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Adsorption of anionic food azo dyes from aqueous solution by silica modified with cetylpyridinium chloride

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Abstract

A silica modified by cetylpyridinium cations for the effective preconcentration of azo dyes from dilute aqueous solutions was proposed. The morphology of the modified silica was studied by SEM and it was shown that the modified silica surface has a 3D framework structure. The fixation of cetylpyridinium cations on the silica surface was confirmed by the diffuse reflectance infrared Fourier-transform spectroscopy method. Using the mathematical planning of the experiment via the method of Latin squares; the conditions for the adsorption of dyes have been optimized. It was found that the maximum dye adsorption occurs at pH 1.5–2.5 where azo dyes are in the ionic form with dissociated sulfo groups. The formation of ion pairs of azo dye anions and cetylpyridinium cations in solution was proved by spectrophotometry and on the surface by diffuse reflection spectroscopy. It is shown that adsorption isotherms are of the H-type and they are well described by the Langmuir equation. The maximum adsorption capacity of the modified silica with respect to azo dyes ranges from 2 to 9 µmol/g and depends on the geometric dimensions and spatial configuration of the dyes molecules. Thermodynamic studies have made it possible to establish the endothermic nature of adsorption. It is shown that the most effective eluent is 1.0×10^{-3} mol dm^{-3} solution of sodium dodecylsulfate in 0.1 mol dm^{-3} solution of NH$_3$·H$_2$O, and desorption itself occurs due to the destruction of ion pairs of dye anions with cetylpyridinium cations fixed on the surface.

Graphical abstract
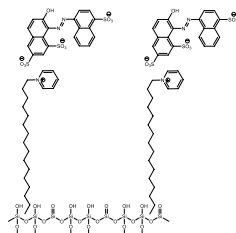
Introduction

To date, synthetic dyes are used more often than natural dyes, and the area of their application is extremely wide. They are mainly used for coloring beverages, confectionery products, and pharmaceuticals to create an attractive appearance for the consumer. However, depending on the concentration, food dyes can have a negative effect on the human body and exhibit carcinogenic, mutagenic, and allergenic properties [1–3]. For example, in [4] the

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toxic effect of tartrazine and carmoisine was studied and it was shown that the use of these dyes adversely affects the biochemical parameters of the liver and kidneys not only at high, but also at low doses. Various methods have been proposed for the identification and quantification of food dyes, among which are chromatography [5, 6], spectrophotometry [7, 8], and capillary electrophoresis [9, 10]. The main problem of determining food colors is the difficulty in extracting them from analytical samples. For this purpose, various extraction methods are used, for example, classical liquid–liquid, homogeneous and cloud point extraction [11, 12]. However, it is assumed that they work with toxic organic solvents and are not always effective enough, so they are used only for a limited range of foods. In our opinion, a convenient method of separating the component to be determined is sorption, since it allows one to combine ease of execution, environmental appeal, and high efficiency [13–16]. In addition, it seems possible to combine sorption preconcentration with various detection methods, such as diffuse reflection spectroscopy and voltammetry [17, 18]. Several studies in this field are known, for example, a method for determining the dye sunset yellow FCF by diffuse reflection spectroscopy after sorption concentration [13, 16] on Al_2O_3 is proposed. However, the extraction of dyes by the proposed sorbents is quite long in time and, in some cases, not quantitative. Thus, the search for cheap, affordable, and efficient sorbents remains an urgent task. Among the many sorbents, silica is advantageous because of its abundance, the simplicity of its surface modification, chemical and mechanical stability, and also whiteness, which allows combining sorption concentrating with analytical signal measurement

using diffuse reflection spectroscopy or tristimulus colorimetry [19, 20].

It is known [21–23] that silica adsorbs surfactants due to the van der Waals forces and these materials are effective for the extraction of anionic substances. In addition, silicas and modified silicas can act as modifiers of carbon-paste electrodes in voltammetry [24] and it can be used in diffuse reflection spectroscopy. Thus, the aim of this paper is to study the adsorption of anionic food dyes with silica modified with cetylpyridinium chloride (CPC) cationic surfactant.

Results and discussion

Characterization of adsorbent

The morphological properties of the silicas were investigated by SEM (Fig. 1). It can be seen from Fig. 1 that the surface of the non-modified silica was rougher than that of CPC-modified silica. The non-modified silica consists of particles with poor agglomeration, and the aggregation takes place after modification by CPC. The CPC-modified silica surface has a 3D framework structure, which is beneficial to obtain high sorption efficiency. The diffuse reflectance infrared Fourier-transform spectroscopy (DRIFT) [25] was used to study the fixing of CPC on the surface. The DRIFT-spectra of non-modified and modified silicas are given in Fig. 2.

