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Adsorption of di-2-pyridyl ketone salicyloylhydrazone on Amberlite XAD-2 and XAD-7 resins: Characteristics and isotherms

P.A.M. Freitas [a,b], K. Iha [c], M.C.F.C. Felinto [d], M.E.V. Suárez-Iha [a,*]

[a] Instituto de Química, Universidade de São Paulo, Caixa Postal 26077, CEP 05513-970, São Paulo, Brasil
[b] Escola de Engenharia Mauá, Instituto Mauá de Tecnologia—IMT, São Caetano do Sul, CEP 09580-900, São Paulo, Brasil
[c] Departamento de Química, Instituto Tecnológico de Aeronáutica—CTA, São José dos Campos, CEP 12228-901, São Paulo, Brasil
[d] Instituto de Pesquisas Energéticas e Nucleares—IPEN, São Paulo, CEP 05508-000, São Paulo, Brasil

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ABSTRACT

The adsorption of DPKSH onto Amberlite XAD-2 (styrene resin) and XAD-7 (acrylic ester resin) has been investigated, at $(25 \pm 1)\,^\circ\mathrm{C}$ and pH 4.7. The experimental equilibrium data were fitted to the Langmuir, Freundlich, and Dubinin–Radushkevich (D-R) models. These three models provide a very good fit for both resins and the respective constants K_L, K_F, and K_{DR} were calculated. For the same DPKSH concentration interval, the minimum time of contact for adsorption maximum at XAD-7 was smaller than at XAD-2 and the maximum amount of DPKSH adsorbed per gram of XAD-2 is smaller than at XAD-7. The investigation indicates that the mean sorption energy (E) characterizes a physical adsorption and the surfaces of both resins are energetically heterogeneous. The constants obtained in these studied systems were correlated and compared with those obtained for the silica gel/DPKSH system.

1. Introduction

Hydrazones are azometines characterized by the triatomic grouping C=N–N and they are interesting substances because some of them may play an important role in the treatment of several diseases, such as leukemia and tuberculosis. Such an activity is directly attributed to the formation of the stable chelates with transition metals that catalyze the corresponding physiological processes [1–3]. In these chelates the aroylhydrazones can behave as bidentate, tridentate, or tetradentate ligands, when they have donor atoms in appropriate positions [1,2,4].

A typical aroylhydrazone used in the present work is di-2-pyridyl ketone salicyloylhydrazone (DPKSH) which was formerly synthesized by Garcia-Vargas et al. [4].

DPKSH with molecular formula $C_{18}H_{14}N_4O_2$ (318.33 $\mathrm{g\,mol^{-1}}$) is a reagent soluble in different organic solvents but only slightly soluble in water. In aqueous/ethanolic solutions, DPKSH presents a tautomeric equilibrium and structures I and II of Fig. 1 represent, respectively, the ketonic and enolic forms.

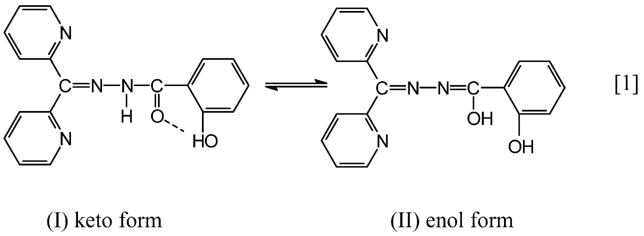

Fig. 1. Tautomeric equilibrium between the ketonic and the enolic forms of DPKSH.

In ethanolic/aqueous solutions and pH between 3.5 and 6.8 the neutral species is predominant and the maximum percentage of this species is found at pH ≈ 5. This neutral species has been used in analytical chemistry as a cromophore reagent for the determination of metallic ions [5,6] and the ligand is coordinated as an anion [7], $[C_{18}H_{13}N_4O_2]^-$.

Considering the several advantageous of solid-phase extraction processes for separation/enrichment of metal ions and the chelate properties of DPKSH, this reagent was used to modify the surface of a silica gel. DPKSH was adsorbed onto a silica surface and the process was evaluated from a quantitative and kinetic point of view [8,9].

