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Carbon/NiO Compositional Fibers

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Abstract

This article presents the results of the synthesis of carbon-NiO composite fibers. Fibers doped with NiO particles are of practical interest for applications in sensors, energy storage systems, photocatalysts, etc. Four-component initial fibers based on polyacrylonitrile (PAN), activated carbon (AC), coal tar pitch (CTP), and NiO particles were obtained. CTP was obtained by thermal treatment of coal tar, AC by carbonization of apricot kernels, NiO by solution combustion synthesis. PAN, CTP, and AC are a source of carbon, but each of them plays a specific role. PAN is the basis of carbon fibers and a fiber-forming material, CTP is a technogenic waste added to replace polymer particles, AC is an additive that could increase the carbon content and the porosity of the final fibers. The fibers were obtained using the electrospinning method, which makes it possible to use complex suspensions and obtain fibers of various diameters. PAN:CTP:AC:NiO fibers were obtained. Next, the processes of stabilization and carbonization of the fibers were carried out. The fibers at each stage were examined by scanning electron microscopy and EDAX. The result of the synthesis was carbon/NiO fibers with a diameter of 100–300 nm. The resulting fibers are promising for practical applications due to the one-dimensional structure of the fibers and better adhesion between the fiber and NiO particles.

1. Introduction

Composite materials consist of two or more components with different physical and chemical properties. The composite consists of a matrix and one or more fillers. By changing the composition, structure, ratio of matrix and filler, it is possible to design composites with a given set of characteristics. The use of composites removes the material load and increases the performance and service life of the material or product.

The development of nanotechnology has led to the creation of a variety of nanocomposites that combine the properties of not only their constituent materials, but also demonstrate completely new properties due to quantum-size effects [1].

Inorganic and organic compounds can serve as a matrix for designing nanocomposites: polymers, metals, ceramics, carbon, etc. When creating nanocomposites, not only the composition is important, but also the final shape of the material. Thus, the composite may have a zero-, one-, two-, or three-dimensional shape.

This article presents the results of designing nanocomposite fibers from carbon and nickel oxide particles. The choice of this composite is due to a wide range of applications of NiO-based composites for various practical applications, including energy storage systems, sensors, photocatalysis,

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etc. And giving a one-dimensional structure to the composite gives it interesting new properties.

1.1. NiO/C composites for energy storage systems

The current energy storage methods have certain shortcomings, so there is an urgent need to design and develop new systems to meet the requirements of modern energy and electronics technology. Recently, energy storage systems based on capacitors and accumulators have been attracting more and more attention. Efforts to improve the energy storage capacity of capacitors are conducted in two directions: to extend the operating voltage range with organic electrolytes and electrolytes based on ionic liquids, and to increase the capacity of electrode structures, firstly, by increasing the capacity of the double electric layer of carbon electrodes. Electrodes based on carbon materials are widespread and highly efficient. Also, in the last decade [2], electrodes based on composite carbon materials have been increasingly used because of their high operating voltage, high-energy capacity, and long lifetime. Carbon-metal oxide composites attract special attention for these purposes. In this case, carbon can have various forms from carbon nanotubes to activated carbon. The composition of the oxide also depends on the purpose of the composite. Composites based on nickel oxide are considered promising for use in energy storage systems, the choice of which is due to the ease of synthesis, stability, price, and environmental safety considerations. The table presents some successful applications of carbon-NiO composite materials in supercapacitors and Li-ion batteries.

In [8] the authors obtained a highly efficient anode for a Li-ion battery based on a one-dimensional PAN-based carbon fibers/NiO composite (CF/NiO). The authors also claim that the resulting CF/NiO composite, due to the combination of high theoretical specific capacity and high electrical conductivity of NiO (~780 $mA \cdot h \cdot g^{-1}$) and the robust mechanical properties of the carbon fiber, demonstrates excellent performance in the storage of lithium-ion batteries. It is noted that the material synthesized by the authors has a high reversible capacity of 648 $mA \cdot h \cdot g^{-1}$ after 150 cycles at a current density of 100 $mA \cdot h \cdot g^{-1}$, which is approximately 2.5 times higher than that of pure CF (~ 260 $mA \cdot h \cdot g^{-1}$). The excellent electrochemical performance can be attributed to the synergistic effect between the one-dimensional structural carbon fibers and NiO, which makes the electrochemical reaction reversible.

