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Characterization of Nanofibers Developed from PS and PCZ with TTIP

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***Abstract*—Polystyrene (PS) is a transparent insulator known for its outstanding dielectric qualities. Capacitors made from polystyrene show minimal dielectric adsorption and practically no leakage. Polycarbazole (PCZ), a kind of conducting polymer, showcases remarkable optical properties. Titanium Isopropoxide, known for being a powerful oxidizing agent, has a large surface area that contributes to its high photocatalytic activity. Electrospinning is a technique based on the use of electrostatic forces for producing fibres with the diameter from ten nanometres to some micrometres. Such fine fibres cannot be produced with conventional methods for the production of nonfibers. The present work is carried out to develop nanofibers samples by combining polymers PS, PCZ with filler Titanium Isopropoxide (TTIP) by electrospinning. The developed viscous solution of was electrospun for different ratio namely 9:1, 7:3 and 5:5 in order to obtain ultra-thin nanofibers samples. The investigation of surface morphology of the nanofibers is carried out by Scanning Electron Microscopy (SEM). The X-ray diffraction (XRD) analysis was used to study structure and crystalline size of nanofibers samples.**

***Keywords*— Electrospinning, Polystyrene, nanofibers, insulating polymers.**

I. INTRODUCTION

Nanofibers are incredibly thin fibers with diameters in the nanometer range. Due to their small size, they exhibit unique properties that differ significantly from those of bulk materials. Nanofibers can be formed through various methods, including Electrospinning, chemical vapor deposition, and template synthesis. Electrospinning is one of the most common techniques, where a polymer solution or melt is spun into fibers under the stimulus of an electric field. Nanofibers have a large surface area-to-volume ratio, which enhances their mechanical strength, electrical conductivity, and thermal properties compared to bulk materials. This makes them useful in a range of applications. Handling and processing nanofibers can be complicated process due to their small size and potential for aggregation. Additionally, understanding their long-term influences on health and the environment is crucial as their use becomes more widespread. The aim is to develop ultrathin nanofibers by combining hybrid polymers namely Polystyrene and Polycarbazole with filler Titanium Isopropoxide (TTIP). The solution is prepared by mixture of PS and PCZ polymer and stirring the hybrid polymers and filler solution to produce viscous solution. The developed viscous solution of corresponding 9:1, 7:3, 5:5 is electro-spun by electrospinning technology in order to obtain ultrathin nanofiber. The study of surface morphology on the developed nanofiber samples were carried out using scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX).

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Any polymers are made up of macromolecules or very large molecules, which are just multiples of simpler chemical building blocks known as monomers [1]. Here two polymers i.e PS and PCZ are used to prepare the solution.

Polystyrene is a synthetic polymer which can be solid or foamed. Polystyrene is a transparent, rigid and brittle polymer which is easy to process. The Conductive polymers (CPs) are polymers which are organic semiconductors for many essential applications because of their physical/chemical tunable properties. It can be tuned for mechanical flexibility and it has very low weight, the reversible doping, good biocompatibility, and scalable production.

TTIP is used as a precursor to produce high-purity TiO_2 for photocatalysis, pigments, and ceramics. In the present research work the combination of PS, PCZ and TTIP filler are synthesized to form the viscous solution to produce nanofibers with the Electrospinning process [4].

Electrospinning is a technique which is widely used to produce nanofibers and other nanostructures. Electrospinning is a straightforward, adaptable and inexpensive approach for creating organic and inorganic nanofibers with sizeable lengths, consistent diameters, and a range of compositions among the known methods for synthesizing nanostructures. The process uses an electric field/force to produce a polymer solution or melt into fine fibers. The solution is placed in a syringe with a needle and an electric voltage of around 5Kv to 25Kv is applied between the needle and the collector plate or drum. The collector is typically grounded or at a different electrical potential. When the voltage is applied, an electric field is created between the needle and the collector. This field exerts a force on the polymer solution or melt, causing it to stretch and form a fine jet. As the polymer travels through the electric field, it elongates and thins out due to the electrostatic forces and the solvent evaporation (if using a solution). The solvent evaporates or solidifies, leaving behind a nanofiber. The nanofibers are collected on the collector, which can be a flat surface, a rotating drum, or a mesh. The orientation of the fibers and its structure of the fibres can be controlled by adjusting the collector setup and process parameters [5]. Here a high voltage of 10-30 kV is used to electrospun a polymer droplet suspended at the end of a syringe needle. As soon as this charge surpasses the surface tension of the solution, charge builds up on this droplet, which elongates and eventually forms a Taylor cone. The PS, PCZ and TTIP mixture solution poured into the syringe to fix in the electrospinning machine where ultrathin nanofibers samples are produced in terms of nanometer range.

