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Thermophysical Treatment of Petroleum Coke-Based Electrode Paste as a New Promising Approach to Integrating the Oil Refining and Metallurgical Industries for Carbon-Graphite Electrode Production

K. A. Krylov*, V. Y. Bazhin, F. Y. Sharikov, B. E. Matylsky, V. M. Dydin

Faculty of Mineral Processing, Saint Petersburg Mining University, St. Petersburg, Russian Federation

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A B S T R A C T

The study focuses on developing a thermophysical treatment technology for petroleum coke to create a homogeneous layered structure required for producing high-performance electrodes in metallurgical and petrochemical industries. The relevance stems from the shortage of anisotropic petroleum coke in the Russian market and the need to enhance the competitiveness of domestic electrode products. The objective of the study is to develop and experimentally validate a technology that improves the structural anisotropy of carbon material. The materials used include samples of green petroleum coke calcined in an inert atmosphere and coke-pitch charge subjected to thermal and mechanical treatment. An experimental setup with an extruder (heating up to 520°C, pressure up to 100 MPa) was developed, alongside structural analysis methods such as FTIR spectroscopy, SEM and XRD analyses. Results confirmed increased material homogeneity, enhanced graphitization tendency, and improved electrode performance, made by treated carbon material. The technology demonstrates potential for industrial implementation, fostering new production chains for carbon-graphite products and increasing oil refining profitability.

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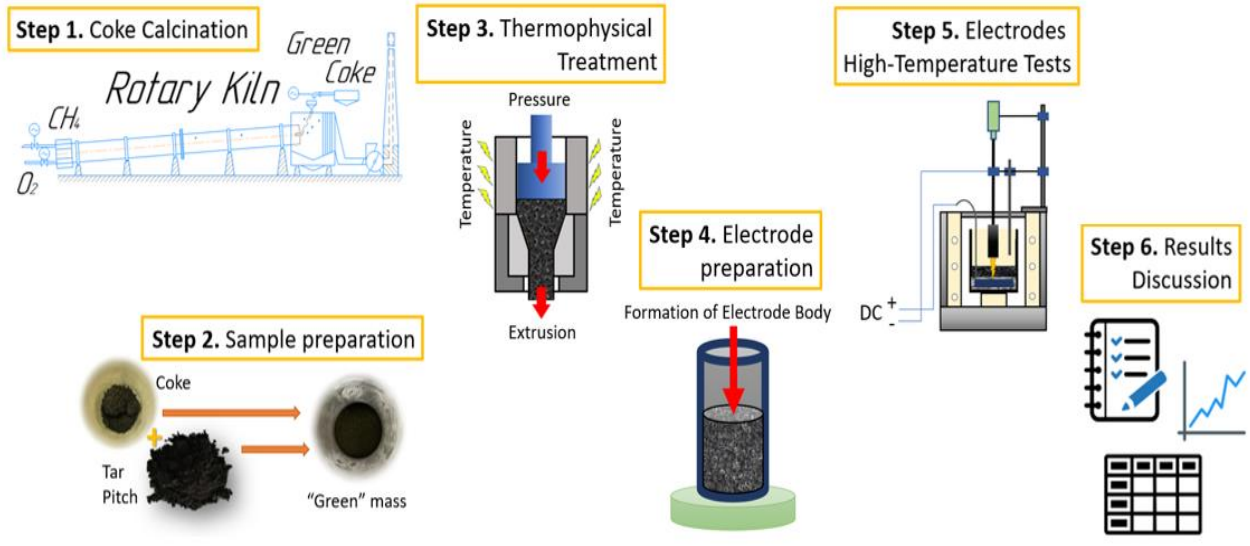
Graphical Abstract



*Corresponding Author Email: krylov-j@yandex.ru (K. A. Krylov)

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1. INTRODUCTION

In recent years, there has been a steady increase in the consumption of coal-graphite and graphitized electrodes worldwide, as well as growing demand for both graphite and carbon-based composite materials (1-3). High-quality coke with an anisotropic structure is required for graphite production. Global consumption of graphitized electrodes is projected reached 980.3 thousand tons in 2024. This is due to the increasing production rates of high-quality metallurgical products and the development of new technologies, such as battery manufacturing, where graphite is a key structural material (4-6).