The main parameter for efficient energy storage is needed in the electrode material to develop a porous structure with high adsorption characteristics. For instance, in research work [9] a method of obtaining carbon sorbents from natural oxidized coal of Barzas deposit is presented. The authors show that sorbents obtained from Barzas natural oxidized activated carbon by potassium hydroxide have a developed porous structure and high adsorption characteristics. In addition, in this work, these parameters are confirmed experimentally and demonstrate the wide applicability of AC as electrode material for energy storage systems. Composites are based on activated carbon obtained from biological waste such as rice husks [10] and walnut shells [11] etc.

In [12], activated carbon was derived from rice husk and further modified by nickel hydroxide. The modified activated carbon was obtained by a two-step chemical procedure and showed a very high specific surface area of 3292 $m^2 \cdot g^{-1}$. The author carried out the modification with nickel hydroxide $Ni(OH)_2$ content of about 9 wt.%. The authors measured the specific capacitance of supercapacitor electrodes based on this activated carbon and it was 236 $F \cdot g^{-1}$, whereas modified electrodes made

Table

NiO/carbon composite materials for energy storage systems and its electrochemical parameters

Composite materials	Type of storage energy	Specific capacity $F \cdot g^{-1}$	Specific surface area $m^2 \cdot g^{-1}$	Electrolyte	Ref.
NiO/AC	Supercapacitor	214.48	2034.38	1 M KOH	[3]
NiO/carbon fibers	Li-ion battery, pseudocapacitor	480	N/A	1 M LiPF6	[4]
NiO/graphene	Supercapacitor	632	89.5	2 M KOH	[5]
NiO/graphene oxide	Supercapacitor	1093	47.5	6 M KOH	[6]
NiO/carbon nanotube	Li-ion battery	1765	39.64	1 M LiPF6	[7]

of this material showed specific capacitance up to 300 $F \cdot g^{-1}$. Chemical modification with nickel increases the specific capacitance of activated carbon by about 27%. These data prove directly that the nickel modification of activated carbon leads to better performance and has an improved electrode material.

The demand for NiO composite has increased dramatically due to the excellent physicochemical properties of the last 10 years for energy storage systems. To obtain a composite based on nickel oxide particles, the electrode component is mixed with nickel oxide.

In [13], the researchers obtain a NiO/AC composite in the form of granules and electrochemical characterization of the electrodes was carried out in three electrode configurations in which the electrolyte is an aqueous 1 M KOH solution. The authors found two charge storage mechanisms:

• a supercapacitor response involving two-layer capacitance and psedocapacitance for AC and reaching a specific capacitance up to 142 $F \cdot g^{-1}$ at 6 $mA \cdot g^{-1}$;

• a battery response due to reversible redox reactions of NiO and reaching a specific capacitance up to 19 $F \cdot g^{-1}$ at 7 $mA \cdot g^{-1}$.

AC are well known to be widely investigated as materials for supercapacitor electrodes, which have such advantages as low cost, large specific surface area and variously shaped porous structure [14]. However, most AC is inferior to metal oxides in terms of capacitance. Therefore, much attention has been paid to improving the electrochemical performance of AC, especially by introducing metals with the creation of composite materials. The most effective component for the improvement of these characteristics of composites is various metals and their oxides. It is a relevant development with a wide range of applications, including an energy storage system.

1.2. Other applications of NiO/C composites

Composite materials based on a carbon matrix modified with nickel oxide particles have a high potential for various applications. Thus, due to the unique electrochemical characteristics of NiO, composite materials based on it can be used in various gas sensitive systems. For example, Fan X. et al. [15] note the possibility of using a p-type composite based on NiO as a sensor for detecting gaseous acetone. Experimental results showed that the applied composite had an optimum sensor operating temperature in the range of 220 °C and sensitivity to acetone concentration of 100 bbp (parts per billion). Despite these comparatively high material sensitivity to the test gas, the sample has a long response and recovery time. Previously, A. Khalil et al. [16] described in detail the influence of various factors on the gas sensitive characteristics of a composite material based on carbon fibers modified with nickel oxide particles. It should be noted that the high sensitivity of nickel oxide to various gases is explained by the dependence of the sizes of nickel oxide crystallites. Thus, with a decrease in the size of crystallites, an improvement in gas-sensitive characteristics is noted. In addition to the size of nickel oxide crystallites, the specific surface area of the composite and the bond between particles play an important role. Therefore, the use of electrospinning for the manufacture of composite materials based on carbon matrices and nickel oxide particles is a relatively simple and effective method for obtaining gas-sensitive materials, however, the issues of optimizing the structure of composite fibers are relevant.