II. MATERIALS AND METHOD

Polymers are the molecules composed of repetitive structural units, called monomers, that are covalently bonded together in long chains. They are fundamental to many materials and applications in modern life, ranging from plastics and fibers to biological molecules and synthetic materials. Nanoparticles are extremely small particles with dimensions ranging from one to hundred nanometers (nm). Due to their size, they exhibit excellent physical, chemical, and biological properties that differ from those of bulk materials. The polymers and fillers are used in this process. The hybrid polymers namely PS, PCZ with the filler TTIP is used to develop nanofibers samples. The steps followed in the implementation of the work is as shown in the below figure 1.

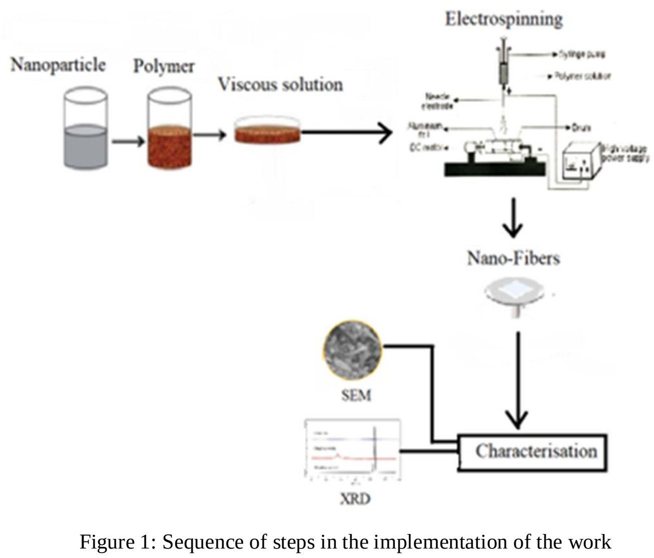

Figure 1: Sequence of steps in the implementation of the work

A. Preparation of Viscous Solution

The preparation of nano composite or the viscous solution is carried out as follows. The hybrid polymers namely PS and PCZ are used to prepare viscous solution. The nanofibers produced for the following combination of polymer solutions. The solution is prepared using two methods which are discussed below.

- Base solution (PS+PCZ) using direct method: 4g of Carbazole dissolved in 96ml of DCM. 15g of polystyrene dissolved in 85ml DMF. The prepared base solution is shown in figure 2

Figure 2: Base solution using direct method

- Base Solution using precipitate method: 60mM Carbazole dissolved in 250ml of DCM and 1.2M APS in 0.5M Hcl. Slowly Carbazole solution was added to the Hcl solution. The mixed solution was kept at room temperature for 24Hrs. The filtration process was carried out and the precipitate was obtained. The precipitate was air dried at ambient temperature. The synthesized PCZ precipitate was mixed in polystyrene solution which was dissolved in DMF. The solution was stirred for 2Hrs. The stirred solution was used for electrospinning process using a voltage of 20-25Kv at a flow rate of 5 ml/hr.The prepared base solution with precipitate is shown in Figure 3.

Figure 3: Base solution using precipitate method.

The prepared base solution of PS, PCZ is added with filler TTIP. The prepared solution is made into four different solutions named as T0, T1, T2 and T3. These names correspond to the ratio of each material as listed in Table 1.

TABLE I: SOLUTION AND ITS COMPOSITION IN PERCENTAGE

Sample	PS+PCZ	TTIP
T0	100	0
T1	90	10
T2	70	30
T3	50	50

B. Electrospinning Process

A nanoparticle is a tiny particle with a size between one and one hundred nanometers. Any member of the family of naturally occurring or artificial substances known as polymers is made up of very big molecules, or macromolecules known as monomers. “Electrostatic spinning” which is also known as electrospinning is one of the easiest and most economical ways to make polymer fibers with diameter in the sub-micron to nano-meter ranges by the application of high electrostatic filed [11]. A grounded collector screen, a syringe pump, a syringe, and a high voltage power source, discharge stick and hazard lamp are the components of Electrospinning setup.
The composite solution (PS, PCZ) of different ratio of without filler TTIP (T0 sample), 9:1 (T1 sample), 7:3 (T2 sample) and 5:5 (T3 sample) samples are taken in a syringe of 2 ml with a needle diameter of 2 mm. The syringe

