



Extraction of Cetyltrimethyl Ammonium bromide with picric acid by spectrophotometry Method

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Abstract : A simple and sensitive spectrophotometric method was developed for the extraction and determination of cetyltrimethyl Ammonium bromide surfactants in real sample. Cationic surfactants are extracted into CH_2Cl_2 as an ion-pair with $(\text{CTA}^+(\text{NO}_2)_3\text{PhO}^-)$ (and the absorbance of the organic phase is measured at 376nm. The Beer–Lambert law was obeyed over the concentration range $100\text{--}3000 \text{ ng mL}^{-1}$ of cetyltrimethyl Ammonium bromide and the detection limit was 7.0 ng mL^{-1} . The relative standard deviations were about 2.1%. Electrolytes in real sample do not interfere. The method was applied to the determination of Cationic surfactants in real samples without interference from matrix substances.

Key words: Cetyltrimethyl Ammonium bromide, Ion-Pair, picric acid, spectrophotometry Method.

Introduction

In chemistry, ion-association is a chemical reaction whereby ions of opposite electrical charge come together in solution to form a distinct chemical entity. Ion-associates are classified, according to the number of ions that associate with each other, as ion-pairs, ion-triplets, etc. Ion-pairs are also classified according to the nature of the interaction as contact, solvent-shared or solvent-separated. The most important factor to determine the extent of ion-association is the dielectric constant of the solvent. Ion-associates have been characterized by means of vibrational spectroscopy. Ion-pairs are formed when a cation and anion come together [1, 2].



There are three distinct types of ion-pair, depending on the extent of solvation of the two ions. An alternative name for a solvent-shared ion-pair is an outer-sphere complex. This usage is common in coordination chemistry and denotes a complex between a solvated metal cation and an anion. Similarly a contact ion-pair may be termed an inner-sphere complex. The essential difference between the three types is the closeness with which the ions approach each other; fully solvated > solvent-shared > contact. With fully solvated and solvent shared ion-pairs the interaction is primarily electrostatic, but in a contact ion-pair there will also be some covalent character in the bond between cation and anion. An Ion-triplet may be formed from one cation and two anions, or from one anion and two cations [3,4]. Higher aggregates, such as a tetramer, $(\text{AB})_4$ may be formed. Ternary ion-associates involve the association of three species.[3] Another type, named intrusion ion-pair has also been characterized [4-17]. Spectrophotometry, polarography, potentiometry, conductometry, and atomistic modeling techniques have been used to better understand dye–surfactant interactions [18, 19]. Emphasis has been placed on methods that can characterize the solution structure of dye–

surfactant mixtures and how that structure correlates with the adsorption results. Two of the possible phenomena that can change adsorption profiles are dye aggregation and ion pair formation between dye and surfactant [20, 21]. Atomistic simulation techniques have been used to obtain a microscopy view of the ion pair formation and resulting solution structure and spectra of dye–surfactant mixtures. Moreover, density functional theory has been used to suggest the preferred aggregation site between the dye and the surfactant [22].

The method is used for determination of cetyltrimethyl Ammonium bromide (CTAB) in the shampoo sample by mean of extraction-spectrophotometric and good result is obtained. The method has also proved to be simple, sensitive, selective and reproducible. Against the previous methods which have been used for measuring this arterialscetyltrimethyl Ammonium bromide (CTAB), in this method there is no need of having complex equipment like HPLC and this one is a cheap and accessible method.

2. Experimental

2.1 Instrumentation

A Shimadzu 2100 UV–visible spectrophotometer (Shimadzu, Japan), A Jenway model 3510 pHmeter was used for pH measurements. An electronic analytical balance (220LA, ADAM) was used for weight the materials.

2.2. Materials

All solutions were prepared with ultra pure water (obtained from HAMILTON, England) Laboratory glass was kept overnight in a 10% (V/V) HNO₃ solution and then rinsed with deionized water. All reagents were made from Merck.

