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A study of adsorption characteristics of activated carbon material for typical organic and inorganic pollutants

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Abstract: The paper presents the results of adsorption studies on the developed activated carbon material (AM), obtained by two activation methods – with one (AM1) and two activators (AM2), respectively, as well as its compacted versions (AMK) using polyvinyl alcohol (PVA), polyvinyl acetate (PVAC) and basalt fiber (BF) as binders, with regard to typical pollutants of aquatic environments – organic dyes and heavy metals. The carbon materials sorption capacity was assessed by the ability to remove dye molecules – "methylene blue" (MB) and "sunset yellow" (SY) using spectrophotometric analysis, as well as by the ability to remove heavy metal salts – lead (Pb^{2+}) using X-ray fluorescence spectrometry. As a result of adsorption kinetic studies, the absorption capacity of the starting material, activated and compacted materials was determined. The sorption capacity for lead for the materials carbonisate and AMK1 was 71 and 65 $mg \cdot g^{-1}$, respectively, the optimal sorption time was 30 minutes; for the materials AM1, AM2, AMK1/PVA, AMK1/PVAC and AMK1/BF 65, 66, 49, 45, 42 $mg \cdot g^{-1}$ accordingly, the optimal sorption time was 15 min. For MB and SY dyes, the parameters were 1000 – 2010 $mg \cdot g^{-1}$, 66 – 972 $mg \cdot g^{-1}$ and 15 min, respectively. To analyze the adsorption mechanisms using kinetic relationships and sorption isotherms, empirical equations of pseudo-first and pseudo-second order, Elovich equation and intraparticle diffusion model were used. The presented results show the possibility of using the developed activated carbon material as an effective sorbent of organic and inorganic pollutants from aqueous solutions.

Keywords: activated carbon material; compaction; adsorption; methylene blue; yellow sunset; lead; kinetics.

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Исследование адсорбционных характеристик активированного углеродного материала по отношению к типовым органическим и неорганическим загрязнителям

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Аннотация: Представлены результаты исследований адсорбционной способности разработанного активированного углеродного материала (АМ), полученного двумя методами активации – с одним (АМ1) и двумя активаторами (АМ2) соответственно, а также его компактированных вариантов (АМК) с использованием в качестве связующих поливинилового спирта (ПВС), поливинилацетата (ПВА) и базальтового волокна (БВ) по отношению к типовым загрязнителям водных сред – органическим красителям и тяжелым металлам. Сорбционная способность углеродных материалов оценивалась по способности удаления молекул красителей – «метиленового синего» (МС) и «желтого «солнечного заката» (СЗ) с помощью спектрофотометрического анализа, а также ионов тяжелых металлов – свинца (Pb^{2+}) с помощью рентгенофлуоресцентной спектрометрии. В результате проведенных адсорбционных кинетических исследований установлена поглотительная способность исходного, активированных и компактированных материалов. Сорбционная емкость по свинцу для материалов карбонизат и АМК1 составила 71 и 65 мг/г соответственно, оптимальное время сорбции – 30 мин; для материалов АМ1, АМ2, АМК1/ПВС, АМК1/ПВА и АМК1/БВ – 65, 66, 49, 45, 42 мг/г соответственно, оптимальное время сорбции – 15 мин. Для красителей МС и СЗ получены значения емкости: 1000 – 2010 мг/г, 66 – 972 мг/г и 15 мин соответственно. Для анализа механизмов поглощения применялись эмпирические уравнения псевдо-первого и псевдо-второго порядка, Еловича и внутричастичной диффузии. Представленные результаты показывают возможность применения разработанного активированного углеродного материала в качестве эффективного сорбента органических и неорганических поллютантов из водных растворов.

Ключевые слова: активированный углеродный материал; компактирование; адсорбция; метиленовый синий; желтый солнечный закат; свинец; кинетика.