was fed by the nanocomposite solution and placed on a pumping machine that rotate clockwise direction and that is provided by a controlled flow rate of 2ml/hr is set through the display. The flow rate is kept constant for all the four samples. The voltage is applied to the needle by 20 kV-25KV for all the four samples. The tip of the needle to the plate collector the distance is 10 cm for all the four samples. A droplet will form on the needle tip or capillary tip as the syringe is pumped, and an electric field created between the two will cause charges to be induced into the hemispherical shape to form on the capillary tip.
The formation of Taylor's Cone, also known as a conical shape, and the execution of a jet of filament occur as the liquid droplet receives a sufficiently high voltage, as the electric field produced balances the surface tension of the droplet. Due to whipping instability, the jet will travel in a straight jet form for about 10 cm before turning into a spiral route that leads to the collector. Before the filament gets gathered on the rotating collector or plate collector, it will first evaporate and then solidify while travelling the distance [12]. In the present work, all the four samples are electros pinned for 1hr each by maintaining optimized settings for the flow, volume of the solution from syringe and the electric field applied in producing the nanofibers [16].
The fiber diameter is affected to some extent by elongation as it begins to occur along the trip distance. Even if this component may have contributed to the outcomes, there is still another significant factor that may have an impact on the development and shape of the fibers. For all four concentrations to achieve statistical significance, the trials were repeated.

C. Characterization

Characterizing nanofibers samples involves a range of different techniques to understand their physical, chemical, and mechanical properties. Scanning Electron Microscopy (SEM) is a powerful imaging technique used to characterize the surface morphology and structure of materials at high resolution. It is particularly useful for analyzing nanofibers samples due to its ability to provide detailed images at the nanometer scale. SEM uses a focused beam of high-energy electrons that scans the surface of a sample. As the electrons interact with the sample, they produce various signals, including secondary electrons, backscattered electrons, and X-rays. Detectors capture these signals to form an image, revealing detailed surface features and composition.
The produced nanofibers samples are characterized by scanning electron microscope (SEM) where the electron beam is used to scan the surface of a sample to create images with an electron microscope and it can only produce two dimensional views. Then it is characterized by an analytical method known as X-ray powder diffraction (XRD) is quick and is generally used to determine the phase of crystalline materials [13].

III. RESULTS AND DISCUSSIONS

The morphological study, XRD of ultrathin nanofibers samples are analyzed using SEM and EDX.
Morphological Study
A scanning electron microscope (SEM) is a kind of electron microscope that can provide¬¬¬¬¬ two-dimensional views of a material.
Electron Microscopes are capable of much higher magnifications and have a greater resolving power than a light microscope, allowing it to see much smaller objects at sub cellular, molecular and atomic level.

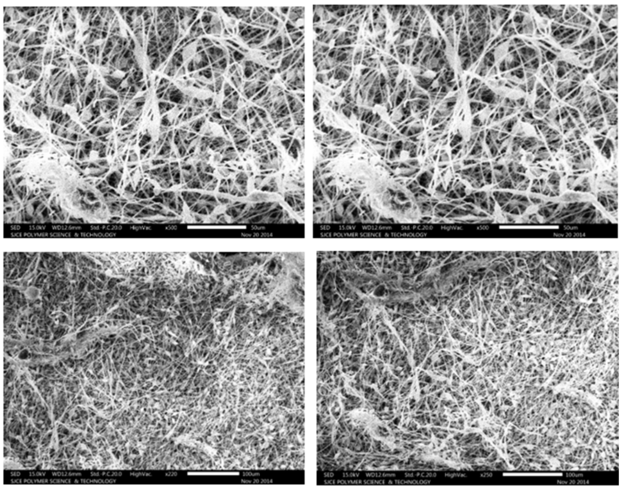

Fig4: SEM images of Base solution nanofiber using direct method

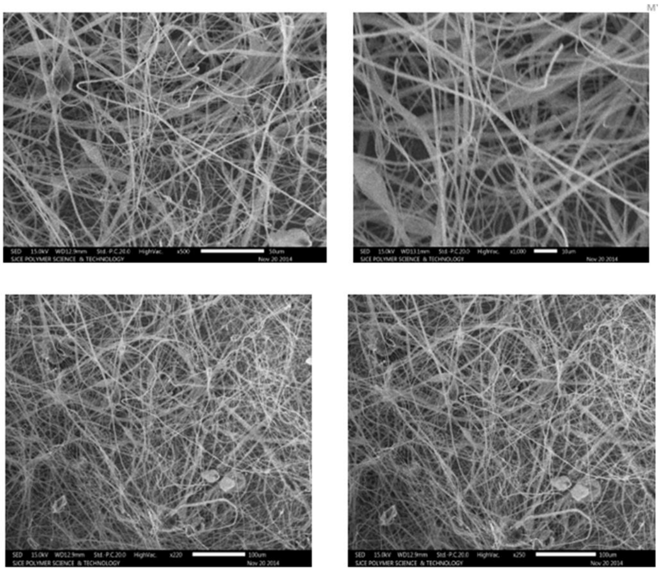

Fig5: SEM images of Base solution nanofiber using precipitate method

EDX or EDS analysis is a widely employed technique together with a scanning electron microscope (SEM). An EDX detector can generate more information about a sample. EDX can be used for both qualitative and quantitative analysis, enabling users to identify both the type of elements that are present as well as the percentage of each element's concentration within the sample. Figure 6 shows the EDX analysis of the samples T0, T1, T2 and T3 respectively. Analysing EDX images confirms the material composition.

