

JELENA BASTIC¹
DEJAN SKALA²

¹Military Technical Institute,
Belgrade

²Faculty of Technology and
Metallurgy, University of
Belgrade, Yugoslavia

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335.665.1+661.185.2 +
+678.066:613.032.4+616-099

COMPARISON OF CALCULATED AND EXPERIMENTALLY DETERMINED EFFECTS OF THE PENETRATION OF HIGHLY TOXIC ORGANIC VAPORS THROUGH A LAYER OF ACTIVATED CHARCOAL CLOTH

Activated charcoal cloth (ACC) beds of uniform cross-sectional area, were subjected to different inlet vapor concentrations and volume flow rates at constant temperature. The time of breakthrough of the vapor from the bed (t_b), in an exit concentration equal to 1% of the inlet concentrations was determined for several bed weights (W) at defined concentrations and flow rates. Using benzene as a reference vapor, the performances of activated charcoal cloth were determined from the adsorption capacity (W_e) and adsorption rate constant (k_v) of the vapor. They were calculated from the straight-line relationship between t_b and W at constant vapor (benzene) concentration and gas flow rate. From these properties, the kinetic adsorption equations for 3,3-dimethylbutoxy-(2)-methylphosphorylfluoride (soman, GD) and bis-(2-chlorethyl)-thioether (S-mustard, HD) were evaluated and compared to experimental data. Fairly good agreement between the calculated and experimentally determined 1% breakthrough time of GD and HD, highly toxic organic vapors, was obtained.

Activated charcoal cloth (ACC) (pyrolysed viscose rayon activated in the gas phase) has the high affinity (i.e., large adsorption capacity) for vapors of many organic compounds. The relative non-specificity of ACC is the major advantage for its use as an adsorbent of toxic compounds (vapors) from moving air streams. A potential disadvantage of ACC is that experimental determinations need to be made for each ACC-vapor combination, from which the kinetic adsorption capacity W_e (g/g) and the adsorption rate k_v (min^{-1}) could be determined. However, adsorption kinetic theory provides the means to predict carbon performance for various vapors [1]. In a recently published paper it was shown how the existing theory of adsorption could be used to characterize an ACC with benzene as the reference vapor, and then to predict, from those data, the performance of such a material in contact with a highly toxic organic vapor [2].

THEORETICAL BACKGROUND

The initial work on predicting the equilibrium vapor adsorption performance of an activated carbon, based on the physical properties of the adsorbents, was proposed by Dubinin [3]. Calculation of the adsorption parameters of the carbon, based on an arbitrarily chosen reference vapor from isotherm data was shown in our recently published paper. The previous work indicated how it is possible by using Dubinin equations to calculate the carbon structural constant k ($[\text{mol}/\text{kJ}]^2$) and maximum adsorption volume W_0 (cm^3/g). These are two basic parameters for a particular carbon and independent of the vapor which is adsorbed. So it might be possible to calculate the carbon adsorption of other vapors at any relative pressure by using the Dubinin-Radushkevich (D-R) equation [3,4].

The adsorption volume W_v (cm^3/g) available in carbon to adsorb the vapor of interest can be determined from the D-R equation:

$$W_v = W_0 \cdot \exp \left[-\frac{k}{\beta^2} \left(R \cdot T \cdot \ln \frac{P_0}{P} \right)^2 \right] \quad (1)$$

where:

$R = 8.314 \text{ J/mol.K}$ is the gas constant,

T, K – the temperature,

β – the affinity coefficient that permits the comparison of the adsorption potential of the test adsorbate to a reference adsorbate (for the reference vapor $\beta=1$),

P_0 – the saturated vapor pressure of the adsorbate at the test temperature,

P – the equilibrium vapor pressure of the adsorbate.

The adsorption volume of a carbon for a vapor is related to the weight of vapor adsorbed per unit weight of carbon by the relationship:

$$W_v \cdot \rho = W_e \quad (2)$$

This relationship uses the concept of volume pore filling [5], which states that an adsorbed vapor fills the pore into which it is adsorbed as if it were a liquid.

