperature as shown in Table III.

Calculations:

Thickness of one carbon fibers sheet (t): = 1 mm
Probe distance (s): 2mm
$F(\frac{t}{s})$ = 2.78 (from standard table)

TABLE III
ELECTRICAL CONDUCTIVITY ON BASIS OF VARIOUS TEMPERATURE OF PAN CARBON FIBERS

Sr No	Temp (°C)	Temp (K)	Voltage (mV)	Resistivity (ρ)	Conductivity (S/cm)
1	30	303	0.13	7.69	85.08
2	40	313	0.12	8.33	92.17
3	50	323	0.11	9.09	100.55
4	60	333	0.10	10	110.61
5	70	343	0.08	12.5	138.26
6	80	353	0.06	16.67	187.35
7	90	363	0.05	20	221.22



(a)

(b)

Fig. 6 Experimental setup for conductivity measurement (a) Four point probe (b) Digital micrometer

III. Results and Discussion

Fungilab Spain Viscometer was used to measure the viscosity of the polymer solution which was found out to be 76.6 centi Poise All the fibers were produced in a controlled atmosphere by adjusting all the parameters viz. solution, process and ambient parameters, in order to obtain ideal and high yielding conditions. Table I shows the optimum processing conditions to produce nanofibers using flat plate and rotating drum collector. Fiber alignment was achieved using different types of collectors used to create nanofiber webs.

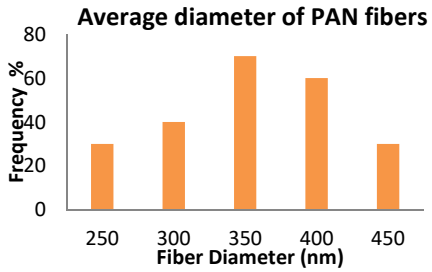


Fig. 7 Fiber diameter distribution of carbon nanofibers (CNFs) from SEM images

The average fiber diameter of the CNFs got from this experiment were evaluated by Image J software; installed in the SEM assembly and the statistical population is shown in Fig. 7, the PAN nanofiber diameter is moderately uniform of 350 nm. There was a slight decrease in diameter of PAN nanofibers after carbonization. Voltage applied during Electrospinning process was in the range of 11 to 13kV. For Flat Plate collector, fibers started forming at 11kV and were discontinuous. When the voltage was increased to 12 kV the fibers were continuous and highly aligned. At 13 kV the degree of alignment decreased. So, 12 kV was set as the optimum voltage for flat plate collector and 13 kV for rotating drum type.

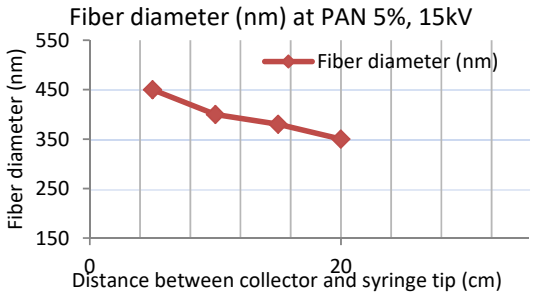


Fig. 8 Effect of separation between syringe head and collector on fiber diameter

It has been demonstrated that the distance between the collector and the syringe tip can likewise influence the fiber diameter and structure as shown in Fig. 8. In short, if the separation is too short, the fiber won't have sufficient time to solidify before going to the collector, while if the separation is too long, fiber along with beads can be formed. Drying of fibers while its travel towards the collector is a very important factor, so optimum separation distance is suggested is 15cm from this study.

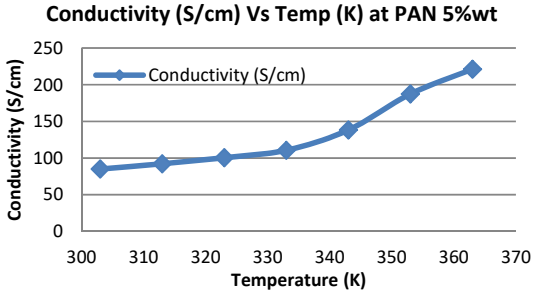


Fig. 9 Relationship between temperature and conductivity of PAN carbon fibers

The process of carbonization led to the formation of three dimensional carbonaceous structures and the fiber diameter reduced by 20-30%. Figs. 10 and 11 show SEM images of electrospun PAN nanofibers for both collectors and Fig. 12 (A) and (B) showed carbonized nanofibers for flat plate and rotating drum collectors. The average fiber diameter of nanofibers produced from PAN solution with concentration 5 wt % after carbonization process was analyzed using the average diameters of the CNFs obtained from this experiment were estimated by ImageJ software (JEOL, Japan) installed in the SEM apparatus and are 365nm and 280nm for flat plat collector and rotating drum collector respectively. The average fiber diameter after carbonization was reduced to 30-44 % of PAN fibers. The measurement of the electrical conductivity of

carbon nanofibers have showed that increasing the oxidation temperature gradually increases the conductivity of the nanofibers is shown in Fig. 9.

