

# Porous Structure and Krypton Sorption Capacity of Carbon Sorbents from a Composite of Hydrolytic Lignin and Phenol-Lignin-Formaldehyde

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**Abstract**—The porous structure of granular activated carbons from a composite of hydrolytic lignin and phenol-lignin-formaldehyde resin was studied. The volumes of characteristic pore types, characteristic adsorption energy, and main technical characteristics were determined. The possibility of obtaining microporous, mechanically strong sorbents based on hydrolytic lignin (hazard class 3 waste) by adding phenol-lignin-formaldehyde resin to the starting composite raw material was shown. Thirty five percent combustion loss was found to be optimum for activated carbons from this type of raw material and makes it possible to obtain a sorbent with a micropore volume of 0.36 cm<sup>3</sup>/g and mechanical abrasion strength of at least 82%, which is equivalent to commercial activated carbons in sorption and technical characteristics. It was shown that the carbonizate from lignin and phenol-lignin-formaldehyde resin has pronounced molecular sieve properties: the pore volume of 0.30 cm<sup>3</sup>/cm<sup>3</sup> is available only for molecules with a diameter of no more than 0.44 nm. The possibility of using active carbons from lignin for sorption of radioactive noble gases was evaluated using krypton sorption as an example.

**Keywords:** porous structure, activated carbon, lignin, sorption

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Due to its physicochemical properties, activated carbon is a unique sorbent that can be used to address issues of chemical and biological safety of humans and environment.

Over the past few years, the average annual growth of global consumption of activated carbon was ~5.5% and at the end of 2019 amounted to ~1.85–1.90 million tons. In 2000, the production of activated carbons in Russia did not exceed 7 thousand t/year, and a noticeable increase in production volume has started only since 2016. In 2020, Russian enterprises produced 12 059.3 tons of activated carbon [1]. The lack of high-quality Russian-made activated carbons leads to problems in some industries such as gas processing, gold hydrometallurgy, purification of gas emissions from nuclear power plants, drinking water supply, soil detoxification, and anti-gas equipment [2].

Microporous granular active carbons are used for adsorption of gases and vapors. Russian industry currently produces the following brands of gas and recuperative active coals from three types of raw materials: AG-2, SKT, AR, SKT-3, ART, BAU (BAU-A, BAU-K, BAU-MF). Coals AG-2 (grades A and B) and AR (AR-A, AR-B, AR-V) are obtained from coal dust and tar by steam-gas activation. Coal SKT is synthesized from peat, and SKT-3 and ART (ART-1 and ART-2),

from peat and coal dust by chemical activation. Coal BAU is obtained from grade A charcoal by processing it with steam at temperatures above 800°C. Activated carbons for degassing are characterized by micropore volumes of 0.24–0.48 cm<sup>3</sup>/g at a total pore volume of 0.52–1.00 cm<sup>3</sup>/g; their bulk density is 0.30–0.60 g/cm<sup>3</sup>.

The main volume (80–85%) of activated carbon is produced from non-renewable resources such as mineral coals, wood, and peat [2, 3]. Chemical activation is an effective method for structuring carbon-containing materials, including peat, to obtain highly active sorbents with a given porous structure. The use of a potassium sulfide solution for peat impregnation and subsequent carbonization and activation makes it possible to obtain active carbons of the SKT type with micropore volumes of 0.35–0.60 cm<sup>3</sup>/g, at least 60% strength, and high sorption properties, including for inert and noble gases, due to the presence of a sulfur heteroatom in the structure of the material [4].

