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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Үлттық ғылым академиясы «ҚР ¥ҒА Хабарлары. Химия және технология сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бүл 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 үсынады. ҚР ¥ҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қогамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» 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 Socia1 Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество в глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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SYNTHESIS OF CARBON NANOFIBERS BASED ON HUMIC ACID AND POLYACRYONITRILE BY ELECTROSPINNING METHOD

Abstract. The article describes a method for obtaining carbon nanofibers (CNFs) based on humic acid from oxidized coal of the Maikuben basin and polycarlonitrile (PAN) by electrospinning in laboratory conditions. The value of the interelectrode voltage was 20-25 kV. The elemental composition was determined and the surface morphology of the studied sample was studied, the type of modification of the carbon fiber was revealed. As a result of energy dispersive X-ray spectroscopy and scanning electron microscopy (SEM), the chemical composition of the initial CNF (C-48.73%) and the diameter of carbon fibers, which ranged from 148.6 nm to 1.36 μm, were found. The processes of oxidation and carbonization of the obtained samples were also carried out. The elemental composition of carbon after oxidation and carbonization was 87.75 and 89.16%, respectively, the diameter of the fibers was 117.5 nm -1.03 microns. The results of Raman scattering of light (RS) of carbonized CNF showed the degree of graphitization - 23.97%, the ratio I (D) / I (G) = 0.7, I (G) / I (D) = 1.4. The resistance of this material was 27 ohms. On the basis of SEM patterns of CNFs based on humic acid and PAN, it was found that the structure of the sample after oxidation and carbonization retains the original fibrous structure. It was also found that the diameter of nanofibers decreases from 1 μm to 117.5 nm, which may be associated with the release of volatile and heterogeneous components of the original product and the formation of a more structural thin porous filament.

Key words: carbon nanofiber (CNF), humic acid (HA), polyacrylonitrile (PAN), electrospinning, formation, oxidation, carbonization.

Introduction. Over the past few decades, lithium-ion batteries (LIB) have been successfully applied as rechargeable power supplies in various electrical products such as MP3 players, laptops and cell phones due to their long lifespan and high energy density [1, 2]. However, their high cost (about USD 5,000 per ton of Li_2CO_3 [3]) and limited onshore lithium reserves [4] have limited their further large-scale application in areas such as hybrid electric vehicles. Therefore, a growing concern lately is the abundant sodium on Earth (about US $ 150 per tonne [2]), which also comes from the alkaline family. Compared to lithium metal anodes, sodium-based metal batteries exhibit a lower theoretical energy density as a result of a higher standard sodium reduction potential and a lower specific capacity [2]. Similar problems with lithium metal anodes are even more problematic for sodium metal anodes due to the higher reactivity of sodium metal and dendrite formation. The safety risk of devices with metallic sodium electrodes at ambient temperatures is also serious due to the low melting point of sodium (98°C) [5]. Therefore, it is important to develop true sodium ion batteries (NIBs), in which sodium ions move between electrodes in the form of a "rocking chair". This design avoids the use of highly reactive sodium metal. So far, a large number of promising cathode materials, including $NaCrO_2$, NaV_6O_{15}, $NaFePO_4$, Na_2MPO_4F и $Na_3M_2 (PO_4)_3$ (M = Fe, Mn, Co и Ni), Na0,44 MnO_2, $NaNi_{1/3}$ $Mn_{1/3}$ $Fe_{1/3}$ O_2. , $Na_{2/3}$ [$MxMn_1$− x] O_2, $NaMF_3$ (M = Fe, Mn, V, Ni), were developed [2–4, 6].

