Effect of micelles on the chemical speciation of binary complexes of Co(II), Ni(II), Cu(II) and Zn(II) with succinic acid

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Abstract: Speciation of Co(II), Ni(II), Cu(II) and Zn(II) complexes with succinic acid in the presence of anionic, cationic and non-ionic surfactants at an ionic strength of 0.16 mol dm^{-3} and temperature 303 K were investigated pH metrically. The existence of different binary complex species was established from modelling studies using the computer program MINIQUAD75. The decreased stability of the complexes with increasing micellar content was explained by electrostatic forces. The influence of the micelles on the chemical speciation is discussed based on the mole fraction of the medium. Distribution diagrams of various species of the complexes in relation to pH are presented.

Keywords: succinic acid; speciation; essential metals; binary complexes; micelles.

INTRODUCTION

Succinic acid (suc) has great potential as a building block chemical,[1] being the precursor for many other chemicals made from renewable resources. It can be produced by fermentation and processed into a variety of products.[2] Succinate is a component of the citric acid cycle. Dialkyl succinates are essential chemicals for industries producing food and pharmaceutical products, surfactants and detergents, green solvents, biodegradable plastics and ingredients to stimulate animal and plant growth. Succinate is also involved in the metabolic pathway that forms part of the breakdown of carbohydrates, fats and proteins into carbon dioxide and water in order to generate energy in all living cells that utilize oxygen as part of cellular respiration.

Speciation studies of essential metal ion complexes of suc are useful[3–6] for the understanding of the role played by active site cavities in biological molecules and the binding behaviour of protein residues with metal ions. Cobalt in the form of vitamin B$_{12}$ is essential for animals. Vitamin B$_{12}$ is synthesized only by micro-organisms, in particular anaerobic bacteria. Nickel is associated with seve-

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ral enzymes[7–9] and any variation in its concentration leads to metabolic disorders.[10] Copper is largely rejected from cells but outside the cell, it is essential for the metabolism of many hormones and connective tissue. The biological functions include electron transfer, dioxygen transport, oxygenation, oxidation, reduction and disproportionation.[11,12] Zinc is the second most abundant essential trace metal after iron and it plays vital roles in biological systems.[13–16] Surfactants are used as detergents, cleaning agents, emulsifiers, in food, pharmaceuticals and cosmetics. Hence, speciation studies of suc with some essential metal ions, such as Co, Ni, Cu and Zn, in surfactant–water mixtures are reported in this paper.

EXPERIMENTAL

A solution 0.050 mol dm^{-3} of succinic acid (R.G., E-Merck, Germany) was prepared in triple distilled water. Aqueous solutions of Co(II), Ni(II), Cu(II) and Zn(II) chlorides (0.050 mol dm^{-3}) were prepared in 0.050 mol dm^{-3} HNO$_3$, to suppress the hydrolysis of the metal salts. A.R. or G.R. samples of sodium lauryl sulphate, SDS, (Qualigens, India), cetyltrimethylammonium bromide, CTAB (Qualigens, India) and Triton X-100, TX100, (E-Merck, Germany) were used as commercial products and their purity was checked by determining the critical micellar concentration (*CMC*) conductometrically. The *CMC* values of SDS, CTAB and TX-100 were 8.1×10^{-3}, 9.2×10^{-4} mol dm^{-3} and 0.54 vol. %, respectively, at 303 K. Sodium nitrate was used to maintain the ionic strength in the titrand. The strengths of alkali and mineral acid were determined using the Gran plot method.[17] To assess the errors that might have crept into the determination of the concentrations, the data were subjected to analysis of variance of one-way classification (ANOVA).