Lignin formed by percolation of sulfuric acid through a wood layer in the technological cycle of hydrolysis production is a complex mixture of products of hydrolytic decomposition of wood, which includes altered lignin of plant fiber, polysaccharides,

**Table 1.** Dependence of the technical characteristics of carbon adsorbents on the combustion loss

| $\alpha$ , %    | Sample<br>(starting raw material) | $\Delta$ , g/cm <sup>3</sup> | $\delta$ , g/cm <sup>3</sup> | $\rho$ , g/cm <sup>3</sup>    |                  | $A$ , % | $H$ , % |
|-----------------|-----------------------------------|------------------------------|------------------------------|-------------------------------|------------------|---------|---------|
|                 |                                   |                              |                              | C <sub>6</sub> H <sub>6</sub> | CCl <sub>4</sub> |         |         |
| 0 (carbonizate) | Lignin                            | 0.35                         | 0.66                         | 1.86                          | 1.47             | 5       | 89      |
|                 | FLA-4                             | 0.53                         | 1.15                         | 1.59                          | 1.50             | 1       | 99      |
|                 | Lignin + 60% FLA-4                | 0.51                         | 1.04                         | 1.64                          | 1.55             | 3       | 99      |
| 15 ± 2          | Lignin                            | 0.29                         | 0.56                         | 1.82                          | 1.61             | 6       | 85      |
|                 | FLA-4                             | 0.44                         | 0.90                         | 1.67                          | 1.62             | 3       | 99      |
|                 | Lignin + 60% FLA-4                | 0.45                         | 0.91                         | 1.70                          | 1.60             | 4       | 98      |
| 25 ± 2          | Lignin                            | 0.28                         | 0.51                         | 1.92                          | 1.71             | 8       | 80      |
|                 | FLA-4                             | 0.42                         | 0.85                         | 1.69                          | 1.64             | 3       | 98      |
|                 | Lignin + 60% FLA-4                | 0.39                         | 0.80                         | 1.79                          | 1.65             | 5       | 97      |
| 35 ± 2          | Lignin                            | 0.25                         | 0.46                         | 1.97                          | 1.82             | 9       | 75      |
|                 | FLA-4                             | 0.39                         | 0.72                         | 1.78                          | 1.71             | 3       | 97      |
|                 | Lignin + 60% FLA-4                | 0.36                         | 0.69                         | 1.92                          | 1.89             | 7       | 96      |

$\alpha$  – combustion loss,  $\Delta$  – bulk density,  $\delta$  – apparent density,  $\rho$  – pycnometric density,  $A$  – ash content,  $H$  – strength.

lignohumic substances, unwashed mineral and organic (mainly sulfuric, formic, and acetic) acids, and ash elements [5]. According to its chemical composition, lignin is a promising material for the production of active carbons. There are more than 2 million tons of hydrolytic lignin in the dumps of hydrolysis enterprises [6]. In view of their environmental hazard and large tonnage, the use of lignin wastes as raw materials for the production of high-quality activated carbons is also very important.

Despite some valuable advantages of lignin, such as the possibility of granulation without a binder and low cost, activated carbons based on it have a large volume of macropores and low mechanical strength. The introduction of some specific binders in lignin, for example, phenol-lignin-formaldehyde resin, will increase the strength of adsorbents, reducing the volume of large pores and increasing that of micropores. The goal of this study was to investigate the porous structure and sorption properties of activated carbon obtained from a composite of lignin and phenol-lignin-formaldehyde resin.

## EXPERIMENTAL

Carbon sorbents from a composite of hydrolytic lignin and phenol-lignin-formaldehyde resin were studied.

Hydrolytic lignin (Petrosirt, on the territory of Leningrad Hydrolysis Plant) and phenol-lignin-formaldehyde resin FLA-4 (Institute of Pulp and Paper Industry, VNIIB) were used as raw materials for the production of activated carbons.

After grinding to a fraction of less than 160  $\mu\text{m}$ , lignin was mixed with water or an aqueous solution of phenol-lignin-formaldehyde resin and molded on a

screw granulator. The resin content in the mixed samples was 60 wt %, providing sufficient strength of the resulting activated carbon. The formed granules were subjected to carbonization at (650–700) $^{\circ}\text{C}$ , followed by activation at (800–850) $^{\circ}\text{C}$ .

The quality of the resulting sorbents was evaluated by the methods adopted for certification of activated carbons.

