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Synthesis, Characterization and CMC Determination of Surfactant-Cobalt(III) Complexes

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ABSTRACT

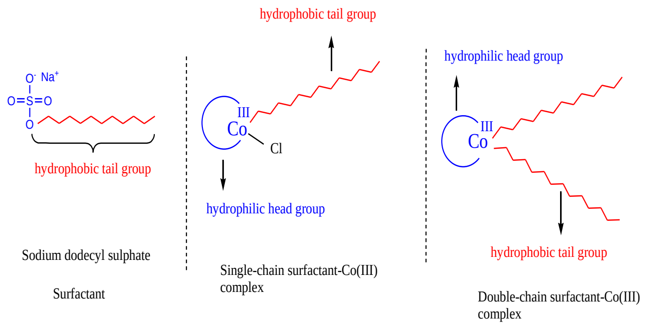
A number of mixed ligand surfactant-Cobalt(III) complexes of the type cis-$[Co(phendione)_2AX]^{2+}$ and cis-$[Co(phendione)_2A_2]^{3+}$ (phendione = 1,10-phenanthroline-5,6-dione, A = dodecylamine or cetylamine, X = Cl) were synthesized and characterized by elemental analysis, UV–Vis, Infra-red and NMR spectroscopy methods. The critical micelle concentration (CMC) values of these complexes in aqueous solution were obtained from conductance measurements. The specific conductivity data (at 303, 308 and 313 K) served for the evaluation of the temperature dependent CMC and the thermodynamics of micellization (ΔG^0_{mic}, ΔH^0_{mic}, ΔS^0_{mic}).

1. Introduction

Studies on the chemistry of surfactant-metal complexes have received a sustained high level of attention from the scientific community for the last decades [1–3], due to their relevance in various redox processes in biological systems. Self-assembly in coordination chemistry consequently provides an important and powerful entry into supramolecular engineering and the associated fields of solid-state and crystal engineering [4, 5]. It potentially also affords novel catalytic systems [6] which may ultimately be induced to offer the selectivity and usefulness of biological catalysts. Recently, there has been a growing interest in the study of processes under conditions globally referred to as restricted geometry conditions, that is under conditions in which a given ligand is forced (or different ligands are forced) to remain totally or partially bound at the surface of some substrate. In a broad sense, the restricted geometry conditions encompass phenomena such as homogeneous, heterogeneous and enzymatic catalysis [1, 7], reactivity in micellar systems and microemulsions [3], molecular machines [8], molecular electronics [9], and trapping of substrates by polyelectrolytes [10], conformational changes of DNA induced by the binding of solutes [11] and so forth. Numerous studies have been performed addressing the dependence of electron transfer on different environments including metalloproteins [12], Vitamin B_{12} [13], liquids [14, 15], micelles [16], vesicles [17] and DNA [18]. It is well known that amphiphilic molecules are characterized by their dual nature. On the same molecule two differentiated parts, the hydrocarbon (hydrophobic) and the ionic (hydrophilic) are found. This general structure, characteristic of surfactants, is responsible for the self-assembly process in solution.

Surface active materials are major building blocks of many physical, chemical and biological systems. They have been introduced into several commercial products such as antiseptic agents in cosmetics and as germicides [1], and also have found a wide range of applications because of their unique solution properties such as detergency, solubilization and surface wetting capabilities, in diverse areas such as mining, petroleum and pharmaceutical industries. It has been observed that several redox reactions in micellar media were influenced by the hydrophobic and electrostatic forces and for a given set of reactions the observed rate depends on the extent of association between the reactants and micellar aggregates [19–23]. A growing number of applications have been developed to study surfactant-metal complexes in this content. They are a good enzymatic model for the catalytic hydrolysis of carboxylic acid esters, since they mimic not only the active center of the metalloenzyme but also the microenvironment of the metalloenzyme. In the course of studies on the effects of surfactant-metal complexes on chemical reactions, catalytic effects were observed above a critical micelle concentration (CMC). Such systems offered the possibility to investigate the effects of the local reactant concentrations in the micellar sub phase, as well as the local microenvironment on the reactivity [24–28]. We have been interested in the synthesis and micelle forming properties of surfactant metal complexes containing lipophilic ligands for a long time [29–33]. As in biology, such compounds may exhibit novel physical and chemical properties with interesting and useful associated applications. Studies on the chemistry of metallomicelles have received a sustained high level of attention due to their relevance in various redox processes in biological systems and act as promising agents for antitumor [34], anthelmintic [35], antiparasitic [36], antibiotics [37] and antimicrobial activities [18], and a boost of interest because of their multiple applications in fields such as medicine [38], magnetic resonance imaging [39] and drug delivery [40].

