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Analysis of simplified equations of adsorption dynamics of HCH

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ABSTRACT

Depending on the system, in which the adsorption process is implemented, equations of kinetics, statics, and dynamics of adsorption can be applied for its mathematical description. The most general model used for description of adsorption process in the batch system is a model based on field equation of adsorbate concentration in the internal structure of the absorbent. Dynamics of adsorption examines the time-spatial distribution of adsorbate in the adsorbent bed. This publication presents an analysis and practical use of selected dynamics equations of the adsorption process for calculating the working time of sorbent deposits in a flow system.

Keywords: Simplified adsorption equations; Adsorption isotherms; Modeling adsorption; Sorption mass transfer

1. Introduction

Adsorption on activated carbon is the most widespread technology used to deal with purification of water contaminated by pesticides [1–5]. These data consist of adsorption isotherms, heats of adsorption, and kinetics, which are required both to evaluate the theories of adsorption equilibrium and to calculate energy balances for adsorption processes. Using these data, a thermodynamic property field (enthalpy, internal energy, and entropy) of adsorbent–adsorbate system can be developed and analyzed for the practical interests of adsorption processes. The porous properties of the assorted adsorbent and the adsorption characteristics of the adsorbent–refrigerant pair influencing directly on the operating behaviors of adsorption processes. Granular activated carbons made from coal or wood have a large surface area and poor adsorption properties. Thus, they are not suitable for industrial applications like environmental protection and medicine. Activated carbon made from coconut shell, which has a high surface area and larger adsorption capacity than other granular activated carbons, is the potential candidate for the process of pesticide adsorption.

Pesticides are toxic chemicals to fight against various diseases and pests. These compounds are carcinogenic, teratogenic, embryotoxic, and mutagenic. Expired or not used, they become very dangerous

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wastes. When improperly stored, they penetrate in uncontrolled way to the natural environment making the threat for all living forms. Over the past years have remained dozens of thousands of tons of pesticide wastes that have been stored since 50s of the twentieth century [6]. There is also a danger of impairing the construction condition along with the occurrence of corroding the concrete bunkers and wells, the outdated pesticides are deposited, and as a consequence, a toxic leakage [4,7,8]. The leakage can be transported by underground water and then in a form of, so-called underground inflow, it is caught by a network of surface waters [6,9,10]. Acceptable concentration of pesticides in drinkable water was tightened following the introduction (March, 2007) of new standards for drinkable water. The amount of a single pesticide compound cannot exceed 0.1 μg/L, while all of the pesticides—0.5 μg/L. It should be mentioned at this point that those strict standards follow the directives of the European Union. There is, consequently, the need to intensify the potable water treatment [1,11]. Additionally, special attention should be given to subsequent stages of the removal of organic compounds residue and pesticide residue.

Therefore, the purpose of this work was to evaluate the adsorption potential of activated carbon for pesticide. The equilibrium of the adsorption was then studied to understand the adsorption mechanism of HCH molecules onto made of coconut shell materials activated carbon.

2. Sorption isotherms

Among the several existing isotherms, the sorption data were subjected to three commonly used isotherms models, namely Langmuir (1918), Freundlich (1894), and BET (1938), to evaluate the maximum saturation capacity of adsorbent [12].

2.1. Freundlich model

The first mathematical fit to an isotherm was published by Freundlich and is a purely empirical formula for microporous and heterogeneous adsorbates:

$$q_e = K_F \cdot c^{\frac{1}{n}} \tag{1}$$

where q_e (mg/g) is the amount of HCH adsorbed on the adsorbent surface at equilibrium, c (mg/L) is the HCH concentration in aqueous solutions at equilibrium, K_F constant is the maximum multilayer adsorption capacity and $1/n$ is a characteristic constant which measures the adsorption intensity (K_F and n are empirical constants for each adsorbent–adsorbate pair at a given temperature). As the temperature increases, the constants k and n change to reflect the empirical observation that the quantity adsorbed rises more slowly and higher pressures are required to saturate the surface.