The bulk density, ash content, and abrasion resistance were determined in accordance with ASTM D 2854, 2866, and 3802, respectively. Apparent density was determined by the volumetric method, and pycnometric density by pycnometric method using benzene and carbon tetrachloride as a pycnometric liquid. The parameters of the microporous structure were calculated from the benzene adsorption isotherm determined by the adsorption-vacuum method. The limiting volume of the sorption space was determined by the desiccator method. The adsorption of krypton was determined in a closed circulation unit by the tracer method.

## RESULTS AND DISCUSSION

The results of the porous and microporous structure studies and technical characteristics of granular activated carbons made from a composite of hydrolytic lignin and phenol-lignin-formaldehyde resin obtained at different degrees of combustion loss were compared.

The dependence of the technical characteristics of the obtained adsorbents on the degree of combustion loss is presented in Table 1.

The data presented in Table 1 show that the carbonizate of the mixed sample has higher apparent density compared with the carbonizate of the lignin

**Table 2.** Parameters of the porous structure of activated coals

| $\alpha$ , % | Sample             | $V_{\Sigma}$ , cm <sup>3</sup> /cm <sup>3</sup> | $V_{ma}$ , cm <sup>3</sup> /cm <sup>3</sup> | $V_{me}$ , cm <sup>3</sup> /cm <sup>3</sup> | $V_{mi}$ , cm <sup>3</sup> /cm <sup>3</sup> |
|--------------|--------------------|---|---|---|---|
| 15 ± 2       | Lignin             | 0.69  | 0.49  | 0.10  | 0.09  |
|              | FLA-4              | 0.46  | 0.24  | 0.08  | 0.14  |
|              | Lignin + 60% FLA-4 | 0.47  | 0.18  | 0.09  | 0.19  |
| 25 ± 2       | Lignin             | 0.73  | 0.50  | 0.09  | 0.10  |
|              | FLA-4              | 0.53  | 0.31  | 0.08  | 0.15  |
|              | Lignin + 60% FLA-4 | 0.55  | 0.24  | 0.10  | 0.23  |
| 35 ± 2       | Lignin             | 0.76  | 0.56  | 0.09  | 0.11  |
|              | FLA-4              | 0.60  | 0.32  | 0.09  | 0.19  |
|              | Lignin + 60% FLA-4 | 0.64  | 0.28  | 0.11  | 0.25  |

$\alpha$  – combustion loss,  $V_{\Sigma}$  – total pore volume,  $V_{ma}$  – macropore volume,  $V_{me}$  – mesopore volume,  $V_{mi}$  – micropore volume.

sample; this suggests potential development of microporosity during further activation.

The pycnometric density increases with the combustion loss for all the samples. At (15 ± 2)% combustion loss, the pore volume is not yet totally available for carbon tetrachloride, while at (35 ± 2)%, the pycnometric densities determined using benzene and carbon tetrachloride coincide almost completely and correspond to the values for activated carbons. This suggests that the carbon skeleton of the adsorbent completely forms at (35 ± 2)% combustion loss.

According to Table 1, an increase in the combustion loss leads to a decrease in the mechanical strength of the samples; for the sample prepared from lignin, the mechanical strength decreases by 20% and amounts to 55%; as a consequence, it is impossible to use this coal in dynamic adsorption processes. The introduction of phenol-lignin-formaldehyde resin in the raw material leads to compaction of the material and an increase in its mechanical strength, which meets the requirements to high-strength industrial activated carbons.

In some applications of activated carbons, e.g., water purification, special requirements are imposed on the ash content of the material. The introduction of low-ash phenol-lignin-formaldehyde resin (whose ash content is 1%) makes it possible to reduce the ash content of activated lignin coals by 20%.

We studied the parameters of the porous structure of the obtained activated carbons (Table 2).

