*Jr. of Industrial Pollution Control 21 (2)(2005) pp 293- 298*


# ESTIMATION OF SURFACTANTS AT PPM LEVEL FROM SYNTHETICALLY POLLUTED WATER

**DILIP B.PATIL*, A. KSHIRSAGAR AND AJAY P. GANORKAR**

*Dcpartment of Chemistry, Institute of Science, Nagpur - 440001, India

**Key words :**

## ABSTRACT

**Large scale use of non-biodegradable surfactants in houses and industries leaves effluents where surfactants persist in it. These surfactants causes water pollution and are having very low concentration. Hence there is a need to develop simple method that is efficient to measure ppm level concentration of surfactants. Several methods have been known in the literature to estimate the amount of surfactant present in the given sample. In present work we have used relatively simple and precise method to estimate surfactant at ppm level from, synthetically polluted water. Part per million level of surfactant were estimated in synthetically polluted water containing 0.001 to 0.007 percent of Nirma. Surf excel, Rin Shakti and Arial. The level of surfactant in synthetically polluted water followed the order Arial > Surf excel > Rin Shakti > Nirma. Present study revealed that minimum amount of surfactant that could be estimated in synthetically polluted water of commercially available detergent like Arial, Surf excel, Rin Shakti and Nirma were 39.0, 50.6, 58.1 and 72.2 ppm respectively.**

## INTRODUCTION

Surfactants have good cleaning properties and do not form insoluble salts with hard water ions. The most objectionable manifestation of the non-biodegradable surfactant is the 'head' of the foam that began to appear in glasses of drinking water in areas where sewage is recycled through domestic water supply.

Household surfactants contain several pollutants which severely affect the water bodies. Present day sewage contains appreciable quantities of surfac-

tants due to effluent of domestic waste and surfactant based industries. Waste water contaminated with surfactant carries huge cap of foam. This visible foam is an anaesthetic for all purposes. Concentration of surfactants in these waste is very low. Various methods have been used by environmentalist to estimate such low concentration [1-5]. Solvent extraction combined with spectrophotometry has been found to be useful in this regards. The use of ion-pair complex formation between methylene blue and surfactant allows the ppm level estimation of surfactant from synthetically polluted water.

Present work describes the preparation of synthetically polluted water of surfactants such as Nirma. Kin Shakti. Surf Lxcel and Arial and its employment in the estimation of amount of surfactant present in commercially available detergents at ppm level.

## MATERIAL AND METHOD

All the chemicals used in the study were of analytical grade. Methylene blue was from BDII chemicals- India, potassium dihydrogen phosphate and concentrated sulphuric acid were from E.Merck India Ltd., Bombay, Ethyl acetate used was purified by the method of Vogel[6]. Several commercially available anionic detergents were used for the study. Surf Excel was from Hindustan Lever Ltd.- Bombay, Nirma was from Nirma Chemicals- Ahmedabad, Arial was from Procter And Gamble Home Products Ltd., Mumbai. Double distilled water was used for the preparation of various solutions.

**Methylene blue reagent :** To prepare methylene blue stock solution, 0.100 gm of solid was dissolved in 100 ml distilled water. 30 ml of this stock solution was transferred to one liter volumetric flask and 500 ml distilled water, 6.8 ml concentrated sulphuric acid and 50 ml potassium dihydrogen phosphate were added to it. The solution was finally diluted to one litre.

**Synthetically polluted water :** Accurately weighed 100 mg each of Nirma. Arial, Surf Excel and Rin Shakti samples were dissolved in one litre of double distilled water. To prepare test solutions 10, 20, 30, 40, 50, —— 80 ml each of the above solutions were diluted to 100 ml. The resulting solution contained 10. 20, 30, ——— 80 ppm of the surfactant.

**Purification of Ethyl Acetate (Atkins, 1990) :** A mixture of one litre of ethyl acetate. 100 ml of acetic anhydride and 10 drops of concentrated sulphuric acid is refluxed for four hours and then fractioned. The distillate is shaken with 20-30 gm of anhydrous potassium carbonate, filtered and redistilled.

