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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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NEWS

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OF THE REPUBLIC OF KAZAKHSTAN
Kazakh national research technical university
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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences 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 geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

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. Web of 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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PREPARATION OF CARBON NANOCOMPOSITES ON THE BASIS OF SILICON-TIN CONTAINING SUBSTANCES

Abstract. The obtained nanocomposites are tin dioxide nanoparticles immobilized on inorganic polymeric network of silicon dioxide and precipitated carbon. Wherein, silicon dioxide provides high adhesion properties and prevents the aggregation of the crystallites of the composite, and the nano- and microparticles of tin dioxide and carbon predetermine the gas-sensitive properties. The film-forming solutions obtained by sol-gel technology contains tetraethoxysilane, tin salts ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$), silicic acid ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) and sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$). The resulting solutions have been applied to metal substrates with subsequent heat treatment. The additional introduction of tetraethoxysilane as a source of SiO_2 in solution-sol has made it possible to obtain xerogels of oxide compositions of 5% SnO_2 – 95% SiO_2 , 30% SnO_2 – 70% SiO_2 . The modification of the content of elements shows that the growth of carbon nanoparticles changes the microstructure of the obtained compound.

Key words: nanocomposites, nanoparticles, silicon-tin containing nanotubes.

Introduction. The priority task of the state economic policy and science is the integrated and rational use of mineral raw materials, combating the negative impact of industrial waste and harmful emissions on the environment. With the involvement of phosphate concentrate obtained on the basis of waste into the production of complex mineral fertilizers, an additional 540 thousand tons of highly concentrated fertilizers sold on the international market at a price of at least \$400 per ton can be obtained [1-4].

At the present time, waste stored in the sludge accumulators of the chemical and phosphorous industry under the open air is exposed to wind and water erosion, which creates unfavorable environmental conditions for nearby areas: removal of dust containing silicon oxide from the dried surface of the sludge collector, flushing fluorine-containing salts from the rainwater waters into the underground waters. The second product of waste processing is a cement clinker. The main components of the clinker are calcium oxide (CaO), silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3), the total content of which reaches 95-97.5%. With the full complex processing of accumulated waste, the ecological problem of environmental pollution with toxic substances will be solved.

Materials obtained on the basis of carbon nanotubes or with the addition of SiO_2 can differ in a wide variety of functional properties. The nanoscale effect of particles of carbon nanotubes is manifested in the unusual properties of materials completely different from the known properties of bulk materials:

- optical, photonic and electronic (glass tubes for optical fibers, color coatings for automotive glasses, photochromic glasses, liquid crystal displays, photonic crystals, solar batteries, gas analyzers, etc.);
- thermal (refractory ceramics, ceramics with low coefficient of thermal expansion, fireproof coatings, thermal insulators);
- mechanical (high-strength polymeric products and ceramics, abrasives, tribological materials);
- chemical (catalysts, membranes, hydrophilic and hydrophobic films, antioxidants);

- biomedical (immobilization of biological molecules, such as proteins, microorganisms, antibacterial substances, biosynthesis, creation of biosensors, removal of toxic organic substances).

The introduction of silica nanoparticles can significantly improve the properties of well-known materials, for example, concrete soil, cement, ceramics, wood.

Methods. Finely divided silica is a light gray or white powder obtained from quartz. It is produced both in Europe and in the USA and it has various product names such as "SSA-1" Halliburton Services "D-66" from "Dowell Schlumberger" or "D-8" from "B.J. Hughes" [5-8].

In the production of glasses, ceramics, mixed inorganic oxides, gels are heated to several hundred degrees to sinter the particles and compress the porous structure to a dense material. In sol-gel technology, the term "sol" refers to colloidal ultradispersed and microheterogeneous systems with a liquid dispersion medium and a solid dispersed phase. Colloidal sols are aggregatively stable disperse systems of various compounds in water (hydrosols of silica, silver, boehmite, titanium dioxide, metal salts), obtained by condensation or dispersive methods. Thus, as a result of the polycondensation of aqueous solutions of silicic acids, silica hydrosols are obtained, which have extremely wide application [9].

