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INVESTIGATION OF THE PHYSICOCHEMICAL PROPERTIES OF CARBON NANOMATERIAL OBTAINED BY THE ELECTRIC ARC METHOD BASED ON ACTIVATED TALDYKOL COAL

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In this study, the modification of ”Taldykol” activated coal using the electric arc discharge method at different currents (100 A, 200 A, and 300 A) and its potential for producing graphene-like nanomaterials is discussed. The treatment of the coal leads to significant changes in its microstructure, including an increase in porosity (from 1.88 to 0.38 μm) and an improvement in adsorption properties, which contributes to effective absorption of gases and liquids. Raman spectroscopy showed a high degree of graphitization of the obtained nanomaterials with G_f values of 83.1% at 100 A, 89.5% at 200 A (lower part of the reactor), 97.5% at 200 A (electrode), and 91.1% at 300 A. Low ID/IG ratios (from 0.01 to 0.1) and high IG/I_2D ratios (from 2.5 to 3.7) indicate minimal defects and high-quality graphitic structures. Based on the gas composition and physicochemical properties data (volatility 42.54%, moisture 29.37%, ash content 19.23%, bulk density 0.70 g/cm³, pore volume 0.59 cm³/g, pH 7.05), the obtained nanomaterials possess high adsorption properties and can be used in fields such as hydrogen storage, energy, filtration, and the development of nanomaterials.

Keywords: carbon nanomaterial, graphite, electric arc discharge, specific surface area, graphitization.

БЕЛСЕНДІРІЛГЕН ТАЛДЫКӨЛ КӨМІРІ НЕГІЗІНДЕ ЭЛЕКТРО ДОҒАЛЫ ӘДІСПЕН АЛЫНҒАН КӨМІРТЕКТІ НАНОМАТЕРИАЛДЫҢ ФИЗИКА-ХИМИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

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Бұл зерттеуде ”Талдыкөл” белсендірілген көмірін электр доғалық разряд әдісімен әртүрлі токтарда (100 А, 200 А және 300 А) модификациялау және оның графен тәрізді наноматериалдарды алу потенциалы қарастырылады. Көмірді өңдеу оның микроструктурасында елеулі өзгерістерге әкеліп, порosity көрсеткішінің 1,88-ден 0,38 мкм-ге дейін артуына және адсорбциялық қасиеттерінің жақсаруына ықпал етеді, бұл газдар мен сұйықтықтарды тиімді сіңіруге мүмкіндік береді. Раман спектроскопиясы алынған наноматериалдардың жоғары графиттену деңгейін көрсетті, мұндағы G_f мәндері 100 А кезінде 83,1%, 200 А кезінде реактордың төменгі бөлігіне 89,5%, 200 А кезінде электродта 97,5%, ал 300 А кезінде 91,1% болды. ID/IG қатынасының төмен (0,01-ден 0,1-ге дейін) және IG/I_2D қатынасының жоғары (2,5-ден 3,7-ге дейін) мәндері дефектілердің минималды екендігін және жоғары сапалы графиттік құрылымдардың бар екенін көрсетеді. Газ құрамы мен физика-химиялық қасиеттер бойынша алынған деректерге (ұшқыштық 42,54%, ылғалдылық 29,37%, күлділік 19,23%, көлемді тығыздық 0,70 г/см³, кеуектің көлемі 0,59 см³/г, pH 7,05) сүйене отырып, алынған наноматериалдар жоғары адсорбциялық қасиеттерге ие және оларды сутекті сақтау, энергия, фильтрация және наноматериалдарды дамыту салаларында қолдануға болады.

Түйін сөздер: көміртекті наноматериал, графит, электр доғасының разряды, меншікті беттік аудан, графиттену дәрежесі.

