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ABSTRACT

of the submitted dissertation for the degree of Doctor of Philosophy

**TRIBOELECTRIC GENERATORS
BASED ON MICRO- AND NANOSTRUCTURED
POLYSILOXANE AND NYLON FILMS**

Speciality: 2222.01 – Physics and technology of nanostructures

Field of science: Physics

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The work was performed at the Nano research Scientific-Research Laboratory of the Excellence Center for Research, Development, and Innovations of Baku State University.

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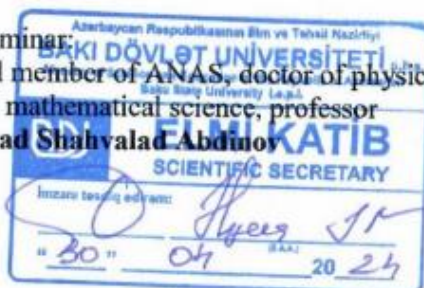
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GENERAL CHARACTERISTICS OF THE WORK

Relevance of the topic and degree of elaboration. Green energy production is considered one of the most urgent problems for all mankind today. Oil, gas, and other traditional resources are gradually losing their relevance, becoming more expensive, and, of course, causing enormous damage to the environment. Therefore, green energy sources that can be used cleanly and safely for the environment – wind, sun, water, biomechanical energy, etc. In terms of cost, the generation of electricity by cheap and technologically easy methods is considered one of the urgent issues, perhaps the most important.

In this regard, generators based on the triboelectric effect (triboelectric nanogenerators (TENG)) can be used for vibration, human movement (stepping, running), water flow, airflow, etc. It is considered a promising method for generating electricity from green energy sources. TENGs are characterized by low cost, miniaturization, flexibility, variety of base material selection, etc. It differs from other alternatives with advantages such as

On the other hand, the increasing use of portable miniature electronic devices (pressure, temperature, humidity, and other types of sensors) requires that they be supplied with electrical energy in various natural conditions. In this sense, TENGs generate electricity from green energy sources, enabling these devices to be conveniently and safely supplied with electricity in harsh natural conditions.

For these reasons, intensive research is currently being conducted in the direction of creating TENGs with various modifications based on materials with triboelectric properties, improving output parameters, and applying them.

In the presented dissertation work, for the first time, the results of research conducted on the creation of micro- and nanostructures on the surface of polysiloxane (PS) and nylon materials, the investigation of the properties of thin-film TENGs prepared by obtaining nylon/TiO₂ and nylon/Fe₃O₄ nanocomposites, and obtaining optimal output parameters have been interpreted.

The object and subject of the research. TENGs based on nanocomposite materials based on nylon, PS, polyurethane, polyvinyl chloride, TiO₂, and Fe₃O₄ with various methods (3D printing, drop casting,

spray coating, and replica molding) were used as research objects. The acquisition technology and the study of the effect of the surface structures formed on the surface of the materials on the physical properties of TENGs constitute the main subject of the research.

Research goals and objectives. The dissertation work aims to form micro- and nanostructures on the surface of PS and nylon materials using different manufacturing technologies, to study the structure and surface morphology of the structures, to study the effect of the structures on the electrical properties of the TENG, to synthesize various metal oxide-based nanocomposite materials in the structure, to use nanocomposites in the TENG is to determine the mechanism of its effect on the output parameters, to determine the areas of application of the developed nanogenerators.

To achieve the goal, the following issues have been resolved:

- preparing TENG based on nylon and PS layers without surface structure and studying their electrical properties;

- fabricate multilayer (origami) TENG based on nylon and PS and determine their output parameters;

- to create micro- and nanostructures on the surface of nylon and PS materials using different fabrication technologies (3D printing, drop casting, spray coating, and replica molding), to study the surface morphology and to investigate the effect on the output parameters of the TENG;

- to synthesize nanocomposite materials based on TiO_2 and Fe_3O_4 nanoparticles by a spray coating method, to investigate their effect on the output parameters of TENG;

- Research increasing the efficiency of TENGs that generate electricity from biomechanical energy associated with wind energy and human movement.

Methods of research. X-ray structural analysis (RSA), scanning electron microscopy (SEM), morphological structure and element composition analysis (EDS), and electrical and dielectric properties measurement methods were applied.

The main provisions defended:

1. It is possible to fabricate TENG based on thin nylon, PS, PU, and PVC thin films obtained by various methods (3D printing, drop casting, spray coating, and replica molding).

2. The surface structures of the nylon and PS layers affect the output parameters of the TENG.
3. It is possible to control the output parameters of the TENG by changing the diameter of the fibers in the nylon layers.
4. The dependence of the efficiency of TENG on the amount of TiO_2 and Fe_3O_4 nanoparticles included in nylon is extreme.
5. The output efficiency of the TEG increases by reducing the size of the conical structures obtained on the surface of the PS sheets with the help of 3D printer technology.
6. Improvement of output parameters of TENG based on nylon / Fe_3O_4 nanocomposite materials is related to the additional electric field generated during contact electrification in Fe_3O_4 .
7. Polyurethane, PS, and nylon-based origami-structured TENGs can be used as biomechanical and wind energy converters. Effect of sizes and shapes of surface structures on electrical properties of TEG.

The scientific novelty of the research.

1. For the first time in the research work, PS and nylon films were used as a hybrid material and it was determined that the conical micro-textures formed on the surface of the PS films increase the active contact area of the materials with each other and ultimately the output power of the TENG.
2. For the first time, the value of the short-circuit current and open-circuit voltage in TENG based on PVC/ TiO_2 and nylon/ TiO_2 nanocomposite materials depends non-monotonically on the amount of TiO_2 nanoparticles and is related to the uneven distribution of polar charges on the surface.
3. It was determined that it is possible to control the output parameters of the generator by changing the diameter of the fibers in TENGs made based on nylon layers with a fibrous or mesh microstructure.
4. For the first time, it was determined that the increase in output power in TENG based on nylon/ Fe_3O_4 nanocomposite materials is due to the additional electric field generated during contact electrification in Fe_3O_4 .
5. The output power of the TENG made by various methods can be increased by forming nanoclusters in volume and artificial textures on

the surface, along with the physical parameters of the contact materials (dielectric permeability, magnetic susceptibility).;

The theoretical and practical significance of research. In the research work, TEGs with different structures based on nylon and PS were prepared and their electrical parameters were investigated. The controllability of surface structures and their effect on TEG performance is of high practical importance. The convenient and safe generation of electrical energy from different types of mechanical energy sources of the obtained TEG samples makes them a new type of energy source for portable electronic devices. Also, the preparation and use of self-powered sensors due to such types of TENGs, health monitoring in daily life, and collection of environmental information in an effective form opens wide opportunities.

Approval and application. The main provisions of the dissertation work and the obtained results were discussed in several national and international scientific conferences and published in their materials:

- 6th International Conference “Nanotechnology” (Georgia, Tbilisi, Georgian Technical University, 4-7 October, 2021);

- 7th International Conference Mtp-2021: Modern Trends In Physics (Azerbaijan, Baku, Baku State University, 15-17 December, 2021);

- Fizika və astronomiyanın problemləri XXI Respublika elmi konfransı (Azərbaycan, Bakı, Bakı Dövlət Universiteti, 21 may, 2021);

- 2nd International Science and Engineering Conference (Azerbaijan, Baku, Baku Engineering University, 26-27 November, 2021);

- VI International Scientific Conference of Young Researchers Dedicated to the 99th Anniversary of National Leader Heydar Aliyev (Azerbaijan, Baku, Baku Engineering University, 28-29 April, 2022);

- 9th International Conference on Materials Science and Nanotechnology for Next Generation “MSNG-2022” (Türkiye, Ankara, Gazi University, 22-24 September, 2022);

- Nanoscience & Nanotechnology Conference (Italy, Rome, Laboratori Nazionali di Frascati, 29 May-1 June, 2023);

- 10th International Conference on Materials Science and Nanotechnology for Next Generation, MSNG2023 (Türkiye, Kayseri, Erciyes University, 27-29 September, 2023).