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1. Introduction

Nowadays, many regions of Russia, especially the industrialized ones, are facing the problem of environmental pollution, especially water pollution. The development of industrial complexes, technologies and materials is directly related to the emergence of new chemical production facilities, which, as a rule, produce large volumes of wastewater that require proper and high quality recycling or treatment. Failure to comply with disposal requirements has serious consequences for the environment and the ecological situation in general. The most typical representatives of toxic pollutants of water resources are various organic dyes and heavy metals (e.g. methylene blue (MB) and sunset yellow (SY) dyes or heavy metals – Pb^{2+}). They are widely used in a variety of industries and, when present in wastewater, have a detrimental effect on flora and fauna, including humans. Therefore, water needs to be treated to remove these pollutants and research in this direction is relevant [1–3].

One of the most popular methods of water treatment is sorption, which is technologically proven, economically justified and in many cases the most effective [1, 3–5].

The most widely used modern adsorption materials are activated carbons [6–9], silica gels and zeolites [10–14]. These form the basis of industrial sorption materials, but do not always fully meet the ever-increasing demands on their efficiency [1, 7].

The solution to this problem – the creation of an effective sorbent – is seen in the development and research of promising activated carbon materials (AMs), which combine a high specific surface area and significant porosity with a predominance of micro- and mesopores, the presence of sufficiently large transport pores, ensuring rapid diffusion of sorbent substances, chemical inertness and stability in real application conditions. This is confirmed by a number of publications on this subject [1, 6, 9, 15–16].

To obtain such carbon materials, different pre-carbonized carbon sources, such as furfural, hydroquinone, dextrin, urotropine, natural carbons, carbon nanotubes, graphene or their combinations, are activated by various gas- or liquid-phase reagents: different acids or alkalis, steam, etc. [17–24].

In practice, the resulting activated highly porous carbon materials can be used in the form of powders, granules or fibers, which often places additional high requirements on the possibility of their molding for convenience of further use in finished products and improvement of sorption properties. They should also have a high specific surface area and large pore volume, have a hierarchical porous structure, be environmentally safe, economical and highly selective, which makes them promising and in demand materials for use in adsorption of organic and inorganic pollutants from aqueous media, including various heavy metals and dyes, i.e. these materials should have a specific surface area of 500–3000 $m^2 \cdot g^{-1}$ and a porosity of more than 1 $cm^3 \cdot g^{-1}$ [1, 6, 8, 9, 25, 26].

The influence of the technological parameters of activation of the initial carbon raw material and the modes of its subsequent compaction on the sorption properties is studied in [27–31], where the authors note their relationship, as well as the possibility of using the results of laboratory studies in real production conditions.

The relationship between adsorption and structural properties of highly porous carbon materials is assessed in [15, 32, 33], where the importance of research in this direction is pointed out.

The analysis of the literature sources shows the undoubted interest in the research of the adsorption activity of sorbents on typical pollutants, which in various works was in the following ranges: on MB dye – 61–1190 $mg \cdot g^{-1}$ [34–39], on SY dye – 44–333 $mg \cdot g^{-1}$ [40–43] and on lead – 40–413 $mg \cdot g^{-1}$ [41–46].

Thus, taking into account the need and relevance of this research direction - development and research of effective sorbents – the aim of this work was to study the sorption activity of the developed activated carbon material in relation to organic dyes and heavy metals.

2. Materials and Methods

2.1. Reagents and techniques for the preparation of activated and compacted carbon materials

Based on preliminary studies, the authors of the paper developed and studied an activated carbon material obtained by two activation methods and its compacted variants with different binders. The work consisted of several stages.

In the first stage, samples of activated carbon material were obtained using one activator – potassium hydroxide (KOH) and two activators – KOH + H_2O. In general, this process was a high temperature chemical activation of the initial carbonisate with the activator(s) at a temperature of 400–7500 °C for two hours in an inert environment [18]. At this stage, AM was obtained with a specific surface area greater than 2700 $m^2 \cdot g^{-1}$ and a pore volume greater than 1.3 $cm^3 \cdot g^{-1}$ [27].