As noted earlier, nickel oxide has good electrochemical characteristics, due to which CF/NiO composite fibers can be used not only as a gas-sensitive material, but also as photocatalysts [17, 18].

This article presents the results of designing a one-dimensional nanocomposite in the form of carbon fibers and metal oxide particles. For this, initial 4-component fibers were obtained from polyacrylonitrile (PAN), coal tar pitch (CTP), activated carbon (AC) and NiO particles using the electrospinning method. The main components for creating initial fibers are PAN and NiO particles.

The role of each component:

PAN is the main matrix, fiber-forming material, and carbon source.

CTP – substitution of a part of polyacrylonitrile and carbon source.

AC is a source of carbon and an increase in the specific surface due to high porosity.

NiO is an active material distributed in a carbon matrix for various applications (gas sensors, energy storage systems, etc.).

Recycling coal tar is a great way to produce carbon fibers. However, obtaining carbon fibers from CTP using the electrospinning method is a difficult task [19]. On the other hand, the addition of CTP to PAN solution in the production of carbon fibers is feasible. The successful use of CTP to replace part of the polyacrylonitrile in the production of carbon fibers by electrospinning is shown in [20].

2. Experimental part

2.1. Materials

For the synthesis of composite fibers based on polyacrylonitrile with the addition of coal tar pitch, activated carbon, and nickel oxide, the following materials were used: polyacrylonitrile with a molecular weight of 150.000 $g \cdot mol^{-1}$ (DFL Minmet Refractories Corp.); coal tar (CT) obtained during the processing of coal from the Shubarkol deposit (Kazakhstan); dimethylformamide (DMF, $(CH_3)_2NC(O)H$, Sigma-Aldrich, 99.9%); nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$); urea (NH_2CONH_2).

2.2. Obtaining composite fibers PAN:CTP:AC:NiO

2.2.1. Synthesis of coal tar pitch by heat treatment

Synthesis of coal tar pitch was carried out by heat treatment of coal tar. The heat treatment was carried out in a tubular CVD furnace with a quartz tube 6 cm in diameter. The CTP was placed in a reactor and heated to a temperature of 400 °C at a heating rate of 5 $°C \cdot min^{-1}$ and kept at this temperature for 60 min at a constant supply of argon (99.993%) at a rate of 400 scm. Next, the furnace was cooled to room temperature also with a constant supply of argon, after lowering the temperature, the sample was removed from the furnace.

2.2.2. Synthesis of activated carbon by carbonization of apricot kernels

Synthesis of activated carbon was carried out according to the method described in detail in [21]. For this, crushed and peeled apricot kernels were processed in several stages: carbonization and chemical-thermal activation.

2.2.3. Synthesis of NiO particles by the solution combustion method

For the synthesis of nickel oxide particles by the solution combustion method, nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) was used as an oxidizer and urea (NH_2CONH_2) as a fuel. According to the stoichiometry, the reagents were dissolved in distilled water, then evaporated to a volume of 5–7 ml. After evaporation, the mixture was heated to 260 °C; after evaporation of the main volume of water and transition to a gel-like system, self-ignition of the solution was observed. After the end of the process, the particles are washed with water until normal pH to remove unreacted reagents.

2.2.4. Electrospinning process

After preparing all the necessary components, PAN:CTP:AC:NiO composite fibers were synthesized by electrospinning with a drum-type collector. As the main fiber-forming solution used 9 wt.% PAN/DMF solution. To do this, a portion of PAN was dissolved in DMF with constant stirring for 5 h at a stirring speed of 500 rpm at a temperature of 80 °C. After that, small portions of CTP, AC, and nickel oxide powder were added to the prepared PAN/DMF solution, the mass ratio of all components was 3:2:2:3 (PAN:CTP:AC:-NiO) excluding DMF. The resulting suspension was also continuously stirred for 5 h on a magnetic stirrer at a stirring speed of 500 rpm, at room temperature. Next, the suspension was placed in an ultrasonic bath for 1 h to evenly distribute the particles of nickel oxide and activated carbon in the suspension.

The resulting suspension PAN:CTP:AC:NiO was filled into a syringe with a volume of 5 ml. The electrospinning process was carried out at an ambient temperature of 20–25 °C and a humidity of 30–35%, the voltage was 15 kV, the feed rate was 1.0 $ml \cdot h^{-1}$, the distance between the needle and the collector was 18 cm, the surface of the collector was covered with aluminum foil.