In spite of the greatest effort and success in the study of surfactant-Cobalt(III) complexes, such complexes still attract much attention due to their interesting properties and the relative simplicity of their synthesis Surfactant-Cobalt(III) complexes are a special type of surfactant, where a coordination complex (containing a central metal ion with surrounding ligands coordinated to the metal) acts as the surfactant (Scheme 1).



Scheme 1 Structure of surfactant and surfactant-Cobalt(III) complexes

Like any other well-known surfactant, e.g. sodium dodecyl sulfate (SDS), these surfactant–metal complexes also form micelles at a specific concentration called critical micelle concentration (CMC) in aqueous solution. In all these surfactant-metal complexes, the metal complex entity containing the central metal ion with its primary coordination sphere acts

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as the head group and the hydrophobic entity of one or more ligands act as a tail part. Recently, there has been increasing interest in the use of these organized media to study the fundamental photochemical reactions of metal complexes in relevance to the conversion of solar energy into other useful forms of energy through photochemical reactions [41, 42]. As a part of our studies on transition metal-based surfactants [29–33] in this paper we report the synthesis, characterization and CMC determination of some more novel surfactant-Cobalt(III) complexes.

2. Experimental Methods

2.1 Materials

All the reagents were of analytical grade (Aldrich and Merck). Ultrapure water, obtained by deionising distilled water (conductivity < 10^{-6} Sm^{-1}) using a Milli-Q Reagent Grade water system, was used for preparative work and to make up solutions for all physical measurements.

2.2 Instrumentation

Electronic absorption spectra were taken in 10^{-3} M perchloric acid medium recorded in Varian Cary 500 Scan UV-Vis-NIR spectrophotometer. Infrared spectra (IR) were recorded on a Jasco FT/IR- 460 plus spectrometer using the potassium bromide discs method. NMR spectra were recorded in appropriate deuterated solvents using a Bruker AC 300F (300 MHz) spectrometer with TMS as internal reference. C, H and N were estimated using a Perkin–Elmer 2400 CHN instrument. Conductance measurements were carried out in aqueous solutions of the surfactant-complexes with an Elico digital conductivity bridge type CM82 and a dip-type cell with a cell constant of 1.0.

2.3 Synthesis of Single Chain Surfactants-Cobalt(III) Complexes

2.3.1 Cis-$[Co((phendione)_2)_2(C_{12}H_{25}NH_2)Cl](ClO_4)_2$

Cis-$[Co(phendione)_2Cl_2]Cl$, (3 g) was dissolved in water (10 mL). To this solution dodecylamine (1 mL), where the amine used was not sufficiently miscible with (soluble in) water, was first mixed with ethanol (2 mL) and then added drop by drop over a period of 30 min. The brown-red colour solution gradually became pale during the reaction. The mixture was set aside at 303 K for 2 days until no further change was observed. Afterwards a saturated solution of sodium perchlorate in very dilute perchloric acid was added. Slowly a pasty solid mass was separated out and it was filtered off, washed with small amounts of alcohol followed by acetone, and then it was dried over air. The semi-dried solid was further dried in a drying pistol over fused calcium chloride and stored in vacuum desiccator.

