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SYNTHESIS OF COMPOSITE MEMBRANE BASED ON GRAPHENE OXIDE AND NANOSTARCH

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Intensive research of nanocomposites contributes to the development of new materials in the fields of medicine, nanoelectronics, high dielectric materials, energy, biotechnology and information technology. Therefore, the synthesis of new materials by modifying of grapheme oxide with nanostarch and the study of its properties are of great interest. In this study, nanocomposite membrane was synthesized by modifying of graphene oxide in a 1:1 volume ratio with nanostarch and their chemical structures were studied by fourier-transform infrared spectroscopy and ultraviolet spectroscopy. The results of the study showed the absorption of the etheric bond C=O in the ultraviolet spectrum at full length was 243 nm. The infrared spectrum showed new etheric bonds O=C-OH at a wavelength of 1150 cm^{-1}. The scanning electron microscopy analysis indicates that grapheme oxide is homogeneously coated by starch, it means that nanostarch was successfully polymerized on the surfaces of graphene oxide nanosheets and formed sandwich-like structures. The X-Ray diffraction analysis indicatedthat the crystalline structure changes to amorphous to a certain extent due to the ester bond formed between graphene oxide and starch. The electrical properties showed the resistivity of the initial graphene oxide was $\rho = 5.53 \cdot 10^3$ Ohm·m and after its modification by starch, the resistivity of the graphene oxide/starch membrane increased by 2 orders, which was equal to $2.59 \cdot 10^5$ Ohm·m. According to the results, high dielectric materials are very important in electrical and electronics engineering. Therefore, there is no doubt that there will be great interest in this field.

Keywords: *graphene oxide, nanostarch, dielectric materials, nanocomposite membrane, resistivity.*

1 INTRODUCTION

Currently, the use of composite materials based on graphene oxide (GO) as a sensitive element of moisture and gas sensors, as sorbents for cleaning water from heavy metal ions and various polluting compounds is being actively researched [1, 2].Some studies have reported the use of chemically modified GO in the treatment of cancer and as a tool for cancer gene therapy based on ribonucleic acid interference [3, 4].

According to the results of many recent studies, it has been established that modification of GO with biopolymers allows obtaining new materials with the most improved properties [5]. Among different biopolymers, starch is very appealing because of its cost effectiveness and easy availability from different natural sources such as vegetables and cereals. Besides the nourishing value, the film forming ability has given to starch a more remarkable role, for example in food packaging. Films based on starchare biodegradable and edible therefore, potential substitutes for many synthetic packaging materials and pose negligible harm to the environment. Unfortunately, the starch-based biocomposites have many disadvantages (e.g. strong hydrophilic character, poor mechanical properties and low thermal stability) compared to conventional polymers, which make it unsatisfactory for practical applications [6–8]. Moreover, the utilization of nano-sized fillers can also increase dispersibility in polymer resin and lead to improved mechanical, thermal and dielectric properties [9–10]. [11] investigated the preparation of antibacterial GO/starch hydrogel using 2-hydroxyethyl methacrylate as a cross-linking agent.

The presence of oxygen functional groups in the chemical structure of GO provides a great opportunity for surface modification of it [12]. In this regard, modification of GO with starch makes it possible to obtain eco friendly, biodegradable, thermally stable, electroconductive and improved films with improved mechanical properties [13–15]. Therefore, obtaining new composite materials by modifying graphene oxide with starch, is attracting the attention of world scientists.

Thus, the purpose of this research work was to modify GO with nanostarch by ultrasonic treatment. The physico-chemical properties of obtained GO/starch composite membrane are studied during the modification.

2 EXPERIMENTAL SECTION

2.1 Materials

Potassium permanganate (State standard (STST) 5777-84), sodium nitrate (STST 4168-79), sulfuric acid (STST 4204-77), hydrochloric acid (STST 3118-77), starch (STST 7699-78), graphite (STST 17022-81), ethanol (STST 5962-2013), deionized water (STST 52501-2005).