Apparatus

The titrimetric data were obtained using a calibrated ELICO (Model LI-120) pH-meter (readability 0.01), which can monitor changes in the H$_3$O$^+$ concentration. The glass electrode was equilibrated in a well-stirred micellar solution containing an inert electrolyte. All the titrations were performed at 303.0±0.1 K in a medium containing varying concentrations of the surfactants (0.5–2.5 % w/w) maintaining an ionic strength of 0.16 mol dm^{-3} with sodium nitrate. The effect of variations in asymmetry, liquid junction potential, activity coefficient, sodium ion error and dissolved CO$_2$ on the response of glass electrode were taken into account in the form of a correction factor, which was discussed in an earlier communication.[18]

Procedure and modelling strategy

For the determination of the stability constants of the binary metal–ligand species, initially titrations of a strong acid with alkali were performed at regular intervals to check whether complete equilibration had been achieved. Then the calomel electrode was refilled with micellar solution (only TX100 and CTAB but not SDS, since it forms a precipitate with KCl) of equivalent composition to that of the titrand. In each of the titrations, the titrand consisted of approximately 1 mmol mineral acid in a total volume of 50 cm^3. Titrations with different ratios (1:2.5, 1:3.5, 1:5) of metal to ligand were performed with 0.40 mol dm^{-3} sodium hydroxide. Other experimental details are given elsewhere.[19]

The computer program SCPHD[20] was used to calculate the correction factor. The binary stability constants were calculated from with the pH-metric titration data using the computer program MINIQUAD75,[21] which exploits the advantage of a constrained least-squares me-

thod in the initial refinement and reliable convergence of the Marquardt algorithm. During the refinement of the binary systems, the correction factor and the protonation constants of suc were fixed. The variation of stability constants with the mole fraction of the medium was analysed on electrostatic grounds based on solute–solute and solute–solvent interactions.

RESULTS AND DISCUSSION

The results of the best-fit models that contain the stoichiometry of the complex species and their overall formation constants along with some of the important statistical parameters are given in Tables I–III. The very low standard deviation in the log β values indicates the precision of these parameters. The small values of U_{corr} (sum of the squares of the deviations in the concentrations of the constituents at all experimental points) corrected for degrees of freedom, indicate that the experimental data can be represented by the model. The small values of the mean, standard deviation and mean deviation for the systems corroborate that the residuals are around a zero mean with little dispersion. For an ideal normal distribution, the values of the kurtosis and skewness should be three and zero, respectively. The kurtosis values in the present study indicate that the residuals form leptokurtic as well as platykurtic patterns[18] and very few form mesokurtic patterns. The values of skewness recorded in the Tables I–III are between –2.88 and –0.01. These data evince that the residuals form part of a normal distribution. Hence, least squares method can be applied to the present data. The sufficiency of the model is further evident from the low crystallographic R-values. These statistical parameters thus show that the best-fit models portray the metal–ligand species in micellar media.

Effect of systematic errors on best-fit model

In order to rely upon the best chemical model for critical evaluation and application under varied experimental conditions with different accuracies of data acquisition, an investigation was made by introducing pessimistic errors into influential parameters, such as the concentrations of alkali, mineral acid, ligand and metal (Table IV). The order of the components that influence the magnitudes of the stability constants due to incorporation of errors is alkali > acid > ligand > > metal. Some species were even rejected when errors were introduced in the concentrations. The rejection of some species and increased standard deviations in the stability constants on introduction of errors confirm the appropriateness of the experimental conditions (concentrations of the constituents) and the choice of the best-fit models.

Effect of the surfactant

Variation of the stability constants (log β) with mole fraction of surfactants in various micellar media exhibits a non-linear decreasing trend. The stability of the complex depends on the polarity of the medium, charge on the Stern layer[22]

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TABLE I. Parameters of the best-fit chemical models of Co(II), Ni(II), Cu(II) and Zn(II)–succinic acid complexes in CTAB–water mixtures

