

SORPTION PROPERTIES OF BROWN COAL
PROCESSING PRODUCTS

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<https://doi.org/10.23939/chcht18.04.493>

Abstract. The paper is devoted to the study of sorption properties of residual coal formed after hydrocavitation treatment of brown coal of the Olexandria deposit. The study aims to investigate the possibility of more efficient usage of brown coal processing products that have undergone cavitation treatment. Hydrocavitation of earthy brown coal provides complete and fast extraction of humus acids during extraction with the sodium hydroxide solution. At the same time, the residual coal is pulverized to the size of 10-20 µm. It was shown that the residual carbon has a high sorption capacity (the degree of extraction of methylene blue from the solution is more than 95%).

Keywords: brown coal, cavitation, sorption, residual coal.

1. Introduction

Brown coal, also known as lignite, is the most abundant solid combustible mineral on the planet and is available in large quantities in Ukraine.¹⁻⁴ Due to its declining use as an energy fuel, there has been a recent increase in research to find ways to utilize it for non-thermal purposes.^{1,4-11}

Chemically, brown coal differs from more metamorphosed coals by the presence of a large amount of oxygen and the acidic properties of its main component, humic acids. The main structural element of brown coal is the aromatic component, on the periphery of which there are numerous side radicals associated with oxygen-containing functional groups: carboxylic, phenolic, hydroxylic, etc.

Sorption properties of brown coal, especially oxidized brown coal, are determined by the high content

of oxygen constituents, which promotes the binding of heavy metal ions into inactive and difficult-to-dissociate forms.^{12,13}

The salts of humic acids - humates - in aqueous solutions are most active against heavy metals. The binding of heavy metal ions occurs due to complexing, ion exchange, and physical sorption.^{14,15} However, brown coal itself has certain sorption properties in relation to, for example, water-soluble dyes.¹⁶ Dyes are on the list of pollutants of natural water bodies. Currently, more than 100 thousand types of dyes are known, about 10-15% of which are not bindable by conventional trapping agents and can get into natural water sources. It should be noted that dyes can color surface waters, negatively affect microorganisms and aquatic fauna, and are toxic to human health even in small quantities.^{16,17}

Numerous natural products and some wastes from industrial production and farms serve as cost-effective and efficient sorbents for the deep treatment of wastewater from dyes. These are such products as ashes and slags, chitosan, chitin, peat, shells of various nuts, and many others.^{16,18}

Taking into account the porosity and surface properties of brown coal, it is possible to recommend this product for solving the problem of wastewater treatment. In the study¹⁶ it is shown that the sorption properties of the studied brown coal in relation to acid dyes allow its use in wastewater treatment on a par with the above-mentioned products.

Currently, the problem of treatment of wastewater from various pharmaceutical industries is acute.^{19,20} Vrchovecká *et al.*²¹ present the results of comparative tests on the treatment of wastewater from some types of pharmaceuticals and initial components for their production. Polyamide nanofibers (PA-nanofibers), biochar, and brown coal were used in the studies. The latter showed the best sorption properties. The degree of water purification from various pollutants ranged from 25.58 to 98.55% (depending on the test conditions).²¹

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Brown coal is effective at purification of soils from heavy metals, which is very important in modern times.²²⁻²⁴ To test the possibility of using brown coal for remediation of soils contaminated with lead and copper, extensive tests were carried out in Poland, at the industrial site of a copper-smelting plant. Brown coal was used in a mixture with lime. The resulting mixture proved to be effective in binding both metals. It should be noted that the binding of lead was much more effective than that of copper. The effective impact of brown coal significantly depends on the uniformity of preparation of the solid phase (mixture with limestone) and the moisture content of the recultivated soil.²²

Thus, based on the analysis of some literature sources, it should be noted that brown coal has certain sorption properties, mainly due to the presence of humic acids in it.

According to the Ukrainian national classification (DSTU 34-72:2015) brown coal coals from Dnipro brown coal basin (Dniprobass) belong to group B1, which is characterized by increased water content, high oxygen content, and, as a consequence, high content of humic acids.

Humic acids extracted from coal by alkaline solutions are the most valuable component and are applied in various fields. Their properties, including sorption properties, are well studied.²⁵ At the same time, the sorption properties of residual coal, which has not undergone extraction and also has oxygen-containing functional groups in its composition, have been studied much less. This coal is used only as an energy fuel.