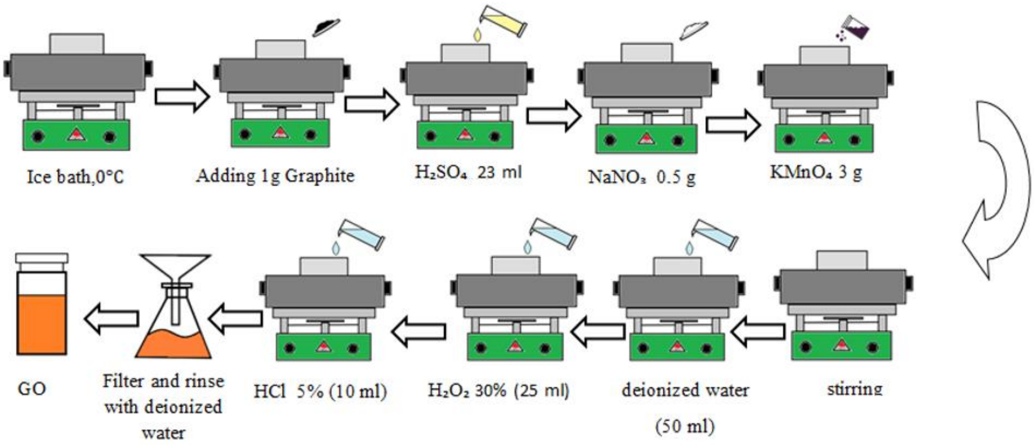

Figure 1. The GO synthesis scheme

2.2 Methods

2.2.1 Synthesis of GO

Graphene oxide was synthesized based on the Hammers method used in the research [16]. In order to do this, a beaker with 1 g of graphite was placed in a container filled with ice at a temperature equal to 0 ºC, then 23 ml of 94% H_2SO_4 was poured, after that left for 15 min and was continuously stirred on a magnetic stirrer. $NaNO_3$ (0.5 g) was added to the obtained suspension, 3 g of $KMnO_4$ was gradually added to it with continuous stirring for 15 min and it was further stirred for 2 hours. Then the temperature was raised to 35 °C and held for 30 minutes. Further, was added 30 ml of deionized water, the temperature was raised to 90 °C and stirring continued for 30 minutes. Finally, 30% $H_2O_2$was added until the color of the mixture changed to light yellow. After the reaction, the product was filtered with filter paper and washed several times with 5% HCl to remove residual metal ions. Finally, deionized water was neutralized by a centrifuge (Centrifuge 5427R Eppendorf) until pH is equal to 7 (Figure 1).

2.2.2 Synthesis of nanostarch

Based on the method described in [17], nanostarch has been isolated from starch by precipitation with ethyl alcohol. The nanostarch suspension was obtained by centrifugation (Centrifuge 5427R Eppendorf) from the initial suspension (Figure 2).

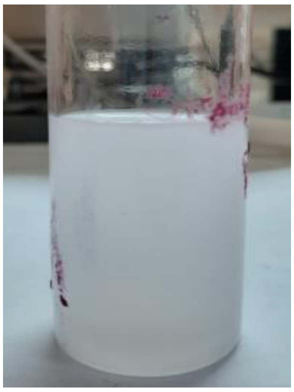

Figure 2. The water suspension of nanostarch

2.2.3 Synthesis of composite membrane based on GO/nanostarch

The membrane based on GO/nanostarch was synthesized, which shown in the scheme Figure 3. First, 1% GO water solution and 1% nanostarch water solution were prepared. The prepared solutions were mixed for 30 min in a magnetic stirrer in a volume ratio of 1:1. The resulting solution was processed in an ultrasonic machine UZTA-0.15/22-0 (Alena, made in Russia) at 45 kHz and 40 ºC for 30 min. The treated solution was poured into a disc-shaped plastic container and dried for 90 min in a drying cabinet at 50 ºC.