Coal-graphite electrodes are essential consumables for many metallurgical units. They are typically made from petroleum and bituminous coke. The structure of the coke influences the tendency of the already formed and baked electrode to undergo further graphitization at temperatures above 2500°C. The electrical resistance of a coal-graphite electrode directly depends on its degree of graphitization, while its chemical and mechanical resistance during further use is largely determined by the homogeneity of the carbon material composition throughout the electrode (7-9). The productivity of metallurgical furnaces, the energy efficiency of the metallurgical industry as a whole, and the quality of the products obtained are heavily dependent on the quality characteristics of the corresponding electrodes (10-12). A relevant issue remains the improvement of the service life of carbon electrodes in ore-thermal furnaces and graphitized electrodes in electric arc furnaces (13-15).

During thermal heating and melting of the charge materials, electrodes undergo significant destruction. The main types of these destructions include mechanical wear of the electrode's end surface throughout its operation in the furnace, the formation of microcracks, gradual burning of the electrode's side surface, or its flaking, caused by chemical erosion (16, 17).

At present, there is only one pilot industrial facility in Russia where needle coke production has been established. In 2019, Public Joint-Stock Company (PJSC) "Gazpromneft-ONPZ" received a patent for the technology of petroleum needle coke production, and only in August 2024, a production unit with a declared capacity of 38,000 tons per year was launched. Clearly, such an amount of needle coke would not be sufficient to fully meet the needs of the domestic chemical-metallurgical sector (18).

As part of the governmental import substitution program, Russian scientists and also electrode factories are actively working on the development of technologies for producing high-quality electrode masses and electrodes using domestic mineral raw materials (19-21). Currently, attempts are being made to apply various organic modifiers and adjust the semicoking processes to improve the properties of domestic electrode masses (22). However, the largest Russian producer of coal-graphite products, LLC "GC "El6" which operates three plants (Chelyabinsk El6, Novosibirsk El6, Novocherkassk El6), still lacks technologies for producing needle coke – a crucial component for manufacturing coal-graphite and graphitized electrodes.

Due to the shortage of domestic products, Russian metallurgical companies are forced to purchase available Chinese electrodes or import needle coke, which is currently also produced in China, the USA, Japan, South Korea, and several other countries, in order to produce the necessary coal-graphite products within Russia. According to analytical research, the majority of advanced technologies for the production of high-quality electrode coke are associated with American companies (23). At the same time, it should be noted that most imported electrode masses based on needle coke exhibit significant deviations in the length of the needle-like phases while maintaining a uniform particle thickness. One of the most likely reasons for these deviations may be poorly selected technological conditions (24, 25).

During the calcination of petroleum coke, the filler material for the electrode mass, the disordered structure of non-graphitized carbon undergoes several changes.

According to Zhang et al. (26) coke calcination is carried out at temperatures up to 1350°C, during which volatile substances are removed from it and its density increases from 1.6 to 2.2 grams/cm^3. Thermal conductivity of calcined coke in relation to green coke increases from 0.4 to 0.9 W/(m-K) at 100°C, its electrical resistance decreases by several orders. If the calcination temperature within the electrode mass differs from the temperature inside a single coke particle, it inevitably leads to the relative misorientation of the carbon layers and their poor aggregation into packets, and some of the carbon may remain in an amorphous state. The electrode formed from such material will not have a uniform structure throughout its volume, which will inevitably lead to the formation of areas with various defects during operation (27-29). The process of structural changes in carbon materials is quite complex and not yet fully understood, although numerous studies exist on possible transformations of the coke structure during thermal treatment.

The scientific and technical interest consist in the development of additional measures for preparing coke for processing into electrode mass, specifically how thermophysical treatment influences the structure and, consequently, the properties of the coal-graphite material. A crucial task is to justify the technological foundations for forming the initial structure of the future electrode mass during the calcination of green coke in rotary kilns (30), as well as during the extrusion process of the liquid-solid phase consisting of calcined coke and pitch during the semicoking process within a specified temperature range. All of this is necessary to determine

the optimal technological parameters for the production of structured electrode mass (31).

Heat treatment at temperatures up to 1450°C has a significant effect on the structural characteristics of petroleum coke and its microstructure. The calcination promotes orderly stacking of aromatic layers, increasing crystallinity. This is reflected in the growth of packing parameters: the height of the pile of layers and the number of layers increases. The interlayer spacing (d002) decreases to 0.344 nm, bringing the structure closer to a graphite-like structure (32). Thus, heat treatment of petcoke can contribute to the formation of an ordered layered structure, which directly correlates with the improvement of the graphitising ability of the material during further processing.