CTAB % w/v	log β(SD)					NP	U_{corr}[a] ×10^{-8}	Skewness	χ^2	R-Factor ×10^3	Kurtosis	pH Range
	ML	MLH	ML$_2$	ML$_2$H	ML$_2$H$_2$							
Co(II)												
0.0	–	9.99(4)	6.43(3)	12.52(8)	–	115	2.56	-1.93	31.09	73	4.07	1.60–8.50
0.5	–	8.87(5)	5.34(4)	11.34(7)	–	93	3.10	0.44	35.02	49	5.04	2.50–7.50
1.0	–	8.71(2)	5.30(3)	11.26(12)	–	98	8.15	0.71	22.04	5.2	9.21	2.00–7.90
1.5	–	8.69(2)	5.27(8)	11.20(10)	–	95	6.53	1.21	39.31	1.4	3.23	2.50–7.50
2.0	–	8.51(4)	5.16(7)	11.15(11)	–	89	4.72	1.04	47.71	65	5.45	2.50–5.50
2.5	–	8.29(2)	5.04(5)	11.06(12)	–	96	3.25	1.74	49.05	1.3	6.05	2.50–6.00
Ni(II)												
0.0	4.47(2)	–	9.58(4)	12.68(7)	–	59	7.32	-0.18	21.92	83	2.93	2.00–7.00
0.5	3.36(1)	–	8.95(7)	11.64(8)	–	95	3.10	-2.42	29.01	9.5	3.42	2.50–6.00
1.0	3.29(2)	–	8.74(5)	11.72(8)	–	105	8.15	-2.54	32.13	72	4.44	1.85–6.50
1.5	3.20(2)	–	8.45(6)	11.51(9)	–	102	6.53	-1.09	13.59	46	8.02	1.85–6.00
2.0	3.11(3)	–	8.32(5)	11.39(10)	–	108	4.72	-1.74	18.92	8.5	7.31	1.85–7.00
2.5	3.03(3)	–	7.58(5)	11.16(11)	–	92	3.25	-1.80	17.41	9.6	4.92	2.50–6.00
Cu(II)												
0.0	–	–	8.95(3)	11.86(5)	16.08(12)	71	0.65	-2.24	22.32	5.2	8.62	2.00–7.50
0.5	–	–	7.95(4)	10.95(6)	15.59(18)	100	6.40	-0.38	24.32	3.2	3.62	2.75–8.50
1.0	–	–	7.63(2)	10.68(4)	15.35(14)	101	8.29	-0.44	38.34	6.3	4.82	1.75–8.00
1.5	–	–	7.34(5)	10.48(6)	15.21(20)	94	8.74	-0.52	48.48	8.7	5.62	2.50–8.00
2.0	–	–	6.98(3)	10.35(5)	14.95(11)	89	8.02	-0.32	34.32	8.3	4.48	2.50–7.50
2.5	–	–	6.81(4)	10.02(6)	14.82(16)	85	4.09	-0.12	4.69	18	3.67	2.50–5.50
Zn(II)												
0.0	–	10.75(3)	6.75(5)	14.87(6)	–	85	2.55	-1.34	19.32	3.9	4.32	2.00–7.50
0.5	–	9.94(2)	5.94(4)	13.91(6)	–	100	5.04	0.03	40.74	40	2.70	1.90–8.50
1.0	–	9.83(2)	5.83(4)	13.82(7)	–	97	2.64	1.35	64.25	2.2	8.32	1.70–7.50
1.5	–	9.52(4)	5.25(5)	13.50(8)	–	98	1.55	2.24	32.32	8.1	7.42	1.80–8.50
2.0	–	8.98(3)	4.93(5)	13.42(5)	–	85	4.24	1.00	44.81	39	6.93	1.75–7.00
2.5	–	8.62(6)	4.57(4)	13.15(7)	–	97	2.10	1.32	53.85	4.3	5.42	2.50–8.50

[a]$U_{corr} = U/(NP – m)\times10^8$, where, m = number of species; NP = number of experimental points

TABLE II. Parameters of the best-fit chemical models of Co(II), Ni(II), Cu(II) and Zn(II)–succinic acid complexes in TX100–water mixtures

