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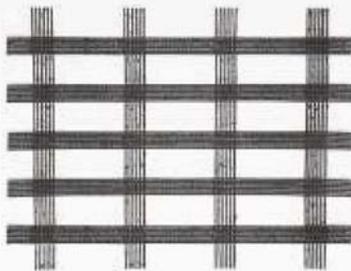
January, 2021



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## CHAIRMAN MESSAGE



Mr. S. K. Saraf  
Chairman, BTRA Governing Council

Dear All,

Greetings...

I am quite pleased to know that 'BTRA Scan' has completed 50 successful years; it is indeed a matter of joy. All this is because of everyone's hard work and co-operation. Without the co-operation of all the staff, scientists and editors seeing this day would have surely been impossible. This magazine covers research articles on recent innovations in the field of sustainable functional textiles, polymers, fibres and textile materials.

I take this opportunity to congratulate the editorial board for bringing out this magazine up to this level, which in itself is an achievement considering the effort and time required. I hope this magazine will continue its successful journey with a new form in future and will keep the readers updated in the field of textiles as it has been doing with each passing day.

**Mr. Sharad Saraf**  
*Chairman, BTRA Governing Council*

## DY. CHAIRMAN MESSAGE



Mr. Narendra Dalmia  
Dy. Chairman, BTRA Governing Council

Dear All,

Greetings...

It gives me great pleasure to know that 'BTRA Scan' completed 50 successful years of publication this year. True to its name, this magazine gives an insight into the range and scope of the innovation and creativity of our staff and research team. This magazine is very informative for the readers on recent innovations in the field of functional textile based on the demand of the time.

I applaud the editorial team for the hard work and dedication they have invested in realizing this goal, and wish all staffs, scientists and readers success in all future endeavours.

**Mr. Narendra Dalmia**  
*Deputy Chairman, BTRA Governing Council*

## EDITOR'S DESK

Dear Readers,

Greetings!!

I am delighted to present to our readers the - Edition of BTRA SCAN. The year of 2020 is momentous for humanity. During COVID 19 pandemic, lockdown and social distancing norms in these countries have taken a toll on socioeconomic activity and growth. Businesses continue to face major challenges as they try to adjust to the new normal. We are all learning to change the way we do things, as the office and the home become one space and restrictions on activities has made it difficult for us to print copies of BTRA Scan and get them delivered to you. But to ensure that our readers stay informed, we have decided to publish this edition as an E - Version for this Issue. I am sure readers will find this issue engaging and informative.

Last few months was most defining period for industries, but textile industries have played a great role to manufacture - mask, gloves, PPE kit, anti-viral textile products, packaging, etc to support our country during this pandemic. Now things are coming to normal, it may take another few month to see a big difference. As we are working unitedly to make the country Atmanirbhar, textile have a big role to play. Overall, within the textile industry, I see tremendous capability. Some more support from the government and the bureaucracy, lower cost of power and a good infrastructure will help us with a big leap forward as regards business.

I wish to assure you that we are on course for a solid recovery and growth. The future is undoubtedly positive for our industry. I feel the year 2021 will be a very good recovery year. Hope, we all are following the safety practices to defeat the pandemic completely from our life soon.

Stay safe, Work safe!

Thank You

**Dr. T V Sreekumar**

*Director, BTRA*

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# Copper Nanoparticles Synthesis and its coating for antimicrobial applications with improved durability



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## ABSTRACT

*The present work describes the synthesis of copper nanoparticles by chemical reduction method and its coating over pre-treated cotton fabric by Pad-Dry-Cure method. Plasma treatment followed by acrylic acid grafting was adopted as pre-treatment over cotton fabric to improve the adhesion between Copper nanoparticles (Cu-NP) over cotton fabric. The deposition of Cu-NPs was analyzed by SEM EDX as well as antibacterial activity. Developed Cu-NP coated cotton fabric showed excellent antibacterial activity as well as durability towards washing.*

## 1. INTRODUCTION:

Bacterial and viral infections are a major concern nowadays, which has been arising from adhesion, growth and proliferation of bacteria/virus on the surfaces. Significant research has been carried out to get novel antibacterial solution through effective use of nanotechnology and its applications [1,2]. The cheaper copper nanoparticles with high effectiveness and long durable properties could match the requirement of antibacterial materials for industrial production. Antibacterial activity of NPs is varied depending on the taxonomical location of microorganisms. For example, it was shown that Cu NPs have higher affinity to the amines and carboxyl groups on the surface of *Bacillus subtilis* than Ag NPs and therefore show higher antibacterial activity [2].

The most common process of conferring the copper nanoparticles to fabrics materials is utilizing some adhesion and cross-linked agents, such as acrylate and polyurethane, to form a composite coating on the substrate [1,3]. Consequently, only the exposed copper nanoparticles on the external surface can be released and show antibacterial property subsequently; meanwhile the majority of nanoparticles are blocked in the inner of composites by adhesion agents [1,4].

In this paper, method of impregnation of CuNP over plasma activated and acrylic acid grafted cotton fabric is proposed in order to get better antimicrobial activity as well as wash durability.

## 2. EXPERIMENTAL:

### 2.1 Material & Method:

Light weight, ready for dyeing 100% knitted cotton fabric was used for the coating of Copper Nanoparticles. Copper (II) Sulphate pentahydrate (*Merck India*, > 98%) and Ascorbic acid (*SDFCL India*) were used for the synthesis of CuNP. All the chemicals were of analytical grade and were used as-received. De-ionized (DI) water was used to conduct all the experimental procedures.

### 2.2 Preparation of Cu NP:

1.6% copper sulphate solution was prepared in distilled water and was heated at 80°C for 10 minutes. Later the 4% ascorbic acid solution was added in the same copper sulphate solution, drop wise with vigorous magnetic agitation. The colour of the copper sulphate solution slowly changes from light blue to light green initially and after 10-15 minutes turns to brown due to suspension of CuNP's.

The reaction condition was then maintained at 80°C for 5hrs. After the reaction, it was allowed to cool at room temperature. The resultant solution containing the nanoparticles was centrifuged for 10 min at 4000 rpm and resulting suspension was redispersed in distilled water. The centrifugation and redispersion process was

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repeated twice with water and finally with ethanol to remove any unreacted agent. Brown suspension of Cu-NPs was allowed to dry at room temperature. Finally, the dried nanoparticles of CuNP were used for further coating over Cotton substrate.

### 2.3 Fabric Treatment:

The knitted cotton fabric sample was pre-treated using dielectric barrier discharge- atmospheric pressure plasma treatment with helium + oxygen gas. The treatment was conducted on equipment PLATEX-600 (make GRINP S.R.L., Italy). Fabric sample of 50cm width was passed in continuous manner through plasma zone under 1.5 kW plasma power. Plasma treatment time and interelectrode spacing was maintained constant at 60sec and 1.0mm respectively for all experimental trials. This plasma treated sample was immersed in to acrylic acid solution (20% V/V). In order to remove the air trapped inside the flask, nitrogen gas was purged in to the solution. Reaction flask was then heated for 60 min at 65° C. Fabric was then removed, washed and dried in open atmosphere.

### 2.4 Cu NP coating over Cotton fabric:

Untreated cotton and pre-treated cotton fabric were dipped in solution containing CuNP (0.5%), and allowed to pass through padding process (2 dip-2 nip) with maintaining 75% pick up. Then samples were dried in 80°C for 15 min and cured at 110°C for 1 min. These CuNP coated fabrics were further analyzed for antibacterial activity, SEM EDX investigations and their durability towards washing.

**2.5 Antibacterial Properties:** An antimicrobial property of CuNP's coated cotton fabrics was studied by AATCC-100 procedure which gives a quantitative estimate of the antibacterial activities of the fabrics.

**2.6 Wash Durability:** The durability of CuNP coated fabrics towards washing was analyzed by gentle washing of samples as per AATCC LP2-2018 method with 5 and 10 numbers of washes.

**2.7 SEM & EDX investigation:** Scanning electron micrographs and Energy dispersive X-ray (EDX) scans were recorded using JEOL JSM IT 200 & Element make EDX, to confirm the surface morphology and elemental composition of the CuNP's coated fabrics

## 3.0 RESULTS & DISCUSSION:

Cu particles in nano scale have been shown to have antibacterial effect on the bacterial cell, functions in multiple ways, including adhesion to gram negative bacterial cell wall due to electrostatic interaction, having effect on protein structure in the cell membrane, denaturation of the intracellular proteins and interaction with phosphorus and sulphur containing compounds like DNA[5].

In our study, it was found that both (with and without plasma pre-treated) Cu NP coated fabrics showed excellent antibacterial activity towards Gram positive (*Klebsiella Pneumoniae*) and Gram negative bacteria (*Staphylococcus aureus*). In case of control (without pre treated) CuNP coated fabric, the antibacterial rate reached to 99.44% towards bacteria *Klebsiella Pneumoniae* and 99.96% towards bacteria *Staphylococcus aureus* after 24 hrs duration. Whereas for plasma pre-treated CuNP coated fabrics, it reached to 99.93% and 99.96% for *Klebsiella Pneumoniae* and *Staphylococcus aureus* respectively.

Wash durability of CuNP coated fabrics (with and without plasma treatment) was then assessed in terms of change in antibacterial activity with 5 and 10 number of washes. It was found that for control CuNP coated samples, complete loss of antibacterial activity due to poor adhesion between substrate and CuNP [Table-1]. For practical use, it is very important to have wash durability in antibacterial textiles. Hence to overcome this, fabric was activated with plasma and grafting treatment in order to improve the adhesion between fabric and antibacterial finish.

Table-1 : Poor adhesion between substrate and CuNP

Sample No.	Bacterial Reduction, (%)	
	<i>Klebsiella Pneumoniae</i> ATCC 4352	<i>Staphylococcus aureus</i> ATCC 6538
Cu-NP 0-Wash	99.44 %	99.96 %
Cu NP - 5 Wash	No reduction	No reduction

These pre-treated CuNP coated samples showed negligible change in antibacterial activity, even after 10 no. of washes [table-2]. This improvement in wash durability of CuNP coated fabric, after pre-treatment of functionalisation and grafting is due to physical interaction and formation of chemical bonding respectively. Interaction of plasma and grafting of acrylic acid leads to formation of more hydrophilic groups( such as C=O, COO) on the surface[6,7], resulting in improved functionality of the fabric, leads to improvement of adhesion of CuNP's over substrate.

To confirm the presence of CuNP's on the fibre surface, Scanning electron micrographs and energy dispersive X-ray (EDX) spectra were generated. Surface morphology of the CuNP coated cotton sample shows the uniform distribution of Cu nanoparticles over the substrate (Fig.1).

Whereas the EDX spectra (Fig.2) shows the signals for Cu along with oxygen and carbon. The appearance of carbon

Table-2 : Wash durability of Pre-treated Cu-NP coated fabrics

Sample No.	Bacterial Reduction, (%)	
	<i>Klebsiella Pneumoniae</i> ATCC 4352	<i>Staphylococcus aureus</i> ATCC 6538
Cu -Np 0-Wash	99.93 %	99.96 %
Cu Np – 5 Wash	99.87 %	99.93 %
Cu Np – 10 Wash	99.90 %	99.94 %

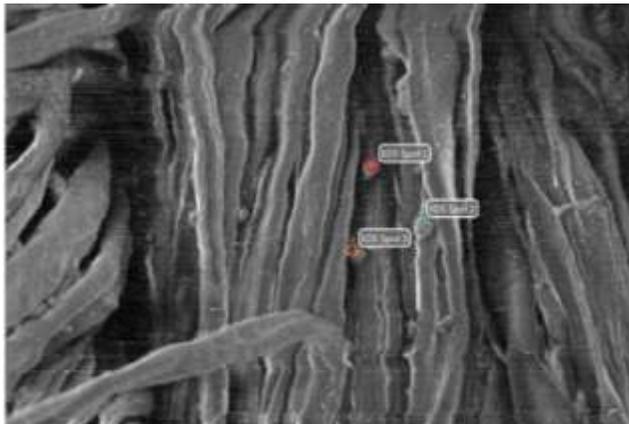


Fig. 1 Scanning electron micrograph (SEM) of CUNP coated sample

and oxygen peaks in the spectrum may attribute to the cotton fabric over which Cu-nanoparticles are coated. The elemental analysis of prepared CuNP coated sample is shown in Table-3.

Overall uniform distribution and improved adhesion of Table-3 Energy dispersive X-ray (EDX) of CUNP coated samples

Elemental analysis weight by weight percentage	
Carbon	70.9
Oxygen	26.3
Copper	2.8

CuNP's over pre-treated cotton fabric leads to excellent antibacterial activity and wash durability in cotton fabric rendering them suitable for commercial applications.

**4. CONCLUSION:**

Highly efficient antimicrobial cotton fabric was obtained by coating CuNP over pre-treated cotton fabric by Pad-Dry cure method. The sample was characterized by SEM, EDX spectra, which confirms the Cu deposition over cotton substrate. Developed Cu-NP coated cotton fabric showed excellent antibacterial activity as well as its durability towards washing as pre-treatment of atmospheric pressure plasma followed by acrylic acid grafting resulted in improved adhesion between CuNP over cotton fabric.

