

# New Indirect Spectrophotometric Method for Determination of Hypochlorite using Nile blue

Nief Rahman Ahmad<sup>1</sup>, Farha Khalaf Omar<sup>2</sup>

<sup>1</sup>Department of Environmental Technology, College of Environmental, University of Mosul, Mosul-Iraq

<sup>2</sup>Department of Chemistry, Education College for Girls, University of Mosul, Mosul-Iraq

## ABSTRACT

A new, simple and sensitive indirect spectrophotometric method for the determination of hypochlorite ion has been developed. The method is based on bleaching the color of Nile blue by hypochlorite and measurement of absorbance at 635 nm. The decreased in absorbance is directly proportional to the concentration of hypochlorite. The calibration graph was linear over the range 0.0-1.6 µg/mL. The apparent molar absorbance and Sandell's sensitivity values are  $2.17 \times 10^4 \text{ l.mol}^{-1}\text{cm}^{-1}$  and  $2.3 \text{ ng.cm}^{-2}$ , respectively. The relative standard deviation (RSD) is less than 1.9 (n=10) and the accuracy (average recovery) is  $100 \pm 1.2$ . The advantages of the proposed method include a high sensitivity and considered to be simple because it does not need heating or solvent extraction steps. The method was applied successfully for determination of hypochlorite in household bleaching products.

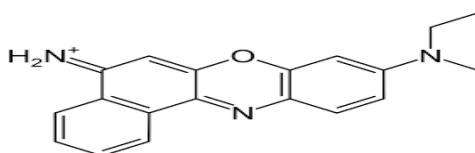
**Keywords:** Hypochlorite ion, Nile Blue, Spectrophotometric, Bleaching Samples.

## HOW TO CITE THIS ARTICLE

Nief Rahman Ahmad, Farha Khalaf Omar, "New Indirect Spectrophotometric Method for Determination of Hypochlorite using Nile blue", International Journal of Enhanced Research in Science, Technology & Engineering, ISSN: 2319-7463, Vol. 7 Issue 1, January-2018.

## INTRODUCTION

Sodium hypochlorite (NaOCl) is a compound that can be effectively used for water treatment and purification. It is used on a large scale for odor removal, bleaching, disinfection, and surface purification[1]. Water chlorination has been the most common disinfectant methods used by waters suppliers[2]. Chlorine is an efficient agent for inactivating several types of microorganisms, it has been preferred as a disinfecting agent to assure the bacteriological quality of the drinking water[3]. The water bacterial contamination also occur in the distribution network; therefore, to prevent this occurrence, a free chlorine residual in excess of  $0.2 \text{ mg L}^{-1}$  must be maintained throughout the distribution lines[3]. Various analytical methods have been utilized for the determination of NaOCl. Some of the examples were colorimetric[4], iodimetric[5-7], chemiluminiscent[8,9], potentiometric [10], spectrophotometric methods[11-13], amperometric [14] and flow injection methods[15,16]. In this work, we intend to develop a simple, sensitive indirect spectrophotometric method for the determination of hypochlorite ion in household bleaching samples using Nile blue which destroyed to a colorless product by hypochlorite ion.



**Chemical Structure of Nile blue**

## EXPERIMENTAL

Spectro Uv / visible dual beam Spectrophotometer [UVS-2700, Labomed, [INC ] with 1.0 cm quartz cells was used.

### Reagents

All chemicals used were of analytical purity grade and all solutions were prepared in distilled water. A hypochlorite stock solution (0.01%) was prepared by dilution of 0.25 ml of 4 % hypochlorite to 100 ml by distilled water, store in a dark bottle and standardized every 4-5 days. This solution stored in a dark bottle to protected from light preferably in a cool place<sup>[17-19]</sup>. Standard solution of hypochlorite (10ppm). This solution was prepared by diluting 10 ml of the stock solution to 100 ml by distilled water in a volumetric flask. Nile blue solution (0.025%) was prepared by dissolved 0.025gm of Nile blue reagent in 50ml ethanol and diluting to 100 ml by distilled water in a volumetric flask. Ten types of household bleach samples of different brands were purchased from local markets include Sehat bleaching liquid /Iran/shampoo cede sehat Co., Fas / bleaching liquid /Iraq /detergent Babil com. 3-TopRox/Syria/Sugar company for chemical industries., Noura /Madar for chemicals com./ Syria., Altunsa/(SYA) / Turkey. Chlormax/ U.A.E/ Sun Shine com., Chlorox/National cleaning product .com /Saudi Arabia, Peros/Turkey/Shop product. Com., Clorite/U.A.E/Pana Star. Com., Shoof/Iraq/Shoof com.