The name of the institution where the dissertation work was

performed. The dissertation work was carried out at the Nano research Scientific-Research Laboratory of the Excellence Center for Research, Development, and Innovations of Baku State University.

The total volume of the dissertation with a sign indicating the volume of the structural sections of the dissertation separately. The dissertation consists of an introduction, 4 (four) chapters, conclusions, a bibliography of 181 sources cited in the work, a list of abbreviations, and covers a total of 195 pages, including 94 pictures and 2 tables. Dissertation volume (excluding gaps in the text and pictures, tables, graphs, appendices, and bibliography) – 220964 (including Introduction – 11395, Chapter I – 65827, Chapter II – 52132, Chapter III – 39349, Chapter IV – 50910, conclusion – 1351) is a sign.

CONTENTS OF THE WORK

In the introduction, the relevance of the research topic is substantiated, the purpose of the work, the issues that need to be solved to achieve the set goal, research objects, scientific innovation, and the main scientific propositions presented for defense are indicated. In addition, information about the practical and scientific importance of the work, its approval, publications, the structure and volume of the dissertation, and the brief content of the chapters of the dissertation were interpreted.

In the first chapter, a literature review on the physical basis of TEGs, operating modes, theoretical models, and parameters affecting their performance is presented. Also, extensive literature analysis was conducted on the perspectives of materials used in TEGs, especially polymer layers. Finally, the creation of surface structures of triboelectric materials and their effect on the output parameters of the TEG, as well as the effect of the acquisition, size, and shape of such structures on the efficiency of the generator, were investigated. The preparation of such surface structures and the practical significance of the obtained TEGs were comparatively analyzed.

The second chapter is dedicated to the methods of creating thin layers with triboelectric properties and structures on their surface. Thus, information about the advantages and disadvantages of each of the methods used in obtaining thin films and their working mechanisms has been interpreted. Also, 3D printing technology, selected

dissolution, and surface anodization methods for obtaining surface structures are provided in detail. Later, the information about the modern research methods used in the structural, morphological, and stoichiometric analysis of the samples is reflected in this chapter. Finally, detailed information on the mechanisms of the methods used in the study of the dielectric properties of triboelectric layers and the output parameters of TEGs in general is provided.

In the third chapter, the output performance of TENGs made based on PVC/TiO₂ nanocomposites and thin films of nylon, PS, and polyurethane without any surface structure, and the electric energy obtained by origami-structured TEGs were analyzed comparatively with single-layer and three-layer TEGs. Also, the generation of wind energy and the biomechanical energy generated in connection with the mechanical movement of people in daily life into electrical energy, the physical basis of the change of this energy depending on the movement speed and frequency were investigated. Also, the possibility of using TEGs as self-powered sensors for monitoring people's health in daily life has been studied.

Initially, TEGs were fabricated based on plain and structured PS and nylon thin films using replica molding and doctor blade coating techniques. Electrical measurements (short-circuit current intensity, open-circuit voltage) were carried out on the prepared TEGs and are shown in Figure 1.

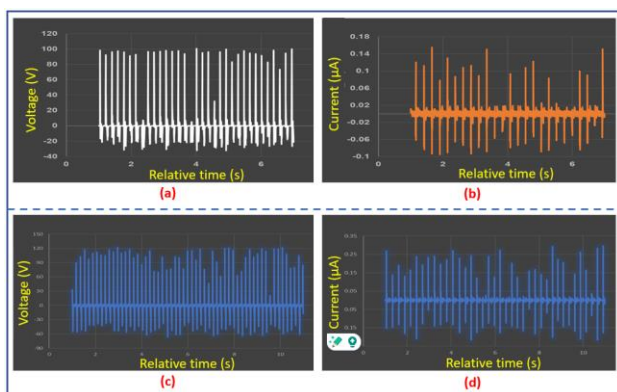


Figure 1. Electrical measurements of TEG. a) voltage-time graph of conventional PS-based TEG; b) current-time graph of conventional PS-based TEG; c) voltage-time graph of structured PS-based TEG; d) current-time graph of a conventional PS-based TEG.

From the electrical measurements, it was determined that the value of voltage and current intensity of structured PS-based TEG is higher than that of conventional PS. Thus, the maximum value of voltage and current intensity of conventional PS-based TEG is 100 V and 0.16 μA , respectively, and the maximum value of voltage and current intensity of structured PS-based TEG is set as 120 V and 0.27 μA , respectively. Compared to ordinary PS, this increase in the value of voltage and current intensity of PS sample-based TEG with a certain structure formed on its surface is explained by the textures on the surface. So, due to such textures, the number of active contact area on the surface increases, which ultimately increases the output parameters of the TEG. It should be noted that the contact of the layers occurs at the atomic level, and the more these contact areas, the greater the number of static charges collected on the surface. The increase in the amount of charge carriers on the surface of triboelectric materials simultaneously increases the potential difference at the electrodes due to electrostatic induction. Also, an increase in the number of charge carriers leads to an increase in the value of the current flowing in the electrode, which is directly proportional to the number of charge carriers.

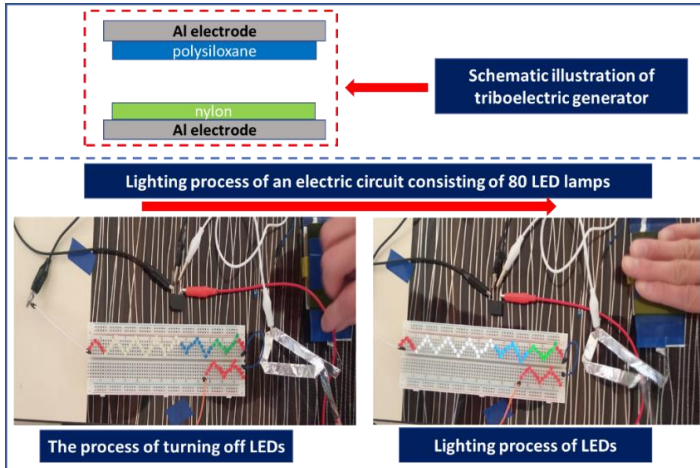


Figure 2. Illumination of 80 LEDs to investigate the practical application of TEG

To check the practical application of the developed TEG, an electric circuit consisting of 80 LED lamps (blue (3.0 V, 30-100 mA), green

(2-2.4 V, 30-100 mA), red (1.8-2.2 V, 30-100 mA), white (5 V, 10 μ A)) was built. The alternating current rectification process obtained from the structured PS-based TEG was performed and the lamps were lit as shown in Figure 2.

For the first time in the generation of wind energy into electricity, TEGs operating based on Scotch yoke were developed using thin nylon film and PS layers as triboelectric material and Al electrode as a metal electrode. The doctor blade coating method was used to obtain PS samples. The thickness of such PS samples obtained in the conducted research was in the range of 90-120 μ m. The thickness of the PS sample obtained in this experiment was determined to be 106 μ m. An example of a TEG designed to generate wind energy into electricity is placed on a Scotch yoke as shown in Figure 3.a. The working principle of the Scotch yoke device is to convert circular motion into forward motion.