2.2. Langmuir model

Langmuir isotherm is a semi-empirical isotherm derived from a proposed kinetic mechanism. Langmuir isotherm is a model for monolayer localized physical adsorption on a homogeneous surface; it may be extended with heterogeneity effects, lateral interactions, and multilayer effects. It is based on four assumptions:

1. The surface of the adsorbent is uniform, that is, all the adsorption sites are equivalent,
2. Adsorbed molecules do not interact,
3. All adsorption occurs through the same mechanism,
4. At the maximum adsorption, only a monolayer is formed: molecules of adsorbate do not deposit on other, already adsorbed, molecules of adsorbate, only on the free surface of the adsorbent.

The Langmuir equation may be written as:

$$q_e = q_m \cdot \frac{k \cdot c}{1 + k \cdot c} \tag{2}$$

where q_e (mg/g) is the amount of HCH adsorbed on the adsorbent surface at equilibrium, c (mg/L) is the HCH concentration in aqueous solutions at equilibrium, q_m (mg/g) is the maximum monolayer adsorption capacity, and k is the constant related to the free energy of adsorption.

2.3. BET model

Molecules frequently form multilayer, that is, some are adsorbed on already adsorbed molecules and the Langmuir isotherm is not valid. In 1938, Stephan Brunauer, Paul Emmett, and Edward Teller developed a model isotherm that takes that possibility into account. The Langmuir isotherm is usually better for chemisorption, and the BET isotherm works better for physisorption for non-microporous surfaces. The BET equation may be written as:

$$\Theta = \frac{1}{1-c} \cdot \frac{k \cdot c}{1+(k-1)\cdot c} \tag{3}$$

where Θ (q_e/q_m) is the fractional coverage, c (mg/L) is the HCH concentration in aqueous solutions at equilibrium, and k is the constant related to the free energy of adsorption.

3. Analysis of simplified equations

Depending on the system, in which the adsorption process is implemented, equations of kinetics, statics, and dynamics of the adsorption can be applied for its mathematical description. The most general model used for description of the adsorption process in the batch system is a model based on field equation of adsorbate concentration in the internal structure of the adsorbent. Dynamics of the adsorption examines the time-spatial distribution of adsorbate in the adsorbent bed. Principles of dynamics determine the degree of usage of adsorptional capacity at any section of bed and at any time of the process duration and also concentration of adsorbate in solution in intergranular space, depending on the hydrodynamic conditions of the process. For the analysis of experimental data, when an accurate description of the adsorbate mass transfer processes is not required, it is assumed that only one component of the stream is stopped. At low concentration of adsorbate its loss during adsorption does not change the values of viscosity and density of the stream. The process runs in isothermal system, and the stream flows in one direction at a constant speed [13–16].

There are known approximate equations in integral form, which describe the dynamics of adsorption at both stages of the process: formation of the stationary front, and its parallel movement down the column. For the analysis of simplified equations of adsorption dynamics activated carbon NP-5 produced by Gryfskand was used as an adsorbent.

4. Material and methods

4.1. Sorbat

On a basis of literature data and own studies, chloroorganic pesticides that most often occurred in drinking water near the graveyards at the highest concentrations was selected as a representative sorbat [2,6,8,10,17]. Individual pure active substances HCH were applied. Technical grade HCH of 99.8 ± 0.2% purity obtained from Institute of Industrial Organic Chemistry Analytical Department in Poland was used as an adsorbat. A sample solution of pesticide was prepared by dissolving 1 g of pesticide in 10 mL of acetone and then diluted to 1 L with double distilled deionized water. The concentrations of prepared solutions were applied: 10 mg pesticide per liter.

4.2. Sorbent

The microporous adsorbent NP-5 manufactured by Gryfsk and Co. Hajnówka, Poland, has been used in the present study (Fig. 1). An activated carbon NP-5 made from coconut shell, which has a high surface area and lager adsorption capacity than other granular carbons.

The porous properties such as, the BET surface area, the pore size, the pore volume, the porosity, and the skeletal density are shown in Table 1. The porosity, total pore volume, average pore diameter, and the surface area of the activated carbon (NP-5)

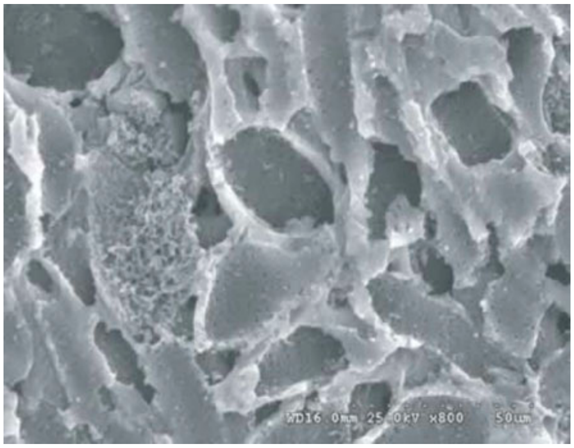

Fig. 1. Scanning electron microscope (SEM) photos of activated carbon type NP-5.