2.2.5. Preparing of C/NiO fibers

The initial PAN:CTP:AC:NiO electrospun fibers were subjected to stabilization and carbonization processes to convert them into C/NiO fibers.

Stabilization of the initial PAN:CTP:AC:NiO fibers was carried out in the air in a tubular CVD furnace with a quartz tube 6 cm in diameter. The reactor was heated to a temperature of 260 °C at a heating rate of 3 $°C \cdot min^{-1}$. The stabilization time was 1 h; after the stabilization process, the heating of the reactor was stopped, and the sample was cooled to room temperature without removing from the reactor in an air atmosphere.

The stabilized fibers were carbonized in the same CVD oven. The quartz tube was preliminarily purged with argon to remove air from the reactor and exclude contact with oxygen. Argon (99.993%) was passed through for 5 min at a volume flow rate of 400 sccm, after which the reactor was heated to a temperature of 700 °C. The heating rate was 2 $°C \cdot min^{-1}$. The carbonization time was 1 h; after the end of the carbonization process, the heating of the reactor was stopped, and the sample

was cooled to room temperature without removing from the reactor in an argon atmosphere.

2.2.6. Characterization methods

The investigation of the structure, size and morphology of the obtained samples was carried out on a Quanta 200i 3D scanning electron microscope (FEI, USA) equipped with an energy dispersive X-ray analysis (EDAX) system with an accelerating voltage of 30 kV (National nanotechnology laboratory of open type (NNLOT), Al-Farabi Kazakh National University, Almaty, Kazakhstan).

For the investigation of coal tar pitches, an automated digital optical microscope Leica DM 600M (NNLOT) was used.

The Raman spectra of the samples were obtained using an NT-MDT NTegra Spectra (NNLOT) spectrometer. Raman spectroscopy was carried out under excitation by unpolarized radiation from a semiconductor diode laser at a wavelength λ_{exc} = 473 nm.

X-ray phase analysis was carried out on a Dron-4 diffractometer (Institute of Combustion Problems, Almaty, Kazakhstan).

3. Results and discussions

3.1. Physical and chemical properties of coal tar pitch

Coal tar was obtained during the processing of coal from the Shubarkol deposit. Coal tar is characterized by low density (1039–1061 kg/m^3), low content of insoluble substances in toluene and absence of substances insoluble in quinoline, high content of phenols and low content of naphthalene.

Morphological properties of CTP were carried out on an optical microscope and a scanning electron microscope. Optical microscopy is an effective method for studying pitches for identifying mesophase centers due to the polarization of incident light. Scanning electron microscopy allows studying the morphology and topography of the sample at high resolution. Previous work in this area [22, 23] showed that the optimal temperature for heat treatment of CT corresponds to a temperature of 400 °C. After heat treatment, coal tar passes from a viscous state to a condensed solid state. Optical microscopy images (Fig. 1a) showed that heat treatment at 400 °C leads to the removal of