Three different methods can be used to calculate the theoretical affinity coefficient [6]. For non-polar and weakly polar adsorbates, the work of Dubinin and co-workers has shown that the affinity coefficient can be expressed as a ratio of the molar volumes of the test adsorbate, V and the reference adsorbate, V_r :

$$\beta = \frac{V}{V_r} \quad (3)$$

where

$$V = \frac{M}{\rho} \quad (4)$$

M – is the molar mass of the adsorbate, g/mol

ρ – the density of the adsorbate, g/cm^3 .

Author address: J. Bastic, Military Technical Institute, Katanićeva
15, Belgrade, Yugoslavia

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More precisely, the affinity coefficient can be calculated from the ratio of the molar parachore of the test adsorbate, Ω , and of some reference adsorbate, Ω_r :

$$\beta = \frac{\Omega}{\Omega_r} \quad (5)$$

where

$$\Omega = \frac{M \cdot \gamma^{\frac{1}{4}}}{\rho} \quad (6)$$

γ – is the surface tension of the adsorbate, g/s².

For polar adsorbates, Reucroft et al. suggested that the ratio of the electronic polarization test adsorbate, P_e and reference adsorbate (P_e)_r can be used to calculate the affinity coefficient $\beta = P_e/(P_e)_r$, where:

$$P_e = \frac{(n^2 - 1) \cdot M}{(n^2 + 2) \cdot \rho} \quad (7)$$

n – is the refractive index of the liquid at the sodium D wavelength.

Jonas and Rehrmann applied predicted techniques of measuring equilibrium vapor adsorption under dynamic (or kinetic) conditions [7]. They showed that a well-packed bed of activated carbon granules exhibited an adsorption capacity very close to its equilibrium adsorption value, which was also confirmed by the results of other investigations [8].

The transition from vapor adsorption prediction at equilibrium to that under kinetic conditions requires knowledge of the adsorption rate constant k_v (min⁻¹) of the carbon which can be determined by means of a reference vapor in the same manner as performed and recently published in the case of adsorption capacity determination [2]. Such a procedure based on k_v determination for a reference substance (i.e. benzene) also provides the possibility of calculating k_v for some other, e.g. toxic, organic vapors. Although the basic physical process of gas adsorption is considered to be kinetically second order [9,10], involving, from a molecular viewpoint, the reaction between an active site on the adsorbent and a free gas molecule, the presence of excess active sites over gas molecules makes the kinetics of adsorption actually pseudo first order [11] with respect to the gas molecules. The pseudo first order adsorption rate constant k_v has not been analyzed theoretically, and its complete functional dependence is therefore not known.

The adsorption rate constant k_v , like the adsorption capacity W_e , of the reference vapor can be determined from the straight lines obtained when t_b (breakthrough time of vapor from the bed) is plotted vs. W (mass of the adsorption bed) by using the modified Wheeler equation [12]:

$$t_b = \frac{W_e}{C_0 \cdot Q} \cdot \left(W + \frac{\rho_B Q}{k_v} \cdot \ln \frac{p}{1-p} \right) \quad (9)$$

where:

t_b – is the breakthrough time, min;

W_e – the kinetic adsorption capacity, g/g;

$p = C/C_0$,

C – the contaminant concentration in the adsorbent bed, mg/dm³;

C_0 – the contaminant concentration at the inlet, mg/dm³;

W – the mass of carbon adsorbent, g;

ρ_B – the packed bed density, g/cm³;

Q – the volumetric flow rate, dm³/min;

k_v – the pseudo-first-order rate constant, min⁻¹.

Knowing W_e and k_v for any reference vapor and calculating these fundamental parameters for some other vapor, permits prediction of the vapor protection of carbon for this vapor under different conditions.

The k_v values of some other (untested) vapors can be calculated from the relationship:

$$k_{vi} = k_{vr} \cdot \frac{M_i}{M_r} \cdot \beta_i \quad (10)$$

where the subscripts represent:

i = some other (untested) vapor; and

r = the reference vapor.