The second stage of work involved obtaining compacted samples using such binders as polyvinyl alcohol (PVA), polyvinyl acetate (PVAC) and basalt fiber (BF), the main technological and process parameters for obtaining which are considered in [29]. Moreover, at this stage, only carbon material activated with one activator (AMK1) was used for compacting. The binder content in different compacted samples was as follows: basalt fibre (LLC ‘Kamenny Vek’, Dubna, Russia) in the amount of 5 % (AMK1/BF), polyvinyl alcohol (TC Spektr-Chem, Moscow, Russia) – 20 % (AMK1/PVA), polyvinyl acetate (JSC ‘Pigment’, Tambov, Russia) – 20 % (AMK1/PVAC).

As a result, a number of samples were prepared for the next (third) stage of the research, including: initial carbonisate, carbon materials activated with one AM1 and two AM2 activators, and carbon materials AMK1/PVA, AMK1/PVAC and AMK1/BF compacted with different binders.

2.2. Adsorption studies

The third stage of the research consisted in determining the adsorption activity of the previously obtained samples on Pb^{2+}, for which batch experiments were carried out in a limited volume. The sorbent weighing 0.01 g was placed in 30 mL of Pb^{2+} solution with $C_0 = 100$ $mg \cdot L^{-1}$ according to Russian Standard 4453-74 at pH = 6. Each tube containing purified solutions and sorbent was shaken continuously for 5, 15, 30 and 60 min on a Multi Bio RS-24 programmable rotator (Biosan, Riga, Latvia). The equilibrium concentration of lead ions was determined by energy dispersive X-ray fluorescence spectrometry (ARLQuant ThermoScientific spectrometer, ThermoScientific, USA).

To study sorption to organic dyes, 30 mL of MB, SY solution with an initial concentration of 1500 $mg \cdot L^{-1}$ at pH = 6 was taken and 0.01 g of sorbent was added. Tubes containing the tested solution and sorbent suspension were placed in a programmable multi-rotator Multi Bio RS-24 (Biosan, Riga, Latvia) and stirred continuously for 5, 15, 30 and 60 min. The optical density of the filtered dye solution was then measured on a PE-5400VI spectrophotometer (Ekroskhim Co., Ltd, St. Petersburg, Russia) at a wavelength of λ = 815 nm for MB and λ = 513 nm for SY.

The static sorption capacity of the sorbents, Q_e, mg/g, was calculated using the formula

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

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

3. Results and Discussion

Figure 1 shows the time dependence of lead ion adsorption on the starting material (carbonisate) and the activated materials AM1 and AM2.

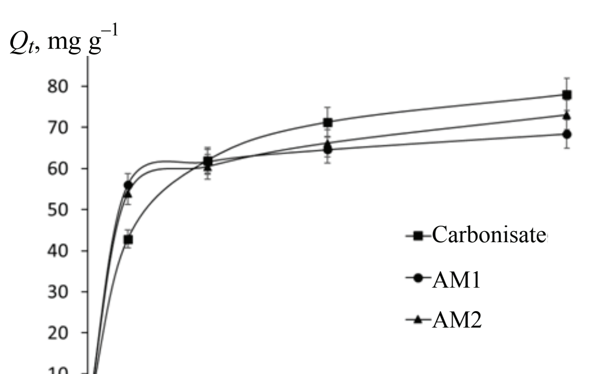


Fig. 1. Kinetic dependence of adsorption of Pb^{+2} ions on carbonisate and the activated materials AM1, AM2

As a result of kinetic studies, carbonisate was found to have an adsorption capacity for the removal of lead ions equal to 71 $mg \cdot g^{-1}$, with the optimal sorption time being 30 min. The adsorption capacity on lead for activated materials AM1 and AM2 is 65 and 66 $mg \cdot g^{-1}$, respectively, the optimal sorption time is 15 min.

In order to describe the ongoing processes of lead ion sorption, namely the mechanisms involved in the transfer of sorbent to the surface and inside the structure of sorbents, the equations of known kinetic models (pseudo-first and pseudo-second order, Elovich equation and intraparticle diffusion model) [47] were applied to the obtained experimental data. Table 1 and Figure 2 show the kinetic data for lead and the results of the mathematical processing of the kinetic data for carbonisate, AM1 and AM2 materials.