2.3. Procedure

In two 25 mL flasks, 5ml solution of picric acid 5×10^{-4} M as a blank sample and the other 5ml solution CTAB with a concentration 25 mg L^{-1} and 5 mL picric acid solution with a concentration of 5×10^{-4} M for sample moved and then diluted with distilled water. Then funnel content into two separate flasks, each containing 5 ml dichloromethane were added and stirred for one minute, the organic phase transferred to the cell separation and absorption solution, and was used in the visible wavelength (376 nm) by UV-Vis spectrophotometer.

3. Results and discussion

3.1. Influence of pH

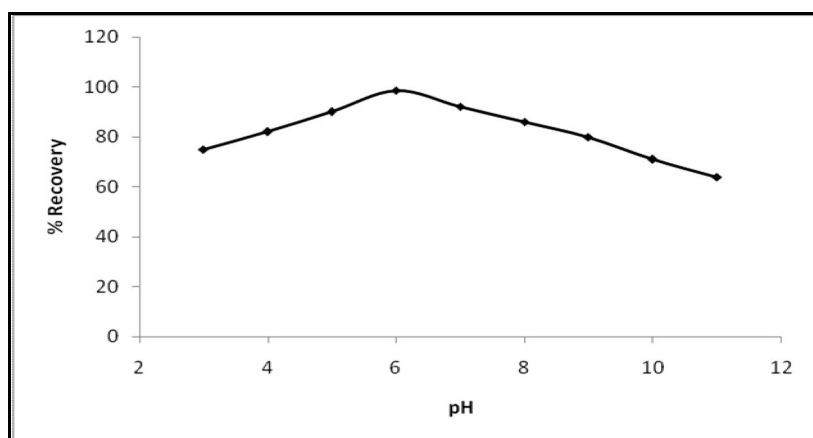


Fig. 1 Effect of pH on the extraction of paired ionic

Since this study is based on the extraction paired ion ($\text{CTA}^+ (\text{NO}_2)_3\text{PhO}^-$) is based, according to studies cationic surfactant CTAB extraction is very sensitive to pH. In order to achieve the highest concentration of

ions paired, mining operation in the buffer acidic and alkaline in the range of pH=3-12 was investigated Fig.1. The results in Fig. 1 shows that the maximum Recovery was achieved at pH of 6.0.

3.2. The effect of NaCl on the extraction of the purposed ion pair

In order to better and faster separation of organic and aqueous phases from solution of the 0.1 mol L^{-1} NaCl with volumes 0-10 mL was used in this study and its effect on the extraction of ion pair were investigated. It was observed that increasing the NaCl solution into an aqueous solution paired ions extracted into the organic phase has no significant effect on extraction (Fig.2). Also, because the organic and aqueous phases without NaCl salt also quickly separated and the increased salt no significant effect on, Thus adding to the concentration of salt NaCl aqueous solution was discarded.

