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INFLUENCE OF BARIUM IONS ON THE PROPERTIES OF SUPRAMOLECULAR SUBSTANCES COCAMIDE DIETHANOLAMINE AND POLYOXOMOLIBDATES

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ABSTRACT

In this work, the effect of solution acidity and the presence of barium ions on the properties of the supramolecular substance diethanolamide of coconut oil fatty acids and molybdenum polyoxometalates was investigated using the UV spectrophotometric method. The possibility of interacting the obtained cocamide diethanolamine-barium complex with polyoxomolybdates to get a new supramolecular substance with electroactive properties was investigated, and the nature of the bond in the obtained substances was determined. The results of these studies can be used in the development of technologies for obtaining new electroactive supramolecular substances based on cocamide diethanolamine and polyoxomolybdates for use in electrochemical sensors.

Keywords: barium, coconut oil fatty acid diethanolamide, cocamide diethanolamine, cocodiethanolamide, molybdenum, nonionic surfactant, polyoxometalates, supramolecular substance, spectrophotometry.

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INTRODUCTION

Cocamide diethanolamine (coconut diethanolamide; diethanolamide condensed with coconut fatty acid; cocoyldiethanolamide) is a mixture of coconut acid ethanolamides, where RC=O represents fatty acids derived from coconut oil [1].

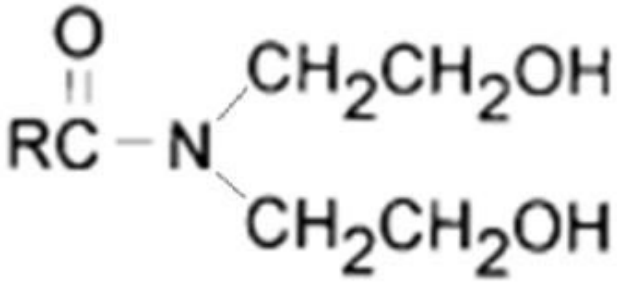

Figure-1. Structural formula of cocamide diethanolamine (n = 5, 7, 9, 11, 13, 15).

The general formula of cocamide diethanolamine (cocamide DEA) can be written as $C_{(7+n)}H_{(15+2n)}NO_3$, where n is the number of carbon atoms in the alkyl chain of the fatty acid. The relative molecular mass depends on the value of n and varies in the range of 231–371 g/mol. In calculations, n = 11 is mainly used, which corresponds to lauric acid, which dominates the composition respectively, the calculated molecular mass of cocamide diethanolamine (CDEA) is 315 g/mol.

Cocamide DEA, due to its amphiphilic structure, is actively used as an auxiliary surfactant in a wide range of industries. Its main advantage is the ability to simultaneously exhibit the properties of an emulsifier, stabilizer, and foaming agent, which determines its functional suitability. This compound demonstrates low acute toxicity, but with prolonged exposure in high doses, it can cause dose-dependent effects. Irritation of human skin is unlikely: a 10% solution of cocoyldiethanolamide did not cause reactions in tests on volunteers; however, professional contact with concentrations > 5% can provoke allergic dermatitis. Cases of sensitization in cosmetic products are rare due to the limitation of concentrations (≤ 10% in leave-on products) [2].

Identification of coconut diethanolamide is carried out by IR spectroscopy [3-4] and high-performance liquid chromatography (HPLC): normal-phase [5] or reversed-phase [6]. N-nitrosodiethanolamine (NDELA) impurities in cocoyldiethanolamide are determined by gas chromatography with mass spectrometry and high-performance liquid chromatography with UV detection [7]. Potentiometric titration, chromatographic methods, capillary electrophoresis, and the most modern method of attenuated internal reflection in combination with Fourier-IR spectrometry (ATR-FTIR) and partial least squares regression are used for quantitative determination of coconut diethanolamide [8].

The second component of the synthesized supramolecular substances - polyoxometalates (POM) - is are coordination compound belonging to polyligand inorganic substances [9-10]. Polyoxometalate salts exhibit ion-exchange properties. Previous studies of the reactions of polyoxomolybdates with other types of organic cationic and nonionic substances (plant polyphenols [11-14], surfactants [15-18], antimicrobial substances [19-21]) indicate the possibility of obtaining electroactive

supramolecular substances of cocamide diethanolamine and polyoxomolybdates.

