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International Journal of Current Research
Vol. 9, Issue, 07, pp.54595-54599, July, 2017

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

ISSN: 0975-833X

RESEARCH ARTICLE

STUDIES ON COMPLEXATION, ACCURACY, PRECISION OF CAS, PCV WITH CTAB IN HEAVY METAL IONS, DETERMINATION

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ARTICLE INFO

Article History:
Received 29th April, 2017
Received in revised form
11th May, 2017
Accepted 16th June, 2017
Published online 31st July, 2017

Key words:

Pyrocatechol Violet,
Cetyl trimethyl ammonium bromide,
Sensitivity,
Precision,
Accuracy.

ABSTRACT

Study of coordination compounds of heavy metal ions is of special interest since past due to their growing applications in science and technology. Complexation behavior of Triphenyl-Methane Dyes viz. Chrome Azurol-S,CAS; and Pyrocatechol Violet PCV, in the presence and absence of surfactant Cetyl Trimethyl Ammonium Bromide has been found to form stable water soluble pink colored complexes. CTAB have shown to increase sensitivity of the color reactions of these complexes with greater solubility and higher stability. The addition of some heavy metal ions such as Cu(II), Ni(II), Cd(II), Hg(II), and Pb(II), to these sensitized modified reagents resulted in the formation of intense, stable colored complexes. The composition of the complexes of metal ions under study remains same in both dyes CAS and PCV as 1:1, both in the absence and presence of CTAB except in case of Cd(II). In case of Cd(II) with CAS it is 1:1 and it changes as 1:2 in presence of CTAB. Dissociation constants pK values of dye- surfactant modified reagents has been calculated. Various analytical parameters including rate of color formation, effect of temperature and stability of color formation, range of adherence to Beers Law, Molar Extinction constants, Sensitivity, were studied for all systems in absence as well as in presence of CTAB.A simple, rapid, highly sensitized stable method has been proposed. Precision and Accuracy of the method suggested for microdetermination of metal ions was determined which shown suggested methods are both precise and accurate.

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Citation: Dr (Mrs.) Suparna Deshmukh, 2017. "Studies on complexation, accuracy, precision of CAS, PCV with CTAB in heavy metal ions, determination", *International Journal of Current Research*, 9, (07), 54595-54599.

INTRODUCTION

The property of formation of colored complexes has been successfully applied for the spectrophotometric estimation of metal ions since last few decades. Reaction of triphenylmethane dyes with 4f and 5f metal ions have been a subject matter of study by several workers (Jarosz, 1988; Shtykov *et al*., 1979; Chernova *et al*., 1979; Cerkova, 1982; West *et al*., 1968). The systematic design of surfactants induced dye metal interactions leading to the sensitized photometric metal ion determination could obviously be facilitated by an accurate model of detail chemistry involved (Mittal and Procd, 1988; Vekhande and Munshi, 1973; Microchem, 1978). Complexation of Cu(II), Fe (II) & Al (III) with Chrome Azurol S in the presence of non ionic surfactants was studied (Chernova, 1977) The present investigation undertaken involves a detail study of the interaction of surfactant Cetyl Trimethyl Aminonium Bromide with triphenylmethane dye Chrome Azurol S (CAS) and Pyrocatechol Violet (PCV). The dye surfactant complexes thus formed was used to study complexation reactions of Cu (II), Ni (II), Cd (II) Hg(II), and Pb(II), and compared with the complexation reaction of these metal ions with Chrome Azurol S and Pyrocatechol Violet in absence of detergents.

MATERIALS AND METHODS

The absorption measurements were done on UV Shimadzu – UV 240 Spectrophotometer. Distilled water blanks were used for pH measurements Elico pH meter LI-10 with glass calomel electrode system was used after standardization. All the reagents used were of BDH Anala R grade purity. The surfactant Cetyl trimethyl ammonium bromide (CTAB) was prepared in 20% aqueous methanol in double distilled water and were standardized by usual procedures. The dye solution was also prepared in double distilled water by dissolving its purified samples and the standard solutions of heavy metals under study were prepared by dissolving analytical grade metal salts in 5% HNO_3.

