

CHAPTER III

Experimental

3.1 Apparatus

1. UV-Visible spectrophotometer (spectronic 20 Genesys, USA).
2. pH meter (Gebrauchsanleitung Mikroprozessor CG 840 Schott Geräte).
3. RC -2 magnetic bar and a magnetic stirrer, Japan.
4. Inverters (Novem NSP Series, USA).
5. Water bath (Mettler, USA).
6. Motor 2 phase (Semco, Germany).
7. Graduated pipettes 1.00, 5.00, 10.00 and 20.00 ml.
8. Separation funnel 100 and 120 ml.
9. Measuring cylinder 10.0 ml.
10. Beakers 25, 50, 100, 125 and 500 ml.

3.2 Chemicals

1. The standard metal solution (1,000 ppm) of copper (II) nitrate: was from E. Merck, Darmstadt, Germany. A standard metal solution was standard solution for atomic absorption spectroscopy (Spectro Pure Grade).
2. Zinc dibutyldithio carbamate (ZDBC MW 475.89): was from Fluka chemie, Switzerland.

3. Surfactant

There were four types of surfactants used in this work which were:

- Nonylphenol ethoxylate (NP(EO)₉ MW 616) was purchased from K.H. CO.,Ltd. NP(EO)₉ containing approximately nine moles of ethylene oxide per mole of nonylphenol.
- Sodium dodecyl benzene sulfonate (LAS MW 284) was supplied by Henkel company with a manufacture reported purity of at least 90%.
- Benzethonium chloride (Hyamine 1622 Mw 448.18) was purchased from Henkel Co. Ltd.,Thailand.
- Coco amidopropylbetaine (Tegobetaine Mw 640) was supplied by Johnson's Johnson Pte.Ltd., Thailand.

4. Solvent

- Ammonia solution (AR Grade) was purchased from BDH Limited, poole, England.
- Chloroform (AR Grade) was purchased from LAB Scan Asia Co. Ltd., Thailand.

Methods

Deionized double distilled water was used throughout this study. All glasswares and sample containers used in this study were free from iron by being soaked overnight in 10% hydrochloric acid bath and thoroughly rinsed several times with deionized double distilled water.

3.3 Preparation of the standard solutions

The standard solution for Visible spectra of Cu – ZDEC complex.

The 100.0 ppm standard solution of copper was prepared by pipetting 5.00 ml of 1,000.0 ppm standard solution of copper into a 50.0 ml of volumetric flask and the content was diluted to the mark with deionized water. A series of copper at the concentration of 1.00, 5.00, 10.00, 15.00 and 20.00 ppm was prepared by further dilution of the 100.0 ppm solution. A 1.0×10^{-2} M ammonia solution was employed for pH adjustment of the solution.

3.4 Copper determination using ZDBC as spectrometric reagents

Reagents

1. Stock solution of 0.1% w/v ZDBC in chloroform.
2. Copper standard solution at the concentration of 1.00, 5.00, 10.00 and 15.00 ppm, pH adjusted by ammonia solution to 4.27-8.27.
3. Stock solution of LAS, NP₉, Hyamine 1622 and Tegobetaine at the concentration 0.1, 0.2, 0.3, 0.4 and 0.5% w/v.

Procedure

The optimum conditions for reducing reaction of copper carbamate stain were studied in order of selected wavelength, optimum surfactant concentration, optimum pH and temperature of copper nitrate standard solution.

Selected Wavelength

Equal volume (10 ml) of ZDBC in chloroform phase was fixed at 0.1% w/v while Cu concentration in aqueous phase at pH 3.30 was varied from 0-15 ppm. After shaking for 10 min at 420 rpm and waiting until the phase separation was completed, the chloroform layer was transferred for spectral study in the range of 330 to 700 nm using chloroform as reference.

Effect of surfactant concentration

A series of solutions containing 1.00, 5.00 and 10.00 ppm of Cu^{2+} in aqueous phase and various surfactant concentrations from 0.1-0.5% w/v were prepared by shaking for 10 min at 420 rpm. The 10 ml of 0.1% w/v ZDBC in chloroform phase was added, and shaken for 10 min at 420 rpm. The absorbances of chloroform layer were measured at 440 nm.