In both cases, an absorption band about 1050 cm^{-1} can be attributed to stretching vibrations of Si–O–Si. The 3750 cm^{-1} band due to OH stretching vibration and according to [26] the bands about 3650 cm^{-1} can be attributed to internal silanols. In the case of CPC-modified silica,
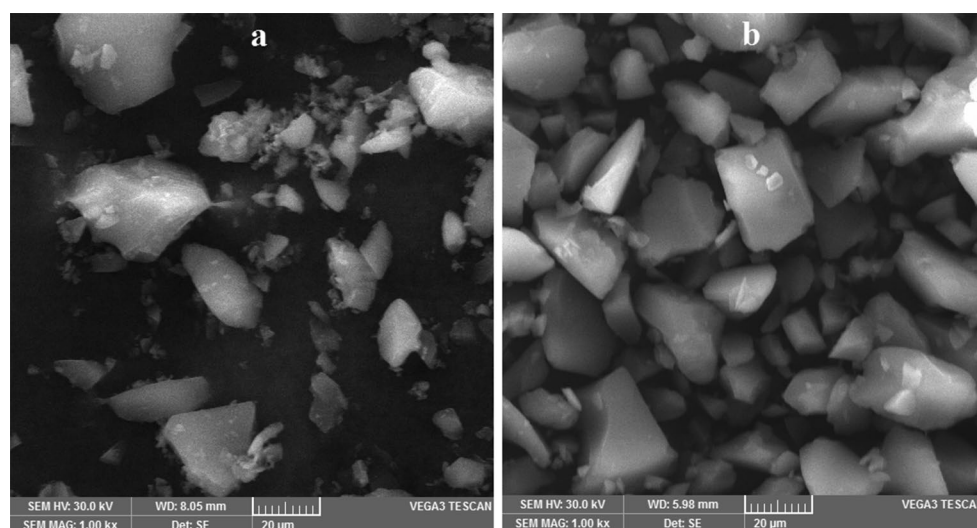
Fig. 1 SEM images of **a** non-modified silica and **b** silica modified with CPC
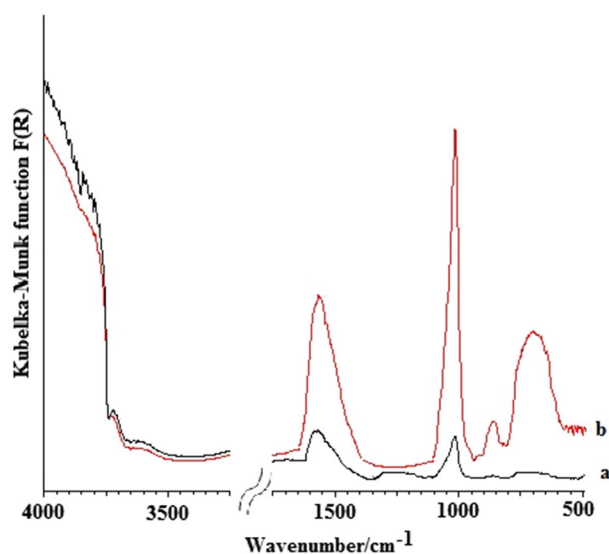
Fig. 2 DRIFT spectra of **a** non-modified silica and **b** silica modified with CPC

absorption bands at 700 and 850 cm^{-1} are additionally visible and which should be attributed to the bending vibrations of the CH groups of the pyridine ring. The 1600 cm^{-1} band in DRIFT spectrum of CPC-modified silica due to C=N stretching vibration and it overlaps the absorption band of H–O–H bending vibrations around 1610 cm^{-1} of adsorbed water in non-modified silica. The vibrations of the carbon chain are invisible, which probably indicates its participation in van der Waals interaction with the silica surface. After the adsorption of studied dyes (Fig. 3) on CPC-modified silica, the absorption bands at 1600 cm^{-1}, 700 cm^{-1} and 850 cm^{-1}

in DRIFT spectra disappear and an absorption band of about 1610 cm^{-1} of non-modified silica is again observed. Such changes are not typical, though they are evidence of surface change. Probably, dye molecules screen CPC cations fixed on the surface, which leads to the disappearance of the corresponding absorption bands. To clearly confirm the formation of azo dye anions and CPC cations ion pairs the UV–Vis absorption spectra in solutions (Fig. 4) and on the modified silica surface (Fig. 5) have been studied.