Procedure

Preparation of mixtures, measurements of absorbance, adjustment of pH etc. were carried out at room temperature. In all the experiments CTAB solution was added to the reagent solutions which was kept for at least 20 minutes for maximum

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decolorizing effect to which metal ions solution was then added. The absorbance readings were recorded only after 30 minutes of additions of the reactants a time necessary for equilibration.

RESULTS AND DISCUSSION

Absorption Spectra

Absorption Spectra of CAS and PCV solution was recorded from pH 1.0 to 12.0. The spectral studies were carried out wit in the presence of ten times by volume of surfactant, CTAB at different values of pH 1.0 to pH12. The λmax values in the absence of, as well as in the presence of CTAB was found to be as given below in Table - 1. For studying the absorption spectra of CAS and PCV, 1.0 ml of 1.0 x 10^{-3} M solution was taken in different volumetric flasks and their pH was adjusted & from 1.0 to 12.0 by using HCl, and NaOH, after which total volume was made upto 25ml with distilled water. The hypsochromic shift in the presence of CTAB was observed in almost all pH values. This was attributed due to formation of dye / surfactant complex.

Table 1. λmax of CAS at different pH values

pH	In absence (nm)	In presence of CTAB (nm)
1.0	470 nm	490
2.0	470	500
3.0	490	500
4.0	490	470
5.0	435	420
6.0	430	415
7.0	420	415
8.0	420	415
9.0	420	415
10.0	420	415
11.0	420	415
12.0	600	610

Table 2. λMax of PCV in the presence and absence of CTAB at different pH values

	λMax (nm)	
pH	In absence of surfactant	In presence of CTAB
1.0	440	440
2.0	440	440
3.0	440	440
4.0	440	440
5.0	440	440
6.0	440	605, (440)
7.0	440	605
8.0	440	605
9.0	590	490
10.0	470	490
11.0	470	490
12.0	500	No sharp peak is obtained

pH 5 & λmax 420 nm is thus selected for further analytical studies of CAS while that of PCV is 435nm. The absorbance of CAS and PCV both, is found to be reduced in the presence of surfactant

Dissociation constant (pK values) of CAS

CAS has 4 replaceable protons in its molecule. One of these corresponds to -SO_3H group, two corresponds to - COOH groups while the fourth corresponds to -OH group. Thus, the following equilibrium can be written for the stepwise dissociation of CAS which represents existence of its four pk values :

$$H_4CAS \rightleftarrows H_3\,CAS^{-1} \rightleftarrows H_2\,CAS^{-2} \rightleftarrows HCAS^{-3} \rightleftarrows CAS^{4-}$$

Table 3. pK values of CAS in absence & in presence of CTAB

	CAS	CAS + CTAB
pK_1	1.80	1.57
pK_2	4.70	4.00
pK_3	7.50	7.20
pK_4	11.50	11.20

The lowering of pK values in presence of CTAB indicates their action on CAS & due to hypsochromic effect of CTAB on CAS. Following equilibrium can be written for the stepwise dissociation of PCV which represents existance of its four pk values :

$$H_4\,PCV\;\; H_a\,PCV^{-1} \overset{p\,K_1}{\rightleftarrows} H_2PCV^{-2}\text{-}HPCV^{-3} \;\; \overset{p\,K_2}{\rightleftarrows}$$

$$HPCV \;\; \overset{pK_3}{\rightleftarrows}$$

In the presence and absence of CTAB, the four pK-values obtained are as shown in table 4.

