

Paweł MUSZYŃSKI¹

SORPTION OF THE SURFACTANT HYAMINE 1622 IN SOILS

SORPCJA SURFAKTANTU HIAMINY 1622 W GLEBACH

Abstract: A study was performed on the sorption of hyamine 1622, a cationic surfactant, from water solutions in 3 soils which, with relation to the character of their parent rocks, are referred to in this paper as a sandy soil, a loess soil and a loamy soil, respectively. The sorption process was executed with the static method: 2-gram weighed portions of soil were placed in polyethylene test tubes, adding 10 cm³ of solution with hyamine concentration in the range from 0.00015 to 0.006 mol · dm⁻³, and then shaken for 6 hours. The level of sorption of hyamine was determined with relation to the concentration and pH of the initial solution, to the time of soil-solution contact, and in the presence of electrolytes KCl and KNO₃ in the surfactant-electrolyte systems = 1 : 1 and 1 : 10.

The experiments showed that with increase in the concentration of the initial solution there was an increase of hyamine sorption in the soils. It was established that the affinity sequence of the studied soils with relation to hyamine was as follows: loamy soil > loess soil > sandy soil. With increase in the pH value only slight changes were observed in the sorption effectiveness in the sandy and loess soils, while in the loamy soil the sorption remained at a constant level. Kinetic tests revealed that in the sandy and loess soils the sorption proceeded the fastest during the initial 15 minutes, and extension of the time of the soil-solution contact to 720 minutes did not produce any notable changes in the level of the sorption. In the loamy soil hyamine sorption at the level of 100 % was observed irrespective of the time of contact of the solution with the soil. The sorption process proceeded in accordance with the mechanism of pseudo second-order reaction. The presence of electrolyte caused both a reduction and an increase in the sorption of hyamine in the sandy and loess soils, but the extent of the changes was related to the type of electrolyte and to the concentration of the surfactant. Electrolyte addition had no effect on hyamine sorption in the loamy soil.

Keywords: cationic surfactant, sorption, soil

Surfactants are compounds with asymmetric structure, composed on a non-polar “tail” which is frequently a long hydrocarbon chain, and a polar “head” which has an ionic or a dipole character [1]. The compounds are commonly used as components of detergents and emulsifiers. Certain surfactants are also used for the removal of organic contaminants and heavy metals from soils. Currently an increase is observed in the level of environmental pollution with those compounds, resulting from intensive economic

¹ Department of Chemistry, University of Life Sciences in Lublin, ul. Akademicka 15, 20–950 Lublin, Poland, phone: +48 81 445 65 56, email: pawel.muszynski@up.lublin.pl

growth. Surfactants penetrate into the environment with industrial and domestic sewage and with wastes of various kinds [2, 3].

Cationic surfactants are characterised by having a polar “head” with a positive charge, eg quaternary ammonium salts [4]. They are substances that have a toxic effect on soil microorganisms and an unfavourable effect on the physicochemical properties of soils. Sorption plays an important role in the fate of surfactants in soils. According to Lee et al [5], the sorption of surfactants is strongly influenced by the properties of soils, such as: organic matter content, clay content, electrolyte content and pH, as well as the molecular structure of the surfactant. In the opinion of Muherei and Junin [6], in water solution, the surfactant exist as monomers at low concentration. When surfactant concentration is above a critical value termed the *critical micelle concentration* (CMC), the surfactant molecules aggregate and form micelles. At concentrations above the CMC, the sorption of the surfactant reaches a plateau.

Hyamine is quaternary ammonium salts of cationic surfactant character [1]. Due to the antibacterial and antifungal properties this compound is used in households and in medical consulting rooms and dentists’ surgeries. After use, hyamine with the wastewater goes to the various components of the environment, including the soil, which is subject to sorption. Knowledge of the sorption is essential to estimate the bioavailability of hyamine and may be used in studies of the degradation of the surfactant. But one article [1] in the literature, there is no information about the sorption of hyamine in soils. Therefore, there is a need for experiments that would provide an explanation of the behaviour of hyamine in soils.