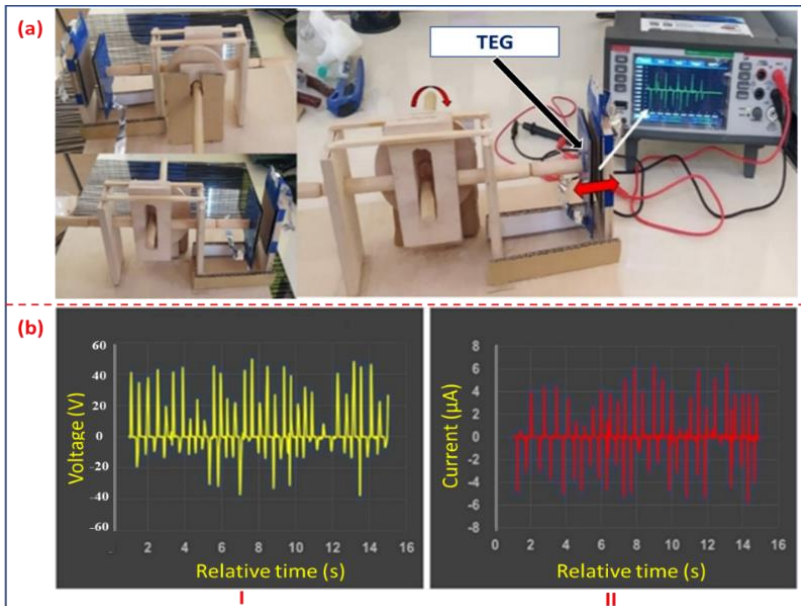


Figure 3. a) Schematic representation of a Scotch yoke-based TEG fabricated using nylon and PS thin films; b) time dependence graphs of voltage (I) and current intensity (II) of the prepared TEG.

The developed TEG was able to efficiently generate wind energy into electricity even at small values of the wind speed. Thus, the maximum

value of the voltage and current intensity of the TEG at the wind speed of 5 Hs was 5 V and 6.2 μ A, respectively, as can be seen in Figure 3.b.

To increase the output efficiency of TEGs, single-layer, three-layer, and five-layer (origami) TEGs have been developed using nylon and PS layers. A drop-casting method was used to obtain thin PS samples. TEGs are mounted on a crankshaft as shown in Figure 4 to generate wind energy into electricity for daily life as a stable energy source, as well as for the development of portable electronic devices. To conduct tests on the prepared TEG samples, an electric cooler was used as a wind source, an anemometer was used to determine the wind speed, and an infrared sensor through an Arduino Nano was used to calculate the frequency of contact of materials in the TEG. First, for the case where the wind speed is 5 m/sec, the experiment was carried out on the TEG with an origami structure, and the rotation frequency of the device was estimated as 5 Hs.

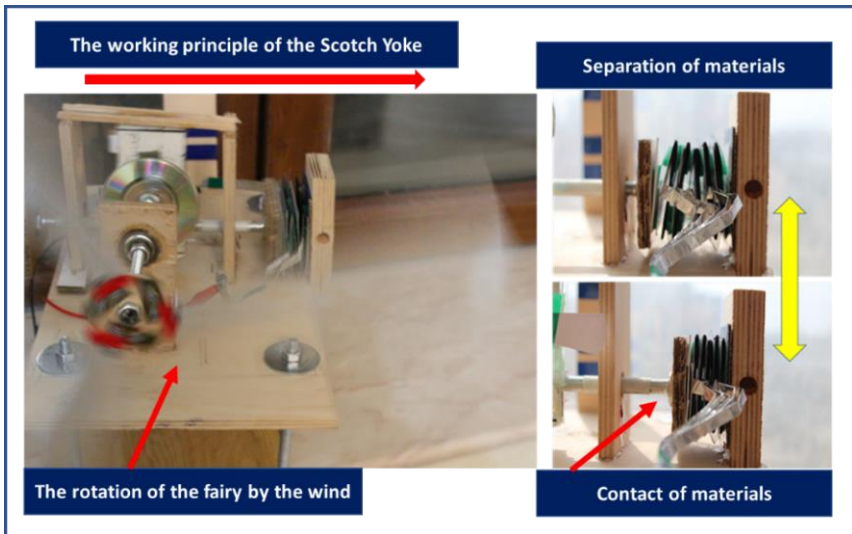


Figure 4. Schematic representation of the operation of the TEG with an origami structure based on the Scotch yoke at a wind speed of 5 m/s

Later, the same experiment was repeated for single-layer and three-layer TEGs. It was determined that TEGs with an origami structure have a higher output power compared to single-layer TEGs. It is clear from experiments that TEG with a 5-layer origami structure has better output

efficiency. So, as shown in Figure 5, the open circuit voltage and short circuit current intensity were determined for each TEG sample at a wind speed of 5 m/s. The maximum values of voltage and current intensity for single-layer TEG were 8 V and 1 μ A, respectively. With the increase in the number of layers, the value of the voltage and current strength of TEG has also increased. Thus, the maximum values of voltage and current intensity for the five-layer TEG were 50 V and 5 μ A, respectively. This increase in voltage and current values is associated with an increase in the number of layers in the TEG. Thus, as the number of layers in the origami structure increases, the contact areas increase, and as a result, the output performance of the TEG increases.

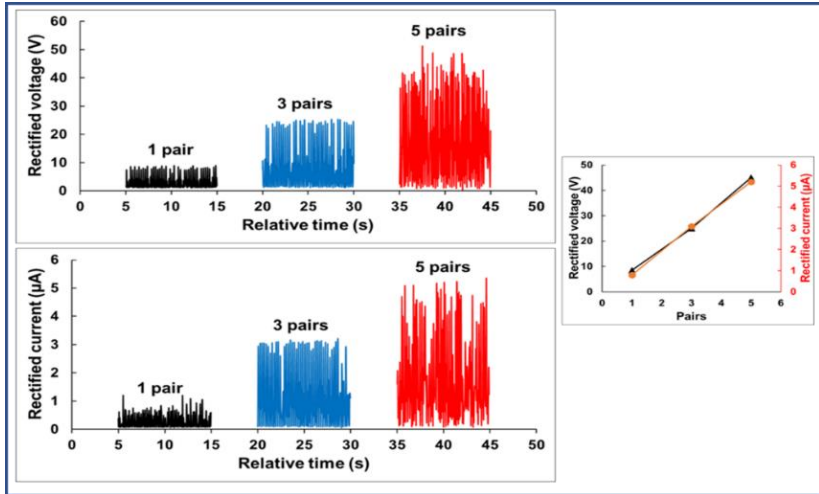


Figure 5. Conducting electrical measurements on single-layer, ternary-layer, and five-layer TEGs at a wind speed of 5 m/s

Also, electrical measurements were made at different values of wind speed on TEG with an origami structure made of 5 layers. It was found that with the increase of the wind speed, the contact-separation frequency of the materials and the value of the voltage and current intensity of the TEG also increased. Thus, at a wind speed of 2 m/s, the maximum values of TEG open circuit voltage and short circuit current were 10 V and 1 μ A, respectively. As shown in Figure 6, when the wind speed increased to 5 m/s, the maximum values of the voltage and current intensity of the origami structure TEG increased to 50 V and 5

μA , respectively.

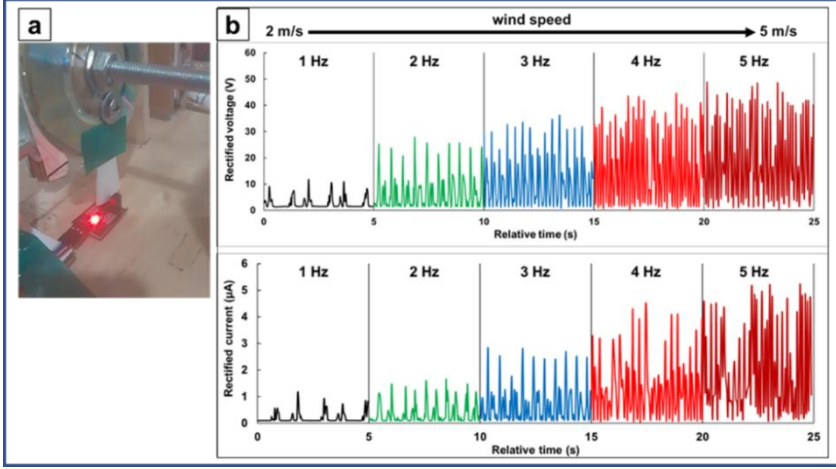


Figure 6. a) Determining the contact-separation frequency with the help of an infrared sensor; b) Determination of TEG voltage and current values with increasing wind speed

With the increase in wind speed, the frequency of contact-separation of materials with each other increases, which in turn ensures the accumulation of more static charges on the surface. Due to the kinetic energy generated during the rotation of the propeller due to the movement of the wind, the sliding arm of the crankshaft moves back and forth. As a result, the kinetic energy becomes the interaction energy of the materials. The mentioned energy conversions are indicated by the following expression (1):

$$E_{a.f} = \frac{1}{2} I_d^2 \omega_d^2 = \frac{1}{2} \left(\frac{1}{2} m_d R_d^2 \right) \omega_d^2 = W_{s.q} = F_{s.q} R. \quad (1)$$

As can be seen from expression (1), the force acting on the materials increases with the increase in the frequency of the sliding arm. Also, it was found that the impact force increases with the increase in the value of the mass and radius of the rotating disk of the crankshaft. However, increasing the size and mass of the disc is not effective for areas with low wind speeds. During the experiments, it was found that the output capacity of the generator increases due to the reduction of the distance between the layers of the origami-structured TEG due to the effect of

the thrust force.