### Recommended procedure

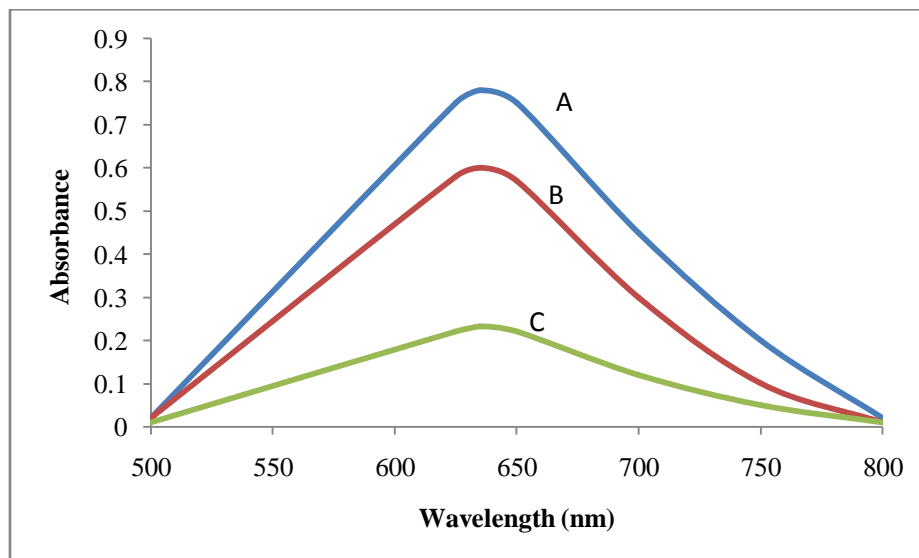
A known volume of standard solution of hypochlorite ion sample containing (0.0-40  $\mu\text{g}$ ) was transferred into a series of 25ml calibrated flask followed by 1ml of 0.025% Nile blue solution ,the volume was made up to the mark with distilled water. The absorbance was measured at 635 nm against distilled water.

### Procedure for the determination of hypochlorite in household bleach

0.25 ml of the household bleach solution was transferred into 1L volumetric flask and diluted up to the mark with distilled water, 3ml of this solution was treated as described above under recommended procedure.

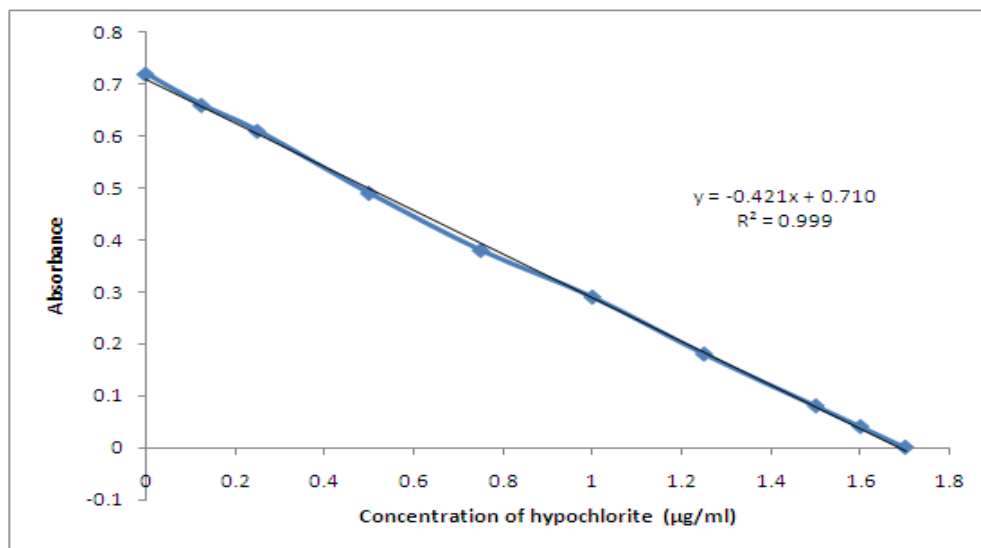
## RESULTS AND DISCUSSION

The proposed method was based on the bleaching the color of Nile blue by hypochlorite solution. The reaction occurred at room temperature and neutral medium (pH6.5-7.3) immediately and remained stable for at least 24h. and measurement of absorbance at 635 nm. The decrease in absorbance is directly proportional to the concentration of hypochlorite, as shown in Figure1.