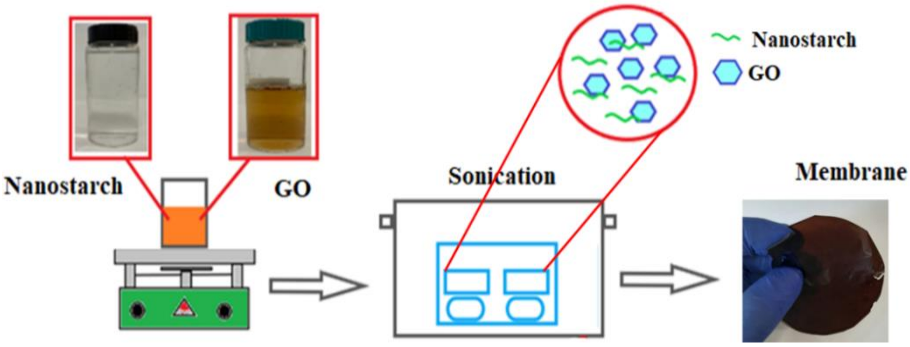

Figure 3. The GO/nanostarch membrane synthesis scheme

2.2.4 FTIR analysis

FTIR analysis of chemical structures of GO and GO/nanostarch membrane were performed on a spectrometer FT-801 (Simex, made in Russia), with resolution 1 cm^{-1}. Measurements were conducted in the region between 450–4700 cm^{-1} according to the standard method using a single-use universal full internal and mirror-diffuse reflection with the upper position of the model, at a temperature 25 °C.

2.2.5 X-ray diffraction

The crystal structures of GO and GO/nanostarch membrane were studied via X-ray diffraction on a X´PertPRO diffractometer (Malvern Panalytical Empyrean, made in Netherlands) using monochromatized copper (CuKα), at a scan speed of 0.05º for 10 s, with a K-Alpha1 wavelength of 1.54187 Å. Measurement in reflection mode, using an aluminium rectangular multi-purpose sample holder (PW1172/01), was performed at a diffraction angle 2θ between 10° and 40°, with the X-ray tube voltage at 45 kV, current intensity at 30 mA, and a measurement time of each step 0.5 s.

2.2.6 SEM analysis

The surface morphology of GO/nanostarch membrane was studied by scanning electron microscope JSM-6390LV (Jeol, made in Japan). Measurements were carried out in high vacuum mode using a secondary electron detector at an accelerating voltage of 15 kV. The surface of the nanostarch film is coated with gold to improve the transfer of electrons. The specimens were mounted on aluminum pins with carbon tape.

2.2.7 Electrical Characterization

The electrical properties of GO and GO/nanastarch membrane were investigated by four-point probes method. Electrical measurements were made using the traditional four-probe method, with current measured on a Keithley 6485 (made in USA) and voltage applied with a Tektronix PWS2326.

3 RESULTS AND DISCUSSION

3.1 UV analysis of nanostarch

Figure 4 shows the UV absorption spectrum of nanostarch. New absorption peaks were not observed in the obtained spectrum. It means that during the synthesis of nanostarch there was not oxidation process and the dispersibility of the aqueous solution was very good. We saw that similar results were obtained in [18].

3.2 FTIR analysis

Figure 5 shows the FTIR spectra of GO, nanostarch and GO/nanostarch membrane. In the spectrum of GO obtained by Hammer's method, the OH group at 3250 cm^{-1} and 1650 cm^{-1}, the carbonyl and carboxyl C=O group at 1750 cm^{-1}, and the C=C bonds of the aromatic ring at 1450 cm^{-1} are observed. Vibration of epoxide functional groups at 1300 cm^{-1} C-O, 1050 cm^{-1} is characteristic of alkoxy bonds [16]. The nanostarch spectrum shows the spectrum of nanostarch at 2850 cm^{-1} CH_2 band, 1430 cm^{-1}group assigned to a symmetric CH_2 bending vibration, 1150 cm^{-1} CO band and at 898 cm^{-1}, assigned to C–O–C stretching [18].