The structured carbon material has an better graphitisation ability and the products made of graphitised material have superior thermophysical properties. Thus, the aim of this study is to enhance the performance characteristics of carbon graphite materials, such as thermal conductivity, electrical conductivity, mechanical strength and chemical stability, by directional modification of their structure. For this purpose it is necessary to solve a number of problems on development of methods of structural modification of carbon matrix, and establishment of interrelation between microstructure and stability of products under operating conditions.

2. EXPERIMENTAL

Based on literature data and the results of previous studies, it can be asserted that the classical one-stage calcination of green coke in a rotary tube furnace does not allow to produce calcined coke with a structure significantly superior to that of the original material.

In the present work, KZ Brand Coke (Table 1) and B grade coal tar-pitch (Table 2) were used. Samples of carbon material at different stages of technological processing were studied using FTIR spectroscopy, X-ray diffraction (XRD analisys) and scanning electron microscopy (SEM analysis).

XRD analysis was employed to evaluate the crystallinity degree and lattice parameters of raw and calcined petroleum cokes. The measurements were performed using a Shimadzu XRD-7000 powder diffractometer (Cu-Kα radiation, 2.7 kW) at room temperature. The diffraction patterns were recorded in the 2θ range of 10-80° with a scanning step of 0.02° and counting time of 2 seconds per point. The degree of crystallinity was assessed based on the intensity and position of characteristic (002) and (100) diffraction peaks. Lattice parameters (d-spacing) and crystallite sizes (Lc and La) were calculated using Bragg's law and the Scherrer equation.

TABLE 1. Specifications of KZ brand petroleum coke produced by LLC "LUKOIL-Volgogradneftepererabotka"

Property	PtW requirements
Mass fraction of total moisture, %	not more than 3,0
Mass fraction of volatile substances, %	not more than 11,0
Ash content, %	not more than 0,6
Mass fraction of sulphur, %	not more than 1.8

TABLE 2. Specifications of B grade coal tar pitch produced by "Chelyabinsk Metallurgical Plant"

Property	CHMK
Softening point, °C	70,5
Volatile yield, %	59,2
Fractional content α (toluene insoluble substances), %	33,9
Fractional content α1 (quinoline insoluble substances), %	13,5

FTIR spectroscopy was used to analyze the chemical composition and functional groups of the coke samples. The measurements were conducted using an IRAffinity-1 spectrometer (Shimadzu, Japan). For sample preparation, potassium bromide pellets containing finely ground coke samples were prepared according to standard methodology. The amount of coke in each pellet was optimized to obtain the best spectral response. The obtained IR spectra allowed identification of residual hydrocarbon functional groups after calcination process.

SEM analysis was performed to investigate the morphology and microstructure of coke samples at different processing stages. A Tescan Vega 3 scanning electron microscope equipped with secondary electron (SE) and backscattered electron (BSE) detectors was used for imaging. Prior to analysis, the samples were ground to particle sizes below 100 microns and mounted on carbon tape. The measurements were conducted at an accelerating voltage of 20 kV and emission current of 120 μA in high-resolution mode.

The combination of these complementary techniques provided comprehensive characterization of structural, chemical and morphological transformations in petroleum coke at different stages of thermophysical treatment.

To test the proposed technology for manufacturing electrode masses from calcined coke, a prototype extruder was created (Figure 1), designed for physical impact on the electrode mass (33, 34). The loading chamber of the laboratory extruder was filled with mixed coke-pitch charge (the ratio of coke and pitch was 3:1, respectively). The coke used was pre-crushed to the size of (-0.5.+0.3) mm. The pitch served as a plasticiser. The chamber with the mixture was heated up to the temperature of 450-630ºC in a crucible mini melting furnace PP-1 (PLAVKA.PRO, Russia), where it was kept

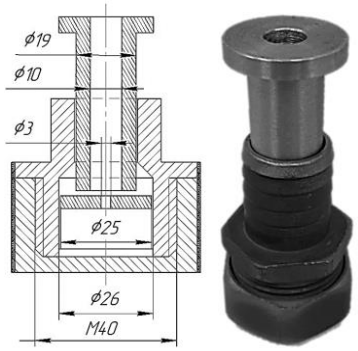


Figure 1. Dependence of the relative expansion of coke particles on the calcination temperature

for some time until complete heating of the charge. A rod was placed in the chamber with heated coke after removal from the furnace and the structure was placed in a hydraulic press, where a pressure of 50-100 MPa was applied to the rod. The experiment was carried out several times at different extrusion pressure, heating temperature and time.

The initial stages of testing demonstrated the feasibility of using the extruder for processing the charge through intermediate pressing of the electrode mass.