## The state of azo dyes in solutions and on CPC-modified silica surface

The electronic absorption spectra of azo dyes (Fig. 3) in aqueous solutions and in the presence of CPC are shown in Fig. 4. It is clearly seen from Fig. 4a–e that the interaction of dyes with CPC cations is indicated by a slight bathochromic shift of the absorption band (~5 nm) and a hypochromic effect. In the case of allura red AC (Fig. 4a), ponceau 4R (Fig. 4b), carmoisine (Fig. 4c), and sunset yellow (Fig. 4d) the formation of ion pairs is further confirmed by the formation of shoulders in the absorption spectra at 535, 545, 560, and 515 nm, respectively. For tropaeolin O (Fig. 4f), a bathochromic shift of the absorption band (~20 nm) and a hyperchromic effect are observed. Probably, the formation of an ion pair is accompanied by tautomerization of tropaeolin O. According to the changes observed in the absorption spectra, it can be concluded that ion pairs are formed with the participation of negatively charged sulfo groups of azo dyes and the cetylpyridinium cation. This is due to the state of these dyes in
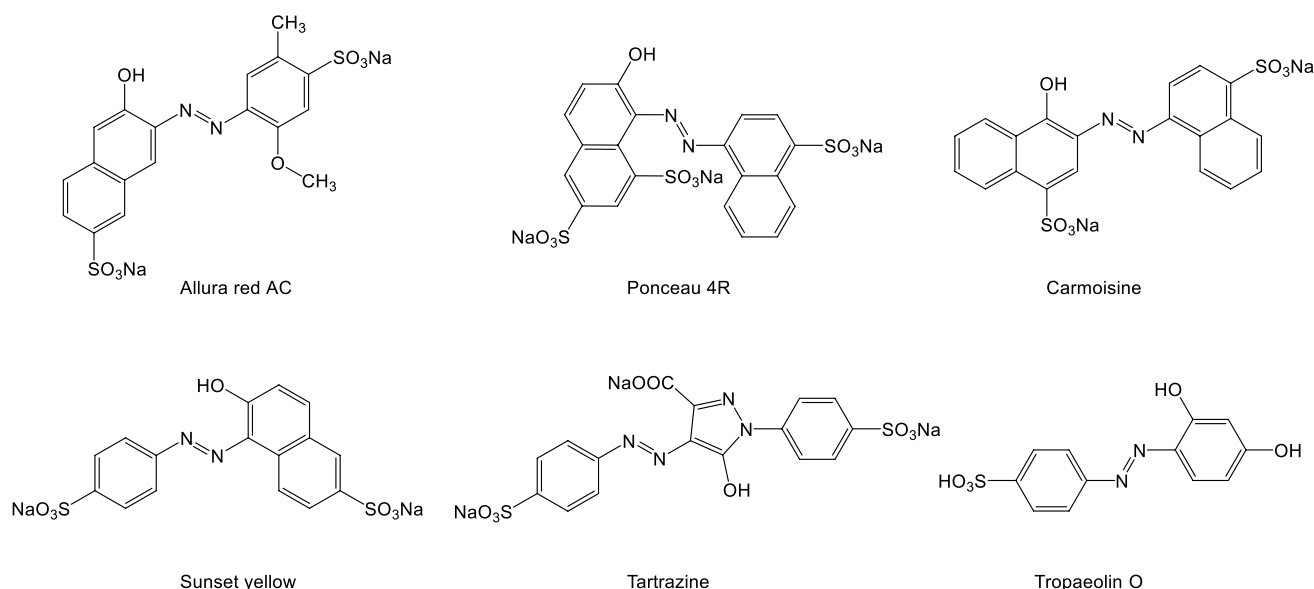
Fig. 3 The structures of the studied azo dyes
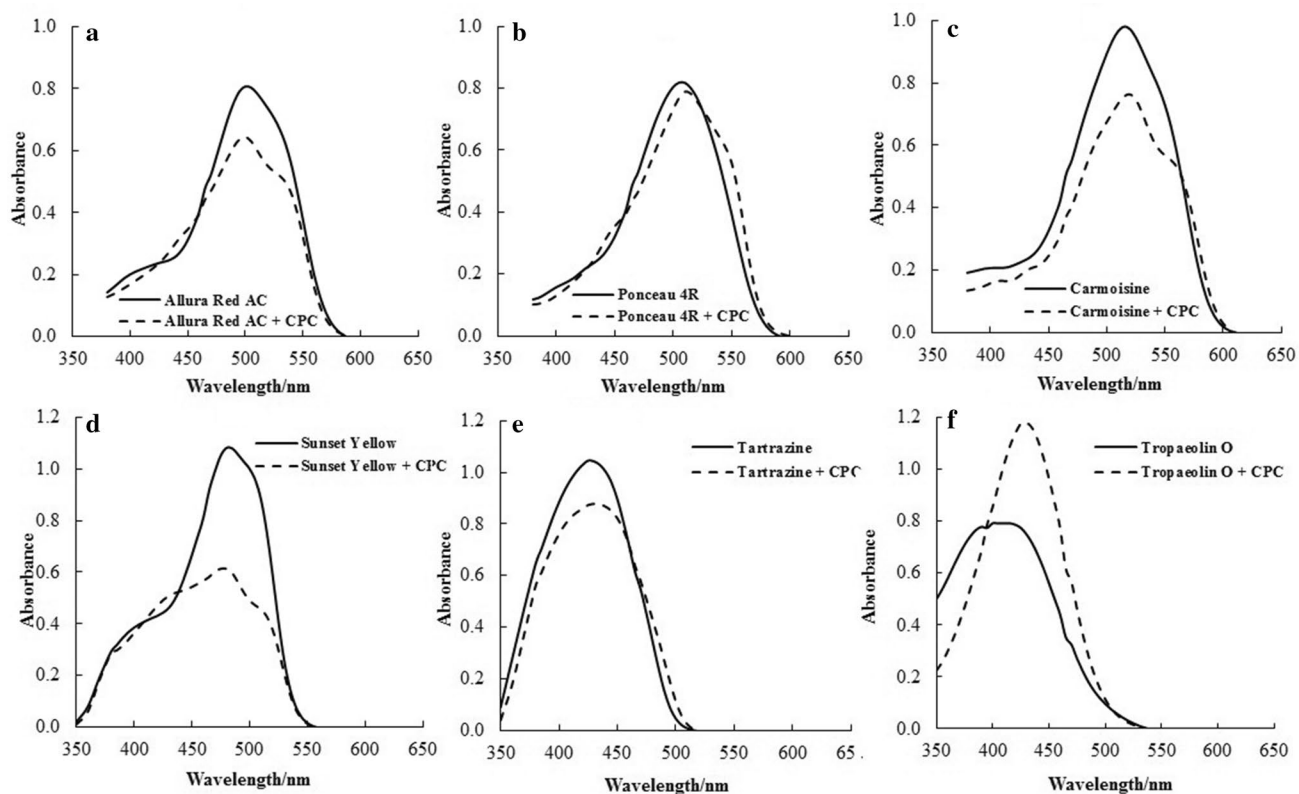
Fig. 4 Electronic absorption spectra of azo dyes and its ionic pairs with CPC in aqueous solutions; $C_{Dye} = 5 \times 10^{-5}$ mol dm^{-3}; $C_{CPC} = 1 \times 10^{-4}$ mol dm^{-3}; $l = 1$ cm
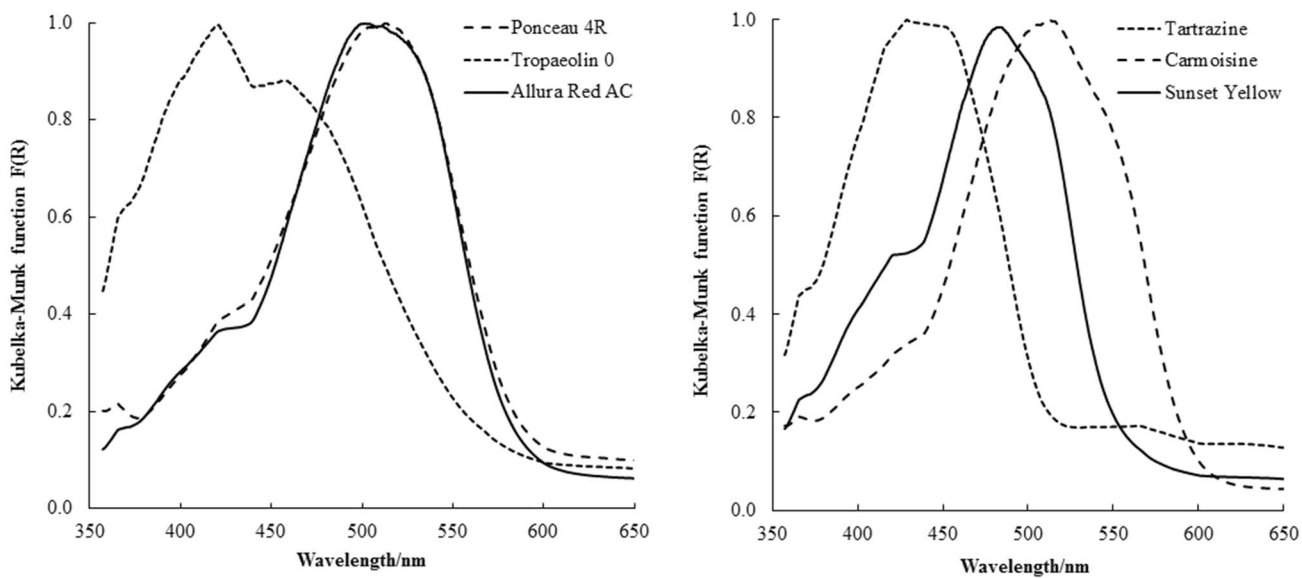
Fig. 5 Normalized diffuse reflectance spectra of azo dyes attached to the CPC-modified silica surface

