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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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ИЗВЕСТИЯ

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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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B. T. Yermagambet[1], M. K. Kazankapova[1], A. T. Nauryzbaeva[1],
Z. A. Mansurov[2], G. T. Smagulova[2], B. B. Kaidar [2]

[1]LLP «Institute of coal chemistry and technology», Astana, Kazakhstan,
[2]RSE «Institute of Combustion Problems», Almaty, Kazakhstan.
E-mail: coaltech@bk.ru, maira_1986@mail.ru, asemai_2296@mail.ru, zmansurov@kaznu.kz,
smagulova.gaukhar@gmail.com, kaydar.bayan@gmail.com

PRODUCTION OF CARBON FIBERS BY ELECTROSPINING METHOD

Abstract. The technique of carbon fibers synthesis by the method of electrospinning from the coal tar pitch of the Shubarkol deposit in laboratory conditions is described, the value of the interelectrode voltage is 18-20 kV. The elemental composition was determined and the morphology of the surface of the investigated sample was studied, the type of carbon fiber modification was revealed. As a result of energy-dispersive X-ray spectroscopy and SEM microscopy, the chemical composition (C – 84.73%, O – 14.05%, Si – 0.49%, S – 0.73%) was determined and carbon fiber diameter constitutes from 2 to 9 μm. Raman spectroscopy showed the presence of D peaks (1367 cm^{-1}) and G (1595 cm^{-1}), which characterizes carbon materials, as well as a peak at 2900 cm^{-1}, which indicates the presence of CH bonds.

Key words: carbon nanofibre, stone-pitch, electric spinning, composite.

Introduction. Currently, in order to reduce dependence on imports or exports of oil and natural gas, most countries are keen to actively develop coal chemistry, namely actively develop modern technologies for deep processing of coal and create materials and products of a new generation on the basis of coals of different grades. It should be noted that the products obtained by deep processing at a cost several times superior to the raw materials. Now it is important to understand which methods and technologies in the field of deep coal processing will be prioritized and will have a positive impact on economic growth.

Among a wide class of nanomaterials, carbon fibers (CF) occupy a separate position, due to the uniqueness of their physicochemical properties and the prospects for practical application [1]. Carbon nanofibers (CNF) are a class of materials in which curved graphene layers or nanocones are composed in the form of a quasi-one-dimensional filament whose internal structure can be characterized by an angle α between the layers of graphene and the fiber axis [2]. CNF attracted great attention of scientists with their potential thermal, electrical, shielding and mechanical properties [3], as well as high tension force, low specific gravity, low coefficient of thermal expansion and chemical inertness. Due to their exceptional properties and low cost, they are now increasingly used in various materials such as composites production [4]. Other areas of nanofiber technology include engineering, wires, capacitors, transistors, diodes for information technology, systems for transport, conversion and storage of energy, such as batteries and fuel cells, and structural composites for aerospace structures.

A unique property of carbon fibers is also their relatively high electrical conductivity. Fibers can be used as heating elements, since their electrical conductivity is close to the electrical conductivity of nichrome [5, 6].

In the early 1970s, Japanese researchers Koyama and Endo [7] were able to produce carbon fibers with a diameter of 1 μm and a length of more than 1 mm by deposition from the gas phase. Later, in the early 1980s, Tibbets [8] in the USA and Benissad [9] in France continued to improve the process of obtaining carbon fibers by gas-phase deposition. In the US, more in-depth studies on the synthesis and properties of these materials were carried out for practical use by R. Terry K. Baker [10].

The first attempt to commercialize carbon fibers grown from the gas phase was undertaken by the Japanese company Nikosso in 1991 under the trademark Grasker [4], the same year Ijima published his famous article on the discovery of carbon nanotubes. Essentially, carbon nanofibers are produced by the same process as carbon fibers grown from the gas phase, only their diameter is generally less than 200 nm. At the present time, many companies around the world are actively involved in the commercialization of the production of carbon nanofibers and the introduction of new technical applications of these materials, the latest of which is a carbon nanofibre-porous composite that is used to eliminate oil spills.