Table 4. Dissociation constants of PCV in the presence and absence of CTAB

pK values	In absence of CTAB	In presence of CTAB
pK1	5.40	4.50
pK2	6.30	6.10
pK3	8.32	8.15
pK4	11.02	10.81

Composition of CAS - CTAB complex

For determination of dye - surfactant composition, the absorption of dye was studied at pH5.0 by keeping the dye concentration same and varying the surfactant concentration.

The concentration of CAS selected were

for system A : 8.0x10^{-5}M; system B, 6.0 x 10^{-5}M & system C, 4.0 x 10^{-5} M.

The composition of dye - surfactant complex was established at a value where further addition of CTAB did not alter the absorbance of the dye. The absorbance decreases till the ratio of dye-detergent is reached to 1:1 which on further addition of CTAB remains constant. This shows a definite ratio of 1:1 of CAS : CTAB complex formed. The modified species called as dye - detergent complex & may be represented as [CAS/ (CTAB)].

Composition of PCV-CTAB Complex

Composition of PCV-CTAB complex was found out by studying. The effect of CTAB concentration on the absorbance of PCV. The experiment was carried out by keeping dye concentration constant and varying the concentrations of surfactants. The composition of PCV- CTAB complex was established at a value where further addition of CTAB did not alter the absorbance of PCV. The curves obtained by plotting the absorbance Vs. the ratio of concentration of PCV: CTAB were found to consist of two different parts. One part shows linear decrease in the absorbance values. Absorbance decreases upto a certain value of PCV: CTAB. Hence composition of PCV:CTAB is 1:1.

Absorption spectra of the complexes

Absorption spectra of CAS, CAS- metal ion CAS - CTAB and CAS - metal ion - CTAB were recorded at the pH of study. The nature of complexes found between CAS and Cu(II), Ni (II),Cd (II),Hg(II), & Pb(II) has been studied in detail. A representative absorption spectrum is shown in the fig. 1 to indicate the methodology used. The metal ions selected for the present study are susceptible to hydrolysis in alkaline range. It was thus necessary to study the complex formation in the acidic range of pH 3.0 to 6.0. The absorption spectra of metal complexes of CAS and PCV was studied in the absence as well as in the presence of CTAB. The bathochromic shift and increase in the absorbance of complexes are attributed due to the early dissociation of phenolic protons of the dye in presence of surfactant. Similar results are obtained with PCV-CTAB chelates.

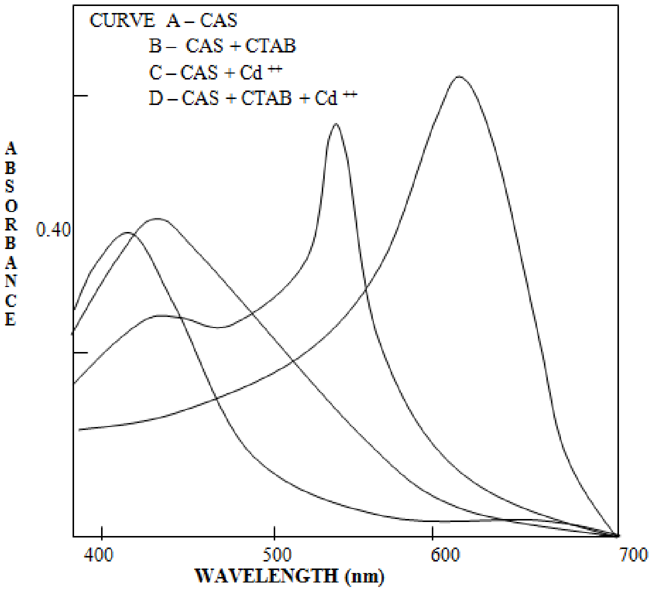

Fig.1. The absorption spectra of Cd (II) complex of CAS

Table 5. λmax of CAS, PCV and its chelates in the absence as well as in the presence of CTAB at pH 5.0 and with M : L as 1:1