Table 1
Porous properties of NP-5

BET Surface area	m^2/g	1,840
Total surface area	m^2/g	1,360
Micropore volume	cm^3/g	0.81
Mezopore's surface area	m^2/g	54
Total pore volume	cm^3/g	0.90
Granulation	mm	0.75–1.2
Dechloration	cm	5–8
Methylene blue	cm^3	min 40
Iodine number	mg/g	1,390
Hardness	%	95–97
Grindability	%	0.3

samples are measured by the adsorption isotherm of nitrogen at 77 K. Samples are first treated for possible outgassing at a temperature of 473 K for 3 h. The surface area is determined by the BET method from the N_2 adsorption isotherm data [17]. It's granular fraction size is $d = 0.75/1.2$ mm. It combines typical properties for carbon and additional ones, such as: highly developed specific surface area, high adsorption ability, high resistance to abrasion of grains, and its grain size is adapted to work effectively during adsorption of impurities from a liquid. NP-5 activated carbon may be used, *inter alia*, for drinking water treatment and wastewater treatment. The adsorbent bulk density is $\rho_N = 410\ \text{kg/m}^3$, porosity of grain is $\varepsilon_Z = 0.18$ and porosity of layer is $\varepsilon_W = 0.45$.

4.3. Sorption procedure

Studies under static conditions were performed in accordance with methodology applied in Belgium, Germany, France, Italy, England, USA, Poland, and other [1,11,17–24]. They were aimed at plotting the adsorption isotherms due to which it is possible to compare the sorption capacities of different adsorbates on different adsorbents. A selected adsorbent, previously degassed, washed with distilled water and dried, was grounded in spherical mortar, and dried in electric drier at 150℃ for 3 h till constant weight. Such prepared sorbent served for weighing following samples: 0.001, 0.002; 0.005; 0.01; 0.025 g per 100 mL solution. Representative samples of the adsorbent were added into the conical flasks with glass stopper and containing working solution of the HCH (10 mg/L). Flasks were shaken in electric oven at constant oscillation amplitude (9) for 24 h, and then remained for 24 h to reach a complete adsorption equilibrium. After that, the samples were subjected to double filtration on soft filter paper. Then, HCH concentration in a filtrate according to obligatory methods was determined using gas chromatograph AGILENT 6890. Freundlich's, Langmuir's, and BET's isotherms [12,18,19] were plotted on a base of obtained results applying IZO software in order to analyze the processes [13–16].

4.4. Analytical procedure

HCH concentrations were determined in collected samples in accordance with obligatory methodology using gas chromatograph AGILENT 6890 equipped with ECD and NPD detectors. The injector temperature was 210℃ and the flow rate of helium was 1.0 mL/min. The column DB (35 m length, 0.32 mm i.d., and 0.5 μm film thickness) temperature was set at 120℃ for 2 min and increased at a rate of 13℃/min to 190℃. The temperature was finally increased to 295℃ and maintained isothermally for 20 min [25–28].

5. Results and discussion

The results of static adsorption process are shown in Table 2. Basing on the data from Table 2, values of coefficients of adsorption isotherms according to Freundlich, Langmuir, and BET were calculated (Figs. 2–4). The chart of Langmuir isotherm in the presented range of changes of equilibrium concentration, corresponds to BET isotherm chart, so it can be concluded that adsorption in a monolayer took place. The concentration of adsorbate in adsorbent monolayer calculated from Langmuir isotherm is $x_m = 977$ g/kg, and on the basis of BET isotherm $x_m = 667$ g/kg.

