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Promising sorbents based on compacted highly porous carbon materials

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Abstract: The paper considers the stages of preparing a compacted highly porous carbon material (HPCM), which involves alkaline high-temperature activation of the initial carbonizate at a temperature of 400–7500 °C for a duration of 2 hours. As a result of activation, a material was produced with a specific surface area of 2600–2700 $m^2 \cdot g^{-1}$ and a pore volume of more than 1.3 $cm^3 \cdot g^{-1}$. The activated material was compacted using binders, which were basalt fiber (HPCM/BF), polyvinyl alcohol (HPCM/PVA) and polyvinyl acetate (HPCM/PVAC). Conditions for compacting were as follows: pressure in the range from 25 to 1600 $kgf \cdot cm^{-2}$, temperature 75–1900 °C and duration between 30 and 150 minutes. As a result, the compacted material had a specific surface area of 1550–2000 $m^2 \cdot g^{-1}$ and a specific pore volume of 0.693–0.849 $cm^3 \cdot g^{-1}$. For the final samples, the sorption capacity for molecules of the organic dye methylene blue (MB) was determined. The kinetic studies showed that the absorptive capacity of h the initial material HPCM was 1691 $m \cdot g^{-1}$, while that of compacted samples of HPCM/PVAC, HPCM/BF and HPCM/PVA was 1611, 1000, and 1270 $mg \cdot g^{-1}$, respectively. In this case, the time for the onset of adsorption equilibrium was 15 min. The presented results show that the compacted carbon material can be a promising sorbent of organic pollutants from aqueous solutions.

Keywords: highly porous carbon material; compaction; specific surface area; adsorption; methylene blue; kinetics.

Перспективные сорбенты на основе компактированного высокопористого углеродного материала

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Аннотация: Рассмотрены этапы получения компактированного высокопористого углеродного материала (ВУМ), предусматривающие проведение щелочной высокотемпературной активации исходного карбонизата при температуре 400…750 °С продолжительностью 2 ч. В результате активации получен материал, обладающий удельной поверхностью 2600…2700 м²/г и объемом пор более 1,3 см³/г. Активированный материал компактирован с применением связующих, в качестве которых использовались базальтовое волокно (ВУМ/БВ), поливиниловый спирт (ВУМ/ПВС) и поливинилацетат (ВУМ/ПВА). Условия проведения компактирования – давление прессования в диапазоне от 25 до 1600 кгс/см², температура 75…190 °С и продолжительность от 30 до 150 мин. В результате компактированный материал обладал удельной поверхностью 1550…2000 м²/г и удельным объемом пор 0,693…0,849 см³/г. Для конечных образцов определена сорбционная емкость по молекулам органического красителя метиленового синего (МС). В результате кинетических исследований выявлена поглотительная способность как исходного материала ВУМ – 1691 мг/г, так и компактированных образцов ВУМ/ПВС – 1611 мг/г, ВУМ/БВ – 1000 мг/г, ВУМ/ПВА – 1270 мг/г. При этом время наступления адсорбционного равновесия составило ≈ 15 мин. Представленные результаты показывают, что компактированный углеродный материал может являться перспективным сорбентом органических поллютантов из водных растворов.

1. Introduction

The development and study of promising highly porous carbon materials with a large specific surface and porosity with a predominance of micro- and mesopores are of interest for many studies, the results of which are in demand in a number of branches of modern industry. These materials seem to be the most promising as universal sorbents for various liquid and gaseous media, devices for storing and transporting fuel, as catalysts, fertilizer carriers, fuel cells, as well as solving environmental problems in such industries as petrochemistry, power engineering, radio electronics, medicine, and agriculture. This is explained primarily by the presence of a balanced combination of a developed system of micro- and mesopores, with a significant specific surface area, corresponding accessible pores and their large volume, with the presence of sufficiently large transport pores that ensure rapid diffusion of sorbed substances, chemical inertness, and stability under real conditions of the use of such materials [1–4].

To produce highly porous carbon materials, various pre-carbonized initial carbon raw materials, including phenol-formaldehyde resin, hydroquinone, carboxymethylcellulose, natural coals, furfural, dextrin, urotropine, carbon nanotubes, graphene, or their combinations are activated with various gas-phase or liquid-phase reagents, such as various acids or alkalis, water vapor, etc. [5–11].

Thus, the authors in [12–16] conducted a study of the impact of activation parameters of the initial carbon raw material and the modes of its subsequent compaction on the sorption characteristics of a highly porous carbon material (HPCM), noting their direct relationship and the possibility of scaling the results of laboratory studies in relation to the possibilities of real production.