The use of methylene blue as a sorbate is widely used to study the sorption properties of various sorbents such as activated carbon,²⁶⁻²⁸ cellulose,²⁹ biomass,^{30,31} and other sorbents.^{32,33} The use of methylene blue is also the basis for assessing the quality of activated carbon according to the standard method. In addition, methylene blue is a type of cationic dye that can harm human health.²⁹

The aim of the work is to test the sorption properties of residual coal after the removal of humus acids from brown coal of the Olexandria deposit.

Main objectives: determination of changes in technical properties, elemental composition, and particle size distribution of coal as a result of hydrocavitation treatment and extraction of humus acids.

2. Experimental

2.1. Materials

The sorption properties of residual coal obtained by hydrocavitation treatment of Olexandria brown coal (fraction – 3-0 mm), which was carried out to ensure the completeness of humus acids extraction, were studied.

Images of the initial and residual (cavitator-treated) coal taken with a BRESSER Science MTL-201 metallographic microscope at a twenty-fold magnification are represented in Fig. 1. The particle size of the residual coal after grinding is 10-20 μm .

Properties of the initial brown coal and the residual coal obtained as a result of processing are given in Tables 1 and 2.

The elemental composition of initial and residual coal, especially for sulfur and oxygen, practically remained unchanged. This is due to the special mode of cavitation, at which there are no mechanochemical transformations in the coal organic mass. The brown coal fraction of 3-0 mm was subjected to cavitation treatment. In this case, the size of coal particles decreased to the value of about 10 μm . The specific surface area of particles in this case decreases significantly, which will positively affect their sorption properties.

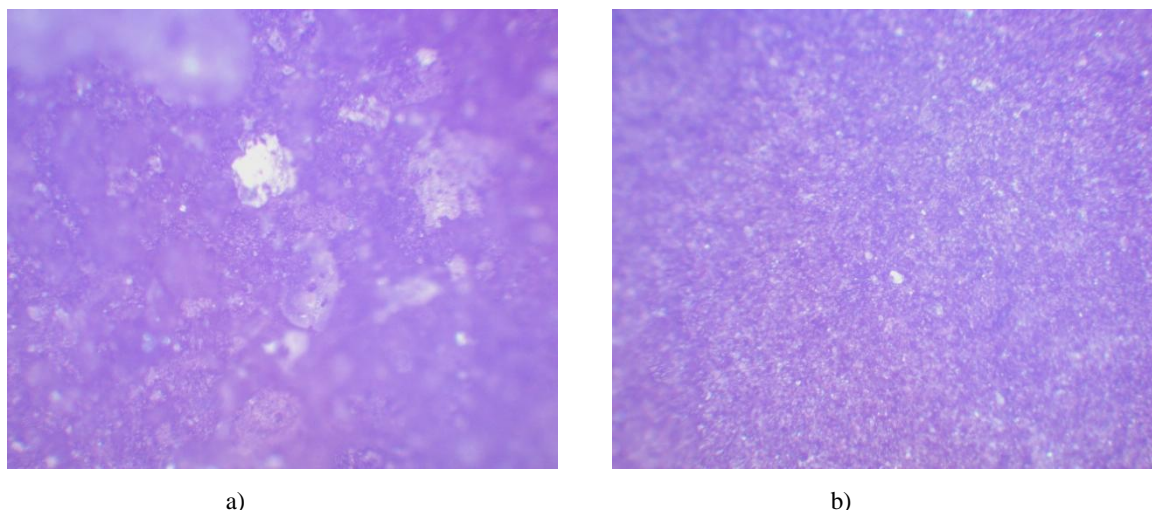


Fig. 1. Images of the initial (a) and residual (b) coal magnified with a metallographic microscope

Table 1. Properties of the initial and residual brown coal

Object	Yield, mass %	A^d , mass %	S^d_t , mass %	V^d , mass %	FC^d , mass %
Brown coal	100.0	43.4	2.54	33.3	23.3
Residual brown coal	22.5	39.6	2.55	42.4	18.0

Table 2. Elemental composition of the investigated products on a dry weight basis

