

THE CHOICE OF SORBENT FOR ADSORPTION EXTRACTION OF CHLOROFORM FROM DRINKING WATER

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Abstract: At present, providing the population with clean drinking water is one of the most important urgent problems of our time. Due to seasonal changes in water composition and violation of water treatment technology, the conventional process is not always effective to ensure water purification of organic compounds. Moreover, more hazardous contaminants may form unlike the previous ones. Humic substances act as the main source of chloroform formation for water decontamination during the water treatment. Adsorption chloroform extraction from water was studied under static conditions using KAU, SKD-515, BAU, AG-3, AG-OV-1 carbon, ABG semi-coke, PFS polymer sorbents and Porolas T and active nonwoven fabric that differ in the production method, structure and specific surface. Main regularities, features and mechanism for adsorption extraction of chloroform from water are identified for test sorbents. The Freundlich and Langmuir equations (theories of monomolecular adsorption), the Dubinin-Radushkevich equation, modified for adsorption from aquatic solution (theory of micro-pore bulk filling) and the BET equation (generalized theory of polymolecular adsorption of Brunauer, Emmett, and Teller) are used to define sorbents and calculate adsorption parameters. To study the potential to increase the sorbent adsorption capacity due variations in the surface structure and chemistry, sorbents were modified by hydrochloric acid solutions and sodium hydroxide. In absence of experimental studies, the technique is developed to define the limiting value of organic compound adsorption not interacting with surface functional groups of carbon sorbents with the developed system of micropores. The sorbent with the best adsorption properties regarding chloroform was recommended.

Keywords: Adsorption, active carbons, polymeric sorbents, chloroform

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INTRODUCTION

For the past decades, the biosphere has been considerably contaminated, including aquatic ecosystems. The health of population depends on the quality of water resources, the rational integrated use thereof and protection [1]. Being the main source for utility and drinking water system, the natural water contains the considerable amount of pollutants of natural and anthropogenic origin, including organic contaminants. In addition, the water treatment process poses the risk of possibility for more dangerous secondary toxic contaminants to generate. The water from natural water bodies is mainly decontaminated by chlorine-containing reagents. In water, chlorine interacts with natural organic substances, including humic substances, producing halogen-organic compounds. The nature of organic contaminants in the

source water, the dose of chlorinating agent, temperature, the length of chlorination and other factors influence the generation of these products and relative content thereof.

For the last decade, people changed their mind as to address the issue of water with the content of halogen-containing compounds in water. Due to the likelihood of adverse effects on health, including metabolic disturbances in case of chronic ingestion intake, the chloroform poses a hazard as the priority and rather popular decontamination by-product available in all water supply systems with chlorinated reagents used. Chloroform accounts for 80–90% of all halocarbons generated in water during chlorination. This compound may be regarded as the indicator of the content of chlorinating products in water [2]. Containing in water in concentrations in excess of the maximum permissible

value, the chloroform has the general toxic and carcinogenic effect on the human body [3]. As classified by the International Agency for Research on Cancer (IARC, Lyon, France) being the part of WHO, chloroform is referred to 2B Group, i.e., to potentially carcinogenic substances (potentially hazardous) to humans. Many authors [4–9] mention the possibility of cancer development in humans, most often pancreatic, bladder, colon and rectal cancer associated with the consumption of chlorinated chloroform-containing water.

The danger of chlorinated water consumption is the ability of chloroform to cause the most severe delayed action. The direct cytotoxic effect of chloroform is identified on liver cells that are responsible for higher permeability of cell membranes [10]. The development of hematopoiesis process disorder is the consequence of changes in oxidative and antioxidant processes proved by the identified dependence of erythrocytes reduction likelihood in blood due to chloroform content.

The data [10] is compiled on the adverse effect of chloroform on children's health to indicate that at the chloroform content of less than 0.3 MPC, the risk assessment for critical organs and systems chronically exposed to chloroform conducted in line with P2.1.10.1920-04 "Guidelines for Risk Assessment for Public Health Exposed to Chemicals Contaminating the Environment" was adequate to define the unacceptable level of risk for blood circulatory system, liver, kidneys, and central nervous system. The more profound examination of children assisted in getting a package of laboratory parameters to identify the development of adverse effects with negative effects of chloroform, supplied with the drinking water.