This work aims to improve the electroactive properties of previously obtained [22] supramolecular substances of coconut diethanolamide and molybdenum polyoxometalates by adding barium salts. To this end, the possibility of chemical reactions between coconut oil fatty acid diethanolamide, barium salts, and polyoxomolybdates to obtain electroactive supramolecular substances was investigated, the nature of the bond in the obtained compounds was determined, and the influence of various factors on the properties of the obtained supramolecular substance was also investigated.

MATERIALS AND DEVICES

Spectrophotometric studies carried out with the spectrophotometer SF-46 (UV- spectra).
The following reagents were used in the work:

- polyoxomolybdate, $H_3PMo_{12}O_{40}x26H_2O$ (analytically pure);
- polyoxomolybdate, $H_5PMo_2Mo_{10}O_{40}x26H_2O$ (analytically pure);
- cocamide diethanolamine, $C_{12}H_{25}ON$ (analytically pure);
- barium chloride $BaCl_2 \cdot 2H_2O$ (analytical grade);
- sodium hydroxide (analytically grade);
- chloride acid (analytical grade).

EXPERIMENTAL PART

To determine the possibility of obtaining supramolecular substances of coconut diethanolamide with barium salts and polyoxomolybdates, it is necessary to determine rational conditions for the reaction. To determine rational conditions, the effect of solution acidity on the properties of cocamide diethanolamine and its complexes with barium was studied by the UV spectrophotometric method. The spectral characteristics of the solutions were recorded at the inherent acidity of the cocamide diethanolamine solution (pH = 6), as well as in the weakly acidic (pH = 4) and alkaline (pH = 8-10) ranges. UV absorption spectra recorded in the range of 200-350 nm in quartz glass cuvettes (optical path length 1 cm). Figure-2 and 3 show the spectral characteristics of solutions of KDEA and its complexes with barium salts.

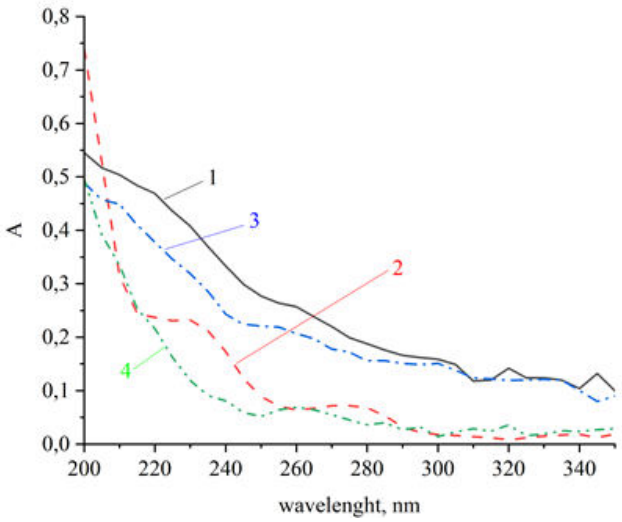

Figure-2. UV absorption spectra in the acidic region: 1,3 - CDEA at pH 4 and 6, respectively; 2,4 - CDEA-Baat pH 4 and 6 respectively

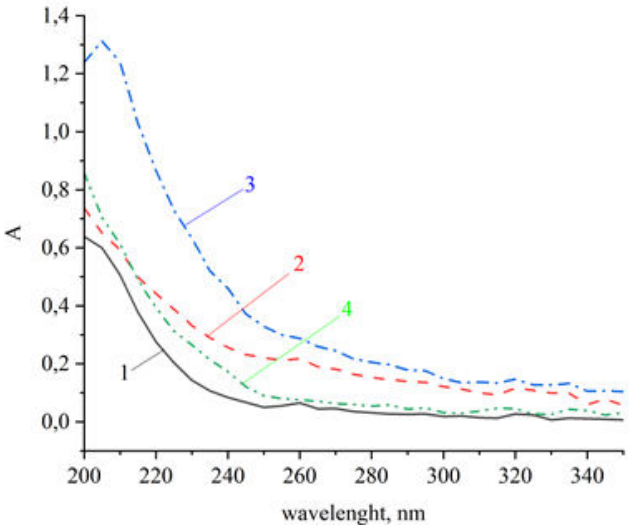

Figure-3. UV absorption spectra in the alkaline region: 1,3 - CDEA at pH 8 and 10, respectively; 2,4 - CDEA-Ba at pH 8 and 10, respectively.