TX100 % v/v	log β(SD)					NP	U_{corr}^{a} ×10^{-8}	χ^2	R–Factor ×10^3	Skewness	Kurtosis	pH Range
	ML	MLH	ML$_2$	ML$_2$H	ML$_2$H$_2$							
Co(II)												
0.0	–	9.99(4)	6.43(3)	12.52(8)	–	115	2.56	31.09	73.2	–1.93	4.07	1.60–8.50
0.5	–	8.64(2)	5.92(4)	11.98(7)	–	110	8.66	35.42	93.2	–1.84	5.94	1.64–8.50
1.0	–	8.39(1)	5.87(4)	11.78(4)	–	125	3.62	98.32	14.2	–2.85	9.34	1.65–8.00
1.5	–	8.27(4)	5.49(2)	11.52(5)	–	52	8.97	25.32	83.2	–1.24	4.85	2.00–5.50
2.0	–	7.97(2)	4.97(3)	10.89(8)	–	50	7.33	29.64	85.4	–1.84	3.32	2.00–5.60
2.5	–	7.64(1)	4.81(3)	10.56(8)	–	65	5.83	34.29	45.9	–1.01	2.83	2.00–6.00
Ni(II)												
0.0	4.47(2)	–	9.58(4)	12.98(7)	–	59	7.32	21.92	83.2	–0.18	2.93	2.00–7.00
0.5	3.36(2)	–	8.75(4)	11.89(7)	–	55	8.09	42.32	84.1	–4.32	4.32	2.50–6.00
1.0	3.29(3)	–	8.65(5)	11.92(7)	–	51	6.66	38.68	49.2	–2.42	6.84	2.50–7.00
1.5	3.20(3)	–	8.53(4)	11.48(4)	–	57	1.89	54.32	49.2	–1.38	5.94	2.00–7.50
2.0	3.11(2)	–	7.97(5)	10.97(4)	–	78	5.74	38.68	18.1	–2.32	4.48	2.50–7.00
2.5	3.02(2)	–	7.62(5)	10.58(5)	–	80	9.22	54.32	317	–1.94	3.42	2.00–6.00
Cu(II)												
0.0	–	–	6.94(3)	11.97(5)	16.08(12)	71	0.65	22.32	5.23	–2.24	8.62	2.00–7.50
0.5	–	–	5.90(7)	10.89(8)	15.76(10)	162	6.37	45.20	3.43	–1.49	3.14	1.50–8.50
1.0	–	–	5.76(5)	10.85(7)	15.89(21)	160	6.44	22.15	8.41	–2.42	2.15	1.50–8.20
1.5	–	–	5.62(6)	10.45(8)	15.35(14)	62	4.09	42.52	9.31	–4.42	4.82	2.50–7.50
2.0	–	–	4.98(6)	9.86(8)	14.98(15)	65	3.60	52.11	8.31	–1.82	3.42	2.50–8.00
2.5	–	–	4.78(7)	9.25(9)	14.53(10)	62	3.87	72.35	1.24	1.39	8.14	2.50–7.50
Zn(II)												
0.0	–	10.75(3)	6.75(5)	14.87(6)	–	85	2.55	19.32	3.94	–1.34	4.32	2.00–7.50
0.5	–	9.87(1)	5.87(7)	13.89(11)	–	148	1.07	38.25	17.3	–1.88	8.32	1.62–8.50
1.0	–	9.82(3)	5.76(8)	13.56(12)	–	132	1.17	57.52	18.3	–2.49	7.92	1.60–8.00
1.5	–	9.51(3)	5.32(7)	13.25(12)	–	84	1.18	45.39	13.4	0.87	4.62	2.00–8.00
2.0	–	8.94(2)	4.95(8)	12.89(11)	–	82	8.46	52.31	13.5	–1.34	5.62	2.00–8.00
2.5	–	8.24(4)	4.78(7)	12.56(10)	–	77	2.27	39.21	42.9	0.47	3.90	2.00–7.80

[a] $U_{corr} = U/(NP - m)\times10^{8}$, where, m = number of species; NP = number of experimental points

TABLE III. Parameters of the best-fit chemical models of Co(II), Ni(II), Cu(II) and Zn(II)–succinic acid complexes in SDS–water mixtures