As per the genetic testing in children [11] who have been long exposed to organochlorine compounds orally and consume the chloroform-containing drinking water, an the unacceptable chronic (non-carcinogenic) pathology risk is formed with the endocrine system. Non-carcinogenic risk is shown as the probability to develop chronic intoxication symptoms over the certain period which is quantitatively associated with the increase in the overall incidence with no any specific forms of disease.

The effect of halocarbons on the female reproductive function was revealed. The consumption of water disinfected with chlorine during pregnancy may result to the birth of children with severe congenital defects, in particular, with heart and brain defects, as considered by the research team headed by Uni Yaakkola from the University of Birmingham (UK). The investigator assessments revealed that the higher content of chlorinated by-products significantly increases the risk of occurrence of three congenital malformations: septum defect between two ventricles (the septum opening results in the mix of arterial and venous blood and insufficient supply of oxygen to blood), "wolf mouth" (cleft in the palate) and anencephaly (partial or even full absence of the cranial vault bones and brain tissue) [12] In addition, a range of compilations indicate on pregnancy disturbance caused by chloroform: delay in fetal development, weight loss of newborns, premature birth [13,14].

Observations in the USA (Iowa State) made it possible to establish that the consumption of chloroform-containing drinking water by the pregnant at concentrations of more than 0.009 mg/dm³ results in the higher risk of the fetal development delay [15]. The maximum permissible concentration of chloroform as per international and national standards for drinking water quality is as follows: in the EEC standards - up to 0.001 mg/dm³, WHO - up to 0.030 mg/dm³, RF - up to 0.060 mg/dm³ (hygienic standards 2.1.5.2280-07), USA - up to 0.100 mg/dm³. In this view, it is necessary to exclude chloroform in drinking water.

One of ways to reduce the chloroform concentration in water is its adsorption by various materials of the developed surface or specific properties. Most often adsorbents are active carbons (AC) and polymeric sorbents with the selectivity of adsorption of organic substances. In this view, this paper aims at the study of adsorption properties of the large group of domestic carbon sorbents with respect to chloroform.

OBJECTS AND METHODS OF STUDY

The research objects were the commercial active carbons based on SKD-515, AG-3, AG-OB-1 made black coal, BAU charcoal, KAU carbon obtained from apricot kernels (produced by the Sorbent OJSC, Perm), ABG semi-coke (produced by Carbonika-F CJSC, Krasnoyarsk), PFS polymer sorbent (PFR), Porolas T polymer sorbent (produced by Sorbent JSC, Perm), activated nonwoven fabric (ANF) that differ in nature, production method, porosity of structure and specific surface area. Brief description of physical and chemical properties of active carbons and polymeric sorbents is presented in Table 1.

The AG-OV-1 carbon is produced from the crozzling coal of coal-bearing base. The main production stages include: grinding, granulation, carbonization at 500–600°C, activation at 900–950°C, sieving and packing. The AG-3 carbon is produced from the Kuznetsk candle coal. Main production stages cover: semi-coking, grinding, granulation, carbonization, steam activation. The SKD-515 carbon is the sorbent on the coal-bearing basis known for the developed porous structure and high internal surface. Main stages of production include two-stage heat treatment, activation and granulation. The coal of KAU grade is made of apricot bone putament by crushing it followed by sieving. A kernel-based sorbent may undergo multiple regeneration as it is known for the large microporous structure and higher strength. The BAU carbon is the wood-based sorbent made from the hardwood. Main stages of production include the following: sieving, activation at 850°C, crushing and grinding. However, this sorbent is recommended for small-sized sorption columns with fixed bed due to low mechanical characteristics (strength, micro- and mesoporous structure). ABG is the brown coal gasified carbon sorbent obtained from the coal-tar raw material (semi-coke). The semi-coke is produced from coal mined in the Kansko-Achinsky Basin section. This unique technology for semi-coke production is low-cost. Polymer phenolic-formaldehyde resin-based sorbent (PFS) [16].