System	λmax (nm)
CAS	430
CAS + CTAB	420
CAS + Cu (II)	540
CAS +CTAB + Cu (II)	625
CAS + Ni (II)	440
CAS+ CTAB + Ni (II)	615
CAS + Cd (II)	545
CAS + CTAB + Cd (II)	625
CAS + Hg (II)	440, (540)
CAS + CTAB + Hg (II)	625
CAS + Pb (II)	440, (540)
CAS + CTAB +Pb (II)	625
PCV	440
PCV + CTAB	440
PCV +Cu(II)	440
PCV+CTAB+Cu(II)	435, (630)

Composition of the chelates

The stoichiometry of complexes is ascertained by Job's method of continuous variation at the pH & wavelength of study. The composition of the complexes at metal ions under study with CAS remains same in the presence and in the absence of surfactants. The reason may lie in the pH selected for study where the dissociation of phenolic hydrogen may not be much accelerated in the presence of surfactants. From spectral studies, it was found that CAS forms complexes M:L ratios either at 1:1 with metal ions Cu(II) & Ni (II) in presence as well as in absence of CTAB while with Cd (II) it is 1:2 in both presence & absence of CTAB. Similarly spectral charecteristics shows that PCV forms only one stable complex with Cu(II), in the presence of CTAB.

Stability Constant

The values of log K of chelates of metal ions under study in absence as well as in presence of CTAB are reported in Table 6

Table 6. Stability Constants of Chelates In Presence and Absence of Surfactant

Chelates	λmax (nm)	Composition M:CAS:CTAB	LogK value By Job's method
Cu(II) -CAS	540	1:1	4.7
Cu(II)- CAS-CTAB	625	1:1:1	5.5
Ni (II) - CAS	540	1:1	5.2
Ni(II)-CAS CTAB	615	1:1:1	5.6
Cd (II) - CAS	545	1:2	10.2
Cd(II)-CAS-CTAB	625	1:2:2	10.6
Hg (II)-CAS	540	1:2	10.5
Hg (II)-CAS-CTAB	625	1:2:2	11.3
Pb (II)-CAS	545	1:2	10.2
Pb (II)-CAS-CTAB	625	1:2:2	11.9
Cu (II)-PCV	440	1:1	3.2
Cu(II)- PCV- CTAB	630	1:1:2	4.6

The results showed that the values of log K in the presence of CTAB are greater than that in the absence. This is due to CTAB reacting earlier with dyes, CAS and PCV to allow an early dissociation of protons from the phenolic group which participates in the complex formation, thus allowing the attachment of the metal ion more easily at the pH of study and therefore increasing the value of stability constant. This is attributed to the tendency of decrease in pK values of dyes CAS, PCV(ligands) in the presence of surfactants.

Analytical applications

A) FOR CAS

It forms stable complexes with Cu, Ni, Cd, Hg and Pb with CTAB.In all the experiments, CTAB solution was added to CAS solution; which was then kept for at least 0.5hr for complete formation of dye - detergent complex, to which then metal ion solution was added. This sequence was fixed by carrying out experiment by varying sequences of additions, but the above one sequence showed to give constant absorbance readings. The temperature was found to have little effect on colour intensity from 25^0C to 60^0C in absence or presence of surfactants in all the systems reported here. The colour was stable up to 3 to 4 hrs after which it changes slightly.

B) FOR PCV

Pyrocatechol Violet is a TPM dye which forms complex only with Cu(II) and CTAB among heavy metal ions under study.

Under the experimental conditions, surfactant solution i.e CTAB solution was added to PCV solution and kept for half an hour for completion of the reaction. The metal ion solution is then added to this PCV -CTAB solution. The temperature was found to have no effect on the colour intensity from 20^0 – 60^{0}C in the presence of CTAB. The stability of the colour was studied at room temperature (30^{0}C), by measuring the absorbance at regular time intervals. The maximum absorbance was achieved 20-25 minutes after the action of reactants and no change was then observed atleast for 3 to 4 hrs. It was observed that for maximum color development three fold excess of PCV over metal concentration is required.

pH range of stability

For studying effect of pH on absorbance of metal chelates in absence as well as in presence of surfactants, the ratio of the dye : surfactant : metal was kept as 3:15:1 i.e. 1:5:0.33 & pH was adjusted from 3 to 6.5 using HCl and NaOH. Total volume was kept as 25ml. The pH range within which absorbance values do not change is taken as pH range of stability of coloured complexes.