The issue of improving the quality of electrode mass was approached comprehensively. To solve the problem of obtaining an ordered structure for the electrode material, an innovative method for producing material with the required structure from calcined coke was proposed. Taking into account the specifics of the laboratory experiment, a sample of an experimental-industrial installation was developed and patented, considering the specifics of the complex reaction of coke-pitch charge calcination and the changes in its physicochemical properties. The operating principle of such an installation allows for simultaneous thermal (coking) and physical (pressure extrusion) treatment of the electrode charge, consisting of coke calcined in a tube furnace and solid pitch. Thus, intermediate treatment of the electrode mass becomes technically possible, which will not only eliminate structural defects in the calcined coke but also improve its overall properties.

Figure 2 presents a schematic diagram of the industrial extruder and its key elements, which will allow for the implementation of the technology for processing needle coke to create structured electrode mass. The device ensures the mixing and continuous heating of the coke-pitch charge and the creation of pressure to form an ordered structure of layers of a specified size with high packing density.

The developed installation for producing structured electrode mass consists of four main units and several auxiliary elements. A detailed description of the diagram, indicating all the structural elements shown, is provided in patent RU2784238C1. In the first unit, the coke-pitch charge is prepared for extrusion and transported to the extruder receiver. The screw, along with its casing and forming nozzle, constitutes the main assembly of the second unit – the extruder. The third unit is the drive mechanism for the screw, while the fourth unit serves as the air-cooling system for the extruded product.

The operating principle of the installation is as follows. At the input to the installation, the crushed coke-pitch charge flows by gravity from the loading hopper into the heating chamber, where it is heated to 200-250°C and charge is transitioned to a liquid-solid state. Under the influence of gravity, the mass flows to a branch integrated into the transport device, which not only feeds the heated mass into the extruder receiver but also mixes the charge. While mixing with the pitch, the coke particles agglomerate, the process of restoring broken layers begins, and pitch starts to penetrate the open pores of these particles.

According to the proposed technology, to form an ordered structure of the carbon mass, it is necessary to supply sufficient heat to the screw casing and forming nozzle to initiate the complex decomposition reaction of the pitch, which releases volatiles and forms semicoke. Thus, the technological treatment of the carbon mass will occur within a temperature range of 250-520°C. While the screw of the extruder is on, the charge will continue to mix, ensuring uniformity in its composition. Just before extrusion, the temperature of the charge reaches 450°C and continues to rise, initiating the semicoking process. Under the pressure generated by the screw (10-100 Pa), the carbon mass enters the forming channels of

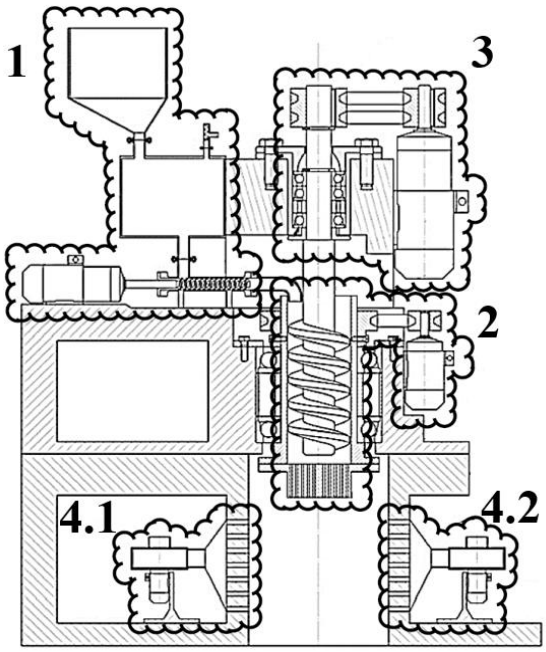


Figure 2. Schematic diagram of the industrial installation for producing structured electrode

the extruder nozzle, where the temperature rises from 450°C to 520°C, completing the semicoking process (35). Upon exiting the nozzle, the structured carbon material is subjected to air cooling, and the gases from the semicoking process are vented. Thus, through thermal and physical influence, the structural inheritance for the entire sample of carbon material is transferred.

3. RESULTS AND DISCUSSION

To confirm the hypothesis of the effectiveness of the proposed electrode mass structuring technology, a number of methodologies were used, which have been well-established in the study of carbon materials. Previous works (36) have extensively examined the kinetics of petroleum coke calcination. Based on the obtained experimental data, a mathematical model of coke calcination was developed, accounting for the peculiarities of the process in a rotary kiln.