The first developments in the production of carbon fibers from the pitch were carried out by Japanese researchers, who are still holding the first place in the world market for the production of carbon fibers. The technology for producing pitch-based carbon fiber includes several stages: preparation of the substrate, synthesis of the fiber by spinning from the melt, stabilization in an oxidizing atmosphere, carbonization in an inert atmosphere, graphitization at an elevated temperature. Activation of carbon fibers produces materials with a large active surface (from 300 to 1000 m^2/g), which are sorbents. The application of catalysts on the fiber allows the development of catalytic systems with a developed surface. Due to the low density (1.7 to 1.9 g/m^3), the specific value of mechanical properties, carbon fibers exceed all known heat-resistant fibrous materials [11, 12]. Special fibers from phenolic resins, lignin, coal and petroleum pitches can be used to produce CNF. In this respect, coal precursors are of particular interest, since they are economically viable, and also present in large quantities in various coal deposits.

Coal pitch - the remnant from the distillation of coal tar after pyrolysis. It is a hard, sometimes viscous mass of black. High-molecular aromatic hydrocarbons, higher phenols and organic bases predominate in the composition of coal tar pitch. The insoluble part includes free carbon (8-30%, depending on the melting temperature of the pitch), ash (0.2% and higher), and asphaltenes. The density of the pitch is 1.2-1.3 g/cm^3. Pitch are not electrically conductive and insoluble in water, they dissolve only in organic solvents (pyridine, benzene, etc.) and are resistant to the action of acids [13-15].

Given the potential of nanofibers, interest in production technology is growing. Among technologies, including templating method, drawing method, phase separation and electrospinning attracted the greatest interest. We will use the method of electrospinning to produce CNF, since this method is economically effective on a semi-industrial scale.

Electrospinning is a process that leads to the formation of nanofibers as a result of the action of electrostatic forces on an electrically charged flow of a solution or a melt [16, 17]. The essence of the method of electrospinning is that the electrical voltage from 1-100 kW is applied to the solution (melt), which is fed through a capillary by means of a dispenser [18]. High voltage induces in the solution similar electrical charges, which, as a result of Coulomb electrostatic interaction, lead to the drawing of the solution into a thin flow [19, 20]. In the process of electrostatic jet stretching, a series of successive splittings into more thin flow can occur under a certain ratio of the values of viscosity, surface tension, and the density of electric charges in the fiber [21]. The resulting flow harden by evaporation of the solvent or as a result of cooling, turning into fibers, under the action of electrostatic forces drift to a grounded substrate having the opposite value of the electric potential. The precipitation electrode (collector) must have good electrical conductivity [22, 23].

The authors of [24] found that by varying the parameters of the process of electrospinning, it is possible to vary the thickness of the filaments in the range from hundreds of nanometers to several microns. The method of electrospinning is good because, unlike usual, mechanical drawing of fibers from a solution, it does not impose high requirements on the chemistry of the process, does not require high temperatures for solidification of the fiber, and therefore it allows the creation of fibers from long and complex molecules, as a result of the struggle between capillary and electrostatic forces. Also, the processes inside the solution, the charged drop itself lengthens, becomes thinner and dries out in flight.

Electrospinning is a universal and effective method for obtaining continuous nanofibers from submicron diameters to nanometer diameters, using a high-potential electric field [25, 26]. The technology can be easily used in the laboratory and can be scaled to the industrial process [27]. Electroconversion of nanofibers from polymer solutions or melts is of practical interest, since they have many potential applications [28].

Optimum nanofibers can be manufactured by controlling the solution, process and environmental parameters, since the characteristics of the electro-cord are determined by these parameters. It is very important to avoid the appearance of bubbles, especially for small nanofibres, by controlling the parameters it is possible to control the pore diameter [28].

A typical electrospinning installation, as shown in Figure 1, consists mainly of three components: a capillary tube with a pipette or a small diameter needle, a high voltage source, a metal collecting screen.