In comparison to silica gel, alumina and activated carbon which have been extensively used to remove organic compounds from aqueous solutions, polymeric adsorbents, namely synthetic resins, possess many striking features such as stable chemical structures, high porosity and surface areas, and great adsorption capacities. XAD resins exhibit good physical properties such as porosity, uniform pore size distribution, high surface area, and chemically homogeneous non-ionic structure. They have been shown to be good adsorbents for great amounts of uncharged compounds [10] and

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[*] Corresponding author. Fax: +55 11 3815 5579.
E-mail address: maevsiha@iq.usp.br (M.E.V. Suárez-Iha).

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Table 1
Physical and chemical properties of adsorbents

Physical/chemical properties	XAD-2	XAD-7
BET surface area ($m^2\,g^{-1}$)	330	450
Particle size (mesh)	20–60	20–40
Average pore volume ($cm^3\,g^{-1}$)	0.65	1.14
Average pore diameter (Å)	90	90
Total porosity ($cm^3\,cm^{-3}$)	0.420	0.550
Dry/wet density ($g\,cm^{-3}$)	1.07/1.02	1.24/1.05
Polarity	Nonpolar	Polar (intermediate)
Structure	Styrene	Acrylic ester
Amount of DPKSH adsorbed ($mg\,g^{-1}$)	2.2	10.4

they have, therefore, been used as supports for immobilization of chelating agents and metal complexes [11]. However, no report was found that employed polymeric resins for adsorption of DPKSH.

In this paper, two commercial Amberlite resins, XAD-2 and XAD-7, were used to study the adsorption of DPKSH and a comparison between these two materials was done.

2. Materials and methods

2.1. Reagents and solutions

All reagents and solvents were of analytical reagent grade. The synthesis of DPKSH was performed as previously reported by Garcia-Vargas et al. [4]. The ligand was obtained from the reaction of stoichiometric amounts of di-2-pyridyl ketone and salicyloylhydrazide in ethanol. The product was collected on a Büchner funnel and recrystallized twice from an ethanol–water solution. After synthesis, the melting point of the product was verified to be $(173 \pm 1)\,°C$ and it is in excellent agreement with the literature [4]. Stock 10^{-3} mol L^{-1} DPKSH solutions were prepared by dissolving the reagent in ethanol 100%.

The pHs of the solutions were adjusted to pH 4.7 with a sodium acetate and glacial acetic acid ($0.1/0.1$ mol L^{-1}) buffer solution.

Aldrich Chemical resins [12,13], Amberlite XAD-2 and XAD-7, were used in this work and their respective physical properties are represented in Table 1. Amberlite XAD-2 and XAD-7 resins have some similar properties but are quite different in their chemical compositions and BET surface areas and average pore volumes. XAD-7 has the larger surface area and average pore volume. XAD-2 is a styrene divinylbenzene copolymer which is not easily wetted and absorbs little water because of its hydrophobic nature. XAD-7 is an acrylic ester polymer of intermediate polarity and this resin is more hydrophilic than styrene divinylbenzene copolymer; accordingly, it is more easily wetted and absorbs more water.

The resins XAD-2 and XAD-7 were thoroughly washed with HCl solution (0.1 mol L^{-1}), in a bath for 15 min to remove any impurity and the reagents NaCl and Na_2CO_3 originally added to the resins in order to prevent the bacterial growth. Then, each resin was collected on a Büchner funnel with a filter paper and repeatedly washed with deionized water, until the filtrate was almost neutral ($pH \approx 6$). Also, the washed resins were dried in a desiccator for 4 days to remove any adsorbed water.

The cuvettes and all glass and plastic vessels used in these experiments were washed with 10% HNO_3 aqueous solution followed by distilled and deionized water to remove any trace of contaminants.

The stock DPKSH solution was stored in plastic vessels to avoid adsorption on glass surfaces.

2.2. Apparatus

The UV–vis spectra were registered on a Hitachi U3000 spectrophotometer using 1.00-cm quartz cells. The pH measurements were performed with a Metrohm 654 pH-meter equipped with a Metrohm glass electrode, the Ag/AgCl reference electrode of which was filled with 3 mol L^{-1} NaCl. Centrifugation of the mixtures was carried out in an Incibras–Spin IV centrifuge. The agitation of the mixtures was performed on a shaker TE-140 Tecnal with controlled velocity. The room temperature was kept constant at $(25 \pm 1)\,°C$, in all experiments.