According to modern concepts, the formation of a gel begins with the formation of a fractal structure of the sol, the growth of fractal aggregates to the extent that they begin to collide and adhere to each other, as percolation theory (percolation theory) describes. Near the gel point, randomly located neighboring clusters consisting of polymers or aggregates of particles join together to form a single structural grid. The gel point corresponds to the percolation threshold, when a single constricting cluster is formed, as it were spread throughout the volume of the sol [11-13]. After passing the gel point, the sol loses its mobility and gelifies, transforming into a "wet gel", since the liquid phase is retained in the spatial structure. Wet gel usually takes the form of the vessel in which the sol was. The formation of the gel does not stop at the gel point, for some time the aging of the gel occurs. The term "aging gel" reflects structural changes occurring after the gel point in the wet gel. In the resulting product, a single giant cluster coexists with a sol containing many small clusters that continuously join the common core - a giant cluster. In addition, in gels, polycondensation reactions that have not passed to the end in sols can be continued, accompanied by syneresis (release of water); there are also processes of reprecipitation of monomers or oligomers; there are also phase transitions of the "solid-liquid" type, as well as the condensation of the structure [14-17].

The third stage of sol-gel technology is drying, i.e., removing liquid from the spatial structure of the gel, resulting in the formation of a xerogel (dried gel). The xerogel volume is 5-10 times less than the volume of the wet gel. When removing free water from the gel, wetting capillary menisci are formed, which leads to an increase in pressure and cracking of the structure. To reduce the capillary pressure, the gel drying is desirably carried out in a vacuum, in the presence of surfactants (surfactants). Drying in supercritical conditions provided in autoclaves allows obtaining porous aerogels [18-20].

In this scientific work, nanoparticles of silicon oxide with tin chloride and sodium silicates with tin chloride sol-gel method were synthesized. The composition of obtained products was determined by chemical, IR-spectroscopic and microstructural X-ray phase analysis. Growth of nanomaterials was carried out on the carbon nanotube CVD method. The investigated silicon-tin nanostructured vitreous substances were deposited from a carbon nanotube of a columnar shape on a metal substrate using the unique technology of ULVAC JAPAN, Ltd., Japan Manufacturing [8, 13,21].

Results and discussion. The obtained nanocomposites were investigated by chemical and physico-chemical methods. Table 1 shows the results of the chemical analysis.

Table 1 shows that with increasing solution concentration the silicon content in silicates increases, the chlorine ion decreases, and the content of tin gradually increases. The results obtained make it possible to convert liquid solutions into gel-like liquid glasses, which can be used as semiconductors.

Research on an IR spectrophotometer allows a more accurate determination of the desired components, which gives a huge advantage over the chemical method of analysis of solutions. Figure 1 shows the IR spectra of compounds obtained by mixing 0.1 N Na_2SiO_3 and SnCl_2 . It can be seen that the strongly pronounced peaks in the 767 cm^{-1} region correspond to tin oxide- SnO_2 , in the region of 958 cm^{-1} there correspond silicon oxide- SiO_2 , and also in the region 1153 cm^{-1} and 2667 cm^{-1} , the slopes corresponding to tin oxide - SnO_2 , weakly expressed peak in the region of 4386 cm^{-1} corresponds to sodium oxide - Na_2O .

Figure 2 shows the IR spectra of products obtained from 0.5 N solutions of SnCl_2 and SiO_2 . A strongly pronounced peak in the 802 cm^{-1} region corresponds to silicon oxide- SiO_2 , weakly expressed peaks in