The objective of the study presented here was examination of the process of sorption of a cationic surfactant, hyamine 1622, in three soils with differing properties, with relation to the concentration of the surfactant, duration of the sorption process, pH of the solution, and an addition of electrolytes.

Material and methods

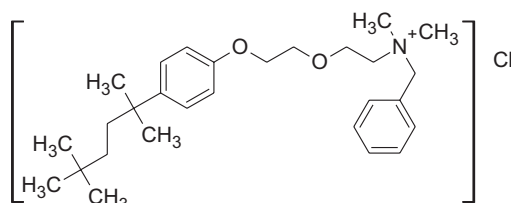
For the sorption experiments three soils were used, the following types of: a grey-brown podzolic soil developed from sand with the particle size distribution of loamy sand, a grey-brown podzolic soil developed from loess, with particle size distribution of silt, and a brown soil developed from boulder loam, with the particle size distribution of sandy clay loam (soil types according to the classification of the Polish Soil Science Society from 1989 [7]; particle size distribution according to the classification of the Polish Soil Science Society from 2008 [8]), further referred to in the paper, due to the character of their parent rocks, a sandy soil, a loess soil and a loamy soil. Samples of the soils were taken from the A_p horizon, air dried, then screened through a sieve with 2 mm mesh, and averaged. The physicochemical properties of soils are given in Table 1.

Grain size composition was determined with the Cassagrande method as modified by Proszynski, organic carbon content with the method of Tiurin, pH in 1 mol · dm⁻³ KCl – electrometrically, specific surface area with the method of water vapour adsorption, hydrolytic acidity (H_h) acc. to procedure PB-38 and sum of exchangeable cations (S) acc. to procedure PN-R-04027:1997 and sorptive capacity (T) as sum H_h and S.

Table 1
Physicochemical properties of soils

Soil	Share of soil fraction [%] with diameter [mm]				pH in 1 mol · dm ⁻³ KCl	Organic carbon content [g · kg ⁻¹]	Specific surface area [m ² · g ⁻¹]	Hydrolytic acidity (H _b) [mmol H ⁺ · kg ⁻¹]	Sum of exchangeable bases (S) [mmol(+) · kg ⁻¹]	Sorpive capacity (T)
	1.0-0.1	0.1-0.02	0.02-0.002	<0.002						
Sandy	80	14	2	4	3.9	5	8.67	35.8	0	35.8
Loess	1	63	27	9	4.3	21.3	20.05	41.7	30	71.7
Loamy	29	19	34	18	5.6	17	49.09	9.2	332.4	341.6

The surfactant used in the experiments was hyamine 1622 (molar mass = 448.1 g · mol⁻¹, CMC = 2.75 · 10⁻³ mol · dm⁻³ [9]) produced by Sigma-Aldrich (Germany). It is a cationic surfactant with the following chemical formula:



The properties of surfactants depend on the CMC, it is a concentration at which they begin to form aggregates. The form in which the surfactant is in solution (monomeric or aggregated) affects its sorption. For this reason, solutions of hyamine with concentrations below and above the CMC were studied.

The sorption experiments were performed under static conditions: 2 grams of soil were added to 10 cm³ of water solution of hyamine with required concentration (0.00015, 0.0003, 0.0005, 0.001, 0.0015, 0.002, 0.003, 0.004, 0.005 and 0.006 mol · dm⁻³) and shaken for 6 hours at room temperature. After that time the soil suspensions were centrifuged at 4000 rev. · min⁻¹ for 20 minutes to separate the liquid phase from the solid phase. Next, a part of the solution taken from above the solid phase of the soil was transferred to polyethylene test tubes and analysed for the presence of hyamine. The level of the surfactant in the solutions was determined with the technique of *high performance liquid chromatography* (HPLC) using a Hypersil Gold RP-C₁₈ column and an UV-VIS detector at $\lambda = 269$ nm. The mobile phase was a system of solvents – acetonitrile/water (95/5, v/v) in an acetate buffer with pH 7.4. Flow rate of 0.5 cm³ · min⁻¹ was applied. The amount of hyamine sorbed by the soil (q , [mol · kg⁻¹]) was calculated from the formula:

$$q = (C_0 - C_{eq}) \cdot V/m \quad (1)$$

where: C_0 and C_{eq} – initial and equivalent concentrations of surfactant [mol · dm⁻³],
 V – volume of the solution [dm³],
 m – mass of the soil [kg].