Materials. As a result of the fact that few anode materials have been studied for NIB, the selection of the appropriate anode materials has become one of the most important tasks in the development of highly efficient NIB. Unfortunately, the outstanding LIB graphite anodes previously put on the market do not have a high reversible capacity in NIB [7]. On the other hand, many other carbonaceous materials such as solid carbon [8,9], carbon fibers [10-13], carbon nanotubes [14], graphene [15], carbon black [16], cellulose [17]], polyparaphenylene [18], porous carbon [19], carbon spheres [20] and petroleum coke [21] have been found to facilitate the sodium ion incorporation / extraction process. Carbon nanomaterials, which are considered one of the most promising candidates among a limited number of anode materials for NIBs, have such advantages as electrical conductivity, heat resistance, environmental friendliness, and cost effectiveness [14]. Recent studies have shown that anodic morphology significantly affects the electrochemical characteristics of NIBs [8-15]. Thus, further use of carbon nanomaterials can provide high NIB performance characteristics. Although various carbon electrodes with excellent mechanical and ionic transport properties have been used in solar cells [22], fuel cells [23], supercapacitors [24], and LIB [25], it is unlikely that the use of nanocarbon anodes has been investigated at NIB.

Due to their outstanding tensile strength and modulus, carbon nanofibers are commercially used in structural composites for superior performance, lightness, and ergonomics. The global carbon fiber market is expected to triple in one decade and surpass 100,000 tonnes in 2020, worth over $ 3 billion. While carbon fiber composites are currently used in airplanes and some racing cars, the cost of CNF is prohibitive for many other sectors of the industry. The high price of CNF is directly related to the cost and yield of the precursor from which it is obtained, and the cost of its conversion.

Electrospinning as a universal methodology for the manufacture of organic, inorganic, and hybrid nanomaterials with controlled dimensions attracts the attention of the whole world in the production of random or oriented continuous nanofibers [26]. Controllable sizes and flexible compositions of electrically spun nanofibers are increasingly being investigated for the targeted study of electrode materials in energy conversion and storage applications [27]. In addition, electrospinning has such advantages as low cost, high yield, simplicity, efficiency, as well as high reproducibility, applicability, and adaptability [28]. Thus, the electrospinning method can be easily applied for the manufacture of various components of energetic materials, ranging from carbon nanofibers to ceramic or metal electrodes [29]. On the other hand, industrial scaling is still largely a challenge for electrospinning, with a lot of room for further improvement. For many types of materials, the achievable lengths of nanofiber electrospinning are still from hundreds of micrometers to millimeters, which is insufficient for most industrial applications. In such cases, the electrospinning method often shows limited performance at the manufacturing stage [30].

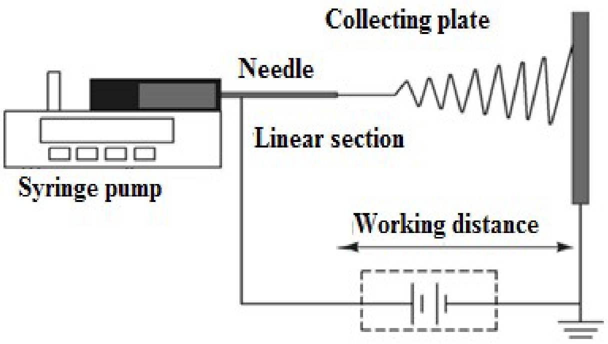


Figure 1 - Schematic diagram of an electrospinning installation with a stationary substrate

Although various anode material precursors have been demonstrated, further improvements are still urgently needed in terms of storage capacity, long cycle performance and speed capability. On the other hand, most electrode materials [14-17] come from expensive chemical raw materials, which are often associated with contamination. Thus, the study of environmentally friendly anode materials remains challenging. As one of the carbonaceous precursors, biomass has been carefully studied for its abundance, ease of access and low cost [31]. In the light of previous studies of biomass carbon [31-33], humic acid (HA) was chosen as the anode material for NIB production. HA is a mixture of aliphatic carbon chains and aromatic organic acids, soluble in water under alkaline conditions, but insoluble under acidic conditions [34]. On the other hand, HAs, as a rule, are complex macromolecules of aromatic units with bound peptides, amino sugars, aliphatic acids, amino acids, and other organic components with a molecular size ranging from about 10,000 to 100,000 [34, 35]. For their flexible chemical properties, these complex carbonaceous materials are considered variable linear polymers that have random helixes of crosslinked bonds in a three-dimensional aspect. As a biomass material with natural environmentally friendly, renewable, low-cost characteristics and a carbon content of more than 40 wt%, obtained from coal, soil or wood, as well as a precursor of fulvic acids and a product of lignin decomposition [36, 37], HA can become potentially reliable a candidate as anode materials.

