ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ

Д.В. Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
электрохимии им. Д.В. Сокольского»

NEWS

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
JSC «D.V. Sokolsky institute of fuel, catalysis
and electrochemistry»

SERIES
CHEMISTRY AND TECHNOLOGY

3 (441)

MAY – JUNE 2020

PUBLISHED SINCE JANUARY 1947

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

Clarivate
Analytics

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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология 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. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық 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 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 в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

«ҚР ҰҒА Хабарлары. Химия және технология сериясы».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» Республикалық қоғамдық бірлестігі (Алматы қ.).
Қазақстан республикасының Мәдениет пен ақпарат министрлігінің Ақпарат және мұрағат комитетінде 30.04.2010 ж. берілген **№1089-Ж** мерзімдік басылым тіркеуіне қойылу туралы куәлік.

Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.

Редакцияның мекенжайы: 050010, Алматы қ., Шевченко көш., 28; 219, 220 бөл.; тел.: 272-13-19; 272-13-18, http://chemistry-technology.kz/index.php/en/arhiv

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Редакцияның мекенжайы: 050100, Алматы қ., Қонаев к-сі, 142, «Д. В. Сокольский атындағы отын, катализ және электрохимия институты» АҚ, каб. 310, тел. 291-62-80, факс 291-57-22, e-mail:orgcat@nursat.kz

Типографияның мекенжайы: «NurNaz GRACE», Алматы қ., Рысқұлов көш., 103.

«Известия НАН РК. Серия химии и технологии».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы).
Свидетельство о постановке на учет периодического печатного издания в Комитете информации и архивов Министерства культуры и информации Республики Казахстан **№10893-Ж,** выданное 30.04.2010 г.

Периодичность: 6 раз в год.
Тираж: 300 экземпляров.

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28; ком. 219, 220; тел. 272-13-19; 272-13-18,
http://chemistry-technology.kz/index.php/en/arhiv

Адрес редакции: 050100, г. Алматы, ул. Кунаева, 142, АО «Институт топлива, катализа и электрохимии им. Д.В. Сокольского», каб. 310, тел. 291-62-80, факс 291-57-22, e-mail:orgcat@nursat.kz

Адрес типографии: «NurNaz GRACE», г. Алматы, ул. Рыскулова, 103.

News of the National Academy of Sciences of the Republic of Kazakhstan. Series of chemistry and technology.
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty).
The certificate of registration of a periodic printed publication in the Committee of Information and Archives of the Ministry of Culture and Information of the Republic of Kazakhstan N 10893-Ж, issued 30.04.2010.

Periodicity: 6 times a year.
Circulation: 300 copies.

Editorial address: 28, Shevchenko str., of. 219, 220, Almaty, 050010, tel. 272-13-19; 272-13-18,
http://chemistry-technology.kz/index.php/en/arhiv

Editorial address: JSC «D.V. Sokolsky institute of fuel, catalysis and electrochemistry», 142, Kunayev str., of. 310, Almaty, 050100, tel. 291-62-80, fax 291-57-22, e-mail: orgcat@nursat.kz

Address of printing house: «NurNaz GRACE», 103, Ryskulov str, Almaty.

N E W S
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES CHEMISTRY AND TECHNOLOGY
ISSN 2224-5286 **https://doi.org/10.32014/2020.2518-1491.52**
Volume 3, Number 441 (2020), 120 – 126

ISRSTI 61.67.33
UDC 678.03:546.26; 677.4

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PRODUCTION OF CARBON COMPOSITE NANOFIBERS BASED ON COAL TAR AND TEXTILE CORD

Abstract. The article conducted experiments on the production of carbon nanofibers based on coal tar and textile cord by method electrospinning in a laboratory setup. Carbon nanofibers (CNFs) were obtained from textile cord carbonizate (TC) and coal tar (CT) from the Shubarkol deposit, and polymethylmethacrylate (PMMA) was used as a binder. The elemental composition was determined and the surface morphology of the samples was studied. As a result of energy dispersive X-ray spectroscopy and SEM microscopy, the chemical compositions of the CNF based on TC + PMMA were determined, which amounted to C-82.69%, O-1.72%, Si-0.46%, Al-15.14%, TC + CT + PMMA: C-88.43%, O-4.06%, Al-7.32%, S-0.19%, the diameter of carbon nanofibers ranged from 64.2 nm to 539.3 nm.