aqueous solutions. In a wide range of pH food azo dyes exist in dissociated by sulfo-groups forms [27, 28]. The stoichiometry of ion pairs is determined by the number of dissociated sulfo groups of the dye [29, 30].

Attaching the azo dye to the modified silica surface can be clearly seen from the corresponding diffuse reflectance spectra (Fig. 5). As can be seen from Fig. 5, the maxima of bands in the diffuse reflection spectra coincide with the absorption spectra of ion pairs (Fig. 4) and are 480, 430, 510, 520, 500, and 430 nm for sunset yellow, tartrazine, ponceau 4R, carmoisine, allura red AC, and tropaeolin O, respectively.

Table 1 The optimal conditions for the sorption of azo dyes by modified silica

Dye	pH	Dosage/g/25 cm^3	t/min	Recovery/%
Allura red AC	2.5	0.3	15	96 ± 1
Ponceau 4R	2.0	0.3	15	95 ± 1
Carmoisine	2.0	0.3	15	97 ± 2
Sunset yellow	2.0	0.4	15	96 ± 1
Tartrazine	1.5	0.4	30	98 ± 1
Tropaeolin O	1.5	0.4	60	93 ± 2

Adsorption of azo dyes by CPC-modified silica

To minimize the time and maximize the information on the optimal conditions for the sorption of azo dyes, the experiment was planned via method of Latin squares (see "Experimental"). Data on optimal conditions are summarized in Table 1.

It can be seen from Table 1 that the sorption of dyes is practically quantitative and is observed at a pH of 1.5–2.5. According to our previous studies [27, 28], in acidic medium (pH 1.5–2.5) the studied azo dyes are in the ionic form with dissociated sulfo groups. A high recovery in a short time suggests chemisorption. The possibility of the formation of ion pairs between existing forms of azo dyes and CPC cations was discussed above. In the case of tropaeolin O, sorption equilibrium occurs within 60 min and the recovery is somewhat lower. This is probably due to the tautomerization of tropaeolin O, which precedes the formation of ion pairs with cetylpyridinium cations.

