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INFLUENCE OF LIQUID-PHASE OXIDATION OF ACTIVATED CARBON "BAU-A" GRADE WITH HYDROGENIC ACID ON ITS SURFACE STRUCTURE

<u>E. Shaimardan^{1,2}, S. K. Kabdrakhmanova^{1*}, M. M. Beisebekov², B. S. Selenova¹, N. Kantay³, K. Akatan³, Zh. Imangazinova⁴, S. Sydykbayeva⁴, Zh. Sagdollin³</u>

¹ Satbayev University, Almaty, Kazakhstan
 ² Scientific Center of Composite Materials, Almaty, Kazakhstan
 ³ S. Amanzholov East Kazakhstan University, Ust-Kamenogorsk, Kazakhstan
 ⁴ I. Zhansugurov Zhetysu University, Taldykorgan, Kazakhstan

*E-mail for contacts: sanaly33@mail.ru

Activated carbon is often used as a carrier, in the manufacture of catalysts and as a sorbent in medicine and pharmaceuticals, as well as in the purification of natural and waste water from various compounds and the concentration of metal ions in the metallurgical industry. A variety of applications for activated carbon causes different requirements for it. The sorption, structural and textural characteristics predetermine the main properties of activated carbon. Improvement of any characteristics of activated carbons, in order to create materials with the required performance properties, is carried out by surface modification with various agents. In this work, liquid-phase oxidation of commercial activated carbon of the brand "BAU-A" with hydrochloric acid was carried out to improve its surface structure and morphology. The IR spectroscopy established oxygen-containing hydroxyl and phenolic, as well as carboxylic, lactone and quinone groups after modification with hydrochloric acid. This in turn affected the morphology of the coal material, which became relatively organized and distinct. Pore volumes after modification with hydrochloric acid decrease in the intensity of areas $2\theta - 29^{\circ}$ and 43° after modification, indicating the removal of certain minerals with a specific crystal structure, such as Na, Ca and Mg.

Keywords: modification, hydrochloric acid, textural characteristics, morphology, activated carbon.

1. INTRODUCTION

In recent years, porous activated commercial carbons have been used in many studies and interest in them is increasing. Due to the developed porosity, activated carbon is of great practical importance as an applied material [1].

Activated carbon is an amorphous carbon with a highly developed porous structure, large specific surface, various chemical characteristics and high surface activity [2]. It is used as an adsorbent [3], catalyst carrier [4], electrode material [5], as well as for separation, concentration and decolorization [6]. The surface of activated carbon reacts with strong oxidizing agents at the appropriate temperature to improve the content of oxygen-containing functional groups such as carboxyl, phenolic hydroxyl, ether and carbonyl, to increase the polarity and increase the hydrophilicity of the surface [7]. Commonly used basic oxidizing agents are HCl, H₂SO₄, HNO₃, HClO₃, H₂O₂ etc. [8]. Various oxidizing agents change the types and amount of oxygen functional groups on the surface by interacting with activated carbon. The higher the oxidation state, the greater the number of oxygen functional groups [9].

An important step in the development of catalytic systems is the search for a catalyst support, which has a great influence on the structure and catalytic properties of the systems. One of the most promising materials for use in catalysis are carbon carriers, which is due to their purity, developed porous structure, high specific surface area and reactivity. Carbon materials are resistant to the action of acidic and alkaline media, they compare favorably in terms of technical, economic and environmental aspects, as well as in terms of the technology for extracting components from spent catalysts [10–11].

Activated carbon (**AC**) is widely used as a carrier in the production of catalysts for the disposal of persistent organic pollutants [12–13]. This is primarily due to the high porosity and specific surface area (500–2.500 m²/g), which is the optimal parameter for the adsorption of pollutants. In addition, activated carbon can be obtained from naturally occurring, easily renewable materials (e.g. coconut, hardwood, bamboo, lignite, peanut husk, rice husk, coconut heart, etc.) [14].

Therefore, the introduction of a certain functional group and a change in the pore structure by physical and chemical transformation of activated carbon will expand the scope of its application and allow obtaining multipurpose, functional and high-quality activated carbon.

In this work, commercial charcoal of the brand "BAU-A" was investigated, modified and the physical and chemical properties were studied before and after modification.