Keywords: carbon nanofiber, coal tar, nanocomposites, electrospinning, textile cord, polymethylmethacrylate.

Introduction. In recent decades, the unique properties of carbon fibers have expanded the scientific base and technology of composite materials [1]. Carbon may exist in the form of tubular microstructures called filaments or fibers. Carbon nanofibers are a class of materials in which curved graphene layers or nanoconuses are folded in the form of a quasi-one-dimensional filament, whose internal structure can be characterized by the angle α between the graphene layers and the fiber axis [2]. Due to their exceptional thermal, electrical, shielding and mechanical properties [3], they have found application in science-intensive industries: mechanical engineering, nuclear energy, aviation and astronautics, the military-industrial complex, construction, in addition, carbon fibers have the potential to be used in various new applications, such as electrodes, catalyst substrates, adsorbents, composites, etc. Composites based on CNFs can be used as promising materials in many fields, such as electrical devices, electrode materials for batteries and supercapacitors, as well as sensors and others because of their large surface area and relatively high electrical conductivity [4].

Carbon fibers have high atmospheric resistance, resistance to light and penetrating radiation. Carbon fibers are bio-resistant and bio-inert, heat resistant and difficult to combust. Of the properties of carbon fibers, a special place is occupied by a high modulus of elasticity and strength, low density, low coefficient of friction, and also high resistance to atmospheric influence and chemical reagents. In addition to high strength properties and low weight, carbon fibers and composites from them (carbon plastics) are black and conduct electricity well. In addition, carbon fibers have a very low, almost zero coefficient of linear expansion, which makes them indispensable in some special applications [5,6].

The global market for carbon fibers and materials based on them has been growing steadily in recent decades, and only in the last eight years has it grown five times in physical terms. Most carbon fiber is produced in a complex and multi-stage process from specially prepared polymer raw materials, mainly polyacrylonitrile or from viscose. As the feedstock, we used coal tar from the coal of the Shubarkol deposit, which are formed during the pyrolysis of the feedstock, as well as textile cord - waste from the processing of car tires, and polymethylmethacrylate was used as a binder.

The initial coal tar has the following characteristics: density at 20 °C - 1070 kg / m^3; viscosity at 80 °C - 2.9-3.3 conventional degrees; coking ability - 2.0-3.5%; flash point - 110-120 °C; softening temperature - 60-70 °C; the yield of volatiles is 83.0%, which are formed during the pyrolysis of raw materials. It is not electrically conductive and insoluble in water, it is soluble only in organic solvents (pyridine, benzene, etc.), and is resistant to acids [7].

Textile cord – is a filament of cord fiber with rubber particles. It is obtained by processing automobile tires into rubber crumb. A car wheel consists of rubber, metal cord (wire) and textile material to give the wheel strength and wear resistance. Textile cord consists of polyester – 60%, polyamide – 37%, viscose – 3.0%, the density of which is 0.1 g / cm^3.

One of the methods for producing chemical fibers is the formation of fibers from solutions under the influence of an electrostatic field (electrospinning of fibers). An electrical voltage of 10 to 30 kV is applied to the solution using a pump syringe. High voltage induces in the solution the same electric charges, which, as a result of the Coulomb electrostatic interaction, lead to the drawing of the solution into a thin stream [8]. The resulting jets are cured by evaporation of the solvent or as a result of cooling, turning into fibers, and drift to a grounded substrate under the action of electrostatic forces [9-11]. Electrospinning is a universal and effective method for producing continuous nanofibers from submicron diameters to nanometer diameters using a high-potential electric field [12,13]. The technology can be easily used in the laboratory and can be scaled to an industrial process [14]. The electrical conversion of nanofibers from polymer solutions or melts is of practical interest, since they have many potential applications [15].