Table 1. Physical and chemical properties of sorbents

Parameter	Sorbent brand							
	KAU	AG-3	AG-OV-1	SKD-515	BAU	ABG semi-coke	PFS	Porolas T
Bulk density, g/dm ³	417	465	531	526	240	490	374	550
Strength, %	90	88	70	75	60	70	86	95
Weight content of total ash, %	5.4	8.0	15.0	31.0	7.0	12.0	1.3	–
pH of water extract	9.25	6.45	6.85	7.65	7.85	7.45	7.85	–
Total pore volume, cm ³ /g	0.97	0.88	0.44	0.7–1.0	0.47	0.5–0.57	1.1	2.52
Pore volume, cm ³ /g								
Micro-	0.31	0.26	0.32	0.28	0.25	0.09	0.34	0
Meso-	0.66	0.09	0.14	0.11	0.10	0.38	0.76	2.52
Macro-	–	0.53	0.44	0.33	–	0.12	–	0
Adsorption activity by iodine, %	78	60	65	56	60	60	226	–
Pellet shape	irregular	cylindrical	irregular	cylindrical	irregular	crushed	globe-shaped	spherical

Table 2. Physical and chemical characteristics of the activated nonwoven fabric

Parameter	Dimension	Value
Width of fabric	mm	400–500
Fabric thickness	mm	3–5
Surface density	g/m ²	50–250
Specific surface area	mg/g	over 1000
Total pore volume	cm ³ /g	over 0.8
Moisture of fabric	%	less than 5
Adsorption capacity for benzene	mg/g	over 280
Adsorption capacity for iodine	%	over 80
Adsorption capacity for phenol	mg/g	over 200

Porolas sorbent, as the highly porous non-ionic polymer, is the polystyrene obtained by granular inert solvent copolymerization of divinylbenzene and ethylstyrene where inert solvents are easily removed from the finished product. The change in the ratio of initial monomers of divinylbenzene and ethylstyrene, the solvent volume and nature (isooctane, benzene, toluene, cyclohexane, isobutanol, etc.) results in the wide range changes in the porous structure of such adsorbents of the similar chemical structure. Highly porous non-ionic polymers are predominantly classified as mesoporous sorbents, with few micropores that may be accessed by organic molecules.

The activated non-woven fabric (ANF) is predominantly produced from viscose production waste. Main production stages include: carbonization in the inert gas (CO₂) and the activation of textile (cellulose, viscose, and synthetic) materials of both woven and non-woven

fabric at 900°C. Carbonization and activation of textile materials commit to micropore formation. The activated non-woven fabric and activated carbon differ in the surface condition by the crystallinity degree (crystallized sections alternate with random sections (amorphous) and degree of carbon surface homogeneity. The activated non-woven fabric surface is more homogeneous as compared with the active coal surface. Sorption carbon fabrics differ with the large structure of the thread porous space forming these fabrics, as well as with high mechanical strength, low air resistance and unique kinetic parameters. The activated non-woven fabric is produced as the fabric with physical and chemical characteristics shown in Table 2.

To clarify peculiarities of chloroform adsorption, the adsorption equilibrium was studied on active coal and polymer sorbent solutions within the range 0.0042 to 25.13 mmol/dm³ in concentration. The chloroform in aqueous solutions was determined as per the common gas chromatography procedure with the electron capture detector [17]. The time to reach the adsorption equilibrium was determined under static conditions by the additional series of tests to be within 24 hours, not longer. The adsorption value (*a*, mmol/g) for test sorbents of the certain weight (*m*, g) was determined by the difference of the initial (*C*₀, mmol/dm³) and equilibrium (*C*_p, mmol/dm³) chloroform concentrations in the aqueous solution of certain volume (*V*, dm³):

$$a = \frac{C_0 - C_p}{m} * V .$$

The test sample adsorption with the exception of polymer sorbents PFS and Porolas T was studied

without any pre-arrangement. In view that PFS and Porolas T have the hydrophobic shell that makes it difficult to study the chloroform adsorption equilibrium from aqueous solutions, it was removed by treating sorbents with ethyl alcohol or acetone for 8 hours followed by washing polymer sorbents with distilled water until the excess solvent was completely removed.