2. MATERIALS AND METHODS

2.1 Materials

For the study, commercial charcoal of the "BAU-A" brand (STST 6217-74, Russia) was chosen: adsorption activity for iodine is not less than 60%, ash mass fraction and moisture mass fraction, are not more than 6%. The hydrochloric acid was used as a modifier HCl 35%

(STST 3118-77, Russia). The reagents were used without further purification.

2.2 Activated carbon modification "BAU-A"

Activated carbon (AC) was modified with hydrochloric acid, concentration 35% in ratios AC:acid – 1:3 (mass.) by continuous stirring at a temperature of 90 °C for two hours [15]. After cooling, the suspension is washed with deionized water at a temperature of 25 °C to pH=6 and placed in a desiccator for 24 hours. After the time has elapsed, they are calcined in an oven at a temperature of 130–150 °C for 4 hours. After the modification, modified **AC** is designated as **AC**_m.

2.3 FTIR spectroscopy

The chemical structure of **AC** and **AC**_m was studied by IR spectroscopy. FTIR analysis was performed on a spectrometer FTIR FT-801 (Simex, Russia), with a resolution of 1 cm⁻¹ and a wavelength 500–4000 cm⁻¹ according to the standard procedure with the production of a tablet with potassium bromide in a ratio of 1/10 substances, at a temperature of 25 °C and a number of scans of 100. Before use, potassium bromide was crushed and calcined at 200 °C for 3 hours.

2.4 Scanning electron microscopy

The morphology and structure of samples of the original AC and modified AC_m were studied on a Zeiss Auriga Crossbeam 540 high-vacuum scanning electron microscope (Germany). The elemental composition of the carrier and catalysts was determined on an energy-dispersive particle distribution analyzer "Thermo Fisher Scientific" (USA).

2.5 Adsorption porosimetry

The textural characteristics of AC and AC_m before and after modification were determined using adsorption porosimetry (low-temperature nitrogen adsorption) on Autosorb 1 (Quantachrome Instruments, USA) and ASAP-2020 (Micromeritics, USA) devices after preliminary evacuation for 20 hours at a temperature of 200-220 °C for sample degassing, followed by experimental data processing by BET and BJH methods (Barrett-Joyner-Halenda). In order to determine the dependence of V on P at a constant temperature (adsorption isotherm), the adsorbent (sample) is degassed in vacuum at a temperature of 200-300 °C. The specific surface area was measured using the Brunauer-Emmett-Teller (BET) method. The error in measuring the specific surface area (Δ) of standard samples on the above instrument is ± 2.8 rel. %. The distribution of total volume and pore size was determined by the Barrett-Joyner-Halend (BJH) method from the desorption isotherm curve. The BJH method is used to generate adsorption and desorption isotherm curves and pore volume size distributions in porous materials.

2.6 X-ray diffractometry

The crystal structures of substances studied by X-ray diffraction on X[']PertPRO diffractometer (Malvern Panalytical Empyrean, Netherlands) using monochromatized copper (CuK α) radiation with a scan step of 0.02°,

K-Alpha1 [Å] 0.1542. The measurement angle was $10-45^{\circ}$, the X-ray tube voltage was 45 kV, the current intensity was 30 mA, and the measurement time at each step was 0.5 s and an aluminium rectangular multipurpose sample holder ((PW1172/01) was used for the measurement in reflection mode.

2.7 TGA analysis

The thermal characteristics of the original and modified activated carbon were studied on a differential thermogravimetric analyzer STA449C (NETZSCH, Germany) in an argon atmosphere within the temperature range of $30\pm5-700\pm5$ °C. The heating rate was 10 ± 1 °C/minutes and mass of the samples was about 20 ± 2 mg.

3. RESULTS AND DISCUSSION

3.1 Physical and chemical characteristics of AC and ACm

The IR spectra of the initial activated carbon **AC** are characterized by the presence of a weak line at 2904–1053 cm⁻¹, corresponding to the stretching vibration of the C–H bond and a weak absorption of the stretching vibration of 3500 cm⁻¹, characteristic of the OH group of carboxylic acid, phenols and adsorbed water (Figure 1a).