Assessing the electrical properties with four probe method showed that the PAN nanofibers had low electrical properties as compared to carbonized PAN nanofibers. If the carbon fibers are made electrically conductive, they can possibly be utilized as terminal materials in batteries. Specifically, they can be utilized as a part of the new carbon fiber battery idea in which the fiber is covered with a thin polymer electrolyte. Batteries made from carbon fibers are light in weight, space efficient, cost effective and ecologically friendly alternative to today's batteries and power storage options.

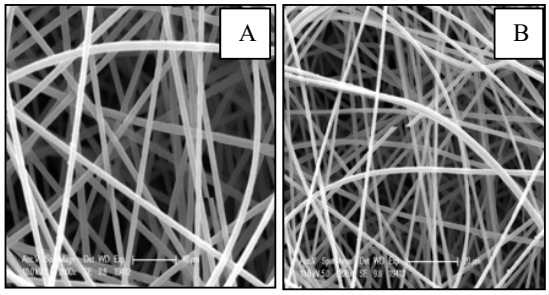


Fig. 10 SEM Images of Electrospun PAN nanofibers (A) at 2000X resolution and (B) at 1200X resolution using Flat plate collector

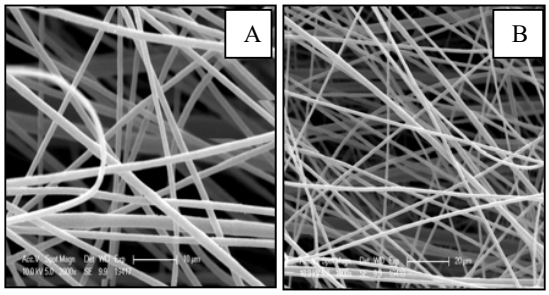


Fig. 10 SEM Images of Electrospun PAN nanofibers (A) at 2000X resolution and (B) at 1200X resolution using rotating drum collector

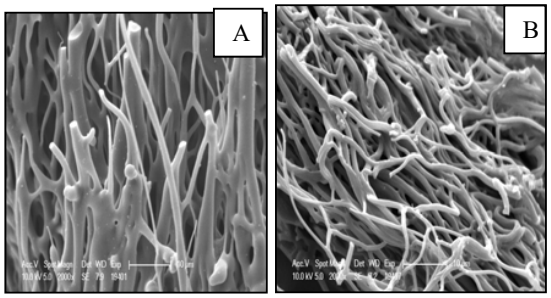


Fig. 12 SEM Images of carbon nanofibers(A)flat plate collector (B) rotating drum collector at 2000X resolution with carbonization

IV. Conclusion

Electrospinning is still the most proficient method for constant manufacturing of nanofibers. In this paper, nonwoven nanofibers were effectively electrospun utilizing electrospinning procedure from polymer solution having concentration 5wt %. All the parameters were optimized to deliver smooth and very oriented nanofibers. Flat plate collector and rotating drum collector were used to accomplish fiber alignment. PAN Nanofibers initially experienced oxidative stabilization followed by carbonization bringing about reduction of fiber diameter. SEM was performed on electrospun carbon nanofibers to study the morphological properties and have shown the orientation of fiber consistency of the fiber diameter. The average fiber diameter produced from PAN solution with concentration 5 wt %, after carbonization process are 365nm and 280nm for flat plate collector and rotating drum collector respectively. A small number of polymers have been electrospun into nanofibers. The electropsinning of PAN nanofiber is slow throughput but better process. The storage of electrospun nanofibers is method is developed to take care of the nanofiber mats. In this experimentation, zip pouch bags were utilized to store the fibers and appropriately dried the fibers before storage to prevent moisture contact. Another test is to create consistent single nanofiber and of uniform diameter. The most optimum voltage is 12 kV for flat plate and 13 kV for rotating drum gave nonstop single nanofiber, but the nanofibers with uniform diameter across still remains a challenge. However, the SEM images have demonstrated that nanofibers were smooth without beads formation. The electrical conductivity of the carbonized nonwoven nanofibers was measured and has shown the significant improvement with increase in the temperature oxidation exposed.

Future focus of this study for synthesized carbon nanofibers is field of the energy applications. Carbon nanofibers will also be used as light weight structural component with energy devices. Batteries made from carbon nanofibers will be light in weight, space and cost effective and ecologically friendly alternative to today's batteries and power storage options.

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