As shown by this study of their porous structure, activated carbons from lignin are characterized by a significant volume of macropores, which increases with the combustion loss, reducing the mechanical strength. At the same time, an increase in the combustion loss for the samples from lignin does not lead to an increase in the micropore volume. The introduction of phenol-lignin-formaldehyde resin in lignin is accompanied by resin penetration into the space between lignin particles and into the lignin structure, forming a denser composite carbon-containing raw material;

this significantly decreases the proportion of macropores and increases the proportion of micropores in the resulting mixed samples of carbon adsorbents. The activation is accompanied by a gradual burnout of the structural fragments of the initial carbon-containing raw materials, which differ in their thermal stability and reactivity. Carbon from lignin is obtained (activated) at earlier stages than carbon from tar (pore opening occurs already at 15% combustion loss). During activation in the mixed sample, the resin component makes a major contribution to the development of the porous and especially microporous structure of the resulting carbon adsorbent. In this case, the micropore volume of the carbon adsorbent from the mixed raw material exceeds the analogous characteristic of coals from both lignin and phenol-lignin-formaldehyde resin.

The parameters of the microporous structure of the obtained activated carbons were calculated from the adsorption isotherms of benzene vapors (Table 3).

As shown by the parameters of the microporous structure, as the combustion loss increases, the size of micropores slightly increases and the characteristic adsorption energy, calculated using the TVFM (theory for volume filling of micropores) equation of M.M. Dubinin, decreases along with an increase in the limiting volume of sorption space; when the combustion loss increases from 15 to 35%, the characteristic adsorption energy decreases from 21 to 19 kJ/mol for the sample from mixed raw material.

Table 4 shows the limiting volumes of the sorption space for adsorbates with different critical diameters of molecules.

According to Table 4, both the micropore volume and size develop at increased combustion losses, which makes it possible to achieve controlled optimum combustion loss. The introduction of phenol-lignin-formaldehyde resin in the sorbent results in increased volume and size of small pores inaccessible for the relatively large benzene–isooctane–carbon tetrachloride molecules, which is clearly seen if we

**Table 3.** Parameters of the microporous structure of activated coals

| $\alpha$ , % | Sample             | $W_0$ , cm <sup>3</sup> /cm <sup>3</sup> | $E$ , kJ/mol | $\bar{X}$ , nm |
|--------------|--------------------|--|--------------|----------------|
| 15 ± 2       | Lignin             | 0.11                                     | 22           | 0.55           |
|              | FLA-4              | 0.14                                     | 21           | 0.56           |
|              | Lignin + 60% FLA-4 | 0.22                                     | 21           | 0.56           |
| 25 ± 2       | Lignin             | 0.11                                     | 21           | 0.58           |
|              | FLA-4              | 0.15                                     | 20           | 0.59           |
|              | Lignin + 60% FLA-4 | 0.23                                     | 20           | 0.59           |
| 35 ± 2       | Lignin             | 0.13                                     | 20           | 0.60           |
|              | FLA-4              | 0.20                                     | 20           | 0.61           |
|              | Lignin + 60% FLA-4 | 0.25                                     | 19           | 0.64           |

$\alpha$  – combustion loss,  $W_0$  – limiting volume of adsorption space,  $E$  – characteristic adsorption energy,  $\bar{X}$  – micropore half-width.

**Table 4.** Limiting volumes of sorption space for various adsorbates (the critical size of sorbate molecules is indicated [7])

| Combustion loss, % | Sample             | $W_s$ , cm <sup>3</sup> /cm <sup>3</sup> |                             |  |                       |                             |
|--------------------|--------------------|--|-----------------------------|--|-----------------------|-----------------------------|
|                    |                    | CH <sub>3</sub> OH<br>0.44 nm            | <i>n</i> -octane<br>0.49 nm | C <sub>6</sub> H <sub>6</sub><br>0.58 nm | Iso-octane<br>0.67 nm | CCl <sub>4</sub><br>0.69 nm |
| 0 (carbonizate)    | Lignin             | 0.12                                     | 0.12                        | 0.08                                     | 0.04                  | 0.03                        |
|                    | FLA-4              | 0.23                                     | 0.20                        | 0.16                                     | 0.11                  | 0.08                        |
|                    | Lignin + 60% FLA-4 | 0.30                                     | 0.07                        | 0.07                                     | 0.07                  | 0.04                        |
| 15 ± 2             | Lignin             | 0.19                                     | 0.18                        | 0.19                                     | 0.17                  | 0.13                        |
|                    | FLA-4              | 0.24                                     | 0.24                        | 0.22                                     | 0.18                  | 0.13                        |
|                    | Lignin + 60% FLA-4 | 0.36                                     | 0.28                        | 0.28                                     | 0.28                  | 0.23                        |
| 25 ± 2             | Lignin             | 0.31                                     | 0.20                        | 0.19                                     | 0.19                  | 0.16                        |
|                    | FLA-4              | 0.25                                     | 0.25                        | 0.23                                     | 0.19                  | 0.14                        |
|                    | Lignin + 60% FLA-4 | 0.41                                     | 0.40                        | 0.31                                     | 0.27                  | 0.25                        |
| 35 ± 2             | Lignin             | 0.41                                     | 0.20                        | 0.20                                     | 0.21                  | 0.19                        |
|                    | FLA-4              | 0.32                                     | 0.30                        | 0.28                                     | 0.23                  | 0.19                        |
|                    | Lignin + 60% FLA-4 | 0.43                                     | 0.38                        | 0.36                                     | 0.37                  | 0.36                        |