As shown in Figure 7, a TEG-based accelerometer for human gait monitoring is computer-designed. The device consists of two parts: a stationary body and a moving mass. The stationary part is made of polyethylene terephthalate (PET) polymer material. The connection between these two parts is provided by a zigzag PET layer. The moving part responds to any mechanical movement or vibration in a sensitive manner. The PS layer placed on the moving part moves up and down according to the vibrations caused by the movement of people. On the stationary body, a nylon layer is placed, and it generates certain electrical signals when in contact with the PS layer.

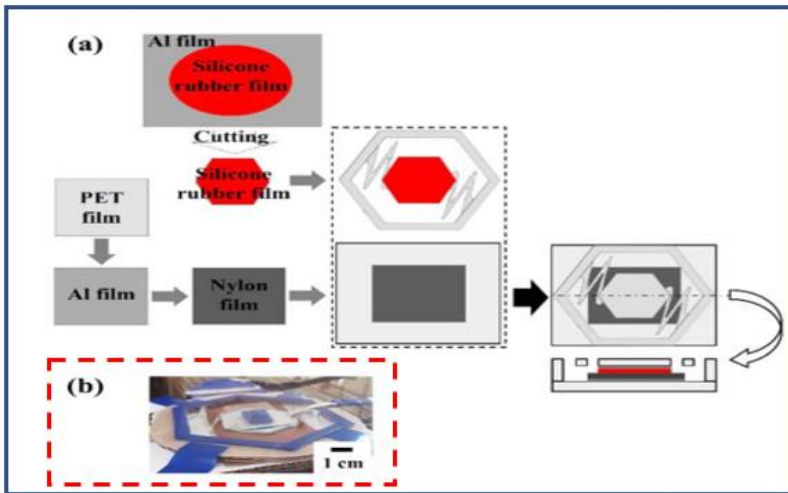


Figure 7. Accelerometer manufacturing scheme: a) TEG manufacturing stage; b) the final shape of the accelerometer

The final system is placed on the upper part of the front of the shoe. Thus, due to the mechanical vibrations generated during the movement of people, the nylon and PS samples are in mutual contact. This converts mechanical energy into electrical energy. Depending on the speed of movement of people, TEG generates different electrical signals. It is possible to perform gait monitoring by simulating the obtained electrical signals.

To conduct tests on the developed system, the TEG was installed on the crankshaft as shown in Figure 8. and driven by an electric motor.

During the engine movement, the crankshaft slide arm moved up and down to enable the TEG to operate. The study of the electrical signals generated by the accelerometer at different frequencies and the output parameters of the TEG were determined and the obtained results are shown in Figure 8.b. From the results, it is known that with the increase in the frequency of rotation of the electric motor, the frequency of contact-separation of the materials with each other increases. This leads to an increase in the output parameters of the TEG. The change of the voltage and current during the frequency change from 1 Hz to 5 Hz is shown in figure 8.b.

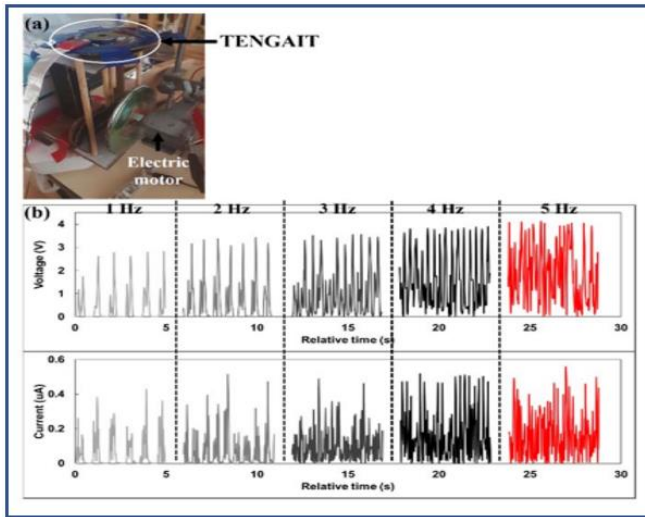


Figure 8. Electrical measurements of TEG: a) accelerometer; b) Variation of voltage and current intensity of TEG as a function of frequency

The maximum values of voltage and current intensity of TEG at 1 Hz value of rotation frequency were 2.5 V and 0.3 μA , respectively. As the frequency increased from 1 Hz to 5 Hz, the values of voltage and current intensity of TEG also increased to 4 V and 0.5 μA , respectively. Thus, this increases in the electrical parameters of the TEG depending on the frequency, while the amplitude value of the force does not change, was explained by the increase in the surface charge density when the materials interact with each other at a higher speed or with an impulse. That is, with an increase in the frequency of contact, the

exchange of charge carriers takes place more intensively in the area where the electron cloud of atoms overlaps.

The hot-pressing method was used to obtain the PVC polymer-based nanocomposite material. PVC was used as the matrix element, and TiO_2 nanoparticles were used as the filler element. Such a nanocomposite material is used as a material with negative triboelectric properties (Figure 9). That is, when the nylon and PVC/ TiO_2 nanocomposite layers are in contact with each other, according to the triboelectric series, the nylon surface is positively charged, and the PVC surface is negatively charged. As a result, by the principle of neutralization of the surface, charges in the opposite direction are transferred to the electrodes. This leads to the generation of opposite-direction displacement current in the electrodes.

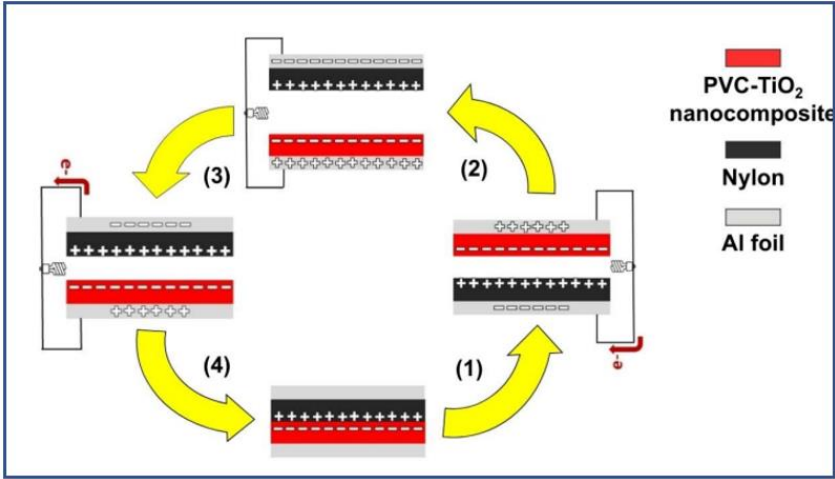


Figure 9. The electricity generation process of PVC/ TiO_2 nanocomposite-based TENGs

According to Maxwell's equation, the current generated during the operation of the TEG is shown in the form of expression (2) as follows:

$$J = \varepsilon_r \varepsilon_0 \frac{dE}{dt} + \frac{dP}{dt}. \quad (2)$$

As can be seen from the expression of the current density (2), the value of the current flowing in the circuit increases with the increase

of dielectric permeability. Since TiO_2 nanoparticles have high dielectric properties, as a result of their penetration into PVC, they form a polymer nanocomposite with higher dielectric permeability compared to ordinary PVC¹. To investigate how TiO_2 nanoparticles affect the output efficiency of the TENG, electrical measurements were performed on two TENG samples. The obtained results are presented in Figure 10. It is clear from the graphs that the open-circuit voltage and short-circuit current intensity of the pure PVC/nylon-based TENG are much lower than that of the nanocomposite material obtained using TiO_2 nanoparticles. Thus, the TENG based on PVC/ TiO_2 nanocomposite was found to have better output capability.