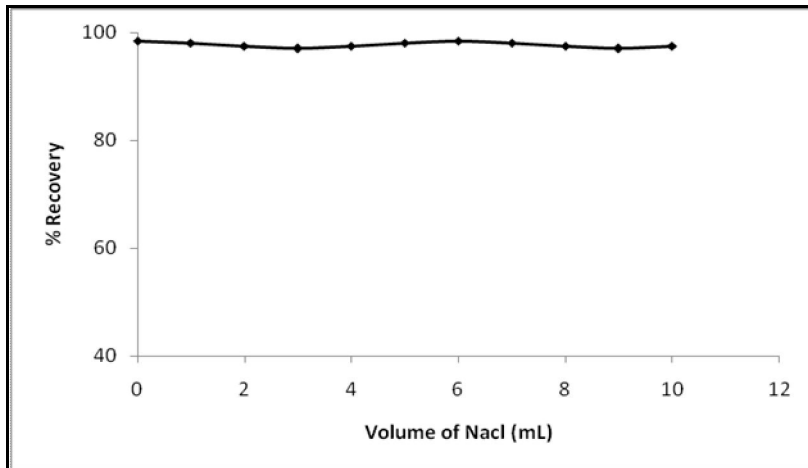


Fig.2. Effect of NaCl on the extraction

3.3. The effect of concentration and volume of picric acid anionic dyes on the extraction of purposed ion pair

In this section to determine the optimal concentration of picric acid anionic dyes have been studied in order to have maximum absorption. Test results showed that by increasing the amount of anionic dyes, the recovery increases and decreased. The volume of 1 mL of $5 \times 10^{-4} \text{ mol L}^{-1}$ of picric acid with maximum absorption for later testing as the optimal volume picric acid anion with concentration $5 \times 10^{-4} \text{ M}$ was selected (Fig.3).

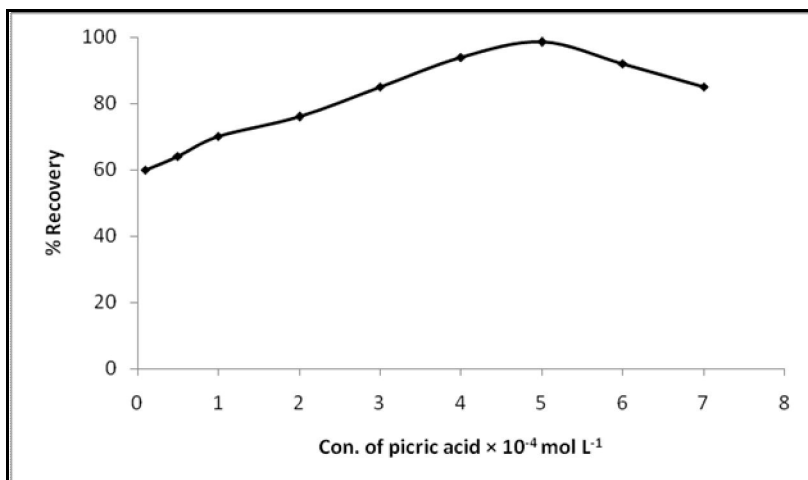


Fig.3. the Effect of concentration of picric acid anion

3.4. The effect of the volume of organic solvent extraction

To investigate the optimization volume of organic solvent was performed in volumes of 3 to 7 mL dichloromethane. According to the results the volume of organic solvent dichloromethane 3 mL having maximum absorption paired ion ($\text{CTA}^+ (\text{NO}_2)_3\text{PhO}^-$) was selected as the optimal volume of extraction solvent.

3.5. Analytical figures of merit

Under the optimum conditions, a linear calibration curve over the range of 100 to 3000 ng mL^{-1} was constructed for CTAB. The equation for the line was $y = 0.4352x - 0.0001$ with regression coefficient (R^2) of 0.9995. Detection limit based on three times standard deviation of the blank ($3S_b$) was 7.0 ng mL^{-1} and the relative standard deviation (R.S.D) was 2.1 %.

3.6. Determination of CTAB in real sample

In order to test the reliability of the proposed methodology suitable for the assaying of CTAB in anti-dandruff shampoo sample. For this purpose a solution of concentrations 30 mg L^{-1} of the anti-dandruff shampoos contain 3% CTAB were prepared and used it to prepare a sample solution with a concentration of 0.6 mg L^{-1} was used to CTAB. After extraction with 3 mL dichloromethane, was called absorption compared to the control solution. The experiment was repeated 3 times for each concentration. The result in Table 1 has been reported.

Table1. Determination of CTAB in anti-Dandruff shampoo

% Recovery	The resulting concentration (mg L^{-1})	concentration of CTAB (mg L^{-1})	volume of CTAB (mL)	number of sample
101	0.609	0.6	0.5	1
99.1	0.595	0.6	0.5	2
101	0.609	0.6	0.5	3

4. Conclusion

This method shown to be efficient, simple, easy, safe, rapid, inexpensive technique to determine CTAB by Uv. Vis. The proposed method has promising application in environmental analysis for trace CTAB.

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