Based on the foregoing, the goal of the forthcoming work was formulated, which is to obtain carbon fibers based on coal tar and textile cord by the method of electrospinning and the study of physicochemical properties.

Research methodology. CNF samples were obtained at the “Institute of Coal Chemistry and Technology” LLP (Nur-Sultan) by the method electrospinning under laboratory conditions.

As feedstock were used coal tar which was obtained from the coal «Shubarkol» deposit, textile cord and PMMA. Samples of textile cord were provided by «Kazakhstan Rubber Recycling» LLP (Nur-Sultan).

We have chosen the electrospinning method for obtaining CNFs, since this method is acceptable in the laboratory, and thin fibers are formed. Electrospinning is good in that, unlike the 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 the fiber to solidify, which means that it allows the creation of fibers from long and complex molecules as a result of capillary and electrostatic forces. Also, the processes inside the solution, the charged drop itself lengthens, thins, and dries in flight.

The technology for producing carbon nanofibers includes the following stages: preparation of raw materials, formation, oxidation (to remove low molecular weight degradation products 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).

To obtain nanofibers based on textile cord, the sample was carbonized at 400 °C in a rotary tube furnace in argon (figure 1).

Figure 1 - High-temperature rotary tube furnace BR-12NRT


a) b)

Figure 2 – Samples of textile cord: a- initial, b-carbonized (400^{0}C)

The obtained carbonized textile cord is crushed and dissolved in 1,2-dichloroethane in an ultrasonic bath at 35 °C for 30 minutes, the frequency is 35 kHz. Polymethylmethacrylate is also soluble in 1,2-dichloroethane (mass fraction of 3%) in an ultrasonic bath under similar conditions. The prepared solutions obtained in the previous stages are mixed in 1:1 ratios in an ultrasonic bath at 35 °C for 40 minutes until a homogeneous mass is obtained. To obtain a nanofiber, dies are used in the form of a needle with a diameter of 0.6 mm. A pulsed high voltage voltage of 20-25 kV is applied to the die. A solution with a solvent is squeezed out of the die with a speed of 5-10 ml / h and precipitated onto a substrate. The distance between the substrate and the syringe was 20 - 30 cm. Next, the finished solutions are formed in the laboratory electrospinning unit [16]. For this the finished mixture is drawn into the syringe and installed on the pump motor, a charge is supplied to the tip of the syringe. Next, the engine turns on, and with the appearance of the first drop, the opposite charge is turned on. The charge of the same name is attracted to the substrate with the opposite charge and nanofibers are formed. Nanofibers are cured by evaporation of the solvent. High voltage is the basis of electroforming.

To obtain composite carbon nanofibers, coal tar and carbonized textile cord (1:1) are used, and polymethyl methacrylate is used as a binder, 1,2-dichloroethane is used as a solvent, the mixture of the starting products is dispersed in an ultrasonic bath at a temperature of 35 ^{0}C, frequency – 35 kHz, holding time 30 min, the prepared solution is placed in an electrospinning unit with a syringe, the set voltage is 20-25 kV.

