Uzbekistan Journal of Polymers
journal homepage: https://uzpolymerjournal.com/
ISSN (online): 2181-3256 (print) 2181-3246

PREPARATION OF NANOCOMPOSITE NANOFIBERS BY ELECTROSPINNING AND THEIR PHYSICOCHEMICAL PROPERTIES

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ARTICLE INFO

Received: 03 September 2025
Revised: 15 October 2025
Accepted: 24 October 2025

Keywords:
Nitron fiber, electrospinning, nanofibers, polyaniline, polymer films, conductivity

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ABSTRACT

In this study, nanofibers based on polyacrylonitrile (PAN) fiber "Nitron" were fabricated using the electrospinning method from a dimethylformamide (DMF) solution. The resulting nanofibers were subsequently coated with a polyaniline (PANI) layer to enhance their functional properties. Scanning electron microscopy (SEM) analysis revealed that the average diameter of the uncoated Nitron-based nanofibers was approximately 149.9 nm, while the PANI-coated fibers exhibited a significantly increased diameter of around 700 nm. Electrical conductivity measurements demonstrated that the PANI-modified nanofibers possess conductive properties, indicating their potential for use in electronic and sensing applications.

Introduction

Currently, the production of electrically conductive fibers and textile materials for both household and technical applications is gaining widespread attention [1-3]. Such materials can be utilized in the development of antistatic fabrics, electromagnetic shielding coatings, flexible conductive components, and in wearable items and garments to enhance functionality without compromising flexibility or user comfort [4-6]. Moreover, the potential applications of conductive materials are much broader, encompassing modern wearable devices for health and fitness monitoring, smart protective clothing capable of intelligently responding to external factors such as heat or moisture, and smart fashion that can dynamically manage user comfort [7].

Various approaches are currently employed to produce electrically conductive fibers [8], one of which involves coating conventional textile fibers with conductive substances. In this context, PAN fibers represent a promising matrix material. PAN fibers are among the most widely used synthetic fibers in industry, marketed under different trade names such as “Nitron,” “Orlon,” “Cashmilon,” “Dralon,” and others [9]. This is due to their high mechanical strength, excellent lightfastness, chemical inertness, moisture resistance, and remarkable thermal stability [10]. Conductive polymers are considered suitable for use as flexible coatings, as they can form interpolymer complexes, ensuring stronger adhesion to the fiber surface. Therefore, the aim of this study is to obtain electrically conductive nanofibers by coating the surface of "Nitron" nanofibers with PANI.

Experimental Section

Aniline (aminobenzene, phenylamine, $C_6H_5NH_2$) of analytical grade (KhimReaktiv, Russia) was used and purified by double distillation prior to use. Ammonium persulfate ($(NH_4)_2S_2O_8$) of

"ACS" grade ("Sigma-Aldrich", USA) was used as the oxidizing agent. The "Nitron" fiber (JSC "Navoiazot", Uzbekistan) is a copolymer of acrylonitrile with methyl acrylate and itaconic acid.

Nanofibers based on "Nitron" were formed using an electrospinning unit CY-ES 200K (China). IR spectra of the samples were recorded using a Nicolet iS50 spectrophotometer (Thermo Fisher Scientific, USA). Scanning electron microscopy (SEM) analysis of the fiber samples was performed using micrographs obtained with a SEM-EDS microscope Jeol JSM-IT200LA (Japan).

The electrical conductivity of the nanofiber samples was measured using a microammeter model M2000.1 at 300 K. A B59 device was used as the voltage source.

