

PRODUCTION OF ACTIVATED ADSORBENT BASED ON SEMICOKE FROM THE COAL SHUBARKOL DEPOSIT FOR GAS PURIFICATION

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Various types of raw materials, including plant precursors, coal raw materials, solid fuel combustion residues, coal mining waste, coke fines, and others, are used for the production of porous carbon materials. One important type of porous carbon materials is microporous sorbents, which have a high volume of micropores. These pores provide carbon materials with the ability to effectively adsorb low-molecular-weight gases and possess molecular sieving properties, allowing their use in gas separation processes.

The main method of increasing the porosity of sorbents and reducing pore sizes is the activation of sorbents at high temperatures using inert gases. This process increases the activity, capacity, and specific surface area of sorbents. The specific surface area of solid bodies plays a key role in their sorption properties at low and medium pressures. The use of activating agents, such as water vapor, in the thermal treatment processes of carbonaceous materials is a promising method for obtaining porous carbon materials.

Keywords: activated carbon, coke, semicoke, pyrolysis, coking, activation, sorbent.

ГАЗ ТАЗАЛАУ ҮШІН ШҰБАРҚӨЛ КӨМІР КЕНІ НЕГІЗІНДЕГІ ЖАРТЫЛАЙ КОКСТАН БЕЛСЕНДЕНДІРІЛГЕН АДСОРБЕНТ ӨНДІРУ

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Кеуекті көміртекті материалдарды өндіру үшін шикізаттың әртүрлі түрлері, соның ішінде өсімдік прекурсорлары, көмір шикізаты, қатты отынның жану қалдықтары, көмір өндіру қалдықтары, кокс үгінділері және т.б. Кеуекті көміртекті материалдардың маңызды түрлерінің бірі микрокеуектердің үлкен көлеміне ие микрокеуекті сорбенттер болып табылады. Бұл кеуектер көміртекті материалдарға төмен молекулалық газдарды тиімді адсорбциялау мүмкіндігін береді және молекулалық елеуіш қасиеттеріне ие, бұл оларды газды бөлу процестерінде қолдануға мүмкіндік береді.

Сорбенттердің кеуектілігін арттырудың және кеуек өлшемдерін азайтудың негізгі әдісі сорбенттерді жоғары температурада инертті газдармен белсендіру болып табылады.

Бұл процесс адсорбенттердің белсенділігін, сыйымдылығын және меншікті бетінің ауданын арттырады. Төмен және орташа қысымда олардың сорбциялық қасиеттерінде қатты денелердің меншікті бетінің ауданы шешуші рөл атқарады. Көміртекті шикізатты термиялық өңдеу процестерінде су буы сияқты активтендіргіштерді қолдану кеуекті көміртекті материалдарды алудың перспективалы әдісі болып табылады.

Негізгі сөздер: белсендірілген көмір, кокс, жартылай кокс, пиролиз, кокстеу, адсорбент.

ПРОИЗВОДСТВО АКТИВИРОВАННОГО АДСОРБЕНТА ИЗ ПОЛУКОКСА НА ОСНОВЕ УГЛЯ МЕСТОРОЖДЕНИЯ ШУБАРКОЛЬ ДЛЯ ОЧИСТКИ ГАЗОВЫХ СРЕД

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Для производства пористых углеродных материалов используют различные виды сырья, включая растительные прекурсоры, каменноугольное сырье, отходы сжигания твердого топлива, отходы угледобычи, коксовую крошку и другие. Одним из важных типов пористых углеродных материалов являются микропористые сорбенты, которые обладают высоким объемом микропор. Эти поры обеспечивают углеродному материалу способность эффективно адсорбировать низкомолекулярные газы и обладать молекулярно-ситовыми свойствами, что позволяет их использовать в газоразделительных процессах.

Основной метод увеличения пористости сорбентов и снижения размеров пор - это активация сорбентов при высоких температурах с помощью инертных газов. Этот процесс увеличивает активность, емкость и удельную поверхность адсорбентов. Удельная поверхность твердых тел играет ключевую роль в их сорбционных свойствах при низких и средних давлениях. Использование активирующих агентов, таких как водяной пар, в процессах термической обработки углеродистого сырья является перспективным методом получения пористых углеродных материалов.

Ключевые слова: активированный уголь, кокс, полукокс, пиролиз, коксование, активация, адсорбент.