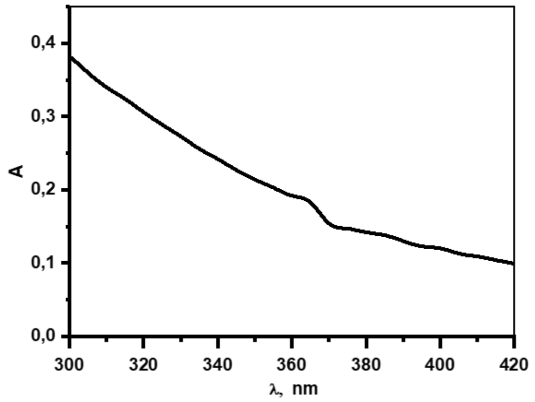

Figure 4. UV spectrum of the nanostarch

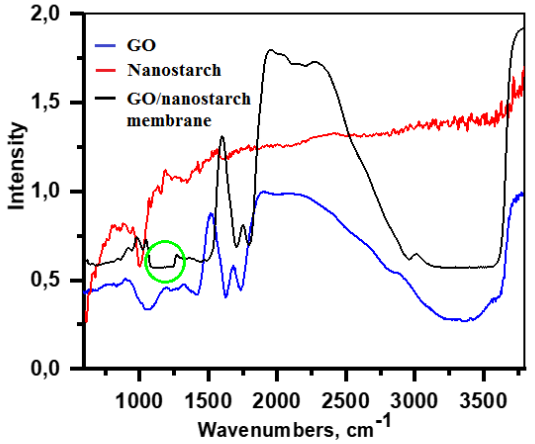

Figure 5. The FTIR spectrum of the GO, nanostarch and GO/nanostarch membrane

And on the GO/nanostarch membrane, OH group at 3250 cm^{-1} and 1650 cm^{-1}, CH group at 2850 cm^{-1}, carbonyl and carboxyl C=O group at 1750 cm^{-1}, aromatic ring C=C bonds at 1450 cm^{-1}. Longitudinal vibrations and deformation peaks, vibrations of epoxide functional groups at 1300 cm^{-1} C-O, 1050 cm^{-1} C-O showed alkoxy bonds [18, 19].The wave number of the absorption region 1150 cm^{-1} showed that it is characteristic of the ether bond formed between the O=C-OH carboxyl group in the GO molecule and the OH groups in the starch molecule [20].This means the formation of a new chemical bond between GO and nanostarch.

3.3 XRD analysis

Figure 6 shows the changes of crystal structures of GO and GO/nanostarch membrane. They were characterized by XRD diffraction peak detected from X-ray diffractogram. The 2θ = 10.5° corresponds to the characteristic peak of GO (001) lattice plane [16, 21]. Additionally, in the GO/nanostarch membrane diffractogram exhibited the crystal plane of GO/nanostarch membrane at 2θ = 13°. This showed that due to the ester bond between the O=C-O carboxyl group in the GO molecule and the OH group in the starch molecule, the crystal structure changed to a certain amount of amorphous form, and the 2θ value decreased.