Object	C^d , mass %	H^d , mass %	N^d , mass %	S^d_t , mass %	O^d_d , mass %	Combustion heat $Q^d_{s,}$ MJ/kg
Brown coal	39.42	3.49	0.32	2.54	10.82	16.84
Residual brown coal	43.08	4.24	0.06	2.55	10.43	19.07

The absorption spectrum (Fig. 2) of the brown coal processing product in the infrared (IR) region (400-4000 cm^{-1}) was studied using a Shimadzu IR Affinity-1S Fourier transform infrared spectrometer. Absorption peaks at 3618.46, 3693.68, 1413.82 cm^{-1} most likely indicate the presence of -OH groups; 3425.58 cm^{-1} corresponds to residual water; 2918.3 and 2848.86 cm^{-1} probably indicates the presence of -CH group; 1581.63 cm^{-1} – aromatic group; 1165 cm^{-1} corresponds to -CH₂ in secondary alcohols and 1099.43 – to C-OH in general; 1031.92 – S-O bond; 1008.77, 796.6, 536.21 and 468.7 cm^{-1} – Si-O-H, Si-O-Si, O-Si-O and Si-O-Si, respectively; 912.33 cm^{-1} – C-H bond; 871.82 cm^{-1} – C-H bond with an aromatic ring; 779.24 cm^{-1} – Si-C bond; 694.37 cm^{-1} – C-S bond.

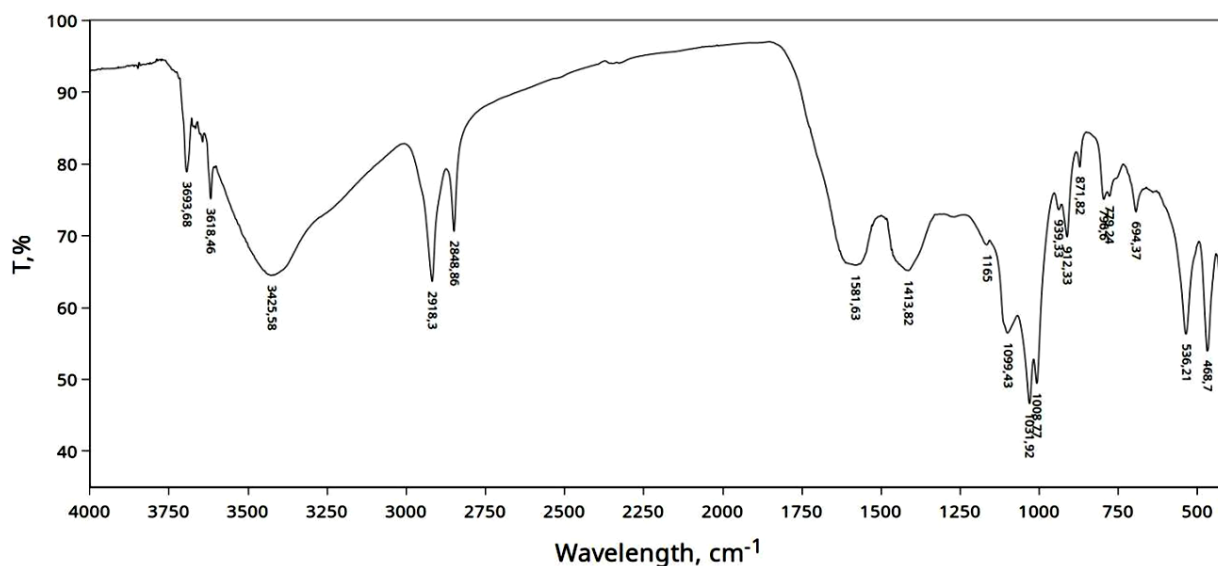
Thus, the brown coal processing product is mainly characterized by the presence of silicon oxide (silicic acid), which can be partially bound to carbon chains. The presence of -CH₂ and -CH groups is observed, indicating the presence of aliphatic compounds. These compounds are likely to be partially oxidized, as indicated by the

presence of C-OH groups. The composition also contains aromatic and polyaromatic hydrocarbons, which is confirmed by the presence of an absorption band of aromatic groups and a C-H bond with an aromatic ring in the spectrum. Probably, the brown coal processing product will exhibit fairly good sorption properties for cationic dyes such as methylene blue due to the presence of -OH groups and aromatic rings.