In our proposed method of producing carbon nanofibers, it includes the following stages: preparation of raw materials, formation, stabilization (to remove low molecular weight products of destruction and the formation of crosslinked and cyclic structures), carbonization (to remove hydrogen and heteroatoms in the form of volatile compounds, the final formation of carbon fibers occurs).

The aim of this work is to synthesize carbon nanofibers by electrospinning from humic acid and polyacryonitrile (PAN).

Methods.

The elemental composition, structure, and dimensions of CNFs were studied by energy dispersive X-ray spectroscopy on an SEM device (Quanta 3D 200i) with an EDAX attachment for energy dispersive analysis. For the study, the samples were fixed on a copper holder using conductive adhesive paper. The energy of the exciting electron beam in the analysis was 15 kV, the working distance was 15 mm.

The study of the type of carbon modification was carried out using Raman spectroscopy using the method of Raman light scattering (RS). The Raman spectra of the samples were recorded on an Integra Spectra scanning probe microscope using a laser with a radiation wavelength of 473 nm. Spectra were recorded with a 20 second accumulation. The samples were applied as a thin replica on a glass substrate. The analyzes used a CCD_3 spectral detector, wavelength K = 632.8 nm (20 mV), the spectral line width was 2.08 cm^{-1}. Installation parameters: power - 35 mW, solid-state laser, grating - 600/600.

Humic acid and polyacrylonitrile are dissolved in N, N-dimethylformamide in an oven at 60°C until homogeneous (Fig. 2). Composition: humic acid - 5%, polyacrylonitrile (PAN) - 3%, N, N-dimethylformamide - 92%.

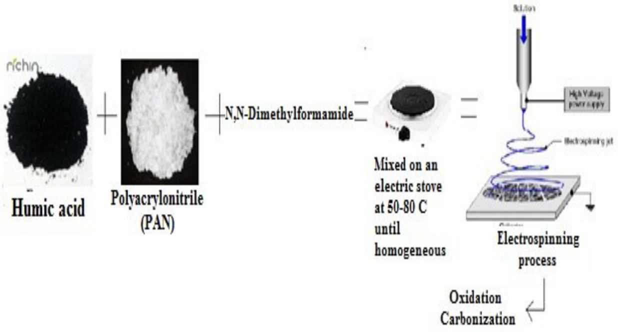


Figure 2 - Scheme of obtaining carbon nanofiber based on humic acid and PAN

The reaction between humic acid and PAN is presented below:

Next, the ready-made solutions are formed in a laboratory electrospinning installation, for this the ready-made mixture is drawn into an insulin syringe and installed on a reversible motor, the prepared solution is placed in a syringe, a charge is applied to the tip of the syringe. Then the engine is turned on, and with the appearance of the first drop, the opposite charge is turned on. A charge of the same name is attracted to a substrate with an opposite charge and nanofibers are formed (Fig. 3). Nanofibers are cured by evaporation of the solvent. The interelectrode voltage was 25 kV, the distance between the receiver and the syringe was 35 cm [39]. High voltage is the basis for electrospinning.

An important factor in choosing the optimal conditions for electrospinning is the diameter of the resulting fibers. It was obtained by solvent evaporation (DMF) followed by solidification of streams of polymer molding solution on a metal collector. Therefore, the fiber diameter depends mainly on the viscosity of the solution; the higher the viscosity, which increases with an increase in the polymer concentration, the larger the fiber diameter, which varies in the range from several tens of nm to microns [38].

The stages of stabilization and carbonization of CNF were carried out in a laboratory reactor. CNF samples are loaded into a horizontal cylindrical quartz reactor. The CNF was stabilized at a temperature of 300°C in an air flow in a laboratory quartz reactor at a heating rate of 5°C / min, the holding time was 1 hour.