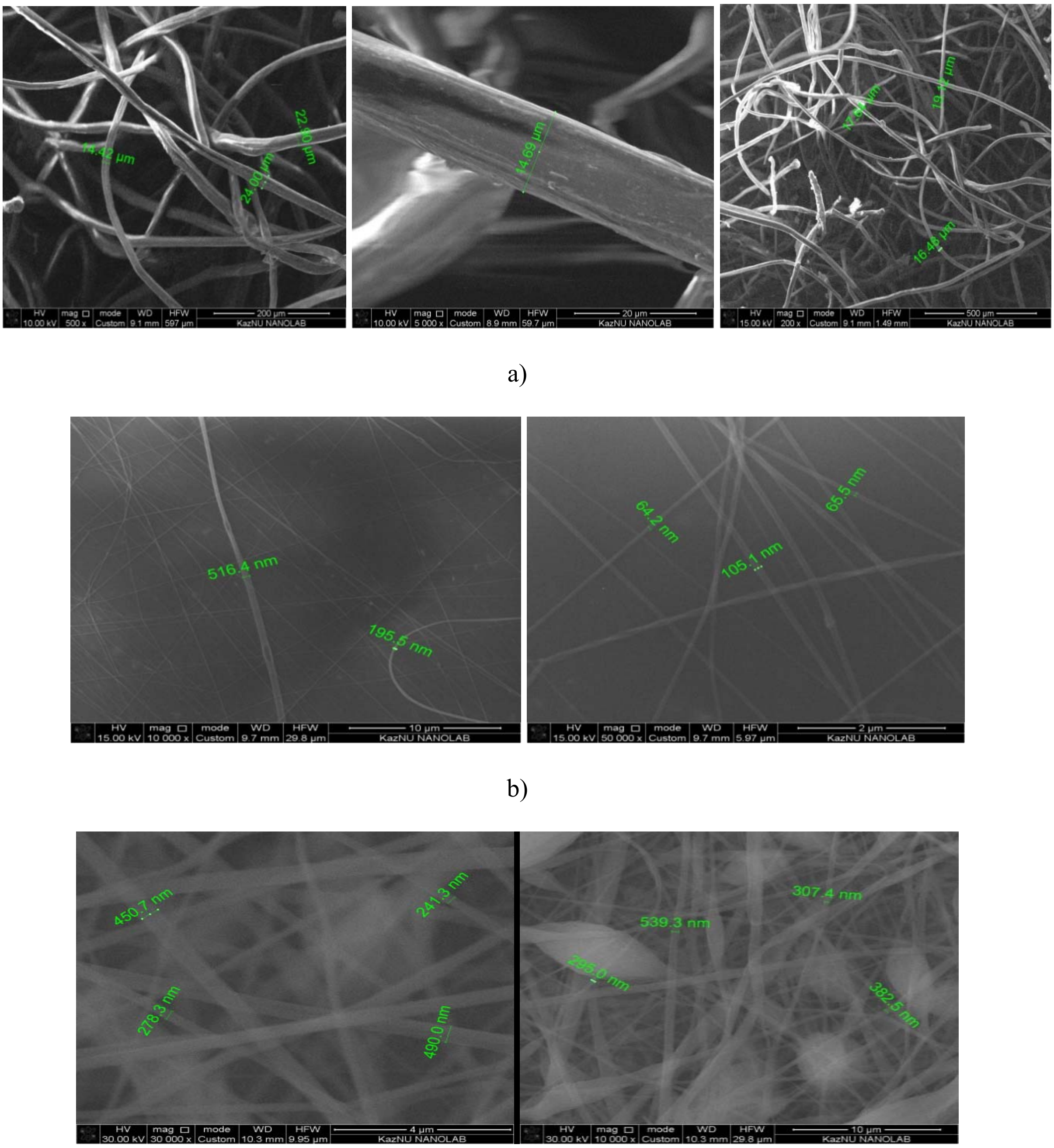
The elemental composition, structure, and dimension of the CNF were studied by energy dispersive X-ray spectroscopy on an SEM instrument (Quanta 3D 200i) with an attachment for energy dispersive analysis from EDAX. For the study, the samples were mounted on a copper holder using conductive adhesive paper. The energy of the exciting electron beam in the analysis was 15 keV, and the working distance was 15 mm. Humidity, ash content and volatility of the samples were determined on a thermogravimetric analyzer «Thermoster Eltra» (according to ASTM D7582-12).