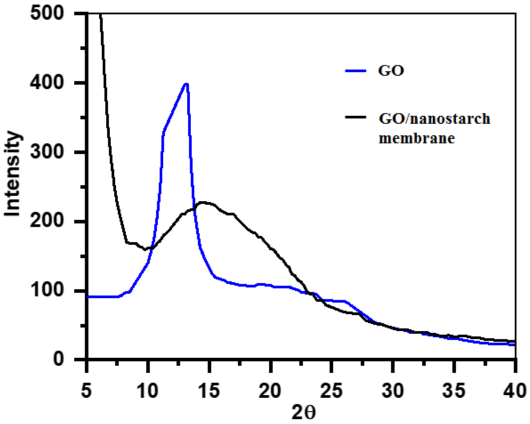

Figure 6. The XRD of the GO and GO/nanostarch membrane

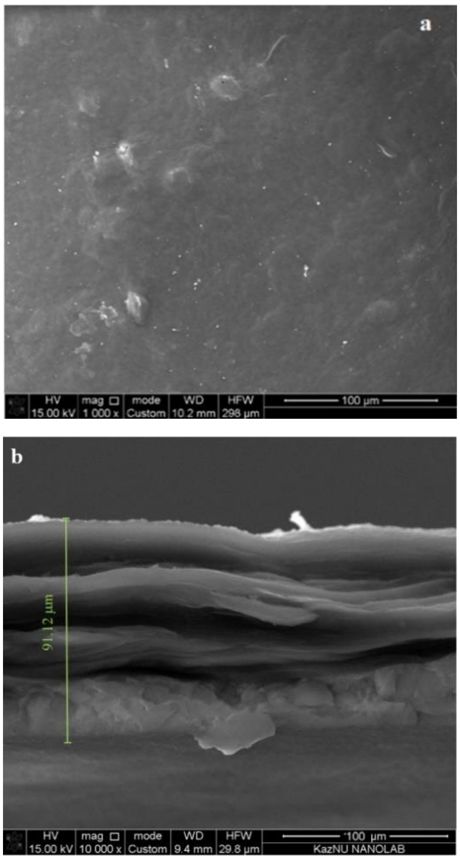

Figure 7. The SEM of GO/nanostarch membrane

3.4 SEM analysis

Figure 7 shows the surface morphology of GO/nanostarch membrane (a) and side micrograph (b). The thickness of the membrane was about 75 µm. The surface of the membrane is smooth and in some parts particles formed by the accumulation of nanostarch were observed (Figure 7a). A layered structure caused by the incorporation of GO was clearly observed in the cross section of the membrane (Figure 7b). It is clear that the GO is homogeneously coated by starch, indicating that nanostarch was successfully polymerized on the surfaces of GO nanosheets to form sandwich-like structures. The cross-section of GO/nanostarch membrane presents a layer-by-layer formation, which is probably caused by the flow assembly effect of GO sheets.

3.5 Electrical Characterization

The electrical characterization of the GO and GO/starch membrane was carried out using Keithley Picoammeter (Model 6485) by four-point probes method. According to the obtained results, the resistivity of the initial GO was $\rho = 5.53 \cdot 103$ Ohm·m and after its modification by starch, the resistivity of the GO/starch membrane increased by 2 orders, which equal to $2.59 \cdot 10^5$ (Figure 8). The increase in the dielectric constant of the composite with the GO is attributed to interfacial polarization occurring at the interface between the nanostarch and GO [22, 23]. In the present study, we can suppose interfacial and space charge polarization in the GO loaded matrix. GO contains polar functional groups, like –OH, –CHO, –CO, and –COOH, which ensures good bonding and appropriate local electrical contacts that may lead to a strong interfacial polarization and play a role in enhancing dielectric constant, whereas, this polarization in the pure sample may be due to the immiscible phase of the polymer matrix. The spatial polarization arising from the trap states results an accumulation of charge carriers and leading to an increase of the value of dielectric constant [22, 24].

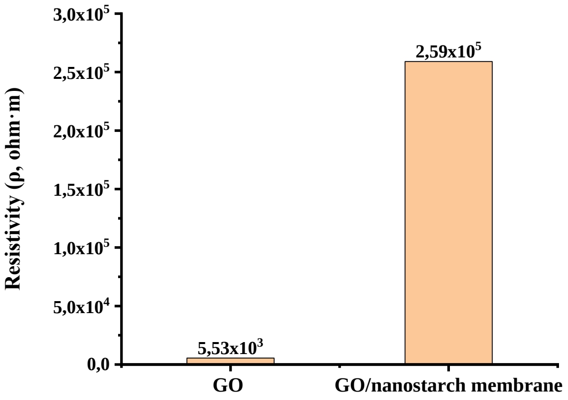

Figure 8. The electrical characterization of the GO and GO/starch membrane

4 CONCLUSION

Synthesized nanocomposite membrane by modifying of graphene oxide (GO) in a 1:1 volume ratio was obtained. Their chemical structure was studied by FTIR and UV-spectroscopy. The results of the study showed the absorption of the etheric bond C=O in the UV spectrum at full length 243 nm. The FTIR spectrum showed all the new etheric bonds O=C-OH at a wavelength of 1150 cm^{-1}. The SEM analysis indicating the GO is homogeneously coated by starch, indicating that nanostarch was successfully polymerized on the surfaces of GO nanosheets to form sandwich-like structures. The XRD analysis indicated this showed that the crystalline structure changes to amorphous to a certain extent due to the ester bond formed between GO and starch. The electrical properties showed the resistivity of the initial GO was $\rho = 5.53 \cdot 10^3$ Ohm·m and after its modification by starch, the resistivity of the GO/starch membrane increased by 2 orders, which equal to $2.59 \cdot 10^5$ Ohm·m. Obtained composite is dielectric and from the above results, we can assume that these membranes can be used in the field of electronics.

Conflict of interests

On behalf of all authors, the corresponding author states that there is no conflict of interest.