2.3. Spectral characteristics of DPKSH

DPKSH spectra for solutions at pH 4.7 and 3% ethanol/water (v/v) were registered in the UV–vis region. The analytical wavelength was established from the DPKSH spectrum and the maximum absorbance value was found at 319 nm. The analytical curve for DPKSH aqueous solutions containing 3% ethanol (v/v) was obtained and the respective parameters were calculated using the least-squares method.

The linear equation can be written as

$$A_{319\ \mathrm{nm}} = 1.98 \times 10^4 C_{\mathrm{DPKSH}} - 0.0293. \tag{2}$$

The correlation coefficient, $r^2 = 0.9996$, reveals the excellent fitting of the experimental data to a linear behavior.

2.4. Adsorption and elution experiments

DPKSH adsorption kinetics and isotherms were determined using the solution depletion method.

For kinetic studies the solutions (sample and blank) were prepared in 10.0 mL volumetric flasks. In stoppered plastic tubes 0.1 g of the resin was kept in contact with an aqueous solution (10.0 mL) of DPKSH (pH 4.7) containing 3% ethanol/water (v/v) and shaken during some specific time periods. The supernatants were separated by centrifuging the mixtures and the concentration of DPKSH was calculated by the absorption measurement at 319 nm (pH 4.7). The blank was prepared in the same way but without DPKSH solution. The temperature was kept constant at $(25 \pm 1)\,°C$ and a constant shaking speed was kept in the shaking table.

For isotherm studies in stoppered plastic tubes 0.1 g of XAD-2 and XAD-7 was shaken separately for specific periods with an aqueous solution (10.0 mL) of DPKSH at pH 4.7. The defined time of contact was based on the kinetic study and the experimental procedure was the same as described above.

The same experimental procedure was used to check the elution of DPKSH loaded in the XAD-7 resin. Nitric acid aqueous solutions at different concentrations (1×10^{-4}; 1×10^{-3} and 1×10^{-2}) and deionized water were used for the elution studies. In stoppered plastic tubes 0.3 g of XAD-7 was shaken for 10 min with 10.0 mL of each eluent. The supernatants were separated by centrifuging the mixtures and the concentration of DPKSH was calculated on the basis of the absorption measurement.

3. Results and discussion

3.1. Kinetics of adsorption

Several experiments were conducted to assess the minimum time necessary to obtain a maximum adsorption of DPKSH onto Amberlite resins XAD-2 and XAD-7. These experiments were carried out at pH 4.7, which was established on the basis of the DPKSH equilibria characterized by the presence of predominantly RN species which corresponds to the neutral DPKSH.

The kinetics of adsorption onto XAD-2 and XAD-7 was studied with a 4.05×10^{-5} mol L^{-1} DPKSH aqueous solution containing 3% ethanol (v/v). The variation of the mass of DPKSH adsorbed per gram of resin as a function of time is shown in Fig. 2, for both

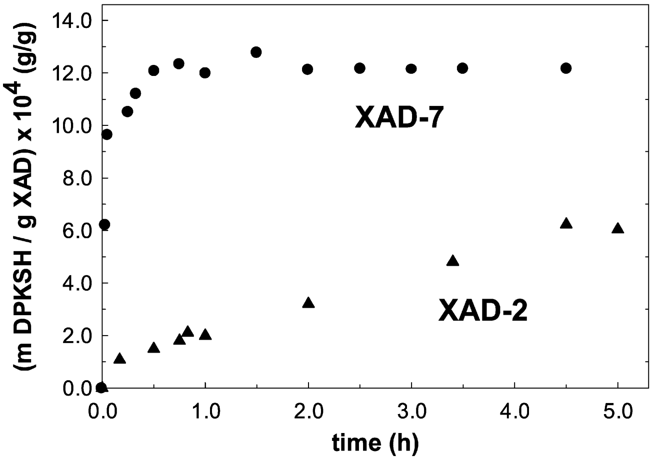

Fig. 2. Dependence of adsorbed mass of DPKSH per XAD-2 and XAD-7 unit mass on time of contact at pH 4.7. DPKSH 4.05×10^{-5} mol L^{-1}.