Introduction. A substantial deficit of high-quality carbon materials, particularly carbon reductants essential for electrothermal processes such as ferroalloy, silicon, and steel production, persists both domestically and internationally. This scarcity stems from the closure, during the 1990s, of semicoke-producing enterprises in Russia (including the Angarsk and Chermkhovo coke and gas plants, each boasting an annual output of 2 million tons of semicoke) and in Eastern Germany. Factors contributing to this cessation encompass the physical deterioration of equipment (notably, coke ovens in Germany constructed during the 1930s-1940s, and plants in Russia relocated from Germany as reparations following World War II), environmental risks associated with production, and the absence of contemporary, efficient, and ecologically sustainable technologies.

The dwindling supply of metallurgical coke has precipitated a surge in its prices. In 2004 alone, the average price of coke across various grades skyrocketed from \$80 per ton to \$250 per ton. This upward trajectory is anticipated to persist, given that by 2010, the resources of nearly all coke plants will have been depleted, and reserves of valuable grades of coking coal (including grades K, KZh, KS) will have been exhausted. Notably, there exists a significant potential for semicoke exportation. In 2004, amidst a shortage of this product, prices for carbon reductants soared to \$300 per ton in Germany, Norway, and Japan. Additionally, in 2017, Russia witnessed a production

volume of 10,468 tons of activated carbons, marking a 10.8% increase compared to the previous year.

The consumption of carbon sorbents, primarily for environmentally sustainable technologies, has been steadily rising by over 5% annually in developed nations in recent years. Conventional methods for producing activated carbons are characterized by a multistage, energy-intensive, and environmentally hazardous process. Prices for activated carbon, comparable in quality, range from \$1200 to \$1500 per ton both domestically in Kazakhstan and abroad. Global consumption of activated carbon totals up to 1.5 million tons annually, whereas in Russia, it amounts to less than 20 thousand tons per year. The production of thermal and electric energy considers the prospect of obtaining valuable by-products such as activated carbon, semicoke, ash-slag materials, and commercial sulfur [1].

Until the advent of the 20th century, most chemical products were derived from the pyrolysis and coking of coal. These processes entail the heating of coals in the absence of air for their thermal decomposition. Coking coal primarily yields coke, extensively utilized in both ferrous and non-ferrous metallurgy for metal smelting. By-products of these processes include a gas-steam mixture containing numerous chemical compounds [2].

Materials and Methods. The production process of granulated activated carbons consists of several crucial stages, including thermo-treatment and activation,

along with additional procedures involving the preparation of a paste-like mass from coke powder and binder, followed by the granulation of the resulting coal-tar composition. The properties of the obtained granules and the parameters of the granulation process are significantly influenced by the characteristics and composition of the raw materials. Noteworthy advantages of these granules encompass their stable shape, mechanical strength, and suitability for employment in cyclic processes with a moving bed [3].

Chromatographic determination of specific retention volumes was carried out on a Kristallux 4000M chromatograph in steel columns 25 cm long, with an internal diameter of 3 mm.

Coke fines manifest visually as solid substances with a grayish-black appearance and a distinct specific odor. The qualitative features of the initial coke fines are delineated in Table 4, demonstrating a high carbon content (approximately 88-90 %) alongside low sulfur and ash content [4].

The applications of activated carbons are diverse, spanning across various industries, including chemical (chemical fibers, dyes, chemical reagents), metallurgical (non-ferrous and ferrous metallurgy), gas and oil refining (separation and purification of process streams), and gas emission control (solvent recovery, gas purification) [5, 6].

A wide range of raw materials can be utilized in the production of activated carbon, encompassing traditional sources such as wood, peat, and coal, as well as unconventional sources like nutshells, barley straw, sewage sludge, liquid oil fractions, fly ash, and used

rubber tires [7-10].

Analysis of methods for obtaining sorbents from carbon-containing raw materials has demonstrated the feasibility of utilizing coal, typically employed as fuel in small autonomous thermal power plants, to produce activated carbon.

The elemental composition of the gas emitted during thermal treatment was analyzed using a "Kristallyuks 4000M" chromatograph. Moisture content, ash content, and volatility of the samples were determined using a "Thermoster Eltra" thermogravimetric analyzer (according to ASTM D7582-12). Various parameters such as total pore volume, bulk density, pH of the aqueous extract, and adsorption activity using methyl orange were determined following standard procedures. The adsorption characteristics of the sorbents (specific surface area) were investigated using the Brunauer-Emmett-Teller (BET) method, with measurements conducted on a KATAKON Sorbtometer M instrument. Chemical analysis and surface morphology were examined using energy-dispersive X-ray spectroscopy on a SEM instrument (Quanta 3D 200i) with an attachment for energy-dispersive analysis from EDAX. Particle size distribution, structural strength, and mass fraction of chlorine, arsenic, and sulfur were determined according to ND methods.