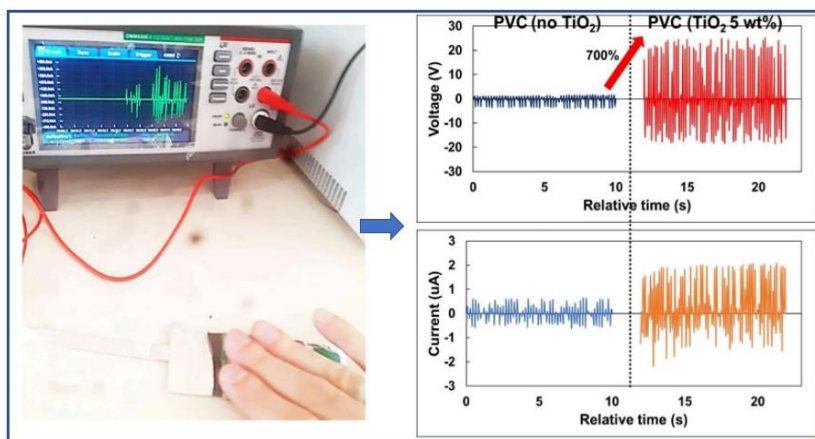


Figure 10. Conducting electrical measurements of TEG based on PVC and PVC/ TiO_2 nanocomposite

The value of voltage and current intensity of the TEG made based on ordinary PVC is set as 3 V and 0.5 μA , respectively. The maximum value of the voltage and current intensity of the 5% PVX/ TiO_2 nanocomposite-based TENG obtained during the study was 24 V and 2 μA , respectively. The 700% increase in the output efficiency of the nanocomposite-based TENG is explained by the fact that TiO_2 nanoparticles have a high dielectric constant. Thus, during the contact of materials, the number of polar dipoles formed inside the composite

¹Fan, F.R., Tian, Z.Q., Wang, Z.L. Flexible triboelectric generator // Nano Energy, – 2012. v. 1, № 2, – p. 328-334.

increases, which in turn has a positive effect on the amount of charge carriers formed on the surface.

It should be noted that since the dielectric constant of PVC is equal to 4, and this value is equal to 110 for TiO_2 , it was determined that nanocomposites with high dielectric properties are effective when used as a material with triboelectric properties in the preparation of TENG. Such materials are considered promising materials in the development of TENG and expanding its practical application.

In the fourth chapter, many research studies were conducted in the direction of determining the effect of micro- and nanostructures created on the surface of triboelectric materials by different methods on the output efficiency of TENG.

3D printing technology was used to investigate the effect of surface textures on TENG output parameters. So, mold samples of different sizes prepared based on this technology were used to create a structure on the surface of PS. The "Tinkercad" program was used to design the molds (for 2-2.5 hours). It was determined that the structures on the substrates obtained after the printing process have a conical structure. Thus, large, medium, small, and micro models, which differ from each other in terms of the diameters of the surface structure, were prepared through the program. The diameters of the cones on these samples were determined as 6.5 mm, 5 mm, 3.5 mm, and 0.5 mm, respectively.

To obtain PS thin films, 31.17 g of PS was first mixed with 1.56 g of cross-linking agent for 15 min. The mixture was then coated onto Al foil using the doctor knife coating method. It should be noted that the thickness of such samples varies from several hundred micrometers. Mold samples printed by a 3D printer are placed on the prepared samples. Certain mass objects are placed on the base for the molding process. The samples were dried at room temperature for one day. For the creation of TEG, nylon with a diameter of 43 μm was used as another material with triboelectric properties, and as a result, 4 TEGs were made based on them. Experiments were performed on such TEGs and the electrical measurements obtained were recorded by a Keithley multimeter. During the experiments, the results for the open-circuit voltage and short-circuit current of the TEG were obtained as shown in Figure 11.

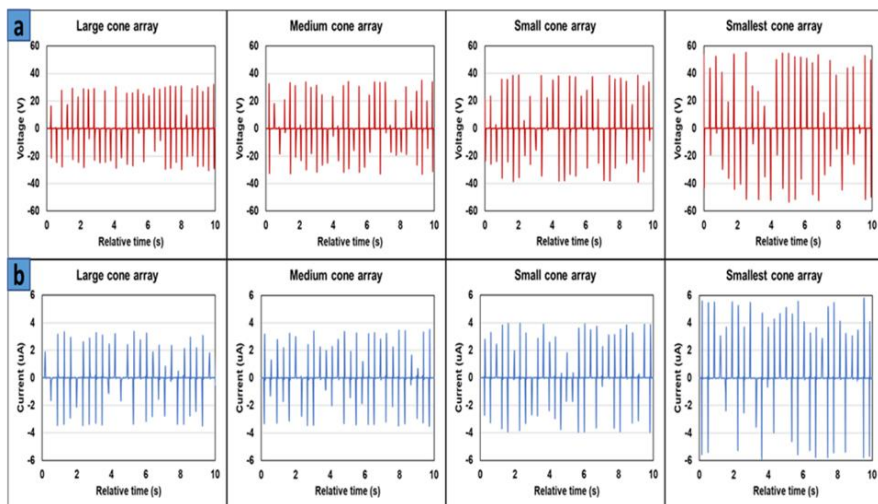


Figure 11. a) Time dependence graphs of the open circuit voltage of surface-structured PS and nylon-based TEGs; b) Graphs of the current intensity of TEGs as a function of time

As shown in Figure 11.a, the value of its open circuit voltage increased with the decrease in the size of the conical structures formed on the surface of the PS layer. Thus, the voltage increased from 25 V to 55 V and reached its maximum value. It should be noted that the greatest increase was registered during the transition to micro size. Also, the same trend was observed in the values of the current strength of TEG. Thus, it was observed that the value of the current increased from 2.8 μ A to 6 μ A with the decrease in the size of the cone-shaped structure formed on the surface of the PS.

This increase in the value of voltage and current intensity of TEG is explained by the presence of artificial textures created on the surface during the molding process. Thus, with the decrease in the size of these structures, the active contact area between PS and nylon increases. On the other hand, due to such rough structures on the surface, it is known that the layers interact with each other more at the atomic level. The fact that output performance of the TEG is the largest in the microarray is explained by the fact that in this case, the number of structures per unit area is greater than in the others. That is, in the case of micro-size, the mutual contact-friction transmission of PS and nylon layers at the

atomic level is maximum, which results in the separation of more static charges on the surfaces. In conclusion, it can be noted that it is possible to control the output efficiency of TEG by artificially creating structures on the surface and changing their dimensions.

Nylon is a polymeric material with a fibrous or mesh structure. To investigate the influence of the surface structure of the nylon material on the output parameters of the TEG, 10, 15, and 20 denier nylons, which differ in the size of the inner fiber diameters, were used. Denier is a property that varies depending on the type of fiber formed in the internal structure of the material, and its abbreviation is DEN. In materials science, it is the expression of the mass of fiber in a 9000 m long thread in grams. In current standards, its quantitative share is estimated at 0.05 g per 450 m. Thus, DEN is a way of expressing the size of the diameter of the fiber.

