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Adsorption Properties of Silochrom Chemically Modified with Nickel Acetylacetonate

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Abstract. One of the areas of development of gas chromatography is the creation of new chromatographic materials that have improved sorption and analytical characteristics. In this work, for the first time, a new sorbent based on Silochrom C-120 modified with nickel acetylacetonate was studied using a complex of physico-chemical methods. It has been established that due to chemical modification of silica gel surface with nickel acetylacetonate the surface area of the specific surface decreases from 112 to 98 m²/g and surface acidity diminishes by 1.2 pH units. Using the thermogravimetric analysis it has been revealed that the obtained sorbent can be used in gas chromatography up to 290°C. Gas chromatography method was used to investigate the adsorption properties of the modified materials. According to the retention data of adsorbates: n-alkanes (C₆-C₉), benzene, ethanol, nitropropane and butanone-2 the differential molar adsorption energy $\bar{q}_{\text{dif}, 1}$, Henry adsorption constants $K_{1,C}$, the differential molar entropy $\Delta\bar{S}_{S1}$ and $\Delta\bar{q}_{\text{dif}, 1}^{\text{(special)}}$ of adsorbates in dispersion and specific interactions were calculated. The influence of the modifying additive on the changings in the thermodynamic retention characteristics of all sorbates because of the manifestation of specific sorbate-sorbent interactions has been shown. The highest values of the thermodynamic parameters were indicative for sorbates forming hydrogen bonds and capable of donor-acceptor interaction.

INTRODUCTION

In the practice of gas chromatography the modification of adsorbents is widely used. This allows not only to avoid a number of their shortcomings, but also to change the chemistry of the surface of adsorbents directly, therefore to expand the range of solved analytical problems.

The evolution of the problems of chemical analysis led to the complication of the structure of the complexes and most of the modern work is devoted to the use of chiral, calixarenes, nematic liquid crystals and fullerenes with an internal metal ion as adsorption layers and binary phase components in gas chromatography [1-3]. A large number of known intracomplex compounds provides for varying of properties of sorbents and combined stationary phases over a wide range due to a change in the nature of the ligands, metals, and characteristics of the carrier surface. In this case, the use of such materials as sorbents is in to a certain extent and their usage for gas chromatography is limited by their solubility in stationary phases, thermal stability and bond strength with surface of the original silica gel.

Liquid crystals are perspective modifiers; their molecules form highly ordered structures on solid substrates, similar to the mesomorphic state. The formation of such structures is confirmed by data scanning tunneling microscopy [4, 5]. Adsorbents modified with liquid crystals are able to exhibit selective properties with respect to isomers of organic substances [6, 7]. Such sorbents cannot be used to separate mixtures of polyaromatic hydrocarbons and other high boiling organic compounds due to their thermal instability.

In the work [8], the adsorption properties of silica modified with stable Ag and Ni nanoparticles obtained in inverse micelles were investigated by gas chromatography. It is shown that the modification of silica gel makes it possible to directly change the adsorption properties of its surface, with the greatest contribution to the energy of adsorption interactions is made by molecules capable of manifesting specific interactions.

Acetylacetonate and metal chelates on their basis are used as catalysts for organic reactions, for the production of films and metal-containing coatings, as well as in the practice of gas chromatography for the purification, separation and analysis of metal traces, complex organic mixtures of various compositions, sorption concentration of microimpurities from atmospheric air and liquid media [9-11].

Complexes based on acetylacetonate after their chemical fixation on the surface of the sorbent are

advantageously distinguished by considerable thermal stability, which allows them to be used for gas chromatographic separation of a wide range of substances.

The purpose of this work is to obtain and study the texture, acid-base and chromatographic properties of Silochrom C-120 chemically modified with nickel acetylacetonate.