Table-1 shows the UV spectra of solutions of cocamide DEA and its complex with barium, depending on the acidity of the studied solution.

Table-1. Spectral characteristics of solutions of CDEA and CDEA-Ba, depending on the pH.

pH of the solution	substance	wavelength, nm	$\varepsilon_{exp.}$, $l \cdot mol^{-1} \cdot cm^{-1}$
4	CDEA	200	54500
		255-260 (shoulder)	26000
		290-295 (shoulder)	17000
		320	14000
		345	13000
	CDEA-Ba	200	148000
		220-230 (shoulder)	46000
		270-275 (shoulder)	14400
		340	3600
		350	3800
6	CDEA	200	49000
		245-250 (shoulder)	22000
		280-300 (shoulder)	15000
		325-335 (shoulder)	12000
	CDEA-Ba	200	50000
		250-250 (shoulder)	6500
		280	4000
		310	2900
		320	3600
		350	2900
8	CDEA	200	64000
		260	6500
		285-295 (shoulder)	2500
		305	2000
		320-325 (shoulder)	2700
		335	1300
	CDEA-Ba	200	73700
		250-260 (shoulder)	22000
		285-290 (shoulder)	14500
		320	11600
		330-335	9000
		345	7800
10	CDEA	205	131000
		245-250 (shoulder)	37000
		290-295 (shoulder)	17500
		320	14700
		335	13000
	CDEA-Ba	200	85800
		260-265 (shoulder)	7100
		280-285 (shoulder)	5400
		315-320 (shoulder)	4500
		335	4300
		350	3200

The obtained absorption spectra of CDEA and CDEA-Ba (Table-1) indicate the presence of a characteristic absorption maximum or shoulder of high intensity in the range of 200-205 nm. For CDEA solutions, the presence of absorption maxima or shoulders of medium intensity at 245-260 nm, 280-300 nm, and 320-335 nm is characteristic, regardless of the acidity of the solution. In the absorption spectra of the compound CDEA-Ba, regardless of pH, there are characteristic absorption maxima or shoulders of high intensity at 270-290 nm (hypsochromic shift of the absorption band of CDEA at 280-300 nm) and low intensity maxima or shoulders at 315-320 nm and 330-350 nm regardless of the acidity of the solution (bifurcation of the absorption band of CDEA at 320-335 nm). For the compound CDEA-Ba, an additional absorption maximum or shoulder of medium intensity at 250-265 nm (bathochromic shift of the absorption maximum of CDEA at 245-260 nm) is also

observed at pH = 6-10, which at pH = 4 shifts to 220-230 nm. When alkalizing the CDEA solution to pH = 8-10, a more dramatic decrease in absorption is observed after the maximum at 200 nm compared to other acidity levels.
If we analyze the change in the absorption of the CDEA-Ba solution compared to the original CDEA solution at a certain pH, we can see that the absorption band in the range of 200-205 nm is preserved, while the other absorption bands are shifted:

- the absorption band at 245-260 nm shifts to 230-235 nm at pH = 4, is preserved at pH = 6-8, and disappears at pH = 10;
- the absorption band at 280-300 nm hypsochromically shifts to 270-290 nm;
- the absorption shoulder at 320-225 nm is divided into two separate absorption bands at 315-320 nm and 330-350 nm.