The effect of sorbents treatment with HCl and NaOH solutions on adsorption properties was evaluated using active coals of AG-3, SKD-515, and AG-OV-1 brands. They were placed in acid (alkali) solution for 24 hours followed by washing with distilled water.

Porous properties of active coals (total surface area, microporous area, volumes) were studied by the method of low-temperature adsorption of nitrogen at 77°C using the Sorbtometr M (manufactured by the Boreskov Institute of Catalysis, Siberian Branch of the RAS, Novosibirsk).

RESULTS AND DISCUSSIONS

Adsorption isotherms refer to one of major evaluation criteria for the sorbent adsorption properties that allow determination of the sorbent activity dependence (sorption capacity) on adsorbate concentration under equilibrium conditions. The chloroform adsorption isotherms are plotted from aqueous solutions presented in Fig. 1 based on experimental data. It was established that the adsorption capacity of test adsorbents

decreases in terms of chloroform in the row as follows: ANF>KAU>SKD-515>BAU>AG-3>AG-OV-1>PFS>ABG>Porolas T.

In the region of low chloroform concentrations (initial section), the adsorption isotherm for all the tested sorbents has a linear pattern, and is, consequently, described by the Henry equation. In the high-concentration region, the adsorption isotherm is L-shaped as per the Hils's classification. The obtained results indicate that the most effective sorbent for chloroform extraction from aqueous solutions is the activated nonwoven material due to the large specific adsorption surface and the AU KAU. However, since they are ore expensive as compared with other sorbents, SKD-515, BAU, AG-3 and AG-OV-1 are the most effective activated carbons for chloroform extraction. Despite the lower adsorption capacity, the ABG semi-coke is the local inexpensive stock that does not require regeneration and, as processed, it may be provided to combustion plant as the solid fuel. The chloroform adsorption volume on PFS polymer sorbents and Porolas T is lower than that on activated carbon. One of reasons for lower chloroform adsorption on polysorbates as compared with activated carbon adsorption is the screening by hydrogen atoms of carbon atoms of polysorbate frame polymer chains that prevents the chloroform adsorbed molecules from approaching the polymer carbon atoms at the distance reached when adsorbed on the surface of carbon materials.