**Solvent extraction :** The surfactants in known volume of the solution was extracted into an immiscible ethyl acetate by solvent extraction. 85 ml each of the synthetically polluted water solution of the surfactant was taken in an extraction column and mixed with 15 ml of distilled ethyl acetate. A stream of air was bubbled through the solution to extract the surfactant into ethyl acetate. It was found that 30 minutes were adequate to completely extract the surfactant into the ethyl acetate. Then, after the ethyl acetate was separated and treated with 0.5 ml of methylene blue reagent, the mixture was shaken



for 5 minutes and was allowed to settle. The aqueous layer was separated and absorbance was measured at 652 nm. The above procedure was repeated for 0.002.0.003-0.007% of all detergents taken for the study.

The optimum conditions for the solvent extraction were established by varying each of the parameters and thereby determined its optimum value.

## RESULTS AND DISCUSSION

The synthetically polluted water of detergent in known volume of solution was extracted into immiscible distilled ethyl acetate layer by solvent extraction. 85 ml of synthetically polluted water of detergent was taken in an extraction column and mixed with 15 ml distilled ethyl acetate. It was estimated at various time intervals 5. 10, 15, 20, 25, 30, 35 and 40 minutes ( **Fig.-1, Table 1** ) and observed that 30 minutes of extraction time was found to be adequate and larger time were unnecessary. By varying the amount of methylene blue reagent from 0.2 to 2.0 ml, it was observed that 1.0 ml of methylene blue gave the maximum optical density ( **Fig.-2, Table-2** ). Similarly by changing the amount of ethyl acetate from 5 to 30 ml of the extraction column, it was observed that 15 ml of the ethyl acetate is sufficient to give maximum optical density ( **Fig.-3, Table-3** ). Further qualitative estimations were conducted under these optimum performance.

In present method, estimation based on the formation of ion pair complex [6-10] between the methylene blue which is cationic dye. and anionic surfactant

**Table - 1**
**Effect of extraction time**

| Extraction time in minutes | Optical density |
|---|---|
| 5 | 0.19 |
| 10 | 0.24 |
| 15 | 0.26 |
| 20 | 0.28 |
| 25 | 0.29 |
| 30 | 0.30 |
| 35 | 0.30 |
| 40 | 0.30 |

**Table - 2**
**Effect of reagent volume**

| Volume of regent in mL | Optical density |
|---|---|
| 0.50 | 0.270 |
| 0.75 | 0.325 |
| 1.00 | 0.375 |
| 1.25 | 0.350 |
| 1.50 | 0.300 |
| 1.75 | 0.225 |

**Table - 3**
**Effect of ethyl acetate volume**

| Volume of ethyl acetate /ml | Optical density |
|---|---|
| 05 | 0.15 |
| 10 | 0.25 |
| 15 | 0.32 |
| 20 | 0.28 |
| 25 | 0.20 |

**Table - 4**
**Variation of lamberts-beers law for Surf Excel**

| Concentration of Surf Excel in % | Optical density | Concentration of Surf Excel in ppm |
|---|---|---|
| 0.00 1 | 0.15 | 8.3 |
| 0.002 | 0.23 | 16.6 |
| 0.003 | 0.31 | 24.3 |
| 0.004 | 0.40 | 33.2 |
| 0.005 | 0.52 | 41.5 |
| 0.006 | 0.60 | 49.8 |
| 0.007 | 0.72 | 58.1 |

**Table - 5**
**Estimation of surfactant in Nirma**

| Concentration of Nirma in % | Optical density | Concentration of Nirma in ppm |
|---|---|---|
| 0.001 | 0.10 | 7.3 |
| 0.002 | 0.18 | 13.7 |
| 0.003 | 0.24 | 18.7 |
| 0.004 | 0.28 | 23.7 |
| 0.005 | 0.34 | 27.0 |
| 0.006 | 0.41 | 32.8 |
| 0.007 | 0.49 | 39.0 |

**Table - 6**
**Estimation of surfactant In Rin Shakti**

| Concentration of Rin Shakti in % | Optical density | Concentration of Rin Shakti in ppm |
|---|---|---|
| 0.001 | 0.11 | 9.1 |
| 0.002 | 0.215 | 17.4 |
| 0.003 | 0.29 | 23.7 |
| 0.004 | 0.37 | 30.9 |
| 0.005 | 0.46 | 37.8 |
| 0.006 | 0.55 | 44.8 |
| 0.007 | 0.62 | 50.6 |