EXPERIMENTAL PART

Materials

As a silica matrix for the synthesis of gas chromatographic sorbents an amorphous porous silicagel Silochrom C-120 (fraction 0.20-0.355 mm, made in Russia, technical conditions 6-09-17-48-82) was chosen. Silochrom C-120 has high thermal, chemical and mechanical resistance; it is a product of polycondensation of silicic acids, the polarity of which depends on the presence of silanol and siloxane groups that exhibit the properties of active surface centers (acid-base properties) [12]. The limitation of the use of untreated silica in gas chromatographic analysis is asymmetric peaks, high retention of organic compounds, and the inability to separate mixtures of analytes. In order to improve chromatographic characteristics, to change the polarity of sorbents and to synthesize selective materials for various classes of organic substances a method of chemical modification was chosen.

As a surface modifier, nickel acetylacetonate was used. The use of chelates of β -dicarbonyl compounds makes it possible to increase the number of active surface centers, which are reflected in the parameters of chromatographic retention due to the appearance of specific intermolecular interactions with sorbate molecules. Activation of the silica gel surface was carried out by boiling in dilute HNO_3 (chemically pure, GOST RF 4461-77, made in Russia). For this, 10 g of silica gel was added to 200 ml of dilute 10% HNO_3 and heated for 2 hours. After this, the hydroxylated sorbents were washed with several portions of bidistilled water until the acid reaction ceased and the samples were dried for 6 hours in an oven at 200°C . To 10 g of hydroxylated silica gel was added methylene chloride (chemically pure, Russia). The mixture was stirred on a magnetic stirrer for 30 minutes. Then, with constant stirring, thionyl chloride (chemically pure, Russia) was added dropwise 20 ml in volume, at room temperature. To carry out the chemical reaction, the sodium salt of acetylacetonate was used on the surface of the sorbent, which was dissolved on a surface of chlorinated SiO_2 by dissolution in dimethylformamide (chemically pure, GOST RF 20289-74, made in Russia). The ligand-modified carriers were treated with an alcoholic solution of nickel, cobalt and copper chlorides. The stages of synthesis and the structure of the resulting chelate-containing materials are shown in the scheme (Fig. 1) [13].

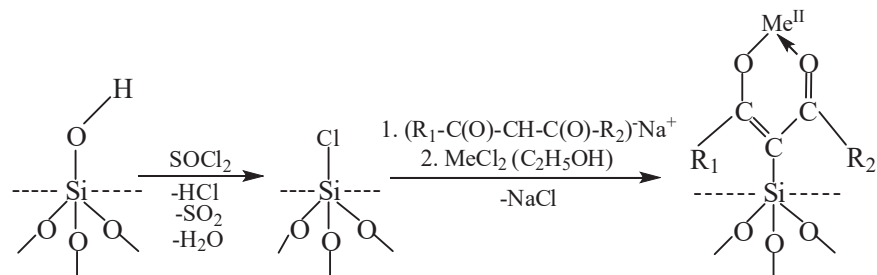


FIGURE 1. The synthesis scheme and the structure of the obtained sorbents with grafted chelates of β -dicarbonyl compounds.

Research Methods

The parameters of the porous structure and the surface area of silica gels were evaluated by the BET method using the low-temperature adsorption isotherm of N_2 (TriStar II 3020, USA). To study the composition and concentration of the grafted layer on the Silochrom S-120 surface the elemental analysis was performed on an automated HCNS-element analyzer Euro EA-3000 (Eurovector, Italy) with Callidus 5.1 software. A sample (1 mg) was burned at a temperature of $1000\text{--}1100^\circ\text{C}$ in an oxidizing medium in an atmosphere of helium. Determination of the mass fraction of carbon, nitrogen, hydrogen (HCNS-analysis) is based on chromatographic determination of the amounts of CO_2 , N_2 , SO_2 and H_2O formed as a result of the sequence of catalytic reactions conducted for complete combustion of the substance and removal of interfering products.