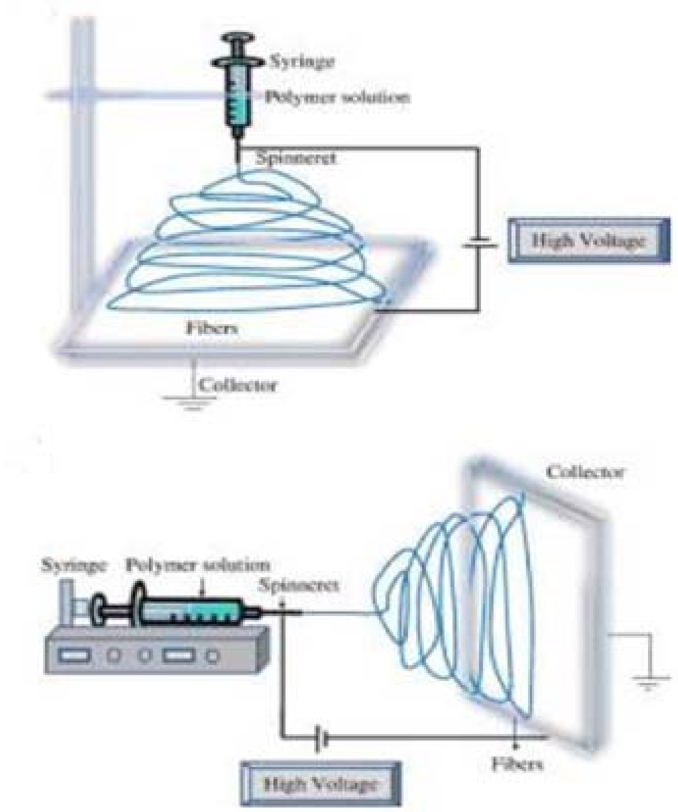


Figure 1 – Schematic diagram of the electrospinning installation

On the basis of the foregoing, the goal of the forthcoming work was formulated, which is to produce carbon fibers on the basis of coal-tar pitch by the method of electrospinning and the study of physico-chemical properties.

Research method. As a raw material, coal tar pitch, which is formed during the pyrolysis of raw materials, from coal of the Shubarkol deposit is used.

In the study, the method of electrospinning was used to produce nanofibers in laboratory conditions. The following instruments were used to prepare the raw material for the production of carbon nanofibers: ultrasonic bath, laboratory electro spinning device, SEM (Quanta 3D 200i) with an attachment for energy dispersive analysis from EDAX, HORIBA Jobin Yvon).

We have chosen the method of electrospinning to produce CNF, since this method is acceptable in laboratory conditions, and thin fibers are formed. Electrospinning is good in that, unlike the usual mechanical pulling of fibers from a solution, it does not impose high requirements on the chemistry of the process, it does not require high temperatures for solidification of the fiber, and therefore it allows the creation of fibers from long and complex molecules as a result of the struggle of capillary and electrostatic forces. Also, the processes inside the solution, the charged drop itself lengthens, becomes thinner and dries out in flight. Despite the complexity of the physical processes of electroforming, this method is characterized by hardware simplicity, high energy efficiency of production, wide versatility to the materials being formed and flexibility in controlling the parameters of the process. All this makes the electrospinning process attractive for industrial production of nanofibres.

Investigation of the elemental composition, structure, and dimension of CNF was performed using energy dispersive X-ray spectroscopy on a SEM device (Quanta 3D 200i) with an attachment for energy-dispersive analysis from EDAX (NNLOT, KazNU named after al-Farabi, Almaty). For the study, the samples were attached to a copper holder using conductive adhesive paper. The energy of the exciting electron beam in the analysis was 15 keV, the working distance was 15 mm.

A study of the type of carbon modification was carried out using Raman spectroscopy using the Raman scattering method. The Raman spectra of the samples were recorded on an Integra Spectra probe scanning microscope, using a laser with a wavelength of 473 nm. The spectra were recorded with a 20 second accumulation. Samples were supported as a thin replica on a glass substrate. The spectral detector CCD3, the wavelength K = 632.8 nm (20 mV), the width of the spectral line was 2.08 cm^{-1}. Installation parameters: power - 35 mW, solid-state laser, grating -600/600.

Results and its discussion. The most important stage in the production of carbon fiber based on coal tar pitch is the process of obtaining the initial fiber. Pitches are a complex mixture of aromatic and aliphatic compounds. The molecular weight of the compounds is relatively small, and only a part of them can be attributed to oligomers. A coarse brittle fiber can be formed from such systems. Consequently, a good quality carbon fiber can not be obtained, so low-molecular volatile compounds must be previously removed to impart fiber-forming properties to the pitch. To increase the molecular weight, the pitch must be heat treated.