ИССЛЕДОВАНИЕ ФИЗИКО-ХИМИЧЕСКИХ СВОЙСТВ УГЛЕРОДНОГО НАНОМАТЕРИАЛА, ПОЛУЧЕННОГО ЭЛЕКТРОРАЗРЯДНЫМ МЕТОДОМ НА ОСНОВЕ АКТИВИРОВАННОГО УГЛЯ ТАЛДЫКОЛЬ

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В настоящем исследовании рассматривается модификация активированного угля ”Талдыколь” с использованием метода электродугового разряда при различных токах (100 А, 200 А и 300 А) и его потенциал для получения наноматериалов на основе графеноподобных структур. Обработка угля приводит к значительным изменениям в его микроструктуре, включая увеличение пористости (от 1,88 до 0,38 мкм) и улучшение адсорбционных свойств, что способствует эффективному поглощению газов и жидкостей. Раман-спектроскопия показала высокую степень графитизации полученных наноматериалов с показателями G_f 83,1% при 100 А, 89,5% при 200 А (нижняя часть реактора), 97,5% при 200 А (электрод) и 91,1% при 300 А. Низкие значения отношения ID/IG (от 0,01 до 0,1) и высокие значения IG/I_2D (от 2,5 до 3,7) свидетельствуют о минимальных дефектах и высококачественных графитовых структурах. На основе данных о газовом составе и физико-химических свойствах (летучесть 42,54%, влажность 29,37%, зольность 19,23%, насыпная плотность 0,70 г/см³, объем пор 0,59 см³/г, pH 7,05) полученные наноматериалы обладают высокими адсорбционными свойствами и могут быть использованы в таких областях, как хранение водорода, энергии, фильтрация и разработка наноматериалов.

Ключевые слова: углеродный наноматериал, графит, электродуговой разряд, удельная поверхность, графитизация.

Introduction. In recent decades, carbon nanomaterials (CNMs) have attracted increasing scientific interest due to their unique physicochemical properties, high specific surface area, electrical conductivity, and chemical resistance. Their applications span a wide range of fields: from energy storage systems and adsorbents to biomedicine and electronics. In this context, the search for environmentally friendly and cost-effective methods for the synthesis of such materials is particularly relevant. The use of activated coals, especially of natural origin, as raw materials for the synthesis of CNMs is a promising direction. This study investigates coal from the Taldykol deposit, emphasizing the importance of local natural resources and their scientific and practical potential.

The aim of this work is to obtain and study carbon nanomaterials based on activated Taldykol coal using the electric arc discharge method, as well as to determine their physicochemical characteristics, morphology, sorption properties, and degree of graphitization.

To achieve this goal, the following objectives were set: modification of activated coal at different currents (100 A, 200 A, 300 A); study of the morphology and structure of the obtained material using SEM and Raman spectroscopy; determination of the elemental composition and degree of graphitization; analysis of the sorption characteristics of the obtained nanomaterials.

The novelty of the research lies in the use of activated Taldykol coal—a local resource—for obtaining graphene-like nanomaterials with a high degree of graphitization. A comprehensive evaluation of the material’s properties under various arc discharge regimes was conducted. Materials with low defect levels and the presence of multilayer graphene were obtained, as confirmed by Raman spectroscopy. This approach has not been previously highlighted in the available literature in the context of this specific coal and

chosen technology. According to reviews [1–5], carbon nanomaterials, including carbon nanotubes, graphene, graphdiyne, and graphone, demonstrate excellent properties when used in nanocomposites, supercapacitors, solar cells, fuel cells, and other devices. Due to their exceptional physicochemical properties, carbon-based nanomaterials have attracted considerable attention, making them promising candidates for a wide range of future applications in various critical fields [1,2].

The growing demand for environmentally friendly and cost-effective synthesis strategies has driven extensive global research efforts in the development of carbon nanomaterials.