Semi-coke fines with a particle size fraction of 3-5 mm (derived from Shubarkol coal, grade D, Kazakhstan) were utilized as raw material, possessing the following technical characteristics: moisture content (W^T) - 7.71%, ash content (A^f) - 6.60%, volatile matter (V^d) - 19.43% [11, 12].



Figure 1 - Pilot-scale rotating drum furnace for thermal treatment of semi-coke fines

To produce the activated adsorbent, crushed and fractionated semi-coke fines (fraction 3-5 mm) are introduced into a rotating drum furnace (refer to Figure 1) for steam activation at temperatures ranging from 700 °C, with a heating rate of 5-10°C/min and a holding period at 700°C for 60 minutes. Subsequently, the samples are extracted and allowed to cool in a dry atmosphere [13-15].

The furnace construction consists of the following main separate units and assemblies:

- Loading hopper with a screw feeder;

- Furnace chamber with a gas heating system to create the desired thermal regime;
- Instrumentation and control devices (thermocouples, flowmeters, pressure gauges);
- Unloading chamber with an unloading gate;
- Collector for removing exhaust gases from the furnace chamber;
- Drive station for rotating the furnace.

The furnace activation scheme is presented in Figure 2.

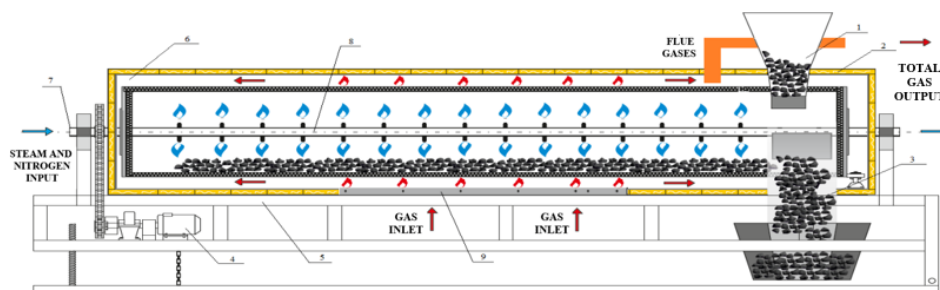


Figure 2 - Schematic diagram of the pilot-scale rotating drum furnace for thermal treatment of semi-coke fines

Gas chromatographic analysis unveiled the primary composition of gases generated during the activation process of semi-coke fines, comprising oxygen, hydrogen, nitrogen, methane, carbon dioxide, hydrogen

sulfide, and carbon monoxide. The gas composition and material balance of semi-coke activation are detailed in Tables 1 and 2.

Table 1 - Gas composition of semi-coke fine (size 3-5 mm) activation at 700°C

T1 C°	Composition of gases, %						
	O2	H2	CO2	N2	CH4	CO	H2S
200	1.809	7.603	12.505	0.239	1.217	-	0.217
300	0.819	4.638	9.617	1.237	1.690	0.290	0.379
400	3.298	4.516	11.089	0.292	1.700	-	0.104
500	0.484	4.818	9.057	3.996	1.768	0.378	0.118
600	3.220	3.575	7.184	0.266	0.466	-	0.183
700	5.613	5.054	3.847	4.596	0.611	0.244	0.051

Table 2 - Material balance of the process of activation of semi-coke breeze (2-5 mm)

№	Name	Content, kg	%
1	Semi-coke breeze, incl.	15	100
Total		15	100
1	Adsorbent	12,4	82,3
2	Resin+water	0,02	0,13
3	Gas	2,63	17,5
Total		15	100

Results and Discussion. The results of the physico-chemical characteristics and elemental composition of the samples are presented in Tables 3-5.

Table 3 - Physico-chemical characteristics of the adsorbent based on semi-coke fines.

Semi-coke fines (3-5 mm)	$W_{rt}, \%$	$A_r, \%$	$V_d, \%$	$V_{\Sigma bywater}, cm^3/g$	$\rho_{bulk}, g/cm^3$	$pH_{aqueous extraction}$	$A_{M.O.}, mg/g$	$A_{M.G.}, mg/g$	$A_{Iodine}, \%$	$S_{BET}, m^2/g$
Initial	7.71	6.6	19.4	0.67	0.67	6	21	95	7.62	8.23
Activated	0.77	13.2	5.99	0.86	0.58	7.5	26.5	100	15.2	227