Table 1 – Results of chemical analysis of initial materials

No	Name of composites	Determination of silicon oxide content in silicates by weight method, %	Determination of the chlorine-ion content by a mercurimetric method, %	Determination of sodium content in bulk method, %	Determination of tin content by weight, %	Determination of the refractive index of a solution
1	0,1 n $\text{Na}_2\text{SiO}_3\text{-SnCl}_2$	31,75	17,129	3,68	84,9	1,3342
2	0,3 n $\text{Na}_2\text{SiO}_3\text{-SnCl}_2$	59,33	16,5387	2,17	89,9	1,3354
3	0,5 n $\text{Na}_2\text{SiO}_3\text{-SnCl}_2$	49,36	10,0413	5,43	91	1,3355
4	0,1 n $\text{SiO}_2\text{-SnCl}_2$	44,55	18,9013	1,69	59,2	1,3362
5	0,3 n $\text{SiO}_2\text{-SnCl}_2$	31,247	21,8547	2,5	69,6	1,3378
6	0,5 n $\text{SiO}_2\text{-SnCl}_2$	30,048	15,948	8,9	87	1,3440

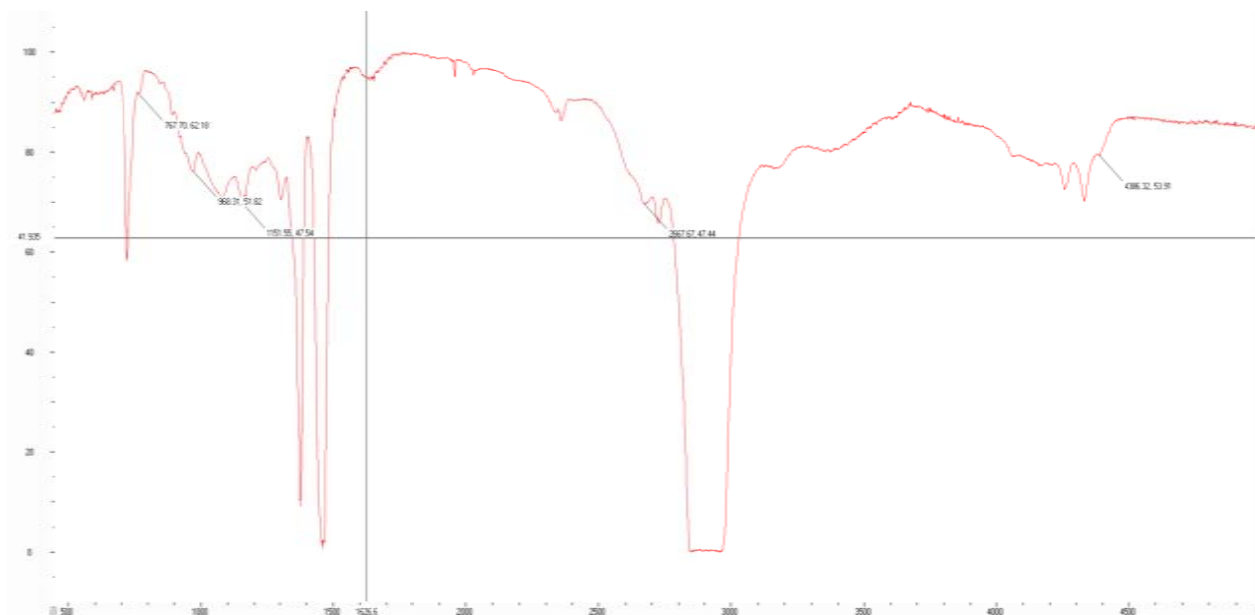


Figure 1 – IR spectra of the resulting compounds of 0.1 N $\text{Na}_2\text{SiO}_3\text{-SnCl}_2$ solution