Equation, which is frequently encountered in the literature [29–31], which allows one to calculate duration of the adsorption process t_S (s) is the relationship resulting from conversion of equation of mass balance for adsorbent layer of height H (m) and bulk density ρ_N (kg/m^3), at initial concentration of medium C_0 (kg/m^3), assumed the final concentration C_E (kg/m^3) and the flow velocity v_P (m/s):

$$t_S = \frac{x_m.\rho_N.H}{v_P.(C_0 - C_E)}\,(\text{s}) \tag{4}$$

Table 2
Results of investigations on influence of activated carbon NP-5 amount *m* (g) change on change of value of equilibrium concentration *C* (g/L), difference between initial and equilibrium concentrations *x* (g/L), and amount of adsorbed mass *a* (mg/g) in the process of static adsorption

C_0 (mg/L)	C (mg/L)	m (g)	V (L)	x (mg/L)	a (mg/g)
1,000E + 001	8,400E − 003	250E − 002	100E − 001	9,992E + 000	3,997E + 001
1,000E + 001	2,530E − 002	480E − 003	100E − 001	9,975E + 000	2,078E + 002
1,000E + 001	3,960E − 002	980E − 003	100E − 001	9,960E + 000	1,016E + 002
1,000E + 001	2,196E − 001	210E − 003	100E − 001	9,780E + 000	4,657E + 002
1,000E + 001	2,202E − 001	130E − 003	100E − 001	9,780E + 000	7,523E + 002

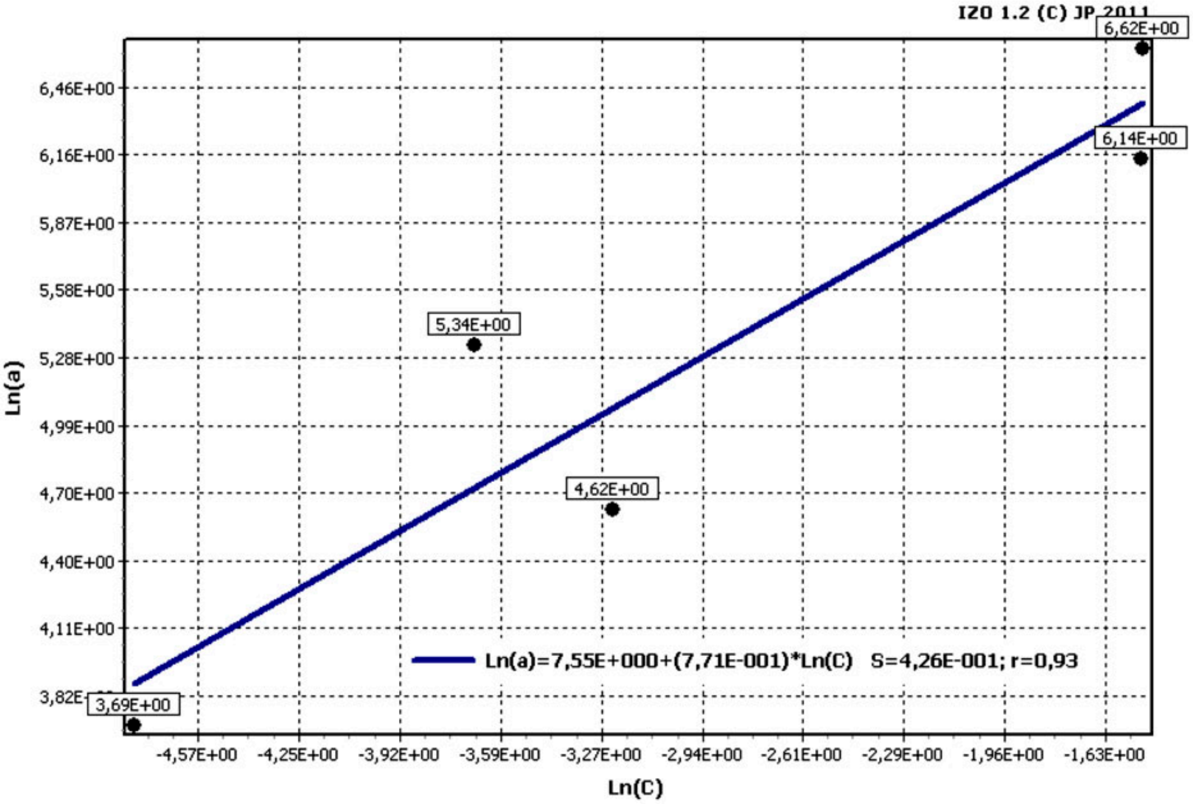

Fig. 2. Freundlich isotherm obtained using the linear method for the sorption of HCH on activated carbon NP-5.