The use of graphene in the preparation of a highly porous carbon material with a hierarchical pore structure for potential applications in electronics, catalysis, and sorption is the subject of studies [17], where the authors pointed out the promise of the obtained materials, the presence of a significant surface area, and the possibility of chemical doping and functionalization.

The evaluation of the adsorption properties of highly porous materials with the determination of their structural characteristics was the subject of research in [18, 19], which considered the mechanisms of adsorption and phase behavior of liquids in ordered nanoporous materials with a well-defined pore structure and prove the importance of studying these mechanisms for improving the characteristics of physical adsorption.

The activation process aimed at increasing and improving the volume of micropores, while maintaining a clear network of mesopores was studied in [20]. It was proven that the applied activator – potassium hydroxide – had the biggest impact on the material characteristics, directly contributing to an increase in the volume of micropores. It was shown that alkaline activation is a suitable method for increasing the adsorption capacity of carbon while maintaining a mesoporous structure.

The study of microporous carbon modified with organic substances as fillers to improve thermal and mechanical properties was conducted in [21]. The prepared sorption material showed a significant increase in characteristics when used in thin-film composite direct osmosis membranes.

The prepared highly porous carbon materials can be used in various forms: in the form of powder, granules, films or fibers, both for absorption, separation, purification of various liquid and gaseous substances, and as carriers for catalysts, medicines, etc. However, high requirements are often imposed on them in terms of the possibility of molding into blocks or granules for the convenience of further use in finished products and improvement of its sorption characteristics [22–26].

They are a type of materials with a high specific surface area, large pore volume, and a hierarchical porous structure [27]; they are environmentally friendly, economical, nontoxic, and selective, which makes them good candidates for adsorption of organic and inorganic pollutants from aqueous media, including heavy metals and dyes [28, 29]. They can also be used for drug delivery, catalysis, storage and transportation of gaseous media, ecology, etc. [30].

In modern industry, various dyes are widely used, one of the most common representatives of which is methylene blue (MB), which has the chemical formula $C_{16}H_{18}ClN_3S$, which is a representative of the group of quinoneimine dyes

Table 1. Comparative characteristics of MB dye molecules on various carbon nanomaterials

Adsorbent	Experiment conditions				Adsorption capacity, mg·g^{-1}	Ref.
	pH	Initial concentration, mg·L^{-1}	Sorbent weight, g	Time, min		
Nanocomposite of saponified polymethyl acrylate with grafted dextrin with embedded nanosilica	7.5	–	20 mg	45	515.40	[36]
Activated charcoal from seed husks	–	316	0.055	19.3	436.68	[37]
Nickel alginate aerogel/activated carbon	–	1000	10 mg	48 h	465.12	[38]
Activated charcoal derived from corn stalks	11	10	1.4	50	90 %	[39]
Activated charcoal made from olive pits	10	–	0.05	24 h	714	[40]
Biochar obtained from leaf litter by slow pyrolysis	12	–	–	–	101.27	[41]
Activated charcoal from walnut shell	–	50	10 mg	540	632	[42]
Activatedcarbon/cellulose	6.9	100	–	24 h	103.66	[43]
Activated charcoal from barley husks	8	50	0.2	180	61.60	[44]

containing a phenothiazine ring [31]. MB is a cationic dye that is not considered highly toxic to human health at low doses and short-term contact [32]. However, long-term exposure or high levels of MB in water can cause detrimental effects on human health. The toxic effects of methylene blue include permanent damage to human eyes, nausea, diarrhea, skin irritation, and possibly cancer [33, 34]. In connection with the development of anthropogenic activities, a large number of toxic contaminants, including the MB dye, affect the environment due to improper disposal of wastewater from various enterprises. In this regard, the problem of purification of aqueous media from industrial dyes, including MB, the decomposition process of which is very complicated, becomes especially urgent [35].

Currently, materials that are used as sorbents for these purposes and their characteristics are presented in Table 1.

On the basis of preliminary studies, the authors of the paper developed and investigated a compacted highly porous carbon material to solve this environmental problem.

Thus, the purpose of this paper is to study the sorption characteristics of a compacted highly porous carbon material with respect to the organic dye MB.