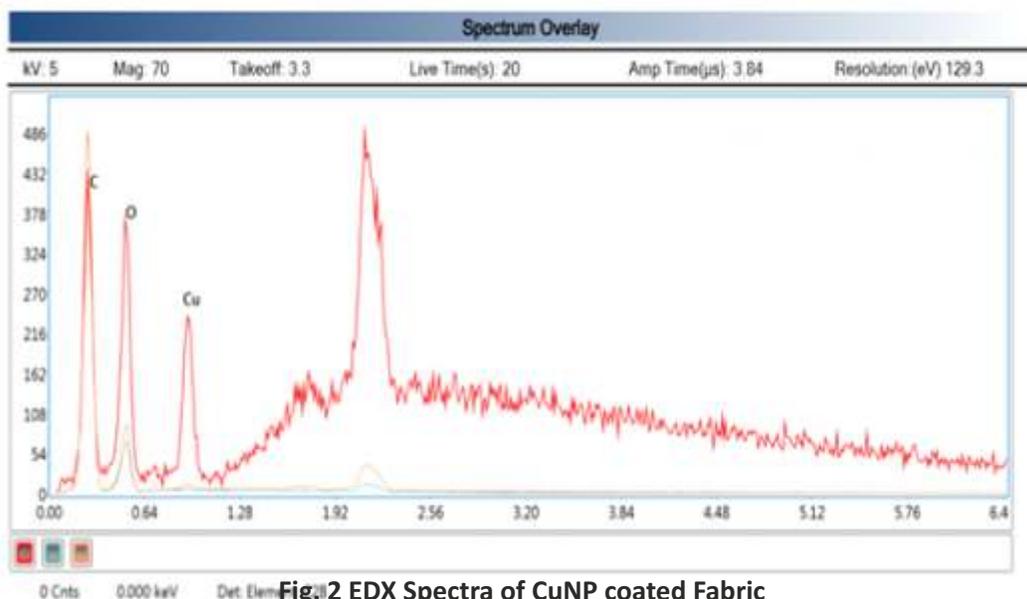


Fig.2 EDX Spectra of CuNP coated Fabric

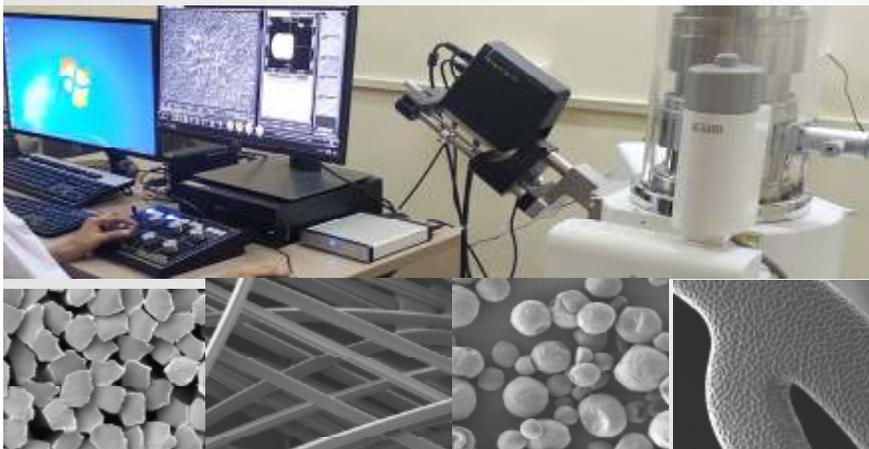
**References:**

1. Sun C., Li Y., Li Z., Su Q., Wang Y., and Liu X; 'Durable and Washable Antibacterial Copper Nanoparticles Bridged by Surface Grafting Polymer Brushes on Cotton and Polymeric Materials' Journal of Nanomaterials, Vol. 2013 [2018], 1-7.
2. Eremenka A.M., I.S.Petrk., Smimova N.P., Rudenka A.V., Marikvas Y.S; 'Antibacterial and Antimycotic Activity of Cotton Fabrics, Impregnated with Silver and Binary Silver/Copper Nanoparticles' Nanoscale Research Letters 11-28, (2016) 1-9
3. Tamayo L. A., Zapata P.A., Rabagliati F.M; 'Antibacterial and non-cytotoxic effect of nano composites based in polyethylene and copper nanoparticles,' Journal of Materials Science: Materials in Medicine, vol.26,no.3,article129,(2015).
4. El. Sadonny M.T., Abd El-Hack. M., Taha A.E., Fouda M.M.G., Ajarem J.S., Maooda S. N.Allam A.A., Elshaer N; 'Ecofriendly Synthesis and Insecticidal Application of Copper Nanoparticles against the Storage Pest Tribolium castaneum' Nanomaterials 10, 587, (2020) 1-16
5. Prabhu S, Poulouse E; 'Silver nanoparticles: mechanism of antimicrobial action, synthesis, medical applications, and toxicity effects'. International Nano Letters, 2, (2012) 32
6. Deogaonkar S.C., ' Dielectric barrier discharge plasma induced surface modification of polyester/cotton blend fabrics to improve polypyrrole coating adhesion and conductivity, The Journal of The Textile Institute,(2020), 1-8
7. Solhi L., Atai M., Nodehi A., Imani M., Ghaemi A., Khosravi K; 'Poly(acrylic acid) grafted montmorillonite as novel fillers for dental adhesives: Synthesis, characterization and properties of the adhesive' Dental Materials , 28 (2012) 369-377.
8. Vankar P.S. and Shukal D; 'Biosynthesis of Silver Nanoparticles by Lemon leaves extract and its application for Antimicrobial finish on fabric' Applied nanoscience 2 (2), 163-168.

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# ELECTROSPINNING OF POLYAMIDE 6 NANOFIBER USING WIRE ELECTRODES

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## ABSTRACT

*Electrospinning is one of the well accepted method to spin the fibers with diameter in nanometer range. In this spinning method, fiber spinning takes place between the electric field created by two electrodes. Various parameters such as polymer viscosity, potential difference, relative humidity and temperature of environment plays important role to decide fiber morphology and diameter. From the technology point of view, there are two types of instrument available i.e. needle type and needle less type. In the needle less method electrode may be cylinder or wire. Effect of parameters on needle type spinning method have been reported by many researchers. In this case, effect of different parameters on electrospinning of Nylon 6 using needle less electro spinning method with wire electrodes have been investigated. Fiber quality such as morphology and diameter have been characterized by scanning electron microscope (SEM). Pore size of the nanofiber mat has been analyzed by using porometer.*

## 1. INTRODUCTION

Fiber having diameter 1  $\mu\text{m}$  (1000 nm) or less, with aspect ratio more than 100:1 is known as nanofiber. Because of its extremely small features nanofibres have great potential to be used in various fields such as filtration, tissue engineering scaffolds, sensors, affinity membranes, catalyst & enzyme carriers, energy storage, release control and recovery of metal ions. A variety of processing techniques such as drawing [1], template synthesis [2,3], template melt extrusion or template assisted extrusion [4], phase separation [5], interfacial polymerization [6,7], self-assembly [8,9], melt blown [10], force spinning [11,12], and electrospinning have been used to prepare polymer nanofibers. All these methods have their own advantage, but electrospinning is a common and a fast-developing technique for the production of nanofibres due to its simplicity, efficiency and cost-effective setup. The industrial and scientific interest of electrospun nanofibre is due to its long length, small diameter and pore size [13-15]. During electrospinning, it is possible to control the fiber diameter and pore characteristics [26, 17]. The electrospinning setup consists of two electrodes of opposite polarity. One electrode is placed into the solution and another one on to a collector. Polymer nanofibres

were formed from the solution by electrostatic forces between two electrodes of opposite polarity [16-20]. The traditional electrospinning setup consists of a needle/nozzle in its jet outlet [21]. In recent years, electrospinning set up has improved much. Multiple needle electrospinning has been developed for enhancing the production rate. But the probability to encounter clogging and the electric field interferences generated by the numerous spinning heads are the disadvantages of multiple needle electrospinning system [22]. Needleless electrospinning system has overcome these problems. It has been found encouraging results for bulk scale production of nanowebs due to its multiple fiber forming jet formation of a suitable support (Rotating roller/ metal edge/ wire) from a polymer solution [23].

Process parameters such as polymer concentration, potential difference between electrodes, relative humidity, distance between the electrodes and chamber temperature plays important role during electrospinning. Effect of those parameters on fiber quality needle spinning method has been studied and reported by many researchers [24-26]. In the case of needleless technology, some parameters behave differently so this study is an attempt to study the effect of different parameters during the spinning of Nylon 6 using wire electrode.



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## 2.0 EXPERIMENTAL

### 2.1 Materials

Nylon 6 is selected for electrospinning, was purchased from the local market of Mumbai (India). Acetic acid (MW 60.05 g/Mol) and formic acid (MW 46.05 g/Mol) obtained from Merck life science PvtLtd., Mumbai (India), was used as it is without further purification. Polypropylene spunbonded non-woven fabric was procured from Techfab (India) Industries Ltd, Daman (U.T). and the Marcy land, soil was procured from soil lab, BTRA.

### 2.2 Methods

#### Spinning solution preparation

The measured amount of solvents like Acetic acid and formic acid 2:1, was taken in a conical flask. The polymer was added slowly with 500-700 RPM stirrer speed and set the temperature of the heating plate at 70°C. The solution was heated and stirred until the polymer dissolved.

#### Electrospinning

The nanofibrous mats were prepared using electrospinning machine from ELMARCO (NS IS500 U) needleless technology. Nylon 6 nanofibrous mats were deposited on the spunbonded polypropylene fabric, for preparing uniform fibers without beads, the parameters such as concentration of polymer, bottom electrode voltage, top electrode voltage, electrode distance and relative humidity was studied.

### 2.3 SEM analysis

Morphology of Nylon 6 nanofibrous mats was examined by Scanning Electron Microscope (SEM JEOL JSM 5400) a voltage of 15kV. All samples were sputter coated with gold prior to SEM analysis. Fiber diameter was measured by image software called Image J (NIH,

<http://rsbweb.nih.gov/ij/>), based on images obtain from SEM analysis. As many as 150 fibers were selected for each sample on different positions. Nanofiber diameter of each sample was estimated from statistics from those fibers.

#### Pore Size Analysis

Quantachrome's 3G porometer with standard test method ASTM D 6767, operating under windows ® the 3G win software was used for the analysis of pore size. This method is based on pore size characteristics of Geotextiles by capillary flow test. Atmospheric conditions were 21±2° C temperature and 60±5 % R.H.

## 3. RESULTS AND DISCUSSION

### 3.1 Effect of polymer concentration on Electrospinning

Effect of polymer concentration on spinnability and fiber diameter was studied by varying the concentration of Nylon 6 in the solution from 11-18 wt%. During the spinning, other parameters such as distance between electrodes, voltage of top electrode, voltage of bottom electrode, width of deposition, carriage speed, RH %, temperature and deposition time were kept fixed. Fiber quality and morphology was investigated by SEM and diameter was measured by image J software. The SEM images of fiber at different concentration and plot of the polymer concentration against diameter and pore are given in Figure 1 and Figure 2 respectively. In the large area, few beads were observed at 11% concentration whereas fibers were bead free from 12 to 18 wt% of polymer concentration. The diameter of the fibers found to be increased with an increase in polymer concentration. As pore size of the mat increases with increase in fiber diameter, for small pore size initial concentrations 11-14% was suitable but based on fiber morphology and uniformity of fiber diameter distribution, 13 wt% kept fixed for further set of experiments.

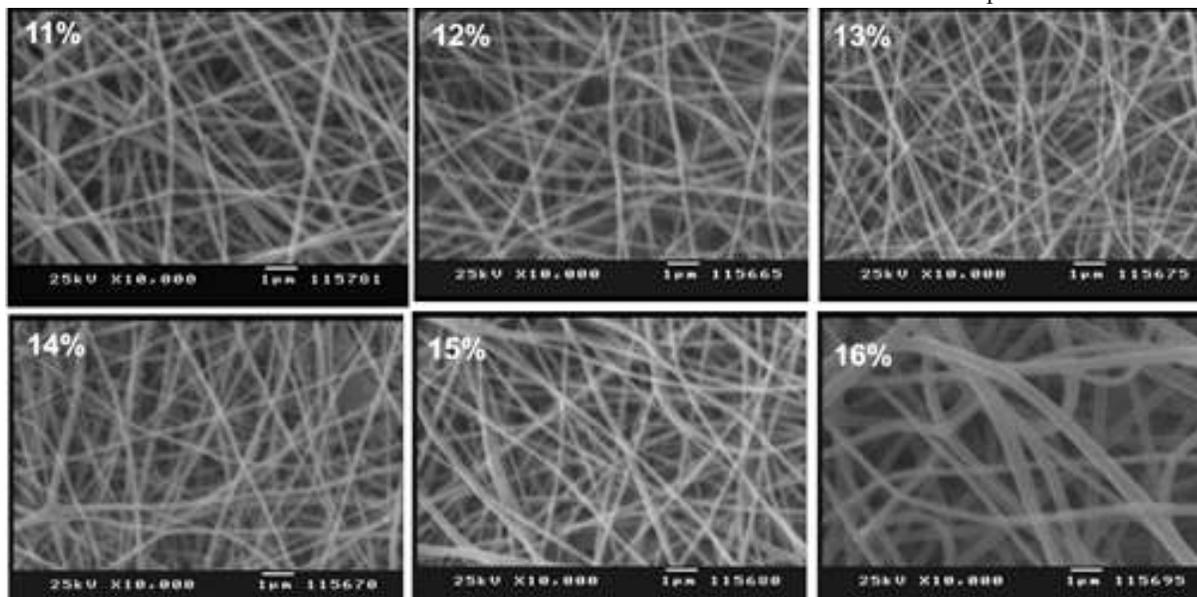


Figure 1: Scanning electron micrograph of nanofiber at different concentration of Nylon 6

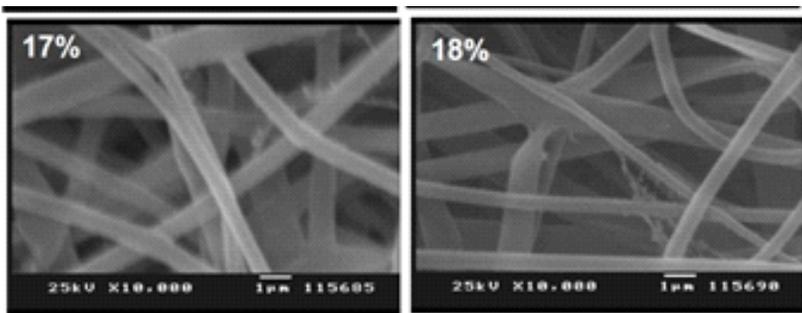


Figure 1: Scanning electron micrograph of nanofiber at different concentration of Nylon 6

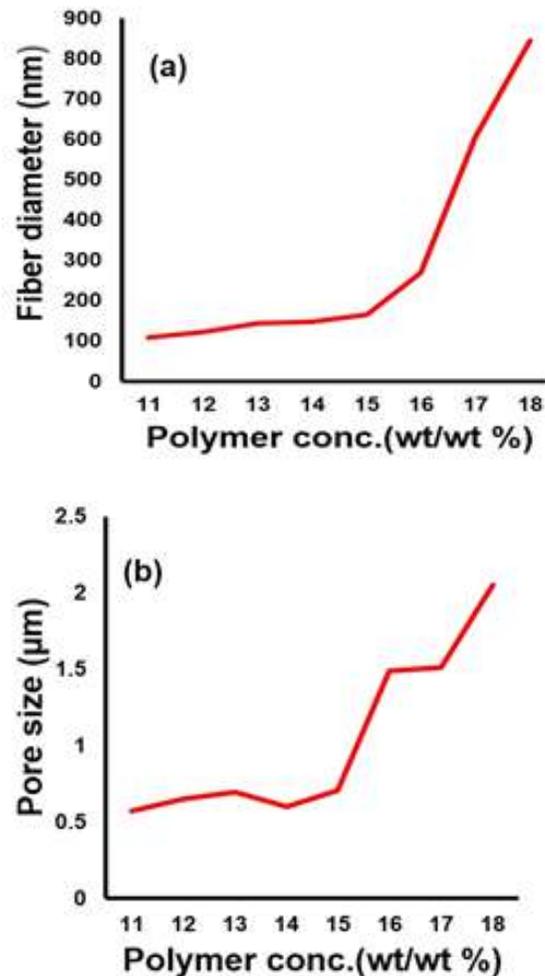


Figure 2: Effect of polymer concentration on (a) fiber diameter and (b) pore size of mat

### 3.2 Effect of positive voltage on fiber diameter and pore size

The effect of positive voltage on the diameter of the nanofiber and pore size of the deposited nanofiber mat was investigated by varying the voltages from 32.5 kV to 50 kV at interval of 2.5 kV. During spinning, other parameters such as concentration, -ve voltage, distance between electrode, relative humidity, temperature and deposition time was kept fix at 13wt %, -15 kV, 160 mm, 45%,  $21\pm 2^\circ\text{C}$  and 5 min respectively. Fiber morphology was investigated by SEM and the diameter of the fiber was measured using image J software. SEM images at

different voltages, plot of voltage verses fiber diameter and pore size are shown in Figure 3 and Figure 4 respectively. Morphology of the fibers found bead free at all the cases but decrease in diameter was observed up to 42.5 kV due to high stretching force on the spinning jet. Beyond the 42.5 kV again increase in fiber diameter was observed up to 50 kV due to reduction in flight time of spinning jet because of high potential difference. Though decrease in fiber diameter was found up to 42.5 kV but after 35 kV this decrease was not significant so 35 kV was considered as optimum and kept fix for further set of experiments.