2.3.2 cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)_2Cl](ClO_4)_2$

cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)Cl](ClO_4)_2$ complex was synthesized by the ligand substitution method, replacing the chloride ion with cetylamine from their corresponding parent complexes [29,43–45] adopting same method as described above for the synthesis of the complex cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)Cl](ClO_4)_2$.

2.4 Synthesis of Double-Chain Metallosurfactants of Cobalt(III) Complexes

2.4.1 cis-$[Co((phendione)_2)_2(C_{12}H_{25}NH_2)_2](ClO_4)_3$

cis-$[Co(phendione)_2Cl_2]Cl$, (3 g) was dissolved in water (10 mL). To this solution dodecylamine (2 mL), where the amine used was not sufficiently miscible with (soluble in) water, was first mixed with ethanol (2 mL) and then added drop by drop over a period of 30 min. The brown-red colour solution gradually became pale during the reaction. The mixture was set aside at 303 K for 3 days until no further change was observed. Afterwards a saturated solution of sodium perchlorate in very dilute perchloric acid was added. Slowly a pasty solid mass was separated out and it was filtered off, washed with small amounts of alcohol followed by acetone, and then it was dried over air. The semi-dried solid was further dried in a drying pistol over fused calcium chloride and stored in vacuum desiccator.

2.4.2 cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)_2](ClO_4)_3$

cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)_2](ClO_4)_3$ complex was synthesized by the ligand substitution method, replacing the chloride ion with cetylamine from their corresponding parent complexes [29,43–45].adopting same method as described above for the synthesis of the complex cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)Cl](ClO_4)_2$.

Safety note. Caution: Perchlorate salts of Co III complexes containing organic ligands are potentially explosive! Although we have experienced no problems with the compounds reported in this work, they should only be handled in small quantities and never scraped from sintered glass frits nor heated in the solid state.

2.5. Elemental Analysis

The purity of the complexes was checked by the analysis of carbon, hydrogen, nitrogen, cobalt and halogen present in them. C, H and N were estimated using a Perkin Elmer 2400 CHN instrument.

2.6 Estimation of Cobalt

A known weight of the complex was taken in a clean dried conical flask and 5 mL of concentrated hydrochloric acid was added to it along with a small piece of tin metal. The contents were heated until a blue colour was obtained. The contents were then transferred quantitatively to a 25 mL standard flask and made up to the mark with concentrated hydrochloric acid. The cobalt concentration was found out from the absorbance value [46] by assuming the molar absorption coefficient of $[CoCl_4]^{2+}$ at 691 nm as 561 $M^{-1}cm^{-1}$.

2.7 Determination of CMC

CMC values of the complexes were calculated using electrical conductance data measured using a digital conductivity meter (Elico CM82). The conductivity cell (dip-type with a cell constant of 1.0) was calibrated with KCl solutions in the appropriate concentration range. The cell constant was calculated using molar conductivity data for KCl published by Barthel et al., [47]. Various concentrations of surfactant-Co(III) complexes were prepared in the range 10^{-6}–10^{-2} $moldm^{-3}$. All measurements were performed in a double-walled glass container, which was maintained at the desired temperature (± 0.1 K) using a circulating water bath. The conductivity of these solutions was measured at 303-313 K. The conductance was measured after thorough mixing and temperature equilibrating at each dilution. The measurement was started with a dilute solution and the subsequent concentrated solutions were prepared by adding a previously prepared stock solution. The establishment of equilibrium was checked by taking a series of reading after 15 min intervals until no significant change occurred.