Acknowledgment

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ГРАФЕН ОКСИДІ МЕН НАНО-КРАХМАЛ НЕГІЗІНДЕГІ КОМПОЗИТТІК МЕМБРАНАНЫҢ СИНТЕЗІ

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Нанокомпозиттерді қарқынды зерттеу медицина, наноэлектроника, жоғары диэлектрлік материалдар, энергетика, биотехнология және ақпараттық технологиялар салаларында жаңа материалдарды дамытуға ықпал етеді. Сондықтан графен оксидін нанокрахмалмен модификациялау арқылы жаңа материалдарды синтездеу және оның қасиеттерін зерттеу үлкен қызығушылық тудырады. Бұл зерттеуде нанокомпозиттік мембрана графен оксидін 1:1 көлемдік қатынасында нанокрахмалмен модификациялау арқылы синтезделді және олардың химиялық құрылымдары инфра қызыл және ультра күлгін-спектроскопия арқылы зерттелді. Зерттеу нәтижелері ультракүлгін спектрдегі C=O эфирлік байланысы 243 нм толық ұзындықтағы жұтылуын көрсетті. Инфра қызыл спектрі толқын ұзындығы 1150 см$^{-1}$ болатын O=C-OH жаңа эфирлік байланыстарды көрсетті. Сканерлеуші электрондық микроскоп талдауы графен оксидін крахмалмен біртекті қапталғанын көрсетеді, бұл нанокрахмал графен оксиді нанопарақтарының беттерінде сәтті полимерленіп, сэндвич тәрізді құрылымдар түзілгенін білдіреді. Рентген дифрактометрі талдауы графен оксиді мен крахмал арасында түзілген күрделі эфирлік байланыстың арқасында белгілі бір дәрежеде кристалдық құрылымнан аморфтыға өзгеретінін көрсетті. Электрлік қасиеттері бастапқы графен оксиднің меншікті кедергісі $\rho = 5{,}53 \cdot 10^3$ Ом·м және оны крахмалмен түрлендіруден кейін графен оксиді/крахмал мембранасының кедергісі 2 ретке өскенін көрсетті, ол $2{,}59 \cdot 10^5$ Ом·м-ге тең болды. Зерттеу нәтижелері бойынша жоғары диэлектрлік материалдар электротехника және электроникада өте маңызды. Сондықтан бұл салаға деген қызығушылықтың зор болатыны сөзсіз.

Түйін сөздер: *графен оксиді, нанокрахмал, диэлектрлік материалдар, нанокомпозиттік мембрана, меншікті кедергі.*

СИНТЕЗ КОМПОЗИЦИОННОЙ МЕМБРАНЫ НА ОСНОВЕ ОКСИДА ГРАФЕНА И НАНОКРАХМАЛА

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Интенсивные исследования нанокомпозитов способствуют разработке новых материалов в области медицины, наноэлектроники, материалов с высокой диэлектрической проницаемостью, энергетики, биотехнологий и информационных технологий. Поэтому синтез новых материалов путем модификации оксида графена нанокрахмалом и изучение его свойств представляют большой интерес. В данной работе синтезированы нанокомпозитные мембраны путем модификации оксида графена в объемном соотношении 1:1 нанокрахмалом и изучена их химическая структура методами инфракрасной и ультрафиолетовой спектроскопиями. Результаты исследования показали, что поглощение эфирной связи C=O в ультрафиолетовом спектре на полной длине составило 243 нм. В инфракрасном спектре обнаружены новые эфирные связи O=C-OH при длине волны 1150 см$^{-1}$. Анализ с помощью сканирующей электронной микроскопии показывает, что оксид графена однородно покрыт крахмалом, это означает, что нанокрахмал успешно полимеризовался на поверхности нанолистов оксида графена и образовал сэндвич-подобные структуры. Рентгенофазовый анализ показал, что кристаллическая структура в определенной степени изменяется на аморфную из-за образования сложноэфирной связи между оксидом графена и крахмалом. Электрические свойства показали, что удельное сопротивление исходного оксида графена составило $\rho = 5{,}53 \cdot 10^3$ Ом·м, а после его модификации крахмалом удельное сопротивление мембраны оксид графена/крахмал увеличилось на 2 порядка и составило $2{,}59 \cdot 10^5$ Ом·м. Согласно результатам, материалы с высокой диэлектрической проницаемостью очень важны в электротехнике и электронике. Поэтому нет никаких сомнений в том, что интерес к этой области будет большим.

Ключевые слова: *оксид графена, нанокрахмал, диэлектрические материалы, нанокомпозитная мембрана, удельное сопротивление.*