The sorption of hyamine was also conducted from solutions with pH 3, 4, 5, 6, 7, 8 and with constant concentration of the surfactant – 0.005 mol · dm⁻³. The pH of the solutions was controlled through dosage of NaOH (1 mol · dm⁻³) or HCl (1 mol · dm⁻³). The use of NaOH and HCl solutions at concentration of 1 mol · dm⁻³ meant that the initial water solutions of were excessively diluted. Whereas, pH control by means of 0.1 mol solutions NaOH and HCl would require the dosage of correspondingly larger amounts of the solutions. The effect of KCl and KNO₃ on the sorption of hyamine was studied at the surfactant/electrolyte molar ratios of 1 : 1 and 1 : 10. In the solutions with electrolytes the concentrations of hyamine were from 0.00015 to 0.006 mol · dm⁻³. The time required to reach sorptive equilibrium was determined through shaking 2 g

samples of the soils from 10 cm³ of hyamine solution with concentration of 0.006 mol · dm⁻³ during 15, 30, 60, 180, 300, 360, 480, 600 and 720 minutes.

The interpretation of results of the sorption kinetics was performed based on a pseudo second-order equation:

$$t/q_t = 1/k_2 \cdot q_{eq}^2 + t/q_{eq} \quad (2)$$

where: q_t – amount of sorbate sorbed during time t [mol · kg⁻¹],

t – time [min],

k_2 – second order rate constant [kg · mol⁻¹ · min⁻¹],

q_{eq} – amount of sorbate sorbed at equilibrium [mol · kg⁻¹].

All experiments were made in three replications.

Results

Figure 1 presents changes in the level of hyamine sorption in relation to equivalent concentration. In the loamy soil the sorption of hyamine varied from 0.00075 mol · kg⁻¹ to 0.03 mol · kg⁻¹, which constituted 100 % of the content of the surfactant in all initial solutions.

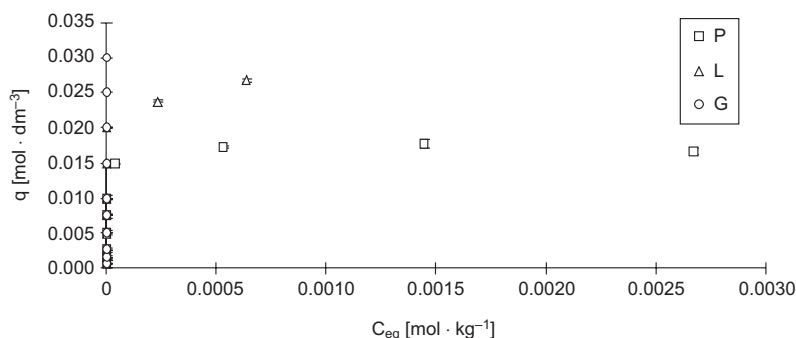


Fig. 1. Isotherms of the hyamine sorption in sandy soil (P), loess soil (L) and loamy soil (G); q – equilibrium sorption capacity; C_{eq} – equilibrium concentration; Error bars represent standard deviation of triplicate assays (shown if larger than the symbols)

The maximum, 100 % degree of sorption was found also in the sandy soil for initial concentrations from 0.00015 to 0.002 mol · dm⁻³, and in the loess soil for concentrations in the range of 0.00015–0.004 mol · dm⁻³. In the sandy soil, at equivalent concentration of 1.45 · 10⁻³ mol · dm⁻³, the sorption reached a maximum of 0.0178 mol · kg⁻¹ and then dropped to the level of 0.0167 mol · kg⁻¹ at concentration of 2.67 · 10⁻³ mol · dm⁻³. Whereas, in the loess soil a gradual increase of hyamine sorption was observed up to a maximum level of 0.0268 mol · kg⁻¹ at equivalent concentration of 6.36 · 10⁻⁴ mol · dm⁻³.