Results and Discussion

Fibers based on acrylonitrile (AN) homopolymers are known to be poorly elastic, difficult to dye, and have limited functionality [10]. Therefore, in most cases, industrial chemical enterprises produce fibers based on acrylonitrile copolymers with other monomers such as methyl acrylate, methacrylic acid, itaconic acid, acrylamide, vinyl acetate, etc. The incorporation of comonomers into the polymer not only alters the physical properties of the resulting fiber but also improves dye absorption, solubility, hygroscopicity, and other characteristics. It is known that "Nitron" fiber is a terpolymer consisting of acrylonitrile (~92.5%), methyl acrylate (~6.0%), and itaconic acid (~1.5%). The presence of itaconic acid in the copolymer provides functional groups that enable the fiber to form complexes with various positively charged compounds, including polymers. In this study PANI, known for its electrical conductivity, was selected as the conductive coating material for the surface of "Nitron" fibers. PANI is easily synthesized by the polymerization of aniline, is sufficiently thermally stable, chemically inert, and considered a safe polymer. Currently, there is no technical feasibility to form polymer fibers directly from PANI solutions, as no solvent capable of fully dissolving this polymer has been found. Furthermore, the melting temperature of PANI exceeds its decomposition temperature, which prevents fiber formation from its melt. However, PANI contains positively charged nitrogen-containing groups along its main chain, which can form strong electrostatic interactions with the negatively charged carboxylic groups present in the AN copolymer, resulting in a conductive coating on the surface of the "Nitron" fibers.

Preliminary experiments showed that commercially produced fibers are not effectively coated with PANI. Therefore, to increase the surface area of the "Nitron" fibers, nanofibers were produced via the electrospinning method. It is well known that nanofibers have a much larger surface area compared to conventional fibers, and consequently, they possess a higher density of negative charges. This facilitates the formation of interpolymer complexes with positively charged PANI on the surface, forming a uniform polymer layer.

To fabricate the nanofibers, a solution of "Nitron" was prepared by dissolving industrial fibers in dimethylformamide (DMF). The nanofibers obtained by electrospinning were analyzed using SEM, and their micrographs at various magnifications are presented in Figure 1.

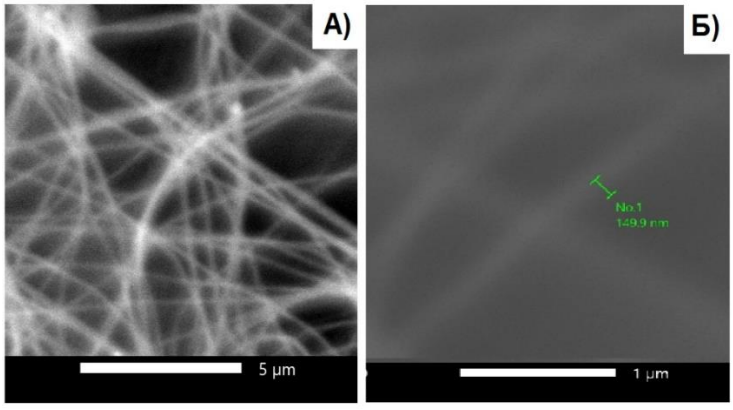


Figure 1. *SEM micrographs of "Nitron" nanofibers obtained by electrospinning*

As seen from the SEM micrographs of the fibers, their average diameter is 149.9 nm (Fig. 1b), which classifies them as nanofibers.

The structure of the obtained “Nitron”-based nanofiber was identified by analyzing its IR spectrum, which is shown in Fig. 2.