Carbon nanomaterials (CNMs) have demonstrated significant potential for enhancing the efficiency of solar cells, batteries, supercapacitors, and fuel cells. Additionally, they are widely used in the development of nanocomposites with improved mechanical strength, thermal and electrical conductivity, as well as in biomedical and other technological applications [3,4]. CNTs also show great promise for advancing materials science, biomedicine, and pharmaceuticals [5].

The arc discharge method is one of the earliest and still effective methods for obtaining CNMs, with the discharge parameters and the composition of the carbon precursor playing a critical role [6–8]. In the works [6] and [8], the importance of synthesis parameter selection for controlling morphology, defectiveness, and degree of graphitization is emphasized. Paradise and Goswami [9] focus on industrial aspects and scaling opportunities for synthesis technologies. Special mention should be made of the research [5], dedicated to new forms of carbon structures and their potential in nanotechnology.

Currently, the global market for carbon nanomaterials is valued at approximately $4 billion, with projections indicating a tenfold increase over the next decade. Consequently, there is a growing need not only to scale up their production but also to reduce manufacturing costs to ensure their sustainable and widespread utilization.

Coal is an abundant and cost-effective natural resource that is considered an environmentally friendly and feasible alternative for the efficient production of carbon nanomaterials.

Since coal is a polyaromatic polymer, it can be thermally depolymerized to break down aromatic compounds, leading to the formation of carbon nanotubes (CNTs) using the arc discharge method [6,7]. (Figure 1).



Fig.1 - Currently used methods for CNTs synthesis

Arc discharge was the first technique that led to the discovery of new nanoscale carbon structures. This method enables the synthesis of fullerenes and carbon nanotubes of various shapes, with diameters ranging from 0.4 nm to several tens of nanometers.

The method is carried out by spraying graphite into an electric arc discharge plasma in an inert gas atmosphere (He or Ar). Graphite electrodes serve as the carbon source, with an arc burning between them. This technique forms the foundation of modern technologies for the large-scale production of fullerenes and enables the synthesis of nanotube-containing materials in quantities sufficient for studying their physicochemical properties [8,9].

However, studies focusing on the use of naturally activated coals (in particular, Kazakh coal) are underrepresented in international literature. This work addresses that gap, demonstrating the potential of local coal as a feedstock for producing CNMs.

In recent years, the relevance of carbon nanomaterials (CNMs) has significantly increased due to global sustainable development goals, the transition to low-carbon energy, and the demand for improved energy storage, environmental remediation, and advanced functional nanocomposites. According to Allied Market Research, the global CNM market is expected to exceed USD 20 billion by 2032, with a projected annual growth rate of over 15% [10].

One of the main challenges in CNM production is the high cost of raw materials and the complexity of scaling synthesis. As a result, there is growing interest in alternative carbon sources, particularly natural coals. Studies show that activated carbon can serve not only as an adsorbent but also as a precursor for the synthesis of graphene-like nanomaterials via arc discharge.

The research [11] demonstrated that thermal treatment of coal in an inert atmosphere promotes the formation of carbon nanostructures with high porosity and sorption capacity. scientific work [12] showed the effectiveness of such materials as catalyst carriers in hydrogen reactions and electrochemical devices.

The arc discharge method remains one of the earliest and most effective approaches for CNM synthesis. Its theoretical principles and practical applications are well described in the Great Russian Encyclopedia and works of scientists Zolotarenko and Kozlov demonstrated the potential of the plasma-arc method for producing high-quality CNMs. The textbook ”Materials and Methods of Nanotechnology” (Ural Federal University) also provides a comprehensive overview of synthesis techniques, including arc discharge [13-16].

Further contributions are presented in a dissertation [17], focusing on nitrogen-doped carbon nanofibers and their nanocomposites synthesized using the arc method.

Utilizing local raw materials, such as activated coal from Kazakhstan, presents new opportunities for localized CNM production and highlights their application potential in energy, ecology, and material science.