SDS % w/v	log β(SD)					NP	U_{corr}[a] ×10^{-3}	Skewness	χ^2	R–Factor ×10^3	Kurtosis	pH Range
	ML	MLH	ML$_2$	ML$_2$H	ML$_2$H$_2$							
Co(II)												
0.0	–	9.99(4)	6.43(3)	12.52(8)	–	115	2.56	–1.93	31.09	73.2	4.07	1.60–8.50
0.5	–	9.25(3)	6.25(7)	12.01(11)	–	57	1.41	–1.18	22.87	35.6	6.29	2.00–7.00
1.0	–	8.79(2)	5.87(6)	11.75(12)	–	53	6.40	–1.24	31.32	46.0	5.52	2.50–6.70
1.5	–	8.53(2)	5.48(8)	11.54(10)	–	56	8.49	–1.33	27.31	3.20	3.39	2.00–6.90
2.0	–	8.23(4)	5.24(8)	11.35(10)	–	55	7.32	–2.30	18.10	5.60	2.36	2.00–7.00
2.5	–	7.56(2)	4.82(7)	10.58(12)	–	54	2.56	–2.25	16.21	81.4	4.42	2.00–6.50
Ni(II)												
0.0	4.47(2)	–	9.58(4)	12.68(7)	–	59	7.32	–0.18	21.92	83.2	2.93	2.00–7.00
0.5	3.98(2)	–	9.01(15)	12.02(14)	–	51	4.02	–0.01	19.47	52.0	4.31	2.00–6.50
1.0	3.59(3)	–	8.56(14)	11.58(18)	–	54	5.88	–0.29	20.25	2.70	3.35	2.00–6.80
1.5	3.52(2)	–	8.31(15)	11.23(15)	–	56	9.24	–0.11	35.90	2.90	8.62	2.00–7.00
2.0	3.21(2)	–	7.86(15)	11.05(20)	–	53	6.40	–0.42	28.98	29.8	5.52	2.50–6.50
2.5	3.01(3)	–	7.25(12)	10.89(22)	–	52	8.97	–0.57	42.45	34.3	4.67	2.00–6.20
Cu(II)												
0.0	–	–	6.94(3)	11.97(5)	16.08(12)	71	0.65	–2.24	22.32	5.20	8.62	2.00–7.50
0.5	–	–	7.15(2)	10.58(8)	15.47(17)	69	9.01	–1.51	19.49	3.90	5.52	2.00–6.80
1.0	–	–	7.32(4)	10.45(7)	15.84(20)	78	2.68	–1.45	27.07	51.2	4.67	1.70–7.40
1.5	–	–	7.54(4)	10.27(7)	15.77(22)	67	8.49	–1.59	32.38	49.7	7.72	2.00–6.50
2.0	–	–	7.18(3)	10.15(8)	15.35(19)	68	3.84	–1.28	17.49	83.2	4.67	2.50–7.00
2.5	–	–	7.21(3)	10.74(8)	15.08(20)	77	1.32	–1.49	27.48	73.5	5.49	1.70–7.50
Zn(II)												
0.0	–	10.75(3)	6.75(5)	14.87(6)	–	85	2.55	–1.34	19.32	3.90	4.32	2.00–7.50
0.5	–	9.66(2)	5.58(8)	13.91(18)	–	92	3.30	–1.59	21.42	4.70	9.82	1.75–7.50
1.0	–	10.71(2)	6.66(5)	14.52(14)	–	87	3.43	–2.88	39.82	93.7	8.84	2.00–7.42
1.5	–	10.05(3)	6.17(7)	14.20(20)	–	79	7.33	–2.21	45.49	52.1	6.95	1.90–7.50
2.0	–	9.89(3)	5.92(7)	13.77(18)	–	84	8.06	–4.45	52.32	45.1	4.59	2.00–7.50
2.5	–	9.74(2)	5.74(8)	13.53(17)	–	81	4.58	–2.45	25.94	83.1	3.23	2.20–7.40

[a]$U_{corr} = U/(NP – m) \times 10^8$, where, m = number of species; NP = number of experimental points

and the electrostatic attraction or repulsive forces operating between the complex species and the micellar surface. The dielectric constant of the medium decreases with increasing concentration of surfactant.[23,24] The charged species will be destabilized due to the decreased dielectric constant of the medium with increasing surfactant concentration. The linear decrease indicates the dominant action of electrostatic forces over non-electrostatic forces on the complex equilibria.