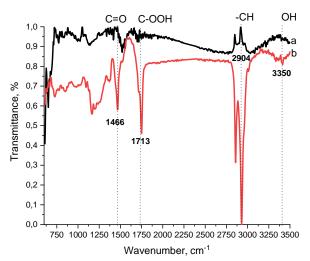


Figure 1. IR spectra: initial AC (a); activated carbon modified by hydrochloric acid AC_m (b)

After modification of AC with hydrochloric acid (AC_m) increases the phenolic hydroxyl group (Figure 1b). In spectrum of AC_m there is a band in the region of 1713 cm⁻¹, related to the C=O stretching vibrations of carboxylic and lactone groups (Figures 1 and 2). The band in the AC_m spectrum at 1495 cm⁻¹ can be attributed to strongly conjugated carbonyls (quinone group) and v(C=C) vibrations in the aromatic ring. The results obtained are in good agreement with previous studies.

The acid treatment of activated carbon removes ions such as Ca, Mg, K and thus increases the carboxyl groups, which contributes to an increasing in polarity. In general, during acid modification, the nature of the surface and the porosity of activated carbon undergoes a certain change, while maintaining the original structure.

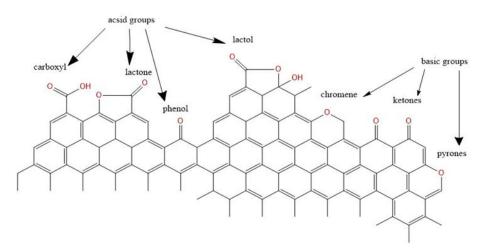


Figure 2. A fragment of the structural formula of modified activated carbon AC_m

The results of SEM with energy dispersive analysis are shown in Figures 3a-3b and in Table 1. The initial **AC** is heterogeneous in its dispersed and morphological composition and has a capillary-tubular structure. Separate fragments are characterized by a sieve-like structure with through cylindrical holes with small white particles on the surface of the pores (Figure 3a). The pores are characterized by relatively the same shape, their sizes are in the range of 1.517–10.97 microns.

The modification of activated carbon with hydrochloric acid resulted in a slight change in the morphology of the carbon material (Figure 3b). A decrease in the number of various fragments is observed. The cellular-porous structure became relatively organized and clear, the number of white particles on the surface of the pores decreases. There is an increase in the number of pores that have acquired the correct shape and the same size. It has been established that hydrochloric acid modification of "BAU-A" activated carbon leads to the destruction of pore walls, contributing to the transition from micro- and mesopores to macropores, which is consistent with previous studies [16-17]. Pore volumes after modification with acid are in the range of 4.264-5.778 μ m, the partition between the pores has a size of 1.618-3.580 µm (Figure 3b). In places, a macrocell is observed, the dimensions of which correspond to 31.57-73.32 microns (Figure 3b).

Comparative data of EDS analysis indicates the absence of elements such as calcium, magnesium, potassium, sulfur and phosphorus after modification with hydrochloric acid (Figure 3a and 3b, Table 1). This agrees with the results of X-ray diffraction analysis, which are presented below. There is also a decrease in the carbon content, while the oxygen content increases (Table 1). Textural characteristics of the initial and modified activated carbon according to nitrogen adsorption isotherms are presented in Table 2 and in Figures 4–5. According to the obtained data, the initial activated carbon can be classified as mesoporous (Table 2). As can be seen from Table 2, when activated carbon is modified, the specific surface area of the sample decreases from 280.8 to 233.8 m²/g, with a corresponding decrease in the total pore volume from 0.22 to 0.19 ml/g and the pore size from 141.19 up to 122.12 nm. The initial and modified activated carbon has a similar pore size distribution (Figure 4). Both samples contain mesopores down to 19 nm with an additional peak at 26 nm indicating the presence of some macropores.