As a result of the experimental data processing, it was found that the mechanism of the sorption process in the removal of lead ions is well described by the pseudo-first order equation and the pseudo-second order equation. It can be noted that the pseudo-second order model has the highest determination coefficients R^2 for the removal of lead ions (for AM2 $R^2 = 0.9977$; for AM1 $R^2 = 0.9994$; for carbonisate $R^2 = 0.9998$). Based on this, it can be assumed that diffusion limitation (internal and external diffusion) and 'sorbate-sorbate' interaction contribute to the rate of the sorption process. It should also be noted that the Elovich model for AM1 material has a high coefficient of determination – $R^2 = 0.9983$.

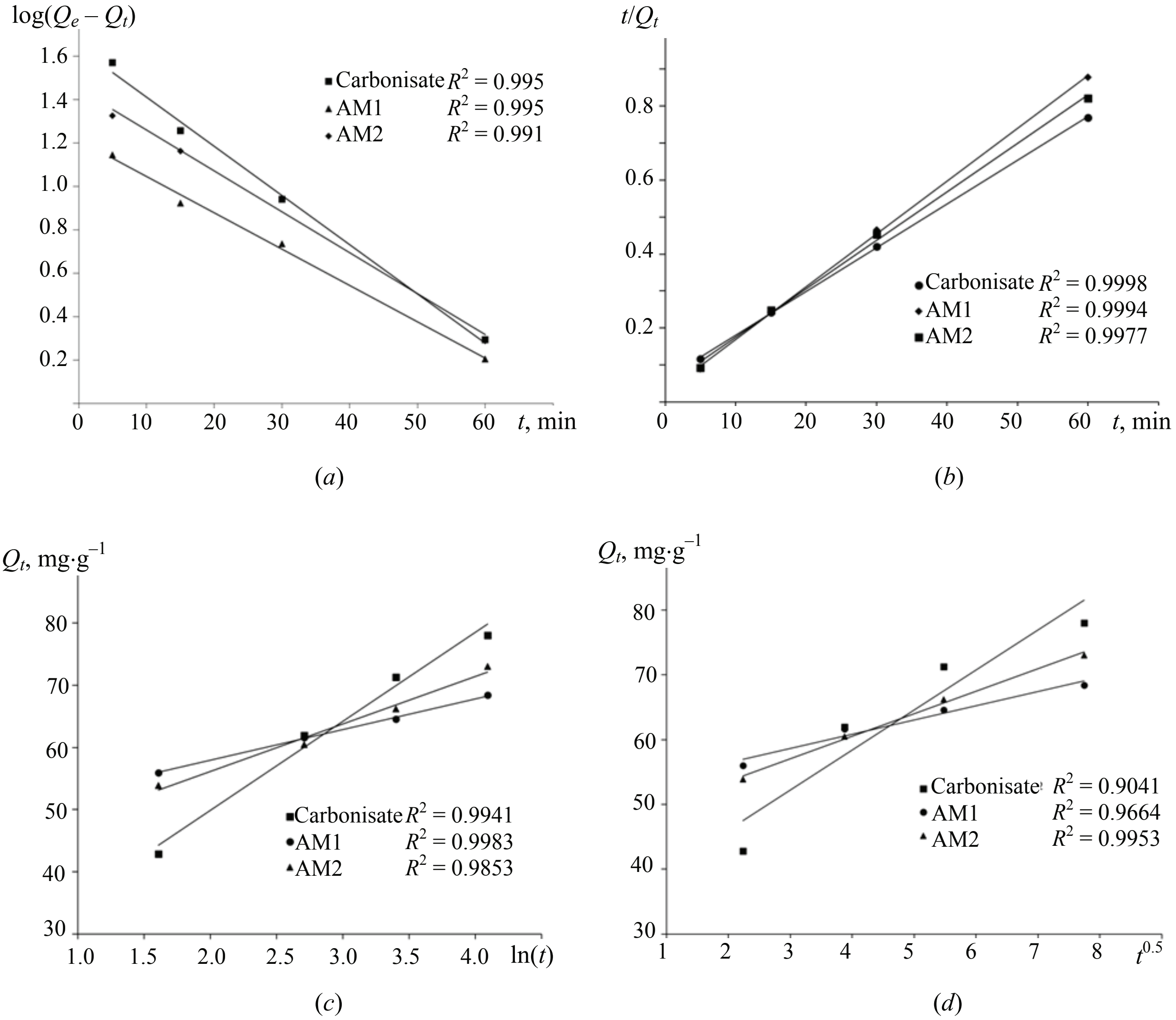
Kinetic studies of the compacted carbon materials – AMK1, AMK1/PVA, AMK1/BF, AMK1/PVAC – have also been carried out.