In order to obtain CNF by the method of electrospinning, coal tar pitch is placed in a quartz reactor and heated to 400^0C at a heating rate of 10-15 ^{o}C/min and with holding at 400 ^{o}C for 3-4 hours (figure 2). The quartz reactor was heated and cooled in an inert argon medium at a gas velocity of 80 cm^3/min. Then the reactor is cooled and intermediate pitches are collected (figure 3).

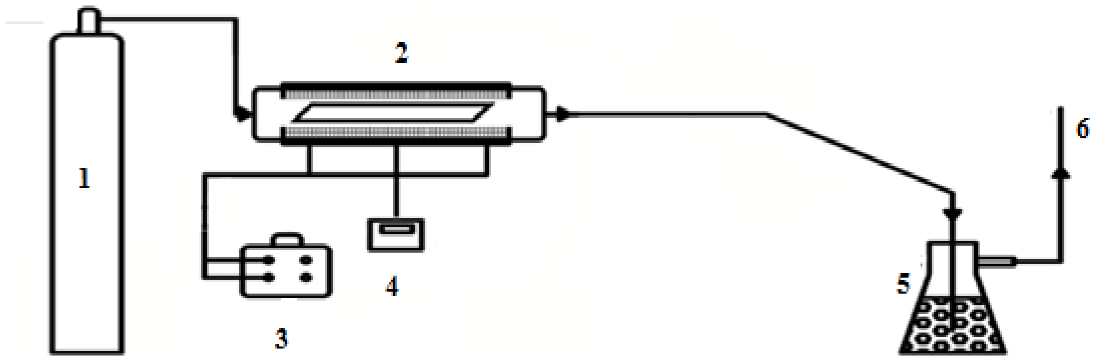
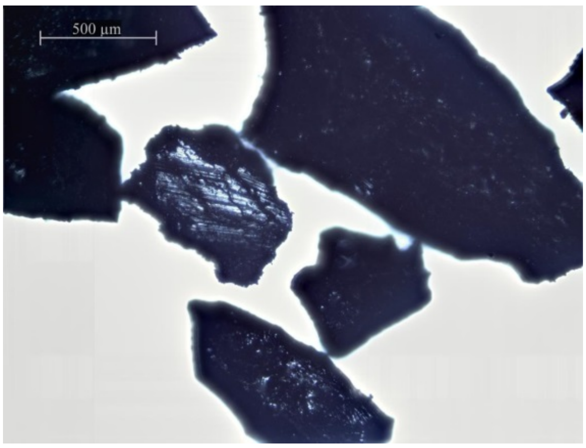


Figure 2 – Schematic diagram of the laboratory installation for the synthesis of CNF:
1 - gas cylinder (argon); 2 - quartz reactor; 3 - LATR; 4-temperature sensor; 5 - a flask to control the gas outlet; 6 - gas outlet



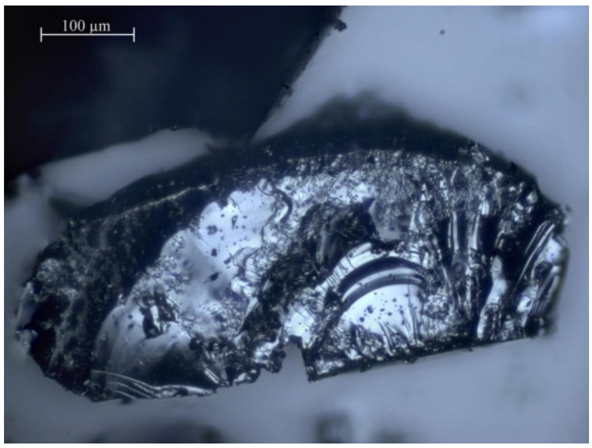
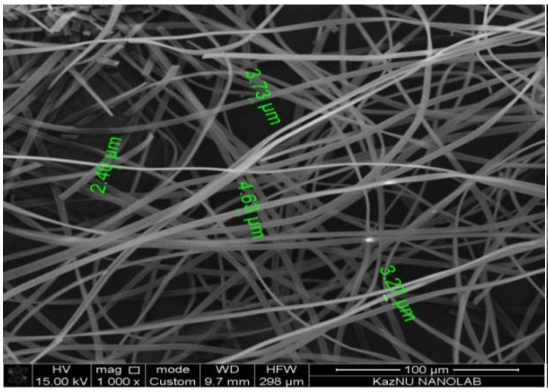