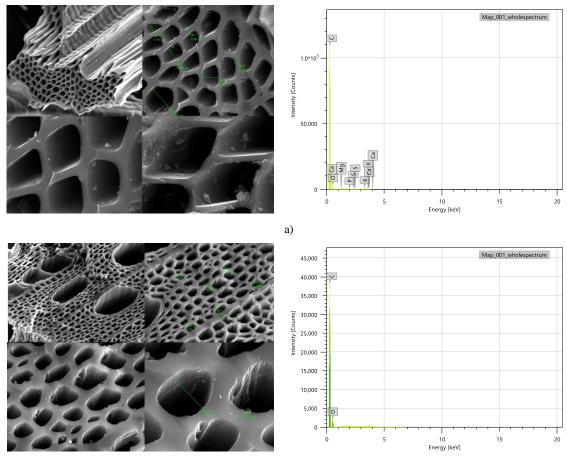
Table 2. Characteristics of the porous structure of the initial AC and hydrochloric acid-modified activated carbon AC_m

Sample	SW (specific pore surface), m ² /g	Dv(r) (Pore Radius₀ – pore size), nm	c – V _P (total pore volume), ml/g		
AC	280,8	141,19	0,19		
ACm	233,8	122,12	0,22		

The treatment of activated carbon with hydrochloric acid increases its adsorption capacity, due to the dissolution of inorganic constituents, and the oxidation of precipitated carbon, as determined by an increase in the oxygen content after **AC** modification by SEM with EDS analyzes (Table 1). This, in turn, affects the porous structure of coal, being localized in the walls of the pores. Liquid-phase modification of activated carbon with concentrated acid increases the number of oxygen functional groups on the surface of carbon, which we have established by IR spectroscopy, with a slight change in the porous structure of activated carbon (Figures 1–2).

Table 1. Results of EDS analysis of the initial AC and hydrochloric acid-modified activated carbon AC_m

Sample	Elements (mass., %)							
	С	0	К	Mg	Ca	S	Р	
AC	89,85±0,06	8,39±0,07	0,71±0,01	0,24±0,01	0,57±0,01	0,05±0,00	0,19±0,01	
ACm	80,21±0,03	19,79±0,06	_	_	-	-	-	



b)

Figure 3. SEM microphotographs with EDS analyses: initial AC(a); hydrochloric acid modified activated carbon $AC_m(b)$

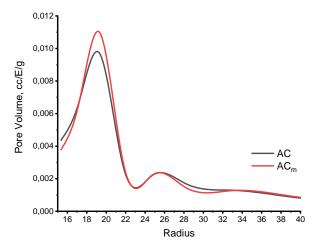


Figure 4. Distribution of pores by size of the initial and modified with hydrochloric acid activated carbon AC_m

The nitrogen adsorption curve belongs to type IV and confirms it as a mesoporous material with the possible presence of interparticle pores (Figure 5). The adsorption and desorption isotherms for the original AC sample are slightly higher than for the modified AC_m sample, which indicates that the total pore volume of AC is greater than.

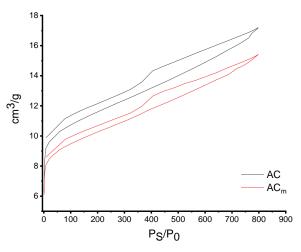


Figure 5. N₂ adsorption-desorption isotherms of the initial and modified activated carbon

SEM data with energy dispersive analyzers and texture characteristics are confirmed by the results of X-ray phase study. X-ray phase analysis of AC and AC_m are presented in Figures 6a and 6b. The X-ray diffraction pattern of AC and AC_m has a similar appearance (Figures 6a and 6b). The activated charcoal is naturally amorphous. According to

the obtained X-ray pattern of the original AC, the diffraction peaks, $2\theta - 23^{\circ}$ (002), 29° ($\overline{2}04$), 43° (101) and 50° (151) indicate a disordered amorphous structure of the substance, which is in good agreement with previous studies [18-20]. The initial AC is characterized by a monoclinic carbon lattice C (002), C (101) and Miller indices C-H-O (204) and C-H-O (151), the intensity of which after modification decreases in the regions $2\theta - 29^{\circ}$ and 43°. This indicates a decrease in some minerals with a certain crystal structure, such as Na, Ca and Mg. After modification with hydrochloric acid, AC_m retains its original amorphous state (Figure 6b). This confirms the broad peaks in the 2 θ region at 23.86° (d - 3.71), 44.08° (d-2.00) and 51.45° (d-1.77). It should be noted that the peak in the region $2\theta - 29^{\circ}$ ($\overline{2}04$) disappeared, which indicates the absence of phosphate and polyphosphate bonds after modification. Perhaps this is due to the hydrochloric acid decomposition of phosphates and polyphosphates during the modification of activated carbon.

