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Determination of Trace Amount of Sodium Dodecyl Sulfate Surfactant by Extraction Spectrophotometric Method

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A simple, sensitive extraction-spectrophotometric method for determination of trace amounts of sodium dodecylsulfate (SDS) is reported. The (SDS^-–OTB^+) ion-pair in buffered media is quantitatively extracted into dichloromethane and its absorbance is measured at 625 nm in room temperature. The effect of different variables such as solvent, volume of extractant, volume of cationic reagent, pH, shaking time is investigated and optimum conditions for quantitative extraction of sodium dodecylsulfate is obtained. Linear calibration curve over the sodium dodecylsulfate concentration range of 0.1-4.0 µg mL^{-1} and regression coefficient of 0.9996 is obtained. The relative standard deviation (RSD) of 7 replicate determinations of sodium dodecylsulfate concentrations of 0.2, 1.0 and 4.0 µg mL^{-1} is 3.75, 1.84 and 0.63 %, respectively. The detection limit of the method is 8.91 ng mL^{-1}. The amount of sodium dodecylsulfate in Karoon river as real sample is determined by standard addition method which is 0.141 µg mL^{-1}. The interference of 22 cationic and anionic species have also been evaluated and it was found that most species have no effect on the extraction selectivity. The method was applied to the Karoon river raw water contaminated with industrial and house sewage. The results showed high potential of the recommended method for the determination of sodium dodecylsulfate in water samples.

Key Words: Determination, Sodium dodecylsulfate, Extraction, Spectrophotometric method, Ion pair, *o*-Toluidin blue.

INTRODUCTION

Sodium dodecylsulfate is linear sulfate (Fig. 1) that widely used in detergent formulations such as dishwashing liquids, hair and body shampoos *etc*. Worldwide industrial production of surfactants increased from 3500 Tons in 1950 to about 4.3 million Tons in 1990. They have been successfully marketed to domestics as well as industrial customers that nowadays they represent one of the most common components of domestic and industrial wastewaters and count among the most widely disseminated xenobiotics in streams and aquatic environments. However, there is wide agreement that their produce in natural fresh-water sources and other ecosystems is undesirable. Excessive foaming in sewage treatment plants causes operational

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difficulties and may also lead to health hazards in the form of airborne pathogens carried on windblown foams. Therefore, many attempts have been made to decrease the amount of surfactants released in the environment[1]. Various analytical techniques have been reported for the determination of SDS including capillary electrophoresis/mass spectrometry[2], turbidimetry[3] and polarographic method[4]. These methods are either insensitive or inconvenient for determination of SDS in river water. Almost all of the methods for the spectrophotometric determination of anionic surfactants depend on the formation of a salt (ion associate) when cationic dye reacts with anionic surfactants. Methylene blue is one of the most frequently used cationic dyes[5]. The methylene blue method, however, is very troublesome and its sensitivity is very low because of the small extractability of the ion associate of methylene blue with anionic surfactants. Recently, several studies for the development of a simpler and more sensitive method have been carried out. Motomizu *et al.*[6] used the cobalt complex cation of 2-(2-pyridylazo)-5-diethyl aminophenol as the cationic reagent and extracted anionic surfactants into benzene. The molar absorptivity was 5.5×10^4 L mol^{-1} cm^{-1} at 550 nm. This paper describes a simple and sensitive method for the quantitative extraction of (OTB^+–SDS^-) ion pair from buffer solution (pH 6.5) into dichloromethane organic solvent followed by spectrophotometric measurements at 625 nm.

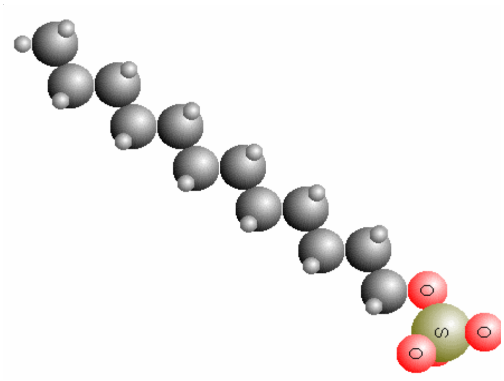

Fig. 1. Structure of sodium dodecylsulfate

EXPERIMENTAL

All the chemicals were of the highest purity available and used without purification. Double distillation deionized water was used throughout.

1000 ppm stock solution of SDS purchased from SDS 99.9 % pure was prepared by dissolving 0.1 g of SDS powder in water and diluting to the mark in a 100 mL volumetric flask with distilled water. Working solutions were prepared by appropriate dilution of the stock solution with water.

o-Tolidine blue (OTB), stock solution (1×10^{-4} M)was prepared by dissolving proper amount of the dyestuff (Merck) in water and diluting to the mark in a 1000 mL volumetric flask.

pH 6.5 buffer solution was prepared by titration 100 mL phosphoric acid 97 % with NaOH (0.1N), the pH measured by a Kent digital pH meter Model EIL 7045/46, equipped with a combined glass electrode, until buffer solution of (pH 6.5) prepared. A Jenway Model 6105 UV/VIS spectrophotometer was used for absorbance measurements.

Recommended procedure: An aliquot of sample solution containing 10 µg mL^{-1} of SDS was placed in a 10 mL volumetric flask. 2.0 mL of *o*-toluidine blue solution (1.0×10^{-4} M) and 1.0 mL of pH 6.5 buffer solution were added and the solution was diluted to the mark with distilled water. The solution was transferred into a 50 mL separatory funnel and 5 mL of dichloromethane was added. The solution was shaken vigorously for 35 s. The phases were allowed to measured at 625 nm against a reagent blank.