The amount of grafted compound N (mmol/g) was calculated under the condition that a grafted monolayer [12]:

$$N = P_C / 1.2n_C,$$

where P_C is the carbon content,%; n_C - number of carbon atoms in the grafted group.

The calculation of the thickness of the grafted layer for porous silica was carried out from the data on the drop in pore volume as a result of chemical modification:

$$h = V_{SiO_2} - V_{modif} / S_{SiO_2}.$$

To calculate the surface concentration of grafted groups, the content of the grafted complex was referred to the unit surface:

$$N (\mu\text{mol}/\text{m}^2) = 106 \times P_C / (1200n_C - P_C \times M) \times S_{SiO_2}.$$

IR spectra were recorded on a Fourier spectrometer «Nicolet 6700» (USA) in the region of 400-4000 cm^{-1} . The Raman spectra were obtained on a «Nicolet NXR 9650» (USA) Raman spectrometer in the region 100-2000 cm^{-1} . The thermal stability of the chelates was determined by the results of thermogravimetric analysis (Netzsch STA 449C, Germany). The thermal analysis of the obtained samples was carried out in an air atmosphere in the temperature range 25-600°C with a heating rate of 10°C/min; crucibles were made from Al_2O_3 . To study the morphology of the surface of silica gels, a scanning electron microscope Hitachi TM-3000 (Japan) was used under conditions of an accelerating voltage of 15 kV. Acid-base properties were studied using the pH-metry method (pH meter Multitest IPL-111, Russia). 20 ml of bidistilled water was introduced into the potentiometric cell and after stabilization of the potential of the glass combined electrode ESK-10601 (pH_0) 0.2 g of the sample were added. Parameters that characterized the acid-base state of the surface of the sorbents under study were chosen pH values of the solution after 5 to 15 seconds of contact between the sample and water. This value gives information on the air-dry state of the surface, as well as on the strength of the primary Lewis acidic or basic centers on the surface. The individuality of the obtained kinetic curves of pH changes of suspensions in time reflects the acid-ground state of the surface, both along the curves, and at extreme pH points or at the moment of equilibrium. The time of stabilization of the pH of the solution is related to the structural transformations of the sample and the change in the acidity of the surface of the solid.

The amount of grafted compound, the surface concentration of the grafted groups, the thickness of the grafted layer were determined from the data on the carbon content in the samples, the change in the specific surface area of the adsorbents before and after chemical modification.

The adsorption of various test compounds (limit and aromatic hydrocarbons, ketones, alcohols, nitro compounds) was studied by gas chromatography on the obtained sorbents. The studies were carried out on a Chrom 5 gas chromatograph with a flame ionization detector in isothermal mode. Glass columns 1.2 m long and 3 mm inside diameter were used. The consumption of carrier gas (helium) is 30 cm^3/min .

The main experimental values were the specific retention volumes per unit surface of the adsorbent $V_{g,1}$, which for small sample volumes are Henry's adsorption constants $K_{1,C}$ (cm^3/m^2). The thermodynamic characteristics of adsorption $\bar{q}_{dif,1}$ and calculated from the linear dependence [14]:

$$\ln K_{1,C} = \frac{\bar{q}_{dif,1}}{RT} + \frac{\Delta S_{1,C}^0}{R} + 1,$$

where $\bar{q}_{dif,1} = -\bar{U}$ - differential molar heat of adsorption,

$$\Delta S_{1,C}^0 = S_1^0 - S_{g,C}^0 - \text{change in standard differential molar entropy.}$$

For all test compounds, the error in determining $V_{g,1}$, $\bar{q}_{dif,1}$ and $\Delta S_{1,C}^0$ on the samples studied did not exceed 10%.