Materials and methods. There are various methods for producing carbon materials, including preparation and modification of the initial carbon, carbonization and subsequent activation with a gas or chemical reagent. The activation medium (N_2, CO_2 or H_2O) also affects the structural properties of activated carbon. Nitrogen has been found to be a good alternative as an activation medium compared to CO_2 and water vapor. The aim of the study is to investigate the physicochemical properties of carbon nanomaterial obtained by the electro-discharge method based on Taldykol activated carbon to assess its structural, morphological, and sorption characteristics.

The chemical analysis and surface morphology of the carbon nanomaterial (CNM) were investigated using energy-dispersive X-ray spectroscopy (EDS) on a scanning electron microscope (SEM) (Quanta 3D 200i) equipped with an EDAX EDS system. The accelerating voltage of the electron beam was set to 15 kV. The elemental composition, structure, and dimensions of the samples were analyzed using energy-dispersive X-ray spectroscopy (EDS) on the SEM (Quanta 3D 200i) with an EDAX energy-dispersive analysis prefix. The modification of the carbon structure was

examined through Raman spectroscopy utilizing the method of combinational light scattering.

Results and discussion. In the present study, the thermal decomposition characteristics of Taldykol coal upon heating to 900°C were investigated, and the resulting material balance was analyzed. The Taldykol coal samples were subjected to thermal treatment at 900°C with a controlled heating rate of 5°C/min. The material balance analysis of the coal activation process (Table 1) revealed that heating to 900°C leads to mass redistribution among the solid residue, liquid products, and gaseous emissions. The analysis of the gas composition during pyrolysis indicated that at the early stages (200–400°C), nitrogen and oxygen dominate, with the oxygen concentration gradually decreasing due to the decomposition of functional groups in the coal. Hydrogen evolution increases with temperature, reaching a maximum at 900°C (1.79%), which may indicate the breakdown of hydrocarbon bonds and the development of a porous structure. Methane formation begins at 600°C, reaching its highest concentration (0.14%) at 800°C, suggesting the thermal degradation of organic compounds. Carbon dioxide is primarily released at lower temperatures, peaking at 16.92% at 200°C, and subsequently decreases, likely due to reduction processes leading to carbon monoxide formation (Table 2).

The results of the physicochemical characteristics of the samples and the elemental composition are presented in Tables 3 and 4.

Table 1 - Material balance of ”Taldykol” coal activation at 900°C with a heating rate of 5°C/min

Input materials				Output products			
№	Name	Content		№	Name	Content	
		g	%			g	%
1	Coal ”Taldykol”	300.00	100.00	1	Product (adsorbent)	158.61	52.66
				2	Resin + water	13.00	4.33
				3	Gas	128.39	42.79
	TOTAL	300.00	100.00		TOTAL	300.00	100.00

Table 2 - Gas composition of ”Taldykol” coal upon activation at 900ºC with a heating rate of 5ºC/min

Temperature	Concentration of gases released during pyrolysis				
	H2	N2	O2	CH4	CO2
200 ºC	4,45	60,08	21,35	-	16,92
300ºC	1,17	67,94	12,06	-	0,06
400ºC	1,14	51,23	14,79	-	0,98
500ºC	0,36	55,4	15,51	0,25	1,48
600ºC	0,55	78,74	18,9	2,13	0,71
700ºC	2,36	68,7	11,19	0,01	0,1
800ºC	1,42	64,23	20,8	0,09	0,22
900ºC	1,79	78,36	18,94	0,02	0,39

Table 3 - Results of physicochemical analysis of activated ”Taldykol” coal

Name of activated adsorbent	W_t^r,%	A^r,%	V^d, %	$V_{\sum}$ (water), cm3/g	ρ_{bulk}, g/cm3	$pH_{\text{aqueous extract}}$	$A_{m.o.}$, mg/g	$A_{m.b.}$, mg/g	A_{iodine}, %
Activated ”Taldykol” coal	29.38	19.23	42.55	0.59	0.70	07.05	39.01	33.50	43.44

Coal ”Taldykol” with such physico-chemical characteristics can be used for the production of nanomaterials. Its high porosity (pore volume of 0.59 cm³/g) and adsorption properties (39.01 mg/g for methyl orange and 43.44% for iodine) indicate a large surface area, which is a key factor for creating and applying nanomaterials, especially in processes where interaction with molecules and particles is important.