materials. The maximum sorption of the DPKSH supernatant solution is achieved after 5 h for XAD-2 and 1 h for XAD-7 under the same conditions. The acrylic ester resin (XAD-7) reaches equilibrium faster than styrene resin (XAD-2) because the former is capable of absorbing more water than the latter, and the acrylic ester resin expands about 20% in diameter after water regain. The expansion of XAD-7 increases the porosity of the beads and permits the solute to attain equilibrium rapidly [14].

3.2. *Adsorption isotherms*

For thermodynamic consideration in terms of adsorption isotherms, many experimental values were obtained for the adsorption of DPKSH on XAD-2 and XAD-7 and were plotted according to Langmuir, Freundlich, and Dubinin–Radushkevich models. All of these data were applied to different mathematical expressions which describe the adsorption equilibria. These equations correspond to the Langmuir, Freundlich, and Dubinin–Radushkevich (D-R) isotherm models [15–17] as cited above.

The Langmuir model [15] was originally developed to represent chemisorption on a set of well-defined localized adsorption sites having the same adsorption energy, independent of the surface coverage and with no interaction between adsorbed molecules. Maximum sorption is achieved when the surface of adsorbent is covered with a monolayer of adsorbate. The familiar form of the Langmuir isotherm based on the kinetic consideration is expressed as

$$m_{\text{ads}} = \frac{m_{\text{ads}}^{\max} K_L C_{\text{eq}}}{1 + K_L C_{\text{eq}}}, \tag{3}$$

where m_{ads} is equal to the amount of DPKSH adsorbed on XAD (g g^{-1}); $m_{\text{ads}}^{\max}$ is the maximum amount of solute adsorbed (g) in gram of XAD and this parameter is called the adsorption capacity, C_{eq} is the equilibrium concentration of DPKSH in solution (g L^{-1}), and K_L is the Langmuir constant (L g^{-1}) which is related to the equilibrium constant of adsorption process.

Fig. 3 shows the experimental data m_{ads} vs C_{eq} for XAD-2/DPKSH (a) and XAD-7/DPKSH (b). The fit of these curves was carried out using the software Jandel Sigma Plot for Windows Version 3.02 by adjusting the experimental data to Eq. (3). The parameters K_L are $(1.24 \pm 0.03) \times 10^2$ and $(1.17 \pm 0.01) \times 10^2$ L g^{-1} and $m_{\text{ads}}^{\max}$ are $(2.2 \pm 0.2) \times 10^{-3}$ and $(1.04 \pm 0.04) \times 10^{-2}$ g DPKSH/g resin for XAD-2 and XAD-7, respectively. The solid lines represented in the curves in Figs. 3a and 3b were calculated using the parameters obtained with the experimental values and an excellent fit was found. As can be observed from the results, K_L values are quite similar but the adsorption capacities, $m_{\text{ads}}^{\max}$, are very different. The found values of $m_{\text{ads}}^{\max}$ have shown that the adsorption capacity for DPKSH on Amberlite XAD-7 is almost five times higher than on XAD-2. The higher amount adsorbed on the XAD-7 resin, when compared to the XAD-2 resin, may be correlated with its higher surface area and the pore size which has an important effect on the adsorption capacity.

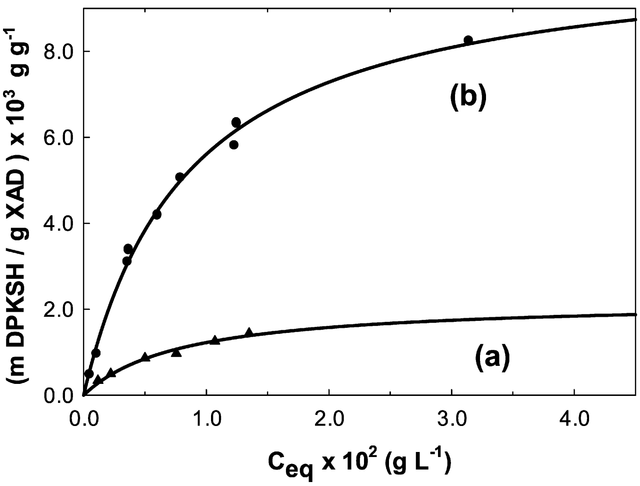

Fig. 3. Adsorption isotherms of DPKSH (aqueous solution containing 3% (v/v) ethanol) onto XAD-2 (a) and XAD-7 (b). $T = (25 \pm 1)\,^\circ\text{C}$. Solid lines correspond to the Langmuir model fitting.