After oxidation, the stage of CNF carbonization was carried out in an inert argon atmosphere at a temperature of 800°C, the heating rate was 5°C / min, and the holding time was 60 minutes. Argon is fed from a cylinder to the reactor at a predetermined flow rate of 20 ml / min, which is set using a flow meter.

Results and discussion.

Electron microscopic images of nanofibers are shown in Figure 3, where carbon nanofibers with a diameter of 148.6 nm to 1.36 µm are clearly visible. The elemental composition of the composite is: C - 98.48%, O - 0.77%, Si - 0.11%, Fe - 0.64%.

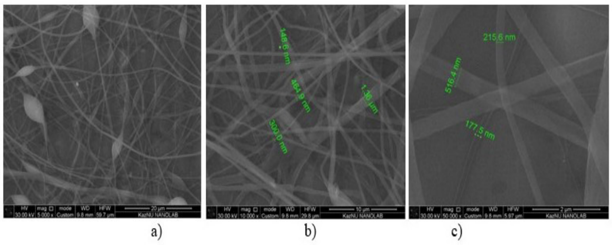


Figure 3 - Electron microscopic images of carbon fiber nanobased on humic acid and PAN: a) x5000, b) x10000, c) x50000

The elemental composition was determined and the surface morphology of the sample under study was studied, the type of CNF modification was revealed. As a result of energy dispersive X-ray spectroscopy and SEM microscopy, a chemical composition was found: C-48.73%. Figure 3 clearly shows CNF particles with a diameter of 148.6 nm to 1.36 µm. CNFs have a structure in cross section - a rounded shape, structural elements take the form of fibrils - threadlike formations.

The resulting fibers are stabilized, for example, by oxidative treatment, making them infusible. For this, CNFs were oxidized, as a result, low-molecular-weight degradation products are removed and cross-linked and cyclic structures are formed (Fig. 4).

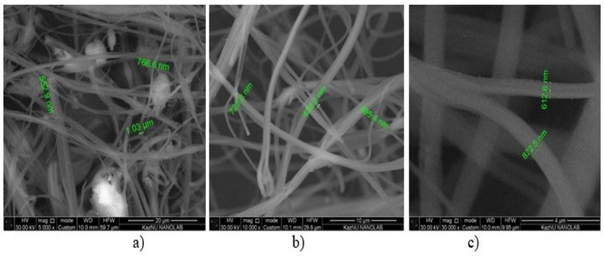


Figure 4 - Electron microscopic images of oxidized carbon nanofiber based on humic acid and PAN: a) x5000, b) x10000, c) x30000

The results on a scanning electron microscope showed CNFs with a diameter of 433.2 nm to 1.03 µm. Figures 4 show CNFs with smooth, filamentous structures. Also visible are spherical agglomerates, which appeared due to adhesion of fibers during deposition and formation during drying. The elemental composition of the composite after oxidation is: C - 87.75%, O - 10.53%, Mg - 0.12%, Al - 0.25%, Si - 0.26%, Cl - 0.10%, K - 0.24%, Ca - 0.13%, Fe - 0.37%, Cu - 0.25%.

At the subsequent stages of high-temperature treatment - carbonization at 800 oC, accompanied by the removal of hydrogen and heteroatoms in the form of volatile compounds, the final formation of carbon fibers occurs (Fig. 5).

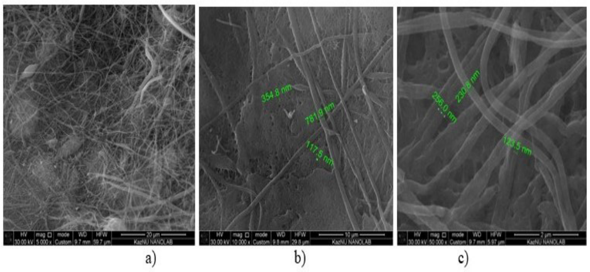
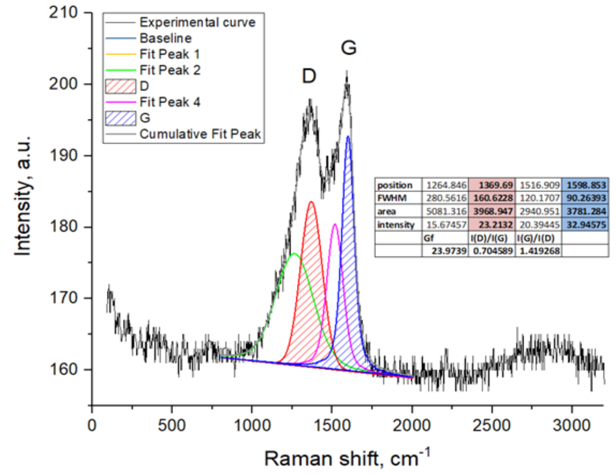