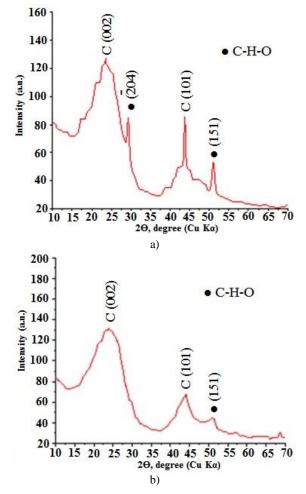


Figure 6. XRD diffraction of: initial AC(a); activated carbon modified with hydrochloric acid $AC_m(b)$

Figure 7 shows the results of thermogravimetric analysis of the initial and modified activated carbon of the "BAU-A" brand. As shown in Figure 7, the decrease

in the bulk of the initial activated carbon occurs in the temperature ranges of $40-150^{\circ}$ C and $250-700^{\circ}$ C. A sharp decrease in mass in the low-temperature zone, i.e. between $40-120^{\circ}$ C, is due to the evaporation of moisture, i.e. release of surface and adsorption water. In the temperature range of $120-700^{\circ}$ C, there is a gradual decrease in the mass of the initial "BAU-A", which is due to the combustion of organic compounds (Figure 7a). After modification with hydrochloric acid, there is a sharp decrease in the bulk in the temperature range of $220-250^{\circ}$ C compared to the initial **AC** (Figure 7b).

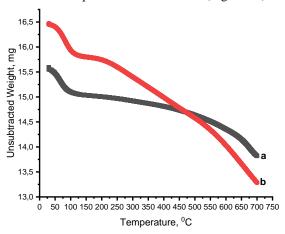


Figure 7. TGA curves of initial AC (a) and hydrochloric acid-modified activated carbon AC_m (b)

This is possibly due to the decrease in mineral constituents and the presence of additional carboxylic, lactone and quinone functional groups after modification, which confirms the above results. In general, the degree of thermal decomposition depends on the functional groups located on the surface of activated carbon. Therefore, an increase or decrease in the thermal decomposition of **AC** indicates the presence of certain functional groups on its surface. Carboxylic acids, carboxylic acid anhydrides and lactone groups decompose at about 400 °C, while other surface functional groups (phenolic, carbonyl, quinones and pyrone structures) require more energy to decompose (above 400 °C).

4. CONCLUSION

Thus, the "BAU-A" charcoal was studied before and after modification with hydrochloric acid. The hydrochloric acid modification of activated carbon leads to an increase in hydroxyl and phenolic groups, as well as the appearance of additional carboxylic, lactone and quinone groups. This, in turn, leads to the fact that activated carbon after acid modification retains its amorphous structure, and the cellular-porous structure becomes relatively organized and clear, due to the washing out of mineral components. This increases the adsorption capacity, polarity and thermal stability of activated carbon. The N₂ adsorption-desorption isotherm of the original and modified activated carbon established mesoporousness with the possible presence of interparticle pores in the structure.

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«БАУ-А» БЕЛСЕНДІРІЛГЕН КӨМІРДІ ТҰЗ ҚЫШҚЫЛЫМЕН СҰЙЫҚ-ФАЗАЛЫ ТОТЫҚТЫРУДЫҢ ОНЫҢ БЕТТІК ҚҰРЫЛЫМЫНА ӘСЕРІН ЗЕРТТЕУ

<u>Е. Шаймардан</u>^{1,2}, С. Қ. Қабдрахманова^{1*}, М. М. Бейсебеков², Б. С. Селенова¹, Н. Қантай³, Қ. Ақатан³, Ж. Имангазинова⁴, С. Сыдықбаева⁴, Ж. Сағдоллин³

¹ Сәтбаев Университеті, Алматы, Қазақстан
 ² Композиттік материалдар ғылыми орталығы, Алматы, Қазақстан
 ³ С. Аманжолов атындағы Шығыс Қазақстан университеті, Өскемен, Қазақстан
 ⁴ Жетісу Университеті, Талдықорған, Қазақстан