RESULTS AND DISCUSSION

Adsorption processes involving chromatographic adsorbents depends not only on the chemical nature of the material, but also on the texture properties of the surface. The porous structure of the investigated materials based on Silochrom C-120 was studied from the experimental data obtained on static low-temperature adsorption of nitrogen. The corresponding isotherm, shown in Figure 2, differs by the presence of a hysteresis loop located in the region of relative pressure $p/p^\circ = 0.9-1.0$, the branches of which are vertical and parallel to each other, which characterizes porous adsorbents. The observed hysteresis loop is of type H1 according to the IUPAC classification, which describes ensembles of rigidly connected globules, uniformly packed and of equal size.

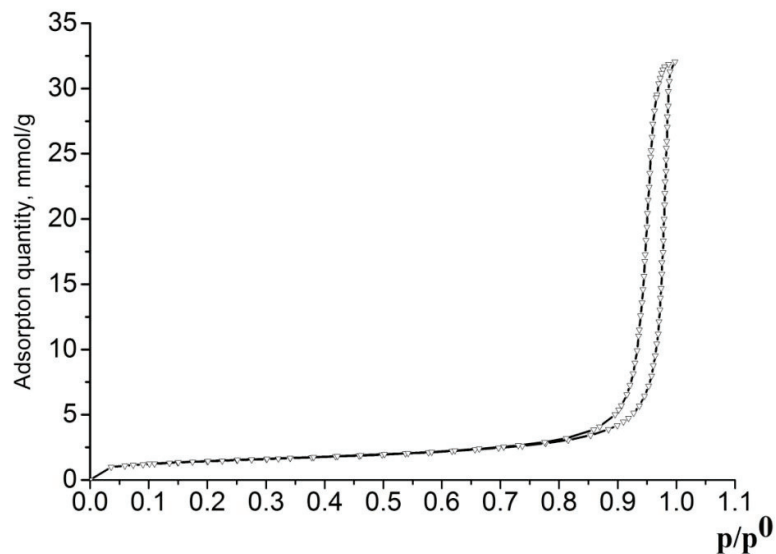


FIGURE 2. Isotherm of static low-temperature nitrogen adsorption at 77 K on Silochrom C-120: N - adsorbed amount of nitrogen (mmol) per 1 g of adsorbent

The pore distribution curve (Fig. 3) shows that Silochrom C-120 is characterized by mesoporosity with a wide distribution of pores in the range of 10-60 nm. With a maximum volume of $0.075 \text{ cm}^3/\text{g} \times \text{nm}$, the pore diameter is 40 nm.

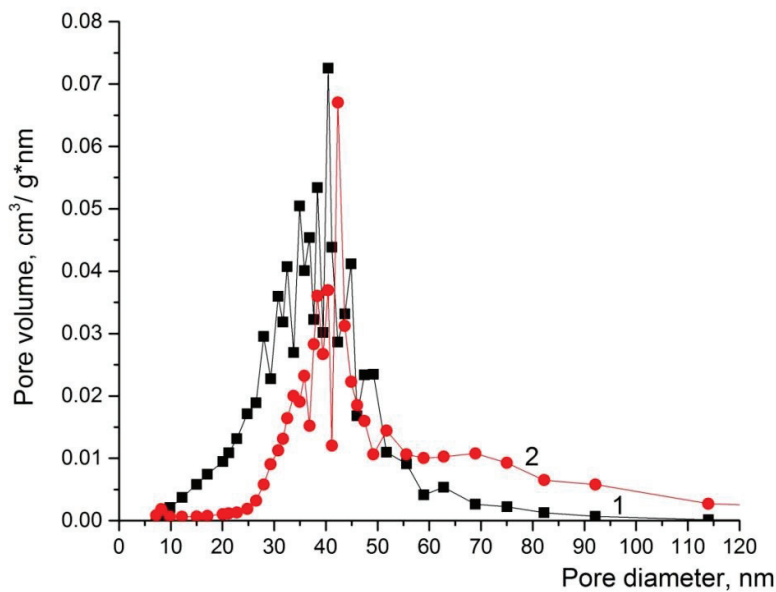


FIGURE 3. Differential curve of pore size distribution for Silochrom C-120 before (1) and after (2) modification

Chemical modification of the initial silica with metal chelates leads to a change in the geometry of the surface, as evidenced by the results of the study of the specific surface area (S , m^2/g) and porosity of the adsorbents presented in Table 1.