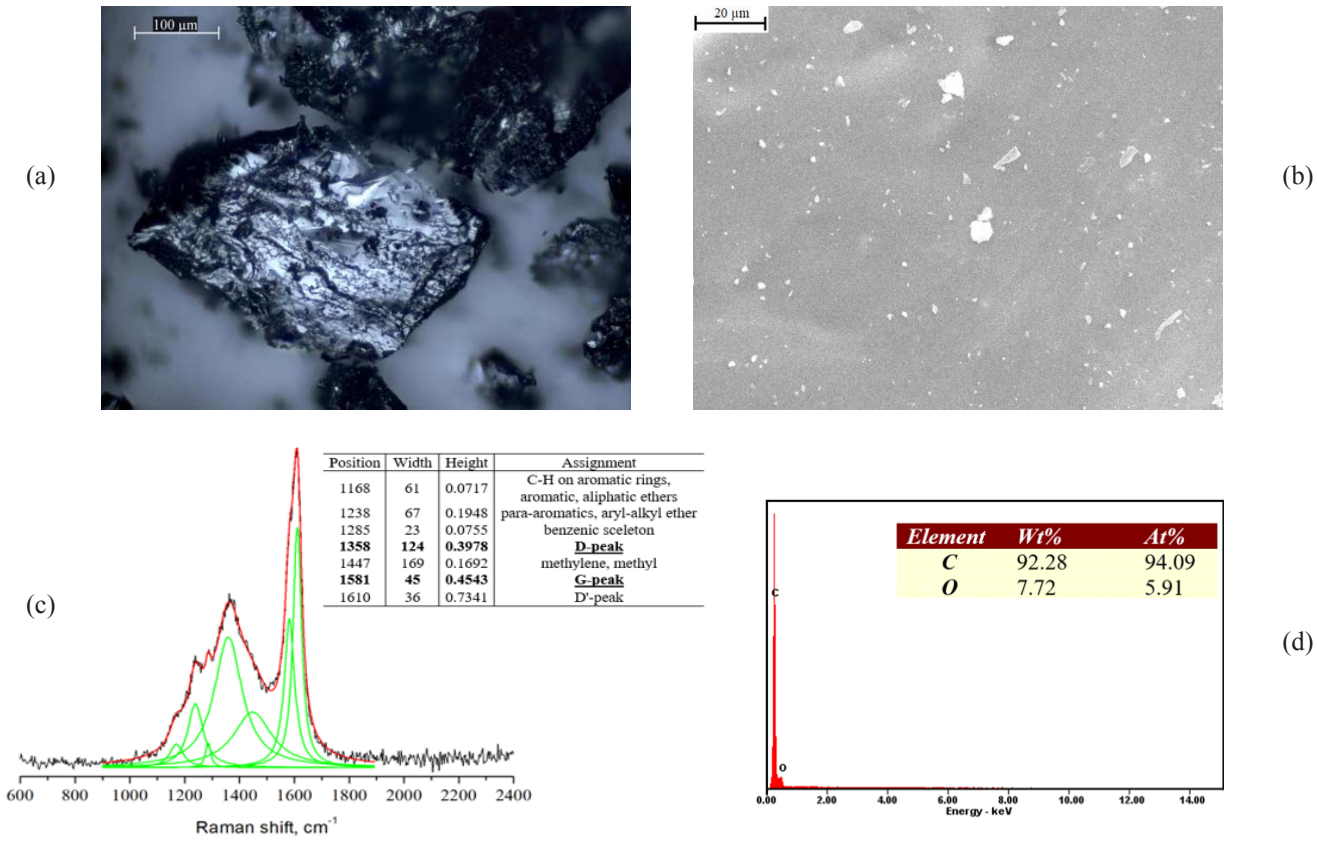


Fig. 1: (a) – optical microscopy image of coal tar pitch obtained after heat treatment at 400 °C; (b) – SEM image of coal tar pitch obtained after heat treatment at 400 °C; (c) – Raman shifts of coal tar pitch obtained after heat treatment at 400 °C; (d) – elemental analysis of coal tar pitch obtained after heat treatment at 400 °C.

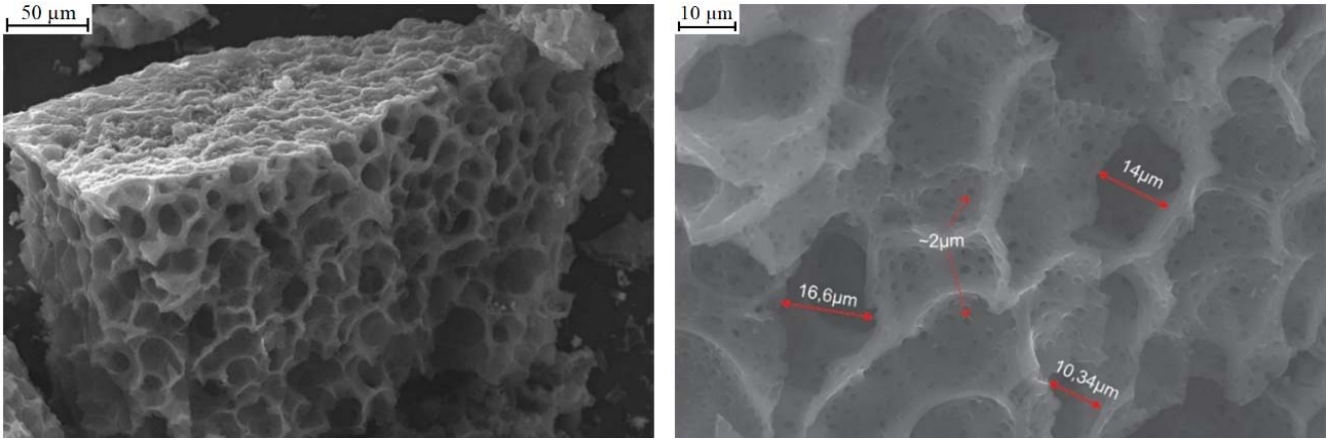


Fig. 2. SEM images of activated carbon obtained from apricot kernel.