Figure 5 - Electron microscopic images of carbonized carbon nanofiber based on humic acid and PAN: a) x5000, b) x10000, c) x50000

The results on a scanning electron microscope after carbonization showed CNFs with a diameter of 123.5 nm to 781.9 nm (Fig. 5). Elemental composition of the composite: C - 89.16%, O – 8.22%, Na – 0.11%, Mg – 0.02%, Al – 0.20%, Si – 0.18%, Cl – 0, 11%, K – 1.11%, Ca – 0.09%, Fe – 0.55%, Cu – 0.24%. The formation of cylindrical fibers along the entire length of the fiber is observed; it has been established that the structure of CNFs after oxidation and carbonization retains the original fibrous structure (Fig. 5).

Raman spectra were recorded to study the degree of graphitization. The results of Raman light scattering (RS) showed the degree of graphitization - 23.97%, the ratio I (D) / I (G) = 0.7, I (G) / I (D) = 1.4 (Fig. 6). The broad bands D (disordered part) and G (ordered graphite structure) suggest that CNFs contain partially graphitized carbon along with amorphous carbon. Graphitized carbon typically consists of assemblies of graphite layers that are expected to act to store ions in fuel cells. The ratio of the relative intensity (ID / IG) of the D and G bands indicates the degree of disorder of the carbon structure. As shown in Figure 6, the ID / IG ratio represents the conversion of disordered carbon to graphite carbon during carbonization. The resistance of this material was 27 ohms.



position	1264.846	1369.69	1516.909	1598.853
FWHM	280.5616	160.6228	120.1707	90.26393
area	5081.316	3968.947	2940.951	3781.284
intensity	15.67457	23.2132	20.39445	32.94575
	Gf	I(D)/I(G)	I(G)/I(D)	
	23.9739	0.704589	1.419268	

Figure 6 - Cattle snapshot of a sample of carbonated carbon nanofiber based on humic acid and PAN

Conclusion. On the basis of SEM drawings of CNFs from HA + PAN, it was found that the structure of CNFs after oxidation and carbonization retains the original fibrous structure. It was also found that the diameter of nanofibers decreases from 148.6 nm -1.36 microns to 123.5 - 781.9 nm, which may be associated with the release of volatile and heterogeneous components of the original product and the formation of a more structural thin porous filament.

Thus, the proposed method for producing CNFs is based on the electrospinning method, which is the most promising method of industrial production and provides a product with a relatively high fraction homogeneity, which determines the achievement of strength characteristics for structural materials. These characteristics make it possible to use CNF obtained from HA and PAN as an electrode material for batteries and supercapacitors. Batteries with high energy density and long service life are widely used in our daily life, such as electric vehicles, mobile phones, laptops, etc.

The ecological effect of the research is to create an environmentally friendly technology based on the processing of secondary raw materials (coal mining waste, like oxidized coals) for the production of carbon fibers and composites based on them. The creation of this technology will make it possible to solve the ecological aspect of the disposal of this type of waste with the receipt of an economically viable product.