According to the calculation of the Henry adsorption constants and the BET constant characterizing the adsorbate-adsorbent interaction force, the BET model describes well the adsorption of nitrogen on the adsorbents under study, as evidenced by the rather high numerical values of TG and BET, as well as the correlation coefficients R. Close to unity ($R = 0.999$) for the studied chelate-containing adsorbents.

TABLE 1. Textural characteristics of the adsorbents under study.

Adsorbent	$S, \text{ m}^2/\text{g}$ (relative error $\Delta \pm 10\%$)	Total pore volume, cm^3/g	Average pore size, nm	BET constant	Henry adsorption constant
Silochrom C-120	112	1.08	38	119	22
Silochrom C-120 + nickel acetylacetonate	98	0.98	36	116	19

For porous silica, the thickness of the grafted layer is determined by the results of a decrease in pore volume as a result of chemical modification, which corresponds to the volume of the grafted layer. According to the results of the elemental analysis, the amount of grafted layer of acetylacetonate is 0.50 mmol /g, the surface density of grafted chelates is 1.60 groups / nm^2 (2.66 $\mu\text{mol} / \text{m}^2$), the volume and thickness of the grafted layer are $1.77 \times 10^{-3} \text{ cm}^3/\text{g}$ and 0.089 nm, respectively.

The results of thermal analysis indicate the stability of nickel acetylacetonate up to 300°C, which allows the use of a sorbent to separate various classes of organic substances in gas chromatography.

The acid-base state of the surface of gas chromatography sorbents based on Silochrom C-120 with grafted layers of nickel acetylacetonate was studied by pH-metry [15]. The decrease in pH values of suspensions in the first seconds indicates the presence of Lewis acidic centers on the surface of the initial and modified sorbents, on which water is sorbed by the acid mechanism. For Silochrom C-120, the main component of which is SiO_2 , the adsorption-desorption equilibrium is achieved in 500 s and the steady-state pH indicates a slightly acidic state of the surface (Fig. 4). Chemical modification of Silochrom C-120 with nickel chelate leads to a general decrease in the acidity of the surface. The difference with the starting silica gel is 1.2 pH units.

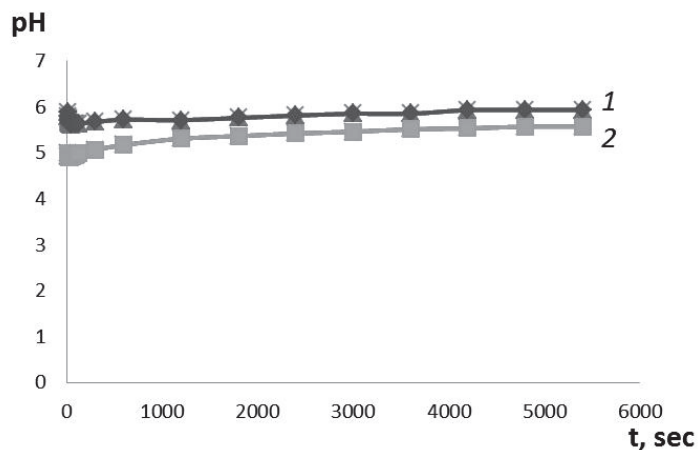


FIGURE 4. Kinetic curves of the pH of aqueous suspensions in time for:
1 - Silochrom C-120; 2 - Silochrom C-120 + nickel acetylacetonate

In the study of thermodynamics of adsorption of organic substances on the investigated materials by gas chromatography at high column temperatures, small volumes of samples of the adsorption isotherm obeys Henry's law constant adsorption equilibrium $K_{1,C}$. To evaluate the thermodynamic parameters of adsorption, substances capable of manifesting various types of intermolecular interactions were chosen as test compounds: alkanes - dispersion, butanone-2 and nitropropane - proton-acceptor and orientation, benzene - π - π interaction, ethanol-orientation and formation hydrogen bonds.