Additionally, the low bulk density (0.70 g/cm³) and moderate ash content (19.23%) can be advantageous in the synthesis of carbon nanomaterials, such as carbon nanoparticles or carbon nanotubes. These properties make ”Taldykol” coal a promising candidate for use in technologies such as the production of nanomaterials for adsorption, hydrogen storage, as well as for the development of catalysts and filtering materials.

Table 4 - Results of the elemental composition of the nanomaterial obtained based on Taldykol coal

№	Name	C	O	Na	Mg	Al	Si	S	Ca	Cl	K	N	Fe	Zn
1	Activated ”Taldykol” coal	70.8	19.9	2.16	0.29	3.16	2.89	0.22	0.49	-	-	-	-	-
2	100 A reactor wall	55.8	32.5	0.62	-	1.52	4.58	0.67	0.40	0.25	0.82	-	-	-
3	100 A electrode surface	23.1	34.7	0.38	-	4.51	17.8	-	-	-	-	19.5	-	-
5	The lower part of the 200 A reactor walls	35.8	23.0	1.73	0.41	05.01	30.0	0.75	0.49	0.28	1.99	-	0.32	0.18
6	The upper part of the wall of a 200 A reactor	45.5	37.1	0.49	0.34	7.26	7.89	0.25	-	-	1.17	-	-	-
7	200 A on the surface of the electrode	29.3	35.8	1.36	0.36	5.66	12.2	0.27	0.43	-	1.11	13.1	0.16	0.27
8	300 A reactor wall	21.9	52.6	-	-	9.65	9.40	-	-	-	-	-	6.33	-
9	300 A electrode surface	-	63.4	4.21	-	-	4.20	-	13.3	0.53	3.95	-	-	-

Elemental analysis confirms that as a result of arc discharge treatment of activated Taldykol coal, there is not only a redistribution of carbon and oxygen, but also intensive formation of

oxide phases of silicon, aluminum, iron, and other metals. This indicates a deep transformation of the material's structure, which must be considered when selecting synthesis conditions for nanomaterials, especially graphene-like structures. At currents of 200–300 A, the formation of oxide phases and high-quality graphene-like structures is observed.

The morphology of the nanomaterials obtained by the electric arc discharge method was studied at different scales using scanning electron microscopy (SEM). Figure 2 shows the results of the SEM analysis.