Results and its discussion. The results of the elemental analysis, presented in table 1, show that after carbonization of the textile cord, most of the volatile components are removed in the form of gaseous products, respectively, the concentration of mineral components increases.

Table 1 – The chemical composition and physico-chemical characteristics of the initial, carbonized textile cord

Denomination	(W^r_t),n %	(A^r), %	(V^d), %	The content of elements, %										
				C	Zn	S	O	Na	Al	Si	Cu	K	Ca	Fe
Initial Textile Cord	-	5,09	75.82	67,9	3,8	0,3	13,9	0,4	0,9	0,7	4,4	-	1,1	6,6
Carbonized Textile Cord (400^{0}C)	0,75	18,73	36,38	69.5	4.3	1.3	3.6	-	0.4	1.1	-	0.5	1.3	4.9

The results of a scanning electron microscope of the initial textile cord, also obtained by CNF based on TC + PMMA and TC + CT + PMMA, are shown in figure 2.



a)

b)

c)

Figure 2 – Electron-microscopic images of the samples: a) initial TC:
b) CNF based on TC + PMMA; c) TC + CT + PMMA

In Figure 2 (a), the fibers of the initial textile cord with a diameter size of 14.42-24.00 µm are clearly visible. In SEM images of CNFs obtained on the basis of TC + PMMA (figure 2(b)), the fiber diameter ranged from 64.2 nm to 516.4 nm, and in Figure 2 (c) CNFs with a diameter slightly higher from 241.3 nm to 539.3 nm are visible. The structural elements of nanofibers take the form of fibrils - filamentary formations, the length of which exceeds their diameter by more than one order. The cylindrical surface of the fibers is formed by hexagons. Due to the existence of penta- and heptagons, structural defects, the formation of bridges and the curvature of the cylindrical surface are observed (Fig. 3 (c)). The CNF obtained on the basis of TC + PMMA compared with TC + CT+PMMA turned out to be more even.

The results of the elemental analysis of the CNF are presented in table 2.

Table 2 - The elemental composition of CNF from TC + PMMA and TC +CT + PMMA

CNF	C, %	O, %	Si, %	Al, %	S, %
TC + PMMA	82,69	1,72	0,46	15,14	-
TC +CT + PMMA	88,43	4,06	-	7,32	0,19

As can be seen from the obtained data, CNF based on TC + CT + PMMA has 5.74% more carbon.

Thus, we obtained carbon nanofibers based on textile cord and “Shubarkul” coal tar using the electrospinning method.

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

The proposed method is unique in that the raw materials that we use to produce carbon fibers are a renewable resource, compared with the technology for producing fibers from many other precursors (nylon, polyester, acrylic, polypropylene, etc.). The prospect of these studies lies in the possibility of large-scale production of carbon fibers from textile cord and coal tar, which will lead to the appearance on the Kazakhstan market of materials and composites based on them of domestic production.

Acknowledgement. This work was carried out as part of the scientific and technical program No. IRN BR05236359 «Scientific and technological support for coal processing and production of high-value coal products» and project No. IRN AP05130707 on the theme «Development of technology and creation of production of carbon nanocomposite materials based on domestic mineral raw materials for gas phase purification and wastewater», funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan.

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ТАСКӨМІР ШАЙЫРЫ МЕН ТОҚЫМА КОРД НЕГІЗІНДЕ КОМПОЗИТТІ КӨМІРТЕКТІ НАНОТАЛШЫҚ АЛУ