It is possible to determine the value of the diameters of nylon fibers with the help of expression (3). Thus, we can calculate the dimensions of the fibers in the nylon layers used in the research work. It should be noted that the diameters of the fibers calculated by this expression are in micrometers. Different constants are added to perform calculations in other systems of units.

$$d_{Nylon} = 11.89\sqrt{DEN/\rho}. \quad (3)$$

Here, d is the diameter of nylon fibers expressed in μm , DEN is the number of deniers, and ρ is the density of fibers expressed in g/ml. The calculated values for 10, 15 and 20 DEN using equation (3) are shown in table 1. Thus, for 10, 15, and 20 DEN nylon, these dimensions were 35 μm , 44 μm , and 51 μm , respectively.

Table 1. Values of diameters of nylon fibers

Nylon	ρ – fiber density (gr/ml)	d –diameter of fibers (μm)	d – diameter of fibers (mil)
1 DEN	1.14	11.13	0.44
3 DEN	1.14	19.29	0.76
6 DEN	1.14	27.28	1.07
10 DEN	1.14	35.22	1.38
15 DEN	1.14	43.57	1.7
20 DEN	1.14	50.81	1.96

To investigate the effect of nylon fiber sizes on TEG output efficiency, 3 TEG samples were fabricated using PS as another triboelectric material. To prepare PS samples, 12.50 g of PS was mixed with 1.3 ml of cross-linking agent for 15 min, and Al was used as an electrode by drop casting method.

Electrical measurements of TEGs based on different micro-sized nylon and PS layers were carried out using a Keithley RMM6500 multimeter. It is clear from the values of voltage and current intensity of TEG that the output parameters of TEG increase with the reduction of the sizes of nylon fibers. Thus, for the case of nylon fibers with a diameter of 51 μm , the open circuit voltage and short circuit current of TEG were determined to be 63 V and 6.3 μA , respectively. While the diameter of the nylon fibers was 44 μm , the voltage and current values of the TEG increased slightly and these values were 75 V and 8 μA , respectively. Finally, when the diameter of the nylon fibers is 35 μm , the value of the voltage and current intensity of the TEG is determined to be 85 V and 8.5 μA , respectively. Summarizing all this, the final result can be said that in TENGs made based on fibrous nylon layers, the output power of the TENG increases up to 140% by reducing the diameter of the fibers from 51 μm to 35 μm . This is because the frictional transmissions falling on a single surface are in the same direction.

Dielectric properties of nanocomposites obtained using nylon layers, TiO_2 and Fe_3O_4 nanoparticles, and output parameters of TENGs made based on such layers were studied. Also, surface morphology, structure analysis, element analysis, and dielectric measurements of the triboelectric layers obtained by different methods were carried out.

Figure 12 shows the results of the X-ray structural analysis of nanocomposites obtained by spraying 5 and 15 times 1% TiO_2 nano-particles on the surface of the nylon layer and immersing them in its solution. The image shows the diffraction patterns of ordinary nylon (Figure 12. a), nylon layers dipped in 1% TiO_2 solution (Figure 12. b), sprayed on the surface 5 times (Figure 12. c) and 15 times (Figure 12.d).

It is related to the α_1 ($2\theta = 20.4^\circ$) peak of the diffraction peaks reflected in RD and corresponds to the distance between the hydrogen-bonded chains. The peak corresponding to α_2 ($2\theta = 23^\circ$) was shown to be related to the separation of hydrogen-bonded layers. The peaks belonging to the β phase are observed at $2\theta = \sim 12^\circ$ and 19° , and the peaks

belonging to the γ phase of pure nylon 6,6 nanofibers are observed at $2\theta = 13^\circ$ (γ_1) and $2\theta = 22^\circ$ (γ_2). The $2\theta = 21^\circ$ and 23° peaks observed in the spectrum are the peaks belonging to the α phase of pure nylon 6. These main peaks are observed in both pure nylon and other samples. A diffraction peak at 28.79° was also observed in the diffraction pattern of pure nylon given in Figure 11.a.

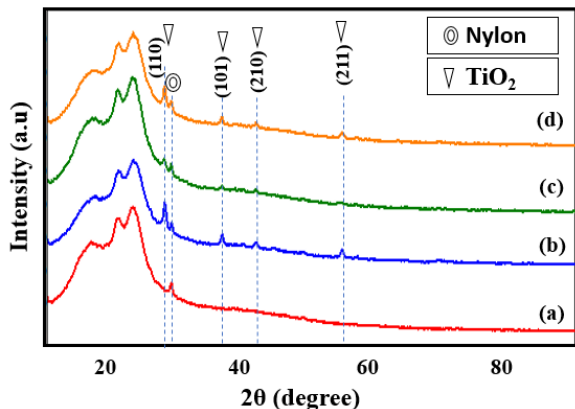


Figure 12. XRD pattern of nylon/TiO₂ nanocomposites: pure nylon (a), immersed in 1% TiO₂ solution (b), sprayed 5 times (c) and 15 times (d)

This is also associated with a characteristic peak belonging to pure nylon. The diffraction peaks at $2\theta = 27.44^\circ$, 36.44° , 44.07° and 54.61° observed in Fig. 11.b, c, and d are (110), (101), (210) and (211) according to the Miller indices. These peaks correspond to the pattern of card JCPDS No: 89-4920, confirming the formation of TiO₂ nanoparticles in the tetragonal phase. So, the distance between the d-atomic layers corresponding to these peaks is equal to 3.2, 2.4, 2.1, and 1.6, respectively. Based on the obtained diffraction patterns, the sizes of TiO₂ nanoparticles were calculated for all three samples using the Debye-Scherrer formula².

Thus, the sizes of nanoparticles for the immersed, 5-, and 15-times sprayed samples were 10.42 nm, 10.12 nm, and 10.38 nm, respectively. The size of nanoparticles for the immersed sample is larger

²Holzwarth, U., Gibson, N. The Scherrer equation versus the 'Debye-Scherrer equation' // Nature nanotechnology, – 2011. v. 6, № 9, – p. 534-534.

compared to the other samples. Because each of the smaller and larger nanoparticles present in the solution is more easily deposited on the surface of the nylon layer. During 5 times of spraying, smaller nanoparticles were formed on the surface of the nylon layer, and during 15 times of spraying, the nanoparticles agglomerated and formed relatively larger nanoparticles.

Surface morphology of conventional nylon (Figure 13. a), 5 and 15 times sprayed 1% nylon/TiO₂ nanocomposites and dipped nylon layers (Figure 13. b, c,d, and e) were studied by SEM and Figure 13- is also shown. From the SEM image, it is clear that nylon is a polymer with a network structure consisting of closely connected fibers.

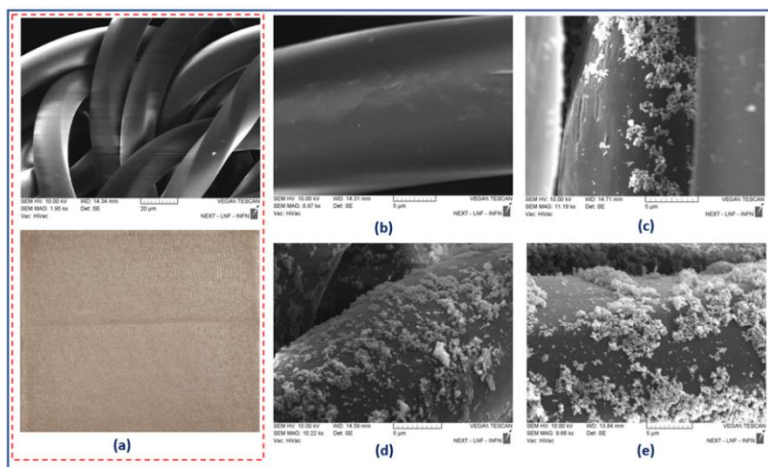


Figure 13. SEM images of nylon thin films. a) pure nylon and 1.95 kx; b) magnification at 8.97 kx of pure nylon; c) nylon thin film sprayed with 1% TiO₂ solution 5 times and 11.19 kx magnification; d) nylon thin film sprayed with 1% TiO₂ solution 15 times and 10.22 kx magnification; e) Nylon thin film immersed in 1% TiO₂ solution and 9.66 kx magnification

With the increasing amount of 1% TiO₂ solution injected on the surface of nylon films, in other words, TiO₂ nanoparticles were distributed in dispersed form on the surface of nylon fibers 5 times, 15 times spray, and dipped case. Since the size of nanoparticles is very small, it is impossible to determine their size. It is seen from the pictures that with the increase in the number of sprayings, the density of TiO₂ distributed in the fibers of the nylon layer increased, and more spherical

particles were formed.