To study the effect of solution acidity on the reaction of the CDEA-Ba complex with polyoxomolybdate ions and to determine rational reaction conditions, the spectral characteristics of supramolecular compounds of the oxide form of polyoxometalate (POM_{ox}) of molybdenum CDEA-Ba-POM_{ox} (Fig. 3) were recorded depending on the pH in the UV region of the spectrum. The obtained spectral data of ternary supramolecular substances of the composition CDEA-Ba-POM_{ox} and CDEA-Ba-POM_{red} were also compared with the previous spectral data obtained for double supramolecular substances without barium salts of the composition CDEA-POM_{ox} and CDEA-POM_{red} [22].

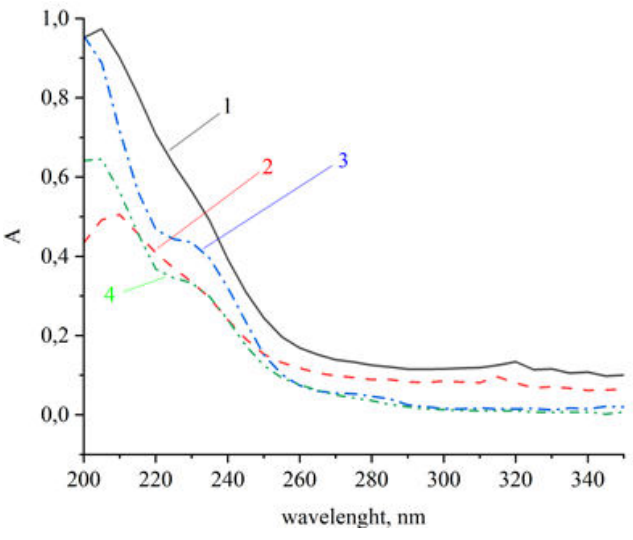

Figure-4. UV absorption spectra of CDEA-Ba-POM_{ox} as a function of pH: 1 - pH = 4; 2 - pH = 6; 3 - pH = 8; 4 - pH = 10.

Table-2 shows the characteristics of the UV spectra of the supramolecular substance based on CDEA and the oxidative form of polyoxomolybdate, depending on the acidity of the studied solution.

Table-2. Spectral characteristics of CDEA-Ba-POM_{ox} depending on the pH.

pH of the solution	wavelength, nm	$\varepsilon_{exp.}$, l·mol^{-1}·cm^{-1}
4	205	324667
	240-245 (shoulder)	130000
	270-275 (shoulder)	45000
	280-285 (shoulder)	40000
	290-300 (shoulder)	38500
	320	44000
	340	36000
	350	33333
6	210	168000
	270-285 (shoulder)	30000
	315	32000
	330	23600
	280-285 (shoulder)	20000
	290-300 (shoulder)	17000
	315	13800
	335-350 (shoulder)	10000
8	200	318000
	225-230 (shoulder)	145000
	270-275 (shoulder)	18000
	280-285 (shoulder)	14000
	290-295 (shoulder)	8000
	310	5600
	335	5600
	345	7000
10	200-205 (shoulder)	213000
	270-275 (shoulder)	16600
	280-285 (shoulder)	7000
	310-320 (shoulder)	3300
	350	2300

For the supramolecular substance based on the oxidative form of polyoxomolybdate CDEA-Ba-POM_{ox} at pH = 4 (Table-2), a general increase in absorption intensity is observed compared to CDEA-POM_{ox} with the preservation of the absorption bands of the starting substances: the intense absorption maximum at 205 nm is a superposition of the absorption maxima characteristic of cocamide diethanolamine, CDEA-Ba and POM_{ox}; the shoulder of average intensity at 240-245 nm can be characterized as a shift to the long-wavelength region of the shoulders of CDEA-Ba and the oxidative form of molybdenum polyoxometalate; the shoulders of average

intensity at 270-275 and 280-285 nm are characteristic of CDEA-Ba; a medium-intensity absorption shoulder at 290-300 nm is characteristic of coconut oil diethanolamide solutions at a given pH; a low-intensity absorption maximum is characteristic of both cocamide diethanolamine and POM_{ox}; two weak absorption maxima at 340 and 350 nm are characteristic of CDEA.