If equations (1), (2) and (10) are inserted into equation (9), one obtains:

$$t_b = \frac{p_i \cdot W_{0r}}{C_{0i} \cdot Q_i} \cdot \exp \left[-\frac{k_r}{\beta_i^2} \cdot \left(R \cdot T \cdot \ln \frac{P_{0i}}{P_i} \right)^2 \right] \cdot \left[W + \frac{\rho_B \cdot Q_i}{k_{vr} \cdot \frac{M_i}{M_r} \cdot \beta_i} \cdot \ln \frac{p}{1-p} \right] \quad (11)$$

MATERIALS

The vapors used as adsorbates in these investigations were benzene as the reference vapor, p.a. grade, from E. Merck, P-6100 Darmstadt, F. R. Germany, and 3,3-dimethylbutoxy-(2)-methylphosphorylfluoride (GD), 90+% bis-(2-chlorethyl)-thioether (HD), 96+%, synthesized in our laboratory. The pertinent physical properties of these compounds and calculated values of the affinity coefficients are shown in a recently published paper [2, Table 1]. In this work only the values of affinity coefficient (β) are given in Table 1.

The activated charcoal cloth (ACC) adsorbent used for testing was CNF 1500-50 grade from Tyobo Co., Ltd., Osaka, Japan, having an internal surface area of 1350 g/m² (BET method).

Table 1. The affinity coefficient for the adsorbate vapor [2]

Benzene	β_{GD}	β_{HD}
1	1.990	1.398
1	1.936	1.535
1	1.678	1.481

ADSORPTION APPARATUS

A detailed description of the adsorption apparatus was recently published [2] and only a schematic view is shown in Figure 1.

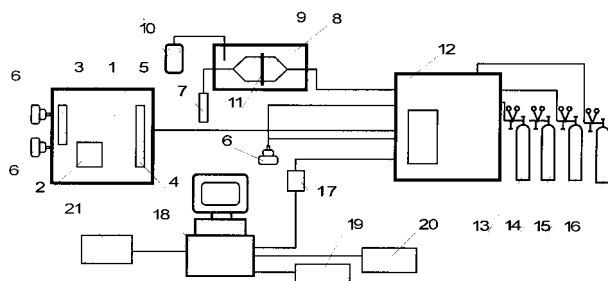


Figure 1. Vapor generation/activated charcoal cloth (ACC) adsorption system: 1 - D ynacabrator; 2 - Chamber for thermostating the diffusion tube; 3, 4 and 7 - Flowmeters; 5 - Switch for changing the vapor flow from one to another; 6 - Canister; 8 - Chamber for thermostating the sample holder; 9 - Sample holder; 10 - Thermometer; 11 - Sample; 12 - GC; 13-16 - Air, hydrogen and nitrogen cylinders for GC; 17 - Interface; 18-21 - Computer, keyboard, printer and plotter.

The apparatus consists of three functionally discrete sections, the first is for specific vapor generation, the second, for measuring vapor adsorption by the charcoal cloth bed, and the third is for vapor detection in the outlet gas phase from the charcoal cloth bed. All the necessary details covering the procedure of each experiment and the corresponding vapor detection is given in a recently published paper [2].

Results and Discussion

Experimental values of the vapor breakthrough time $t_b = t_{1\%r}$ as a function of the ACC bed weight W for benzene as a reference vapor at defined concentrations in the range from 1.7 to 33 mg/dm³ and a volumetric

Table 2. Regression equations of benzene 1% breakthrough times for ACC and adsorption parameters derived from experimental time-weight curves*

Regression line	Calculated parameters	
$t_{1\%r} = \frac{W_{er}}{C_{0r} \cdot Q_r} \cdot \left(W + \frac{\rho_B Q}{k_{vr}} \ln \frac{p}{1-p} \right)$	W_{er} g _{6H6} /g _{ACC}	k_{vr} min ⁻¹
$t_{1\%} = 142.65 \cdot W - 4.29$	0.433	2294
$t_{1\%} = 389.10 \cdot W - 12.24$	0.460	765
$t_{1\%} = 503.28 \cdot W - 16.75$	0.313	2071
$t_{1\%} = 1082.81 \cdot W - 38.84$	0.185	1920
$t_{1\%} = 180.37 \cdot W - 6.05$	0.385	3080
$t_{1\%} = 468.13 \cdot W - 13.42$	0.336	1201
$t_{1\%} = 925.93 \cdot W - 29.81$	0.374	599

* W_{er} was calculated knowing C_{0r} and Q_r from the slope of t_b vs. W , and the k_{vr} value, knowing ρ_B , p ($p = C_b/C_o = 0.01$) and C_{0r} , from the line intercept of equation 9.

flow rate of vapors from 0.028 to 0.15 dm³/min, corresponding to a superficial linear velocity from 2.8 to 15 cm/min, were obtained and subjected to linear regression analysis, in accordance with the Wheeler equation (9). The results of these analyses are shown in Table 2 and confirmed by high values of the correlation coefficient (>0.984 ; Table 2 in [2]), indicating a high degree of confidence for $t_{1\%r} = f(W)$ relationship obtained.