Analysis of the kinetic data (Fig. 2A) indicates that in the sandy and loess soils the intensity of the sorption was high already at the initial stage of the process. In the 15th

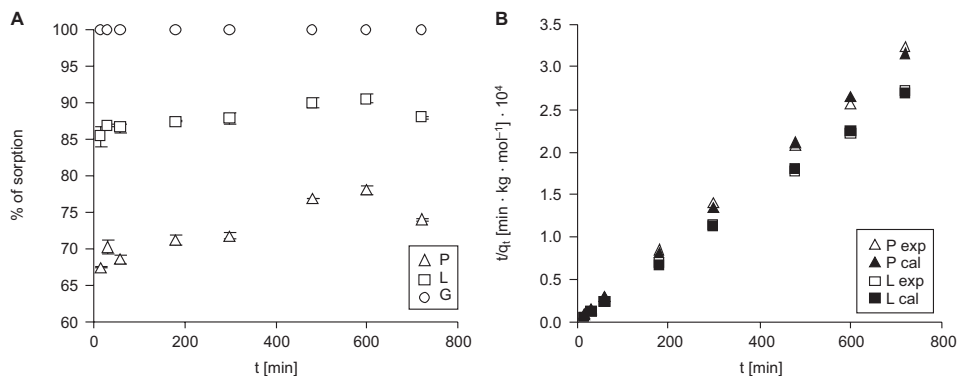


Fig. 2. A: Kinetics of hyamine sorption in sandy soil (P), loess soil (L) and loamy soil (G); B: Comparison between experimental (exp) and calculated (cal) values of t/q_t versus time (t) for the sorption of hyamine in sandy (P) and loess (L) soil; q_t – amount sorbed after time t; Error bars represent standard deviation of triplicate assays (shown if larger than the symbols)

minute the sorption was at the level of $0.0202 \text{ mol} \cdot \text{kg}^{-1}$ in the sandy soil and $0.0256 \text{ mol} \cdot \text{kg}^{-1}$ in the loess soil. Relative to the initial content of hyamine in the solution, that constituted 67.5 and 85.4 %, respectively. However, the effectiveness of the sorption changed only slightly with the passage of time of the contact between the initial solution and the sandy and loess soils. For example, after 60 and 300 minutes the following values of the sorption were obtained: 0.0205 and $0.0215 \text{ mol} \cdot \text{kg}^{-1}$ for the sandy soil, and 0.026 and $0.0263 \text{ mol} \cdot \text{kg}^{-1}$ for the loess soil. Compared with the sorption level on the 15th minute, after 60 and 300 minutes the sorption increased by 0.8 and 4.2 % in the sandy soil, and by 1.3 and 2.3 % in the loess soil. In the loamy soil the sorption was $0.03 \text{ mol} \cdot \text{kg}^{-1}$ (100 % of the initial content in the solution) and did not depend on the duration of the soil-solution contact.

The results of kinetic studies were analysed using the pseudo second-order model. The second order rate constant, k_2 , and the amount of hyamine sorbed at equilibrium, q_{eq} , were obtained from the slope ($1/q_{\text{eq}}$) and the intercept ($1/k_2 \cdot q_{\text{eq}}^2$) of the linear plot of experimental data of t/q_t versus t. Average values of k_2 and q_{eq} were as follows: $k_2 = 80.1 \cdot 10^{-5} \text{ kg} \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$, $q_{\text{eq}} = 229 \text{ mol} \cdot \text{kg}^{-1}$ for sandy soil and $k_2 = 276 \cdot 10^{-5} \text{ kg} \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$, $q_{\text{eq}} = 267.9 \text{ mol} \cdot \text{kg}^{-1}$ for loess soil. The kinetic parameters k_2 and q_{eq} were used to calculate theoretical values of q_t by application the equation 2. Fig. 2B shows the comparison between the experimental data and calculated data, which are obtained from the pseudo second-order equation. It is apparent from Fig. 2B, that the calculated data were similar to the experimental data or were the almost the same. This may indicate that the sorption of hyamine followed in accordance with the pseudo second-order reaction mechanism. Fig. 2B does not present the relation q_t/t in the function of time for sorption in the loamy soil due to the lack of any effect of time on hyamine sorption in that soil.

