Extractive Spectrophotometric Determination of Nickel (II)

Science

KEYWORDS : HHMCP, Nickel (II), extractive spectrophotometric determination, solvent extraction

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ABSTRACT

2-[(E)-N-(2—{[2-[(E)-[(2-hydroxyphenyl) methylildene] amino] phenyl} (methyl) amino} phenyl) carboximidoyl] phenol (HHMCP) was synthesized and employed to develop an extractive spectrophotometric method for the determination of Ni (II). The reagent forms a complex with Ni (II) and can be quantitatively extracted in Chloroform at pH = 7.0. The extracted species showed an absorption maximum at 495 nm with molar absorptivity of 0.75 ×102 L mol–1 cm–1. A systematic study of the extraction was carried out by varying the parameters like pH, reagent concentration and equilibration time. The method has been successfully applied for the determination of Nickel in synthetic mixtures and alloy samples.

INTRODUCTION

The significance of nickel as a transition metal lies in its wide spectrum of applications covering many frontier areas of study, particularly in industrial and consumer products. Even though nickel is not considered to be as toxic as most of the heavy metals, it is an equally harmful element. Hence, owing to the significance of nickel, it's determination from associated elements by extractive spectrophotometry has been of considerable importance

Nickel is widely used in electroplating, the manufacture of Ni-Cd batteries, rods for arc welding, pigments of paints, ceramic, surgical and dental prostheses, magnetic tapes and computer components and nickel catalysts. Nickel enters waters from dissolution of industrial processes and waste disposal1. Nickel was thought to be essential for plants and some domestic animals2, but not considered to be a metal of biological importance until 1975, when Zerner discovered that urease was a nickel enzyme[3]. Nickel is essential constituent in plant urease. Jack beans and soybeans generally contain high concentration of nickel1. Compared with other transition metals, nickel is moderately toxic element, and still at low concentration produces a general toxic effect on the human organism, causing nasopharynx and lung diseases, malignant tumors and dermatological diseases[4]. Nickel-containing sewage is harmful after ingress into water. This fact explained the importance of the monitoring of nickel concentration in natural and waste water samples. Flame and graphite furnace atomic absorption spectrometry and spectrophotometric methods provides accurate and rapid determination of nickel in natural and waste waters[5]. However, very frequently a direct determination cannot be applied due to low concentration of analyte or matrix interferences.

Nickel is a moderately toxic element compared to other transition metals. Environmental pollution monitoring requires determination of nickel in trace levels in various samples. Recently, numerous methods have been published on the preconcentration of nickel, alone or in mixtures, by cloud point extraction (CPE) method prior to its determination using spectrometric techniques [6-10.]

A wide variety of reagent has been reported for the spectrophotometric determination of nickel. However, these methods suffer from limitations such as critical pH[11-13,] requirement of masking agent1 or other agents[14, 15,] requirement of heating[16], and interference from some ions[11, 17]etc. A spectrophotometric extractive method for the determination of Ni (II) was developed in the present study.

MATERIALS AND METHODS

All absorbance measurements were made on Systronics Digital Double Beam spectrophotometer model-2101 with 1 cm quartz cell. Standard volumetric flasks, 125ml separatory funnels, beakers were used for volumetric measurements. All dilutions were made using double distilled water. Solvents like chloroform, ethanol were used after double distillation. All interfering ion solutions were prepared in double distilled water

A stock solution of Ni (II) was prepared by dissolving 1 g Nickel chloride hexahydrate in 250 ml double distilled water and standardized. A working solution of 100µg/ml was prepared by dilution of the stock solution with double distilled water in a standard volumetric flask. 2-[(E)-N-(2—{[2-[(E)-[(2-hydroxyphenyl) methylildene] amino] phenyl} (methyl) amino} phenyl) carboximidoyl] phenol (HHMCP), (10-2 M) was always prepared by dissolving 0.478 g of HHMCP in 100 ml chloroform and used. .