The Freundlich isotherm [16], one of the more widely employed mathematical descriptions, usually fits the experimental data over a wide range of concentrations. This isotherm gives an empirical expression encompassing the surface heterogeneity and the exponential distribution of active sites and their energies. The Freundlich model is expressed as

$$m_{\text{ads}} = K_F C_{\text{eq}}^{1/n}, \tag{4}$$

where m_{ads} and C_{eq} have the same definitions as previously presented for the Langmuir isotherm, K_F is an empirical constant which provides an indication of the adsorption capacity of the adsorbent, and $1/n$ indicates the energetic heterogeneity of adsorption sites. The logarithmic form of Eq. (4) gives the following linear equation:

$$\log m_{\text{ads}} = \log K_F + \frac{1}{n} \log C_{\text{eq}}. \tag{5}$$

The plots of $\log m_{\text{ads}}$ vs $\log C_{\text{eq}}$ are shown in Fig. 4 for XAD-2/DPKSH (a) and XAD-7/DPKSH (b). The Freundlich constants $1/n$ and K_F were determined from the slope and the intercept of the plots. The numerical value of adsorption capacity K_F and n indicating the energy and intensity are, respectively, (18 ± 2) mg g^{-1} and (1.7 ± 0.1) for XAD-2/DPKSH, and (170 ± 10) mg g^{-1} and (1.3 ± 0.1) for XAD-7/DPKSH. The solid lines represented in the curves in Figs. 4a and 4b were calculated using the parameters obtained with the experimental values and an excellent fit was achieved as can be observed. The high correlation shows that the model is very suitable for describing the tested adsorption systems in the studied concentration ranges.

The Freundlich adsorption isotherm does not predict any saturation of the solid adsorbent surface; thus, infinite surface coverage is predicted mathematically. The values of K_F are quite different; adsorption on XAD-7 shows a value 10 times higher than the XAD-2 value which represents a stronger interaction between DPKSH and the resin XAD-7 than that with XAD-2. The values of $1/n$ obtained for the adsorption of DPKSH in both resins were less than 1 (0.59 for XAD-2 and 0.77 for XAD-7), indicating favored adsorption. The n parameters of the Freundlich equation reveal that