Аннотация. Мақалада электроспининг әдісі арқылы зертханалық қондырғыда тоқыма кордынан көміртекті наноталшықтар алу бойынша тәжірибелер көрсетілген. КНТ алуда тоқыма кордының карбонизаты (ТК), «Шұбаркөл» кен орны көмір шайыры (КШ) және полиметилметаакрилат (ПММА) қолданылды. КНТ алу үшін зертханалық жағдайда қолайлылығы себепті электроспиннинг әдісі қолданылды. Оның механикалық созу әдісінен артықшылығы үдерістің химиясына қатаң талап қойылмайды, талшықтың кебуі үшін жоғары температура қажет етпейді. Яғни, капиллярлық және электростатикалық күшінің нәтижесінде ұзын және күрделі молекулалардан талшық алуға мүмкіндік береді. Тоқыма корд негізінде наноталшық алу үшін үлгіні 400 ^{0}С температурада айналмалы түтік пеште аргон ортасында карбондау үдерісі жүргізілді. Алынған карбондалған тоқыма корды үгітіліп, ультрадыбыстық ваннада 35 ^{0}С температурада 30 мин. бойы 35 кГЦ жилікте 1,2-дихлорэтанда ерітілді. Полиметилметаакрилат (массалық үлесі 3 %) ерітіндісі де 1,2-дихлорэтанда ультрадыбыстық ваннадағыға ұқсас жағдайда дайындалды. Дайын ерітінділер 1:1 қатынаста араластырылып, ультрадыбыстық ваннада 35 ^{0}С температурада 40 мин бойы бірыңғай масса алынғанша ұсталды. Наноталшықты алу үшін арнайы ине тәрізді филлер қолданылады, диаметрі 0,6 мм. Оған жоғары вольтті кернеу 20-25 кВ беріледі. Фильерден ерітінді 5-10 мл/сағ жылдамдықта шығып, қабылдағышта қона бастайды. Филер мен коллектор арасындағы қашықтық 200-бен 300 мм арасын қамтиды. Арықарай ерітінді электроспиннинг аппаратына беріледі, онда ерітінді шприцке жиналып, сорғыш қозғалтқышқа (двигатель) орналастырылады, шприц ұшына заряд беріліп, двигатель қосылады, бірінші тамшының пайда болуына қарама-қарсы заряд қосылады. Бірдей зарядтар қарсы зарядталған коллекторға тартылып, наноталшық түзеді. Еріткіш ұшқан кезде наноталшық түзіліп, жоғары кернеу электротүзілудің негізі болып саналады. Композитті наноталшық алу үшін таскөмір шайыры мен тоқыма корды (1:1), байланыстырғыш ретінде полиметилметакрилат, ал еріткіш ретінде 1,2-дихлорэтан қолданылды, алынған қоспа ультрадыбыстық ваннада 35 ^{0}С температурада, 35 кГЦ жилікте, 30 мин. бойы ұсталды, алынған ерітіндіден электоспиннинг әдісімен наноталшық алынды. Энергодисперсиялық рентгенді спектроскопия талдау нәтижесінде және СЭМ көмегімен КНТ құрамы мен морфологиясы анықталды, ТК+ПММА негізінде алынған КНТ құрамы С-82,69%, О-1,72%, Si-0,46%, Al-15,14%, ал ТК+КШ+ПММА: С-88,43%, О-4,06%, Al-7,32%, S-0,19 % көрсетті, КНТ диаметрі 64,2-539,3 нм. ТК+ПММА негізінде алынған көміртекті наноталшықтың ТК+ КШ+ПММА негізінде түзілген наноталшыққа қарағанда диаметрі кіші, бірыңғай наноталшық түзілді.

Түйін сөздер: көміртекті наноталшық (КНТ), таскөмір шайыры, нанокомпозиттер, электроспиннинг, тоқыма корды, полиметилметакрилат.