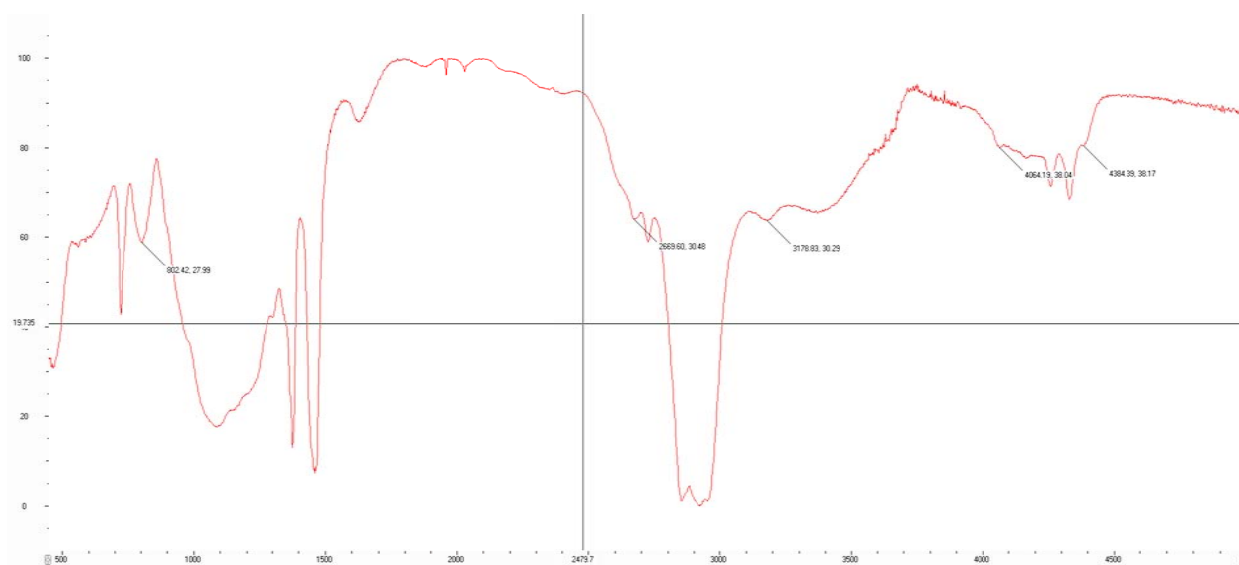


Figure 2 – IR spectra of the resulting compounds of a 0.5 N solution of $\text{SnCl}_2\text{-SiO}_2$

the 2669 and 3178 cm^{-1} region correspond to SnO_2 tin oxide, a weakly pronounced peak at 4064 cm^{-1} corresponds to silicon oxide- SiO_2 .

A scanning electron microscope (SEM) is simpler and more versatile for practical applications. The principle of the JEOL JSM-6490LV raster low-vacuum electron microscope (Japan) is based on the use of certain effects arising from the irradiation of the surface of objects by a finely focused beam of electrons. These effects are the basis for obtaining a variety of information: the relief of the surface of the sample, the chemical composition and the crystallographic orientation of the volumes adjacent to the surface. Electrons emitted by the substance, various kinds of radiation, are captured by special sensors and after amplification are used to control the brightness of the cathode ray tube on which the image is formed. At the same time, a certain point on the screen of the cathode-ray tube corresponds to each point on the surface of the sample [4, 5].

Figure 3 shows microstructure images of products obtained from 0.1 N Na_2SiO_3 and SnCl_2 (initial) (a) and 0.3 N SnCl_2 - SiO_2 solutions (initial) (b).