Figure 3 – Carboniferous coal tar pitch

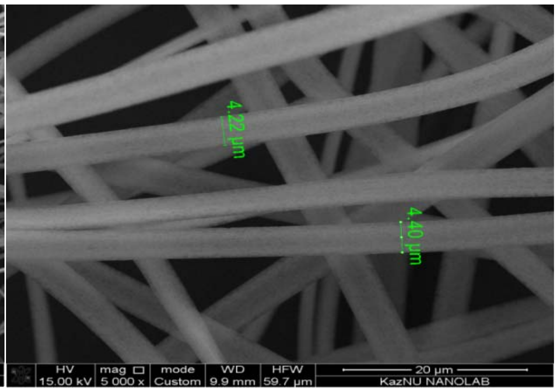
The resulting pitch is crushed, 1,2-dichloroethane is added, afterwards the resulting mixture is placed in an ultrasonic bath for 10-20 minutes for complete mixing. Polymethyl methacrylate is used as a binder, which is also mixed with 1,2-dichloroethane in an ultrasonic bath for 10-20 minutes. The resulting mixtures in 1: 1 ratios are placed in an ultrasonic bath for 20-30 minutes.

Ready mixture is dialed into an insulin syringe and installed on the reversing motor. A charge is applied to the tip of the syringe. Then the engine starts, and with the advent of the first drop the opposite charge is turned on. The same charge is attracted to the substrate with the opposite charge and nanofibers are formed (figure 4). Nanofibers are cured by solvent evaporation. The value of the interelectrode voltage was 18-20 kV. High voltage is the basis of electroforming.

Figure 4 –
Carbon nanofibre obtained
by the method of electrospinning
Electron microscopic images
of nanofibres are shown in figure 5



а)



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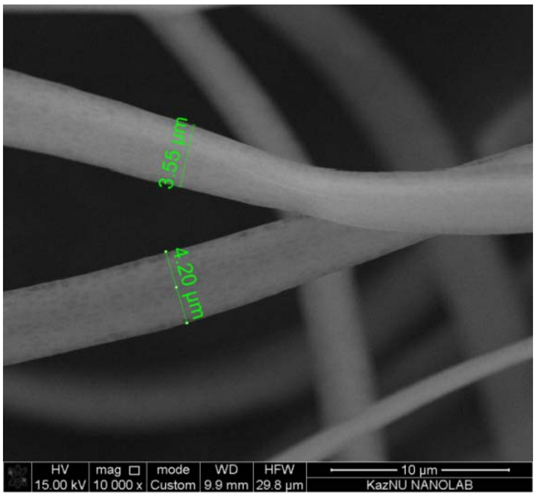

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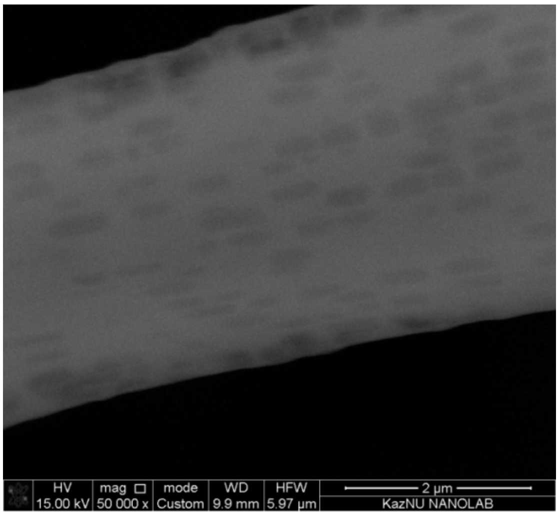