Assuming the adsorbent bed height H = 0.5 m, the initial concentration $C_0 = 0.2$ mg/L, the final concentration $C_E = 0.001$ mg/L, medium flow velocity through the bed $v_p = 3.6$ m/h, and the concentration of adsorbate in adsorbent monolayer $x_m = 997$ g/kg, duration of the adsorption process, calculated using Eq. (4), is about $t_S = 13.02$ years.

Since the duration of the adsorption process t_S (s), calculated on the basis of the transformation of the balance Eq. (4), is greater than the actual sorbent bed working time, the following equations are used in practice, *inter alia*, equation according to Żuchowicki, Zabieziński and Tichonov and equation according to Bohart–Adams [29,31].

5.1. *Żuchowicki, Zabieziński and Tichonov equation*

A formula is derived from the kinetics equation of non stationary external diffusion for the adsorptional system described by Langmuir equation and expressing the duration of column work time at complex parameters of the process:

$$t_S = \frac{x_m \cdot \rho_N}{v_P . C_0} . \left\{ H - \frac{v_P}{k_E} . \left[\frac{1}{w} . \ln\left(\frac{C_0}{C_E} - 1\right) + \ln\left(\frac{C_0}{C_E}\right) - 1 \right] \right\} (\mathrm{s}) \tag{5}$$

On the basis of the value of averaged diffusion coefficient $D = 4.41\mathrm{E}{-10}\ \mathrm{m}^2/\mathrm{s}$, layer porosity $\varepsilon_W = 0.45$ and mean diameter of adsorbent grains $d = 0.98$ mm, the value of external coefficient of mass transfer is $k_E = 0.0543\ \mathrm{s}^{-1}$. Terminal rate of adsorption calculated on the base of Langmuir isotherm for the initial concentration $C_0 = 0.2$ mg/L is $x_0 = 498$ mg/g, hence adsorbate concentration in the stream equivalent to half of terminal rate of adsorption ($x_{0.5} = 249$ mg/g) is $C_{0.5} = 6.58\mathrm{E}{-2}\ \mathrm{g/dm}^3$. Value of $C_{0.5}$ (g/dm^3) depends only on value of coefficient a (–) of Langmuir adsorption isotherm. Whereas the value of the parameter w (–) in Eq. (5), defined as quotient of the initial concentration C_0 (g/dm^3) and adsorbate concentration in the stream equivalent to half of terminal rate of adsorption $C_{0.5}$, is $w = 3.04$. For such parameters duration of the adsorption process, calculated using equation of Żuchowicki, Zabieziński and Tikhonov, is $t_S = 5.75$ years [26].