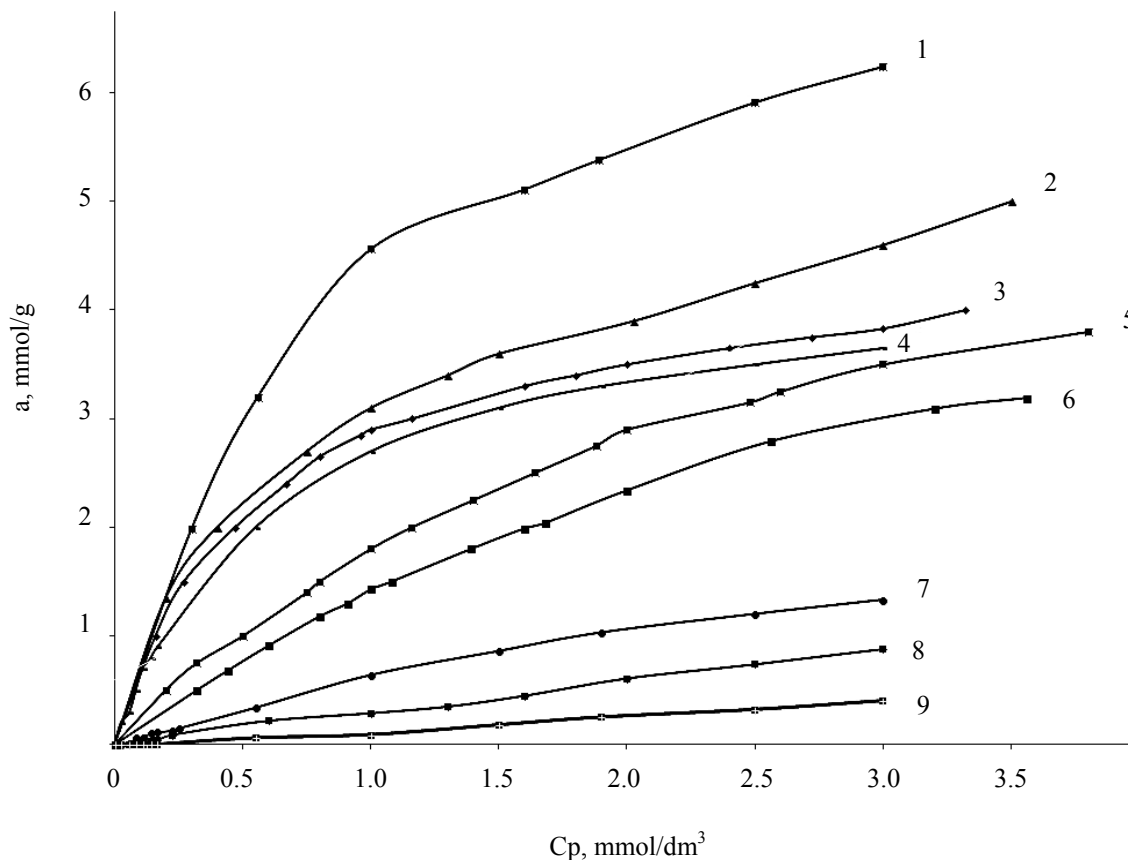


Fig. 1. Isotherms of chloroform adsorption from aqueous solutions on sorbents as follow: (1) ANF, (2) KAU, (3) SKD-515, (4) BAU, (5) AG-3, (6) AG-OV-1, (7) PFS, (8) ABG, (9) Porolas T.

For more detailed characterization of test sorbents and to calculate main adsorption parameters, monomolecular adsorption theories were used (Freundlich and Langmuir equation), the theory of micropore bulk filling (Dubinin-Radushkevich equation modified for aqueous solution adsorption) and the generalized theory of polymolecular adsorption of Brunauer, Emmett and Teller (BET). Theoretical isotherms of chloroform adsorption are calculated by parameters obtained shown in Fig. 2 using the example of AU AG-3. All adsorption isotherms are reviewed in appropriate linearization coordinates of these

equations. The activated carbon chloroform adsorption isotherms in coordinates of equations of Langmuir, Freundlich, Dubinin-Radushkevich, and BET are shown in Fig. 3 using the example of AU SKD-515. A comparative analysis of experimental and theoretical adsorption isotherms indicated that all the equations used well describe adsorption isotherms experimentally obtained on AU. Consequently, all adsorption equations may be used to describe the adsorption process on these sorbents. The calculated values of adsorption parameters for chloroform adsorption are given in Table 3.

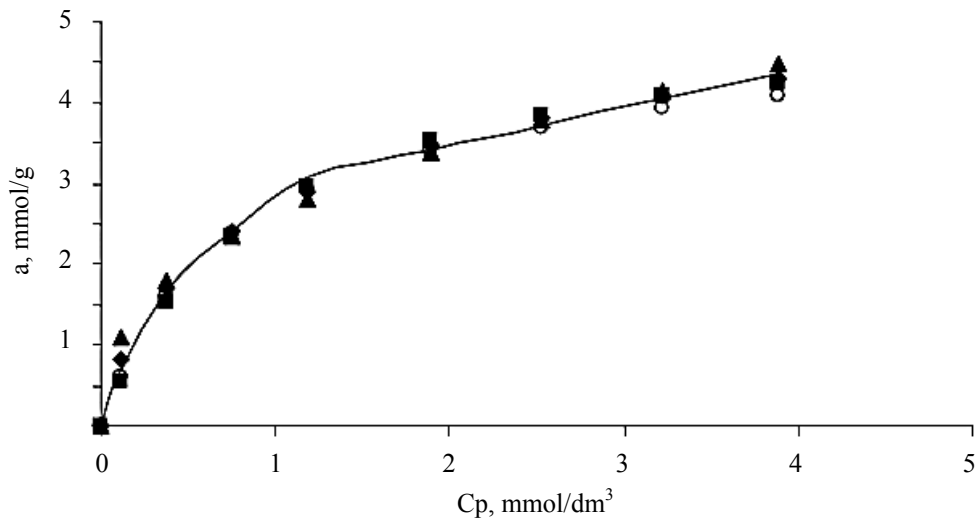


Fig. 2. Experimental (–) and calculated chloroform adsorption isotherms by equations of Langmuir (■), Freundlich (▲), Dubinin-Radushkevich (◆), BET (○) from aqueous solution on AU AG-3.