The petroleum coke used in the laboratory experiment was pre-calcined. The process was conducted in a muffle furnace under an inert atmosphere at temperatures ranging from 20 to 1200°C for two hours, simulating calcination in a rotary kiln. FTIR spectroscopy was used to assess the calcination results. Tablets of potassium bromide (KBr) and the sample coke were prepared according to a standard procedure. The mass of KBr remained constant (around 300 mg), and the coke amount varied from 0.5 mg to 20 mg to obtain FTIR spectra, in which the absorption characteristic of the corresponding functional groups was most clearly visible. Measurements were taken in the wavenumber range from 400 to 4000 cm^{-1} with a step of 2 cm^{-1}. The FTIR spectroscopy results, shown in Figure 3, revealed that the green coke contains residual volatile hydrocarbons, as evidenced by characteristic peaks: aliphatic alkyl groups C_nH_m of varying lengths (methyl groups – absorption at 1375 cm^{-1}; 2950-3100 cm^{-1} and longer alkyl groups – 1450 cm^{-1}) and condensed aromatic rings (700-900 cm^{-1} and 2950-3100 cm^{-1} – associated CH_2 and CH_3 groups).In contrast, the calcined coke lacks these pronounced peaks, indicating the near absence of moisture, residual volatile hydrocarbons, hydrogen, and heteroatomic compounds. This is attributed to cracking and pyrolysis reactions that break C-H, C-C, C-N, and C-S bonds.

To estimate the degree of crystallinity and lattice parameters of coke samples before and after calcination, X-ray diffraction was used. XRD analysis of petroleum cokes reveales that the structure of the samples undergoes significant changes depending on heat treatment. Figure 4 shows the obtained X-ray diffractograms, illustrating the structural differences between the samples. For raw coke, a high proportion of the amorphous phase is observed, along with asymmetry of the d_{002} peak and the absence of the d_{100} peak, indicating a lack of an ordered

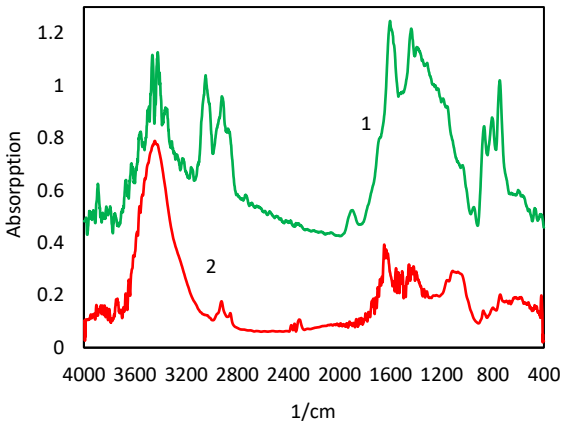


Figure 3. FTIR spectroscopy results for samples at different stages of technological processing (1 – green coke; 2 – calcined coke)

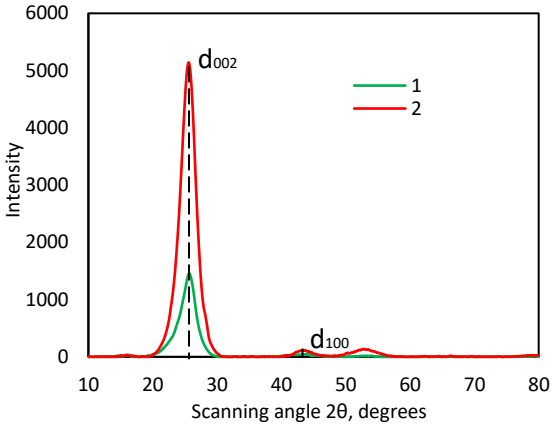


Figure 4. X-ray diffractograms of samples with different thermal history (1 – green coke; 2 – calcined coke)

crystal lattice. At 1200°C, the amorphous phase nearly disappears, the d_{002} peak becomes symmetric, and its intensity increases, suggesting the formation of a crystalline structure (Table 3). Analysis of crystallite parameters (L_a and L_c), indicates the onset of anisotropic particle formation in the material at this temperature.

To examine the morphology of the coke samples at various stages of processing, scanning electron microscopy (SEM) was employed (See the experimental details above). Figure 5 presents images of the original microstructure of green and petroleum coke samples calcined calcined at 1200°C in a rotary kiln. The surface of the calcined sample shows disoriented anisotropic crystallites of various sizes.