Dielectric measurements of nylon/TiO₂ nanocomposites were performed and the results are shown in Figure 14. From the analysis of the results, it was found that the dielectric permeability of nanocomposite materials deposited on nylon increases compared to ordinary nylon. This is due to the high value of the dielectric constant of TiO₂ nanoparticles compared to the usual nylon layer. This, in turn, affects the arrangement of dipoles of atoms and molecules. From the results, it was determined that the dielectric permeability gradually increased with the increase in the amount of TiO₂ within the nanocomposite.

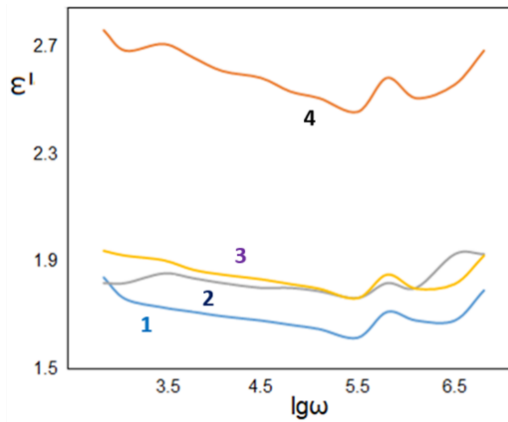


Figure 14. Investigation of dielectric measurements of nylon/TiO₂ nanocomposite materials: 1) pure nylon; 2) 5 times sprayed with 1% TiO₂; 3) nylon thin film sprayed with TiO₂ 15 times and 4) dipped in 1% TiO₂ solution

Figure 15. a and b show the changes in the open circuit voltage and short circuit current values of the nanocomposite-based TENG. It is clear from the diagrams that with the increase in the amount of TiO₂ nanoparticles, the maximum values of the voltage and current of the TENG increased in a certain range in direct proportion. According to the capacitor model of the TENG, this increase is explained by the increase in the dielectric constant value as shown in Figure 14. In the next increase in the amount of TiO₂, the decrease in the voltage and current intensity values of the TENG was explained by the fact that the triboelectric materials are less in contact with each other.

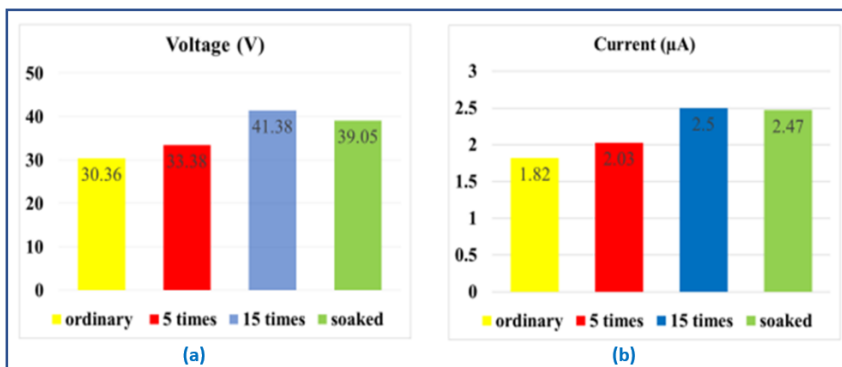


Figure 15. Variation of output performance of nylon/TiO₂ nanocomposite-based TENG depending on the number of sprays: a) voltage; b) current intensity

It is clear from the electrical measurements that the output parameters of nanocomposite-based TENGs are higher than those of conventional nylon. This increase was attributed to the formation of polar charges on the surface. Thus, nylon/TiO₂ nanocomposite materials were found to be effective as triboelectric materials in TENGs. By changing the amount of filler, the efficiency of the TENG can be increased by determining the optimal parameters.

To investigate the effect of magnetic nanoparticles on the output parameters of TENGs, 1% Nylon/Fe₃O₄ nanocomposite materials were prepared using spray coating technology. The output performance of such nanocomposite-based TENGs obtained was compared with the output performance of conventional nylon-based TENG. Thus, 2 samples were obtained by spraying 1% Fe₃O₄-ethanol solution on the surface of nylon 5 times and 15 times, as well as samples dipped in this solution. At the same time, the influence of the nanocomposite-based TENG on the output parameters was investigated due to the changes in the distribution of Fe₃O₄ nanoparticles on the surface of the nylon with the application of an external magnetic field after immersion in the solution. Figure 15 shows the RD images of 1% Nylon/Fe₃O₄ nanocomposite material.

Figure 16.a.1 shows the RD plots for pure nylon. The 2θ=21° and 23° peaks are diffraction peaks of pure nylon 6 belonging to the α phase. Figure 16.a.2 shows the RD view of pure cardboard, and Figure 16.a.3

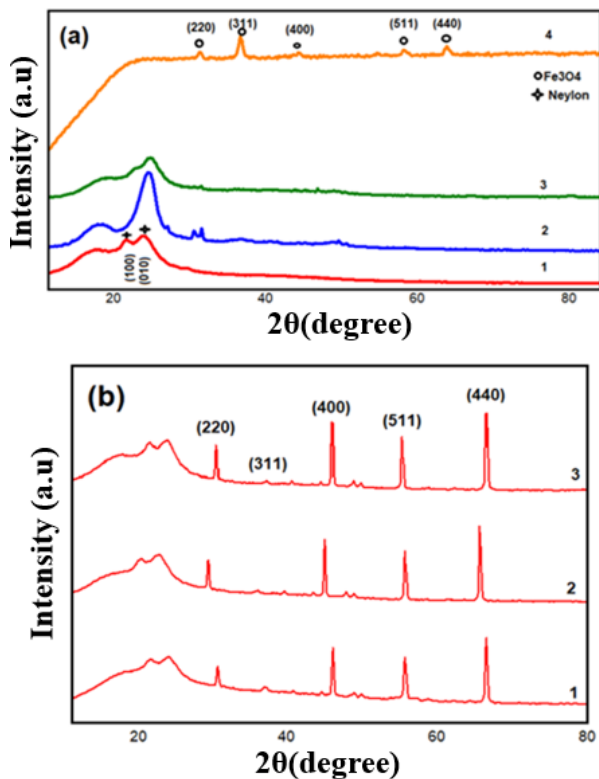


Figure 16. XRD pattern of nylon/ Fe_3O_4 nanocomposites: a) pure nylon (1), cardboard (2), nylon/cardboard (3), Fe_3O_4 nanoparticles (4); b) Sprayed and immersed in 1% Fe_3O_4 solution 5 times (1), 15 times (2) and immersed (3)

shows the RD view of nylon/cardboard. Here, the broad diffraction pattern observed in the range of $\sim 20\text{-}30^\circ$ is attributed to cardboard and nylon, respectively. Figure 16.a.4 shows the RD picture related to Fe_3O_4 nanoparticles. The observed peaks at $2\theta=31^\circ, 35^\circ, 43^\circ, 58^\circ$, and 63° correspond to Miller indices (220), (311), (400), (511), and (440), respectively. From comparison with the literature, it was determined that the formed nanoparticles correspond to the JCPDS card 00-003-0863 and cubic reverse spinel structure³.