**Table - 7**
**Estimation of surfactant in Arial**

| Concentration ofAerial in % | Optical density | Concentration of Aerial in ppm |
|---|---|---|
| 0.001 | 0.17 | 14. 1 |
| 0.002 | 0.275 | 22.4 |
| 0.003 | 0.38 | 31.1 |
| 0.004 | 0.49 | 39.8 |
| 0.005 | 0.62 | 50.6 |
| 0.006 | 0.76 | 61.8 |
| 0.007 | 0.875 | 72.2 |

solution of synthetically polluted water of commercially available detergents. The ion-pair complex is insoluble in aqueous layer but it is soluble in ethyl acetate layer. Therefore dye remains in aqueous layer and ion-pair enters into the ethyl acetate layer.

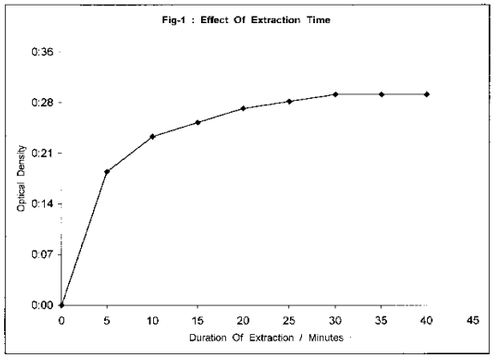
The ion-pair complex formed between methylene blue and surfactants have broad absorption spectrum with A.max 652 nm. Since Xmax is distinctly in the visible region, intensity of ion-pair complex depends upon the concentration of surfactant in synthetically polluted water of detergents. The results of extraction at optimum conditions shows that by changing the amount of detergents in synthetically polluted water from 0.001 percent to 0.007 percent, plot of observed optical density and concentration of detergent ( in ppm ) was found to be satisfactory and linear over the range of concentrations from 7.3 ppm to 72.2 ppm of surfactant ( **Fig.- 4 to 7, Table- 4 to 7** ) and it obeyed the Lamberts-Beer's Law.

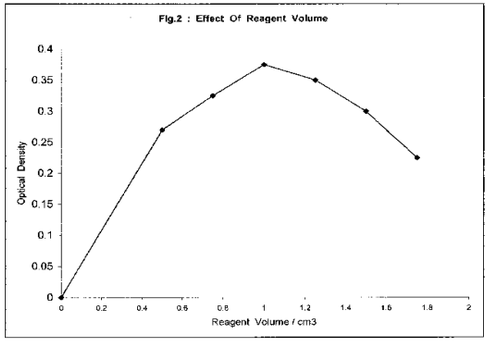
The concentration of surfactant ( in ppm ) estimated from Surf excel was 8.3 to 58.1. The concentrations of surfactants ( in ppm ) that could be estimated from synthetically polluted water of commercially available detergents were in range of 7.3 ppm to 39.0 ppm for Nirma, 9.1 ppm to 50.6 ppm for Rin Shakti, 8.3 ppm to 58.1 ppm for Surf excel and 14.1 ppm to 72.2 ppm for Arial. In present study detergent Surf excel was taken as the reference standard.

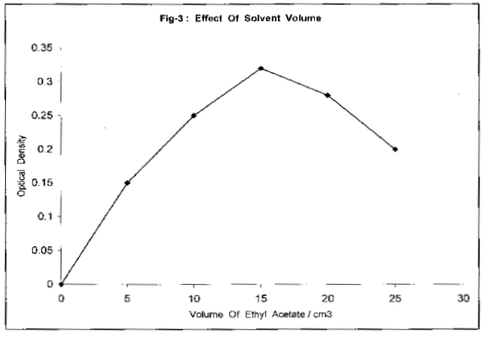
## CONCLUSION

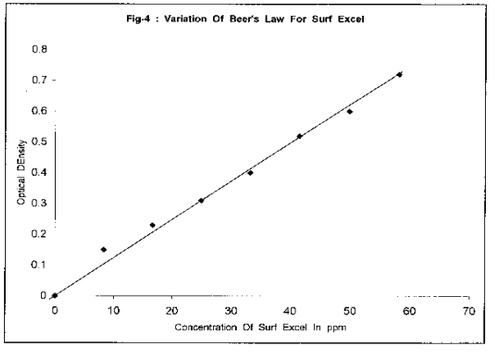
Comparing the estimations of surfactants in the synthetically polluted water






solutions of commercially available detergents, it was found that the concentration of surfactant in Rin Shakti and Nirma is lower than that of Surf excel. Only Arial is having higher amount of surfactant.