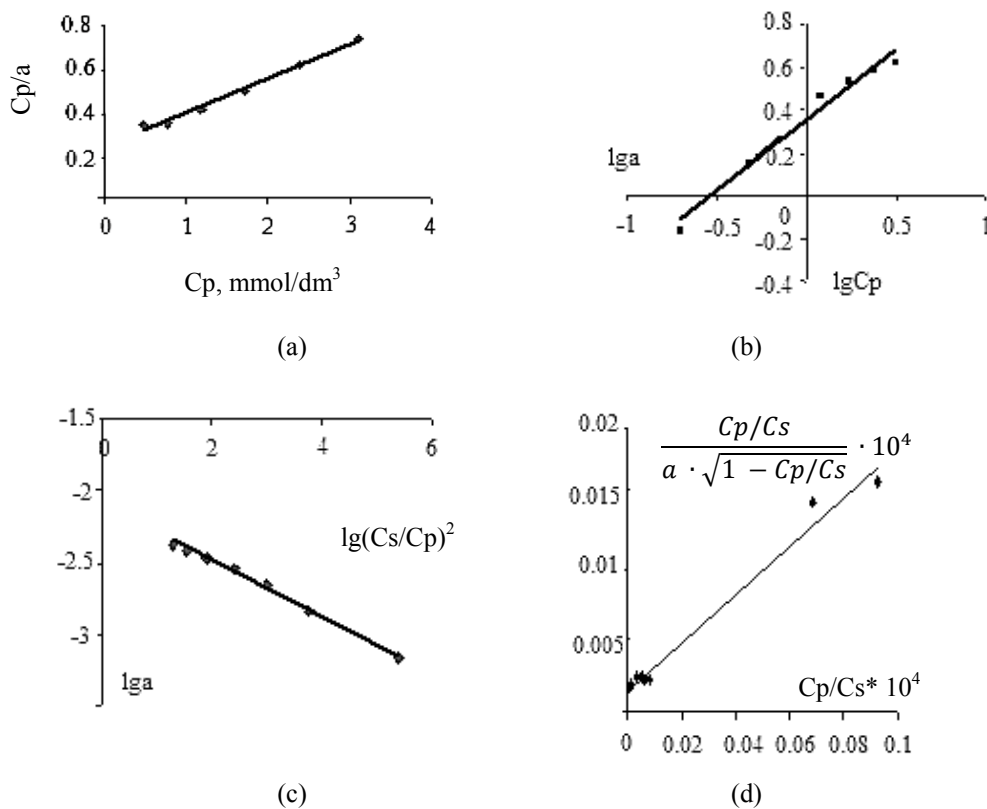


Fig. 3. Isotherms of chloroform adsorption on AU SKD-515 in the linearization coordinates of equations Langmuir (a), Freundlich (b), Dubinin-Radushkevich (in), BET (g).

Table 3. Parameters of chloroform adsorption from aqueous solutions by sorbents under static conditions

Sorbent make	Adsorption parameters calculated by the equation of									
	Langmuir		Freindlich		Dubinin-Radushkevich			BET		
	a_{max} , mmol/g	K	β , mmol/g	1/n	a_{max} , mmol/g	E_0 , kJ/mol	W , dm ³ /kg	a_{max} , mmol/g	K	Q, kJ/mol
AG-3	5.27	1.07	2.63	0.82	6.85	10.49	0.64	4.6	58.75	10.13
AG-OV-1	–	–	2.27	0.81	6.54	10.10	0.1892	–	–	8.23
SKD-515	6.26	0.67	0.52	0.85	7.22	8.50	0.86	5.53	33.49	8.77
KAU	–	–	–	–	7.96	10.40	0.85	–	–	–
PFS	–	–	–	–	4.56	11.70	0.49	–	–	–
ABG	4.99	0.14	0.70	0.35	3.54	7.03	0.38	3.22	9.86	5.91
BAU	–	–	0.62	0.82	5.63	10.00	0.3218	–	–	14.15
Porolas T	–	–	1.6975	0.38	–	–	–	–	–	–
ANF	–	–	0.5537	0.09	5.30	14.12	0.49	–	–	14.55