3. Results and Discussion

3.1 Spectroscopic Characterization

3.1.1 Infrared Spectra

For the assignment of a geometrical configuration to these complexes, infrared spectroscopy was used. Several attempts have been made to relate differences in infrared spectra to geometrical configuration of complexes. Various workers have employed [48–50] the NH_2 deformation mode in the 1700–1500 cm^{-1} region, CH_2 rocking mode in the 900–850 cm^{-1} and Co–N stretching mode in the 610–500 cm^{-1} region to distinguish between the cis and Trans isomers of surfactant-Cobalt(III) complexes. The cis-isomers always show two peaks, whereas the trans-isomers usually have only one. Strukl and Walter [51] and Schilt and Taylor [52] studied the infrared spectra of several polypyridyl complexes. They observed that the spectral shape of the complexes were quite similar, taking into account the similarity of both ligands. In the spectra of the phendione complexes, strong bands were observed in three frequency regions namely between 700 and 900 cm^{-1}, between 1100 and 1250 cm^{-1} and between 1718 and 1687 cm^{-1}. Strong bands in the 723–897 cm^{-1} region were attributed to the aromatic hydrocarbons of the phendione ring. The band near 714 cm^{-1} was assigned to the out of the plane motion of hydrogen atoms on the heterocyclic rings and the band near 826 cm^{-1} to the hydrogens on the centre ring. The multiple splitting which these bands exhibit in most of the spectra probably arise from out-of-plane hydrogen motion other than the one in which all atoms move in phase, and also possibly from overtones of low lying fundamentals in resonance.

Only three strong bands are present, one near 750 ascribed to out of plane bending of ring hydrogens, and one near 1450 cm^{-1} which is probably a ring frequency, and a ring frequency near 1550 cm^{-1}. Numerous weak bands were observed between 900 and 1400 cm^{-1}. The C=C and C=N stretching vibrations of the heterocyclic ring stem exhibit in the region at 1420, 1465, 1570 and 1590 cm^{-1}. Perchlorate bonds at ca. 1120, 935, 640 and 440 cm^{-1} belong to an ionic species; this means that this counter-ion is not involved in the cobalt-ligand coordination [53]. The IR spectroscopic data therefore clearly indicate a cis configuration for the surfactant-Cobalt(III) complexes.

3.1.2 ^1H NMR Spectra

The ^1H NMR spectra offer a conclusive proof of the configuration of the isomers in solution. The methylene protons of the long chain moieties

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(dodecylamine/cetylamine) give rise to a multiplet usually at 1.2–1.8 ppm, whereas the terminal methyl group of the hydrocarbon chain substituent gives a triplet at 0.84 ppm. It is well established that bis-type surfactant-Cobalt(III) complexes of phendione take only the cis-configuration because of the repulsion between the ligands. The cis configuration of all the present complexes was confirmed by the ^{1}H NMR. The spectra of the phendione complexes exhibit patterns similar to those of the phenanthroline complexes 7.65, 8.42 and 8.97. The assignment of the phendione complexes were made in a manner similar to the case of their respective parent complexes [54, 55].

3.1.3 Electronic Absorption Spectra

The wavelength of the first absorption maximum in aqueous perchlorate medium for each complex is given in Table 1. It has been observed that the absorption spectra [56, 57] of the cis-$[Co(phendione)_2(A)X]^{3+}$ cations (X = Cl) showed some differences in the longest wavelength absorption band (d–d transition).

Table 1 Microanalysis and visible spectra of some surfactant-Cobalt(III) complexes

Surfactants	λ_{max}	Yield	%Calculated (Found)			
	$(\varepsilon)^a$	(%)	Co	C	H	N
cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)Cl](ClO_4)_2$	342 (91)	56	6.96 (7.11)	51.47 (51.62)	4.67 (4.80)	8.33 (8.39)
cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)_2](ClO_4)_3$	337 (93)	59	5.37 (5.43)	52.92 (53.31)	6.10 (6.19	7.71 (7.74)
cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)Cl](ClO_4)_2$	359 (98)	63	6.53 (6.64)	53.60 (53.85)	5.28 (5.33)	7.81 (7.92)
cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)_2](ClO_4)_3$	344 (102)	67	4.87 (4.99)	55.96 (56.21)	6.87 (6.96)	6.99 (7.08)