$\alpha$  – combustion loss,  $W_s$  – limiting volume of sorption space.

compare the limiting volumes of the sorption space. At a combustion loss increased from 25 to 35%, the limiting volume of the sorption space does not change, while the proportion of pores of more than 0.69 nm increases from 61 to 84%.

In contrast to the samples from lignin and phenol-lignin-formaldehyde resin, the carbonized mixed sample has a pronounced molecular sieve effect, the pore volume of 0.30 cm<sup>3</sup>/cm<sup>3</sup> being available only for molecules with a diameter of 0.44 nm. In this case, only the pore volume of 0.04 cm<sup>3</sup>/cm<sup>3</sup> is available for the carbon tetrachloride molecule. Activation of the material to 35% combustion loss leads to equalization of pore volumes available for molecules with different critical diameters. For example, for methanol molecules (0.44 nm), the available pore volume for activated carbon from mixed raw material is 0.43

cm<sup>3</sup>/cm<sup>3</sup>, while for *n*-octane molecules (0.49 nm) and up to carbon tetrachloride (0.69 nm), it is 0.36–0.38 cm<sup>3</sup>/cm<sup>3</sup>, which are close values.

While having high mechanical strength, activated carbon from lignin and phenol-lignin-formaldehyde resin is not inferior to industrial coals used for purification of gases and vapors in the parameters of the porous structure and adsorption from the liquid phase (Table 5).

Thus, the results of these studies showed that the introduction of phenol-lignin-formaldehyde resin in lignin at the mixing stage makes it possible to obtain activated carbons with a micropore volume of 0.36 cm<sup>3</sup>/g and mechanical abrasion strength of at least 82%. It was found that 35% combustion is optimum for activated carbons from this type of raw material. The carbonizate of lignin and phenol-lignin-

**Table 5.** Characteristics of activated coals

| Sample                                      | $V_{ma}$ , cm <sup>3</sup> /g | $V_{me}$ , cm <sup>3</sup> /g | $V_{mi}$ , cm <sup>3</sup> /g | $\Pi$ , %   |
|---|-------------------------------|-------------------------------|-------------------------------|-------------|
| Lignin + 60% FLA-4<br>(35% combustion loss) | 0.41                          | 0.16                          | 0.36                          | 82          |
| BAU-A*                                      | 0.35–1.45                     | 0.08–0.10                     | 0.22–0.25                     | at least 60 |
| ADG-II                                      | 0.13–0.25                     | 0.03–0.12                     | 0.50–0.85                     | at least 75 |
| SKT-3                                       | 0.25–0.32                     | 0.06–0.09                     | 0.37–0.46                     | at least 71 |
| ATsB-1                                      | 0.17–0.19                     | 0.16–0.26                     | 0.42–0.47                     | at least 75 |

$V_{ma}$  – macropore volume,  $V_{me}$  – mesopore volume,  $V_{mi}$  – micropore volume,  $\Pi$  – mechanical abrasion strength; \*: characteristics of commercial activated coals [8].