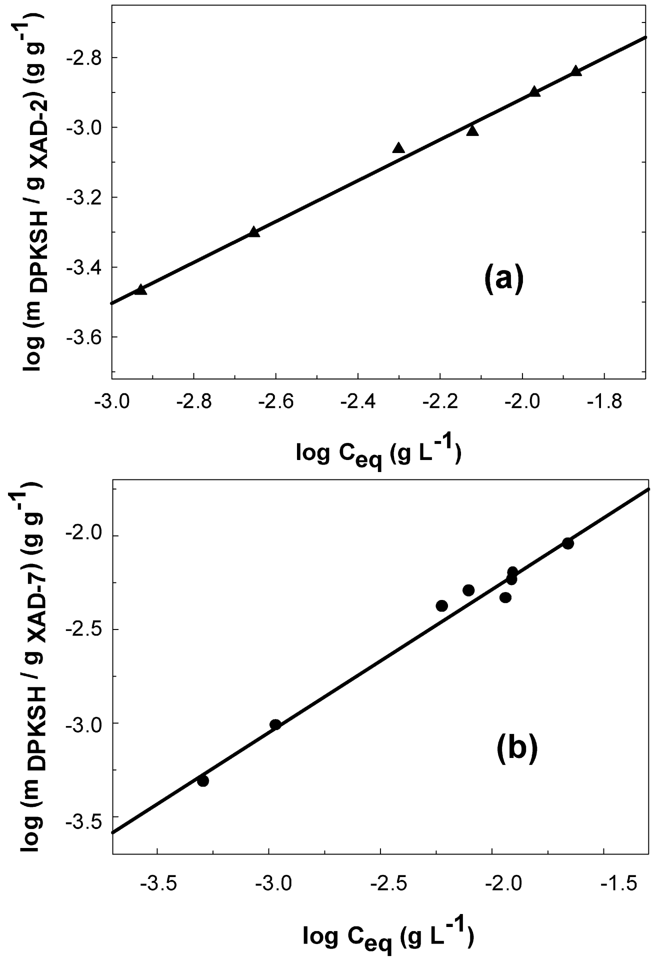

Fig. 4. Adsorption isotherms of DPKSH (aqueous solution containing 3% (v/v) ethanol) onto XAD-2 (a) and XAD-7 (b). $T = (25 \pm 1)\,^\circ\mathrm{C}$. Solid lines correspond to the Freundlich model fitting.

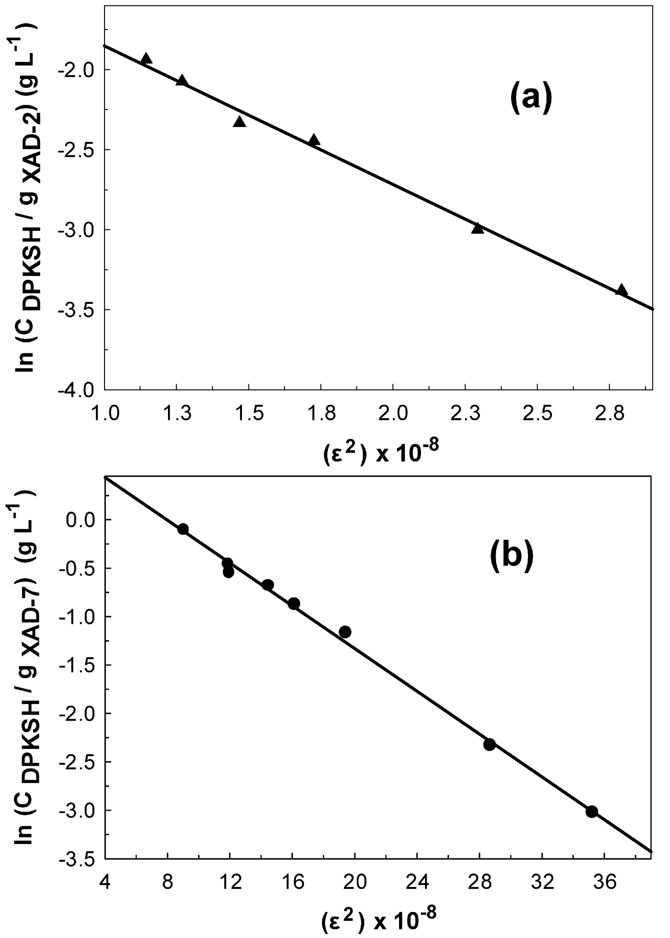

Fig. 5. Adsorption isotherms of DPKSH (aqueous solution containing 3% (v/v) ethanol) onto XAD-2 (a) and XAD-7 (b). $T = (25 \pm 1)\,^\circ\mathrm{C}$. Solid lines correspond to the Dubinin–Radushkevich (D-R) model fitting.

the adsorption sites have a narrow energy distribution since its values were close to 1 (1.7 and 1.3).

Although the Langmuir isotherm describes adsorption on strongly homogeneous surfaces, which is not the case for the adsorbents used in the present study because they exhibit a chemical heterogeneity, the n parameter of the Freundlich adsorption model reveals adsorption sites with low energetic heterogeneity. Based on this reason it can be seen from the obtained data that the Langmuir equation can be fitted with desirable r^2 equal to 0.9806 and 0.9943 with a monolayer capacity of 2.2 and 10.4 $\mathrm{mg\,g^{-1}}$, respectively, for XAD-2 and XAD-7.