As a result of kinetic studies of sorption capacity of compacted carbon materials (Fig. 3), it was found that the initial AMK1 has an adsorption capacity for removal of lead ions equal to 65 $mg \cdot g^{-1}$, optimum time of sorption 30 min. With the addition of a binder, the highest sorption capacity is shown by the AMK1/PVA compacted carbon sorbent – 49 $mg \cdot g^{-1}$, optimum sorption time 15 min. When basalt fibre (AMK1/BF) and polyvinyl acetate (AMK1/PVAC) were used, the adsorption capacity was 45 and 42 $mg \cdot g^{-1}$, respectively, optimum sorption time 15 min.

Table 1. Parameters of lead ion sorption kinetics on carbonisate, AM1 and AM2 materials

	Pseudo-first order			Pseudo-second order		
	$\log(Q_e - Q_t) = \log Q_e - \frac{k_1}{2.303} t$			$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{1}{Q_e} t$		
	Q_e	k_1	R^2	Q_e	k_2	R^2
Carbonisate	43.511	0.0523	0.995	84.7458	0.00224	0.9998
AM1	16.319	0.0385	0.995	69.93007	0.0078	0.9994
AM2	28.138	0.0435	0.991	76.3359	0.00399	0.9977
	Elovich equation			Intraparticle diffusion model		
	$Q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t$			$Q_t = k_{id} t^{0.5} + C$		
	α	β	R^2	k_{id}	C	R^2
Carbonisate	62.4998	0.0699	0.9841	6.1843	33.644	0.9041
AM1	80493.4	0.2025	0.9983	2.1906	52.052	0.9664
AM2	1552.34	0.1307	0.9853	3.4665	46.643	0.9953

* Q_e – amount of adsorbed contaminant on the adsorbent surface at the moment of equilibrium, $mg \cdot g^{-1}$; Q_t – amount of adsorbed contaminant on the adsorbent surface at time t, $mg \cdot g^{-1}$; k_1 – pseudo-first order adsorption rate constant, min^{-1}; k_2 – pseudo-second-order adsorption rate constant, $g \cdot (mg \cdot min)^{-1}$; α – adsorption constant, $1 \cdot (min \cdot mg/g)^{-1}$; β – surface coverage and chemisorption activation energy, $g \cdot mg^{-1}$; k_{id} – internal diffusion coefficient, $1 \cdot (mg/g \cdot min)^{-1}$; C – boundary layer thickness, $mg \cdot g^{-1}$.



(*a*) (*b*)

(*c*) (*d*)

Fig. 2. Results of mathematical processing of the experimental kinetic dependencies using pseudo-first order models (*a*); pseudo-second order models (*b*); Elovich equation (*c*); intraparticle diffusion model (*d*)

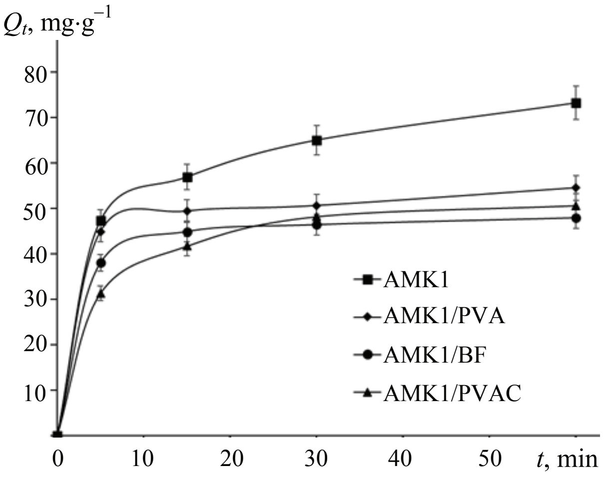


Fig. 3. Kinetic dependences of the adsorption of lead ions on the compacted carbon sorbents AMK1, AMK1/PVA, AMK1/BF, AMK1/PVA

The experimental data obtained for AMK1, AMK1/PVA, AMK1/BF and AMK1/PVAC samples were also processed by the equations of known kinetic models (Table 2, Fig. 4).