The method used to estimate surfactant is relatively simple, precise and accurate. As the water used is synthetically polluted water solution of commercially available detergents, the method can also be applied to estimate amount of surfactant at part per million level in drinking water samples.

## ACKNOWLEDGEMENT

Author sincerely thanks Prof. M. N. Dcshpande, Director, Institute of Science, Nagpur and Dr. Mrs B. D. Saraf. H. O. D. Chemistry Department, for encouragement and facilities.

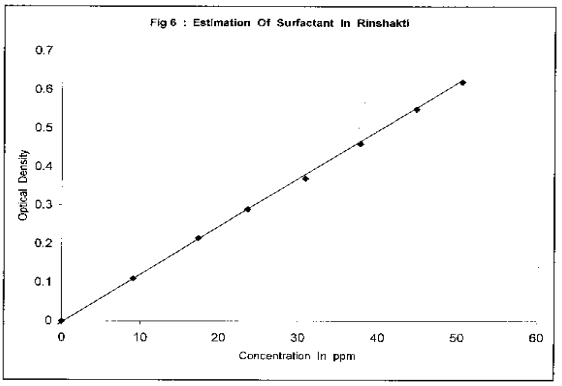
## REFERENCES

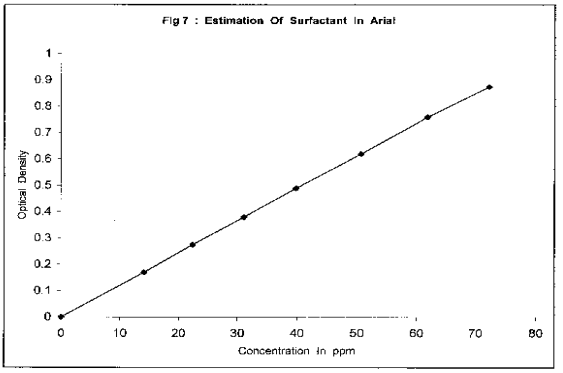
Atkins, P.W. 1990. *Physical chemistry*, 4th Edition, LLBS Society and Longman.

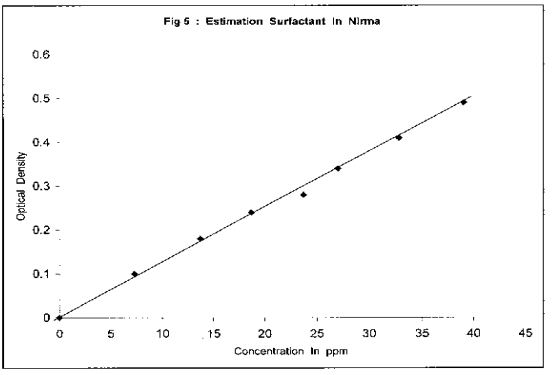
Basself, J., Venry, R.C., Jeffry, G.H. and Mendnam, J. 1978. *Vogcls text book of quantitative inorganic chemistry*, 4th Edition, ELBS and Longman.

Barrow, G.M. 1992. *Physical chemistry*, 5th Edition, Tata McGraw-Hill Publications.

Castellan, G.B. 1986. *Physical chemistry*, 3rd Ed., Narosa Publishing House, New Delhi.




Glasstone Samuel, 1951. *Text book of physical chemistry.* 2nd Edition, D. Ran Mostrand Co. Inc.,Newyork. p.1206.

Hanson, N.W. 1973. *Official standards and recommended methods of analysis.* 2nd Edition, The Society of Analytical Chemistry, London .

Moore, W. J. 1986. *Basic Physical chemistry,*6th Ed.Prentrice-Hall of India Pvt. Ltd. N.Delhi.

S.U.I. Board of consultants of Engineers, 1982. *Household detergents and Industrial Sur factants.* Delux Ed., Roopnagar, N. Delhi.

Vogel, A. I. 1970. *A textbook of practical organic chemistry,* 3rd Edition, EEBS and Longman Group Ltd.

Welchar, E. J. *Standard methods of chemical analysis,* 6th Ed.,Vol. (3), Instrumental Methods Part-B, Van Mostrand Rein Hold Company, London.