^{a} λ in nm; ε in $M^{-1}cm^{-1}$

The Trans form showed a lower intensity (e < 50 $M^{-1}cm^{-1}$) whereas the cis form showed a higher intensity (e > 70 $M^{1}cm^{1}$). This observation can be used to determine the geometrical configuration of the present cations, whose longest wavelength absorption maxima are shown in Table 1. Similarly, the position of λ_{max} suggests that the strength of the bidentate ligands. The lower λ_{max} values for the complexes containing the phendione ligand indicate that in these complexes the basal planes are comparatively more distorted than in the bpy containing complexes. By comparing the spectra of the corresponding butylamine complexes it reveals that both the ordinary and the surfactant complexes have the same structure and the replacement of lower aliphatic amine ligands by long chain aliphatic amine ligands does not affect the symmetry of the surfactant complexes. Consequently, it does not seem that micellization greatly influences the structure of the polar head.

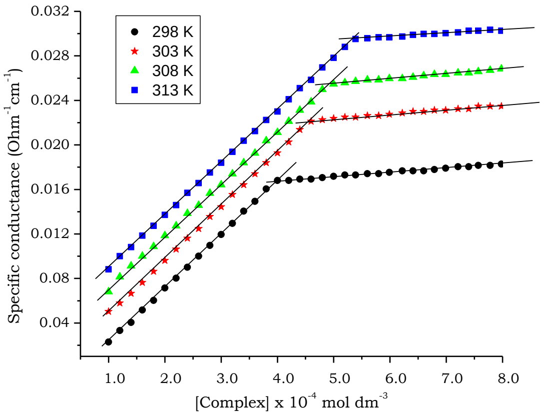

Fig. 1 Electrical conductivity *vs* [Co(III)] in aqueous solution

3.2 Critical Micelle Concentration Values (CMC)

The CMC values were computed from the slope of [Co(III)] versus specific conductance data. The complex concentration at which the micellization starts was evident from the change in the slope of the plot and that particular concentration is the CMC under the experimental conditions. The CMC values were determined at three different temperatures (303, 308 and 313 K). At all temperatures a break in the conductance versus concentration plots, characteristic of micelle formation, was observed. The CMC values were determined by fitting the data points above and below the break to two equation of the form y = mx + c and solving the two equations simultaneously to obtain the point of intersection. Least-squares analysis was employed and correlation coefficients were greater than 0.98 in all the cases. The conductivity measurements at three different temperatures were repeated three times and the accuracy of the CMC values (Table 2) was found to be within ±3% error. Fig. 1 illustrates the plot for the complex cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)Cl](ClO_4)_2$; similar plots (not shown) were obtained for all the remaining complexes. It is observed that the increase in the alkyl chain on polar head group, the CMC shows a decrease. This may be due to an increase in hydrophobic character of the molecule in the coordination sphere with that of dodecyl/cetylamine.

Table 2 CMC values of some surfactant-Cobalt(III) in aqueous solution

Complexes	CMC (moldm^{-3})				ΔG^0_{mic} (kJmol^{-1})	ΔH^0_{mic} (kJmol^{-1})	$T\Delta S^0_{mic}$ (kJmol^{-1})
	298 K	303 K	308 K	313 K			
cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)Cl](ClO_4)_2$	3.95 x 10^{-4}	4.54 x 10^{-4}	4.93 x 10^{-4}	5.38 x 10^{-4}	−27.34 ± 0.1	−15.46 ± 0.3	17.17 ± 0.1
cis-$[Co(phendione)_2(C_{12}H_{25}NH_2)_2](ClO_4)_3$	4.55x 10^{-4}	4.91 x 10^{-4}	5.32 x 10^{-4}	5.67 x 10^{-4}	−29.84 ± 0.3	−18.62 ± 0.1	16.25 ± 0.2
cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)Cl](ClO_4)_2$	7.17 x 10^{-4}	7.41 x 10^{-4}	7.68 x 10^{-4}	7.91 x 10^{-4}	−33.43 ± 0.2	−21.85 ± 0.1	14.38 ± 0.2
cis-$[Co(phendione)_2(C_{16}H_{33}NH_2)_2](ClO_4)_3$	7.52 x 10^{-5}	8.09 x 10^{-5}	8.34 x 10^{-5}	8.65 x 10^{-5}	−36.24 ± 0.1	−25.49 ± 0.2	11.95 ± 0.1