The pseudo-second-order model had the highest determination coefficients R^2 for the removal of lead ions (for AMK1 $R^2 = 0.9948$; for AMK1/PVA $R^2 = 0.9988$; for AMK1/BF $R^2 = 1$; for AMK1/PVAC $R^2 = 0.9997$). Accordingly, it can be concluded that the reaction between adsorbate and sorbent functional groups is strictly stoichiometric (one molecule occupies one position on the sorbent), i.e. there is a chemical interaction between Pb^{+2} ions and sorbent functional groups. For the kinetic data for the AM1 material, high coefficients of determination are also observed for the Elovich ($R^2 = 0.9936$) and pseudo-first order ($R^2 = 0.9926$) models.

Table 2. Parameters of lead ion sorption kinetics on the materials AMK1, AMK1/PVA, AMK1/BF, AMK1/PVAC

	Pseudo-first order			Pseudo-second order		
	Q_e	k_1	R^2	Q_e	k_2	R^2
AMK1	38.318	0.0495	0.9926	78.125	0.00259	0.9948
AMK1/PVA	10.416	0.0299	0.9741	55.56	0.00964	0.9988
AMK1/BF	9.667	0.038	0.9169	49.02	0.01378	1
AMK1/PVAC	25.067	0.0504	0.9656	54.054	0.00475	0.9997
	Elovich equation			**Intraparticle diffusion model**		
	α	β	R^2	k_{id}	C	R^2
AMK1	178.749	0.0959	0.9936	4.6843	37.938	0.9866
AMK1/PVA	139636	0.2716	0.9741	1.6489	41.861	0.9605
AMK1/BF	14111.3	0.2520	0.9238	1.6675	36.221	0.8022
AMK1/PVAC	86.5644	0.1245	0.9725	3.4523	26.252	0.8829