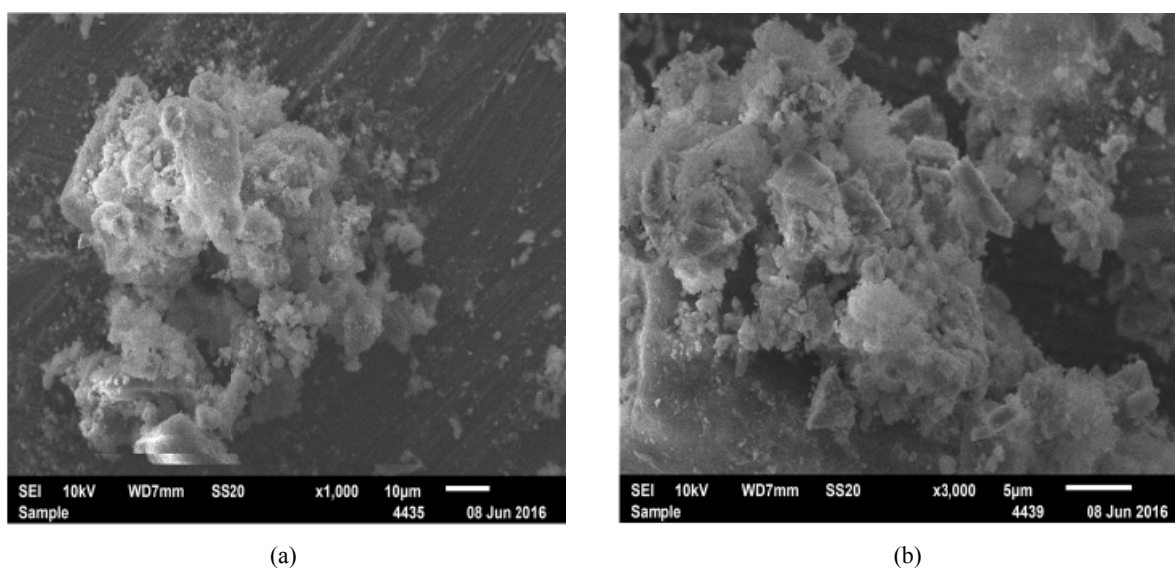


Figure 3 – Microstructure of nanocomposites obtained from Na_2SiO_3 - SnCl_2 (a), microstructure of nanocomposites obtained from SnCl_2 - SiO_2 (b)

Table 2 – Results of X-ray phase analysis of products synthesized from 0,1 N Na_2SiO_3 - SnCl_2 solution

Results of elemental analysis in weight, % ratios									
Range	In stats.	O	Na	Al	Si	S	Cl	Sn	Total
Spectrum 1	Yes	52,57	0,09	0,05	27,65	0,06	0,61	18,97	100
Spectrum 2	Yes	52,57	0,11	0,04	27,55	0,04	0,64	19,05	100
Spectrum 3	Yes	53,14	0,16	0	29,56	0,05	0,69	16,39	100
Average	Yes	52,76	0,12	0,03	28,25	0,05	0,65	18,14	100

Table 3 – Results of X-ray phase analysis of products obtained from 0.3 N SnCl_2 - SiO_2

Results of elemental analysis in weight,% ratios							
Range	In stats.	O	Al	Si	Cl	Sn	Total
Spectrum 1	Yes	41,82	0,12	10,99	4,07	43,01	100
Spectrum 2	Yes	44,17	0,05	15,34	3,62	36,82	100
Spectrum 3	Yes	44,06	0,07	14,39	3,69	37,78	100
Spectrum 4	Yes	41,72	0,04	11,09	4,19	42,95	100
Average		42,94	0,07	12,95	3,89	40,14	100

Table 3 shows that the elemental analysis reduces the oxygen content, the content of tin and silicon increases. The change in the content of elements shows that with the growth of the nanoparticles the microstructure of the obtained compound changes.

In this work, composites based on $\text{SiO}_2\text{-SnCl}_2$ and $\text{Na}_2\text{SiO}_3\text{-SnCl}_2$ with different tin dioxide content were obtained by sol-gel technology on substrates of oxidized single-crystal silicon, further tablets are prepared from the obtained products. The finished tablets allows the carbon deposition in columnar-shaped carbon nanotubes on a metal substrate using the unique technology of ULVAC JAPAN, Ltd. The precursors for the preparation of sols are tetraethoxysilane, five-water tin tetrachloride, ethyl alcohol; the catalyst is hydrochloric acid. The firing was carried out at a temperature of 600 °C.