To determine the adsorption mechanism the isotherms were constructed at 293 K and 315 K (Fig. 6). As can be seen from Fig. 6 the adsorption isotherms are H-type with the exception of the tropaeolin O adsorption isotherm, which is L-type according to the Giles's classification [31]. This indicates a high affinity of the adsorbates to the surface and confirms the formation of ion pairs of azo dyes anions with
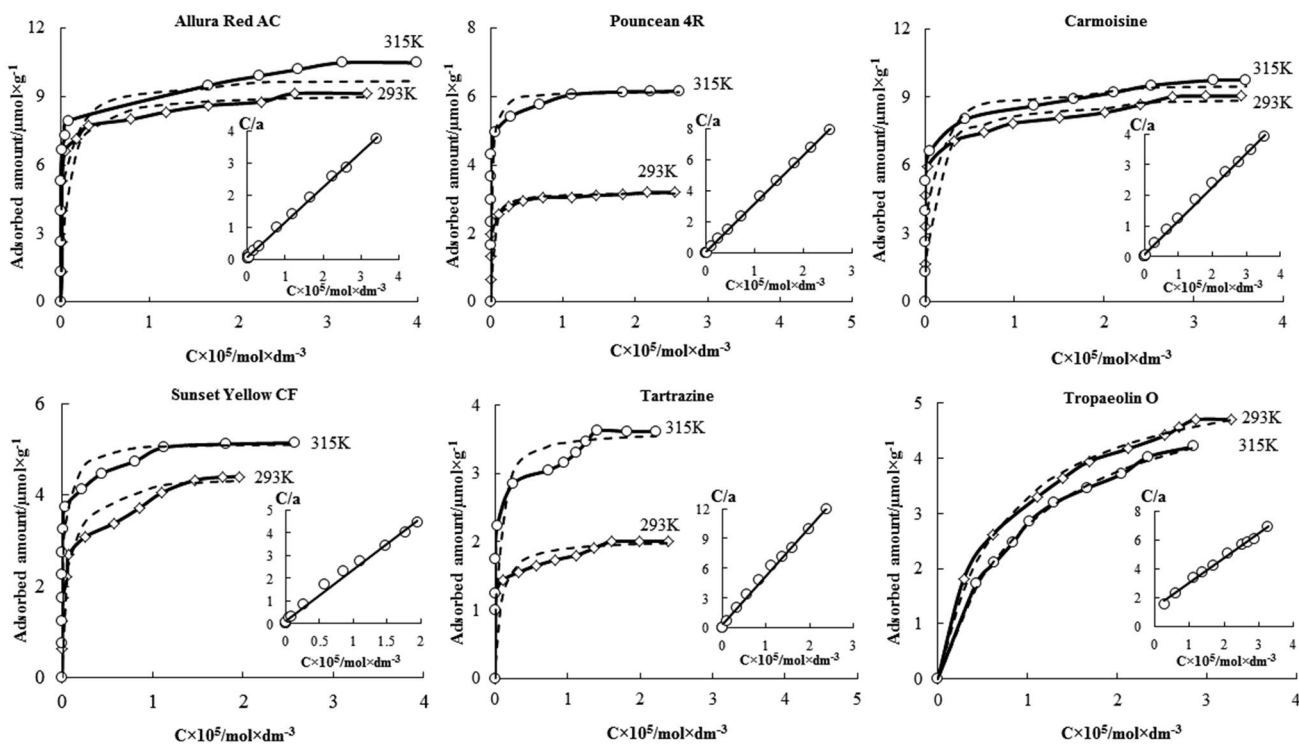
Fig. 6 Experimental adsorption isotherms of azo dyes at 293 K and 315 K (the isotherms calculated by the Langmuir equation are plotted with a dashed line). Inserts—adsorption isotherms at 293 K linearized in the coordinates of the Langmuir equation

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Table 2 Adsorption and thermodynamic properties of the studied systems at 293 K and 315 K

Dye	T/K	a_∞/µmol/g	$K \times 10^{-6}$	ΔG^0/kJ/mol	ΔH^0/kJ/mol	ΔS^0/J/mol K
Allura red AC	293	9.15	1.41	−34.50	27.00	209.88
	315	9.27	3.06	−36.44		
Ponceau 4R	293	3.21	3.55	−36.74	21.43	198.53
	315	6.17	6.91	−38.45		
Carmoisine	293	9.05	1.21	−34.11	12.00	157.39
	315	9.32	1.83	−35.13		
Sunset yellow	293	4.44	1.73	−34.99	42.34	263.90
	315	5.14	5.82	−37.92		
Tartrazine	293	2.04	1.39	−34.46	12.74	161.12
	315	3.62	2.01	−35.41		
Tropaeolin O	293	6.94	0.22	−29.92	−12.23	60.41
	315	5.77	0.15	−31.52		

CPC cations attached to the silica surface. In the case of tropaeolin adsorption, the change in the mechanism is probably due to the tautomerization of the dye. It was found that Langmuir adsorption model fit closely with these experimental data, as indicated by the determination coefficient R^2 (0.95–0.98). In addition, Table 2 summarizes the characteristics of the azo dyes adsorption by modified silica, such as maximum adsorption capacity (a_∞), the constant of adsorption equilibrium (K), and the corresponding thermodynamic properties.

As can be seen from Table 2, the standard Gibbs energy takes on negative values. The positive values of ΔH^0 indicate the endothermic adsorption process. The exception is tropaeolin O, its adsorption is an exothermic process.

When studying the desorption of azo dyes from the surface of a modified silica, it is established that solutions of mineral acids practically do not elute the dyes. When organic solvents are used, the degree of desorption does not exceed 30%, and the most effective eluant is an 1.0×10^{-3} mol dm^{-3} solution of sodium dodecylsulfate in 0.1 mol dm^{-3} solution of NH$_3$·H$_2$O. One can assume that dodecylsulfate anions replace the azo dye anions and form new ion pairs (cetylpyridinium cation–dodecylsulfate anion) on the silica surface.