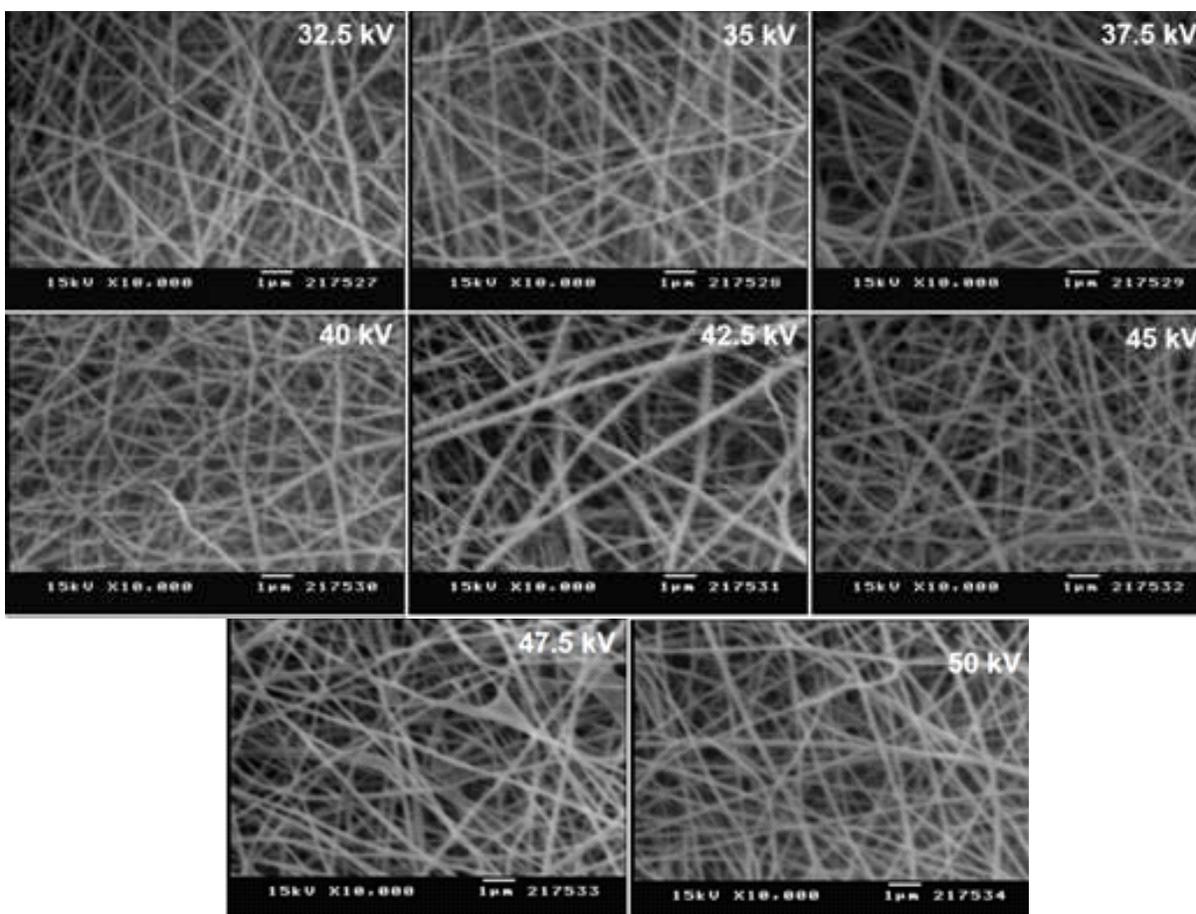


Figure 3: Scanning electron micrograph of nanofiber at different positive voltage

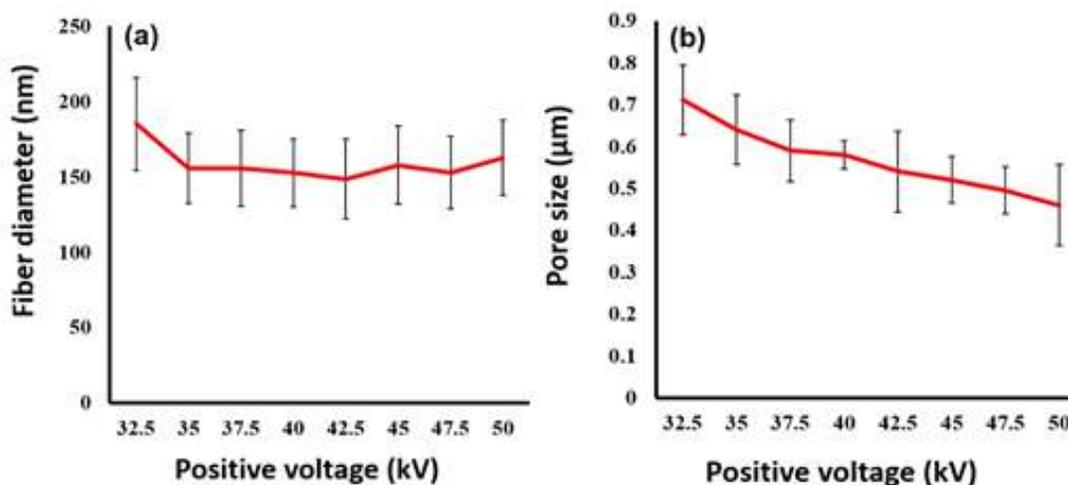


Figure 4: Effect of positive voltage on (a) fiber diameter and (b) pore size of mat

### 3.3 Effect of negative (collector) voltage on fiber diameter and pore size

Effect of negative voltage on spinnability, fiber quality and fiber diameter was investigated by changing the negative voltage from 0 to 15 kV at interval of 2.5 kV. During this study, other parameters such as polymer

concentration, positive voltage, distance between electrode, relative humidity, temperature and deposition time were kept fixed at 13 wt%, 35 kV, 160 mm, 45%,  $21 \pm 2^\circ\text{C}$  and 5 min respectively. SEM images at different voltages, plot of voltage versus fiber diameter and pore size are shown in Figure 5 and Figure 6 respectively.

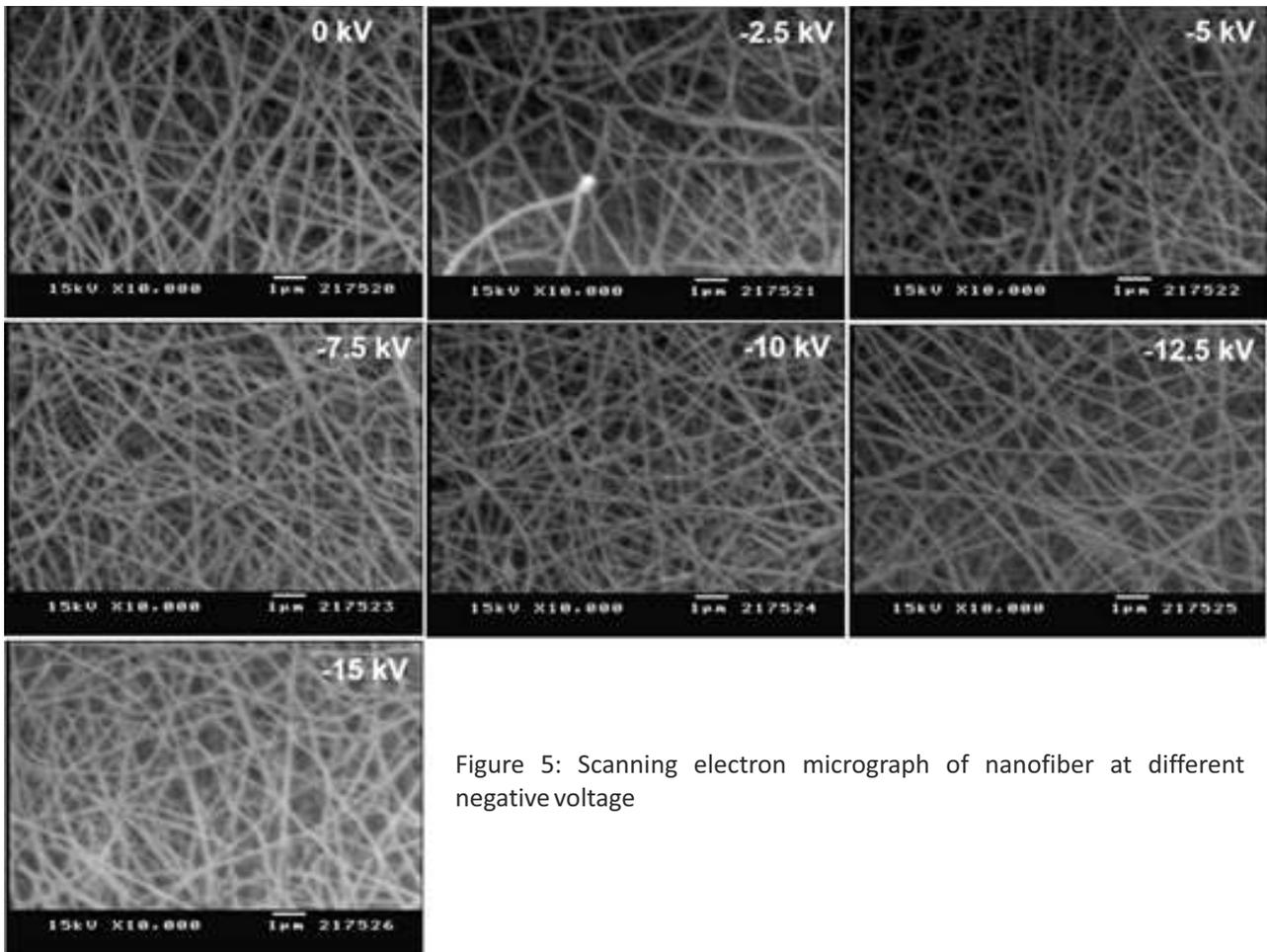


Figure 5: Scanning electron micrograph of nanofiber at different negative voltage

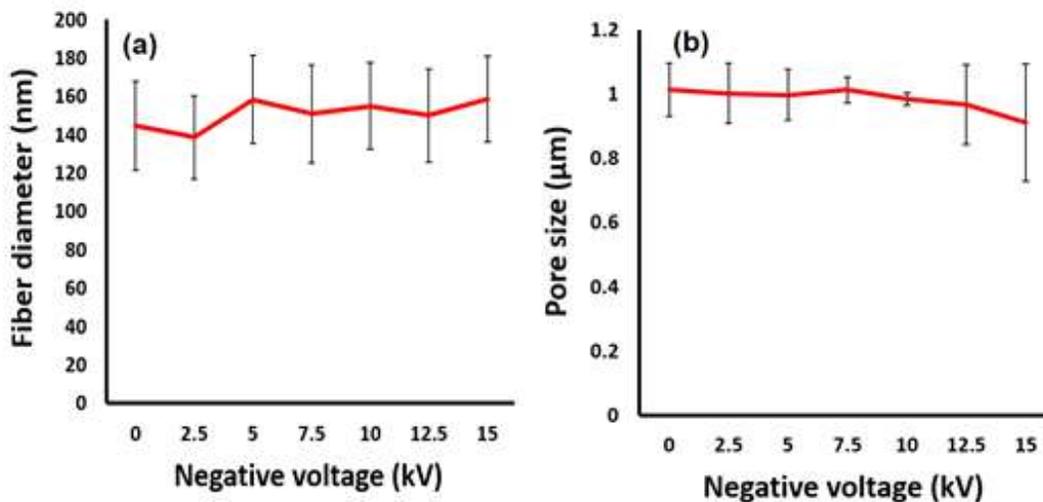


Figure 6: Effect of negative voltage on (a) fiber diameter and (b) pore size of mat

From the given data it can be inferred that there is no significant effect of negative voltage on fiber diameter, whereas slow decrease in pore size was observed with an increase in negative voltage after -7.5 kV. Higher the collector voltage beyond a threshold value lowers the

spread area so fiber density is high at that area. This increase in deposition density at higher collector voltage causing the reduction in pore size. To achieve the small pore size, -15 kV was kept fix for further set of experiments.

### 2.4 Effect of Distance between electrodes

Further the effect of distance between the two electrodes on fiber quality, diameter and pore size of the nanofiber mat during spinning was investigated by changing the distance from 130 to 190 mm at interval of 10 mm. During this study, other parameters such as concentration, bottom

electrode voltage, top electrode voltage, relative humidity percentage, temperature and deposition time was kept fix at 13wt %, +35 kV,-15 kV,45%,21±2°C and 5 min respectively. SEM images at different distance, plot of voltage verses fiber diameter and pore size are shown in Figure 7 and Figure 8 respectively.