2. Materials and Methods

2.1. Reagents and method for preparing a compacted highly porous carbon material

At the first stage of research aimed at obtaining a compacted highly porous carbon material, the issues of chemical activation were worked out, which made it possible to determine rational operating parameters and materials: the initial carbon raw material for carbonizate is a mixture of dextrin (Dekstrinzavod CJSC, Murom, Russia) and graphene oxide (Nanotechcenter Ltd., Tambov, Russia), an activator is potassium hydroxide (RM Engineering, Moscow, Russia), the ratio of components of the reaction mixture of carbonizatewas potassium hydroxide: 1 : 3, activation temperature range 400–750 °C, the duration of the main stage was 2 hours [7, 9].

Basalt fiber (HPCM/BF) (KamennyVek Ltd., Dubna, Russia) in the amount of 1–10%, polyvinyl alcohol (HPCM/PVA) (TK Spektr-Khim, Moscow, Russia) and polyvinyl acetate (HPCM/PVAC) (JSC Pigment, Tambov, Russia) – 10–30 % were used as a binder.

2.2. Characterization

The diagnostics of the characteristics of the activated carbon material before compaction, i.e., its specific surface area and porosity, was carried out on the analytical complex Nova Quantachrome E1200 (Quantachrome, Boynton Beach, USA). As a result, a material was obtained with a specific surface area of 2600–2700 $m^2 \cdot g^{-1}$ and a pore volume of more than 1.3 $cm^3 \cdot g^{-1}$ [7, 9].

2.3. Adsorption studies

To analyze the efficiency of sorption of the synthetic organic MB dye on compacted carbon sorbents, batch experiments were carried out. The sorbent weighing 0.01 g was placed in 30 mL of MB solution with $C_0 = 1500$ $mg \cdot L^{-1}$ according to the Russian standard 4453-74. Each test tube with the solutions to be purified and the sorbent was continuously shaken on a programmable rotator Multi Bio RS-24 (Biosan, Riga, Latvia) for 5, 15, 30, and 60 min. The equilibrium C_e concentration of MB molecules was determined from the optical density measured on a PE-5400V spectrophotometer (Ekros, St. Petersburg, Russia) at a wavelength of $\lambda = 815$ nm.

The static sorption capacity of Q_e sorbents, $mg \cdot g^{-1}$, was calculated by the formula:

$$Q_e = \frac{(C_0 - C_e)V}{m}, \qquad (1)$$

where C_0 and C_e are initial and final concentrations of substances in solution, $mg \cdot L^{-1}$; V is the volume of the solution, L; m is the sorbent sample weight, g.

2.4. Compaction of activated carbon material

For compaction of the activated carbon material, a laboratory setup was developed based on the "IP 100 M – Auto" testing press (Plant of testing instruments and equipment "ZIPO", Armavir, Russia) (Fig. 1).

During this stage, compaction modes were investigated; they included several series of experiments with different pressing pressures, temperatures, and process durations. The amount of the binder was established as a result of preliminary studies by the authors and analysis of literary sources.

For the next stage of research (determination of sorption activity), the samples prepared under the compaction parameters given in Table 2 were used.
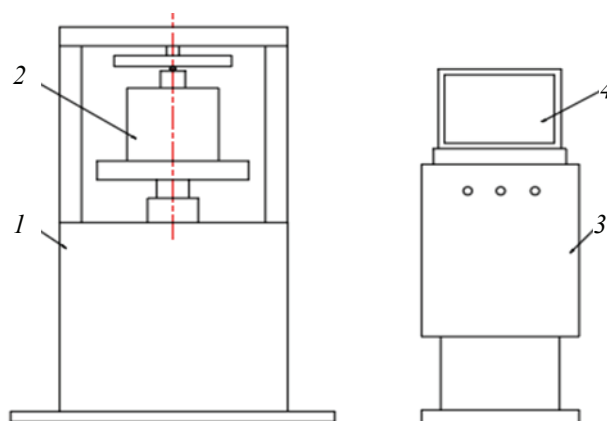

Fig. 1. Laboratory setup for studying the compaction modes of a highly porous carbon material:
1 – press; *2* – heated mold; *3* – press control cabinet;
4 – monitor for setting and displaying the operating modes

Table 2. Compaction mode parameters and characteristics of the prepared samples (blocks)