The values of the limit adsorption volume of tested sorbents (0.49–0.86 cm³/g) indicate that the chloroform adsorption is subject to the volume mechanism of microporous filling. The values of characteristic energy (7.03–14.12 kJ/mol) allow assuming the larger microporous structure of activated carbons.

Isotherm forms and values of adsorption heat allow preliminary conclusion on the mechanism of chemical compound interaction with the carbon sorbent surface. Isotherms of L type show that the interaction between adsorbed molecules of chemical compounds is insignificant, and the activation energy does not depend on the extent of sorbent surface filling. Isotherms of L class and calculated adsorption heats (5.91–14.55 kJ/mol) indicate that the chloroform adsorption from aqueous solutions is of physical pattern and is due to van der Waals forces.

To clarify the mechanism of chloroform adsorption, the potentiometric titration as per Böhm was performed as well as characteristics of the sorbent porous structure (porometry). The porosimetry data allow identification of the non-specific interaction share (dispersion interaction), and titration data define the fraction of specific interaction due to hydrogen bond of the surface oxygen-bearing function groups of AU with the adsorbate, that is, the amount of OBF on the sorbent surface. Main characteristics of the porous structure of tested AU and the potentiometric titration data are shown in Tables 4 and 5.

The porosimetry data indicated (Table 4) that the volume of micropores in AU is pro rata their adsorption capacity in terms of chloroform, the adsorption of which is of physical nature and is predominantly shown in micropores. There is the strong dependence of the adsorption capacity on the number of micropores: KAU>SKD-515>BAU>AG-3>AG-OV-1>ABG. This is what explains the high adsorption capacity of sorbents with a large number of micro-pores.

The chemical condition of the surface may affect the adsorption behavior of sorbents, either. Yet, all carbons possess adequate function groups that interact with active sorbate groups, increasing the adsorption capacity (Table 5). Data comparison of adsorption capacity, structure and surface chemistry confirm that non-specific interaction is the only mechanism for chloroform adsorption. Chloroform with no groups

capable of interacting with function groups on AU surface is adsorbed only in pores with the clear structure-specific dependence of adsorption reported.

Based on experimental data review, the technique to calculate the coefficient b is offered that describes the relationship between the micropore volume (V_{mi} , cm³/g) and the value of the limiting adsorption (a_{max} , mmol/g), which allows without additional studies to determine the adsorption limit of any porous material by with respect to chloroform. The values of the coefficient b are shown in Table 5.

Table 4. Main characteristics of the test sorbent porous structure

sample	S_{micro} , m ² /g	V_S , cm ³ /g	V_{meso} , cm ³ /g	V_{micro} , cm ³ /g
KAU	1419	0,730	0.110	0.620
BAU	586	0,455	0.103	0.352
AG-3	490	0,340	0.060	0.270
SKD-515	404	0,561	0.202	0.359
AG-OV-1	369	0,459	0.241	0.218
ABG	–	0,260	0.240	0.020

Table 5. Surface condition of activated carbons

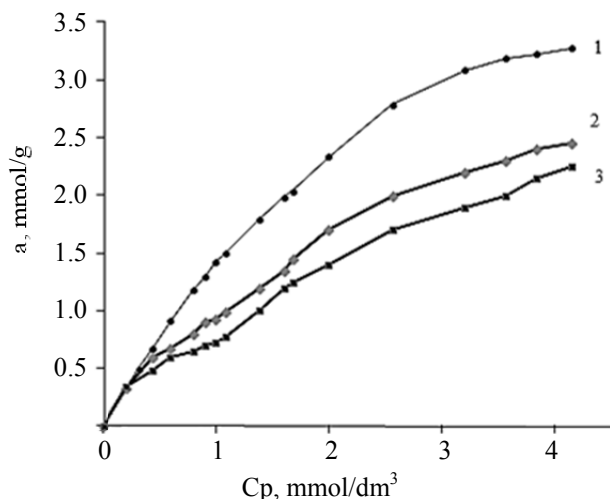
AU	The content of active oxygen in mmol-eq/g of carbon (n_{kfg} , $\mu\text{mol-eq/m}^2$)		
	-OH phenolic	-COOH _{heav} carboxylic	-COO-lactone
SKD-515	0.181	–	0.157
AG-OV-1	0.213	0.032	0.078
AG-3	0.321	0.035	0.039
ABG	0.130	0.020	0.040
KAU	0.194	0.090	0.060