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ГУМИН ҚЫШҚЫЛЫ МЕН ПОЛИКАРИЛОНИТРИЛ НЕГІЗІНДЕ ЭЛЕКТРОСПИННИНГ ӘДІСІМЕН КӨМІРТЕКТІ НАНОТАЛШЫҚТЫ СИНТЕЗДЕУ

Аннотация. Мақалада зертханалық жағдайда электроспиринг әдісімен Майкүбен бассейнінің және поликарлонитрилдің (ПАН) тотыққан көмірінен гумин қышқылына негізделген көміртегі наноталшықтарын (КНҚ) алу әдісі сипатталған. Элементтің құрамы анықталды және зерттелген үлгінің беткі морфологиясы зерттелді, көміртек талшығының модификация түрі анықталды. Энергодисперсиялық рентген-спектроскопия және сканерлейтін электронды микроскопия (СЭМ) нәтижесінде 148,6 нм-ден 1,36 мкм-ге дейінгі бастапқы КНТ диаметрі мен химиялық құрамы (С-48,73%) анықталды. Алынған сынамалардың тотығу және көміртектену процестері де жүргізілді. Тотығудан және көміртектенуден кейінгі көміртектің элементтік құрамы сәйкесінше 87,75 және 89,16% құрады, талшықтың диаметрі 117,5 нм -1,03 мкм. Карбонизацияланған КНТ жарықтың комбинациялық шашырауының (ЖКШ) нәтижелері арқылы графиттену дәрежесі анықталды - 23,97%, I (D) / I (G) = 0,7, I (G) / I (D) = 1,4. Бұл материалдың кедергісі 27 Ом құрады. Гумин қышқылы мен ПАН негізіндегі КНТ үлгілерінің СЭМ үлгілері негізінде тотығу мен карбонизациядан кейінгі үлгінің құрылымы бастапқы талшықты құрылымды сақтайтындығы анықталды. Сондай-ақ, наноталшықтардың диаметрі 1 мкм-ден 117,5 нм-ге дейін төмендейтіндігі анықталды, бұл бастапқы өнімнің ұшқыш және гетерогенді компоненттерінің бөлінуімен және құрылымдық жағынан жіңішке кеуекті жіпшенің пайда болуымен байланысты.

Түйін сөздер: көміртекті наноталшық, гумин қышқылы, электроспиннинг, полиакрилонитрил, натрий-иондық батареялар, литий-иондық батареялар, тотығу, карбонизация.

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СИНТЕЗ УГЛЕРОДНЫХ НАНОВОЛОКОН НА ОСНОВЕ ГУМИНОВОЙ КИСЛОТЫ И ПОЛИАКРИОНИТРИЛА МЕТОДОМ ЭЛЕКТРОСПИННИНГА

Аннотация. В статье приведена методика получения углеродных нановолокон (УНВ) на основе гуминовой кислоты из окисленного угля Майкубенского бассейна и поликарилонитрила (ПАН) методом электроспиннинга в лабораторных условиях. Значение межэлектродного напряжения составило 20-25 кВ. Определен элементный состав и изучена морфология поверхности исследуемого образца, выявлен тип модификации углеродного волокна. В результате энергодисперсионной рентгеновской спектроскопии и скнирующей электронной микроскопии (СЭМ) обнаружен химический состав исходного УНВ (С-48,73%) и диаметр углеродных волокон который составил от 148,6 нм до 1,36 мкм. Также проведены процессы окисления и карбонизации полученных образцов. Элементный состав углерода после окисления и карбонизации составил 87,75 и 89,16 % соотвественно, диаметр волокон - 117,5 нм -1,03 мкм. Результаты комбинационного рассеяния света (КРС) карбонизованного УНВ показал степень графитизации – 23,97 %, соотношение I(D)/I(G)=0.7 , I(G)/I(D)=1.4. Сопротивление данного материала составил 27 Ом. На основание СЭМ рисунков УНВ на основе гуминовой кислоты и ПАН, установлено, что структура образца после окисления и карбонизации сохраняет исходную волокнистую структуру. Также было обнаружено, что диаметр нановолокон уменьшается от 1 мкм до 117,5 нм, что может быть связано выделением летучих и гетерогенных компонентов исходного продукта и образованием более структурной тонкой пористой нити.

Ключевые слова: углеродное нановолокно, гуминовая кислота, электроспиннинг, полиакрилонитрил, натриево-ионные батареи, литий-ионные батареи, окисление, карбонизация.

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