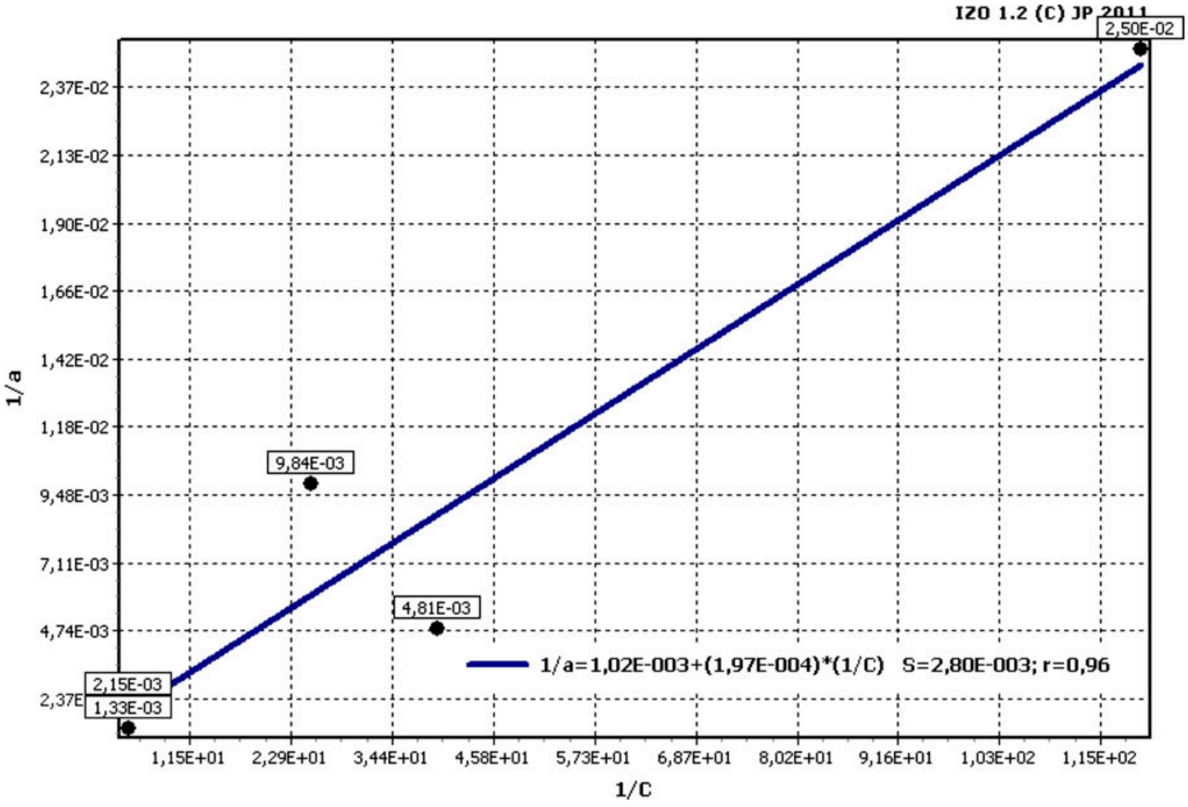

Fig. 3. Langmuir isotherm obtained using the linear method for the sorption of HCH on activated carbon NP-5.

5.2. Simplified version of Żuchowicki, Zabieziński and Tichonov equation

In Eq. (6) the component: $w^{-1} \cdot \ln(C_0 \cdot C_E - 1)$ is omitted. As a result, the equation (6) refers to the adsorption isotherm only by value of concentration of adsorbate in the adsorbent monolayer x_m (g/kg). However, elimination of this component does not change the value of duration of the adsorption process t_S (s) since the first part of Eqs. (5) and (6), i.e. $x_m \cdot \rho_N \cdot v_P^{-1} \cdot C_0^{-1}$ has a much bigger value than the value of the part concerning the difference between the adsorbent bed and the adsorption front heights.

$$t_S = \frac{x_m \cdot \rho_N}{v_P . C_0} . \left\{ H - \frac{v_P}{k_E} . \left[\ln\left(\frac{C_0}{C}\right) - 1 \right] \right\} (\mathrm{s}) \tag{6}$$

After substitution of the same values of the independent variables as in Eqs. (4) and (5) into Eq. (6) calculated duration of the adsorption process, according to simplified Żuchowicki, Zabieziński and Tichonov equation [8], is also $t_S = 5.75$ years.

5.3. Bohart–Adams equation

In Eq. (7), only the parameter w (–) is omitted. As a result, Eq. (7) similar to Eq. (6) refers to the Langmuir adsorption isotherm only by the value of concentration of adsorbate in adsorbent monolayer x_m (g/kg). The lack of w (–) parameter in Eq. (7) does not result in a significant change of the value of duration of the adsorption process t_S (s) since as in Eq. (6), the first part of Eqs. (5)–(7) i.e. $x_m \cdot \rho_N \cdot v_P^{-1} \cdot C_0^{-1}$ is much bigger than the part concerning the difference of adsorbent bed and the adsorption front heights [28].

$$t_S = \frac{x_m \cdot \rho_N}{v_P . C_0} . \left\{ H - \frac{v_P}{k_E} . \left[\ln\left(\frac{C_0}{C} - 1\right) + \ln\left(\frac{C_0}{C}\right) - 1 \right] \right\} (\mathrm{s}) \tag{7}$$

In order to compare, after substitution into Eq. (7), values of the independent variable parameters, the same as in Eqs. (4)–(6) it was calculated, based on a simplified equation Bohart–Adams [31] that the duration of the adsorption process is $t_S = 1.51$ years.