Parameter	Composites		
	HPCM/PVA	HPCM/BF	HPCM/PVAC
Step wise heating and pressing	75 °C, 3.5 kN, for 15 min	75 °C, 3.5 kN, for 15 min	75 °C, 1.5 kN, for 1 min
	90 °C, 7.5 kN, for 60 min	90 °C, 7.5 kN, for 60 min	90 °C, 3.5 kN, for 1 min
	120 °C, 7.5 kN, for 60 min	120 °C, 7.5 kN, for 60 min	130 °C, 7.5 kN, for 1 min
	190 °C, kN, for 120 min	190 °C, kN, for 120 min	
Mold cooling and pressing-off	Up to 40–50 °C	Up to 40–50 °C	Up to 40–50 °C, placing the sample in an oven for 120 min at a temperature of 80 °C
Block weight, g	16.3–16.8	24.5–25.5	32–33
Height × diameter, mm	(24.5–25,5) × 40	(24–25) × 40	(33–234) × 40

3. Results and Discussion

3.1. Finding specific surface area and porous structure parameters

The resulting compacted carbon materials had the following characteristics: specific surface area 1550–2000 $m^2 \cdot g^{-1}$, specific micropore volume 0.693–0.849 $cm^3 \cdot g^{-1}$.

An analysis of the causes of the difference in the physical and structural characteristics of the resulting materials requires additional research and the accumulation of experimental data, since the mechanism of the binder impact on the carbon material is not fully understood at the moment. At the same time, the authors are inclined to believe that polymeric binders (PVA and PVAC) form a certain spatial structure that provides relatively good permeability and contact surface of the block. In the case of a fiber (BF), it is assumed that a rigid spatial framework is formed in a compacted sample, practically without transport pores.

At the same time, the parameters of compacted materials confirm the correctness and promise of this line of research and can be considered a starting point for further work within this stage, which involves further testing of the pressing parameters and the study of binders, including combinations of polymers with fibers.

3.2. Kinetic studies

As a result of kinetic studies in a static mode, graphs of the dependences of the adsorption capacity of the synthesized materials on the time of contact with a pollutant, the MB dye, were plotted (Fig. 2).

As a result of kinetic studies, it was found that the initial HPCM has an absorption capacity of 1691 $mg \cdot g^{-1}$ when removing MB dye molecules. When a binding agent is added, the compacted carbon sorbent
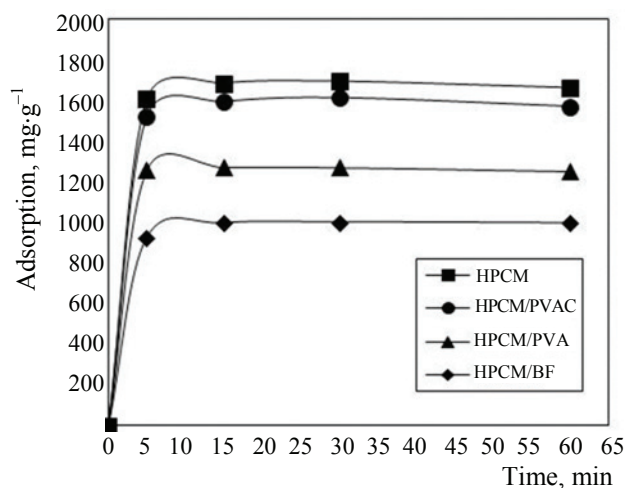
Fig. 2. Adsorption kinetics of MB dye molecules on compacted carbon sorbents HPCM, HPCM/PVA, HPCM/PVAC, HPCM/BF

HPCM/PVA exhibits the highest absorption capacity – 1611 $mg \cdot g^{-1}$. In turn, when using basalt fiber (HPCM/BF) and polyvinyl acetate (HPCM/PVAC), the adsorption capacity is 1000 and 1270 $mg \cdot g^{-1}$, respectively.

To describe the process of sorption of MB molecules (Table 3), namely, the mechanisms involved in the transfer of sorbate to the surface and inside the structure of sorbents, the obtained experimental data were processed by equations of known kinetic models (equations of pseudo-first and pseudo-second order, the Elovich equation and the intraparticle diffusion equation) [45].