Figure 3 presents the relation of hyamine sorption in the soils in the function of pH values of the initial solution. It can be concluded that increase in pH value did not cause

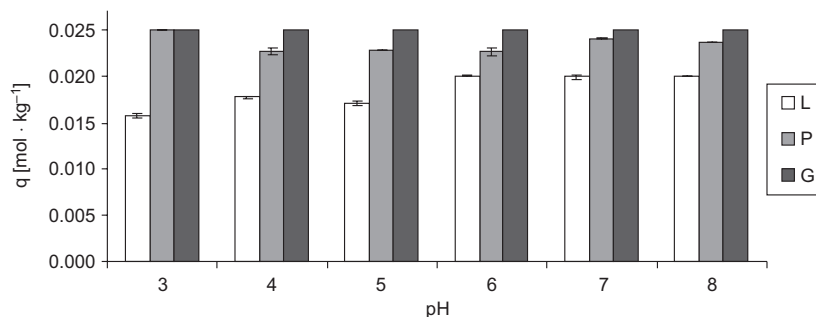


Fig. 3. Sorption of hyamine from solution of different pH in sandy soil (P), loess soil (L) and loamy soil (G); q – equilibrium sorption capacity; Error bars represent standard deviation of triplicate assays (shown if larger than the symbols)

any change in the level of hyamine sorption in the loamy soil. In the case of the loess soil, a slight decrease of hyamine sorption was observed within the range of pH 4 to pH 8. In the case of the sandy soil, increase in pH resulted in a slight increase of sorption until a constant level was reached within the pH range from 5 to 8.

The results of experiments concerning the sorption of hyamine from solutions with an addition of KCl are presented in Fig. 4. In the sandy soil the effect of KCl was bidirectional. At the molar ratio of 1 : 1, a decrease of hyamine sorption from solutions with concentrations of 0.002–0.005 mol · dm⁻³ was observed relative to the solutions without KCl. Whereas, for the highest initial concentration an increase of the sorption by 11.5 % was noted. Also in the case of solutions with the molar ratio of 1 : 10 a similar direction of changes in the level of sorption was observed, i.e. a decrease by 4 %

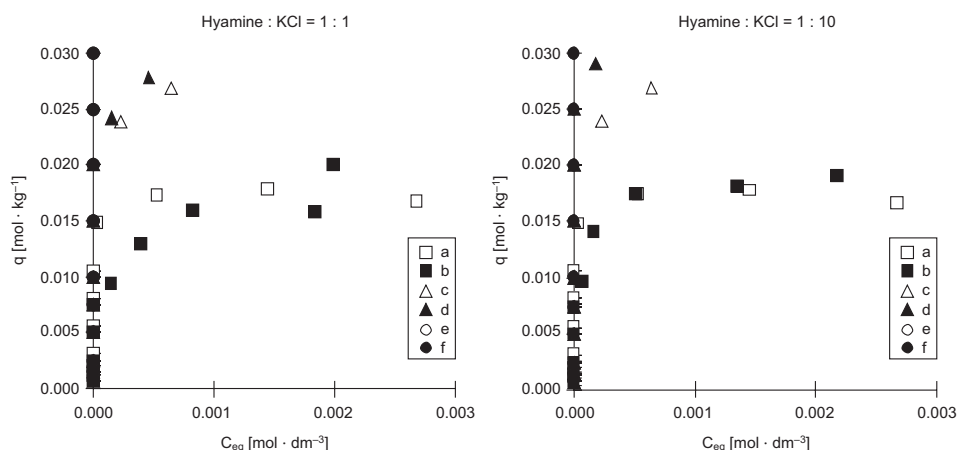


Fig. 4. Effect of KCl on the sorption of hyamine in sandy soil (a – without KCl, b + KCl), loess soil (c – without KCl, d + KCl) and loamy soil (e – without KCl, f + KCl); q – equilibrium sorption capacity; C_{eq} – equilibrium concentration; Error bars represent standard deviation of triplicate assays (shown if larger than the symbols)