TABLE IV. Effect of errors in influential parameters on the stability constants of Ni(II) in Ni(II)-Suc in 1.0 %v/v TX100–water mixture

| Component | Error, % | $\log \beta$(SD) | | |
		MLH	ML$_2$	ML$_2$H
Alkali	0	3.29(3)	8.65(5)	11.92(7)
	−5	4.05(32)	Rejected	Rejected
	−2	3.55(11)	8.97(43)	12.07(32)
	+2	Rejected	8.84(10)	12.23(23)
	+5	3.92(12)	Rejected	Rejected
Acid	−5	Rejected	8.37(22)	Rejected
	−2	3.11(22)	8.05(43)	11.39(41)
	+2	Rejected	Rejected	12.30(35)
	+5	Rejected	9.67(44)	Rejected
Ligand	−5	3.82(24)	8.75(10)	12.09(70)
	−2	3.79(29)	8.71(10)	12.05(66)
	+2	3.85(49)	8.67(62)	12.03(84)
	+5	3.65(97)	8.78(14)	11.12(25)
Metal	−5	3.82(38)	8.82(41)	11.03(70)
	−2	3.79(37)	9.07(41)	12.01(70)
	+2	3.82(36)	8.77(42)	12.00(69)
	+5	3.88(35)	8.84(55)	12.08(69)

Distribution diagrams

Suc has two dissociable carboxyl protons, its various forms are LH$_2$, LH$^-$ and L^{2-} in the pH ranges 3.0–6.5, 3.0–7.0 and 4.5–7.0, respectively. The protonation equilibria of L-aspartic acid, citric acid and succinic acid in anionic and cationic micellar media were earlier reported.[25,26] The plausible refined species are MLH, ML$_2$ and ML$_2$H for the Co(II) and Zn(II) systems; ML, ML$_2$ and ML$_2$H for Ni(II); ML$_2$, ML$_2$H and ML$_2$H$_2$ for Cu(II) in anionic, cationic and non-ionic micellar media.

Typical distribution diagrams of suc in CTAB–water mixtures are shown in Fig. 1, which indicates that ML$_2$ had formed to an extent of 60 % at pH values above 5.0. The formation of the various binary complex species is shown in the following equilibria:

$$M(II) + LH_2 \rightleftharpoons MLH^+ + H^+ \qquad (1)$$

$$MLH^+ \rightleftharpoons ML + H^+ \qquad (2)$$

$$M(II) + 2LH_2 \rightleftharpoons ML_2H_2 + 2H^+ \tag{3}$$
$$ML_2H_2 \rightleftharpoons ML_2H^- + H^+ \tag{4}$$
$$ML_2H^- \rightleftharpoons ML_2{}^{2-} + H^+ \tag{5}$$
$$M(II) + 2LH^- \rightleftharpoons ML_2H^- + H^+ \tag{6}$$
$$ML + LH^- \rightleftharpoons ML_2{}^{2-} + H^+ \tag{7}$$
$$M(II) + LH^- \rightleftharpoons ML + H^+ \tag{8}$$
$$MLH^+ + LH_2 \rightleftharpoons ML_2H_2 + H^+ \tag{9}$$
$$MLH^+ + LH^- \rightleftharpoons ML_2H^- + H^+ \tag{10}$$
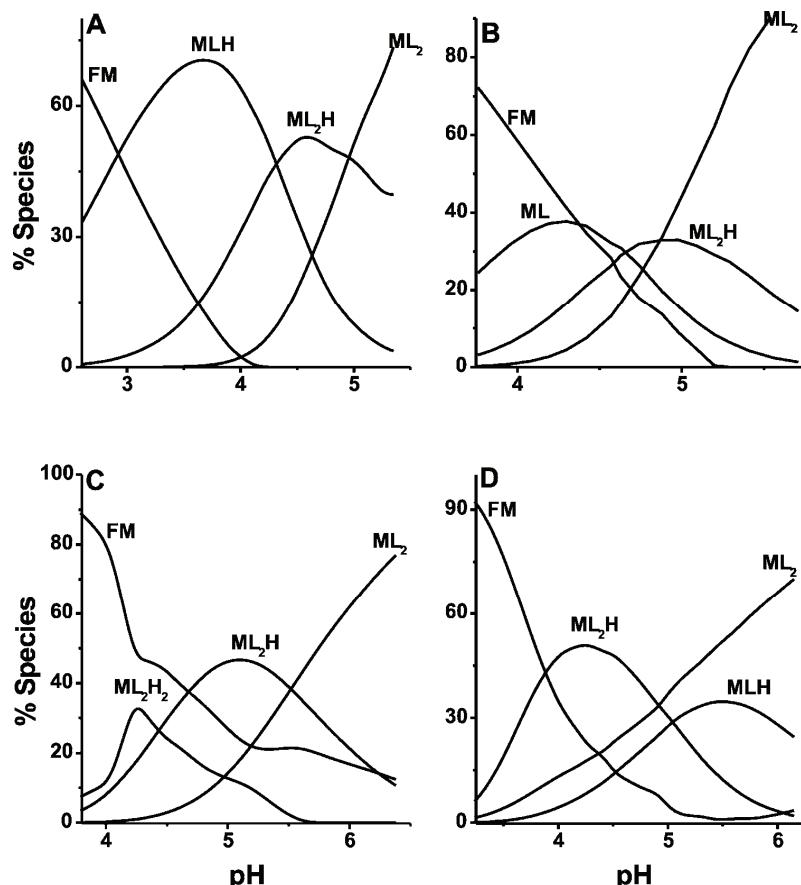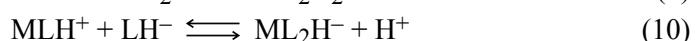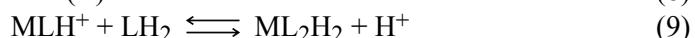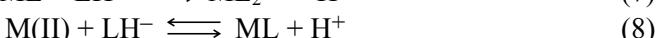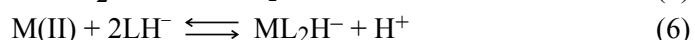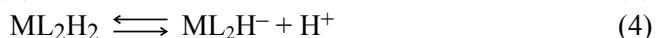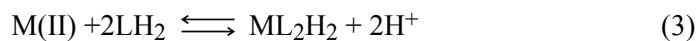
Fig. 1. Distribution diagrams of succinic acid complexes in 1.5 % w/v CTAB mixture. A) Co(II), B) Ni(II), C) Cu(II) and D) Zn(II).

For Co(II) and Zn(II), Eq. (5) is relevant because decreases in the percentage of ML_2H^- and increases in ML_2 were observed at the same pH (Figs. 1A and 1D). For Ni(II), Eqs. (5) and (8) are applicable (Fig. 1B). In the case of Cu(II),

Eqs. (3)–(5) are more appropriate because decreases in the percentage of ML_2H_2 and increases in the percentage of ML_2H^- were observed at the same pH (Fig. 1C).

CONCLUSIONS

The common species formed due to interaction of succinic acid with the studied metals are ML, MLH^+, ML_2^{2-}, ML_2H^- and ML_2H_2. The non-linear decrease of stability constants with mole fraction of the surfactant indicates the dominance of electrostatic forces over the non-electrostatic forces and a decreased dielectric constant with increasing surfactant concentration. The order of the components in influencing the magnitudes of the stability constants due to incorporation of errors is alkali > acid > ligand > metal.

И З В О Д

УТИЦАЈ МИЦЕЛА НА ХЕМИЈСКУ СПЕЦИЈАЦИЈУ БИНАРНИХ КОМПЛЕКСА Co(II), Ni(II), Cu(II) И Zn(II) СА ЋИЛИБАРНОМ КИСЕЛИНОМ

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Изучавана је специјација Co(II), Ni(II), Cu(II) и Zn(II) комплекса са ћилибарном киселином у присуству анјонских, катјонских и нејонских површински активних супстанци при јонској јачини од 0,16 mol dm^{-3} и температури 303 K, пехаметријски. Моделовањем помоћу компјутерског програма MINIQUAD75 утврђено је постојање разних бинарних комплексних врста. Смањење стабилности комплекса са повећањем мицеларног садржаја приписано је електростатичким силама. Утицај мицела на хемијску специјацију дискутован је на основу молских удела. Приказани су дијаграми расподеле разних врста комплекса у зависности од pH вредности.

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