**Table 6.** Adsorption coefficients for krypton

| $\alpha$ , %    | Sample             | $\Gamma_{Kr}$ , cm <sup>3</sup> /cm <sup>3</sup> |     |       |       |       |
|-----------------|--------------------|--|-----|-------|-------|-------|
|                 |                    | +20°C  | 0°C | –20°C | –40°C | –60°C |
| 0 (carbonizate) | Lignin             | 23   | 35  | 56    | 99    | 193   |
|                 | Lignin + 60% FLA-4 | 14.5   | 18  | 25    | 35    | 52.5  |
| 15 ± 2          | Lignin             | 42   | 58  | 85    | 135   | 235   |
|                 | Lignin + 60% FLA-4 | 58   | 83  | 118   | 186   | 312   |
| 25 ± 2          | Lignin             | 49   | 68  | 98    | 152   | 262   |
|                 | Lignin + 60% FLA-4 | 89   | 116 | 158   | 229   | 347   |
| 35 ± 2          | Lignin             | 56   | 77  | 111   | 176   | 287   |
|                 | Lignin + 60% FLA-4 | 105  | 135 | 178   | 246   | 363   |
|                 | SKT-3              | 36   | 52  | 81    | 162   | 332   |

$\alpha$  – combustion loss,  $\Gamma_{Kr}$  – adsorption coefficient of krypton,  $t$  – temperature.

formaldehyde resin has a pronounced molecular sieve effect, the pore volume of 0.30 cm<sup>3</sup>/cm<sup>3</sup> being available only for molecules with a diameter of 0.44 nm. The obtained activated carbon is not inferior to industrial Russian coals in the porous structure parameters, exceeding them in mechanical abrasion strength by a factor of 1.1–1.7.

One of the applications of activated carbons is air purification from radioactive inert gases formed during fuel fission. Sorption of noble gases can be increased by lowering the temperature of exhaust gases and cooling the sorbent. We have studied the possibility of using activated carbons based on lignin and phenol-lignin-formaldehyde resin for purification from radioactive noble gases in a wide temperature range. Table 6 gives the adsorption coefficients for krypton, which are equal to the volume of dry air completely purified from krypton under static conditions. Activated carbon of the SKT-3 type, which is one of the best Russian carbon adsorbents for this purpose, was used as a reference for comparative assessment of the sorption properties of noble gases.

As shown by our studies, the adsorption of krypton by a sample obtained from a mixed raw material (lignin + 60% FLA-4) is higher than that on SKT-3 coal

over the entire temperature range. It can be assumed that the high sorption capacity for krypton (with a molecular size of 0.39 nm) in a sample from a mixed raw material is high due to the presence of a significant volume of pores with a small size ( $d = 0.44$  nm) determined for methanol (Table 4). The significant sorption capacity for the poorly sorbed noble gas krypton both by SKT-3 active carbon and by the sample obtained from mixed raw materials (lignin + 60% FLA-4) is due to the presence of a sulfur heteroatom in the coals, which greatly increases the dispersion interaction of the adsorbent with the krypton molecules. The presence of residual sulfur in the samples is determined by the technology of their preparation. For coals of the SKT series (potassium sulfite peat), sulfur is introduced at the stage of coal formation, while for the sample obtained in this work, sulfur is contained in the starting lignin formed during the percolation of sulfuric acid through a layer of crushed wood. Washing of the samples does not completely remove sulfur from the adsorbents; as a result, the sulfur heteroatom present in coal enhances the sorption properties of the adsorbents. The peculiarity of the introduction of sulfur in the structure of the material and its state in the adsorbent largely determine the sorption characteristics of activated carbon. According

to Table 6, at 20°C the difference in the sorption capacity of coals is significant, probably due to the different states of sulfur in coal as a consequence of different methods of its introduction in the adsorbent. When the temperature decreases to –60°C, this selectivity is lost because of the critical effect of dispersion interaction due to the size factor under the given conditions.

As shown by our studies, the adsorption capacity of the obtained activated carbons for krypton exceeds the sorption capacity of SKT-3 coal over the entire temperature range.

#### CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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SPELL: 1. lignohumic