The pseudo-second order model has high correlation coefficients $R^2 = 0.9999$ for all compacted carbon sorbents HPCM, HPCM/PVA, HPCM/PVAC, HPCM/BF. Accordingly, we can conclude that the reaction between the adsorbate and the functional groups of the sorbent proceeds strictly stoichiometrically (one molecule occupies one

Table 3. Description of the sorption process of MB dye molecules

Sorbents	Pseudo-first order $\log(Q_e - Q_t) = \log Q_e - \dfrac{k_1}{2.303}t$			Pseudo-second order $\dfrac{t}{Q_t} = \dfrac{1}{k_2 Q_e^2} + \dfrac{1}{Q_e}t$		
	Q_e	k_1	R^2	Q_e	k_2	R^2
HPCM	362	–0.0014	0.1387	1666	0.0120	0.9999
HPCM/PVA	400	–0.0062	0.4072	1666	0.0018	0.9999
HPCM/PVAC	232	0.0009	0.3039	1250	–0.0032	0.9999
HPCM/BF	141	–0.0066	0.3716	1000	0.0050	0.9999

Continued Table 3

Sorbents	Elovich equation $Q_t = \dfrac{1}{\beta} \ln(\alpha\beta) + \dfrac{1}{\beta} \ln t$			Intraparticle diffusion equation $Q_t = k_{id}\, t^{0.5} + C$		
	α	β	R^2	k_{id}	C	R^2
HPCM	$40{,}28 \cdot 10^{36}$	0.0478	0.3859	6.9197	1616.4	0.2519
HPCM/PVA	$22{,}95 \cdot 10^{9}$	0.0136	0.7397	28.866	1516.4	0.564
HPCM/PVAC	$96{,}65 \cdot 10^{13}$	−0.4771	0.0559	−1.6535	1270.0	0.1711
HPCM/BF	$11{,}72 \cdot 10^{14}$	0.0351	0.7113	11.113	925.43	0.5335

Note: Q_e is the number of adsorbed dye molecules on the adsorbent surface at the moment of equilibrium, mg·g^{-1}; Q_t is the number of adsorbed dye molecules on the surface of the adsorbent at time t, mg·g^{-1}; k_1 is the pseudo-first order adsorption rate constant, min^{-1}; k_2 is the pseudo-second order adsorption rate constant, g·(mg·min)$^{-1}$; α is the constant adsorption constant, (min·mg·g^{-1})$^{-1}$; β is the degree of surface coverage and chemisorption activation energy, g·mg^{-1}; k_{id} is the internal diffusion coefficient, (mg·(g·min)$^{-1}$)$^{-1}$; C is the thickness of the boundary layer, mg·g^{-1}.
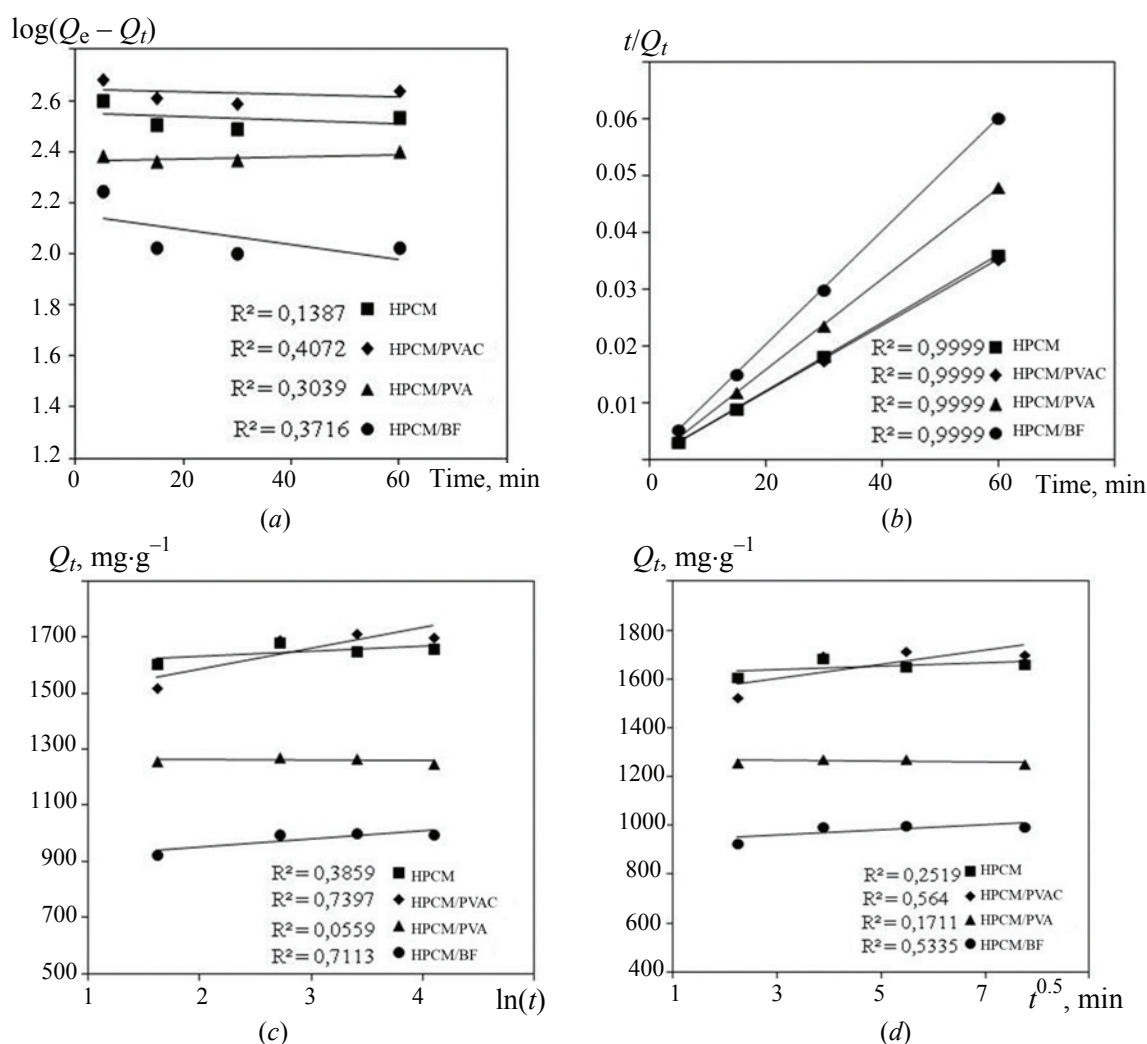