for concentrations of $0.002\text{--}0.003\text{ mol} \cdot \text{dm}^{-3}$ and an increase by 8.5 % for the concentration of $0.006\text{ mol} \cdot \text{dm}^{-3}$.

The effect of KCl on hyamine sorption in the loess soil was observable in the case of solutions with the highest initial concentrations: 0.005 and $0.006\text{ mol} \cdot \text{dm}^{-3}$ (Fig. 4). At both molar ratios of the surfactant to the electrolyte, ie 1 : 1 and 1 : 10, an increase of hyamine sorption was observed relative to the sorption from solutions without any addition of KCl. The greatest changes were noted for sorption from the solutions with the molar ratio of 1 : 10.

The sorption of hyamine at that molar ratio from the solution with concentration of $0.005\text{ mol} \cdot \text{dm}^{-3}$ was higher by 4.8 % with relation to the sorption from solution without any content of KCl. For the solution with concentration of $0.006\text{ mol} \cdot \text{dm}^{-3}$ the increase in sorption was higher at 7.8 %.

The effect of KNO_3 on the sorption of hyamine in the sandy and loess soils had a character similar to that of the effect of KCl in the sandy soil (Fig. 5).

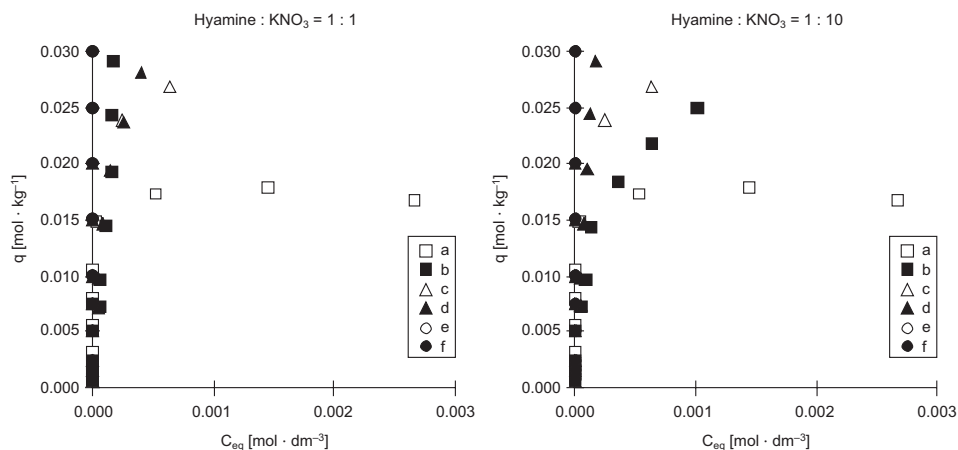


Fig. 5. Effect of KNO_3 on the sorption of hyamine in sandy soil (a – without KNO_3 , b + KNO_3), loess soil (c – without KNO_3 , d + KNO_3) and loamy soil (e – without KNO_3 , f + KNO_3); q – equilibrium sorption capacity; C_{eq} – equilibrium concentration; Error bars represent standard deviation of triplicate assays (shown if larger than the symbols)

At the surfactant/electrolyte molar ratios of 1 : 1 and 1 : 10 a decrease of sorption in the sandy soil was noted for initial concentrations from 0.0015 to $0.003\text{ mol} \cdot \text{dm}^{-3}$, and in the loess soil also for the concentration of $0.004\text{ mol} \cdot \text{dm}^{-3}$. It should be emphasised that the decrease was slight and did not exceed 4.5 % in the sandy soil and 3.5 % in the loess soil. In the range of concentrations from 0.004 to $0.006\text{ mol} \cdot \text{dm}^{-3}$ an increase of sorption was observed in the sandy soil, from 10.3 to 41.6 % at the molar ratio of 1 : 1, and from 4.7 to 27.8 % at the ratio of 1 : 10. In the loess soil a notably smaller increase was noted (a maximum of 7.9 %) for initial concentrations of 0.005 and $0.006\text{ mol} \cdot \text{dm}^{-3}$.