On the assumption that the kinetic adsorption capacity closely approximates the equilibrium capacity value at the same relative pressure and temperature, in accordance with the D-R equation (1), the experimental values of W_{er} as a function of $RT \cdot \ln \left(\frac{1}{P_{rr}} \right)$ were derived from data presented in Table 2:

$$\ln W_r = 1.9555 - 6.18 \cdot 10^3 \cdot \left(RT \cdot \ln \frac{1}{P_{rr}} \right)^2$$

$$(r = 0.985) \quad (12)$$

The maximum adsorption volume W_{or} (0.629 cm³/g; determined from the intercept) and structural constant k ($6.18 \cdot 10^{-3}$ [kJ/mol]⁻², from the slope) as the characteristic properties of ACC were derived from equation (12). The obtained relationship between k_{vr} and the corresponding P_{rr} and Q_r (Table 2, [2]), is:

$$k_{vr} = 20419 \cdot Q + 1763.3 \cdot P_{rr} - 0.4$$

$$(r = 0.993) \quad (13)$$

and the model equation which evaluates the possibility of the calculation of the 1% breakthrough time of benzene vapor, in accordance to the Wheeler equation (9), is:

$$t_{1\%r} = \frac{p_r \cdot W_{or}}{C_{0r} \cdot 10^{-3} \cdot Q} \cdot \exp \left[-k_r \left(R \cdot T \cdot \ln \frac{1}{P_{rr}} \right)^2 \right] \cdot \left(W + \frac{\rho_B \cdot Q_r}{k_{vr}} \cdot \ln \frac{p}{1-p} \right) \quad (14)$$

A comparison of the calculated and experimentally determined 1% breakthrough times for benzene as a referent vapor could be derived:

$$t_{1\%,mod,r} = 1.023 \cdot t_{1\%,exp,r} - 0.049$$

$$(r = 0.997) \quad (15)$$

This is fairly good agreement between calculation and experiments because in equation (15), both the slope (1.023) and intercept (0.049) do not significantly differ from the expected values (1 and 0).

The models of the 1% breakthrough times of GD and HD as representatives of highly toxic organic vapors, through the ACC layer, were determined by evaluating the values of the maximum adsorption capacity ($\rho_i \cdot W_{or}$) (g/cm³), the affinity coefficient β_i , (Table 1), and the adsorption rate constant k_{vi} , by means of equation (10) of these adsorbates ($i = GD, HD$), on the same adsorbent (ACC) characterized in advance by using benzene as a reference substance.

The model was experimentally tested for GD vapor by determining the 1% breakthrough times of GD vapor, at a concentration of $C_0 = 0.561 \text{ mg/dm}^3$ ($s = 1.7\%$) ($P_r = 0.166$) and volumetric flow rates of vapor from 0.05 to $0.10 \text{ dm}^3/\text{min}$, corresponding to a superficial linear velocity from 5 to 10 cm/min , through ACC beds with mass W from 0.064 to 0.130 g , at $23 \pm 1^\circ\text{C}$. The experimentally determined 1% breakthrough time value of GD vapor from the corresponding breakthrough curves is shown in Figure 2.

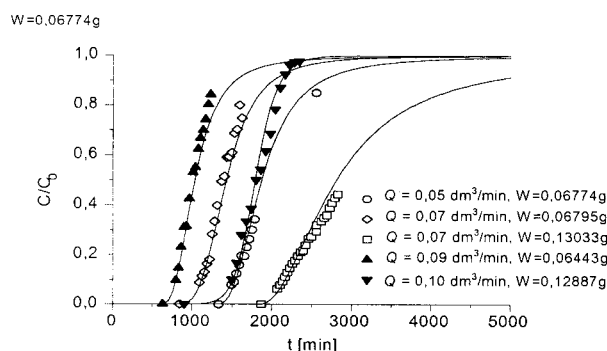


Figure 2. The breakthrough curves of GD at $23 \pm 1^\circ\text{C}$ and at concentration $C_0 = 0.561 \text{ mg/dm}^3$, for different masses of ACC and volumetric flow rates

The comparison of experimental and evaluated 1% breakthrough times values is shown in Table 3. An affinity coefficient (β) for GD of 1.678 was used for this analysis which is calculated from the ratio of the electronic polarization of benzene and GD [2]. This value of β gives the best agreement between the calculated and experimentally determined values (Table 3).