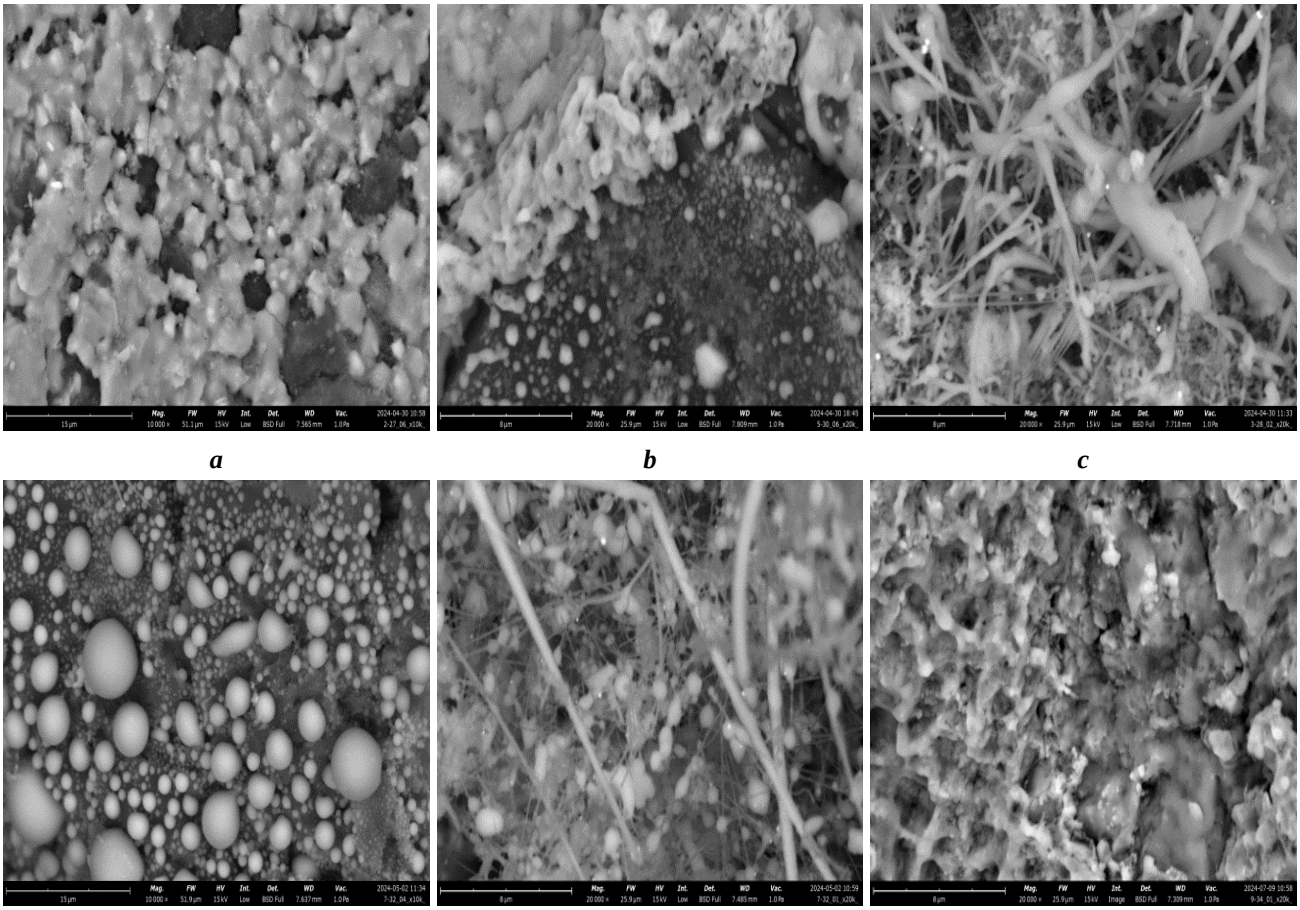


Fig.2 - Results of SEM analysis of carbon nanomaterial obtained on the basis of "Taldykol" activated carbon by the electric arc discharge method:

a- reactor wall 100 A; b, c - electrode surface 100 A; d, e - electrode surface 200 A; f - electrode surface 300 A

The treatment of "Taldykol" coal using an electric arc discharge at various currents leads to significant changes in its microstructure, expanding its potential for use in high-tech applications. After treatment at a current of 100 A, SEM images (a) show pores ranging from 1.88 to 0.38 µm on the reactor walls, indicating an increase in the coal' s porosity. This enhances its adsorption properties, allowing the coal to efficiently absorb various substances, including gases and liquids. After treatment at 100 A, SEM images (b) of the electrode surface reveal the formation of silicon oxide micro-particles ranging from 0.36 to 0.85 µm, which may improve the thermal and chemical properties of the coal, as well as make it suitable for use as a catalyst or filtering component.

Additionally, SEM image (c) after treatment

at 100 A shows fibers on the electrode surface with diameters ranging from 0.62 to 0.24 μm, which may indicate the formation of carbon nanostructures, enhancing the coal' s adsorption properties and mechanical strength. After treatment at 200 A, SEM image (d,e) of the electrode surface shows the formation of silicon oxides ranging from 0.33 to 1 μm, indicating continued microstructural changes that contribute to improved physicochemical characteristics of the coal.