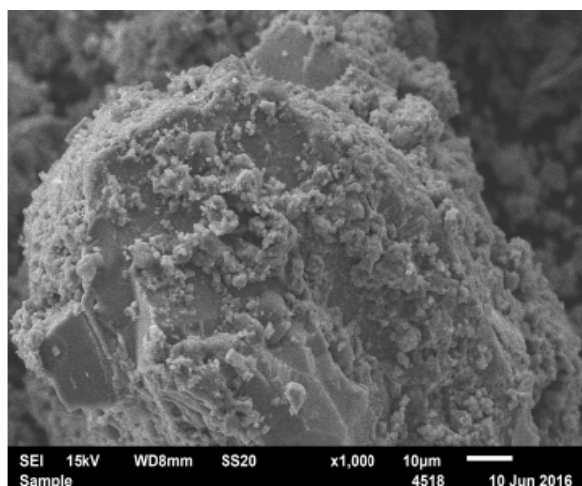


Figure 4 – Microstructure (SEM) of nanocomposites obtained from $\text{Na}_2\text{SiO}_3\text{-SnCl}_2$ compounds after growing nanoparticles on the carbon nanotube CVD methods

Table 4 – Results of X-ray phase analysis after growing a nanoparticle of a carbon nanotube by CVD methods

Results of elemental analysis in weight,% ratios							
Range	O	Na	Al	Si	C	Sn	Total
Spectrum 1	30,17	4,90	0,13	3,97	5,51	55,32	100,00
Spectrum 2	31,60	2,74	0,06	3,96	3,97	57,68	100,00
Spectrum 3	31,07	3,05	0,09	4,20	5,18	56,40	100,00
Average	30,94	3,56	0,09	4,05	4,89	56,47	100,00

Table 4 shows that the elemental analysis reduces the oxygen content, the content of tin and silicon increases, carbon deposits. The change in the content of elements shows that with the growth of carbon nanoparticles, the microstructure of the resulting compounds changes.

From table 5 it is seen that the elemental analysis of the oxygen content decreases, the content of tin and silicon increases, carbon is deposited. The change in the content of elements shows that with the growth of carbon nanoparticles, the microstructure of the resulting compounds changes.

Figures 6 and 7 shows the microstructures of nanocomposites synthesized by the sol-gel method using tin chlorides and sand-like waste, which is accumulated in the fluorine-containing sludge accumulator in the feed phosphate workshop of the Mineral Fertilizers plant in the Taraz city, which contains SiO_2 .

According to the results presented in tables 6 and 7, elemental analysis showed a decrease in the oxygen content, an increase in the content of tin and silicon, and carbon deposition.

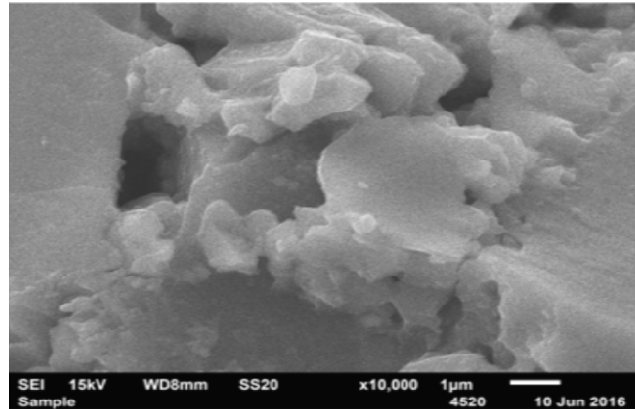


Figure 5 – Microstructure (SEM) of nanocomposites obtained from $\text{SiO}_2\text{-SnCl}_2$ compounds after growing a nanoparticle by a carbon nanotube CVD to methods

Table 5 – Results of X-ray phase analysis after growing nanoparticle carbon nanotube CVD methods

Results of elemental analysis in weight, % ratios							
Range	O	Na	Al	Si	C	Sn	Total
Spectrum 1	29,50	0,50	0,16	2,03	8,40	59,42	100,00
Spectrum 2	29,49	0,47	0,12	2,15	8,60	59,18	100,00
Spectrum 3	29,44	0,52	0,27	2,10	8,63	59,03	100,00
Average	29,48	0,50	0,18	2,09	8,54	59,21	100,00