The relative differences between the experimentally determined 1% breakthrough time values and those calculated lie in the range from -5.9 to $+3.8\%$, with a mean deviation of 2.3% . The correlation between these values for GD vapor was also derived as a regression line:

$$t_{1\%,\text{mod}} = 0.990 \cdot t_{1\%,\text{exp}} + 21.40 \quad (r = 0.995) \quad (16)$$

The experimental values of the GD vapor breakthrough time, $t_{1\%,\text{exp,GD}}$ as a function of ACC bed mass W , (Table 3) at a concentration of $C_0 = 0.556$

Table 3. Comparison of experimentally determined and calculated 1% breakthrough times of GD vapors, $C_0 = 0.561 \text{ mg/dm}^3$, through ACC layers at $23 \pm 1^\circ\text{C}$

W (g)	Q (dm^3/min)	$t_{1\%,\text{exp}}$ (min)	$t_{1\%,\text{mod,eval}}$ (min)	dev (%)
0.06774	0.05	1411	1359	+ 3.8
0.06795	0.07	963	966	- 0.3
0.13033	0.07	1952	1958	- 0.3
0.06443	0.09	696	705	- 1.3
0.12887	0.10	1270	1350	- 5.9

mg/dm^3 ($P_r = 0.164$) and volumetric flow rate of $Q = 0.07 \text{ dm}^3/\text{min}$, were obtained and subjected to linear regression analysis, in accordance to the Wheeler equation (9). The result of such an analysis is the following equation:

$$t_{1\%,\text{GD}} = 15845.4 \cdot W - 114.3 \quad (17)$$

The value for the kinetic adsorption capacity $W_{e,\text{GD}}$ was calculated by using the value of the slope of equation (17), and C_0 and Q conditions. Furthermore, the $k_{v,\text{GD}}$ value (6692 min^{-1}) was calculated from the intercept of the line (equation 17) taking into account values of $W_{e,\text{GD}}$ ($0.617 \text{ gGD/gACC} = 3.385 \text{ mmolGD/gACC}$) and knowing parameters p_B , p and C_0 . The adsorption rate constant $k_{v,\text{GD}}$ could also be determined from equations 10 and 13, taking into account the calculated values of k_v for benzene at $P_r = 0.164$ and for $Q = 0.07 \text{ dm}^3/\text{min}$, and the relation between $k_{v,\text{GD}}$ and k_v . Such an experimentally determined value differs by only 0.6% to the calculated one (6731 min^{-1}) if $\beta = 1.678$ was used as the affinity coefficient of GD.

The model for calculating 1% breakthrough times for HD vapor was experimentally tested by determining the 1% breakthrough times of HD vapor at a concentration of $C_0 = 0.188 \text{ mg/dm}^3$ ($P_r = 0.242$) and volumetric vapor flow rates of 0.10 and $0.15 \text{ dm}^3/\text{min}$, corresponding to superficial linear velocities of 10 and 15 cm/min , through ACC layers, at $23 \pm 2^\circ\text{C}$. The experimentally determined breakthrough curves of HD under these conditions are shown in Figure 3.

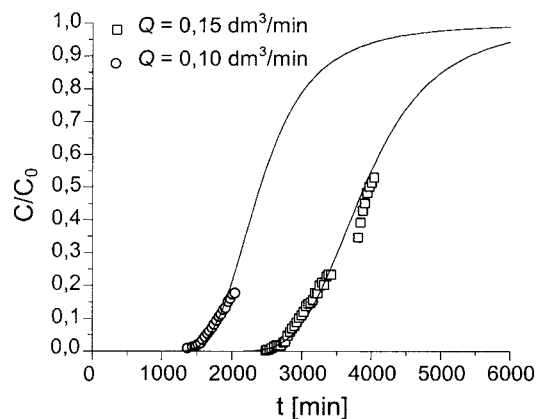


Figure 3. The breakthrough curves at concentration $C_0 = 0.188 \text{ mg/dm}^3$, for various volumetric flow rates, through ACC layers at $23 \pm 2^\circ\text{C}$

The comparison of the experimentally determined and calculated 1% breakthrough times is given in Table 4. In this case the value of 1.398 was used as an affinity coefficient of HD, calculated from the ratio of the molar volumes of benzene and HD, giving the best agreement between calculation and experiment.