**Figure 1: Absorption spectra of Nile blue, A:** Nile blue alone against distilled water, **B:** Nile blue+10  $\mu\text{g}$  hypochlorite ion against distilled water, **C:** Nile blue+35 $\mu\text{g}$  hypochlorite ion against distilled water

Under the experimental conditions described, Beer's law is obeyed over the concentration range 0.0-1.6  $\mu\text{g}/\text{ml}$  Figure 2, with correlation coefficient of 0.999, intercept of -0.4215 and slope of 0.7106.



**Figure 2: Calibration graph of hypochlorite ion.**

The accuracy and precision of the method was established by analyzing the standard hypochlorite solution at three different levels. The average recovery which is a measure of accuracy is  $100 \pm 1.2$  revealing high accuracy of the method. The relative standard deviation (RSD), which is an indicator of precision is less than  $\pm 1.9\%$ . The results are compiled in (Table1).

**Table1: Optical characteristics and statistical data for regression equation of the proposed method**

| Parameters   | Value   |
|--|---|
| $\lambda$ max (nm)   | 635   |
| Beer's law limit ( $\mu\text{g} \cdot \text{ml}^{-1}$ )                      | 0.0-1.6   |
| Molar absorptivity ( $\text{l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ ) | $2.17 \times 10^4 \text{ l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ |
| Corrélation coefficient ( $r^2$ )  | 0.999   |
| Régression équation ( $Y = a \times + b$ )                                   |   |
| Slope (a)  | -0.4215   |
| Intercept (b)  | 0.7106  |
| Recovery %   | $100 \pm 1.2$   |
| Relative standard deviation (%)  | $< \pm 1.9\%$   |

### Application to real samples

To demonstrate the practical applicability of the proposed method, hypochlorite ion in household bleach samples. Ten types of household bleach samples of different brands were purchased from local markets and analyzed by the proposed method. The results were also compared statistically by student t-test with those obtained by standard method<sup>[20]</sup> at 95% confidence level. The calculated t- values did not exceed the theoretical values indicating that there was no significant differences between the precision of the proposed and literature method as cited in (Table2).

**Table2: Determination of hypochlorite ion in different household bleach samples.**

| Sample  | Amount of hypochlorite % * |                 |               |
|---|----------------------------|-----------------|---------------|
|   | Proposed method (DPD)      | standard method | methodt-value |
| 1- Sehat bleaching liquid /Iran/shampoo<br>cede sehat co. | 2.9                        | 2.95            | 0.95          |
| 2-Fas / bleaching liquid/Iraq/detergent<br>babil com      | 4.1                        | 4.08            | 0.88          |
| 3-Top Rox/Syria/Sugar company for                         | 3.88                       | 3.9             | 1.04          |

|  |              |          |        |
|--|--------------|----------|--------|
| chemical industries                      |              |          |        |
| 4-Noura /Madar for chemicals com./Syria  | 4.06         | 4.08     | 0.63 . |
| -Altunsa/(SYA)/Turkia5                   | 5.12         | 5.14     | 0.99   |
| 6- Chlormax/U.A.E/ Sun Shine co.         | 5.22         | 5.23     | 1.21   |
| 7- Chlorox/ National cleaning product    | 5,22         | 5.19     | 1.65   |
| .com/Saudi Arabia                        | 4.144.121.84 | 4.884.92 | 0.66   |
| 8-Peros/Turkia/Shopy product.co          | 4.10         | 4.12     | 1.94   |
| 9- Clorite/U.A.E/Panastar. co Shoof/Iraq |              |          |        |
| /Shoof co.10-                            |              |          |        |

\*Average of ten determinations.

T values (n=10, at 95% confidence level tabulated value 2.101).

## CONCLUSIONS

For the first time, Nile blue has been used as a chromogenic reagent for the spectrophotometric determination of hypochlorite. The proposed method, which is simple and rapid, offers the advantages of sensitivity and wide range of determinations without the need for extraction or heating. The method does not involve any difficult reaction conditions and can be compared favorably with the standard method. The proposed method has been successfully applied to the determination of hypochlorite in household bleach various materials.

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