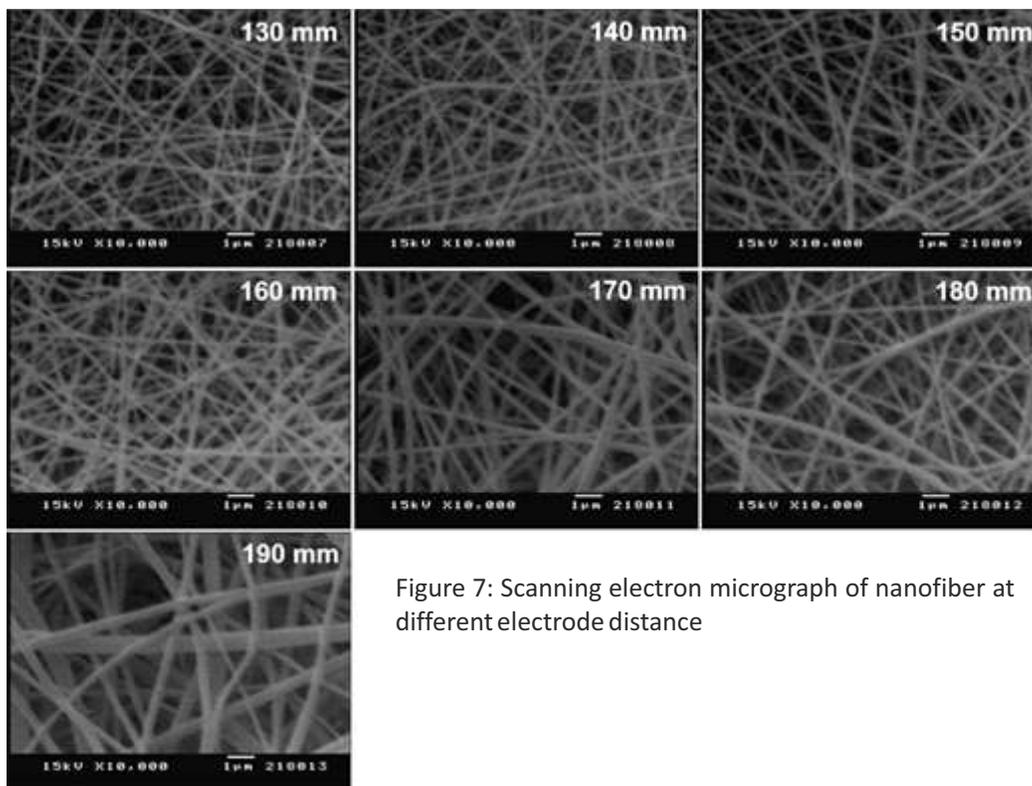


Figure 7: Scanning electron micrograph of nanofiber at different electrode distance

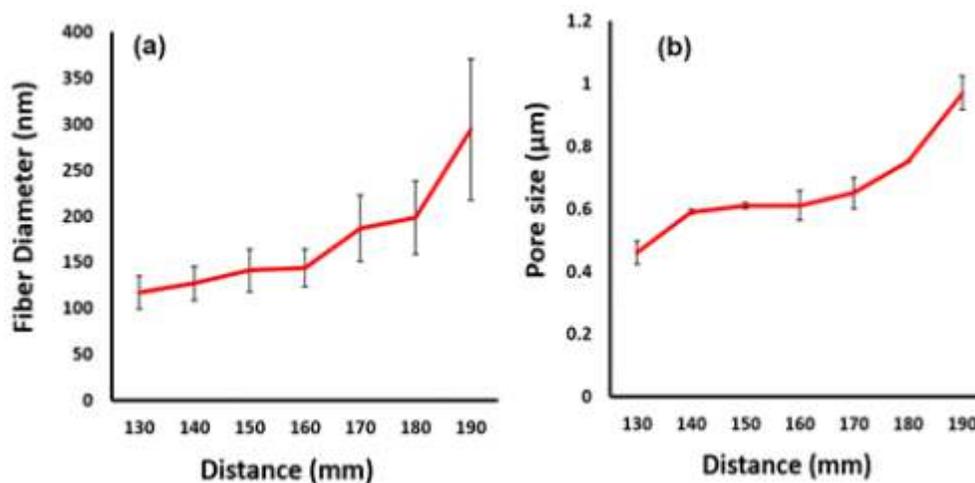


Figure 8: Effect of distance between electrode on (a) fiber diameter and (b) pore size of mat

When the voltage between electrode is fix, fiber diameter increases with an increase in distance between electrodes because strength of electric field reduces at higher distance which applies a low stretch on the solution. Increase in fiber diameter also leads to increase in pores size of the mat.

### 3.5 Effect of Relative Humidity

Effect of environmental relative humidity percentage on the spinning and fiber quality was investigated by changing relative humidity percentage from 45 to 70 % inside the spinning chamber at interval of 5%. During this study, other parameters such as polymer concentration,

positive electrode voltage, negative electrode voltage, distance between the electrodes, temperature and deposition time was kept fix at 13wt%, +35 kV, -15 kV, 130 mm, 21±2°C and 5 min respectively. SEM images at different RH%, plot of voltage versus fiber diameter and pore size are shown in Figure 9 and Figure 10 respectively.

Fiber quality and morphology was found similar in the above range of RH% because Nylon6 is well spinnable

within that range. At 70% RH small increase in diameter and significant increase in pore size was observed. The charge transfer between environment and solvent also helps for the evaporation of solvent some more the water vapor more is the charge diffusion. Thus, the fibersolidifies before further elongation and causes thicker fiber. Pore size is increasing with the lower web density and web density of nanofibres generally reduces with increase in fiber diameter.

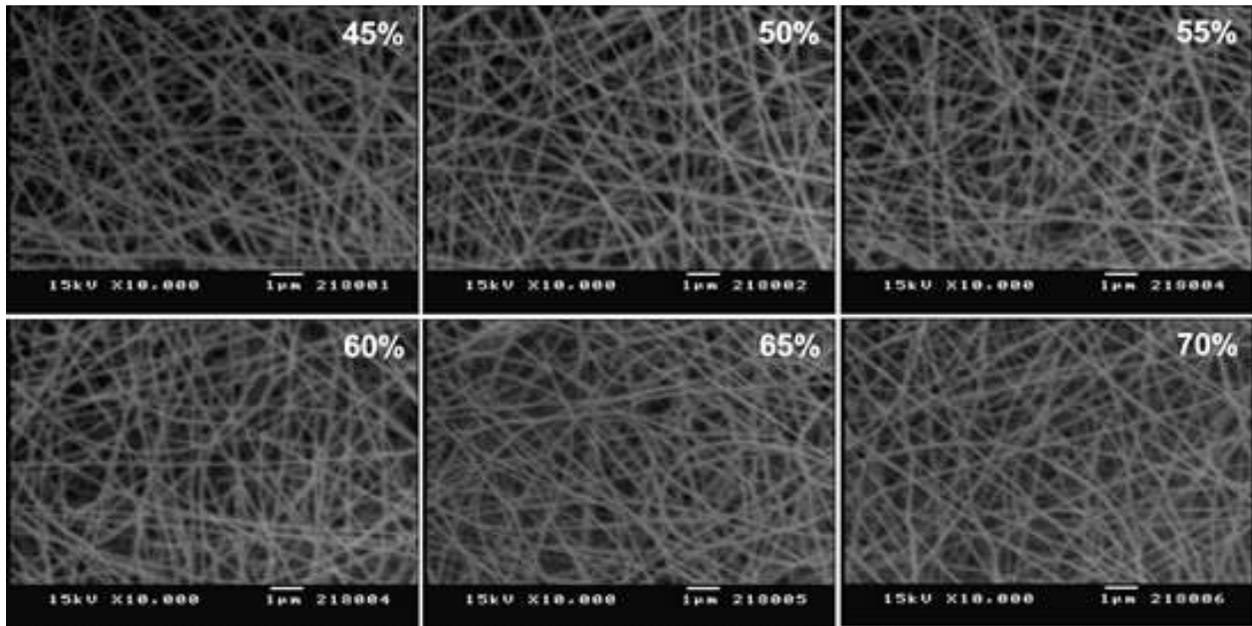


Figure 9: Scanning electron micrograph of nanofiber at different relative humidity percentage

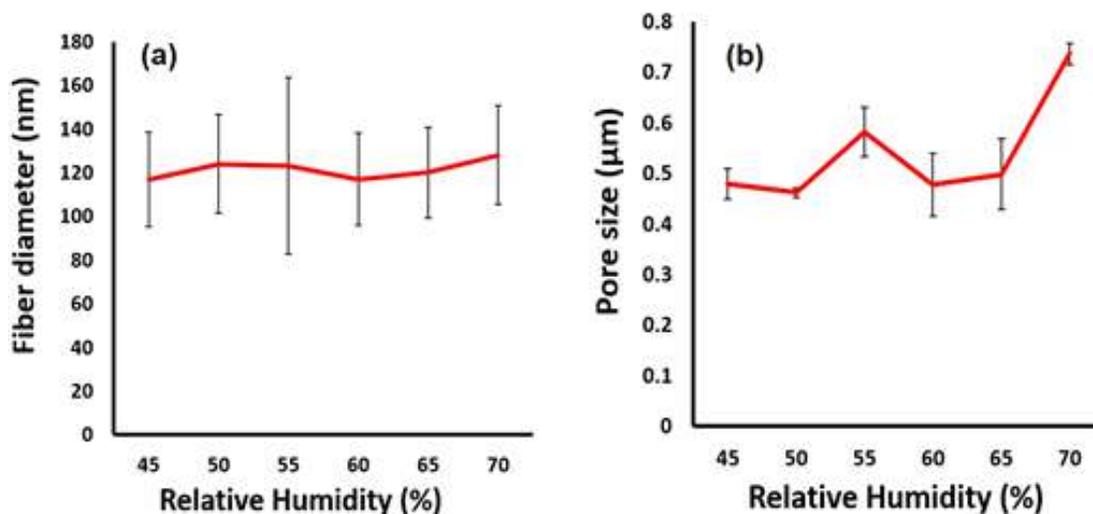


Figure 10: Effect of RH% on (a) fiber diameter and (b) pore size of mat

### 3.6 Effect of deposition time

The pore size of the nanofiber web depends on fineness of fiber and web density. To reduce the pore size up to minimum level, fiber diameter and web density need to be standardized. After standardization of related parameters to spin fine fibers, web density was standardized by

changing the deposition time from 0.5 to 5 min. At 5 min of deposition pore size was found 0.5  $\mu\text{m}$  compared to 48  $\mu\text{m}$  in existing microfiber media. Pore size values at different time is given in Table 6 and plot of pore size against deposition time is given in Figure 13. There were small changes in pore size after 2 min of deposition.

Table 6. Pore size at different deposition time

Time of deposition (min)	Pore size ( $\mu\text{m}$ )
0.5	9.96
1.0	1.63
2.0	0.70
3.0	0.63
4.0	0.61
5.0	0.50

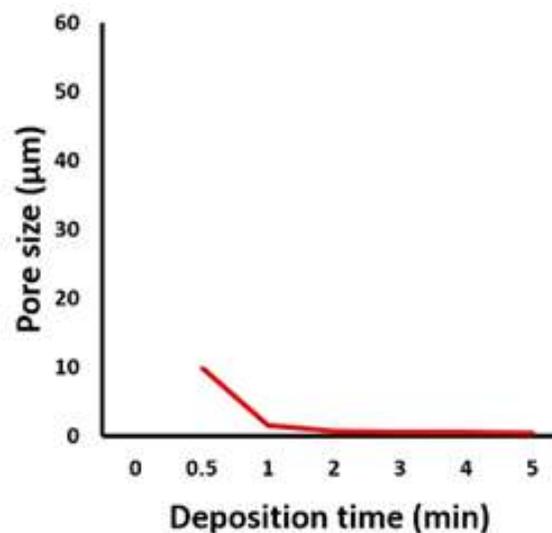


Figure 11. Effect of deposition time on pore size

## 4. CONCLUSION

Polyamide 6 solution in acetic acid and formic acid solvent system was successfully spun using electrospinning system with wire electrode. Increase in fiber diameter as well as pore size was observed with increase in polymer concentration from a certain percentage at which fiber morphology was found acceptable. At standardized concentration, increase in positive electrode voltage could not help to make the fiber finer after a threshold value due to the increase in force and reduction in time for molecular chain relaxation. There was no significant effect of negative electrode voltage on fiber diameter but decrease in pore size was observed at higher voltage due to less spreading of fibers. Increasing trend in fiber diameter was found by increasing the spinning distance because of reduction in charge

density per unit volume. Polyamide 6 was found spinnable within a range of relative humidity. As usually reduction in pore size value was observed with increase in deposition time. For the used polymer at 13% concentration, positive voltage, negative voltage, distance between the electrodes and relative humidity was 35kV, -15kV, 130mm and 45to70% respectively for getting finer fiber and pore size.

**References-**

1. H. Y. Liu, L. Xu, and Q. L. Sun, Highly Aligned Electrospun Nanofibres by Hot-drawing, *Thermal Science*, Vol. 19, No. 4, p 1357-1360, 2015
2. L. Costa, M. Al-Hashimi, M. Heeney, A. Terekhov, Template-Synthesis of Conjugated Poly(3-Hexylselenophene) (P3HS) Nanofibers Using Femtosecond Laser Machined Fused Silica Templates, *electronic devices & materials*, Vol 2(51), p 2957-2960, 2017
3. Y. Song, J. Wei, J. Liu, Template Synthesis of Hollow Carbon Nanofibers, *Microsc. Microanal.* 21 (Suppl 3), p 989-990, 2015
4. Huayi Li, Yucaike and Youliang Hu, Polymer nanofibers prepared by template melt extrusion, *Journal of Applied Polymer Science*, volume 99, issue 3, PP 1018 – 1023, 2006.
5. Mohammad Raoufi, Neda Aslankoochi, Christine Mollenhauer, Heike Boehm, Joachim P. Spatzab and Dorothea Bruggemann, Template-assisted extrusion of biopolymer nanofibers under physiological conditions, *Integrative Biology*, 8, PP 1059-1066, 2016.
6. M. Deka, A. K. Nath, A. Kumar, Ionic conduction and phase separation studies in PEO-P(VdF-HFP)-LiClO<sub>4</sub>-dedoped polyaniline nanofiber composite polymer electrolytes — I, *Indian J of Physics*, volume 184, issue 10, PP 1299-1305, 2010
7. Jie Li, Jing Fang, Mu Cui, Hai Lu, Zhi-an Zhang, Yan-qing Lai, Electrochemical performance of interfacially polymerized polyaniline nanofibres as electrode materials for non-aqueous redox supercapacitors, *Journal of central south university of technology*, volume 18, issue 1, PP 78–82, 2011
8. A Huczko, M Osica, A Rutkowska, M Bystrzejewski, H Lange and S Cudziło, A self-assembly SHS approach to form silicon carbide nanofibres, *Journal of physics: Condensed matter*, volume 19, number 39, 2007
9. Marco Rolandi, Ranieri Rolandi, Self-assembled chitin nanofibers and applications, *Advances in Colloid and Interface Science*, volume 207, PP 216-222, 2014
10. Nitilaksha Hiremath, and Gajanan Bhat, Melt blown Polymeric Nanofibers for Medical Applications- An Overview, *Nanoscience and Technology*, volume 2, issue 1, PP 1-9, 2015
11. Bharath Raghavan, Haidy Soto, Karen Lozano, Fabrication of Melt Spun Polypropylene Nanofibers by Forcespinning, *Journal of Engineered Fibers and Fabrics*, Volume 8, Issue 1, PP 52-60, 2013
12. Horacio Vasquez, Horacio Gutierrez, Karen Lozano, Gerardo Leal, Titanium Dioxide Nanofibers through Forcespinning, *Journal of Engineered Fibers and Fabrics*, Volume 10, Issue 2, PP 129-136, 2015
13. Rajkishore Nayak, Rajiv Padhye, Illias Louis Kyratzis, Yen Bach Truong and Lyndon Arnold, Recent advances in nanofibre fabrication techniques, *Textile Research Journal*, volume 82, issue 2, PP 129–147, 2011
14. C.J. Thompson, G.G. Chase, A.L. Yarin, D.H. Reneker, Effects of parameters on nanofiber diameter determined from electrospinning model, *Polymer*, volume 48, PP 6913-6922, 2007
15. A Berteau, L R Manea, A Popa and A Berteau, The Influence of Process Parameters on the Characteristics of Electrospun 3D Nanostructures, *IOP Conf. Series: Materials Science and Engineering*, volume 209, article ID 012074, PP 1-6, 2017
16. Hale Karakas, Electrospinning of Nanofibres and their Applications, *MDT'Electrospinning*, PP 1-35
17. Liu, Y.; He, J.H.; Yu, J.Y. & Zeng, H.M.: Controlling numbers and sizes of beads in electrospun nanofibers, *Polymer International*, volume 57, issue 4, pp. 632-636, 2008
18. Bhardwaj, N. & Kundu, S.C. Electrospinning: A fascinating fiber fabrication technique, *Biotechnology Advances*, volume 28, PP 325-347, 2010
19. Atefe Rezaei, Ali Nasirpour, and Milad Fathi, Application of Cellulosic Nanofibers in Food science Using Electrospinning and Its Potential Risk, *Comprehensive Reviews in Food Science and Food Safety*, Vol.14, PP 269-284, 2015