According to the developed technology, portions of coke blend were placed in the loading chamber of the laboratory extruder and heated in the muffle furnace, followed by extrusion of the heated carbon-graphite mass using the press and piston of the laboratory extruder.

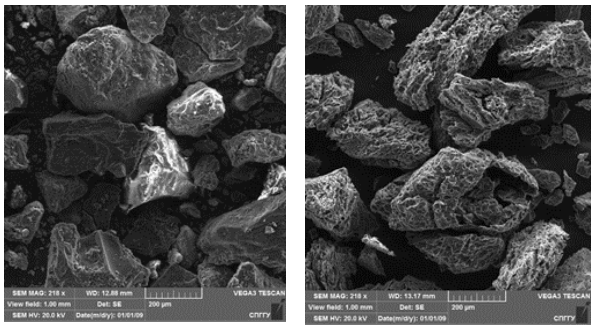
TABLE 3. Calculated Structural Characteristics of Coke Samples with Different Thermal History

Sample	Reflection (002)				Reflection (100)				La/Lc
	θ, rad	β, rad	d_{002}, Å	Lc, Å	θ, rad	β, rad	d_{100}, Å	La, Å	
Green coke	0,223	0,043	3,466	9,754	-	-	-	-	-
Calcined coke	0,224	0,046	3,480	30,748	0,378	0,061	2,088	60,232	1,959

The experiment was repeated several times with different technological parameters (matrix pressure, heating temperature of the blend before extrusion). The resulting test samples of carbon-graphite material were analyzed using scanning electron microscopy.

The X-ray diffraction analysis results for the carbon material samples generally confirmed the assumption about the thermo-physical transformation of the carbon-graphite material with increasing anisotropy. The most effective results were obtained when processing the samples in the temperature range of 450–520°C and pressure of 60–80 MPa. These conditions promoted the formation of an ordered layered structure in the carbon-graphite material. Figure 6 shows the typical microstructure for one of the samples at different stages of technological processing.

The green coke in its original state (Figure 6a) is a non-graphitizable material, where in some sections highly disoriented layers of varying thickness and orientation are observed, and no signs of the layered phase are visible. The calcined coke (Figure 6b) shows the formation of early stages of an ordered structure with some germs of the layered phase locally present. Along with relatively ordered structures, the material still contains amorphous carbon. The extruded sample blend (Figure 6c) exhibits a fully ordered structure, with layers flattened and packed tightly into packets, and the interlayer distances averaged to 12-20 microns. Thus, it