3.3 Thermodynamics of Micellization

The study of CMC versus temperature is often undertaken to obtain information on hydrophobic and head group interactions. This involves deriving various thermodynamic parameters of micelle formation. Two models are generally used, the mass-action or equilibrium model and the phase separation or pseudo-phase model. The mass-action model assumes that an equilibrium exists between the monomeric surfactant and the micelles. The phase separation model, assumes that the aggregates have their counterions in the surrounding atmosphere in a separate phase [41-43]. According to these models, the standard Gibbs free energy of micelle formation per mole of monomer, ΔG_m^0, is given by

$$\Delta G_m^0 = RT\,(2-\alpha_{ave})\ln CMC \qquad (1)$$

where R, T and α_{ave} are gas constant, absolute temperature and average degree of micellar ionization, respectively.

The enthalpy of micelle formation can be obtained by applying the Gibbs-Helmholtz equation to the Eq. (1),

$$\Delta H_m^0 = -\,RT^2\,(2-\alpha_{ave})\,d\ln CMC\,/\,dT \qquad (2)$$

Once the Gibbs free energy and the enthalpy of micelle formation are obtained, obviously, the entropy of micelle formation can be determined by,

$$\Delta S_m^0 = (\Delta H_m^0 - \Delta G_m^0)\,/\,T \qquad (3)$$

The thermodynamic parameters of micellization for the cationic surfactant are compiled in Table 2. The observed more negative Gibbs free energy of micellization indicates more favored micellization for the system under study when compared to hexadecyltrimethylammonium bromide (CMC = 9.0 x 10^{-4} moldm^{-3}). Moreover, since the changes of CMC with temperature are small, the value of ΔH_m^0 and ΔS_m^0 must be rather inaccurate and should be considered as only approximate. Nuselder and Engberts [44] have suggested that for negative ΔH_m^0 values, London-dispersion forces play major role in the micelle formation. Positive values of ΔS_m^0 clearly indicate that the micellization of the studied surfactants in aqueous solution is governed mainly by hydrophobic interactions between the surfactant cations resulting in the breakdown of the structured water surrounding the hydrophobic groups and indicates that the cationic surfactants formation is entropy driven process. The observed increase in the entropy values indicates that increasing head group polarity favors the micellization process. As mentioned in our previous reports [33-35], the CMC values for surfactant-Co(III) in the present study are also very low compared to that of simple organic surfactants. Thus it is suggested that these metal surfactant complexes have more capacity to associate themselves forming aggregates compared to those of ordinary

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synthetic organic surfactants. Moreover, introduction of a metal complex to the hydrophilic part of the amphiphile can remarkably enhance the ability of aggregation.

4. Conclusion

The mixed ligand surfactant-Cobalt(III) complexes containing 1,10-phenanthroline-5,6-dione and dodecylamine or cetylamine were synthesised. The surfactant-Cobalt(III) complexes were characterized adopting various spectroscopic techniques and elemental analysis. The critical micelle concentration (CMC) values of these complexes in aqueous solution were obtained from conductance measurements. The specific conductivity data (at 298, 303, 308 and 313 K) served for the evaluation of the temperature dependent CMC and the thermodynamics of micellization ((ΔG^0_{mic}, ΔH^0_{mic}, ΔS^0_{mic}). Increase in the alkyl chain on polar head group, the CMC shows a decrease. Surfactant-Cobalt(III) complexes have more capacity to associate themselves forming aggregates, compared to those of ordinary synthetic organic surfactants.

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