In the loamy soil the presence of KCl and KNO_3 did not cause any change in the level of hyamine sorption (Figs. 4 and 5). In this system, as in the case of sorption from

the solutions without any electrolyte, the level of sorption from all initial solutions was 100 %.

Discussion

The form of isotherms of hyamine sorption from the soils under study conforms to that of isotherms type L and H acc. to the classification by Giles and indicates strong interactions between hyamine and the soils [10]. This is supported by the 100 % sorption of hyamine from all solutions in the loamy soil, and by the high levels of hyamine sorption in the sandy and loess soils both from solutions with low and with medium initial concentrations of the surfactant. Also the results of the kinetic tests, and especially the high level of sorption in the initial phase of the experiment, suggest high affinity of hyamine to the soils used in the study. The sorptive properties of the studied soils with relation to hyamine were, however, notably varied, as indicated by the various forms of the isotherms in the area of high equivalent concentrations. Such behaviour of surfactants is frequently observed in soils with a high level of clay minerals and a relatively low organic matter content (< 5 %) [11]. The soils used in this study were characterised by a low content of organic carbon (below 3 %) and distinctly varied levels of clay fractions. It should be emphasised that the sequence of affinity to hyamine, determined on the basis of sorption levels, coincided with the hierarchy of the soils in the order of increasing content of clay fractions: sandy soil < loess soil < loamy soil. This sequence is also in agreement with the sorptive capacity of the soils under study.

The study demonstrated that in the sandy and loess soils the intensity of the sorption from solutions with higher initial concentrations was distinctly lower compared with that from solutions with lower concentrations. Xu and Boyd [12] presented the opinion that cationic surfactants are bound by soils in an alternating manner. Sorptive sites with negative charge attract the positively charged “head” of the surfactant, while its non-polar “tail” is oriented towards the soil solution. With increase in the concentration of the solution more and more sorptive sites are fitted in this way with the surfactant ions. It can be assumed that in the sandy soil, with lower sorptive capacity relative to the loess soil, saturation of the sorptive sites proceeded faster. For this reason, for solutions with the highest initial concentrations only slight changes were observed in the level of hyamine sorption in the sandy soil.

The lower sorption of hyamine from the solution with concentration of $0.006 \text{ mol} \cdot \text{dm}^{-3}$ observed in the sandy soil relative to the solution with concentration of $0.005 \text{ mol} \cdot \text{dm}^{-3}$ could be a result of electrostatic repulsion between the positively charged “heads” of the polar part of the surfactant [4]. As a result of that, hyamine cations were forced away from the active surface of the soil sorbents into the soil solution. Consequently, hyamine ions had difficult access to the sorptive sites and, therefore, the sorption of the surfactant decreased. With increase of the concentration of the solution, the forces of repulsion increase as diminish the distances between equally charged ions of the surfactant. This explains why a decrease of hyamine sorption was observed in the case of the solution with the highest concentration.

Analysis of the results revealed that the process of hyamine sorption in the loamy soil did not depend on the pH of the initial solution, and in the case of the sandy and loess soils the effect of pH of the solution on the sorption of the surfactant was slight. This should be attributed to the considerably stronger buffering properties of the loamy soil compared with the weaker regulating properties of the sandy and loess soils, resulting from the physicochemical properties of the soils studied.