2.2. Methods

Hydrocavitation treatment was carried out in 1% NaOH solution at the solid-to-liquid ratio (S:L) equal to 1:10, at ambient temperature for 10 min. The scheme of the cavitation unit is described in detail by Kravchenko *et al.*³⁴ Individual parts of this scheme were used in this study. The block diagram of the unit is presented in Fig. 3.

The following equipment and reagents were used in this research: Denver Instrument TP-214 assay balance, KFK-3 photocolimeter, T-23 centrifuge, shaking apparatus, bi-distilled water, and methylene blue (analytically pure).

**Fig. 2.** Absorption spectrum of the brown coal processing product in the infrared region (T as transmittance, %)

Three series of product samples (0.0628–0.0715 g; 0.0280–0.0374 g; 0.0142–0.0173 g) were taken in borosilicate glass test tubes. Five cm³ of methylene blue aqueous solutions in concentrations from 99.9 mg/dm³ to 999 mg/dm³ were added. The tubes were inverted several times and shaken on a shaking apparatus for 60 min at a speed of 2 oscillations per second. The test tubes were then centrifuged for 20 min at 3000 rpm, followed by 3 mL sampling from the center of the tube, as the sorbent residue was distributed both at the top and bottom of the

tube and centrifuged for 30 min at 4000 rpm. The light absorbance of the prepared samples was measured in a cuvette with an optical path thickness of 1 cm at a wavelength of 625 nm using a pre-built graduated dependence of the methylene blue concentration (C) on the light absorbance (A) (see Fig. 4).

In cases where the light absorbance value exceeded 0.717, the samples were diluted so that the light absorbance of the sample was within the graduated dependence.

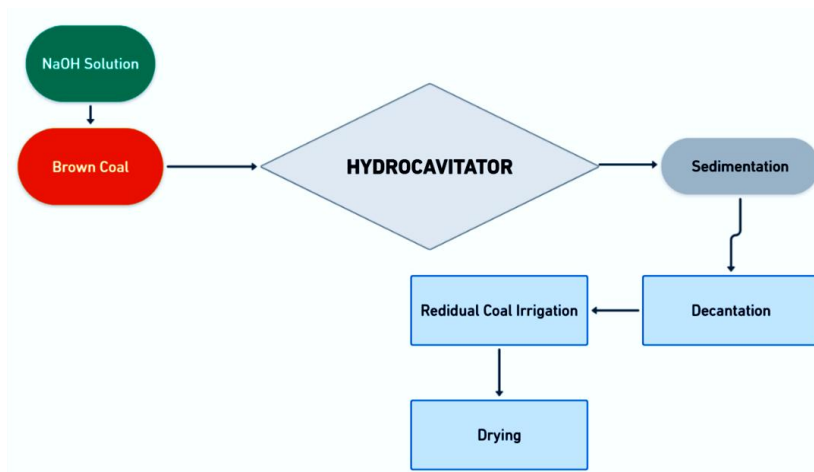


Fig. 3. Block diagram of brown coal hydrocavitation unit

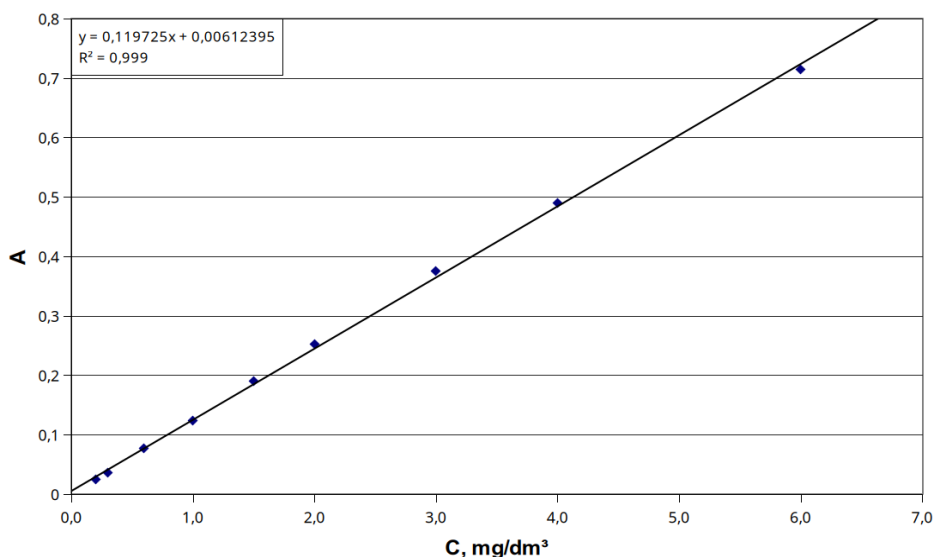


Fig. 4. Graduated dependence for determining the concentration of methylene blue in a solution