20. Anton Formhals, Richard Schreiber Gastell, Process and apparatus for preparing artificial threads. US Patent Nr. 1,975,504, 1934
21. Guocheng Zhu, L Y Zhao, L T Zhu, X Y Deng and W L Chen, Effect of Experimental Parameters on Nanofiber Diameter from Electrospinning with Wire Electrodes, IOP Conf. Series: Materials Science and Engineering, volume 230, Article ID 012043, PP 1-12, 2017
22. Claudio Migliaresi, Giuseppe Alberto Ruffo, Fabio Zomer Volpato and Dario Zeni, Electrospinning for Advanced Biomedical Applications and Therapies, Advanced Electrospinning Setups and Special Fibre and Mesh Morphologies, PP 23-68
23. Hosne Ara Begum, Md. Khalilur Rahman Khan, Study on the Various Types of Needle Based and Needleless Electrospinning System for Nanofiber Production, International Journal of Textile Science, volume 6, issue 4, PP 110-117, 2017
24. Amir Houshang Hekmati, Abosaeed Rashidi, Reza Ghazisaeidi, Jean-Yves Drean, Effect of needle length, electrospinning distance, and solution concentration on morphological properties of polyamide-6 electrospun nanowebs, TRJ, 83(14), 2013.
25. Sheng Xie, Yongchun Zeng, Effects of Electric Field on Multineedle Electrospinning: Experiment and Simulation Study, Industrial & Engineering Chemistry Research. 2012, 51, 14, 5336–5345
26. Ying Yang, Zhidong Jia, Qiang Li and Zhicheng Guan, "Experimental investigation of the governing parameters in the electrospinning of polyethylene oxide solution," in IEEE Transactions on Dielectrics and Electrical Insulation, vol. 13, no. 3, pp. 580-585, June 2006.

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# INDIAN CARBON FIBRE HISTORY AND PRESENT CHALLENGES

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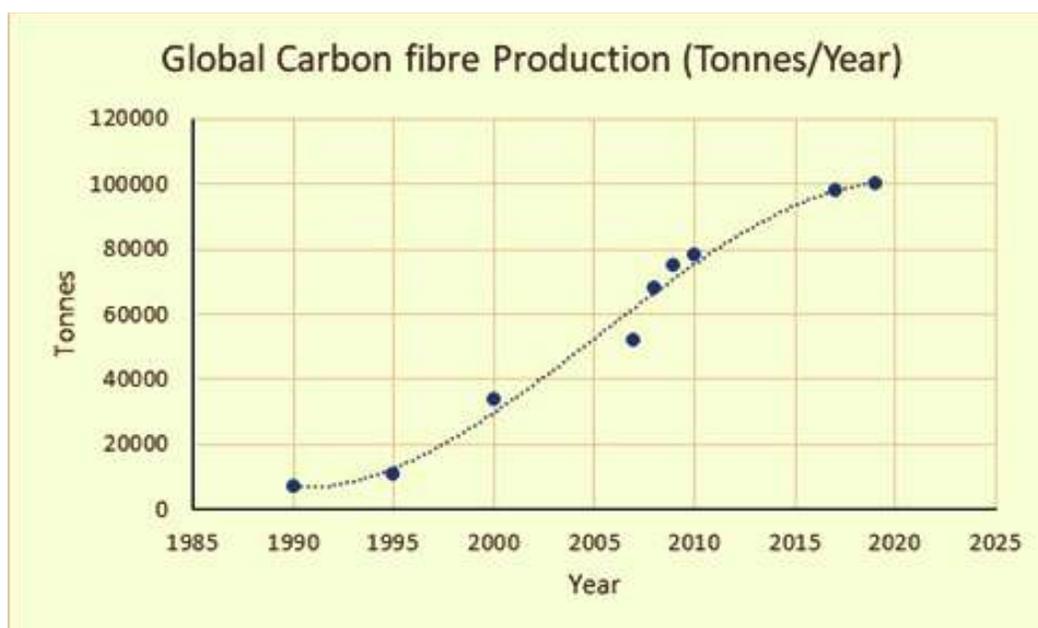


## Introduction

Carbon fiber is a unique material among high-performance fibers which is used for aerospace to industrial applications. It consists of majorly carbon (>95%) with some amounts of other elements such as oxygen, nitrogen, etc. The importance of carbon fiber is derived from its high tensile properties such as strength, modulus, and low elongation to stress. If specific strength is compared, this amazing material is fifty times stronger than steel. The specific strength of steel is ~60 kN.m/kg and that of carbon fiber is about 3000 kN.m/kg. When carbon fiber is used in aircraft, 20% of its weight can be reduced compared to aircraft with aluminum which is very important for better fuel efficiency and higher payload. 40% of the structural weight of aircrafts are

made of carbon fiber composites. Apart from high-end aerospace applications, carbon fiber finds its place in industrial and sports goods. It is used in bridge repair, sports cars, tennis rackets, lightweight bicycles, etc.

Dramatic growth in worldwide production and consumption of carbon fibre is being witnessed due to the large demand for carbon fibre in the fields of sports and leisure sector, automobile industries, civil and marine engineering apart from its conventional application in the aerospace industry. The following figure gives an idea of the growth in carbon fibre production worldwide in the last thirty years [1,2]. There has been significant growth in industrial carbon fibre production especially large tows carbon fibres such as 24k and 48k.

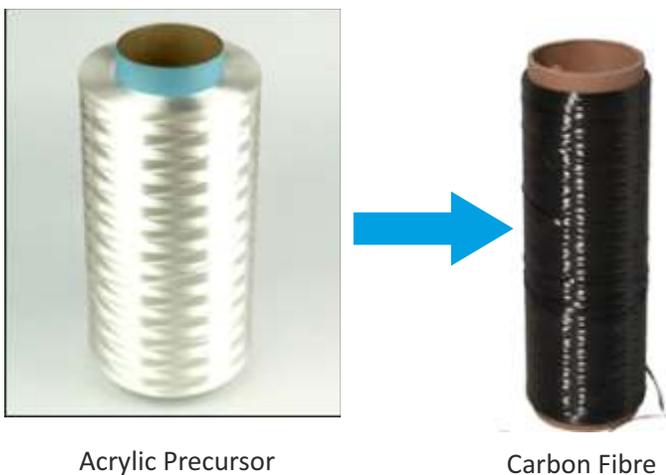


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## History of Carbon Fibre

### Worldwide

In 1860, Joseph Swan and in 1879 Thomas Edison produced carbon fibre and found application as the filament in a light bulb. Cellulosic fibres were charred to produce carbon fibre those days. Edison used cotton fibres and heated at very high temperatures in an inert atmosphere to remove all elements except carbon. In 1958, Roger Bacon from Union Carbide produced the high-performance carbon fibres using viscose rayon. These continuous carbon fibres were made by heat-treating rayon at high temperatures in an inert atmosphere until all other elements except carbon were eliminated. In the early 1960s Japanese scientist Akio Shindo developed Polyacrylonitrile (PAN) based carbon fibres which lead to today's high tenacity acrylic precursors and high strength carbon fibres such as T-300 and other carbon fibre variants. During the 1960s United Kingdom-based Courttelle, Japanese Toray, and American company Union Carbide were the major players in carbon fibre production. After year 1970, several manufacturers started production of carbon fibre across the world including Mitsubishi (PAN and pitch-based), Toho Tenex, Zoltek, Cytec, Aksa, etc. Recently China also is a major producer of carbon fibre. Today 95% of the world carbon fibre is being manufactured from acrylic precursors. The remaining 5% is being produced from viscose rayon, pitch, etc.



Acrylic Precursor

Carbon Fibre

### India's encounter with Carbon fibre

In India, Indian Petrochemical Corporation Ltd (IPCL) was producing carbon fibre since 1989 under the trade name Indocraft. It had a production capacity of 12 metric tonnes. The plant was closed down in 1998 due to non-availability of precursor fibres. Also, 12 MT is not economically viable. IPCL was importing the PAN precursor from Courttelle, Mitsubishi and others. Today, an economically viable production capacity is 1500 Tonnes per annum.

Though, India had nearly ten years of experience in small

scale carbon fibre production at IPCL, India is yet to commercially produce good quality acrylic precursors suitable for T-300 and above carbon fibres. Carbon fibre and precursor technology are not available as the material is extensively being used in defence applications. Carbon fibre producers do not disclose how their product is manufactured. The United States require carbon fibre manufacturers to secure an export license for each carbon fibre transaction with foreign interest. Most of the machinery and precursor technology falls under the export control rules where such critical technologies are regulated and restricted.

### Carbon fibre research in India

#### Academic research

India's carbon fibre research started as early as 1980 at National Physical Laboratory, (NPL), Delhi. These researches were primarily led by three scientists, O. P. Bahl, L. M. Manocha and R. B. Mathur [3,4] Their work majorly focused on the characterization of commercially available precursors, their chemical modification, the effect of stabilization processes and carbon fibre properties. In the same period of time, researchers at Indian Institute of Technology, Delhi (IIT) published work on the synthesis of Polyacrylonitrile, their copolymers and fibres [5]. Some basic research on acrylic precursors also happened in Central Leather Research Institute (CLRI), Madras [6]. After the carbon fibre plant at IPCL was commissioned, there was an urgent need of acrylic precursor. The plant was offered precursor for a limited time (five years) due to restrictions. India was supposed to create the precursor facility by that time. The Government of India identified several institutes for precursor research and development. IPCL had an acrylic fibre plant those days which was producing textile grade acrylic fibre. Textile grade acrylic fibres are low strength having comonomers suitable for dye sites. Precursor grade acrylic fibre has to be high tenacity and devoid of bulky groups used for attracting cationic dyes. Precursors should have monomers with carboxylic acid groups to facilitate thermo-oxidative stabilization (Initiation of nitrile cyclization) in an air atmosphere [7]. Given the above IPCL R&D also initiated precursor research [8].

In the meantime, in 1993, a major turning point in precursor research happened when a joint project funding was sanctioned to IIT Delhi by Department of science and technology (DST) and Aeronautic Research and development board (ARDB), Government of India. The project amount was about one crore rupees. The project was headed by Prof. Pushpa Bajaj, Prof. A K Gupta and Prof. Kushal Sen [9] at the Department of Textile Technology, IIT Delhi under the close monitoring of Dr A P J Abdul Kalam, then scientific advisor to defence minister.

### Mission mode development at IIT Delhi (1993-1998)

The major focus of the DST/ARDB sponsored project was the development of high tenacity acrylic fibres as a precursor for carbon fibre. Table 1 shows the properties of commercial precursors. Precursor with similar properties may provide carbon fibres with quality equivalent to T300.

oxidative stabilization during PAN to carbon fibre processes. Several methods of polymerizations were attempted including solution polymerization [13], emulsion polymerization [9] and solvent water suspension polymerization [14]. In these papers, they have reported the synthesis of PAN with Methyl acrylate (MA), Methacrylic acid (MAA) and Itaconic acid (IA)

Table 1- Properties of PAN Precursor Fibres.

Property	Commercial Precursor I	Commercial Precursor II
Probable Polymer composition	AN/MA (98:2)	AN/MA/MAA (96:3:1)
Intrinsic Viscosity (dl/g in DMF) at 25°C	1.64	1.6
Filament diameter ( $\mu\text{m}$ )	12	11.4
Denier/ Filament	1.21	1.1
Density ( $\text{g}/\text{cm}^3$ )	1.184	1.182
Tensile strength (GPa)	0.68	0.66
Initial Modulus (GPa)	12.2	8.25
Breaking elongation (%)	11	16
Extent of order (%)	59	47
Molecular orientation (%)	86	83
Exothermic initiation Temp ( $^{\circ}\text{C}$ )	205	200
Exothermic Heat ( $\Delta\text{H}$ ) (J/g)	2602	2315

The precursor produced by any carbon fibre manufacturer differs from those of its competitors, and the processing details that give each brand its signature characteristics and are considered to be their intellectual property. Carbon fiber manufacturing process is difficult to copy and any professional consultation is very expensive. The availability of experienced manpower in this field also is very rare. Only very few manufacturers offer precursor production line and there are only a few organizations which provide oxidation oven and carbonization furnace. The whole manufacturing facility or manufacturing line is capital intensive. It can be in the tune of 700 crores for a 1500 tonnes per annum plant. It can take up to two years for complete installation and commissioning of the line to make it operational. The team at IIT Delhi headed by Professor Bajaj published several research papers on the production of precursor and carbon fibre [8, 10, 11, 12]. It is evident from their publications that they made significant achievements in the synthesis of high molecular weight co- and terpolymers of polyacrylonitrile (PAN). The comonomers used are vinyl acids and esters mentioned in Table 1. The vinyl comonomers facilitate the easy drawing of filaments during spinning and thermo-

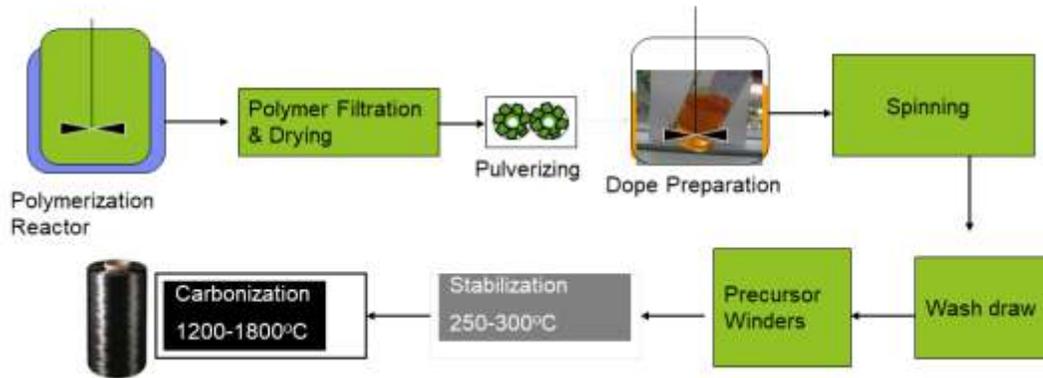
comonomers. Intrinsic viscosities in the range of 1.2 to 3dl/g were reported depending on the polymerization method with molecular weight distribution (Mw/Mn) as low as 2.5. A fibre spinning (wet and dry-jet-wet) machine was installed at IIT Delhi with coagulation, washing, drawing and winding systems. High tenacity acrylic precursors of strength as high as 0.65 GPa were reported by IIT Delhi team in the year 1998 [11]. The institute also reported some efforts on stabilization and carbonization of PAN precursor to produce carbon fibre.