Effect of pH of copper standard solution

The procedure for chloroform extraction was similar to that of previously described, using 1.00, 5.00 and 10.00 ppm of Cu^{2+} solution and varying pH from 4.27 to 8.27, and 0.1-0.5% w/v of the surfactant, 0.1% w/v of ZDBC.

Effect of temperature of copper standard solution

The procedure for chloroform extraction was similar to that of previously described. The experiment was performed at different temperature of copper standard solution from 20°C to 40°C.

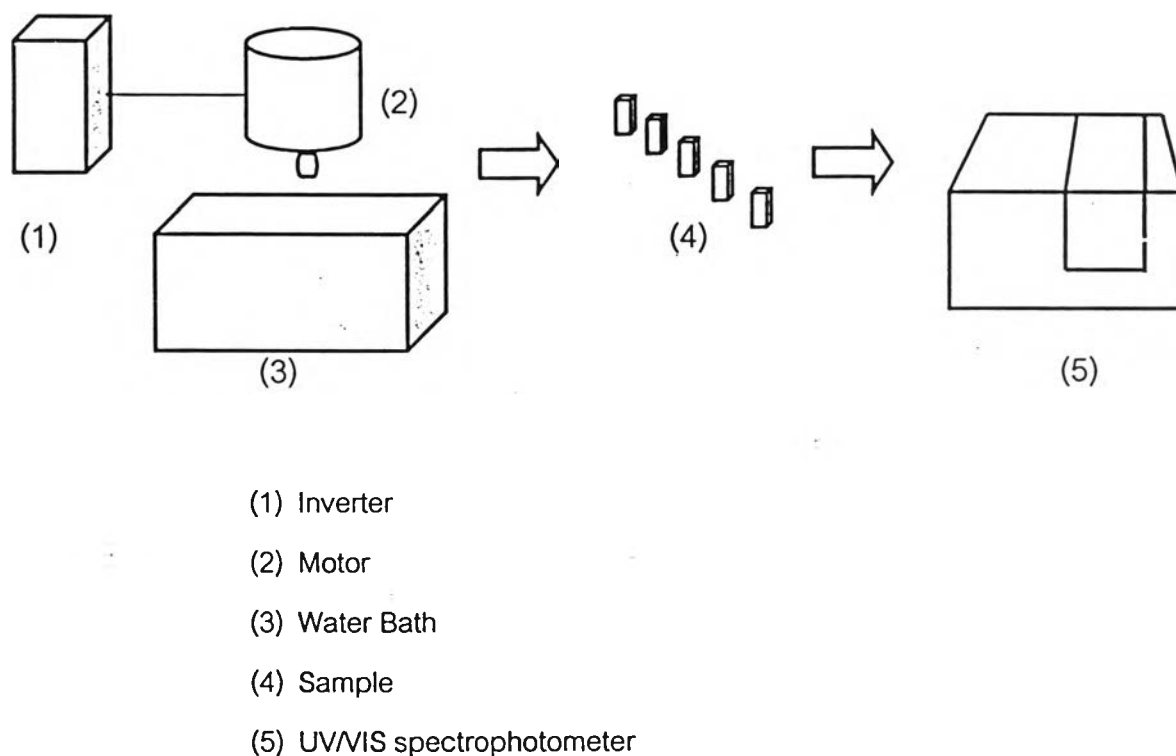


Fig 3.1 A schematic illustration of the experiment setup.

3.5 Formation of copper stain on gloves.

Six pieces of gloves, identified as A, B, C, D, E and F were used having sizes is 1.5x3.5 inch.

The piece A was dipped in 10 ppm copper nitrate standard solution for 10 min. While pieces B, C, D, E and F were dipped in the mixture of 10 ppm copper nitrate standard solution and 0.1, 0.2, 0.3, 0.4, and 0.5 %w/v of nonylphenol ethoxylate(NP₉), respectively. Observation color of pieces A to F.