According to the retention times of sorbates at different temperatures, differential molar changes in the internal adsorption energy $-\Delta\bar{U}$, equal to the differential heat of adsorption $\bar{q}_{\text{dif}, 1}$ at a constant volume, were calculated. For specifically sorbed substances, the contribution of the energy of the specific interaction, $\Delta_{\text{qdif}, 1}$

(special), to the total adsorption energy, which was estimated from the difference $\bar{q}_{\text{dif}, 1}$ sorbate and non-specifically sorbing normal alkane with the same polarizability α was also determined:

$$\Delta \bar{q}_{\text{dif}, 1(\text{spec})} = \bar{q}_{\text{dif}, 1(\text{sorbat})} - \bar{q}_{\text{dif}, 1(\text{n-alkanes})}.$$

Table 2 shows data on the Henry adsorption constants at 150°C on adsorbents based on Silochrom C-120 with a chemically grafted layer of nickel acetylacetonate.

According to the obtained results, the values of $K_{1,C}$ n-alkanes (C₆-C₉) adsorbed due to dispersion interactions on the initial and modified silica are significantly different, while the Henry's adsorption constant ($K_{1,C}(\text{modif}) / K_{1,C}(\text{Ex})$) is different in 2-4 times (Table 2). This can indicate both an increase in the nonspecific activity of the sorption centers and an increase in their concentration. Aromatic hydrocarbons in addition to dispersion interactions with the surface of the hydroxylated Silochrom C-120 are capable of interacting with the π -electrons of the aromatic nucleus with the -OH groups. Therefore, benzene is sorbed on hydrophilic silica much more strongly than n-alkanes with the same number of carbon atoms in the molecule.

For ethanol, butanone-2 and nitropropane adsorbed on the surface of chemically modified silica due to donor-acceptor, electrostatic interactions and the formation of hydrogen bonds, Henry's constants are almost double. The causes of changes in $K_{1,C}$ in the application of complex compounds are associated with the action of energy and entropy factors. The values of $\bar{q}_{\text{dif}, 1}$ and $\Delta S_{1,C}$ are presented in Table 2.

Chemical modification of the surface of Silochrom C-120 with nickel acetylacetonate leads to an increase in the heats of adsorption of organic substances from 14 to 69 kJ / mol (Table 2). Comparing the values of the change in the entropy of one compound upon transition from the initial compound to the adsorbent with a layer of nickel acetylacetonate, it can be concluded that the mobility of adsorbate molecules has changed in the adsorbed state. The increase in $\Delta S_{1,C}$ in the transition from SiO₂ to chelate-containing materials indicates a decrease in the mobility of molecules on the surface of modified adsorbents.

TABLE 2. Thermodynamic characteristics of adsorption of test compounds on the initial Silochrome C-120 (I) and modified nickel (II) acetylacetonate: Henry's adsorption constants ($K_{1,C}$) and the values of $K_{1,C}(\text{modif}) / K_{1,C}(\text{I})$.

Compound	$K_{1,C}(\text{cm}^3/\text{m}^2)$		$K_{1,C}(\text{modif})/K_{1,C}(\text{I})$	$\bar{q}_{\text{dif}, 1}$		$\Delta S_{1,C}^0$	
	I	II		I	II	I	II
n-Hexane	0.04	0.08	2.0	14	-70	69	-193
n- Heptane	0.05	0.14	2.8	14	-42	60	-165
n- Octane	0.07	0.24	3.42	26	-67	57	-154
n- Nonan	0.10	0.41	4.1	28	-65	62	-161
Benzene	0.12	0.14	1.2	38	-117	49	-140
Ethanol	0.40	0.74	1.9	39	-108	55	-141
Butanone-2	1.09	1.85	1.7	35	-90	39	-194
Nitropropane	0.26	0.96	3.7	36	-110	38	-97

In Figure 5 it is shown the logarithm of Henry's adsorption constants as a function of the inverse temperature on the initial and chemically modified Silochrom C-120 for the compounds under study.