Fig. 3. Results of mathematical processing of experimental kinetic data:
a – the pseudo-first order model; *b* – the pseudo-second order model;
c – the Elovich equation model; *d* – the intraparticle diffusion equation model

position on the sorbent), i.e. a chemical interaction occurs between the dye molecules and the functional groups of the sorbent.

Analyzing the differences in the adsorption characteristics of the studied samples, it can be assumed at this stage of research that when PVA is used as a binder, a certain stable spatial structure is formed that ensures good transport permeability of the adsorbed substances, when PVAC is used, such a structure is formed somewhat worse, with the possible closure of part of the pores directly by the binder. In the case of application as a binder fiber, a significant drop in the sorption capacity can be explained by the formation of a rigid spatial framework in a compacted sample without the formation of transport pores, i.e. only the outer surface of the sample remains active.

4. Conclusion

The conducted studies have shown the relevance of the development of compacted carbon materials for their use as sorption materials. The main stages of obtaining these materials were determined, which included the selection of the initial carbon raw material, the activator and process parameters for high-temperature chemical activation, the establishment of process modes for compacting the prepared activated carbon material (pressure, duration and temperature) – for which a laboratory setup was developed and various binders were selected (for which polyvinyl alcohol, polyvinyl acetate and basalt fiber were used).

The characteristics of the compacted carbon material were found: the specific surface area and pore volume amounted to 1550–2000 $m^2 \cdot g^{-1}$ and 0.693–0.849 $cm^3 \cdot g^{-1}$, respectively. The maximum adsorption capacity for methylene blue for the initial HPCM was 1691 $mg \cdot g^{-1}$, while for materials with binding agents HPCM/PVA, HPCM/BF, and HPCM/PVAC it was 1611 $mg \cdot g^{-1}$, 1000 $mg \cdot g^{-1}$, and 1270 $mg \cdot g^{-1}$, respectively. The time of adsorption equilibrium of the process when using carbon sorbents HPCM, HPCM/PVA, HPCM/PVAC, HPCM/BF was ≈ 15 minutes. Also, experimental kinetic data were described using the known equations of kinetic models (the pseudo-first and pseudo-second order equations, the Elovich equation and the intraparticle diffusion equation). As a result, it was found that the experimental dependences have high correlation coefficients R^2 with the calculated values obtained using the pseudo-second order model. This brings us to the conclusion that chemical sorption of the MB dye has a predominant effect on

compacted carbon sorbents. A direct dependence of the sorption capacity of compacted samples and their strength characteristics on the type and content of the binder used and compaction modes was established, which can be explained by the obtained structure of the material, however, these areas are separate stages of research that are not included in this study.

Thus, it can be concluded that the compacted carbon material prepared with various binders can be an effective adsorbent of pollutants from aqueous solutions, which makes it a promising material for solving environmental problems.

7. Conflict of interests

The authors declare no conflict of interest.

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