Conclusion

A modified silica for the effective concentration of azo dyes from dilute aqueous solutions was proposed. The morphology of the modified silica was studied by SEM and it was shown that the CPC-modified silica surface has a 3D framework structure, which is beneficial to obtain high sorption efficiency. The fixation of cetylpyridinium cations on the silica surface was confirmed by the diffuse reflectance infrared Fourier-transform spectroscopy method. Using the mathematical planning of the experiment via method of Latin squares the conditions for the adsorption of dyes

have been optimized. It was found that the maximum dye adsorption occurs at pH 1.5–2.5 where azo dyes are in the ionic form with dissociated sulfo groups. The formation of ion pairs of azo dye anions and cetylpyridinium cations in solution was proved by spectrophotometry and on the surface by diffuse reflection spectroscopy. It is shown that with the exception of tropaeolin O adsorption isotherms are of the H-type. The adsorption isotherms of the azo dyes studied are well described by the Langmuir equation. The maximum adsorption capacity of the modified silica with respect to azo dyes ranges from 2 to 9 µmol/g and depends on the geometric dimensions and spatial configuration of the dyes molecules. Thermodynamic studies have made it possible to establish the endothermic nature of adsorption. It is shown that the most effective eluent is 1.0×10^{-3} mol dm^{-3} solution of sodium dodecylsulfate in 0.1 mol dm^{-3} solution of NH$_3$·H$_2$O, and desorption itself occurs due to the destruction of ion pairs of dye anions with cetylpyridinium cations fixed on the surface.

Experimental

Reagents

Synthetic food azo dyes used in the work, tartrazine (E102), sunset yellow FCF (E110), allura red AC (E129), ponceau 4R (E124), and carmoisine (E122) were obtained from Rocha DyeChem (India) and tropaeolin O was received from UkrLaborReactive (Ukraine). The dyes were purified by recrystallization from methanol. Distilled water was used to prepare the solutions. The acidity of the medium was achieved by the use of sulfuric acid, sodium hydroxide, and acetate buffer solution. All other reagents were analytical reagent grade. As an adsorbent, the L5/40 silica (Chemapol, Czech Republic) modified with cetylpyridinium chloride (Merck, Germany) was used.

Apparatus

The absorption spectra of the solutions of the investigated dyes were recorded on a SF-56 spectrophotometer (OKB "Spectr", Russia) in quartz cells. The diffuse reflectance spectra were measured with a Specord M40 spectrophotometer (Carl Zeiss, Germany). Diffuse reflectance infrared Fourier-transform spectra (DRIFT-spectra) were obtained on an infrared spectrometer Frontier FT-IR Spectrometer (Perkin-Elmer, USA) and processed according to Kubelka–Munk theory [25]. Microphotographs of the sorbent surface were obtained on a scanning electron microscope VEGA 3SBH (Tescan, Czech Republic). The pH of the solutions was measured on an I-160 potentiometer (ZIP, Belarus) with combined glass electrode ESKL-08M (ZIP, Belarus) calibrated using standard buffer solutions. Adsorption was studied in a static mode and mixing was performed with a shaker AVU-1 (Kiev experimental plant of medical products, Ukraine).

Preparation of CPC modified silica

Weigh 10 g silica on the analytical scales and place in a conical flask. 100 cm^3 solution of cetylpyridinium chloride with a concentration of 1×10^{-3} mol dm^{-3} is added, capped, and mixed with a shaker for 40 min. After that, the sorbent is filtered, washed with distilled water, and dried.

Adsorption process study

To optimize the conditions of sorption, the mathematical planning of the experiment using the method of Latin squares was used. Latin squares are a square table consisting of characters that are arranged in rows and columns. The number of rows and columns is the same, and each character meets one another only once. The experimental design is shown in Table 3.

Under the factors A, B, C, the conditions of dyes sorption of—A—pH (1–3), B—adsorbent dosage (0.1–0.4 g), and C—phase contact time (0–60 min) are coded. The data of the table are subjected to a simple processing, determining the sums of the results of the experiments for each factor, taking into account only those experiments in which the given level is found (ΣA_1, ΣA_2, ΣA_3, ΣA_4, ΣB_1, ΣB_2, ΣB_3, ΣB_4, ΣC_1,

Table 3 Planning an experiment using the method of Latin squares

$A_1B_1C_1$	$A_2B_1C_2$	$A_3B_1C_3$	$A_4B_1C_4$
$A_1B_2C_4$	$A_2B_2C_1$	$A_3B_2C_2$	$A_4B_2C_3$
$A_1B_3C_3$	$A_2B_3C_4$	$A_3B_3C_1$	$A_4B_3C_2$
$A_1B_4C_2$	$A_2B_4C_3$	$A_3B_4C_4$	$A_4B_4C_1$

ΣC_2, ΣC_3, ΣC_4). Optimal conditions are those for which the sum of quotients is maximal.