Table 6. Relationship between the volume of micropores and the value of limiting sorbent adsorption

Carbon grade	KAU	AG-3	SKD-515	BAU
a_{∞} , mmol/g	7.96	6.85	7.22	5.63
V_{mi} , cm ³ /g	0.31	0.26	0.28	0.25
$b = a_{\infty} / V_{mi}$	25 ± 7	26.4	25.8	22.5
b_{cp}	25.10 ± 2.04			

Table 7. Henry's Constants for Treated and Technical Sorbents

AU grade	Black AU	AU treated with HCl	AU treated with NaOH
SKD-515	7304	7389	7293
AG-3	6328	6337	6326
AG-OV-1	3813	3798	3798

**Fig. 4.** Isotherms of chloroform adsorption from aqueous solutions on AU AG-OV-1: (1) technical; (2) treated with HCl; (3) treated with NaOH.

The applicability of the proposed method is verified by comparing the value of limiting adsorption calculated by the proposed method and the value obtained during the test on the example of halocarbon adsorption (chloroform, carbon tetrachloride, difluorochloromethane, trifluoromethane) with activated carbon AG-OV-1. The discrepancy between experimental data and calculated values of the limiting adsorption does not the error of chloroform concentration determination in the solution (10–12.5%). This technique to find the limiting adsorption of sorbents in terms of chloroform can be recommended for other class substances not interacting with surface function groups of sorbents with the large system of micropores.

To increase the adsorption capacity of AU, the effect of sorbent pretreatment with hydrochloric acid and sodium hydroxide solutions was investigated. It is found that the isotherm pattern of chloroform adsorption within the initial section with industrial activated carbons treated with acid (alkali) is similar,

that is, in the region of low chloroform concentrations, the amount of chloroform adsorption by carbon sorbents does not almost depend on the method of preparation thereof. The Table 7 presents similar values of Henry's constants rated for treated and commercial carbons that, in turn, indicate on zero influence of the sorbent preparation technique on the chloroform adsorption in the low-concentration region.

The data in Fig. 4 show based on the example of AU AG-OV-1 that regions of high chloroform concentrations report on the decrease in the adsorption value for all sorbents treated with HCl or NaOH solutions. Such effect is probably due to the influence of acid or alkali where a portion of micropores transformed to mesopores with the sorption to be considerably lower than in micropores.

A decrease in the ash content verifies a change in the carbon structure caused by the treatment. The content is determined by gravimetry (on average, the ash content of carbon reduced by 18%). Yet, to note that the partial sorption of reagents (HCl or NaOH) may occur during the treatment resulting in the reduction of the sorption space, and, consequently, to the decrease in the value of ultimate adsorption of carbon to chloroform. Therefore, to purify the drinking water of chloroform, it is practicable to recommend the use of technical sorbents (without pretreatment).

CONCLUSIONS

Thus, the study of adsorption properties of test sorbents under static conditions indicates on their high adsorption capacity in terms of chloroform and allows their arrangement as per extraction efficiency in a series: ANF>KAU>SKD-515>BAU>AG-3>AG-OV-1>PFS>ABG>Porolas T that is related to the different structure of test sorbents. Studies show that the chloroform adsorption predominantly takes place in micropores due to non-specific interaction. The activated carbon treatment with HCl or NaOH solutions results in the decrease in adsorption capacity of sorbents due to changes in their structure by the treating reagent. AU SKD-515 can be recommended for adsorption extraction by the results of studies.

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