Adsorption can be defined as a process of molecules accumulating from a bulk solution onto the exterior and interior surface of an adsorbent, and may include various interactions such as hydrophobic, electrostatic attraction, and hydrogen bonding. Physical properties of the resins like surface area, pore distribution, pore radius, and porosity and chemical characteristics of the DPKSH (adsorbate) solution can also play an important role in determining states and equilibria of adsorption [18] and the adsorption may be characterized as physical or chemical. Then, in order to distinguish between physical and chemical adsorption, the experimental data were applied to a Dubinin–Radushkevich (D-R) isotherm model [17]. In this model, the feature is the heterogeneity of energies over the surface. The linear form of D-R isotherm is given by

$$\ln C_{\mathrm{ads}} = \ln K_{\mathrm{DR}} - B\varepsilon^2, \tag{6}$$

where C_{ads} is the amount of DPKSH adsorbed per unit mass of the XAD ($\mathrm{mol\,L^{-1}\,g^{-1}}$), K_{DR} is the maximum amount of DPKSH adsorbed, and B is a constant with dimensions of energy and Polanyi potential $(\varepsilon) = RT\ln[1 + (1/C_{\mathrm{eq}})]$, where R is a gas constant in $\mathrm{kJ\,K^{-1}\,mol^{-1}}$ and T is the temperature in Kelvin.

The plots of $\ln C_{\mathrm{ads}}$ vs ε^2 in Fig. 5 for XAD-2/DPKSH (a) and XAD-7/DPKSH (b) show the linear behavior between $\ln C_{\mathrm{ads}}$ and ε^2. The calculated values of B and K_{DR} from the slope and the intercept are $(-4.2 \pm 0.2) \times 10^{-9}$ and $(6.17 \pm 0.01) \times 10^{-3}$ $\mathrm{mol\,g^{-1}}$ for XAD-2/DPKSH and $(-5.5 \pm 0.2) \times 10^{-9}$ and $(6.63 \pm 0.01) \times 10^{-2}$ $\mathrm{mol\,g^{-1}}$ for XAD-7/DPKSH.

According to Saeed et al. [19] if the surface is considered heterogeneous and an approximation to a Langmuir isotherm is chosen as a local isotherm for all sites that are energetically equivalent then the quantity $B^{1/2}$ can be related to the mean sorption energy (E) as

$$E = \frac{1}{\sqrt{-2B}}, \tag{7}$$

which is the free energy of the transfer of 1 mol of solute from infinity to the surface of Amberlite resin. The numerical values of E calculated from Eq. (7) are 10.8 and 9.54 $\mathrm{kJ\,mol^{-1}}$ for XAD-2/DPKSH and XAD-7/DPKSH, respectively.

The IR spectra and elemental analysis of both resins and of DPKSH were obtained. The IR spectra of each resin, loaded with DPKSH or not, were quite similar. No significant difference was observed probably due to the low mass of DPKSH adsorbed per gram of each resin. The ν (C–O) phenolic group in 1134 $\mathrm{cm^{-1}}$ ob-

Table 2
Parameters of the equations of Langmuir, Freundlich, and Dubinin–Radushkevich (D-R) for adsorption of DPKSH on different adsorbents

Adsorbent	Langmuir		Freundlich		Dubinin–Radushkevich		
	K_L ($\text{L}\,\text{g}^{-1}$)	$m_{\text{ads}}^{\text{max}}$ ($\text{g}\,\text{g}^{-1}$)	K_F ($\text{mg}\,\text{g}^{-1}$)	n	K_{DR} ($\text{mol}\,\text{g}^{-1}$)	B ($\text{J}^2\,\text{mol}^{-2}$)	E ($\text{kJ}\,\text{mol}^{-1}$)
Silica (pH 1)	5.3	1.5×10^{-3}	3.9	1.2	3.25×10^{-3}	-7.4×10^{-9}	8.22
Silica (pH 4.7)	144	2.7×10^{-4}	1.7	1.9	6.2×10^{-4}	-3.8×10^{-9}	11.5
XAD-2	124	2.2×10^{-3}	18	1.7	6.17×10^{-3}	-4.2×10^{-9}	10.8
XAD-7	117	1.04×10^{-2}	170	1.3	6.63×10^{-2}	-5.5×10^{-9}	9.54