most volatile components and an increase in the number of mesophase centers. Mesophase particle diameters range from 3 to 16 µm. It was also found that the CTP obtained at a temperature of 400 °C has a layered structure, which is associated with an increase in the degree of graphitization of the sample. The resulting coal tar pitch has a porous structure (Fig. 1b), which is due to the removal of low-boiling fractions in the form of vapors, which lead to the formation of loose, spongy material.

Raman spectroscopy makes it possible to estimate the degree of graphitization of CTP depending on the heat treatment temperature. The Raman spectrum of pure graphite has two main peaks: a D-peak at a wavelength of 1355 cm^{-1} and a G-peak at 1575–1582 cm^{-1}, due to carbon atoms in graphite structures with sp^2 hybridization. The investigated sample (Fig. 1c) shows a change in the intensities and frequencies of the G and D peaks and a shift of the D peak to the range of 1600–1610 cm^{-1}, which is explained by the transition from a disordered to a more ordered structure and the formation of nanocrystalline mesophase centers.

Elemental analysis of coal tar pitch was carried out by the EDAX method (Fig. 1d). Thermal treatment at 400 °C leads to the complete removal of sulfur from the composition of CTP. The content of carbon and oxygen in the composition of the pitch is 92.28 wt.% and 7.72 wt.%, respectively.

3.2. Physicochemical properties of activated carbon

Activated carbon from AK was investigated using scanning electron microscopy and Raman spectroscopy.

Microphotographs of activated carbon particles are shown in Fig. 2. Analysis of SEM images of activated carbon shows that the sample is a highly

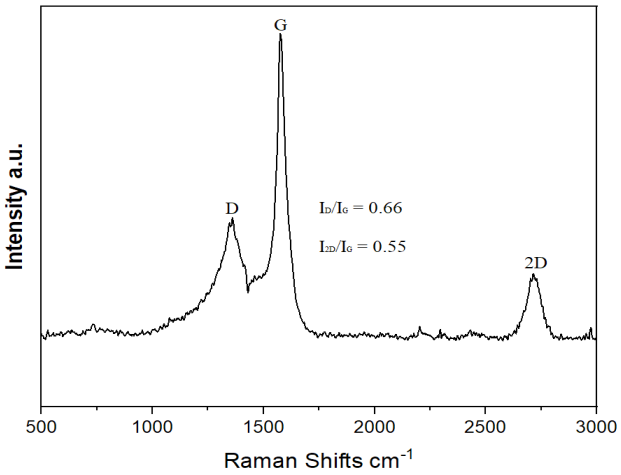


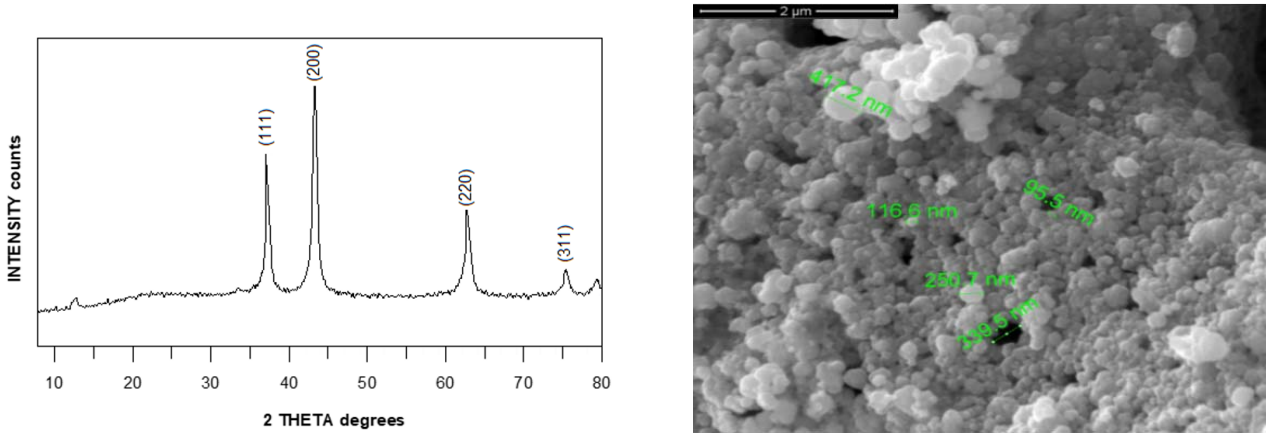
Fig. 3. Raman shifts of activated carbon obtained from apricot kernel.