Green coke **Calcinated coke**

Figure 5. SEM analysis results for green and calcined coke samples

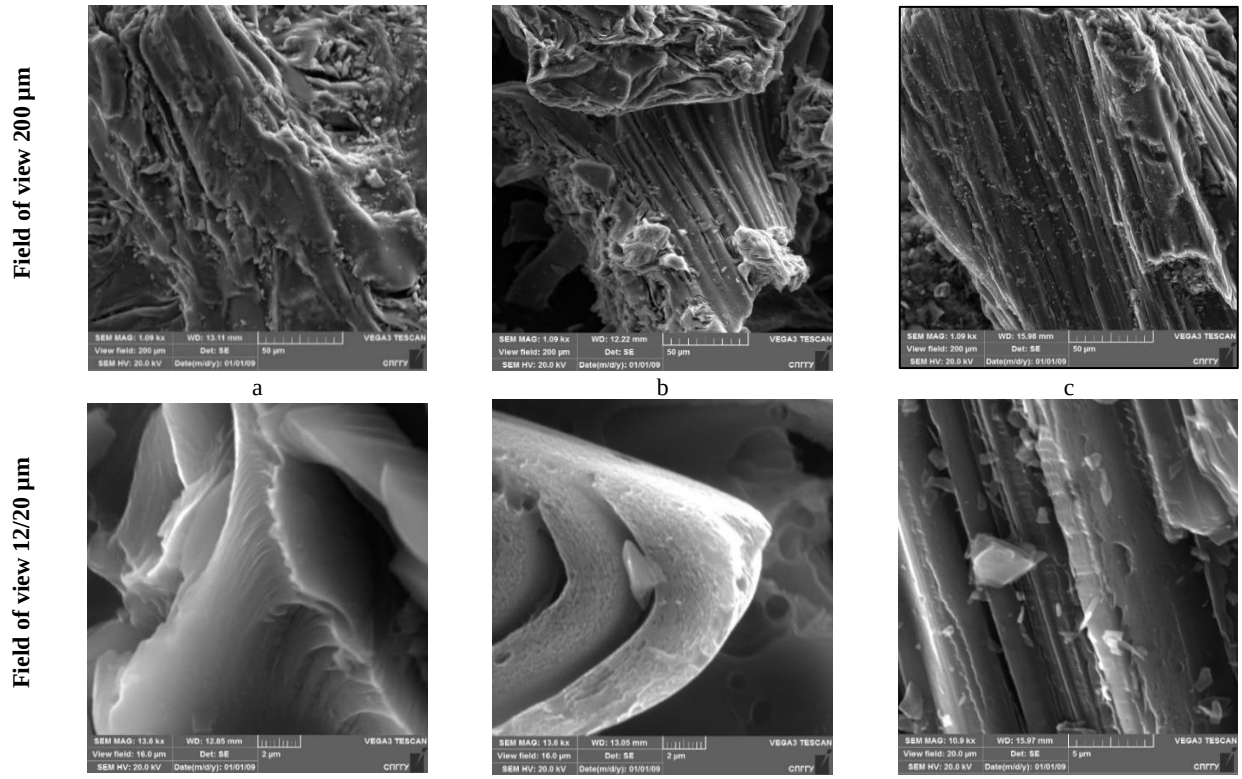


Figure 6. Microstructure of carbon-graphite material samples at different stages of technological processing: a – original green coke; b – structure of calcined coke; c – structure of carbon-graphite material after thermo-physical processing

was proved that thermo-physical treatment can improve the structural anisotropy of the carbon-graphite material. These factors influence the material's tendency to graphitize. Since the thermal expansion of the electrode made from the structured carbon-graphite blend is expected to be lower, this will inevitably impact the potential quality of the electrode.

The operational characteristics of carbon-graphite, graphitized, or graphite products depend on the fraction and granulometric composition of the initial material, the presence of impurities and ash, the pressing technology, and the time and temperature of the material calcination. The calcined coke was processed according to the standard technology to prepare the electrode mass, which was then pressed and fired to form the electrode. Similarly, an electrode was prepared from the material that had undergone thermo-physical treatment. Both electrodes (Figure 7) were tested under conditions similar to those of a smelting process in the furnace. The temperature range was from 700 to 1650°C. The electrode made from the material processed by extrusion (Figure 7b) showed fewer signs of degradation and retained its initial shape better than the electrode made from the standard carbon-graphite mass (Figure 7a).

After testing, the electrodes made from both standard and structured carbon-graphite masses were analyzed to determine the specific electrical resistance, bending strength, density, and linear thermal expansion coefficient. The results were compared with the quality characteristics of the MCGM electrode brand manufactured by the LLC "GC "El6". For clarity, Table 4 presents a comparative analysis of the characteristics of the MCGM electrode and the results of tests conducted on previously prepared electrodes.

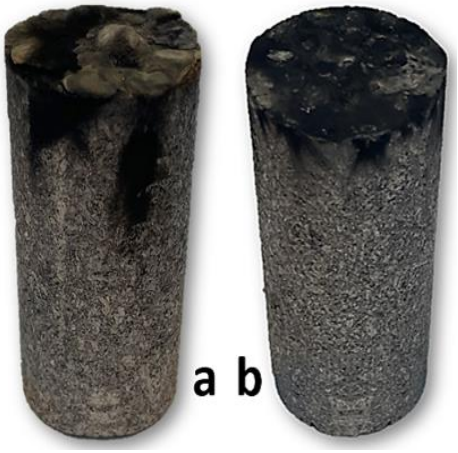


Figure 7. External appearance of electrodes formed from standard carbon-graphite mass (a) and from structured post-extrusion electrode material (b), after testing under simulated smelting furnace conditions

The samples made from the structured carbon-graphite material demonstrated superior performance compared to traditional electrodes made from standard carbon-graphite mass and practically correspond to the characteristics of MCGM brand electrodes (37).

The significant improvement in thermo-physical and mechanical properties of the material suggests that the proposed technology could serve as a basis for the production of more efficient electrodes for smeltingprocesses. In this regard, it is proposed to introduce an additional stage in the production cycle of carbon graphite electrodes - thermophysical treatment of carbon material after calcination. In the standard flow chart (Figure 8) this stage is highlighted by a green colour.