served for the adsorbate does not appear in the spectra of XAD-2 and XAD-7 loaded with DPKSH but a strong peak in 1151 cm^{-1} appears in the XAD-7 spectrum due to the ester group. The carbonyl stretching mode, ν (C=O) in 1636 cm^{-1}, is observed in both DPKSH and XAD-7/DPKSH. The oxygen, carbon, and hydrogen contents of XAD-7 are about 30.6, 62.1, and 7.3%, respectively. The nitrogen content of XAD-7 loaded with DPKSH is 0.17% and it corresponds to 0.0121 g of DPKSH ($C_{18}H_{14}N_4O_2$) per gram of resin. This value may be compared with the $m_{\text{ads}}^{\text{max}}$ found as $(1.04 \pm 0.04) \times 10^{-2}$ g DPKSH/g XAD-7 and a very good agreement was achieved.

All results obtained for XAD-2 and XAD-7 loaded with DPKSH are shown in Table 2. The same adsorbate was used to study the behavior of silica gel as adsorbent, and the data were published elsewhere [8] and the parameters obtained from the three isotherm models are also shown in Table 2. The adsorption of DPKSH onto silica gel follows Langmuir, Freundlich, and D-R adsorption models at both pH (4.7 and 1) studied. The maximum amount of DPKSH (g) per gram of silica obtained from the Langmuir isotherm model at pH 1 was higher than at pH 4.7.

As can be observed from Table 2, except for silica (pH 1), the values of K_L are quite similar and it can be assumed that the equilibrium constants of adsorption processes are very close. The difference with silica (pH 1) may be ascribed to the predominance of the DPKSH in the protonated form, because the other systems were studied at pH 4.7, with predominance of the neutral form of DPKSH.

Parameters n evaluated from the Freundlich isotherm are 1.2 (silica/pH 1), 1.9 (silica/pH 4.7), 1.7 (XAD-2), and 1.3 (XAD-7). These values characterize the presence of energetically heterogeneous adsorption sites in all of the adsorbents used [20]. The values of the mean sorption energy (E) may also be compared and all of them characterize a physical adsorption.

Considering the highest adsorption shown by the XAD-7, it is worthwhile to investigate the elution behavior of DPKSH from the resin and the possibility of its recycled use. The elution was checked with deionized water and HNO_3 aqueous solution at different concentrations. The lowest concentration of HNO_3 ($1 \times 10^{-4}\ \text{mol}\,\text{L}^{-1}$) led to an elution of 4.5% of the total adsorbed DPKSH, after two washes of the loaded resin. The highest concentration of HNO_3 ($1 \times 10^{-2}\ \text{mol}\,\text{L}^{-1}$) led to an elution of only 14% of the total adsorbed DPKSH, also after two washes. Two washes with deionized water led to an elution of 3% and after six washes only 16% of the total adsorbed DPKSH was eluted.

4. Summary

The adsorption capacity of DPKSH onto both resins was studied and it was found to be higher onto XAD-7 than XAD-2. The adsorption equilibrium data were analyzed using three adsorption models and the results have shown that the adsorption behavior of DPKSH by XAD-2 and XAD-7 could be described by Langmuir, Freundlich, or Dubinin–Radushkevich models. The maximum amount of DPKSH (g) per gram of XAD for XAD-7/DPKSH is higher than that for the XAD-2/DPKSH system. Dynamic studies indicate that XAD-2 resin needs more time to achieve adsorption equilibrium than XAD-7 resin.

The constants obtained from two isotherms related to the ratio between grams of DPKSH and volume (L) or mass of resin, that is, K_F and K_{DR} show the same behavior as the maximum amount of the DPKSH adsorbed onto the XADs. The relationship between K_F (XAD-7/XAD-2) values is 9.4 and between K_{DR} (XAD-7/XAD-2) values is 10.7. The different characteristics of isotherms investigated for XAD-2 (styrene resin) and XAD-7 (acrylic ester resin) may be ascribed to the different hydrophobicities of the two kinds of resins. The former is strongly hydrophobic because of its hydrocarbon structure, but the latter is partially hydrophilic due to the presence of polar carboxylic ester groups.

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