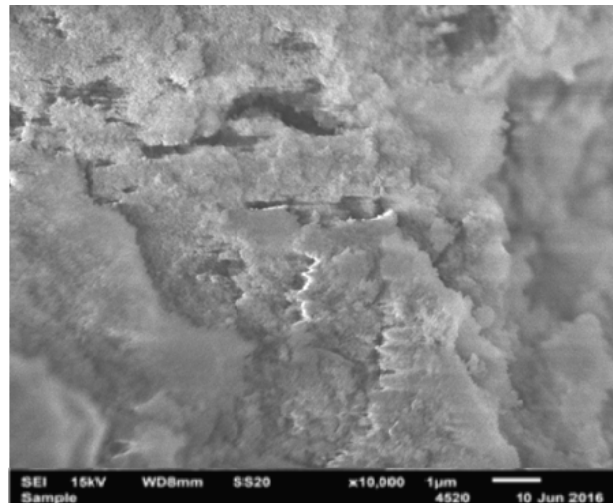
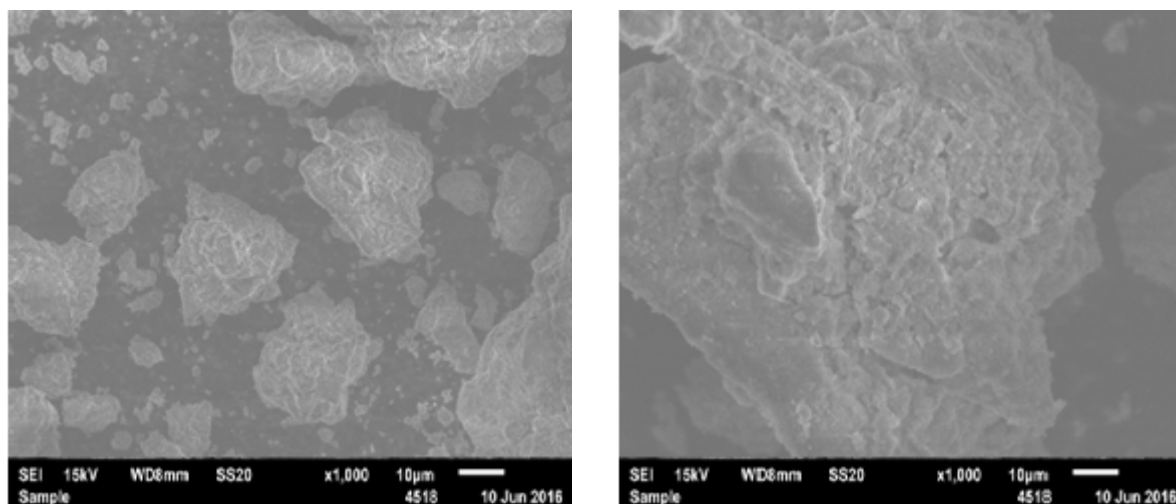


Figure 6 – Microstructure of the obtained products mixture of 1.5 N SnCl_2 solutions and sand waste, in which the composition contains SiO_2 (initial) in SEM - JEOL JSM-6490LV (Japan) low-vacuum electron microscope, 1 nm in volume

Table 6 – Results of X-ray diffraction analysis of the obtained $\text{SnCl}_2\text{-SiO}_2$ starting compounds (from sandy wastes)

Results of elemental analysis in weight,% ratios								
Range	In stats.	O	Al	Si	F	Ca	Sn	Total
Spectrum 1	Yes	31,80	0,12	10,99	0,010	15,00	43,01	100
Spectrum 2	Yes	32,10	0,05	15,34	0,012	15,62	36,82	100
Spectrum 3	Yes	32,06	0,07	14,39	0,013	16,90	37,78	100
Spectrum 4	Yes	32,70	0,04	11,09	0,012	16,25	42,95	100
Average		32,95	0,05	13,95	0,012	15,56	40,14	100



(a) (b)

Figure 7 – Microstructure of nanocomposites obtained from SiO₂ compounds (from sandy wastes) - SnCl₂ after growing a nanoparticle in a carbon nanotube CVD methods, 1 nm (a), microstructure of nanocomposites obtained from SiO₂ compounds (from sandy wastes) - SnCl₂ after growing nanoparticles on a carbon nanotube CVD method (b)

Table 7 – Results of X-ray phase analysis of products obtained from SiO₂ compounds (from sandy wastes) – SnCl₂