³Zhou, X. Fabrication of cluster/shell $\text{Fe}_3\text{O}_4/\text{Au}$ nanoparticles and application in protein detection via a SERS method / X.Zhou, W.Xu, Y.Wang [et al.] // The Journal of Physical Chemistry C, – 2010. v. 114, № 46, – p. 19607-19613.

Figure 16.b.1, 2, and 3 show the RD view of the composite layers obtained by spraying 1% Fe_3O_4 solution 5 times and 15 times on the cardboard/nylon layer and immersing the cardboard/nylon layer, respectively. Here too, it was determined that the diffraction pattern of the obtained Fe_3O_4 nanoparticles corresponds to the cubic reverse spinel structure. The characteristic peaks attributed to Fe_3O_4 are manifested by the increase in the intensity of the characteristic peaks associated with the increase in the number of sprayings and the increase in the amount of Fe_3O_4 nanoparticles in the fibers of the nylon layer as a result of immersion. According to the Debye-Scherrer formula, the size of the nanoparticles formed on the surface of the nylon layer after spraying 1% Fe_3O_4 nanoparticles 5 times was 38.51 nm, the size of the nanoparticles formed as a result of spraying 15 times was 38.17 nm, and the nanoparticles formed on the surface of the nylon obtained after immersing the nylon layer in the solution its size was determined to be 29.63 nm. It can be concluded from this that as a result of spraying 5 times, only large nanoparticles adhered to the surface of the nylon layer, and as a result of spraying 15 times, relatively small particles were also formed. However, as a result of immersing the nylon layer in the solution, both large nanoparticles and small nanoparticles were formed on its surface.

To determine the distribution of Fe_3O_4 nanoparticles on the surface of a nylon thin film, SEM images of the nanocomposite samples obtained by the deposition of Fe_3O_4 nanoparticles on the surface of pure nylon were analyzed and shown in Figure 16. Based on obtained SEM images, it can be seen that pure nylon consists of a large number of fibers. In the SEM images of nanoparticle deposited samples, it is seen that Fe_3O_4 nanoparticles are formed on the surface of nylon fibers (Figure 16. a). The coating of these nanoparticles on the nylon surface occurs due to Van der Waals forces. Thus, Figure 16. b shows the SEM image of the nanocomposite sprayed 5 times on the surface of nylon. As can be seen from the SEM images, the amount of Fe_3O_4 nanoparticles is relatively small, so the nylon surface is not completely covered. Figure 16. c, d, and e show the SEM images of the nanocomposite layers obtained by spraying 15 times, immersion in the solution, and applying an external constant magnetic field after immersion, respectively. It is clear from the images that increasing the amount of Fe_3O_4

nanoparticles (dip or spray number) resulted in greater surface coverage of the nylon fibers. Also, with the increase in the amount of nanoparticles, it was observed that the nanoparticles formed on the surface of nylon fibers agglomerate and form larger particles. To determine the distribution of Fe_3O_4 nanoparticles on the surface of nylon, the SEM images of the nanocomposite samples obtained from the precipitation of Fe_3O_4 nanoparticles on the surface of ordinary nylon were analyzed and shown in Figure 17. If we look at the obtained SEM images, it can be seen that ordinary nylon consists of a large number of fibers.

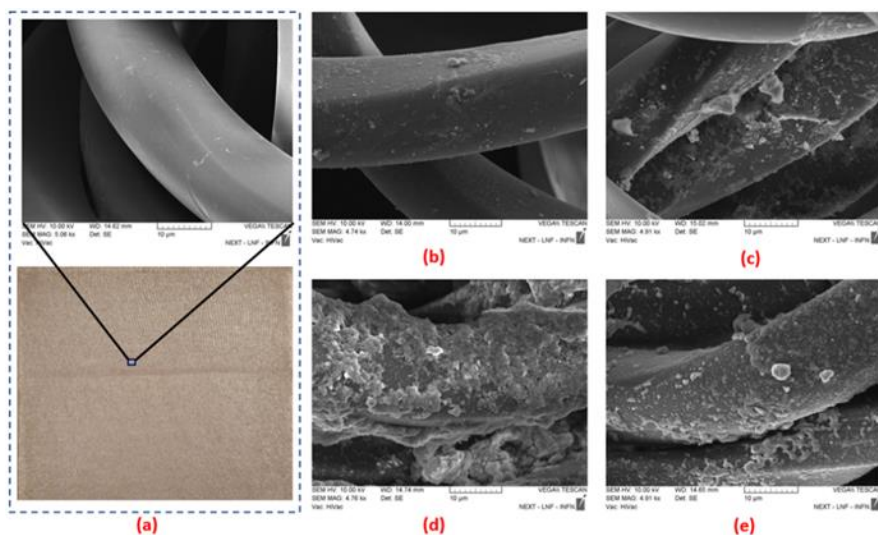


Figure 17. At different magnifications a) pure nylon; b) 5 times; c) 15 times spraying; d) immersed; e) SEM images of nylon nanocomposite thin film with applied magnetic field after immersion

In the SEM images of nanoparticle deposited samples, it is seen that Fe_3O_4 nanoparticles are formed on the surface of nylon fibers (figure 17. a). The coating of these nanoparticles on the nylon surface occurs due to Van der Waals forces. Thus, in picture 17. b, the SEM image of the nanocomposite sprayed 5 times on the surface of nylon is given. As can be seen from the SEM images, the amount of Fe_3O_4 nanoparticles is relatively small, so the nylon surface is not completely covered. Figure 17.c,d, and e show the SEM images of the nanocomposite layers obtained by spraying 15 times, immersion in the solution, and

applying an external constant magnetic field after immersion, respectively. It is clear from the images that with the increase in the amount of Fe_3O_4 nanoparticles (the number of dipping or spraying), more coverage of the surface of the nylon fibers occurred. Also, with the increase in the amount of nanoparticles, it was observed that the nanoparticles formed on the surface of nylon fibers agglomerate and form larger particles. Using nanocomposite materials formed on the surface of nylon sheets, 4 TENG prototypes were additionally prepared, and their output parameters (open circuit voltage, short circuit current intensity) were measured. From the obtained results, it was determined that with the increase of Fe_3O_4 nanoparticles on the surface of the nylon layer, the output parameters of the TENG first increase and then remain unchanged. In the case of a composite material formed under a magnetic field, this value is higher. This is associated with the orientation of the magnetic domains in the field direction.

RESULTS

1. The efficiency of TENGs obtained based on nylon/ TiO_2 nanocomposite materials increases with the initial increase in the amount of nanoparticles, the generation of more polar charges on the surface, the decrease in the output parameters of the TENG with the subsequent increase in the amount of TiO_2 is related to the uneven distribution of nanoparticles.

2. In TENGs based on fibrous nylon layers, the output power increases by 140% by reducing the fiber diameter from 51 μm to 35 μm , which is due to the unidirectionality of the frictional transmissions per unit area.

3. For the first time, it was determined that the increase in output power in the TENG made based on nylon/ Fe_3O_4 nanocomposite materials is due to the additional electric field generated during contact electrification in Fe_3O_4 .

4. It was determined that the conical micro-textures formed on the surface of the PS films increase the active contact area of the materials and ultimately the output power of the TENG.

5. The output power of the TENG made by different methods can be increased by forming nanoclusters in volume and artificial textures

on the surface, along with the physical parameters of the contact materials (dielectric permeability, magnetic susceptibility).

6. The prepared origami-structured TEGs can be used for the safe generation of electrical energy from various types of mechanical energy sources, as a power source for portable electronic devices (light-emitting diode-based irradiators, bio- and thermosensors, pressure recorders, etc.).

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1. Ağayev, Ş.M., J.Kim, Güləhmədov, O.G., Triboelektrik generatorlar və onların tətbiq sahələri // Azərbaycan xalqının Ümummilli lideri Heydər Əliyevin anadan olmasının 98-ci ildönümünə həsr olunmuş “Gələcəyin alimləri” tələbələrin VI Respublika elmi konfransı, – Bakı: – 3 May, – 2021, – səh.114–115.

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