The equivalent initial amount of methylene blue per 1 g of product (E_0 , mg/g) was calculated by the following formula:

$$E_0 = \frac{C_0 \cdot V}{m} \quad (1)$$

where C_0 is an initial concentration of methylene blue, mg/dm³; V is a volume of the solution reacted with the product, equal to 0.005 dm³; m is a sample weight, g.

The results were evaluated using three adsorption models, namely the Langmuir isotherm, the Freundlich isotherm, and the Temkin isotherm.

The calculation of the maximum adsorption capacity G_{∞} was performed using the linear form of the Langmuir equation:²⁶

$$\frac{1}{G} = \frac{1}{G_{\infty}} + \frac{1}{G_{\infty} \times K} \times \frac{1}{C}, \quad (2)$$

where C is an equilibrium concentration of methylene blue, mg/dm³; G is the amount of adsorbed methylene blue, mg/g; K is the adsorption constant of methylene blue, dm³/mg.

The Freundlich isotherm was used to estimate the species dependence:

$$\log G = \log K_f + \frac{1}{n} \times \log C \quad (3)$$

where K_f is a constant denoting the adsorption capacity, mg/g.

The value of $1/n$ in Eq. (3) refers to an empirical parameter related to the adsorption intensity. When the value is between 0 and 1, this indicates favorable conditions. If $1/n > 1$, the adsorption process is cooperative; if $1/n = 1$, the process is concentration-independent; and if $1/n < 1$, the process is normal.²⁶

The Temkin isotherm estimation was performed by the formation of the following dependence:²⁶

$$G = \frac{RT}{BT} \ln AT + \frac{RT}{BT} \ln C \quad (4)$$

where BT and AT are constants; R is the universal gas constant; T is the temperature.

The extraction rate of methylene blue from the solution was calculated using Eq. (5):

$$W = \frac{C_0}{c} \quad (5)$$

3. Results and Discussion

Based on the experimental data, the adsorption isotherms of methylene blue were constructed as the

amount of adsorbed methylene blue (G , mg/g) vs. the equivalent amount of methylene blue per 1 g of product. The resulting graph is shown in Fig. 5.

The approximation of the data obtained by the logarithmic dependence is characterized by high values of the determination coefficients (Fig. 5). Deviations from linearity in Fig. 5 are observed at adsorption values above 50 mg/g, with almost the entire amount of sorbate being adsorbed on the sorbent. With an increase in the amount of methylene blue above 50 mg/g of sorbent, a significant range of values is observed, which probably indicates the heterogeneity of the sorption properties of the studied product.

Fig. 6 presents the dependence of the decimal logarithm of the adsorbed methylene blue amount (G , mg/g) on the decimal logarithm of the equilibrium concentration of methylene blue to check whether the adsorption dependence corresponds to the Freundlich isotherm.

The presented linear dependence (Fig. 6) is an approximation to the Freundlich isotherm model, which assumes that the adsorption process takes place on a heterogeneous surface. On the other hand, in the Freundlich adsorption isotherm model, the nature of the adsorption process is multi-layered. The absorptivity K_f was calculated based on Eq. (3) and is equal to 31 mg/g, assuming a favorable environment and normal adsorption process.

Fig. 7 shows the dependence of the adsorbed methylene blue amount (G , mg/g) on the equilibrium concentration of methylene blue, which is approximated by a logarithmic curve corresponding to the Temkin isotherm. Here, we observe a higher correlation coefficient than for the Freundlich isotherm approximation of the data.