The project was discontinued from IIT Delhi in the year 1998 and further research was initiated at National Aerospace Laboratory, Bangalore.

After concluding the project at IIT Delhi, a new project was initiated at NAL. With the focus on indigenizing the carbon fiber development from polyacrylonitrile based precursor fiber, CSIR-NAL established an integrated facility for carbon fiber and prepregs in 2003 with the leadership of Shri M K Sridhar. NAL claims to have developed a process for making carbon fibre equivalent to T-300 or above and certified by CEMILAC for standard modulus grade carbon fiber production. CSIR-NAL is

Process - Acrylic Precursor & Carbon Fibre

Production Route: Polymerization, Wet Spinning, Stabilization, Carbonization



Development of Carbon Fibre at National Aerospace Laboratories (NAL), Bangalore

now offering this standard modulus grade carbon fiber technology, certified by Centre for Military Airworthiness and Certification, to any Indian organization. The followings are the complete process and properties of carbon fibre as per the NAL website [15].

- a. Acrylonitrile polymerization (CSTR mode)
- b. Carbon fiber precursor by wet spinning
- c. Heat treatment of precursor fibers to carbon fibers

Standard modulus grade carbon fiber properties:

Filament Count	Tensile Strength [GPa]	Tensile Modulus [GPa]	Filament diameter [ $\mu\text{m}$ ]	Density [g/cc]	Carbon [%]
6K	3.5±0.1	240	7.0	1.78	>94

The technology was transferred to Kemrock Industries and Exports Ltd, Vadodara. Kemrock created a 300 TPA facility in 2011 at their composite manufacturing centre which included systems for suspension polymerization, thermo-oxidative stabilization and carbon fibre processing. Kemrock, however, could not commercially produce and sell carbon fibre of required properties in the domestic or international market. Kemrock, later on, stopped all their operations including composite manufacturing. In the year 2017-18, Kemrock was taken over by Reliance Industries Ltd and renamed as Reliance Composite Solutions. Reliance started manufacturing glass fibre composites in 2018 and reports are that they are working on operationalizing the carbon fibre production facility.

NAL continued to progress in improving the properties of their carbon fibre and in a recent expression of interest in January 2019, NAL intent to operate their 3TPA plant in a continues process through GOCO model.

Carbon Fibre Research at Indian Space Research Organization

Carbon fibre is one of the essential materials in space technology. Large-scale rocket parts of more than eight meters in diameter are made of carbon fiber skins with an aluminium honeycomb core. For reentry, Orion multipurpose crew vehicle uses a 5-meter diameter carbon fiber heat shield that is manufactured as a

sandwich structure featuring carbon fiber skins and a titanium honeycomb core. Similar ablative material was used for the Apollo missions also [16]. Indian space research organization (ISRO) also uses carbon fibre in several rockets and satellite parts. To fulfil the internal requirement of ISRO, a team of scientists lead by Dr C P R Nair worked on the development of acrylic precursor. Several reports were published in international journals in this regard [17]. The main focus was on the synthesis of various copolymers, dope rheology, the effect of temperature etc.

Economics

The quality of carbon fibre produced depends on the quality of precursor fibre. Quality of precursor depends on the quality of the polymer. So, it is important to have the right kind of polymer and precursor to produce good quality cost-competitive carbon fibre. The quality of polymer and precursor properties influence the process temperature and speed of production. Both these parameters will decide the final price of carbon fibre. The

scale of production is also an important deciding factor on the selling price of carbon fibre. 1500 TPA is the typical scale for commercial production of carbon fibre. Carbon fibre production is a very energy-intensive process and an increase in nitrile cyclization temperature, pre-carbonization and carbonization can affect the carbon conversion, process speed and resulting carbon fibre properties. Thus, before establishing a carbon fibre business, it is important to understand the commercial scalability of the technology. The cost of production of carbon fibre is important when a material has to compete with imported and time-tested products from Toray, Mitsubishi, Zoltec etc. It is important to understand what is the conversion of carbon fibre or how much carbon fibre can be produced from 1 kg of PAN precursor. The conversion is primarily related to the composition, molecular weight, molecular weight distribution, fibre denier, strength, processing temperature and treatment time, etc (Table 1).

If a production capacity of 1500 TPA carbon fibre of quality equivalent to T-300 and a net profit of 10% and 20% are considered, following calculation shows that the simple payback period is about 14 years.

<b>Commercial Production Capacity - 1500 TPA</b>	
Cost of T-300	- \$ 22/ kg
Net Profit margin @ 10%	- \$ 2.2/kg
Total Profit/ year	- 1500 x 1000 x 2.2 - \$33,00,000
In Rs-	~ 25 crores
If net profit margin @ 20%	- \$4.4/ kg
Total Profit/ year	- 1500 x 1000 x 4.4 - \$66,00,000
In Rs-	~ 50 crore
Capital Investment	- 700 crores
Simple Pay Back	- 14 years

Simple payback of fourteen years is huge. The industry usually considers three to five years a viable payback period. To make it more attractive to business, the profit margin has to be increased by reducing production cost.

Carbon fibre production is an energy-intensive process. 40-50% of the cost of carbon fibre is due to the precursor. The precursor cost is about \$3-4/kg. Minimum of two kilograms of the precursor is required for preparing one kilogram of carbon fibre. The remaining cost is for thermal stabilization, carbonization, surface treatment etc. Thermo-oxidative stabilization is done at 200-250°C, pre-carbonization at 600-800°C (LT furnace) and carbonization at 1200-1800°C (HT furnace). A massive portion of the cost of production is associated with the energy required for the process mentioned above.

According to an analysis, only China electricity rate/ unit supports sustainable carbon fibre production. Approximate cost of electricity in China, United States and India are Rs-3, 6 and 8/kWh. To link with the global benchmark, subsidized electricity is required. It is also important to have uninterrupted power supply during the heat treatment process. Any power failure will reduce the processing temperature, stabilization and carbonization process will not be completed leading to reduced mechanical properties.

The main raw material for making acrylic precursor is Acrylonitrile (ACN). It is important to ensure a constant supply of raw materials with consistent quality to produce superior precursor fibres. Reliance Industries Ltd (RIL) was the only company producing ACN in India. The production was stopped for the last several years. It should be revived for a steady supply of quality raw material. Similarly, Dimethyl Formamide is one of the main solvents for PAN. Only limited companies are producing them in India.

### Need of Government Support and Conclusion

Indian industry is new to high-performance fibre production. Great effort and support from the government are required to gain industry confidence to initiate production of such materials in the country. The main reasons for the reluctance of the industry to initiate the production of high-performance fibers are:

1. Non-availability of reliable inhouse technology, international technology provider due to export control restrictions and product guarantee.
2. High energy cost makes the business less viable. The government may consider special incentives and special energy pricing for carbon fibre industry.
3. The government should ensure uninterrupted, high-quality power supply.
4. Buyback of carbon fibre produced- The government should come forward to buy all the carbon fibre produced as the domestic requirement is only 300TPA.
5. Support CF composite industry for full capacity utilization- The minimum economically viable production is 1500 TPA and the domestic demand being 300 TPA, it is a must to enhance our carbon fibre composite industry. Special incentives should be considered for both CF and composite industry for some period of time till the business becomes competitive and self-sustainable.
6. Permission for exporting carbon fibre after satisfying internal demand. If any industry produces more than 300 TPA (present demand), they should be permitted to export excess production.

*Disclaimer: The views and opinions expressed in this article are those of the authors and do not necessarily reflect the official policy or position of BTRA or of the government. Examples of analysis performed within this article are only examples. They should not be utilized in real-world analytic products as they are based on limited and open source information.*

#### References:

1. Business News on Carbon Fibre, JEC Composite Magazine, 41, 18, 2008.
2. Vicki Mcconnell, Making of carbon fibre, Composite world, 31/03/2020
3. S.S.Chari, O.P.Bahl, R.B.Mathur, Fibre Science and Technology 15, 2, 153-160, 1981.
4. O. P. Bahl, R. B. Mathur, T. L. Dhani, Polymer engineering and science, 24, 7, 455-459, 1984
5. A. K. Gupta, R. P. Singhal, P. Bajaj, V. K. Agarwal, Journal of applied polymer science, Volume28, Issue3, March 1983, Pages 1167-117
6. Nandi Joseph, A. K. Murthy, K. T. Joseph, M. Santappa, Journal of Macromolecular Science: Part A - Chemistry: Pure and Applied Chemistry, A18(2), 153-158, 1982
7. P Bajaj, TV Sreekumar, K Sen. Polymer 42 (4), 1707-1718, 2001
8. J. V. Prasad, U. S. Satpathy, M. Jassal, A. Pantar, S. Satish, International Journal of Polymeric Materials and Polymeric Biomaterials, 18, Issue 1-2, 105-115, 1992
9. A. K. Gupta, D. K. Paliwal, Pushpa Bajaj, Journal of Macromolecular Science, Part C, Polymer Reviews, 31, 1, 1-89, 1991.
10. P. Bajaj, D. K. Paliwal, A. K. Gupta, Journal of applied polymer science, 49, 5, 823-833, 1993
11. P. Bajaj, A. K. Roopanwal, Journal of Macromolecular Science, Part C, Polymer Reviews, 37, 1, 97-157, 1997.
12. P. Bajaj, T.V. Sreekumar and K.Sen, Chem. Fibre. Intern. 48 (4), P 308-315, 1998.
13. P. Bajaj, K. Sen, S. Hajir Bahrami, Journal of applied polymer science 59, 10 1996, 1539-1550
14. P. Bajaj, TV Sreekumar, K Sen. Journal of applied polymer science 79 (9), 1640-1652, 2001.
15. <https://www.nal.res.in/en/technology/standard-modulus-grade-carbon-fiber-technology>
16. Scott Francis, Composite world, 31/03/2020
17. Renjith Devasia, C. P. Reghunadhan Nair & K. N. Ninan, Journal of Macromolecular Science, Part A, Pure and Applied Chemistry 39, 7, 693-708, 2002.

## ETP PROBLEMS ! DON'T WORRY,

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**Contact: The Bombay Textile Research Association**

Email : [tsd@btraindia.com](mailto:tsd@btraindia.com) Phone : +91 22 62023636 Ext-214

# TEXTILE PROCESS AUDIT: AN APPROACH TOWARDS CONTINUAL IMPROVEMENT

**Vijay Shirole\***

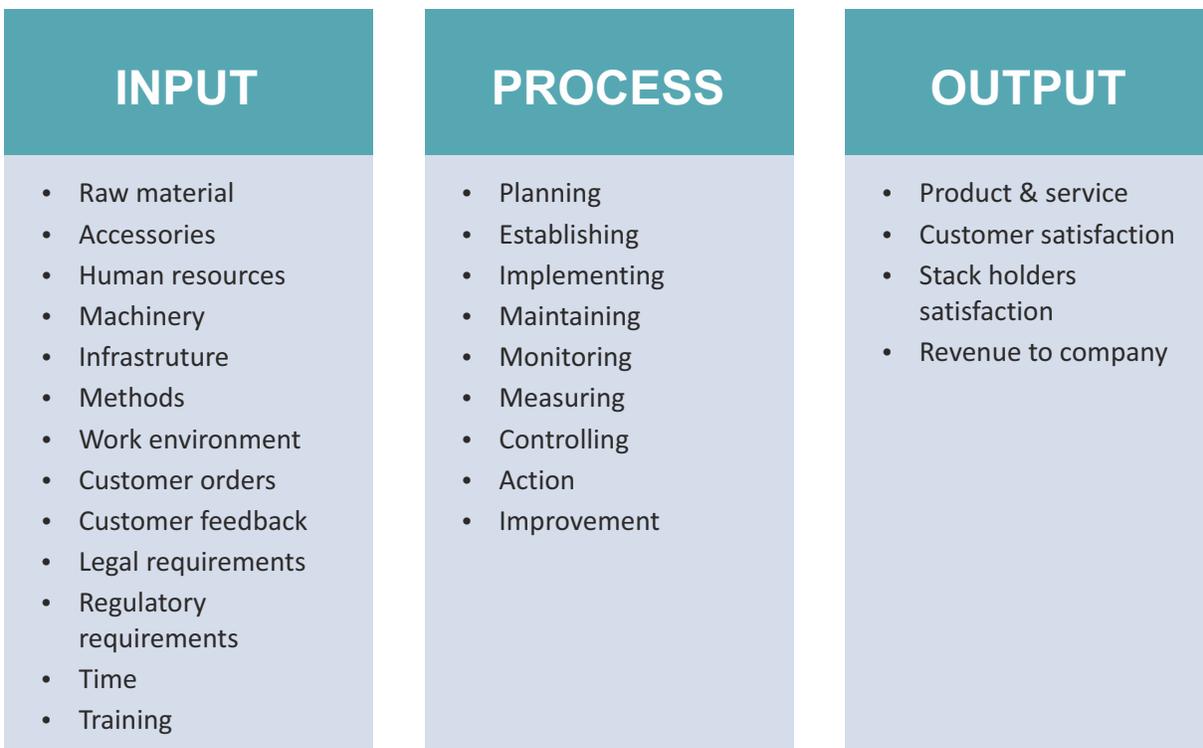
Technical Services Dept., The Bombay Textile Research Association,  
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## WHAT IS A PROCESS?

In short, we can say “it is a series of steps that lead to a desired result.” In other way we can say a process audit is an evaluation of the sequential steps and interactions of a process within a system. For doing any process, we have

to give some inputs and when we give any input we look for the desired output after the process. The following things are taken into consideration during this flow.



## PROCESS AUDIT:

A process audit may check conformance to defined requirements such as time, speed, accuracy, temperature, pressure, composition, component mixture, responsiveness. It may involve special processes such as

heat setting, pretreatment, dyeing, finishing etc.