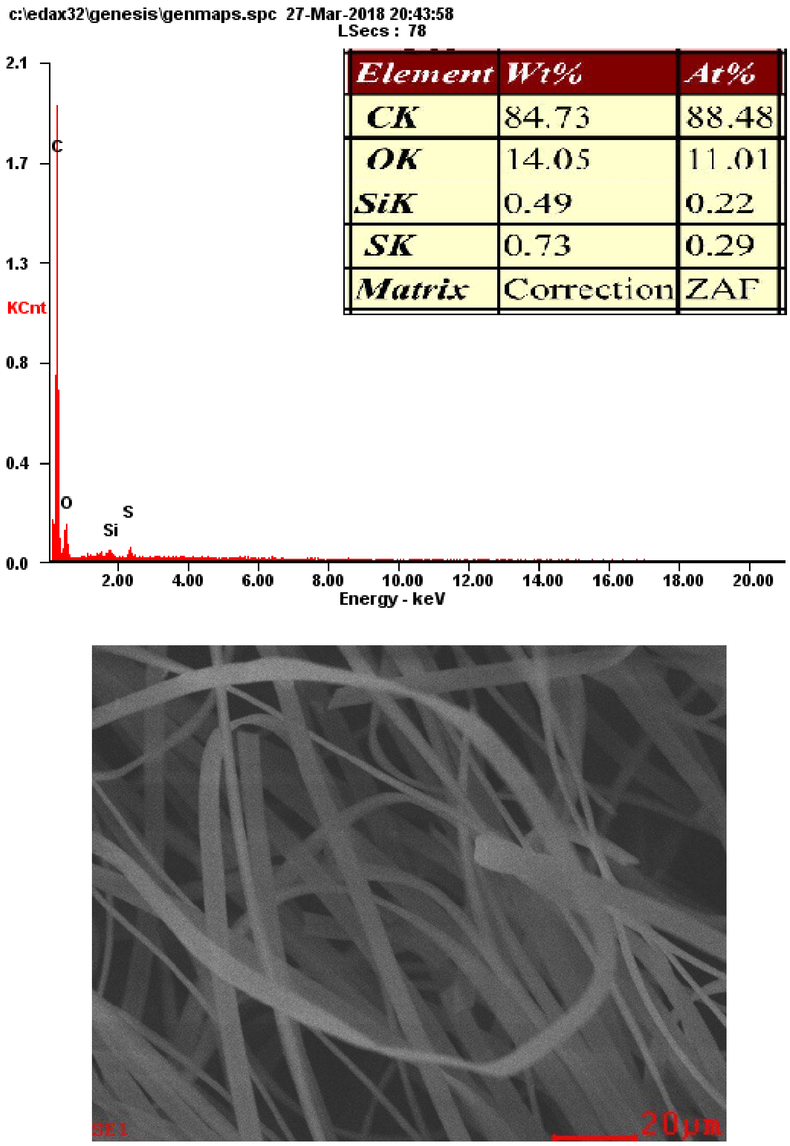
г)

Figure 5 – Electron microscopic images of carbon fibers: a) x1000, b) x5000, c) x10 000, d) x50 000

The process of electroforming begins when the electrostatic forces between the charges accumulated in the molding solution and the electric field between the electrodes overcome the surface tension of the molding solution. The repulsive force between the charges of the same name stretches the viscoelastic flow of the molding solution.

In figure 5, nanofibers with a diameter of 2 to 9 μm are clearly visible, the structural elements take the form of fibrils-filamentary formations whose length exceeds their diameter by more than an order of magnitude.

The results of the elemental analysis are shown in figure 6.

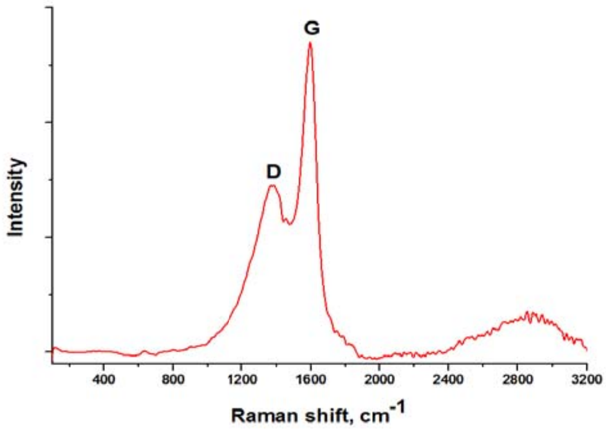


Element	Wt%	At%
CK	84.73	88.48
OK	14.05	11.01
SiK	0.49	0.22
SK	0.73	0.29
Matrix	Correction	ZAF