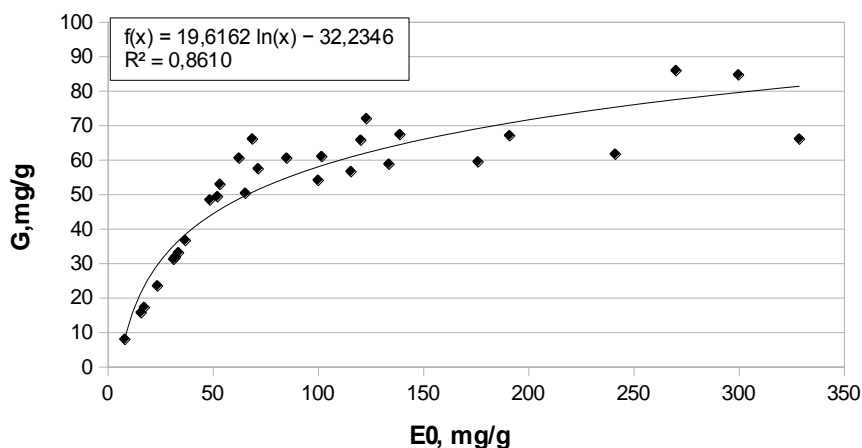


Fig. 5. Dependence of the amount of adsorbed methylene blue (G , mg/g) on the initial equivalent amount of methylene blue

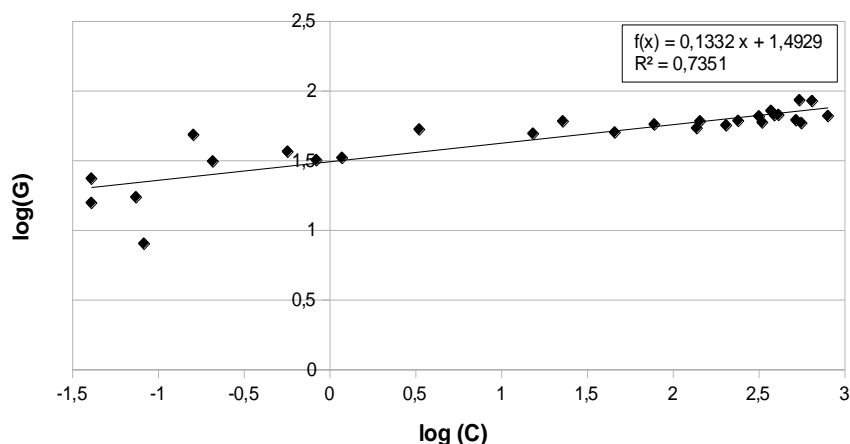


Fig. 6. Dependence of Briggsian logarithm of the adsorbed methylene blue amount (G , mg/g) on Briggsian logarithm of the methylene blue equilibrium concentration

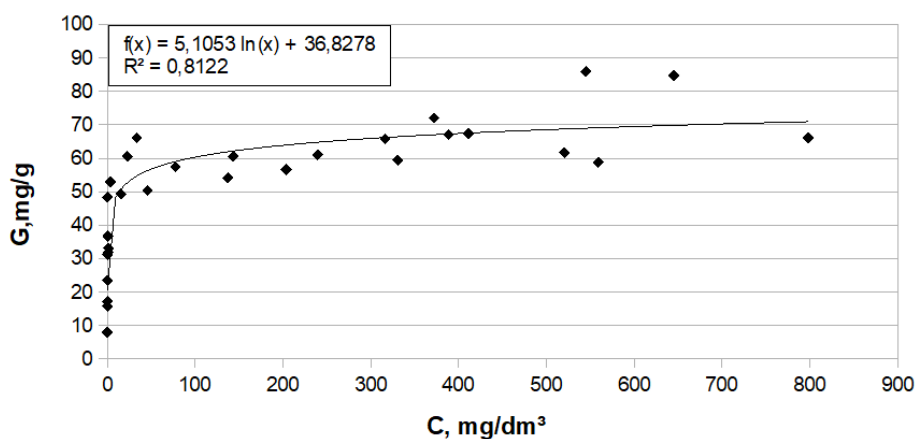


Fig. 7. Dependence of the adsorbed methylene blue amount (G , mg/g) on the equilibrium concentration of methylene blue

The dependences used to calculate the adsorption maximum values according to the Langmuir isotherm are shown in Fig. 8. Langmuir isotherm (Eq. 2) poorly describes the experimental data, high correlation coefficients are observed only due to the significant contribution of some points.