Table 4 - Elemental composition of the samples

Semicoke fines (fraction 3-5 mm)	Elemental composition, wt. %									
	C	O	Na	Mg	Al	Si	K	Ca	Fe	S
Initial	90.06	8.00	0.06	0.03	0.46	0.34	0.07	0.39	0.26	0.33
Activated	88.1	4.21	0.61	0.13	2.51	3.18	0.23	0.45	0.31	0.23

Table 5 - Test Results of Activated Semicoke Fines (fraction 3-5 mm)

No	Test Indicator	Name of Test Method Nominal	Value, %
1	Particle Size Distribution	ST RK 2405-2013	
	>5 mm 8		8
	3-5 mm		57.9
	1-3 mm		31.9
	0.1-1 mm		1.9
	<0.1 mm 0.3		0.3
	Total		100
2	Structural Strength, PS	ST RK 2243-2012	93.86
3	Mass Fraction of Chlorine on Dry Fuel Basis, Cl _d	GOST 9326-2002, (ISO 587-97)	0.1
4	Mass Fraction of Arsenic on Dry Fuel Basis, As _d	GOST 10478-93, (ISO 601-81, ISO 2590-73)	0.0002
5	Mass Fraction of Total Sulfur on Dry Fuel Basis, S _d	GOST 8606-93, (ISO 334-92)	0.23

As a consequence of steam activation, there is a notable increase in the total pore volume due to water and the adsorption activities of the sample, as assessed by methylene orange, methylene blue, and iodine. Furthermore, the specific surface area of the sample experiences a substantial augmentation from 8.23 m²/g to 227.01 m²/g, representing an almost 28-fold increase. This phenomenon can be attributed to the liberation of volatile constituents during thermal treatment and steam activation, along with the emergence of additional microporous structures.

The content of chlorine, arsenic, and sulfur in the activated semi-coke fines adheres to standard specifications, with levels not exceeding 0.1%, 0.002%, and 1.2%, respectively.

Electron microscopic analysis of the samples is depicted in Figures 3 and 4. The initial sample of semicoke fines exhibits macropores characterized by diameters ranging from 2.22 μm to 39.50 μm. Following steam activation, pores are observed with smaller sizes ranging from 214 nm to 515.7 nm.

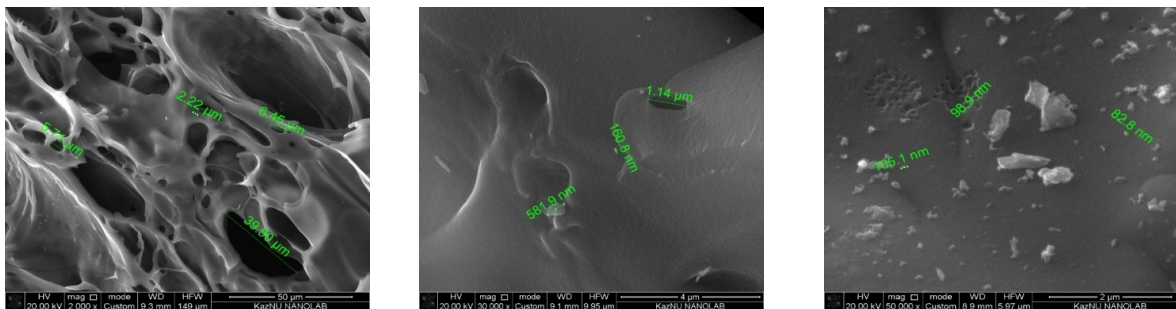


Figure 3 - Electron microscopic images of the initial semi-coke breeze:

a – x2000, b – x30 000, c – x50 000

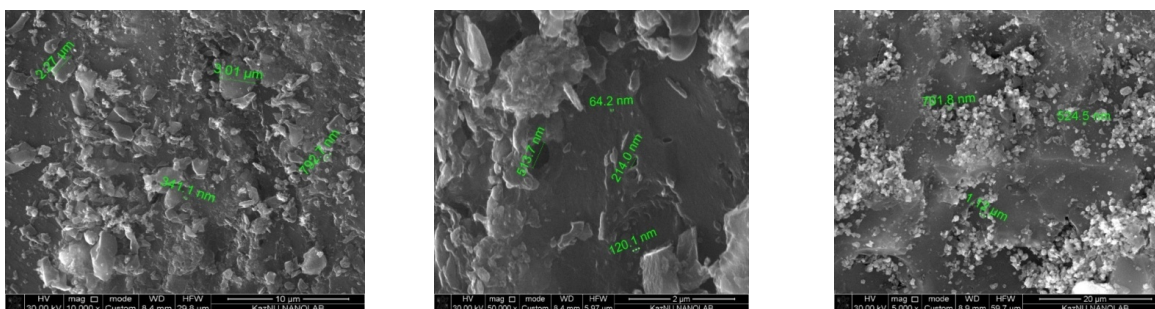


Figure 4 - Electron microscopic images of the activated semi-coke breeze:

a – x2000, b – x30 000, c – x50 000

An activated adsorbent based on semi-coke breeze has been tested for gas purification. An analysis of the kinetic characteristics and sorption capacity was carried out using gas chromatography for the retained volumes of nitrogen, oxygen, hydrogen, carbon monoxide, carbon dioxide, methane, propane, butane, sulfur oxides and nitrogen oxides. The sorption-kinetic characteristics of the adsorbent sample were determined by gas chromatography. From the chromatography data, the specific retained volumes of gases were determined, and the separation selectivity coefficients were determined from their ratio. A sample fraction of 0.2-0.054 mm was sifted out on a laboratory vibrating sieve and packed into chromatographic columns using a vacuum pump. Columns with sample samples were purged in a chromatograph thermostat at a temperature of 150⁰

C for 2 hours. The temperature of the columns is 30⁰C, the temperature of the detector - katharometer and evaporator is not less than 40⁰C. The speed of the carrier gas – argon – is 7 cm³/min. Sample volume – 0.1 ml. Gas samples were taken using rubber samplers from gas cylinders. The specific retention volume was calculated based on the retention time of the analyte gas minus the release time of the helium sample, multiplied by the carrier gas velocity and divided by the mass of the sample in the column. The release time of a helium sample - a gas not absorbed by adsorbents - is determined by the voids in the capillaries in front of the column, after it in front of the detector, as well as the voids between the adsorbent particles in the column, and inside the adsorbent. The test results are presented in Table 6.

As the test showed, the presented adsorbent based

on semi-coke breeze has developed porosity with the presence of micropores in the range of 0.3-2 nm. This is confirmed by the results of gas adsorption chromatography.

Table 6 - Results of studies of the specific retained volume of gases, selectivity coefficients for adsorbent separation

№	Defined indicators	Test results	Units
Specific retention volumes of gases (according to gas chromatography) at 300C			
1	Nitrogen	2,27	cm ³ /g
2	Oxygen	3,47	cm ³ /g
3	Methane	7,84	cm ³ /g
4	Hydrogen	0,73	cm ³ /g
5	Carbon monoxide	3,9	cm ³ /g
6	Carbon dioxide	121,4	cm ³ /g
7	Propane (C ₃ H ₈) at T=140oC	5,4	cm ³ /g
8	Butane (C ₄ H ₁₀) at T=140oC	113,4	cm ³ /g
9	Sulfur dioxide SO ₂	complete absorption	cm ³ /g
10	Nitrogen dioxide NO ₂	complete absorption	cm ³ /g
Gas separation selectivity coefficient (ratio of retained volumes)			
1	CH ₄ /N ₂	3,4	-
2	CH ₄ /CO	2	-
3	CO/H ₂	5,3	-
4	CO ₂ /CH ₄	15,5	-

Conclusions. Based on the findings of the investigation of physical and chemical properties conducted at the laboratory of LLP "Coal Chemistry and Technology Institute," the obtained sample "Activated semi-coke fines (fraction 3-5 mm)" conforms to the classification ST RK 2246-2012. It can be categorized as gas grade marks: AG-3 and AG-5, suitable for gas and liquid adsorption, manufacturing absorbents and catalysts, as well as for respiratory protection equipment (AG-2B) and manufacturing absorbents and catalysts (AG-2A). Moreover, it complies with recuperative activated carbons (AR-A, AR-B, AR-C), intended for capturing and recycling volatile solvent vapors, particularly at high concentrations.

The study showed the prospects of commercializing the technology for the synthesis of carbon adsorbents based on semi-coke breeze for their further use in air purification processes and flue gas capture, gas

purification, ventilation, filters and sorption plants.

The technical outcome of the research encompasses enhanced energy efficiency of the process, augmented specific surface area, specific yield of solid product, strength, porosity, and adsorption activity, alongside reduced volatility attributed to steam thermal activation.

Financing. *The research was carried out with the financial support of the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AR19577512. Development of scientific and technical foundations for the production of microporous carbon nanomaterials for the separation and storage of hydrogen).*

Acknowledgements. The authors express their gratitude to the director of Industrial Solutions and Services LLP, Tolukpaev B.Zh. for his assistance in conducting research and testing the resulting products in industrial conditions.

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