structured material with the inclusion of macropores with a diameter of 2–20 µm. Figure 3 shows the Raman spectrum of activated carbon from AK. It is known that Raman spectroscopy is an informative method for studying graphene [24]. Analysis of the Raman spectra showed the presence of multilayer graphene structures ($I_{2D}/I_G = 0.55$).

The peaks of the Raman spectra in the 2D region indicate that the formed structure consists to a large extent of multilayer graphenes. All spectra have a D-peak, which indicates the presence of deformations in the crystal structure, as well as mechanical stresses.

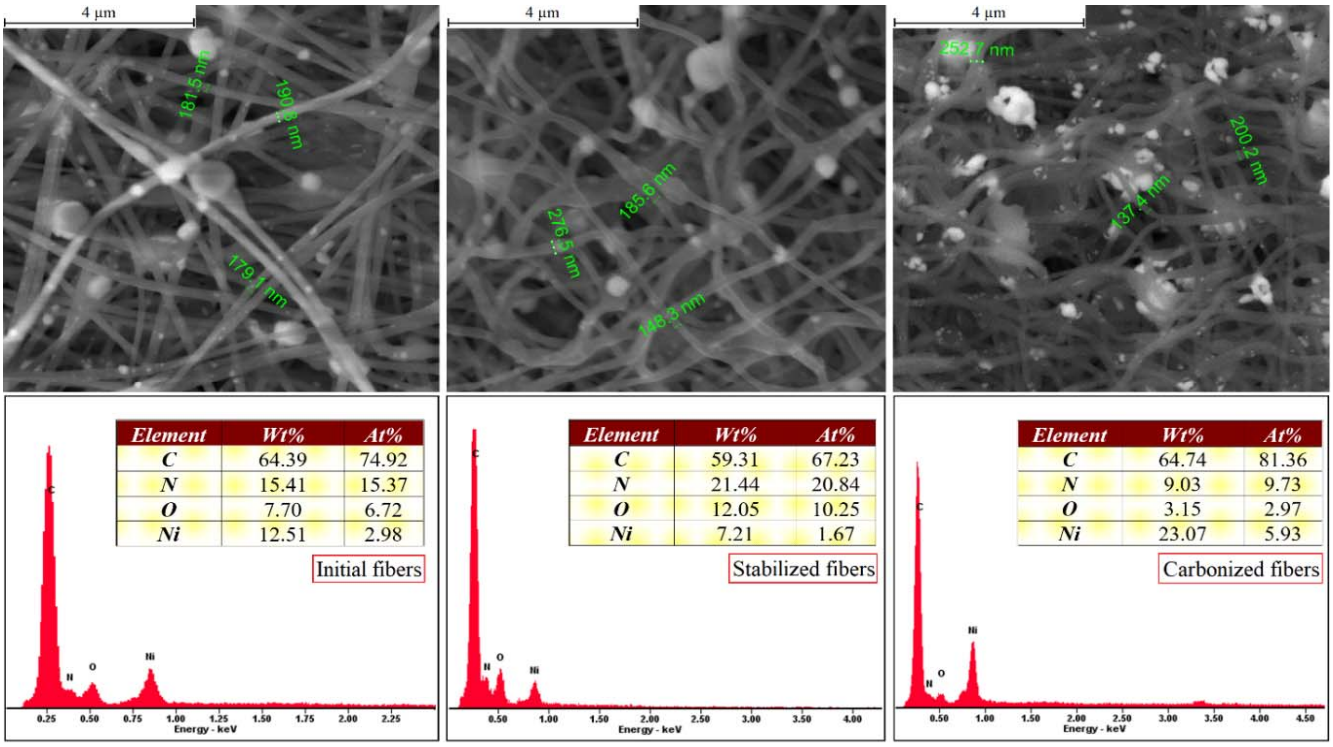
3.3. Physicochemical properties of NiO particles

Figure 4a presented an X-ray diffraction pattern of NiO powder. The synthesized nickel oxide is characterized by a nanocrystalline structure with diffraction peaks at 37.46°, 43.44°, 63.08° and 75.72°, which corresponds to the crystal planes of the nickel oxide phase (111), (200), (220) and (311).