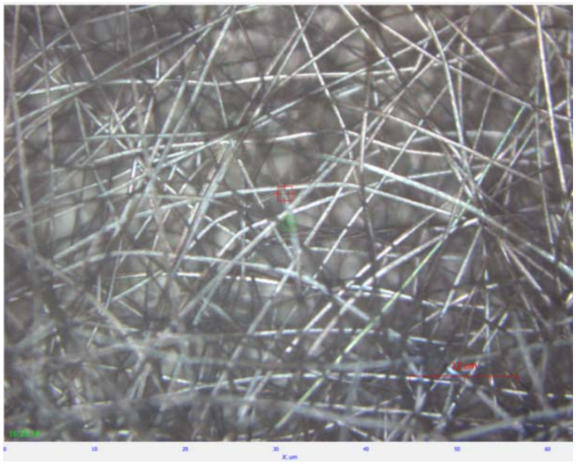
Figure 6 – Elemental analysis of carbon fiber

As shown in figure 5, the elemental composition of carbon fibers is composed by weight %: C – 84,73; O – 14,05; Si – 0,49; S – 0.73.

Figure 7 shows the Raman spectrum of the sample in the wave interval 200-3200 cm^{-1}. There are peaks D and G, which characterize carbon materials. The peak position is D – 1367 cm^{-1}, the intensity is 2900 units, the peak position is G – 1595 cm^{-1}, the intensity is 5401 units. There is also a peak at 2900 cm^{-1}, which indicates the presence of CH-bonds, which indicates the inclusion of polymers in the composition.



a)



б)

Figure 7 – Raman spectrum (a) and optical pattern (b) of carbon fibers

Further, we carry out a study on the oxidation and graphitization of the obtained CNF, since the formed fiber, as a rule, is characterized by low strength and increased brittleness. Such properties are natural for fibers from oligomers, which are essentially pitches. To increase the strength and impart a non-meltability, the formed fibers are oxidized in a gas or liquid medium. Oxygen (air), air with additives of ozone, oxygen or chlorine, a pair of nitroaromatic compounds (nitrobenzene, nitrophenol), dioxide and sulfur trioxide, ozone oxides serve as oxidants [5]. Since oxidation is carried out at elevated temperatures, it is heated at a low rate to produce a spun fiber. The oxidation temperature in the air stream is 280 °C for 1 hour at a heating rate of 1 °C. The oxidized fibers are carbonized at 800 °C for 10 minutes at a heating rate of 5 °C under a nitrogen atmosphere [6]. However, if the carbon content in the fiber is 95%, the heating rate can be increased to 10°C/min. In this case, the fiber yield reaches 85-90%.

The carbonized fiber is graphitized under tension both during electrical heating and when electric current is passed through the fiber. Elastic-strength indexes of fibers from pitch can be significantly increased by stretching during heat treatment at temperatures above 2800^0C. Next, the finished fiber is wound on the coils [5].

Conclusions. Thus, the proposed method for producing carbon nanofibers is built on the basis of the method of electrospinning, which is the most promising method of industrial production and provides a product with a relatively high fraction uniformity, which determines the achievement of the strength characteristics required for structural materials.

In the case of providing Kazakhstani producers with a high quality product that meets all the requirements of the consumer, the need for importing UNV will decrease. There is no market for the production of carbon fiber based on coal pitch, although raw materials are not only cheap, but also affordable, and the resulting product, in the final analysis, far exceeds analogues in terms of elasticity. Unique properties allow the use of carbon fiber in various areas of human life. However, at the moment, industrial technologies for the production of functional carbon fiber from coal are only under development.

The ecological effect of our study is to create an environmentally friendly technology based on the processing of secondary raw materials (coal tar and other coal mining waste) to produce carbon fibers and composites based on them. The creation of this technology will allow to solve the ecological aspect of utilization of this type of waste with obtaining an economically viable product.

Our technology is unique in that the raw materials we use to produce carbon fibers are a renewable resource, compared to the technology of obtaining fibers from a variety of other precursors (nylon, polyester, acrylic, polypropylene, etc.). Prospectivity of these studies lies in the possibility of large-scale production of carbon fibers from coal tar pitch, which will lead to the appearance of materials and composites based on domestic production on the Kazakhstan market.