Beer's Law and Photometric Ranges

The linearity between the absorbance of the chelates in the absence as well as in the present of CTAB Vs concentration of metal ions was tested by varying the metal ions concentration and keeping the reagent concentration constant at the pH and wavelength of study selected for various chelate systems. The effective photometric range was evaluated by Ringbom plots (10) a plot of log of metal ion concentration Vs. % transmittance.

Table 7. pH range of stability, Concentration Range for to Beer's Law & effective Photometric Range

pH of study = 5.0

System	λmax	pH range of stability	Range of Beer's law	Photometric Range.
Cu(II)-CAS	540	3.5-5.5	0.29-0.64	0.29-0.39
Cu(II)-CAS-CTAB	625	4.5-6.0	0.1-0.35	0.15-0.22
Ni (II) - CAS	540	4.0-6.0	0.31-0.49	0.31-0.35
Ni (II)-CAS	615	4.5-5.5	0.24-0.59	0.41-0.52
Cd(II) - CAS	545	4.5-5.5	0.45-1.50	0.69-1.25
Cd(II)-CAS-CTAB	625	4.5-5.5	0.18-0.99	0.45-0.90
Hg (II)-CAS	540	4.5-5.5	0.80-1.34	1.07-1.78
Hg (II)-CAS-CTAB	625	4.5-5.5	0.89-2.02	1.25-2.00
Pb (II)-CAS	545	5.0-6.0	1.24-4.14	1.62-3.22
Pb (II)-CAS-CTAB	625	4.5-5.5	0.74-1.84	0.91-1.99
Cu(II)-PCV	440	4.5-6.0	0.62-0.74	0.40-0.80
Cu(II)-PCV-CTAB	630	5.5-6.5	0.42-2.26	0.62-1.75

Sandell's Sensitivity and Molar Absorptivity of the System

Sensitivity of colour reaction of metal ion with CAS for log Io/It =0.0001, as defined by Sandell are listed below in Table 8.

Procedure for the Microspectrophotometric Determinations of Cu (II), Ni(II), Cd (II), Hg (II), Pb (II) with CAS and Cu(II) with PCV in the presence of surfactant CTAB

pH of the CAS and PCV solution is adjusted containing that concentration of metal ion as mentioned under the effective photometric determination in Table 7 is also adjusted to pH 5.0 and added to the CAS and PCV solution or modified CAS solution (the modified Dyes is prepared by adding five fold excess of CTAB solution to CAS solution and keeping it for half an hour for the complete formation of dye surfactant complex). Total volume was made upto 25 ml with distilled water having pH adjusted to 5.0 and the absorbance of the solution is measured at its wavelength of study against the modified reagent solution as blank. The amount of metal ion present in unknown solution can then be obtained by comparing its absorbance from the calibration curve obtained under similar conditions.

Table 8. Sandell's Sensitivity and Molar Absorptivity at pH and wavelength of study

Systems	wavelength of study (nm)	pH of study	Sandell Sensitivity SX10^{-2}	Molar absorptivity EmX10^{-3}
Cu(II)-CAS	540	5.0	0.73	13.00
Cu(II)-CAS-CTAB	625	5.0	0.20	41.70
NI(II)-CAS	540	5.0	0.78	10.40
NI(II)-CAS-CTAB	615	5.0	0.30	20.00
Cd(II)-CAS	545	5.0	1.12	10.80
Cd(II)-CAS-CTAB	625	5.0	0.63	16.70
Hg (II)-CAS	540	5.0	2.70	9.10
Hg(II)-CAS-CTAB	625	5.0	1.50	15.00
Pb (II)-CAS	545	5.0	2.10	8.93
Pb (II)-CAS-CTAB	625	5.0	1.40	19.20
Cu(II)-PCV	440	5.0	1.05	4.40
Cu(II)-PCV-CTAB	630	5.0	1.62	6.20