According to the results of approximation by linearization in Fig. 8a, an average correlation between values is observed that is significantly lower than the results obtained using the Freundlich and Temkin isotherms and does not describe the data sample well, which may be due to both the deviation from the Langmuir sorption isotherm and the large variation in the data due to sample inhomogeneity. The calculated value of $G_{\infty} = 52$ mg/g, $K = 8.5$ dm³/mg. Fig. 8b demonstrates a lower correlation between the values of $G_{\infty} = 40$ mg/g, $K = 14$ dm³/mg than in Fig. 8a, and given that the majority of the points mostly lie below the trend line, this likely indicates a deviation of the sorption processes that took

place during the experiment from those described by the Langmuir isotherm. Fig. 8c shows a high correlation between the values of $G_{\infty} = 57$ mg/g and $K = 5.7$ dm³/mg. Fig. 8d shows a high correlation between the values of $G_{\infty} = 64$ mg/g and $K = 0.92$ dm³/mg. Thus, with a decrease in the sorbent charge, an increase in G_{∞} and a decrease in K are observed, which indicates a more efficient interaction of a smaller amount of sorbent and is probably due to its hydrophobicity.

The obtained adsorption maximum values are lower than some of the results obtained, which may be due to both the heterogeneity of the sorbent and the deviation of the adsorption processes from the model used in the Langmuir equation. It should be noted that the result of $G_{\infty} = 52$ mg/g corresponds to the limit of a range when the degree of methylene blue removal from solution is predominantly at a level higher than 95% (Fig. 7).

The comparison of the product with different sorbents is presented in Table 3.