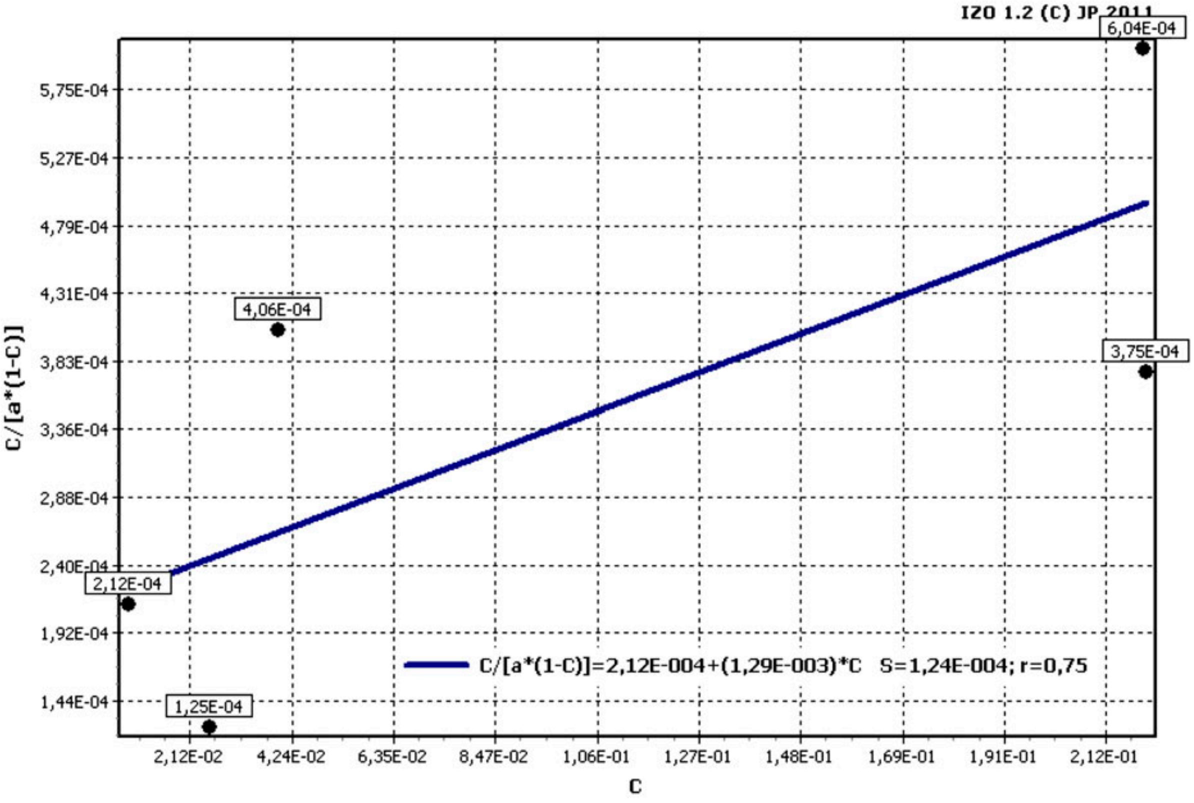

Fig. 4. BET isotherm obtained using the linear method for the sorption of HCH on activated carbon NP-5.

6. Conclusions

The present study indicates the suitability of the activated carbon NP-5 for removal of graveyard's chloroorganic pesticide HCH from aqueous solutions. The adsorption process is described using Freundlich, Langmuir, and BET formulae. The Freundlich and Langmuir models were fitting better the experimental data [32]. According to the three used models, important gaps in the adsorption capacities of the NP-5 adsorbents have been noted.

The group of isotherms according to Giles' classification (L) was achieved for chloroorganic pesticide. The HCH molecules bind to the adsorbent through only one grouping and the adsorption becomes progressively easier as the absorbed quantity increases. Thus, the first fixed molecules facilitate the adsorption of the following molecules because of the lateral attraction. The shape of these isotherms indicates that the chloroorganic pesticide HCH is adsorbed as a monolayer, and that there is no strong competition between the pesticide molecules and water to occupy the adsorption surface sites. In this case, the longitudinal axes of the adsorbed molecules are parallel to the adsorbent surface. This type of isotherm is relative to microporous adsorbents with a diameter lower than 25 Å, the adsorbent being saturated at the moment of the monolayer replenishment.