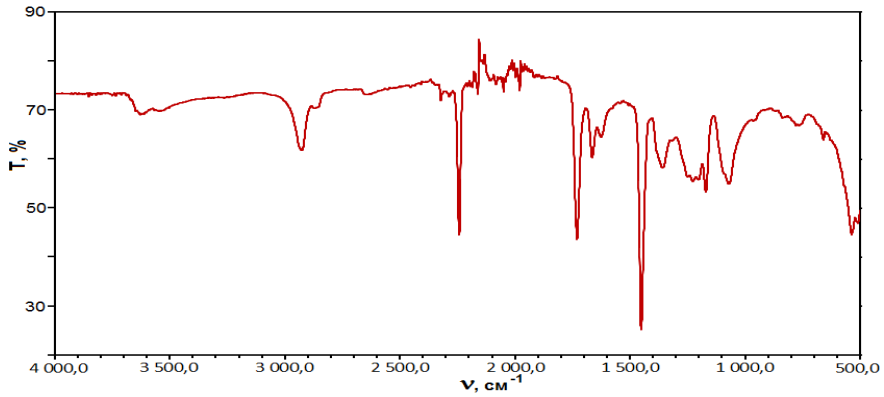


Figure 2. *Result IR spectrum of “Nitron” nanofiber*

As seen in Fig. 2, the IR spectrum of the fiber exhibits an intense peak at 2242.7 cm^{-1}, corresponding to the stretching vibrations of the –CN groups from the acrylonitrile units in the copolymer. The methyl acrylate units present in the copolymer can be identified by the stretching vibrations of the carbonyl group at 1731 cm^{-1} and by several bands in the 1300–1100 cm^{-1} region, corresponding to C–O–C vibrations of ester groups. The intense peak at 1664 cm^{-1} is characteristic of the carbonyl group belonging to the itaconic acid component in the copolymer. The –OH groups of itaconic acid form peaks around 1452 cm^{-1}, while the –C–O– groups are observed near 1356 cm^{-1} [11]. Analysis of the IR spectrum of the “Nitron” nanofibers shows that the chemical structure of the initial copolymer remains unchanged during the fiber formation process.

To coat the surface of the “Nitron”-based nanofibers with a PANI layer, oxidative polymerization of aniline was carried out in a reaction vessel where the nanofibers were immersed. Aniline polymerization was performed in an aqueous solution in the presence of a strong oxidizing agent, ammonium persulfate ($(NH_4)_2S_2O_8$).

During the reaction, the surface of the nanofibers turned black-a typical color of PANI-indicating the formation of a polymer layer on the fiber surface. After completion of the polymerization reaction, the resulting nanofibers were washed first with distilled water, followed by ethanol and acetone. To dope the PANI layer, the samples were treated with a 0.1 N HCl solution and then dried at 333 K to constant weight. SEM micrographs of the resulting nanofibers are shown in Fig. 3.

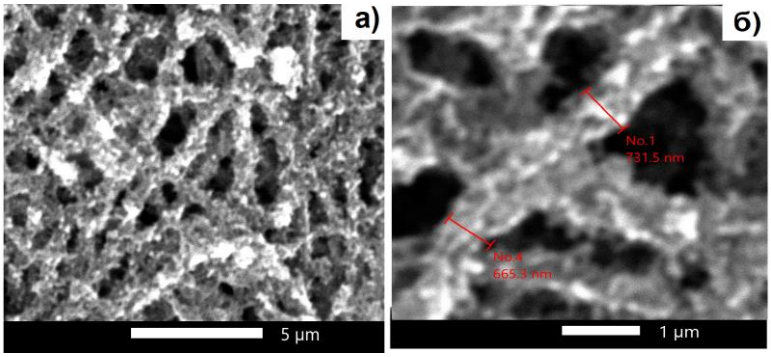


Figure 3. *SEM micrographs of “Nitron” nanofibers coated with a PANI layer*

As seen in the SEM micrographs presented in Fig. 4, the overall appearance of the PANI-modified “Nitron” nanofibers differs significantly from that of the original fibers shown in Fig. 1.

It is also evident that the average diameter increases more than 4.5 times (average ~698.4 nm) compared to the unmodified nanofibers. The chemical composition of the modified fibers, determined by energy-dispersive spectroscopy (EDS, Fig. 4), also differs from that of the original fibers. Composition of the original "Nitron" nanofiber: C – 62.25 ± 0.03 wt.%, N – 30.86 ± 0.09 wt.%, O – 6.89 ± 0.04 wt.%. Composition of the fiber coated with PANI: C – 63.81 ± 0.04 wt.%, N – 25.18 ± 0.03 wt.%, Cl – 6.78 ± 0.01 wt.%, O – 4.23 ± 0.03 wt.%. The element distribution maps show that the elements are uniformly distributed across the surfaces of both types of fibers.