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ПОЛУЧЕНИЕ УГЛЕРОДНЫХ КОМПОЗИТНЫХ НАНОВОЛОКОН НА ОСНОВЕ КАМЕННОУГОЛЬНОЙ СМОЛЫ И ТЕКСТИЛЬНОГО КОРДА

Аннотация. В статье приведены результаты эксперимента по получению углеродных нановолокон на основе текстильного корда методом электроспиннинга на лабораторной установке. Были получены углеродные нановолокна (УНВ) из карбонизата текстильного корда (ТК), каменноугольной смолы (КС) месторождения «Шубарколь» и полиметилметакрилата (ПММА). Нами выбран метод электроспиннинга для получения УНВ, так как этот метод является приемлемым в лабораторных условиях: его преимущество в том, что, в отличие от обычного, механического вытягивания волокон из раствора, он не предъявляет высоких требований к химии процесса, не требует больших температур для затвердевания волокна, а, значит, позволяет создавать волокна из длинных и сложных молекул в результате борьбы капиллярных и электростатических сил. Для получения нановолокон на основе текстильного корда проводился процесс карбонизации образца при 400^{0}С во вращающейся трубчатой печи, в среде аргона. Полученный карбонизованный текстильный корд измельчается и растворяется в 1,2-дихлорэтане в ультразвуковой ванне при 35 ^{0}С в течение 30 мин, частота – 35 кГЦ. Полиметилметакрилат также растворяется в 1,2-дихлорэтане (массовая доля 3 %) в ультразвуковой ванне в аналогичных условиях. Готовые растворы, полученные в предыдущих стадиях, смешиваются в соотношениях 1:1 в ультразвуковой ванне при 35 ^{0}С в течение 40 мин до получения однородной массы. Для получения нановолокна использовался фильер в виде иглы диаметром 0,6 мм. К фильеру прикладывалось импульсное высоковольтное напряжение 20-25 кВ. Из фильеры выдавливали раствор с растворителем со скоростью 5 - 10 мл/час и осаждали на подложку. Далее готовые растворы формируются в лабораторной установке электроспиннинга, для этого готовая смесь набирается в шприц и устанавливается на насосный двиагатель, на кончик шприца подается заряд, далее включается двигатель, и с появлением первой капли включается противоположный заряд. Одноименный заряд притягивается на подложку с противоположным зарядом и образуются нановолокна. Нановолокна отверждаются за счет испарения растворителя. Расстояние между приемником и шприцем составило 20-30 см. Высокое напряжение являяется основой электроформования. Для получения композитных углеродных нановолокон использовались каменноугольная смола и текстильный корд (1:1), а в качестве связующего – полиметилметакрилат, в качестве растворителя – 1,2-дихлорэтан, смесь продуктов также подвергают диспергированию в ультразвуковой ванне при температуре 35 ^{0}С, частота – 35 кГЦ, время выдержки – 30 мин, приготовленный раствор помещают в установку электроспиннинга со шприцем, установленное напряжение – 20-25 кВ. Определен элементный состав и изучена морфология поверхности исследуемых образцов. В результате энергодисперсионной рентгеновской спектроскопии и СЭМ-микроскопии были определены химические составы УНВ на основе ТК+ПММА, который составил С-82,69%, О-1,72%, Si-0,46%, Al-15,14%, ТК+КС+ПММА: С-88,43%, О-4,06%, Al-7,32%, S-0,19 %, диаметр углеродных нановолокон составил от 64,2 нм до 539,3 нм. УНВ, полученные на основе ТК+ПММА, по сравнению ТК+ КС +ПММА, получились более ровными и с меньшим диаметром нановолокон.

Ключевые слова: углеродные нановолокно, каменноугольная смола, нанокомпозиты, электроспиннинг, текстильный корд, полиметилметакрилат.

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ISSN 2518-1491 (Online), ISSN 2224-5286 (Print)

Редакторы: *М. С. Ахметова, Г. Б. Халидуллаева, Д. С. Аленов*
Верстка на компьютере *А.М. Кульгинбаевой*

Подписано в печать 08.06.2020.
Формат 60x881/8. Бумага офсетная. Печать – ризограф.
10,8 п.л. Тираж 300. Заказ 3.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-18, 272-13-19