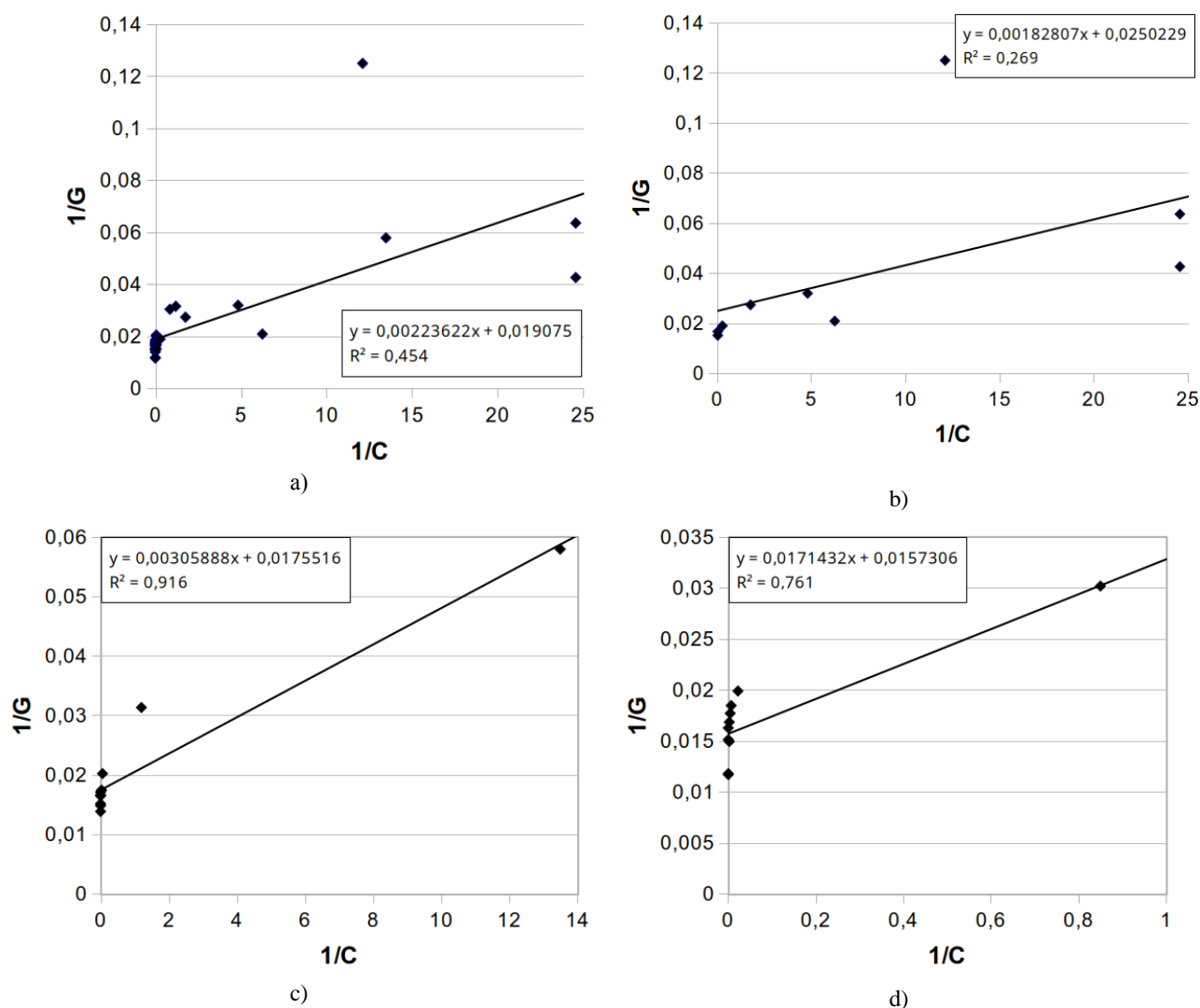


Fig. 8. Dependence of $1/G$ on $1/C$ (for calculations according to Eq. 2) for all results (a); for weights from 0.0628 to 0.0715 g (b); from 0.0280 to 0.0374 g (c); from 0.0142 to 0.0173 g (d)

Table 3. Comparison of sorption properties of different materials

Material	MAX value obtained during product analysis	Activated carbon	Modified activated carbon	Cellulose	Biomass	Various sorbents
Adsorption amount, mg/g	86	322 ²⁷ 153.8 ²⁸ 13 (CNS pyrolyzed carbon) ²⁹	235.5 ²⁸	55 (unmodified) >300 (unmodified) ³⁰	203.33 ³²	891(starbons®) ³² 284.9 (poly(2-acrylamido-2-methyl-1-propanesulfonic acid-co-2-hydroxyethyl methacrylate) hydrogel) ³³

4. Conclusions

The study of the absorption spectrum of the brown coal processing product in the infrared region indicates the presence of aliphatic compounds in the product, which are

likely to be partially oxidized. It also contains aromatic or polyaromatic hydrocarbons. It is worth noting the presence of silicon oxide (silicic acid).

The research of methylene blue adsorption on the coal processing product demonstrated that the product

provides effective sorption (the degree of methylene blue removal from solution is more than 95%) in the range of up to 52 mg of methylene blue in solution per 1 g of sorbent. At higher concentrations, in some cases, the sorption reached 86 mg/g, but the residual concentration of methylene blue in solution was 544 mg/dm³ (68% removal of methylene blue from solution is observed).

A comparison of different adsorption models showed that the highest correlation coefficients when using all the results were observed for the approximation by the Temkin isotherm ($R^2 = 0.812$) and the Freundlich isotherm (0.735).

Comparison of the adsorption properties of the product with different sorbents shows that the adsorption of methylene blue is 3-10 times lower than for specialized sorbents. The main disadvantage of the sorbent is its inhomogeneity (the presence of fractions of different densities) and hydrophobicity, which was observed during the study, both visually and when comparing data using different sorbent weights. Therefore, for the effective use of the sorbent, it is necessary to provide conditions under which the entire active surface of the sorbent is wetted.

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Received: July 01, 2024 / Revised: September 28, 2024 /

Accepted: October 07, 2024

СОРБЦІЙНІ ВЛАСТИВОСТІ ПРОДУКТІВ ПЕРЕРОБКИ БУРОГО ВУГІЛЛЯ

Анотація. Роботу присвячено дослідженню сорбційних властивостей залишкового вугілля, що утворюється після гідрокавітаційного впливу на буре вугілля Олександрійського родовища. Метою роботи є вивчення можливості ефективного використання продуктів переробки бурого вугілля, що пройшло кавітаційну обробку. Гідрокавітація землистого бурого вугілля забезпечує повне і швидке вилучення гумусових кислот після екстракції розчином їдкою натру. При цьому залишкове вугілля подрібнюється до розмірів 10-20 мкм. Показано, що залишкове вугілля має високу сорбційну здатність (ступінь вилучення метиленового синього з розчину – понад 95%).

Ключові слова: буре вугілля, кавітація, сорбція, залишкове вугілля.