The method of studying sorption was as follows. The masses (0.1–0.4 g) of the modified silica are weighed on the analytical scales and placed in Erlenmeyer flasks with a volume of 100 cm^3, which contain 50 cm^3 of the dye solution with a concentration of $(1–5) \times 10^{-5}$ mol dm^{-3} and the corresponding pH value. The flasks are closed with stoppers and placed on a shaker for stirring for a preset time (0–60 min). After the sorption is completed, the sorbent is separated from the equilibrium solution by filtration. The first portions of the filtrate (~ 2 cm^3) are discarded and the sample of the solution is taken to determine the absorbance. The equilibrium concentration of the dye is determined spectrophotometrically.

Recovery (R) was calculated by formula:

$$R = \frac{C_0 - C_e}{C_0} \times 100\%, \tag{1}$$

where C_0—initial concentration of the dye in the solution, mol dm^{-3}; C_e—the equilibrium concentration of the dye in the solution, mol dm^{-3}. Equilibrium experiments were carried out under optimal conditions for each dye. The adsorbed amount (a) of each dye at equilibrium was calculated by the following equation:

$$a = \frac{\left(C_0 - C_e\right) \times V}{m}, \tag{2}$$

where C_0 and C_e—initial concentration and equilibrium concentration of the dye, mol dm^{-3}; V—the volume of the dye solution, dm^3; m—adsorbent dosage, g. Each experiment was repeated at least three times, and the deviation between parallel experiments did not exceed 4%.

Thermodynamic parameters, such as standard Gibbs energy (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0) are necessary for estimating the influence of temperature on the adsorption process and allow estimating the energy changes of the system. These parameters are calculated from the equation:

$$\Delta G^0 = -RT \ln K, \tag{3}$$

where R is the universal gas constant (8.314 J mol^{-1} K^{-1}), T—the temperature (K), and K—the equilibrium constant of adsorption, taking into account the adsorption of solvent molecules.

The Langmuir equation:

$$a = a_\infty \frac{\beta C}{1 + \beta C}, \tag{4}$$

where a is the adsorbed amount of each dye at equilibrium, a_∞ is the maximum adsorption capacity, β—adsorption coefficient, depending on the adsorption energy and temperature,

C is the equilibrium concentration of the dye in the solution, mol dm^{-3}. The constant of the adsorption equilibrium K, taking into account the adsorption of water molecules, is calculated by the formula.

$$K = \beta \times \gamma \times \frac{S_0}{S},$$ (5)

where γ—number of moles of solvent per 1 kg of its mass (for water $\gamma = 55.5$); s_0 and s—areas that occupy the water and dye molecules, respectively, on the adsorbent surface.

To determine the adsorption coefficient (β), the equation of the Langmuir adsorption is linearized in the coordinates $\frac{C}{a} = f(C)$.

The values of the dye areas required for calculations, adsorption equilibrium constant of (K) were calculated in the Marvin 5.9.1 program. For tartrazine, sunset yellow FCF, allura red AC, ponceau, carmoisine, and tropaeolin O, they are respectively equal to 5.4, 4.9, 6.2, 6.5 6.3, 3.3 nm^2, and the water molecule area is 0.096 nm^2.

The change in the enthalpy of the adsorption process (the thermal effect of the adsorption process) is calculated from equation

$$\Delta H^0 = -\frac{R \times \ln \frac{K_{T_2}}{K_{T_1}}}{\left(\frac{1}{T_2} - \frac{1}{T_1}\right)},$$ (6)

where R is the universal gas constant (8.314 J mol^{-1} K^{-1}), T—the temperature (K); K_{T_1} and K_{T_2}—the adsorption equilibrium constants at temperatures T_1 and T_2, respectively. The change in the entropy of the adsorption process is calculated from the equation:

$$\Delta S^0 = \frac{\Delta H^0 - \Delta G^0}{T},$$ (7)

where ΔH^0—the change in the enthalpy of the adsorption process, kJ/mol; ΔG^0—the change in the free energy of the adsorption process, kJ/mol.

Desorption procedure

First of all, samples of modified silica with the maximum possible content of the corresponding dye on the surface were obtained. As eluents, solutions of mineral acids (HCl, H$_2$SO$_4$, and HNO$_3$ with concentration 0.1–1.0 mol dm^{-3}) and alkalis (NaOH and NH$_3$·H$_2$O with concentration 0.1–1.0 mol dm^{-3}), ethanol, acetone, anionic surfactant sodium dodecylsulfate with concentration 1.0×10^{-4}–1.0×10^{-2} mol dm^{-3} and mixtures thereof were tested.

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