RESULTS AND DISCUSSION

Since SDS^- anion forms a fairly stable ion-pair with *o*-toluidine blue cationic dyestuff as counted ion in aqueous buffer solution, it is expected to result in a sensitive and useful spectroscopic method for trace sodium dodecylsulfate determination.

In preliminary experiment, it was found that the counter anion plays an important role in the information of a stable ion-pair and among the dyestuffs examined such as methylene blue, briliant cresyle blue, thionine and *o*-toluidine blue, best results were obtained with *o*-toluidine blue dyestuff.

Choice of organic solvent: The extraction process was performed with some common organic solvents such as dichloromethane, chloroform, carbon tetrachloride and benzene. It was found that the ion-pair is readily extractable in dichloromethane, while in other solvents used, the coloured complex could not be extracted into the organic phase as completely as dichloromethane.

Effect of *o*-toluidine blue concentration: The effect of *o*-toluidine blue concentration on the extraction of SDS^-–OTB^+ ion-pair was investigated and the results are shown in Fig. 2. It is seen that the absorbance of the organic phase increase with increasing *o*-toluidine blue concentration in the aqueous phase. The concentration 2×10^{-4} M of *o*-toluidine blue in the final solution was chosen as the optimum concentration of *o*-toluidine blue.

Effect of pH: The effect of different buffer solutions on the extraction process of SDS^-–OTB^+ ion-pair was studied. On the basis of the results presented in Table-1, 1.0 mL of phosphoric acid-sodium phosphate buffer solution (pH 6.5) gives the best results.

Effect of shaking time: The effect of shaking time on the extraction of SDS^- OTB^+ ion-pair was studied. Shaking time was found to be constant for the extraction of ion-pair.

Analytical performance: Under the optimum conditions described above, linear calibration graph for sodium dodecylsulfate was obtained in the concentration range of 0.1-4 µg mL^{-1}. The regression equation for sodium dodecylsulfate is A_{SDS} =

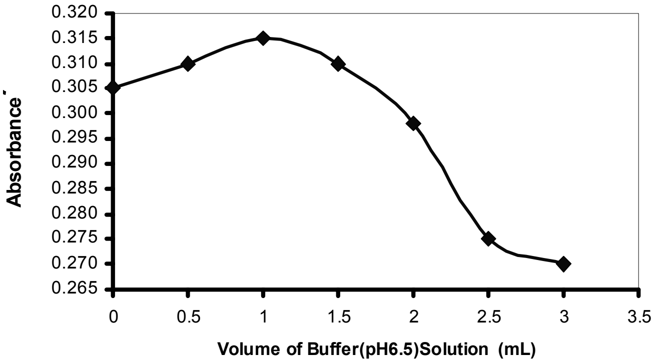

Fig. 2. Effect of buffer solutions on the absorbance of extracted SDS^-OTB^+ ion-pair

TABLE-1
EFFECT OF BUFFER SOLUTION (pH 6.5) ON THE EXTRACTION OF SDS

No.	Volume of buffer (pH 6.5) solution (mL)	ΔA (Abs.)
1	0.0	0.315
2	0.5	0.310
3	1.0	0.315
4	1.5	0.310
5	2.0	0.298
6	2.5	0.275
7	3.0	0.270

$0.3253C_{SDS}-0.0211$ (where A_{SDS} is the absorbance of the sample against a blank solution and C_{SDS} is the concentration of sodium dodecylsulfate in µg mL^{-1}) with a correlation of 0.9996.The relative standard deviation (RSD) of 10 replicate determinations of 0.2, 1.0 and 4 µg mL^{-1} of sodium dodecylsulfate are 3.75,1.84 and 0.36 %, respectively and the limit of detection (LOD) of the method is 8.91 ng mL^{-1}.

Effect of interferences: In order to study the influence of various cations and anions on the determination of sodium dodecylsulfate, a fixed concentration of sodium dodecylsulfate, 0.1 µg mL^{-1}, was taken with different amounts of foreign ions and the recommended procedure was followed. A relative error of ± 3 % with respect to the absorbance difference for the sodium dodecylsulfate solution was considered tolerable. Tolerance limits are as follows: SO_4^{2-}, Na^+ (1000 Folds), I^-, Br^-, CH_3COO^-, PO_4^{3-},ClO_3^-, K^+, Li^+, $C_2O_4^{2-}$, (250 folds). The results show that most of the cations and anions used have no considerable effect on the determination of sodium dodecylsulfate, while SO_3^{2-}, $S_2O_3^{2-}$, N_2H_4 and ascorbic acid do interfere.

Application: The proposed method was applied to the determination of sodium dodecylsulfate content in Karoon river in Ahwaz. The results are shown in Table-2.

Standard addition method used and the amount of recovery calculated, it shown that the results are satisfactory for determination of anionic surfactant in river waters.

TABLE-2
DETERMINATION OF THE SDS CONTENT IN KAROON RIVER BY THE PROPOSED METHOD

No.	Absorbance	Concentration
1	0.024	0.1386
2	0.025	0.1417
3	0.026	0.1447
4	0.025	0.1417

Conclusion

The proposed method has been applied to the determination of concentration of alkyl sulfate surfactant, SDS (sodium dodecylsulfate, lauryl sulfate) that is widely used in detergent formulations such as dishwashing liquids, hair and body shampoos *etc.*, This method is applicable to real samples and river waters in which sodium dodecylsulfate react with cationic dye [*o*-tolidine blue (OTB)] to form ion-pair and subsequently these ion-pair are extracted into organic solvent layer and the absorbance of $SDS^{-}OTB^{+}$ in the organic phase is measured. The results by this method are in good agreement with those other methods[7].

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