A process audit examines the resources (equipment, materials and people) used to transform the inputs into outputs, the environment, the methods (procedures and instructions) followed and the measures collected to determine process performance.

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E-mail : [tsd@btraindia.com](mailto:tsd@btraindia.com)

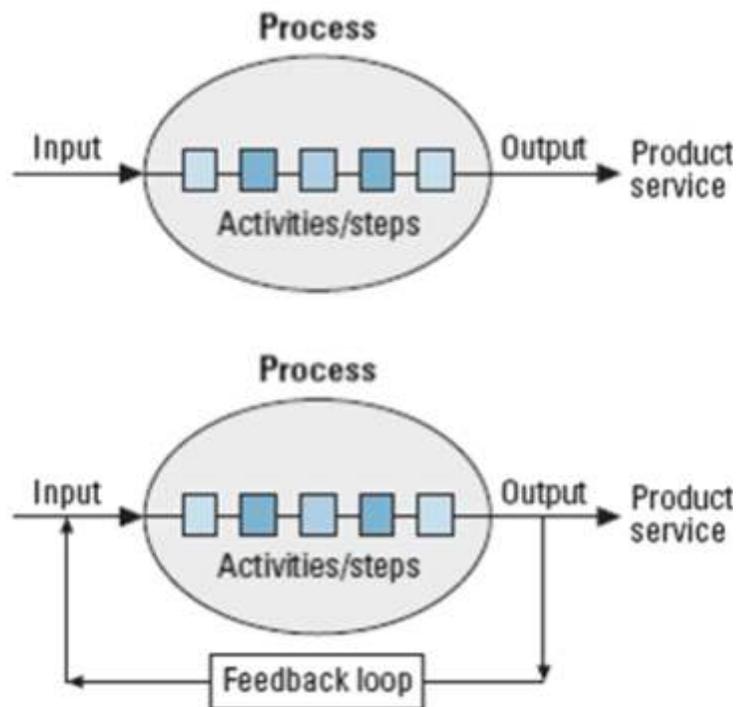
A process audit checks the adequacy and effectiveness of the process controls established by procedures, work instructions, flowcharts, training and process specifications.

By its very nature, process auditing implies an action, such as transforming inputs into outputs. Process auditing is evaluating the steps and activities that create the action

or transform the inputs into outputs. This is a very useful approach because it focuses on the work cycle and deliverables instead of isolated requirements/controls.

The process model in Figure 1 shows inputs, outputs and sequential steps. Some process models also show a feedback loop that is essential for control of a process.

**FIGURE 1** Process Diagrams With and Without a Feedback Loop



There are 2 types of audit methodology

1. Auditing by element
2. Auditing by Process

As we are mainly concerned in the methodology of auditing by process here we describe the methodology in brief,

**AUDITING BY PROCESS**

Auditing a process or system using process techniques verifies conformance to the required sequential steps from input to output. Process auditors use models and tools such as simple flowcharts, process maps or process flow diagrams.

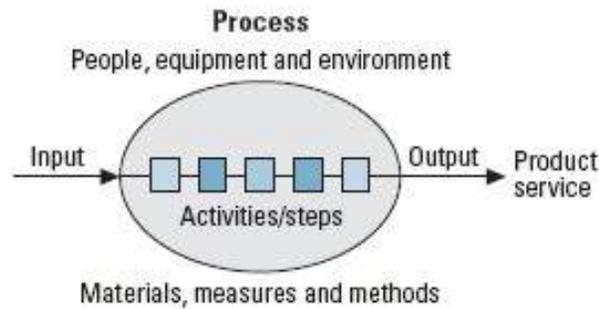
In the process diagram (see Figure 1), the boxes in the center could represent a flowchart of the sequential steps.

Flowcharts typically identify inputs, people, activities or steps, measures and outputs. The auditor normally gets this information from a procedure or flow charts provided by the audited organization.

Processes can be described using the process elements so called PEEMMM (see Figure 2):

- People involved.
- Equipment needed.
- Environmental requirements.
- Measures to test or monitor.
- Methods to follow.
- Materials used or consumed.

**FIGURE 2** Process Elements



**Process: underwriting**

**People:** Underwriter, account manager and customer.

**Equipment:** Computer and software.

**Environment:** Office.

**Materials:** Financial, security, logistical information provided by customer and account manager.

**Measures:** Financial and security risk.

**Methods:** Organizational procedures and industry standards.

**CONTROL POINTS AND CHECK POINTS UNDER THE SCOPE OF AUDIT:**

The control points and check points are under the scope of the audit,

CONTROL POINTS	CHECK POINTS
Process parameters	<ul style="list-style-type: none"> <li>Level of adherence to process parameters.</li> <li>Calibration of equipment’s monitoring the process.</li> <li>Suitability of process parameter decided to get the results.</li> </ul>
Selection of raw materials	<ul style="list-style-type: none"> <li>Quality of Raw materials received.</li> <li>Handling and storage systems.</li> </ul>
Selection and training of employees	<ul style="list-style-type: none"> <li>Competency levels of men available and men employed.</li> <li>Process performance.</li> <li>Work practices.</li> <li>Housekeeping practices.</li> </ul>
Maintenance of machines	<ul style="list-style-type: none"> <li>Adherence to maintenance schedules.</li> <li>Suitability of maintenance schedules and plans for the production and quality expectation.</li> <li>Condition of machine parts.</li> <li>Maintenance practices.</li> <li>Results of maintenance.</li> </ul>
Rejection Rates	<ul style="list-style-type: none"> <li>Whether acceptance criteria are clear to all on the shop floor?</li> <li>Whether process parameters are as per standards specified?</li> <li>Rejection Machine wise, Shift wise, operator wise and material wise.</li> </ul>
Delivery schedule	<ul style="list-style-type: none"> <li>Whether production started in time?</li> <li>Utilisation of machines.</li> <li>Productivity of each machine.</li> <li>Whether quality approved?</li> </ul>

**CHECKPOINTS DURING PROCESS AUDIT**

The following things are checked during process audit

1. Machine details and its condition
2. Product design and its process flow
3. Production process documentation – standard operating procedure (SOP) and work instruction
4. The process flow or sequence of individual machine.
5. Process control parameters & Specification limits of process control parameters
6. Calibration of measuring equipment's and calibration criteria and its frequency.
7. Daily Process control round data by QC or management representative
8. Deviation if found necessary corrective action to take
9. Action plan for implementation for corrective / preventive action

**AREAS UNDER THE SCOPE OF PROCESS AUDIT**

The following areas are under the scope of Process audit

1. General Information (No of staff, Production facility, Location)
2. Production capacity.
3. Production management systems ( production Planning & executions )
4. Quality – In house testing facility
5. Purchase - Procurement
6. Store - Incoming material
7. Manufacturing quality
8. Process control
9. Occupational Health and Safety (OSH)
10. Production - Non confirming material

11. Monitoring and measurement
12. Maintenance
13. Environment
14. Training

**STEPS IN PROCESS AUDIT:**

1. Notification by letter, by email to the factory.
2. Plan the audit
3. Initial General meeting with factory senior management persons to give information about the purpose of meeting.
4. To fill up questionnaire needed to do the process audit.
5. Document check - under the scope of audit.
6. The factory round to know the factory with respect to Housekeeping and cleaning, Safety, Material handling, Maintenance, Machinery conditions, Process control, Environment, Calibration, etc.
7. Suggestion for the improvement during factory round.
8. Discussion on the problems faced by the factory during fabric processing and their remedies to overcome the problem.
9. Closing meeting with factory management persons.
10. Preparation of draft audit report with audit findings and conclusion.
11. Feedback from the factory.

The Bombay Textile Research Association (BTRA) is conducting such process audits in the textile factories for their continual improvement all over India. The factories which want to avail this service can furthermore contact with Technical service Department of BTRA, Mumbai. The email id for communication are [tsd@btraIndia.com](mailto:tsd@btraIndia.com) and [info@btraIndia.com](mailto:info@btraIndia.com).

**Reference:**

1. Processing audit techniques – by JP Russell & Associates – 2002-2009.

## ABSTARCT

**Effect of heat treatment on the microstructural properties of silica embedded cobalt ferrite nanocomposites :**

*Meenakshi Bansal , Dharamvir Singh Ahlawat , Amrik Singh , Vijay Kumar & Shish Pal Rathee*

*Nanocomposite , published 14 Dec 2020*

<https://doi.org/10.1080/20550324.2020.1865711>

Silica coated cobalt ferrite (CoFe<sub>2</sub>O<sub>4</sub>:SiO<sub>2</sub>) nanocomposites were synthesized by coprecipitation technique using metal nitrates as precursors. The as-prepared samples were heat treated at different temperatures of 250, 500 and 750 C for 24 h. Structural, thermal, and morphological behavior of nanocomposites are investigated by XRD, FTIR, TGA-DTG, and SEM characterization results, useful in biomedical applications. With increasing calcinations temperature from 250 to 500 C and 750 C an increase in crystallite size of CoFe<sub>2</sub>O<sub>4</sub>:SiO<sub>2</sub> nanocomposites has been determined from 20.26 to 28.95 nm and 38.76 nm by Williamson–Hall method, respectively. Furthermore, by increasing the temperature from 250 to 750 C the lattice parameter and strain values have been found to increase from 8.0321 to 8.0691 Å and 1.01 10<sup>2</sup> to 3.75 10<sup>3</sup> , respectively. Analysis of TGA results found no weight loss when the sample was heated beyond 700 C and thus complete decomposition of precursors has led to the formation of stable nanocomposite structures at high temperatures. SEM analysis of synthesized samples at 750 C revealed well developed nanoparticles of CoFe<sub>2</sub>O<sub>4</sub>: SiO<sub>2</sub> with inter-granular porosity.

**Nano engineered electro spun fibers and their biomedical applications: a review**

*Xi Zhang , Xuetao Shi , Julien E. Gautrot & Ton Peijs*

*Nanocomposites: published on 29 Dec 2020*

<https://doi.org/10.1080/20550324.2020.1857121>

Electrospun fibers have received significant interests for various application areas such as filtration, composites and biomedical products due to their large surface area, good continuity, high porosity and many other unique properties. In bio-related applications, electrospun fibers have been used for in-situ drug delivery, tissue engineering scaffolds and wound dressing. In more recent years, there has been a drive toward novel electrospun fibers with added functionalities. Nanoengineering of electrospun fibers has introduced many of such novel properties. Through this review, researchers are provided with a state of the art overview of nanoenhanced electrospun fibers with added functionalities. Examples of some nanoengineered fibers include; surface functionalization, multi-component fibers, porous nanofibers, the creation of surface nano-topographies, and the incorporation of nanoparticles to create hierarchical fibrous structures for tailoring of physicochemical properties with a special focus on biomedical applications.

**Structure and properties of thermomechanically processed silk peptide and nanoclay filled chitosan**

*Pei Chen , Fengwei Xie , Fengzai Tang & Tony McNally*

*nanocomposites 2020, vol. 6, no. 3, 125–136*

<https://doi.org/10.1080/20550324.2020.1820796>

While chitosan has great potential for biomedical and wider application due to its appealing characteristics such as biocompatibility and inherent antimicrobial activity, its properties usually need to be further tailored for specific uses. In this study, the effect of inclusion of silk peptide (SP) and nanoclays (montmorillonite, MMT and sepiolite, SPT) on the properties of thermomechanically processed chitosan were examined. Blending SP with chitosan led to a material with greater elasticity and surface wettability. For the chitosan matrix, addition of either MMT or SPT resulted in increased mechanical properties with MMT being more effective, likely due to its 2D layered structure. For the chitosan/SP matrix, while inclusion of MMT caused increased mechanical properties and thermal stability, SPT was more effective than MMT at reducing surface hydrophilicity and SPT fully counteracted the increased surface hydrophilicity caused by SP. Thus, this work shows the different effects of MMT and SPT on chitosan-based materials and provides insights into achieving balanced properties.

**Structural and dielectric characterization of (PVP/PEO)/Al<sub>2</sub>O<sub>3</sub> nanocomposites for biodegradable nanodielectric applications**

Priyanka Dhatarwal & R. J. Sengwa

[Advanced Composites and Hybrid Materials volume 3, pages 344–353 \(2020\)](#)

Polymer host matrix and inorganic nanofiller comprising hybrid nanocomposites of integrated promising properties are the most appealing multifunctional materials for developing advanced flexible-type electrical and electronic devices and also a variety of daily life quality products. This research reports the structural parameters and dielectric properties of biodegradable poly(vinyl pyrrolidone)/poly(ethylene oxide) (PVP/PEO) blend matrix and alumina (Al<sub>2</sub>O<sub>3</sub>) nanoparticle-based polymer nanocomposite (PNC) films. Porous spherulitic morphology of the host PVP/PEO matrix, polymer-polymer interactions, and the structures of PEO crystallites are found largely influenced by the Al<sub>2</sub>O<sub>3</sub> nanofiller, whereas the degree of crystallinity of the PNC films increases slightly. The dielectric permittivity of the PNC films over 20 Hz to 1 MHz frequency range confirmed some increase in dielectric polarization, but they exhibited anomalous behavior when the Al<sub>2</sub>O<sub>3</sub> content increases in gradual steps from 1 to 5 wt%. In these hybrid materials, the interfacial polarization and dielectric relaxation predominantly contributed in the dielectric permittivity and electric modulus dispersion at the lower frequencies. The detailed analysis of dielectric properties authenticates that these nanocomposites could be used as prominent biodegradable nanodielectrics for polymer nanotechnology-based eco-friendly future-generation numerous types of flexible devices/products.

**Potential Application of Green Composites for Cross Arm Component in Transmission Tower: A Brief Review : M.**

R. M. Asyraf,<sup>1</sup> M. R. Ishak,<sup>1,2,3</sup> S. M. Sapuan,<sup>3,4</sup> N. Yidris,<sup>1</sup> R. A. Ilyas,<sup>5,6</sup> M. Rafidah,<sup>7</sup> and M. R. Razman

*Hindawi International Journal of Polymer Science Volume 2020, Article ID 8878300, 15 pages*

<https://doi.org/10.1155/2020/8878300>

Recently, advanced technologies exploit materials from nonrenewable resources such as petroleum, natural gas, metal ores, and minerals. Since the depletion of these resources and environmental issues, it has brought attention to researchers to progress in the development of biodegradable materials from green composites. Most biofibres and biopolymers are obtained from agricultural waste products either from stem, leaf, stalk, or fruit. Nowadays, green composites with well-regulated life span have been widely discussed in numerous fields and applications. Some studies have shown that biofibres and biopolymers have comparable mechanical, thermal, and physical properties with glass fibre and other synthetic polymers. Thus, researchers are progressively narrowing down the development of green composite materials in many high strength applications, such as house deck and automotive components. This review focuses on the background of green composites (natural fibres and biopolymers), the manufacturing processes, potential applications in cross arm structures, and testing evaluations. This article also focuses on the specific current cross arm configurations and the pultrusion process to form squared hollow section beams. Many open issues and ideas for potential applications of green composites are analysed, and further emphases are given on the development of environmentally friendly material structures. Hence, the article is expected to deliver a state-of-art review on manufacturability and perspectives of natural fibre reinforced biopolymer composite cross arms for transmission towers.