The difference between the experimentally determined 1% breakthrough time values and those calculated from the corresponding linear regression was $+4.1\%$ and -9.0% , with a mean deviation of 6.6% .

Table 4. Comparison of experimentally determined and calculated 1% breakthrough times of HD vapor, $C_0 = 0.188 \text{ mg/dm}^3$, through ACC layers at $23 \pm 2^\circ\text{C}$.

W (g)	Q (dm^3/min)	$t_{1\%, \text{exp}}$ (min)	$t_{1\%, \text{mod, eval}}$ (min)	dev (%)
0.06969	0.10	2558	2457	+ 4.1
0.07027	0.15	1490	1637	- 9.0

CONCLUSIONS

Activated charcoal cloth CNF 1500–50 grade from Tyobo Co. was characterized applying a dynamic method of analysis by using, in the first step, benzene as the reference vapor. These data were the starting information necessary for developing models which could be used for predicting the 1% breakthrough time in the case of other toxic organic vapors (soman – GD, and S–mustard – HD).

Comparison of the calculated and experimentally determined 1% breakthrough time data indicates that: the relative error in the prediction of 1% breakthrough times for GD is in the range –5.9 to +3.8% (average deviation 2.3%) and from +4.1 to –9.0% (av. dev. 6.6%) for HD. The best fitting of 1% breakthrough time data was obtained when the affinity coefficient value was calculated by the method of the ratio of electronic polarizations for GD and a reference compound (benzene), and by the method of the ratio of molar volumes for HD and a reference compound.

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IZVOD

POREDENJE IZRAČUNATE I EKSPERIMENTALNO ODREĐENE KRIVE PROBOJA VISOKOTOKSIČNIH ORGANSKIH PARA KROZ ADSORPCIONU TKANINU OD UGLJENIČNOG MATERIJALA

(Naučni rad)

Jelena Bastić¹, Dejan Skala²

¹Vojnotehnički institut, Beograd, ²Tehnološko–metalurški fakultet, Beograd

Karakteristike zaštitnog sloja (tkanine) od ugljeničnog materijala (ACC) ispitivane su na konstantnoj temperaturi u cilju eliminisanja visokotoksičnih organskih para iz gasovite faze. U ovim ispitivanjima su, u cilju određivanja krive proboja pare kroz sloj tkanine (t_b), kada se nakon prolaza gasovite faze kroz adsorpcioni sloj može odrediti koncentracija toksične pare od 1% u odnosu na koncentraciju u gasovitoj fazi, korišćene različite koncentracije visokotoksične organske pare i protoci gasovite faze kroz definisan poprečni presek zaštitnog materijala od ugljenične tkanine.

Osobine zaštitnog materijala su testirane sa benzenom kao referentnim jedinjenjem na osnovu određivanja njegovog adsorpcionog kapaciteta (W_e) i konstante brzine adsorpcije (k_v), a iz pravolinijske zavisnosti parametra t_b od mase adsorbenta W za određen protok gasovite faze i koncentraciju benzene. Na osnovu ovih osobina definisana je kinetika adsorpcije 3,3–dimetilbutoksi–(2)–metilfosforilfluorida (soman, GD) i bis–(2–hloretil)–tioetra (S–mustard, HD). Dobijeno je dobro slaganje između izračunatih i eksperimentalno određenih vrednosti vremena za 1% proboja visokotoksičnim parama pri primeni zaštitnog materijala ugljenične tkanine (od –5,9 to +3,8% za GD i od +4,1 do –9,0% za HD).

Key words: Adsorption • Activated charcoal • Toxic organic vapors • Breakthrough time • Activated charcoal cloth • Adsorbent • Dynamic methods • Models of penetration •

Ključne reči: Adsorpcija • Aktivni ugalj • Toksične organske pare • Kriva proboja • Dinamička metoda • Modelovanje •