Thus, the treatment of "Taldykol" coal with an electric arc discharge significantly improves its microstructure, forming pores, fibers, and oxides, thereby expanding its potential in various industries such as adsorption, catalysis, and nanomaterial development.

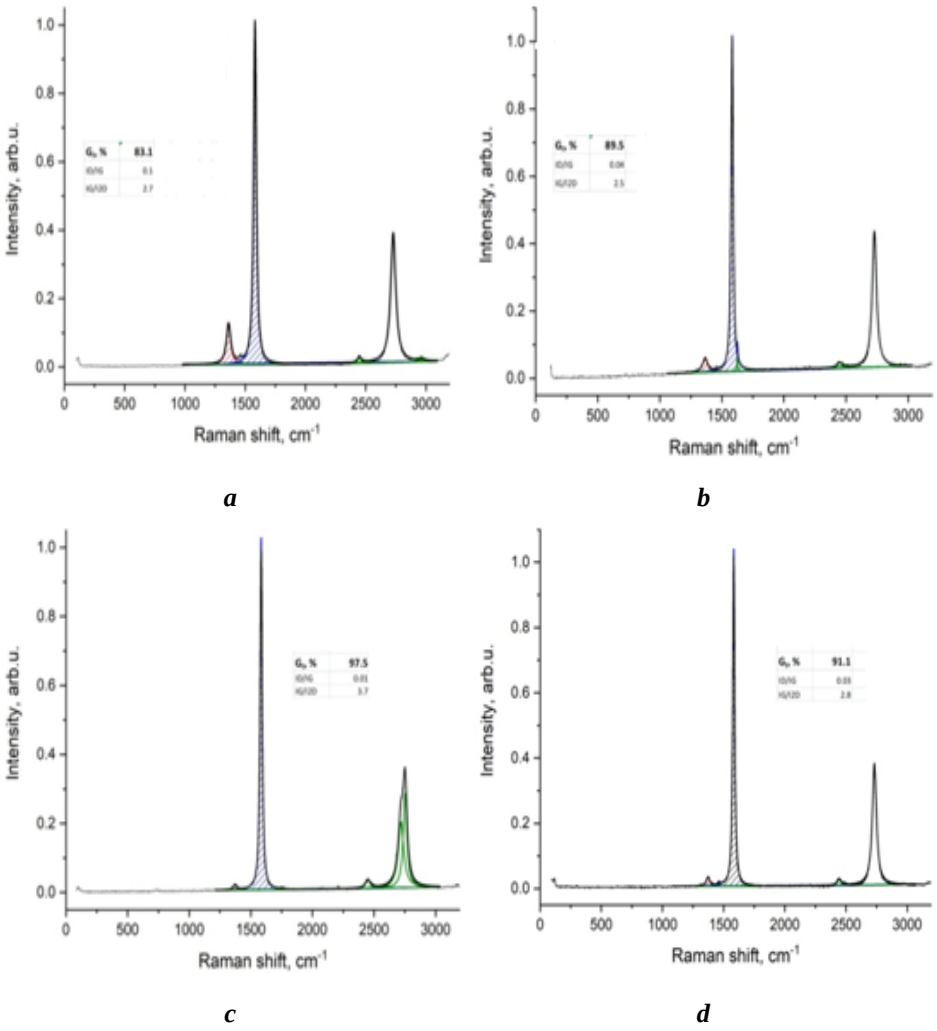


Fig.3 - Results of Raman spectroscopy analysis of carbon nanomaterial obtained on the basis of "Taldykol" activated carbon by the electric arc discharge method:

a - reactor wall 100 A; b - reactor wall 200 A; c - electrode surface 200 A; d - reactor wall 300 A

Raman spectroscopy plays a key role in the study and characterization of nanomaterials, providing important information about the structure, composition, and properties of the materials, which is necessary for their further use in various high-tech applications. The results of Raman spectroscopy are shown in Figure 3.