The UV spectra of the compound at the acidity of the studied solution, pH = 6, are characterized by the following absorption bands: an intense absorption maximum at 210 nm, characteristic of cocamide diethanolamine, CDEA-Ba, and POM_{ox}; an absorption shoulder of medium intensity at 270-285 nm, characteristic of solutions of coconut oil diethanolamide at this pH; low-intensity absorption maxima at 320 and 330 nm, which are characteristic of POM_{ox} and CDEA-Ba.

The following absorption bands are observed in the UV absorption spectra of the supramolecular compound at pH = 8: a high-intensity absorption maximum at 200 nm characteristic of solutions of coconut oil diethanolamide and CDEA-Ba; a medium-intensity absorption shoulder at 225-230 nm is characteristic of POM_{ox}; medium-intensity absorption shoulders at 270-275, 280-285 and 290-295 nm are characteristic of cocamide diethanolamine and CDEA-Ba; a low-intensity absorption maximum at 310 nm is characteristic of the oxide form of molybdenum polyoxometalate and CDEA-Ba; low-intensity absorption maxima at 335 to 345 nm are characteristic of CDEA and CDEA-Ba.

For supramolecular compounds of the composition CDEA-Ba-POM_{ox} at pH = 10, the following absorption bands are characteristic: a high-intensity absorption shoulder at 200-205 nm is characteristic of solutions of cocamide diethanolamine, CDEA-Ba, and POM_{ox}; medium-intensity absorption shoulders at 270-275 and 280-285 nm are characteristic of coconut oil diethanolamide and CDEA-Ba; a low-intensity absorption shoulder at 310-320 nm is characteristic of the chromophore system of cocamide diethanolamine, CDEA-Ba, and POM_{ox}; a low-intensity absorption maximum at 350 nm is characteristic of CDEA-Ba.

As a result of the formation of supramolecular substances CDEA-Ba-POM_{ox}, an increase in optical density is observed while maintaining the absorption maxima of the starting substances, which indicates the ionic-associative nature of the bond in the obtained supramolecular substance. The presence of an ionic bond indicates the presence of electroactivity in this substance, which can be used in the development of electrochemical sensors based on it. In general, for supramolecular compounds CDEA-Ba-POM based on the oxidative form of polyoxomolybdate, a significant increase in absorption is observed at the corresponding wavelengths than to compounds of the CDEA-POM composition.

Similar studies were conducted for the reduced form of polyoxometalate (Figure-5).

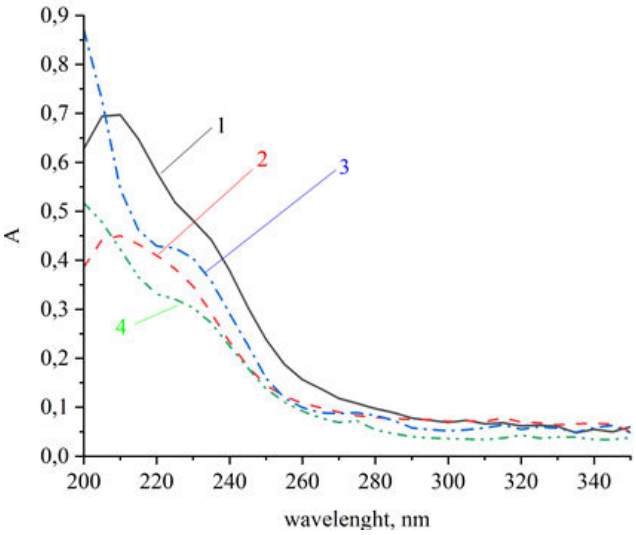

Figure-5. UV absorption spectra of CDEA-Ba-POM_{red} depending on pH:1 - pH = 4; 2 - pH = 6; 3 - pH = 8; 4 - pH = 10.

Table-3 shows the characteristics of the UV spectra of the supramolecular substance based on CDEA and the oxidative form of polyoxomolybdate, depending on the acidity of the studied solution.