Precision and Accuracy

Precision and Accuracy is studied on the basis of the data obtained regarding the i) Root mean square deviation(α), ii) the most probable analytical error(€), the difference between the arithmetic mean (X), and the true or most expected value(Xt), of absorbance for the systems studied. These values are reported in Table 9.

Table 9. Precision Data for Absorbance Measurement

System	$\overline{X}$	∂	€	$\overline{X}$ -€	$\overline{X}$ +€
Cu(II)-CAS	0.793	0.008	0.0093	0.783	0.802
Cu(II)-CAS-CTAB	0.310	0.002	0.0023	0.308	0.312
Ni(II) -CAS	0.469	0.005	0.0058	0.463	0.475
Ni(II)-CAS-CTAB	0.075	0.004	0.0046	0.070	0.079
Cd(II)-CAS	0.584	0.006	0.007	0.577	0.591
Cd(II)-CAS-CTAB	0.091	0.003	0.0035	0.088	0.094
Hg(II)-CAS	0.636	0.003	0.0035	0.633	0.639
Hg(II)-CAS-CTAB	0.292	0.002	0.0023	0.289	0.294
Pb(II)-CAS	0.496	0.006	0.007	0.489	0.503
Pb(II)-CAS-CTAB	0.083	0.004	0.0046	0.078	0.087
Cu(II)-PCV	0.277	0.0045	0.0045	0.332	0.421
Cu(II)-PCV-CTAB	0.234	0.003	0.0035	0.230	0.237

Table 10. Accuracy Data of Determinations Expressed in Terms Of Δ‹€

System	$\overline{X}$	X_t	$\Delta = X_t - \overline{X}$	€
Cu(II)-CAS	0.793	0.791	-0.002	0.0093
Cu(II)-CAS-CTAB	0.310	0.311	0.001	0.0023
Ni(II)-CAS	0.469	0.470	0.001	0.0058
Ni(II)-CAS- CTAB	0.075	0.074	-0.001	0.0046
Cd(II)-CAS	0.584	0.583	-0.001	0.0070
Cd(II)-CAS-CTAB	0.091	0.090	-0.001	-0.001
Hg(II)-CAS	0.636	0.635	-0.001	0.0035
Hg(II)-CAS-CTAB	0.292	0.292	0.000	0.0023
Pb(II)-CAS	0.496	0.495	0.001	0.0070
Pb(II)-CAS-CTAB	0.083	0.083	0.000	0.0046
Cu(II)-PCV	0.351	0.344	0.002	0.0052
Cu(II)-PCV-CTAB	0.234	0.235	0.001	0.0035

Conclusion

It is observed that the stabilities of the complexes have increased in the presence of surfactants. One of the most important application of the coloured complexes is its use in the spectrophotometric determination of the metal ions with organic dyes. A good reagent has to possess properties like stability of the species, rate of colour formation, specificity of colour reactions and the sensitivity of colour reaction. The use of TPM dyes has its origin only because of these desired properties. The use of micelle forming surfactants in increasing the sensitivity of the colour reactions is one of the important aim of study. The increased sensitivity of the system in the presence of surfactants is the net advantage for microspectrophotometric determination of metal ions using triphenyl methane dyes as reagents. Statistical theoretical treatment on the systems under study reveals that the proposed method for microdetermination of metal ions under study, is both precise and accurate. These metal ions when present in water at ppm level as a pollutant thus may be detected by using this method.

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