*Байланыс үшін E-mail: sanaly33@mail.ru

Белсендірілген көмір көп жағдайда тасымалдаушы ретінде катализатор өндірісінде және медицинада мен фармацевтикада саласында сорбент ретінде, сонымен қатар табиғи және металлургия өнеркәсібінің қалдық суын әртүрлі қосылыстардан тазарту және де металл иондарын концентрациялау үшін кеңінен қолданылады. Белсендірілген көмірді әр салада қолдану, оған қойылатын талаптың да әр қилы болуына жол ашады. Сорбциялық, құрылымдық және текстуралық сипаттамалар белсендірілген көмірдің негізгі қасиеттерін анықтайтындығы белгілі. Қасиеті алдын ала белгіленген материалдар алу үшін белсендірілген көмірлердің беттік құрылымын алдын ала түрлі агенттермен модификациялау жүзеге асырылады. Зерттеу жұмысында «БАУ-А» маркалы тауарлық белсендірілген көмірді тұз қышқылымен сұйық-фазалы тотықтыру арқылы оның беттік –құрылымы мен морфологиясын жақсарту жұмысы орындалды. ИҚ-спектроскопия әдісі көмегімен тұз қышқылымен модификацияланған белсендірілген көмір құрылымынан оттекті гидроксил және фенол, сонымен қатар карбоксил, лактон және хинон функционалды топтарды анықталды. Модификация үрдісі өз кезегінде көмірдің морфологиясына әсер етіп, оның құрылымын ұйымдасқан түрге ауыстырады. Тұз қышқылымен модификациядан кейін кеуек өлшемі 4,264–5,778 мкм дейін төмендеп, макрокеуек өлшемдері 31,57–73,32 микронға сәйкес келді. ХRD талдауы модификациядан кейін 20 – 29° және 43° аймақтардың қарқындылығының төмендеуін анықтады, бұл белгілі бір кристалдық құрылымы бар кейбір минералдардың, мысалы, Na, Ca, Mg жойылғанын көрсетеді.

Түйін сөздер: модификация, тұз қышқылы, текстуралық сипаттама, морфология, белсендірілген көмір.

ВЛИЯНИЕ ЖИДКОФАЗНОГО ОКИСЛЕНИЯ АКТИВИРОВАННОГО УГЛЯ МАРКИ «БАУ-А» СОЛЯНОЙ КИСЛОТОЙ НА ЕГО ПОВЕРХНОСТНУЮ СТРУКТУРУ

<u>Е. Шаймардан^{1,2}, С. К. Кабдрахманова^{1*}, М. М. Бейсебеков², Б. С. Селенова¹, Н. Кантай³, К. Акатан³, Ж. Имангазинова⁴, С. Сыдыкбаева⁴, Ж. Сагдоллин³</u>

¹ Сатпаев Университет, Алматы, Казахстан
 ² Научный центр композитных материалов, Алматы, Казахстан
 ³ Восточно-Казахстанский университет им. С. Аманжолова, Усть-Каменогорск, Казахстан
 ⁴ Жетысу Университет, Талдыкорган, Казахстан

*E-mail для контактов: sanaly33@mail.ru

Активированный уголь часто применяется в качестве носителя, в изготовлении катализаторов и как сорбент в медицине и фармацевтике, а также при очистке природных и сточных вод от различных соединений и концентрирования ионов металлов в металлургической промышленности. Разнообразие областей применения активированного угля обуславливает и различные требования к нему. Сорбционная, структурная и текстурная характеристика предопределяет основные свойства активированного угля. Улучшение каких-либо характеристик активированных углей, с целью создания материалов с требуемыми эксплуатационными свойствами, осуществляется путем модификации поверхности различными агентами. В работе проведено жидкофазное окисление коммерческого активированного угля марки «БАУ-А» соляной кислотой для улучшения его поверхностной структуры и морфологии. ИК-спектроскопия установила кислородсодержащие гидроксильные и фенольные, а также карбоновые, лактоновые и хиноновые группы после модификации соляной кислотой. Это в свою очередь повлияло на морфологию угольного материала, которая стала относительно организованной и четкой. Объемы пор после модификации соляной кислотой уменьшилась до 4,264–5,778 мкм, размеры макроячеек соответствуют 31,57–73,32 мкм. XRD-анализ установил уменьшение интенсивности в областях 20 – 29° и 43° после модификации, указывающей на удаление некоторых минеральных веществ с определенной кристаллической структурой, таких как Na, Ca, Mg.

Ключевые слова: модификация, соляная кислота, текстурные характеристики, морфология, активированный уголь.