It should be noted that for all adsorbents there is a linear relationship between $\ln K_{1,C}$ of sorbates and $1000/T$ as well as monotonic decrease in the values of $K_{1,C}$ with increasing of temperature. In the transition from initial to modified adsorbents in the case of hexane adsorption (Fig. 5a), a sharp change in the slope of the direct dependence of $\ln K_{1,C}$ from $1000/T$ is observed, which can indicate both an increase in the nonspecific activity of the sorption centers. In the case of adsorption of benzene, an increase in the strength of π - π interactions in the initial SiO₂-SiO₂ + nickel acetylacetonate series is observed (Fig. 5b).

As a result of the adsorption of ethanol (Fig. 5c), the slope of the direct dependence of $\ln K_{1,C}$ is changed from $1000/T$, which is related to the ability of the sorbate to exhibit orientational interactions and the formation of hydrogen bonds with the grafted layer: metal atoms in the chelate ring are capable of accepting electron pairs Oxygen atoms of the sorbate, and oxygen atoms in the ethoxy group - to form hydrogen bonds with alcohols. In the case of adsorption of butanone-2, a sharp change in the slope of the line $\ln K_{1,C}$ from $1000/T$ (Fig. 5d) is observed, due to strong donor-acceptor interaction with the grafted layer. Nitropropane is characterized by an increase in the values of Henry's constants as a result of chemical modification due to orientational and proton-acceptor interactions with the sorbent surface.

The obtained materials are stored at room temperature and can be used in gas chromatographic analysis without changing the properties for a long time and in the temperature range up to 290 °C.