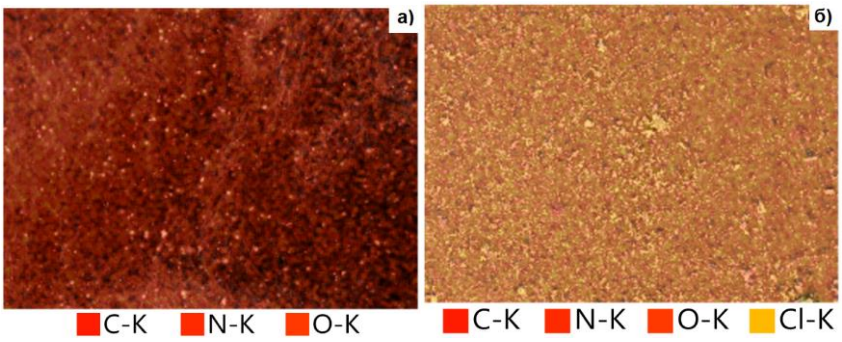
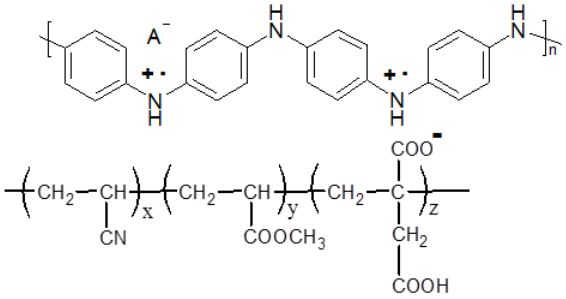


Figure 5. *Energy-dispersive elemental mapping of "Nitron" nanofiber (a) and nanofiber coated with a PANI layer (b)*

Thus, based on the obtained data, it can be concluded that, unlike the industrial "Nitron" fiber, the nanofibers derived from it are capable of forming polymer complexes with PANI, which can be schematically illustrated as follows:



Electrical conductivity is an important property in nanofibers—especially in nanocomposite materials—for several key reasons, depending on the intended application [12].

To investigate the electrophysical properties, the nanofibers were connected to a DC power source, and their electrical conductivity and current–voltage (I–V) characteristics were determined. The results are presented in Fig. 5.

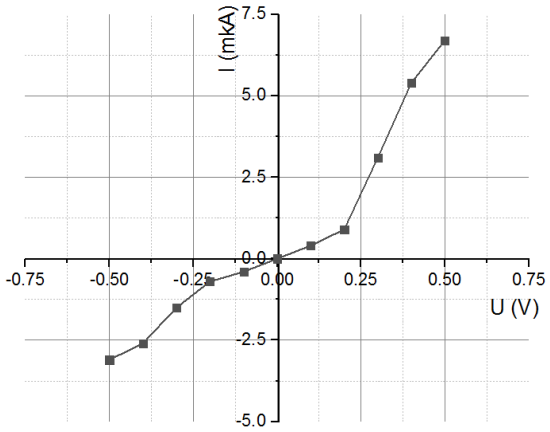


Figure 5. *Current–voltage (I–V) characteristics of forward and reverse current conductivity for "Nitron" nanofiber samples coated with a PANI layer. Temperature: 300 K*

As seen in Fig. 5, the “Nitron” nanofibers modified with PANI and doped with HCl exhibit identical forward and reverse current conductivity values at a temperature of 300 K.

Conclusions

Nanofibers based on the industrial "Nitron" fiber—a terpolymer of acrylonitrile, methyl acrylate, and itaconic acid—were successfully fabricated via electrospinning from a DMF solution. After coating with polyaniline (PANI), the average fiber diameter increased to approximately 698.4 nm. Elemental mapping confirmed the presence and uniform distribution of PANI on the fiber surface. FTIR analysis showed that the chemical structure of the original Nitron copolymer remained unchanged during both fiber formation and surface modification.

Electrical measurements demonstrated that the PANI-coated nanofibers exhibit measurable and symmetric current–voltage (I–V) behavior at room temperature, indicating the successful introduction of conductive properties. This enhanced conductivity is attributed to strong electrostatic interactions between the positively charged nitrogen groups in the PANI chains and the negatively charged functional groups in the Nitron nanofibers.

Overall, this study confirms the feasibility of producing conductive nanocomposite fibers through the surface modification of electrospun PAN-based nanofibers with PANI. These materials show promise for use in sensors, flexible electronics, and other advanced functional applications where both electrical conductivity and high surface area are critical.

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