The analysis of elementary phenomena of mass transfer and, on its basis, determination of factors limiting the course of adsorption, makes it possible to select the optimum, due to effects, and duration time of process, conditions in adsorption systems for water treatment.

The analysis of adsorption dynamics equations, presented in this paper, allows one to note that an increase in the value of coefficient a (–) with the constant value of the coefficient b (–) and a decrease in the value of coefficient b (–) with the constant value of the coefficient a (–) of Langmuir adsorption isotherm, and the preservation of constant values of other input parameters causes an increase in quantity of adsorbed mass in monolayer x_m (g/kg), and thus, a decrease in the value of averaged diffusion coefficient D (m^2/s) as well as an increase in the value of criterion Schmidt number Sc (–). While values of hydraulic diffusion coefficient D_H (m^2/s), internal k_I (s^{-1}) and external k_E (s^{-1}) coefficients of mass transfer are lower. This, in turn, directly affects a decrease in the general mass transfer coefficient k_G (s^{-1}), an increase in the height of adsorption front h_F (m) and a decrease in velocity of adsorption front migration v_F (m/s). In conclusion, the described change of adsorption isotherm coefficients according to Langmuir, increases duration of adsorbent bed working time t_S (h). It may be noted that the number of units of mass transfer X_S (–) depends only on the values of the coefficient b (–) of Langmuir adsorption isotherm. The number of units of mass transfer X_S (–) increases as a result of a decrease in coefficient b (–). The lower value of grain diameter d_W (m) or layer porosity ε_W (–) of adsorbent lower the value of Reynolds criterion number Re (–), hydraulic diffusion coefficient D_H (m^2/s) and height of the adsorption front h_F (m). While, values of the following parameters increase: module of mass penetration j_D (–), internal k_I (s^{-1}) and external k_E (s^{-1}) mass transfer coefficients and thus, general mass transfer coefficient k_G (s^{-1}) and, eventually, duration of adsorbent bed working time t_S (h). An increase in porosity of adsorbent grains ε_Z (–) causes a decrease in the height of adsorption front h_F (m) and an increase in the internal mass transfer coefficient k_I (s^{-1}) and what's involved, the value of general mass transfer coefficient k_G (s^{-1}) and finally, duration of adsorbent bed working time t_S (h). While an increase in bulk density of activated carbon ρ_N (kg/m^3) results in lower velocity of migration of adsorption front v_F (m/s), and at the same time an increase in duration of adsorbent bed working time t_S (h). Lowering the value of the initial concentration C_0 (mg/dm^3), while keeping constant values of other input parameters cause a decrease in the value of: averaged diffusion coefficient D (m^2/s), hydraulic diffusion coefficient D_H (m^2/s), internal k_I (s^{-1}), and external k_E (s^{-1}) coefficients of mass transfer and thus, general mass transfer coefficient k_G (s^{-1}). The number of mass transfer units X_S (–) and velocity of migration of adsorption front v_F (m/s) also decrease. Reduction in value of the initial concentration C_0 (mg/dm^3) causes an increase in the value of Schmidt criterion number Sc (–), height of adsorption front h_f (–), and duration of adsorbent bed working time t_S (h). While, decrease in flow velocity through the bed v_P (m/s) results in decrease of: Reynolds criterion number Re (–), hydraulic diffusion coefficient D_H (m^2/s), height of adsorption front h_F (m), and velocity of adsorption front migration v_F (m/s). The value of mass penetration module j_D (–) and duration of adsorbent bed working time t_S (h) are increasing. The change of temperature of medium directed to the sorption process T (℃) directly affect values of adsorption isotherm coefficients and change of other parameters. An increase in medium temperature T (℃) causes lowering of coefficient of dynamic viscosity η_D (kg/m h), criterion Schmidt number Sc (–), module mass penetration j_D (–), and height of the adsorption front h_F (–). While, an increase in medium temperature T (℃) increases value of averaged diffusion coefficient D (m^2/s), criterion Reynolds number Re (–), hydraulic diffusion coefficient D_H (m^2/s), internal k_I (s^{-1}) and external k_E (s^{-1}) mass transfer coefficients, and general mass transfer coefficient k_G (s^{-1}). Finally, duration of adsorbent bed working time t_S (h) also increases.

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