Based on the study presented here it can be stated that the effect of electrolyte on the sorption of hyamine, or the lack of it, was related with the sorptive capacity of the soils used in the study. In the sandy and loess soils two opposite effects were observed, ie a decrease and an increase in the presence of an electrolyte with relation to sorption from solutions with no content of KCl or KNO₃. It should be noted that no such effect was found in the loamy soil, with the highest sorptive capacity, where the sorption of hyamine was total from all the solutions. Studies by Bouchard et al [13] and Brownawell et al [14] indicate that surfactant ions sorbed by a soil may be exchanged for cations of an electrolyte present in the solution. This would suggest that competition for sorptive sites between hyamine cations and K⁺ ions could had been the cause of reduced sorption of the surfactant in the sandy and loess soils. That effect was less pronounced in the loess soil due to its higher sorptive capacity. Whereas, the increased sorption of hyamine in the presence of the electrolytes could have been caused by the compression of the positive charge of the “heads” of hyamine by anions Cl⁻ and NO₃⁻. The result of that was a reduction of the mutual repulsion of hyamine cations, which stimulated the process of sorption in the soils [15].

Conclusions

1. The level of hyamine sorption was related to the sorptive capacity of the soils and basically increased with increasing concentration of the initial solution.
2. In the sandy and loess soils the effectiveness of hyamine sorption displayed slight changes with extension of the time of sorption and with increase in the pH of the initial solution. In the loamy soil the sorption of hyamine was at a constant level irrespective of the time of contact of the soil with the solution and of the pH of the solution.
3. The presence of an electrolyte did not cause any change in the level of hyamine sorption in the loamy soil. In the sandy and loess soils the effect of electrolytes on the sorption of hyamine depended on the kind of electrolyte used, on the sorptive capacity of the soil, and on the concentration of the surfactant.

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SORPCJA SURFAKTANTU HIAMINY 1622 W GLEBACH

Katedra Chemii
Uniwersytet Przyrodniczy w Lublinie

Abstrakt: Badano sorpcję hiaminy 1622, kationowego surfaktantu, z wodnych roztworów w 3 glebach, które ze względu na charakter skał macierzystych, nazwano w pracy odpowiednio glebą piaszczystą, lessową i gliniastą. Proces sorpcji prowadzono metodą statyczną: do probówek z polietylenu odważano 2 g gleby i dodawano 10 cm³ roztworu o stężeniu hiaminy w zakresie od 0,00015 do 0,006 mol · dm⁻³, a następnie wytrząsano przez 6 godz. Określono wielkość sorpcji hiaminy w zależności od stężenia i pH roztworu wyjściowego, czasu kontaktu gleba – roztwór oraz w obecności elektrolitów KCl i KNO₃ w układach surfaktant-elektrolit = 1 : 1 i 1 : 10.

Badania wykazały, że ze wzrostem stężenia roztworu wyjściowego następowało zwiększenie sorpcji hiaminy w glebach. Ustalono, że szereg powinowactwa badanych gleb względem hiaminy układał się następująco: gleba gliniasta > gleba lessowa > gleba piaszczysta. Wraz ze wzrostem pH obserwowano niewielkie zmiany efektywności sorpcji w glebie piaszczystej i lessowej, natomiast w glebie gliniastej sorpcja utrzymywała się na stałym poziomie. Badania kinetyczne wykazały, że w glebie piaszczystej i lessowej sorpcja zachodziła najszybciej w ciągu pierwszych 15 min, a wydłużenie czasu kontaktu gleba-roztwór do 720 min nie powodowało wyraźnych zmian w poziomie sorpcji. W glebie gliniastej stwierdzono 100 % stopień sorpcji hiaminy niezależnie od czasu kontaktu roztworu z glebą. Proces sorpcji zachodził zgodnie z mechanizmem reakcji pseudodrugiego rzędu. Obecność elektrolitu powodowała zarówno zmniejszenie, jak i zwiększenie sorpcji hiaminy w glebie piaszczystej i lessowej, ale w stopniu zależnym od rodzaju elektrolitu oraz stężenia surfaktantu. Dodatek elektrolitu nie wpływał na sorpcję hiaminy w glebie gliniastej.

Słowa kluczowe: surfaktant kationowy, sorpcja, gleba