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Б. Т. Ермағамбет[1], М. К. Казанкапова[1], А. Т. Наурызбаева[1]
З. А. Мансуров[2], Г. Т. Смагулова[2], Б. Б. Кайдар[2]

[1]«Көмір химиясы және технология институты» ЖШС, Астана, Қазақстан,
[2]ҚР БҒМ ҒК «Жану проблемалары институты» ШЖҚ РМК, Астана, Қазақстан

ЭЛЕКТРОСПИННИНГ ӘДІСІМЕН КӨМІРТЕКТІ ТАЛШЫҚ АЛУ

Аннотация. Зертханалық жағдайда «Шұбаркөл» кенішінің көмірінен алынған пек негізінде электро-спиннинг әдісімен көміртекті талшық алудың әдістемесі келтірілген, электродаралық кернеу 18-20 кВ құрады. Зерттелетін үлгінің элементтік құрамы, беттік құрылымы, көміртекті талшықтың модификация типі анықталды. Энергодисперсиялық рентгенді спектроскопия және СЭМ микроскопия нәтижесінде химиялық құрамы анықталды (С – 84,73%; О – 14,05 %; Si – 0,49 %; S – 0,73%) және көміртекті талшықтаң диаметрі 2-ден 9 мкм аралықты құрыды. Раман спектроскопия нәтижелері көміртекті материалдарды сипаттайтын D (1367 $см^{-1}$) және G (1595 $см^{-1}$) шыңдарын көрсетті, сонымен қатар СН-байланысына тән шың 2900 $см^{-1}$ анықталды.

Түйін сөздер: көміртекті талшық, таскөмір шайыры, электроспиннинг, композит.

Б. Т. Ермағамбет[1], М. К. Казанкапова[1], А. Т. Наурызбаева[1]
З. А. Мансуров[2], Г. Т. Смагулова[2], Б. Б. Кайдар[2]

[1]ТОО «Институт химии угля и технологии», Астана, Казахстан,
[2]РГП «Институт проблем горения», Алматы, Казахстан

ПОЛУЧЕНИЕ УГЛЕРОДНЫХ ВОЛОКОН МЕТОДОМ ЭЛЕКТРОСПИННИНГА

Аннотация. Приведена методика получения углеродных волокон из каменноугольного пека место-рождения «Шубарколь» методом электроспиннинга в лабораторных условиях, значение межэлектродного напряжения составило 18-20 кВ. Определен элементный состав и изучена морфология поверхности исследуемого образца, выявлен тип модификации углеродного волокна. Выявлено, что в результате энергодисперсионной рентгеновской спектроскопии и СЭМ микроскопии обнаружен химический состав (С – 84,73%; О – 14,05 %; Si – 0,49 %; S – 0,73%) и диаметр углеродных волокон который составил от 2 до 9 мкм. Результаты Рамановкой спектроскопии показал наличие пиков D (1367 $см^{-1}$) и G (1595 $см^{-1}$), которые характеризует углеродные материалы, так же обнаружен пик на 2900 $см^{-1}$, который указывает о наличии СН-связей.

Ключевые слова: углеродное волокно, каменноуглыный пек, электроспиннинг, композит.

Information about authors:

Yermagambet Bolat Toleukhanuly, Doctor of Chemical Science, Professor, Director of LLP "Institute of Coal Chemistry and Technology", Astana, Kazakhstan; bake.yer@mail.ru; https://orcid.org/0000-0003-1556-9526

Kazankapova Maira Kuttybaevna, Phd, Senior Researcher of LLP "Institute of Coal Chemistry and Technology", Astana, Kazakhstan; maira_1986@mail.ru; https://orcid.org/0000-0001-9016-3062

Nauryzbayeva Asemay Turlanovna, bachelor, Junior Researcher of LLP "Institute of Coal Chemistry and Technology", Astana, Kazakhstan; asemai_2296@mail.ru; https://orcid.org/0000-0003-4912-3856

Mansurov Zulhair Aimukhametovich, Doctor of Chemical Science, Professor, General Director of RSE «Institute of Combustion Problems», Almaty, Kazakhstan; zmansurov@kaznu.kz; https://orcid.org/0000-0002-8956-216X

Smagulova Gaukhar Tolbayevna, PhD, Leading Researcher of of RSE «Institute of Combustion Problems», Almaty, Kazakhstan; smagulova.gaukhar@gmail.com; https://orcid.org/0000-0002-2943-5222

Kaidar Bayan, PhD student, Researcher of of RSE «Institute for Problems of combusting», Almaty, Kazakhstan; kaydar.bayan@gmail.com; https://orcid.org/0000-0001-6327-7531

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