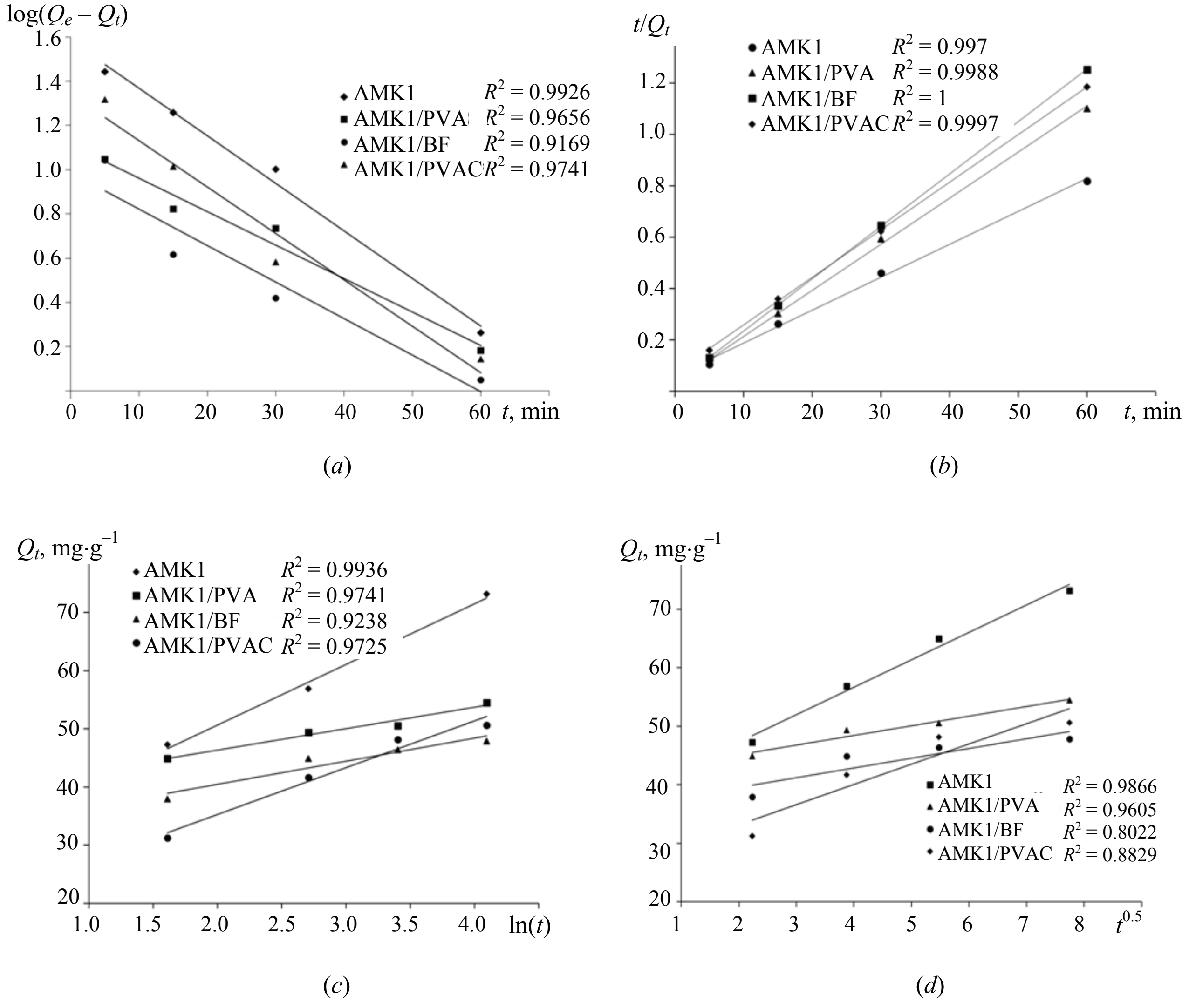
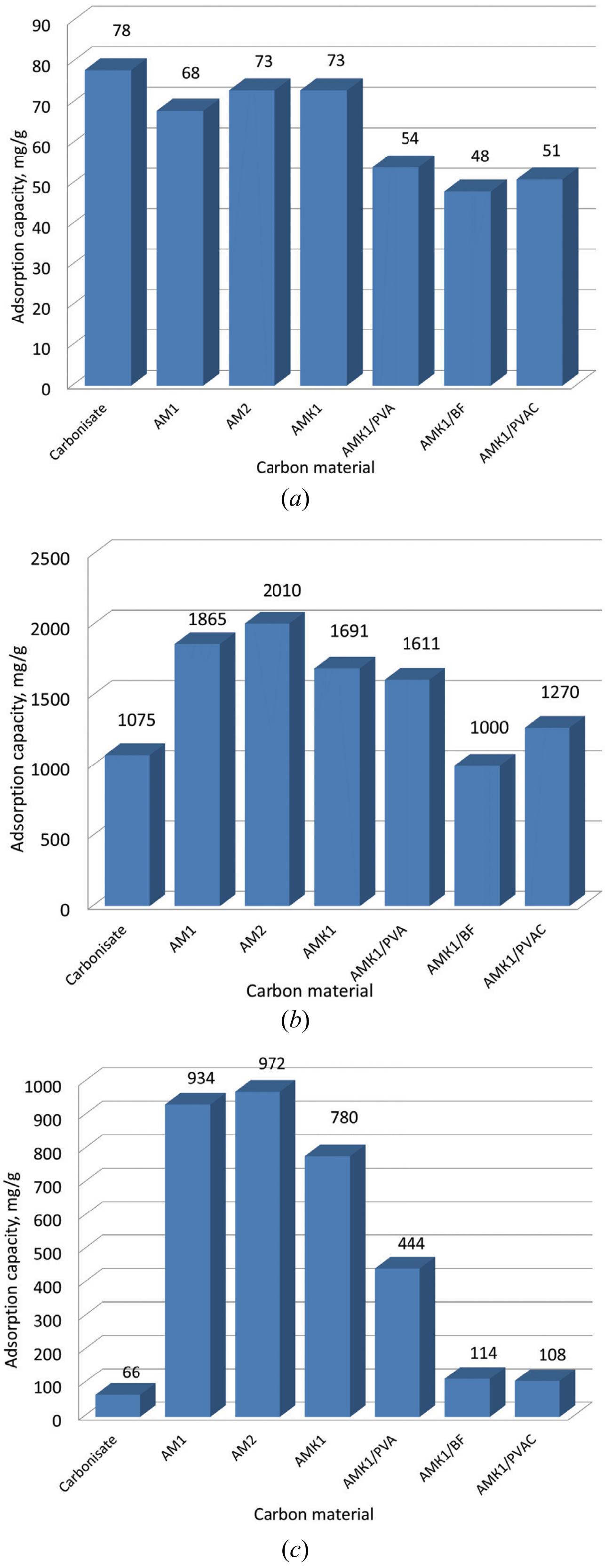