TABLE 4. Quality characteristics of electrodes

Property	MCGM	Electrode from standard material	Electrode from structured material
Apparent density, g/cm³	1.58	1.56+5%	1.63+5%
Specific electrical resistance, μΩ·m	25	30+5%	24+5%
Bending strength, MPa	8.5	6-10+5%	9+5%
Linear thermal expansion coefficient from 20 to 520°C, 10^{-6}/K	3.5	3.6+5%	3.4+5%

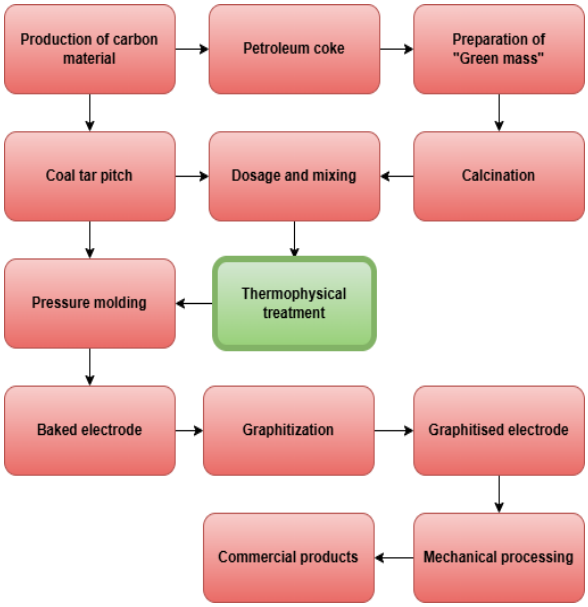


Figure 8. Modified carbon-graphite electrode production cycle

5. CONCLUSIONS

The conducted research confirms the feasibility of introducing the thermo-physical treatment operation for the electrode mass after the calcination stage in a rotary kiln. The developed original design of the installation not only increases the process productivity but also ensures the production of high-quality anisotropic material with specified geometric parameters of the layered phase (length and thickness).

Moreover, the process allows for the possibility of utilizing alternative carbon sources, such as coal or biomass, through blending and other methods, broadening the flexibility of raw material selection. Such coke can be successfully used in the production of large-sized electrodes for ore-thermal or electric arc furnaces.

Experiments with electrode masses on the developed extruder demonstrated that under optimally selected parameters (coking temperature, matrix pressure, screw rotation speed), uniform structuring of the electrode mass is achieved. The main results of the study include:

1. It was established that the quality of carbon-graphite products is determined by all stages of the technological process. Additional thermo-physical treatment at the pressing and forming stages significantly improves the properties of the electrode mass.
2. At an extrusion pressure of 60–80 MPa, stable formation of layered phases is ensured.
3. The bonding of the layered phases occurs through thin layers of coke at the temperature of the extruder outlet, that promotes the formation of a strong structure.

The key factor determining the material's ability to graphitize is its structural anisotropy, specifically the mutual arrangement of carbon networks in the initial material. The parallel arrangement of layers in packets and their minimal misorientation contribute to the flattening and aggregation of layers into groups, which facilitates the graphitization process. Thus, the proposed technology for processing "green" mass opens new opportunities for further processing of carbon material into high-quality carbon-graphite products with high added value.

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Persian Abstract

چکیده

این مطالعه بر توسعه یک فناوری تصفیه ترموفیزیکی برای کک نفتی برای ایجاد یک ساختار لایه‌ای همگن مورد نیاز برای تولید الکترودهایی با کارایی بالا در صنایع متالورژی و پتروشیمی متمرکز است. این ارتباط از کمبود کک نفتی ناهمسانگرد در بازار روسیه و نیاز به افزایش رقابت محصولات الکترود داخلی ناشی می شود. هدف از این مطالعه توسعه و تأیید تجربی فناوری است که ناهمسانگردی ساختاری مواد کربن را بهبود می بخشد. مواد مورد استفاده شامل نمونه‌های کک نفتی سبز کلسینه شده در اتمسفر خنثی و بار کک‌پیچ تحت عملیات حرارتی و مکانیکی است. یک راه‌اندازی آزمایشی با یک اکسترودر (گرمایش تا ۵۲۰ درجه سانتی‌گراد، فشار تا ۱۰۰ مگاپاسکال) در کنار روش‌های تحلیل ساختاری مانند طیف‌سنجی FTIR، SEM و آنالیز XRD توسعه یافت. نتایج افزایش همگنی مواد، افزایش تمایل گرافیتی شدن و بهبود عملکرد الکترود ساخته شده توسط مواد کربنی تیمار شده را تایید کرد. این فناوری پتانسیل اجرای صنعتی، تقویت زنجیره های تولید جدید برای محصولات کربن–گرافیت و افزایش سودآوری پالایش نفت را نشان می دهد.