(a) b

Fig. 4. (a) – XRD-patterns of nickel oxide obtained by solution combustion synthesis; (b) – SEM image of nickel oxide obtained by solution combustion synthesis.



(a) (b) (c)

Fig. 5. SEM images and EDAX analysis of (a) initial; (b) stabilized and (c) carbonized fibers.

As can be seen from the SEM micrographs (Fig. 4b), the sizes of nickel oxide particles range from 95.5 to 417.2 nm and have a spherical structure; the formation of agglomerates is also observed.

3.4. Physicochemical properties of C/NiO composite fibers

The composite fibers obtained by electrospinning were investigated by SEM, EDAX (Fig. 5) and Raman spectroscopy (Fig. 6). The initial PAN:CTP:AC:NiO fibers have a diameter of 100–200 nm. There are large amounts of crystalline inclusions of NiO particles. The particles are located on the surface of the fibers; NiO agglomerates are also present. As can be seen from the results of the analysis, the PAN:CTP:AC:NiO composite fibers change the process of stabilization and carbonization, and the diameter of the fibers increases and amounts to 100–300 nm. Crystalline inclusions of NiO are also presented in large quantities. After heat treatment, NiO particles have better adhesion to the fiber surface.

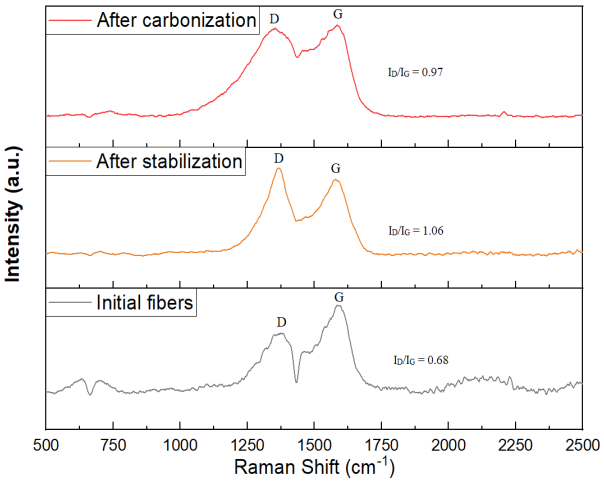


Fig. 6. Raman shifts of composite fibers (initial stabilized and carbonized).

As can be seen from the results of the EDAX analysis, the resulting C/NiO composite fibers consist of carbon, oxygen, nitrogen, and nickel. After stabilization, the carbon content decreases, and the oxygen content increases due to the addition of oxygen molecules from the air and further cyclization processes. After the carbonization process, the content of oxygen and nitrogen decreases due to the removal of volatile components, and the nickel content increases due to a general decrease in weight and, as a result, an increase in the mass fraction of nickel oxide.

4. Conclusion

A one-dimensional C/NiO composite material was obtained by electrospinning. For this, coal tar pitch, nickel oxide particles, and activated carbon were synthesized with further electrospinning of PAN:CTP:AC:NiO fibers and further stabilization and carbonization processes.

Coal tar pitch has been characterized as a layered material with a high content of mesophase particles ranging in size from 3 to 16 μm. The high carbon content and the presence of mesophases make CTP an acceptable material for producing fibers by electrospinning and can be an alternative for partial replacement of the fiber-forming polymer PAN in the electrospinning process. Also, the addition of activated carbon particles makes it possible to increase the total carbon content and increase the specific surface area due to the high porosity of AC particles.

It was also shown that NiO particles with particle sizes up to 500 nm can be synthesized by a simple and efficient solution combustion method, which was confirmed by the results of X-ray phase analysis and SEM.

The resulting coal tar pitch, activated carbon, and nickel oxide particles were used to obtain PAN:CTP:AC:NiO composite fibers by electrospinning. After that, the obtained fibers were subjected to heat treatment, because of which PAN:CTP:AC:NiO passed into C/NiO fibers. SEM showed that nickel oxide particles are located both on the surface and inside the fibers. In this way, carbon-based fibers with NiO particles were obtained. The potential application of the obtained composite fibers can be gas sensors, electrodes of pseudocapacitors, catalysts, etc.

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