Fig. 4. Results of the mathematical processing of experimental kinetic dependencies using pseudo-first order models (*a*); pseudo-second order (*b*); Elovich equation (*c*); intraparticle diffusion (*d*)



(*a*)

(*b*)

(*c*)

Fig. 5. Comparative results of studies on the adsorption activity of activated carbon materials: *a* – on the sorption of lead ions Pb^{2+}; *b* – MB molecules; *c* – SY molecules

Summarising the studies on the determination of the sorption activity of the developed activated carbon material on typical organic [29, 30] and inorganic pollutants from aqueous solutions (including its compacted variants) (Fig. 5), it can be concluded that the developed material shows comparable or higher adsorption capacities on typical pollutants [34–46].

However, a number of peculiarities can be noted in the course of this study. The activated material obtained shows a rather low sorption activity towards inorganic substances (lead), but in general it is comparable with the results obtained by other authors [42–46]. Moreover, the compacted samples showed slightly worse results, which is explained by the presence of a binder, which is an inert ballast, as well as the formation of a new structure of the material when it is compacted with another.

Analysing the results obtained for dye MB (a typical representative of cationic dyes), it can be seen that the developed activated material, including its compacted variants, shows higher activity in comparison with analogues [34–39] and can be of real practical interest for application in industrial production.

The results for dye SY, an anionic type dye, show a higher efficiency in comparison with the studies of other authors [40, 41], but it can be noted that the sorption activity of the compacted samples also decreases several times with respect to the initial ones.

Thus, the results of the studies on organic dyes MB and SY may indicate a significant influence of the adsorption capacity of the binder used, which can be traced for all the samples studied. At the same time, it should be noted that at this stage of the research, the preferred binder is PVA. Further studies are needed to determine the mechanisms of influence of the technology of obtaining the materials.

4. Conclusion

The paper presents the results of studies of the adsorption activity of the developed carbon material and its compacted variants using different binders. The adsorption capacity of the original, activated and compacted materials was determined. The sorption capacity for lead for carbonisate and AMK1 materials was 71 and 65 $mg \cdot g^{-1}$, respectively, the optimum sorption time was 30 min; for AM1, AM2, AMK1/PVA, AMK1/PVAC and AMK1/BF materials 65, 66, 49, 45, 42 $mg \cdot g^{-1}$, respectively, the optimum sorption time was 15 min. For organic dyes – MB and SY, the capacity was 1000 – 2010 $mg \cdot g^{-1}$, 66 – 972 mg·g^{-1}, respectively, at a sorption time of 15 min. The obtained experimental kinetic data were described using known equations of kinetic models (pseudo-first and pseudo-second order, Elovich equation and intraparticle diffusion model). According to the results of the studies, it is possible to note a high sorption activity of the developed carbon material for the extraction of cationic and anionic dyes, as well as similar activity with respect to Pb^{2+} ions, which is comparable with analogues. This may open up prospects for its use in solving a number of environmental problems.

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6. Conflict of interests

The authors declare no conflict of interests.

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