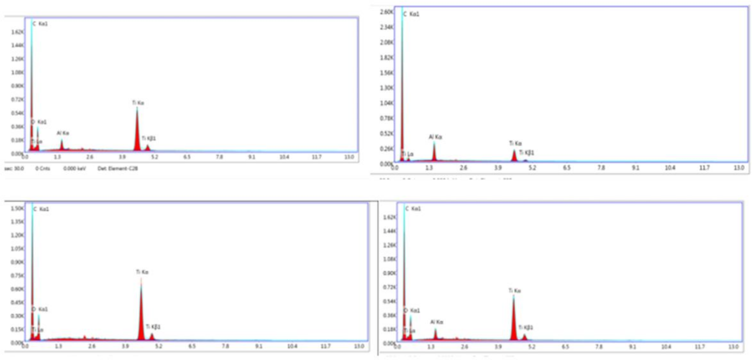

Figure 6: EDX images of the samples T0, T1, T2 and T3

IV. CONCLUSIONS

The following conclusions are drawn from the work carried out

- The nanofibers samples are developed by combining hybrid polymers viz., PS and PCZ with TTIP filler.
- Electrospinning of developed solution to obtain ultrathin nanofiber samples.
- The SEM images show fiber forming bead shape in direct method, whereas bead free structure is obtained for precipitate method.
- The best nanofiber diameter is obtained for 7:3 ratio.

REFERENCE

[1] Xiao-Xiong Wang, Gui-Feng Yu, Jun Zhang, Miao Yu, Seeram Ramakrishna, Yun-Ze Long. "Conductive Polymer Ultrafine Fibers via Electrospinning: Preparation, Physical Properties and Applications ". Journal of Progress in Materials Science, June 2020.

[2] Aditya Sengupta, Soumen Das, Shalini Dasgupta, Pavel Sengupta, and Pallab Datta. "Flexible Nanogenerator from Electrospun PVDF−Polycarbazole Nanofiber Membranes for Human Motion Energy-Harvesting Device Applications". ACS Biomater. Sci. Eng. 2021, 7, 1673−1685

[3] Hameed Naser1,2 · Sabah M. Mohammad1 · Haider Mohammed Shanshool4 · Z. Hassan1 · A. M. Alghareeb Abbas1,3 · Suvindraj Rajamanickam Bimetallic impact on the energy band gap of the polymers PS, PMMA, and PVA nanocomposites, Optical and Quantum Electronics (2024) 56:897 https://doi.org/10.1007/s11082-024-06795-9

[4] Firas K. Mohamad Alosfur, Aseel Adnan Ouda, Noor j. Ridha, Saleh H. Abud Structure and optical properties of TiO2 nanorods prepared using polyol solvothermal method , Conference Paper in AIP Conference Proceedings · August 2019 DOI: 10.1063/1.5123095.

[5] B Raghvendra, T Sankrappa , Amarkumar Malge "Structural, optical and electrical conductivity studies in polycarbazole and its metal oxide nano composites" Springer nature 2022.

[6] Aditya Sengupta, Soumen Das, Shalini Dasgupta , Pavel Sengupta and Pallab Datta, "Flexible nanogenrator from electrospun PVDF-Polycarbazole Nanofiber memnranes for human motion energy- Harvesting device applications", ACS publications, 2021

[7] Liju Fan, Zefang Yu, Z\Xin Wei, Zhanjun Dong, Jing An "Polystyrene electrospun nanofibers as effective sorbents for the removal of atypical antipsychotics: kinetic and thermodynamic studies" Arabian Journal of Chemistry , 2022

[8]] D. Aussawasathien, J.H. Dong, L. Dai. "Electrospun polymer nanofiber sensors". Synthetic Metals 154 (2005) 37–40

[9] Yu G-F, Yan X, Yu M, Jia M-Y, Pan W, He X-X, et al. "Patterned, highly stretchable and conductive nanofibrous PANI/PVDF strain sensors based on electrospinning and in situ polymerization". Nanoscale. 2016;8:2944-50