The results obtained after the treatment of "Taldykol" activated coal using the electric arc discharge method at various currents demonstrate

high-quality graphene-like nanomaterials with different degrees of graphitization. At a current of 100 A and 75 V a graphene-like material with a graphitization degree of Gf = 83.1% was obtained from the reactor wall. The ID/IG ratio of 0.1, IG/I_2D ratio of 2.7, and I_2D/IG ratio of 0.4 indicate a high-quality carbon structure with a low defect count and well-defined graphite layers. At a current of 200 A the lower part of the reactor yielded a graphene-like material with a higher graphitization degree of G_f = 89.5%. The ID/IG ratio of 0.04, IG/I_2D ratio of 2.5, and I_2D/IG ratio of 0.4 confirm an improved structure with reduced defectiveness and a more pronounced graphite nature. Moreover, the material consists of multilayer graphene (5-10 layers), indicating its high quality. At 200 A on the electrode a graphene-like nanomaterial with a graphitization degree of G_f = 97.5% was obtained. The ID/IG ratio of 0.01, IG/I_2D ratio of 3.7, and I_2D/IG ratio of 0.2 show nearly ideal structure with minimal defects and maximum graphitization. This material represents high-quality graphite with a well-developed crystalline structure. Finally, at a current of 300 A a graphene-like material with a graphitization degree of G_f = 91.1% was obtained from the reactor wall. The ID/IG ratio of 0.03 and IG/I_2D ratio of 2.8 confirm a high-quality structure. The material consists of multilayer graphene (7-10 layers) and also exhibits high-quality characteristics.

Thus, the results of ”Taldykol” coal treatment using electric arc discharge show that increasing the current improves the degree of graphitization and the quality of the obtained graphene-like nanomaterials. The materials produced can be applied in energy storage, catalysis, and other high-tech fields requiring high-quality carbon nanomaterials.

Conclusion. The treatment of activated Taldykol coal using the electric arc discharge method at various currents (100 A, 200 A, and 300 A) significantly alters its microstructure, opening new prospects for the application of the obtained nanomaterials in high-tech fields. After treatment at 100 A and 75 V, pores ranging from 1.88 to 0.38 μm were observed on the reactor walls, indicating a significant increase in porosity. These changes enhance the coal’ s adsorption properties, allowing it to effectively absorb various substances, including gases and liquids. After treatment at 100 A, the surface of the electrode exhibited the formation of micro-particles of silicon oxides with sizes ranging from 0.36 to 0.85 μm, which can improve the thermal and chemical characteristics of the coal, as well as expand its use as a catalyst or filtration component.

Raman spectroscopy, conducted for the analysis of the obtained nanomaterials, showed a high degree of graphitization. Specifically, at currents of 100 A, 200 A, and 300 A, the degree of graphitization was as follows: for 100 A (75 V), G_f = 83.1%; for 200 A at the lower part of the reactor, G_f = 89.5%; for 200 A at the electrode, G_f = 97.5%; and for 300 A at the reactor wall, Gf = 91.1%. These values indicate a high level of graphitization with increasing current, which is confirmed by low ID/IG ratios (from 0.01 to 0.1) and high IG/I_2D ratios (from 2.5 to 3.7). The obtained nanomaterials also contain multilayer graphene with a layer count ranging from 5 to 10.

Thus, the treatment of Taldykol coal using electric arc discharge significantly improves its microstructure, increasing porosity, adsorption properties, and the degree of graphitization. The results of Raman spectroscopy confirm the high quality of the obtained graphene-like nanomaterials with low defectiveness. These materials have potential applications in fields such as energy, catalysis, filtration, and the development of nanomaterials requiring high-quality carbon materials.

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