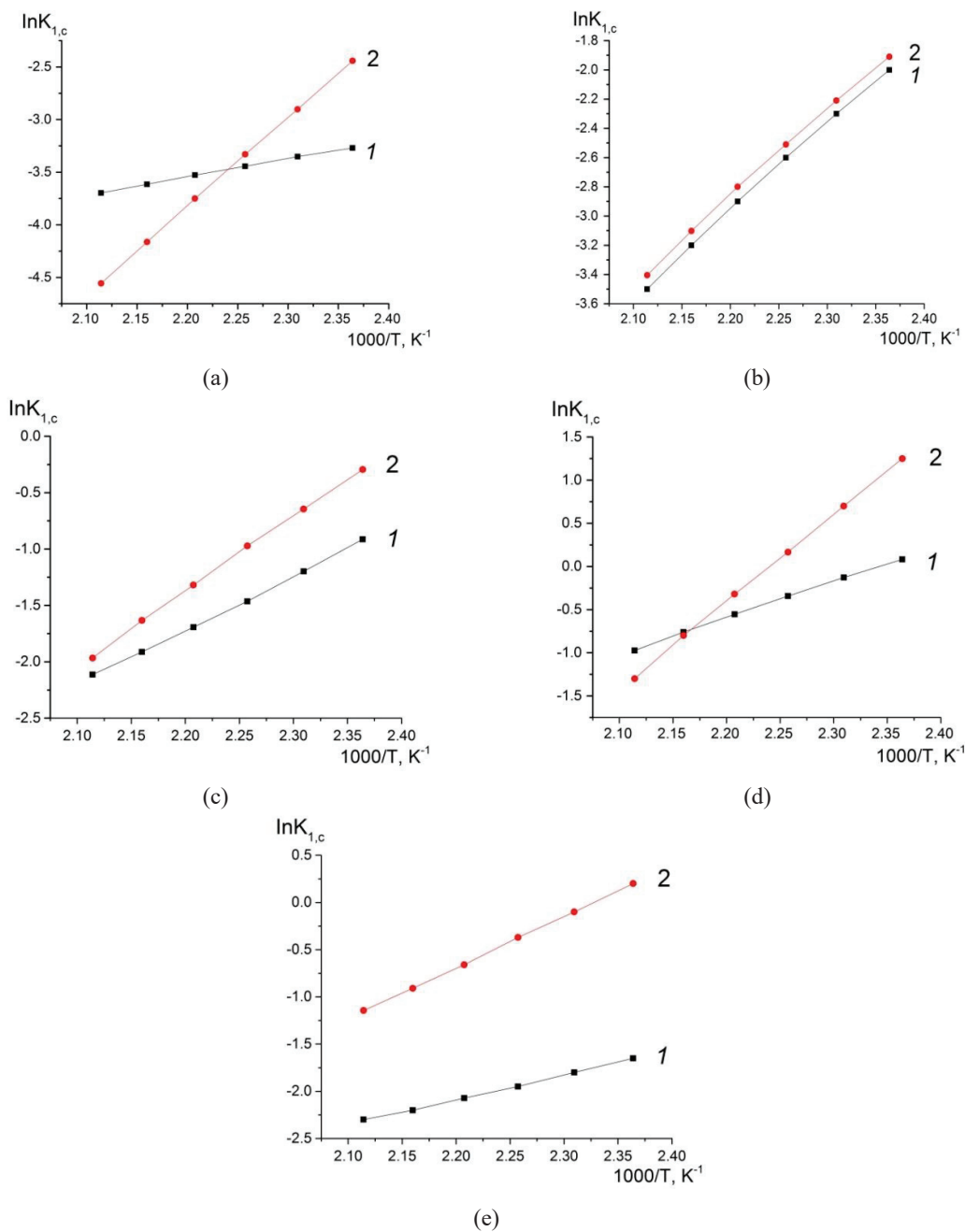


FIGURE 5. Dependences of the logarithm of the Henry constant of adsorption of hexane (a), benzene (b), ethanol (c), butanone-2 (d) and nitropropane (e) on the reverse temperature on the original Silochrom C-120 (1) and chemically modified nickel acetylacetonate (2)

CONCLUSION

In this paper, for the chelate-containing adsorbents based on SiO_2 , the specific surface area, porosity, concentration of the grafted groups is determined. It is shown that when the SiO_2 is modified, the specific surface area decreases from 112 to 98 m^2/g . Chemical modification of Silochrom C-120 leads to a decrease in the acidity of the surface of 1.2 units of pH. Based on the results of the thermal analysis, the thermal stability of the chelates was evaluated, the maximum permissible temperature of the synthesized materials is 300°C. In the

result of chemical modification of Silochrom with nickel acetylacetonate, a significant increase in retention parameters and thermodynamic characteristics of adsorption is observed in comparison with SiO₂ and sorbents with adsorption layers by metals chelates [16]. For the studied sorbent an increase in the Henry adsorption constants is observed for alkanes due to dispersion interactions. The values of K_{1,C} increase by 2-4 times. There is an increase in the Henry adsorption constants for alkanes prone to dispersion interactions. The values of K_{1,C} increase by 2-4 times. On the modified sorbent in contrast to the original Silochrom C-120 the adsorption constant for nitropropane increases by 3.7 times. This is the result of strong orientational and donor-acceptor interaction with nickel acetylacetonate. For ethanol and butanone-2, the Henry constant on the modified sorbent increased by a factor of 2. For all investigated sorbates there is an increase $\Delta\bar{q}_{\text{dif},1}$ and $\Delta S_{1,C}$. This is due to strong dispersion and donor-acceptor interactions with the surface of the chemically modified Silochrom SiO₂.

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