Range	In stats.	Results of elemental analysis in weight,% ratios							Итого
		O	Al	Si	F	C	Ca	Sn	
Spectrum 1	Yes	31,80	0,12	10,99	0,010	3,05	15,00	43,01	100
Spectrum 2	Yes	32,10	0,05	15,34	0,012	2,99	15,62	36,82	100
Spectrum 3	Yes	32,06	0,07	14,39	0,013	3,09	16,90	37,78	100
Spectrum 4	Yes	32,70	0,04	11,09	0,012	4,04	16,25	42,95	100
Average		33,95	0,05	12,95	0,012	3,09	15,56	40,14	100

Conclusion. Analysis of the set of experimental data and the results of scanning electron microscopy showed that the sol-gel technology is an effective and promising way to control the nanocrystalline structure of tin dioxide layers.

The change in the content of the elements shows that when the carbon nanoparticles are grown, the microstructure of the obtained compound changes.

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КРЕМНИЙ-ҚАЛАЙЫҚҰРАМДАС ЗАТТАРДЫҢ НЕГІЗІНДЕ КӨМІРТЕК НАНОКОМПОЗИТТЕРІН АЛУ

Аннотация. Алынған нанокөміртектер кремний диоксиді және тұндырылған көміртегінің бейорганикалық полимерлі торларына иммобилизденген қалайы диоксидінің нанобөлшектері болып табылады. Сондай-ақ, кремний диоксиді жоғары адгезиялық қасиеттерге ие және композиттің кристаллиттерінің агре-

гациялануына жол бермейді, ал қалайы мен көміртегі диоксидінің нано- және микроэлементтері газға сезімтал қасиеттерді алдын ала айқындайды. Золь-гель технологиясымен алынған үлдіртүзгіш ерітінділердің құрамында тетраэтоксисилан, қалайы тұздары ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$), кремний қышқылы ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) және натрий силикаты ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$) бар. Алынған ерітінділер ары қарай термиялық өңдеу арқылы металл тасымалдағыштың бетіне отырғызылды. Ертіндіге SiO_2 көзі ретінде тетраэтоксисилан ерітінділерін қосымша енгізу 5% SnO_2 – 95% SiO_2 , 30% SnO_2 – 70% SiO_2 құрамдас оксидті ксерогельдерді алуға мүмкіндік берді. Элементтердің құрамын өзгерту көміртегі нанобөлшектерін өсіру кезінде алынған қосылыстың микроқұрылымының өзгеретінін көрсетеді.

Түйін сөздер: нанокөміртектер, нанобөлшектер, кремний-қалайы нанотүтікшелері.

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ПОЛУЧЕНИЕ УГЛЕРОДНЫХ НАНОКОМПОЗИТОВ НА ОСНОВЕ КРЕМНИЙ-ОЛОВСОДЕРЖАЩИХ ВЕЩЕСТВ

Аннотация. Полученные нанокөміртектер представляют собой наночастицы диоксида олова, иммобилизованные в неорганической полимерной сетке диоксида кремния и осажденного углерода. При этом диоксид кремния обеспечивает высокие адгезионные свойства и предотвращает укрупнение кристаллитов композита, а нано- и микрочастицы диоксида олова и углерода определяют газочувствительные свойства.

Полученные методом золь-гель технологии пленкообразующие растворы содержат тетраэтоксисилан, соли олова ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$), кремниевую кислоту ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) и кремнекислый натрий ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$). Полученные растворы наносились на металлические подложки с последующей термообработкой. Дополнительное введение в растворы-золи тетраэтоксисилана, как источника SiO_2 , позволило получить ксерогели оксидных составов 5% масс. SnO_2 – 95% масс. SiO_2 , 30% масс. SnO_2 – 70% масс. SiO_2 . Изменение содержания элементов показывает, что при наращивании углеродных наночастиц изменяется микроструктура полученных соединений.

Ключевые слова: нанокөміртектер, наночастицы, кремний-оловосодержащие нанотрубки.

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