**Study on Properties of Heat-Resistant Hybrid Resin Containing Silicon and Composites**

Lixin Xuan,<sup>1,2</sup> Quan Zhou,<sup>3</sup> Zhiqiang Wang,<sup>2</sup> and Tao Su

*Hindawi International Journal of Polymer Science Volume 2020, Article ID 4591028, 8 pages*

<https://doi.org/10.1155/2020/4591028>

In recent years, one kind of novel hybrid polymer containing silicon has already been reported in the field of high-temperature resistance polymer. Gradually, it has been a research hotspot in the field of high-performance matrix resins because of excellent heat resistance and dielectric properties. The composite was prepared by M-aminophenylacetylene terminated polymethyldiphenylethynyl silane (MDPES-2) as a matrix and nonalkali glass cloth as reinforced material using a hot press process. The cure reaction of MDPES-2 was characterized. Meanwhile, heat resistance, mechanical properties, and dielectric properties of MDPES-2 composites were systematically studied in this paper. The results showed that flexural strength at room temperature is 321 MPa and flexural strength retention at 240° C was 98.3%. Flexural strength retention after thermal treatment at 500° C for 7 min was 84%. In addition,  $\epsilon$  and dielectric dissipation factor ( $\tan \delta$ ) were 3.9 and  $2.0 \times 10^{-3}$  (10 GHz).

**Performance of polypropylene textile encased stone columns :**

Meenakshi Bansal , D. Rathod, M.S. Abid, S. K. Vanapalli

*Geotextiles and Geomembranes*, [Volume 49, Issue 1, February 2021, Pages 222-242](#)

This paper explores the potential use of a woven polypropylene textile for encapsulating stone columns and improving performance of a local soft soil in Warangal city of India. A series of axial load tests were performed on stone columns of various diameters and under various encapsulation conditions that include single and double layers and other combinations. Load carrying capacity of stone column increased twice its original capacity when encapsulated with different geofabric materials. Performance enhancement strongly correlated to the tensile strength of encasement material and encapsulation condition. In addition, the influence of lateral thrust on group of stone columns arranged in square and triangular patterns were investigated. Irrespective of the material used, lateral displacement reduced by half for encased stone columns. Apart from tensile strength of encasing material, the amount of material used for encasement in the form of additional encasement layer was found to be crucial. The cost of using the polypropylene encasing material is only a third of the commercial geotextiles; however, the performance is inferior to woven geotextiles but far superior to non-woven geotextiles.

**The characterisation of geosynthetic interface friction by means of the inclined plane test**

P. Pavanello, P. Carubba, N. Moraci

*Geotextiles and Geomembranes*, [Volume 49, Issue 1, February 2021, Pages 257-275](#)

The paper focuses on the evaluation of the shear strength in conditions of low normal stress of various geosynthetic-geosynthetic interfaces, which are typical of landfill cover systems, by means of the inclined plane test, with the aim of studying the friction mobilisation in relation to various kinematic behaviours. The results of three different methods to evaluate the angle of friction were analysed, together with the sensitivity of the in-terfaces in relation to the wear effect and the influence of the state of hydration. The results showed very different responses of the interfaces to the shear stress, which involved three main types of sliding mechanisms, referred to as sudden, gradual and uneven sliding. Another outcome observed was that the shear strength of geosynthetic-geosynthetic interfaces cannot always be properly characterised following the procedure proposed by the European standard for soil-geosynthetic interfaces (EN ISO 12957-2), since the actual mobilised kinematic behaviour should be taken into consideration. In this regard, the paper provides some hints on the choice of the more representative parameter of friction for each type of sliding. A particular focus was given to the case of gradual sliding interfaces, for which the static friction is difficult to detect due to the very slow movements; for practical purposes, the design friction of these interfaces should be evaluated by using an adequate safety factor with respect to the friction evaluated at 1 mm of displacement.

**An Investigation of Using Thermoset Polymer Type Liquid Additives to Improve Cement Grout Performances in Rock Bolting Applications :**

E. Komurlu

*International Journal of Geosynthetics and Ground Engineering*, volume 6, 52 (2020)

Effect of thermoset type polymeric additives on load-bearing capacities of grouted rock bolts was investigated with both laboratory and site studies. For the specimen preparation, two different silicone-based polymeric materials were added with different amounts in the cement and water mixes. Additionally, cement grouts without the polymeric additive were also used to compare results obtained from different grout usage cases. The additives are in the liquid phase while adding into the fresh cement mix and start to solidify due to the polymerization after being mixed in the grout material. Support performances of various grouted rock bolt specimens were tested under axial and shear loading conditions. According to the results of this study, use of the polymeric additives with an amount of 3.5% by weight in total mix significantly improves the ductility in support reactions of grouted bolts and increases the energy absorption capacity by high rates over 50%.

**EPS Geofoam as a Wave Barrier for Attenuating High-Speed Train-Induced Ground Vibrations: A Single-Wheel Analysis :**

M. R. Khan, S. M. Dasaka

*International Journal of Geosynthetics and Ground Engineering*, volume 6, 43 (2020)

Vibrations induced by high-speed trains in the ground are a common cause of concern in the urban railway lines due to construction of buildings and inhabitation of residents close to railway lines. The present study aims to evaluate the potential use of geofoam as an in-fill material for passive trenches to reduce the vibrations generated by railways.

Considering the higher frequencies of railway vibrations in comparison with earthquake excitations, characterization of vibration attenuation efficiencies of geofoam in-filled trenches is done in the scale of peak particle velocities (PPV). A comprehensive study is conducted on the effects of buffer stiffness, geofoam density, and dimensions and location of the in-filled trench away from the railway track. Results indicate that having low buffer stiffness for the geofoam in-filled trenches are efficient in reduction of railway-induced ground vibrations. Grades of geofoam with smaller densities are observed to have better vibration attenuation characteristics as well. It is also characterized that the location of the trench close to the railway embankment results in an overall reduction in the vibration levels in the ground. A higher depth of in-filled trench is requisite, if the trench is located at higher distance from the track. It is ascertained that geofoam in-filled trenches are highly suitable for attenuating the train-induced vibrations in the native sub-soil.

### **Microbial Electrochemical System: A Sustainable Approach for Mitigation of Toxic Dyes and Heavy Metals from Wastewater**

*Bikash Kumar ; Komal Agrawal ; and Pradeep Verma*

*Journal of hazardous toxic and radioactive waste, Vol. 25 Issue 2- 2021*

Anthropogenic activities have lead to the accumulation of toxic and hazardous waste materials, such as dyes and heavy metals, into the environment. The entry of these waste materials into the food chain has emerged as a threat to food and health security. As a result, several conventional physical and chemical-based systems were developed to remove these toxic dyes and heavy metals from wastewater. However, because the conventional methods were costly, energy intensive, and ineffective, microbe-based bioremediation systems gained attention of the scientific community. Among several bioremediations approaches, the microbial electrochemical system (MES) has shown promising results in the selective removal of dyes and heavy metals from wastewater. The chemical energy of the biodegradable substrates is converted to electrical energy using the inherited electrochemical system of the electroactive microorganisms. This is collectively termed a “microbial electrochemical/bioelectrochemical system” (MES/BES). The MES in presence of electroactive microbes can remediate the toxic compounds from a wide range of wastewaters. Thus, the present review provides detailed insight into the principle, mechanisms, electrochemistry, and biochemical capabilities of electroactive microbes during bioremediation of heavy metals and dyes. In addition, the paper discusses the challenges faced in designing large-scale MES and its implementation/commercialization. The future prospect and strategies for the development of a self-sustainable multipurpose MES for bioremediation and recovery of toxic and value-added compounds, respectively, have also been elaborated.

### **Degradation of Direct Blue 1 through Heterogeneous Photocatalysis with TiO<sub>2</sub> Irradiated with E-Beam**

*Elvia Gallegos, Florinella Munoz Bisesti, Katherine Vaca-Escobar, Cristian Santacruz, Lenys Fernandez, Alexis Debut, Patricio J. Espinoza-Montero Processes, 2020, 8, 1181.*

Most dyes used in the textile industry are chemically stable and poorly biodegradable, therefore, they are persistent in the environment and difficult to degrade by conventional methods. An alternative treatment for this kind of substance is heterogeneous photocatalysis using TiO<sub>2</sub>, so, in this work, it is proposed to degrade Direct Blue 1 (DB1) using microparticulate TiO<sub>2</sub> irradiated with e-beam at three different doses: 5, 10 and 20 kGy (J/kg). The DB1 degradation was implemented in a batch reactor (DB1 initial concentration = 50 mg L<sup>-1</sup>, pH 2.5, TiO<sub>2</sub> concentration = 200 mg L<sup>-1</sup>). We have demonstrated that the photocatalytic power of TiO<sub>2</sub>, when irradiated with e-beam (5, 10, 20 kGy), varies slightly, with minor effects on photodegradation performance. However, the dose of 10 kGy showed a slightly better result, according to the DB1 photodegradation rate constant. Adsorption process was not affected by irradiation; its isotherm was fitted to Freundlich's mathematical model. The DB1 photodegradation rate constants, after one hour of treatment, were: 0.0661 and 0.0742 min<sup>-1</sup> for irradiated (10 kGy) and nonirradiated TiO<sub>2</sub>, respectively. The degradation rate constant has an increase of 12.3% for irradiated TiO<sub>2</sub>. Finally, there was no evidence of mineralization in the degradation process after 60 min of treatment. According to the results, the irradiation of microparticulate TiO<sub>2</sub> with e-beam (10 kGy) slightly improves the photodegradation rate constant of Db1.

### **3. Toxicity and color reduction of a textile effluent containing reactive red 239 dye by electron beam irradiation**

*Vanessa S.G. Garciaa, Jorge M. Rosab, Sueli I. Borrely, Radiation Physics and Chemistry*

*Volume 172, July 2020, 108765*

Textile effluents are a mixture of dyestuff, surfactants, dispersants, acids, alkalis and bleaching agents, among other compounds, and some of these are highly soluble and relatively recalcitrant. Suitable improvement of textile effluents may

require combined treatment processes, such as Conventional Treatments and Advanced Oxidative Process (AOPs). Electron beam irradiation (EBI) has been proposed as a possible technology for the treatment of textile effluents. In this context, the aim of the present study was to evaluate the efficiency of an Electron Beam treatment applied to toxicity and color reduction of a textile effluent containing reactive Red 239 dye. Effluent COD and TOC were also evaluated. The assessed effluents were submitted to EBI treatment at doses ranging from 0.5 to 15 kGy. *Vibrio fischeri* bacteria and *Daphnia similis* crustaceans were exposed to both irradiated and non-irradiated effluents, the toxicity was evaluated through EC50 (median effective concentration) calculations. EB irradiation successfully reduced effluent toxicity and color. The EC50 for *D. similis*<sub>(48h)</sub> were of  $6.31\% \pm 3.19$  (non-irradiated) and  $27.56\% \pm 9.31$  (10 kGy), and for *V. fischeri*<sub>(15min)</sub> of  $7.41\% \pm 1.52$  (non-irradiated) and  $31.89\% \pm 10.99$  (10 kGy), respectively. Approximately 70% toxicity reduction was obtained for both organisms, while 95% color reduction was obtained by applying 5 kGy.

#### **Electron beam irradiation post treatment for degradation of non-biodegradable contaminants in textile wastewater.**

*S. C. Deogaonkar, P. Wakode, and K. P. Rawat, Radiat.*

*Phys. Chem., vol. 165, p. 108377, Dec. 2019.*

Wet processing is one of the major streams in textile engineering refers to textile chemical processing. Textile wet processing involves three stages mainly pre-treatment or preparation (de-sizing, scouring), coloration (dyeing and printing) and finishing. In de-sizing and scouring processes sizing material (starch/ PVA) and natural impurities (oil/wax/fat) are removed respectively. Dyeing and printing are the processes for coloration of fabric with natural or synthetic dye and for producing any pattern on the fabric respectively. Textile wet processing produces high load of pollutants to textile effluent treatment plant (ETP) of which aim is to safely discharge of water to meet the consent limits provided by environmental regulatory authority. In this paper, the degradation of simulated textile waste water arising from various processes viz. de-sizing, scouring, dyeing and printing was evaluated by treatability study with activated sludge process. The results show de-sizing, dyeing and printing processes mainly to be contributing to non-biodegradable contaminants in textile effluent treatment plant (ETP). In this study, electron beam radiation technology was applied to enhance the biodegradability of simulated textile effluent (mixed) pre-treated by activated sludge process. This mixed effluent comprises of all constituents related to scouring, de-sizing, dyeing and printing process with known concentrations has never been considered before, in any of the available literature on EB irradiation of Textile waste waters. Most of the studies on EB irradiation on wastewater showed focus on single pollutant. The results showed that E-beam irradiation pre-treatment did not improve the biodegradability of textile effluent even when it was irradiated up to 80 kGy. However, in contrast, e-beam irradiation as post treatment to biologically treated sample could significantly enhance the biodegradability at very low dose 1 kGy where hydroxyl radical (OH) played a very active role. The values of COD, BOD, and BOD/COD ratio were compared before and after E-beam treatment. The absorbance spectra in UV-Visible range were obtained for mixed effluent.

#### **Facile method to fabricate carbon fibers from textile-grade polyacrylonitrile fibers based on electron-beam irradiation and its effect on the subsequent thermal stabilization process**

*S. H. Yoo et al.,*

*Carbon N. Y., vol. 118, pp. 106–113, Jul. 2017.*

An electron-beam was irradiated on textile-grade polyacrylonitrile (PAN) fibers at various electron doses (200, 500, 1000, and 1500 kGy) prior to the thermal stabilization to fabricate carbon fibers (CFs). Textile-grade PAN fibers experienced superficial fusion between filaments during the thermal stabilization due to a large amount of co-monomer contents. However, electron-beam irradiation prior to thermal stabilization prevented the superficial fusion. Furthermore, the total stabilization time required to achieve the same degree of stabilization was effectively reduced as 64% by electron-beam irradiation. Various radicals were formed within textile-grade PAN fibers and they were recombined to crosslink with each other by gradually increasing the temperature. As a result, the elongation of irradiated fibers was suppressed and the glass transition temperature shifted to higher temperatures compared with the pristine fiber by increasing the electron dose. On the other hand, the onset and peak temperature of cyclization reaction for irradiated fibers were shifted to lower temperatures which indicated that the cyclization reaction was initiated at lower temperatures than pristine fibers. The tensile strength, Young's modulus, and strain-to-failure of resulting CFs were  $1.83 \pm 0.23$  GPa,  $147.44 \pm 4.55$  GPa, and  $1.30 \pm 0.15\%$ , respectively.

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