Table-3. Spectral characteristics of CDEA-Ba-POM_{red} depending on the pH.

pH of the solution	wavelength, nm	$\varepsilon_{exp.}$, l·mol^{-1}·cm^{-1}
4	205-210 (shoulder)	208400
	270-275 (shoulder)	35400
	295	21900
	340	16500
6	210	135000
	260-270 (shoulder)	29700
	290-295 (shoulder)	22500
	315	23400
8	200	261500
	220-225 (shoulder)	128000
	255	35700
	270-275 (shoulder)	26100
	315	19200
	345	18900
10	200	155200
	265-275 (shoulder)	23400
	320	12900
	350	11400

As evidenced by the experimental data obtained for the supramolecular substance of the composition CDEA-Ba-POM_{red} at pH = 4, the following are characteristic: a high-intensity absorption shoulder at 205-210 nm is characteristic for solutions of cocamide diethanolamine, CDEA-Ba, and the reduced form of molybdenum polyoxometalate; a medium-intensity absorption shoulder at 270-275 nm is characteristic for the CDEA-Ba solution; low-intensity absorption maxima at 295 and 340 nm are characteristic for coconut oil diethanolamide at this pH.

At pH = 6, the following absorption bands are characteristic for supramolecular substances: a high-intensity absorption maximum at 210 nm is characteristic for solutions of CDEA, CDEA-Ba, and the reduced form of molybdenum polyoxometalate; a medium-intensity absorption shoulder at 260-270 nm is characteristic of CDEA-Ba solutions; a low-intensity absorption shoulder at 290-295 nm is characteristic of coconut oil diethanolamide and CDEA-Ba solutions; a low-intensity absorption maximum at 315 nm is characteristic of CDEA-Ba and POM_{red} solutions, and a low-intensity absorption maximum at 340 nm is characteristic of CDEA-Ba.

The UV absorption spectrum of the supramolecular substance at pH = 8 contains the following absorption bands: a high-intensity absorption maximum at 200 nm is characteristic of cocamide diethanolamine and CDEA-Ba solutions; a high-intensity absorption shoulder at 220-225 nm is characteristic of the reduced form of molybdenum polyoxometalate; a medium-intensity absorption maximum at 255 nm and an absorption shoulder at 270-275 nm are characteristic of CDEA and CDEA-Ba; a low-intensity absorption maximum at 315 nm is characteristic of CDEA and POM_{red}; a low-intensity absorption maximum at 345 nm is characteristic of CDEA and CDEA-Ba solutions. The following absorption bands are characteristic of a supramolecular substance at pH = 10: a high-intensity absorption maximum at 200 nm is characteristic of cocamide diethanolamine, CDEA-Ba, and the reduced form of molybdenum polyoxometalate; a medium-intensity shoulder at 265-275 nm is characteristic of CDEA-Ba; a low-intensity absorption maximum at 320 nm is characteristic of POM_{red}; a low-intensity absorption maximum at 350 nm is characteristic of CDEA-Ba solutions.

Since the study of the spectral characteristics of supramolecular complexes of coconut oil diethanolamide, barium salts, and polyoxomolybdates shows a significant increase in absorption while maintaining the main absorption bands of the starting substances, it can be concluded that the obtained supramolecular complexes are associated with an ionic nature of the bond, and therefore should be characterized by the presence of electroactivity.

CONCLUSIONS

a) The effect of solution acidity on the properties of cocamide diethanolamine and its complex with barium salts was studied by UV spectrophotometry. When the CDEA-Ba complex is formed, a shift in absorption bands is observed in comparison with CDEA, which indicates a change in the chromophore system.

b) The synthesis reaction of supramolecular substances of cocamide DEA with barium salts and various polyoxomolybdates was studied using the UV spectroscopic method, depending on the acidity of the solution. The associative nature of the interaction of the reacting compounds was confirmed.

c) When using barium salts in the synthesis reaction of supramolecular substances and the formation of substances of the CDEA-Ba-POM composition, a significant increase in absorption is observed in comparison with substances of the CDEA-POM composition, which can affect the electroactive properties of the obtained supramolecular substances and can be used to improve the electrode characteristics of potentiometric sensors sensitive to CDEA.

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