EXTRACTION PROCEDURE

To an aliquot of solution containing 100 g of Ni (II) in a separatory funnel, 10 ml of buffer solution of pH 7 and 12 ml 10-2 M HHMCP in chloroform were added. After shaking for 4 minutes, separatory funnel was kept for equilibrium and allowed to separate into two layers. The absorbance of the extracted yellow complex was recorded at 495 nm against chloroform black. A calibration graph was prepared and unknown amount of Ni (II) was determined from the calibration curve. Raffinates were analyzed for determination of Ni (II).

RESULTS AND DISCUSSION

a) Absorption Spectrum:

After extraction, Ni (II): HHMCP complex present in organic phase was scanned from 200 nm to 1000 nm against reagent blank. Maximum Absorbance value was observed at 495 nm. Therefore 495 nm was selected for the absorbance measurement throughout the experiments.

b) Effect of pH:

The absorbance of the organic phase was measured as a function of pH of the aqueous phase. The complexation of Ni (II) was carried out at pH range from 1-12. The data obtained shows maximum absorbance at pH 7.0. In more acidic or more basic solutions, it was found that absorbance decreases (Figure 1)

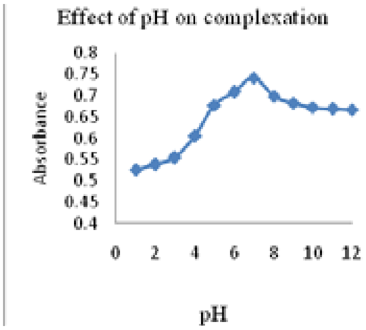

Fig. 1 Effect of pH

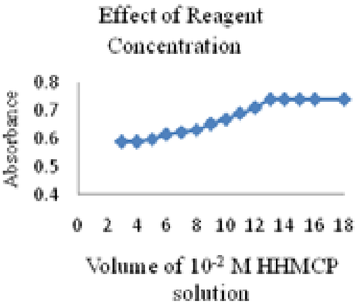

Fig.2 Effect of HHMCP reagent concentration

c) Effect of HHMCP reagent concentration

The minimum amount of reagent required for complete complexation of 100µg of Ni (II) was studied by varying the concentration of HHMCP. The results obtained from the plot of absorbance versus concentration of HHMCP indicate that 13 ml of 10^{-2} M reagent solution was sufficient for the quantitative extraction and spectrophotometric determination of 100µg Ni (II) (Figure 2). Addition of more reagent did not interfere with complexation and extraction of the complex. Further study of complexation was carried out by using 14 ml of 10^{-2} M HHMCP solution in chloroform to ensure the complete complexation.

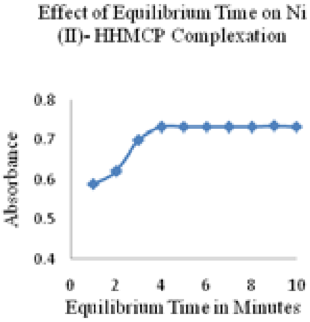

Fig.3 Effect of Effect of equilibrium time

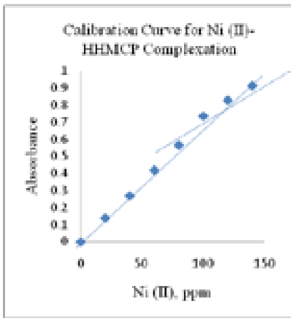

Figure 4: Calibration Curve for Ni (II)-HHMCP Complexation

d) Effect of equilibrium time

The minimum equilibrium time for complete complexation of 100µg Ni (II) was studied by varying the equilibrium period from 5 seconds to 10 minutes. The results obtained from the plot of absorbance versus equilibrium time indicated that minimum 1 minute equilibrium time was required for the quantitative extraction and spectrophotometric determination of 100µg of Ni (II) (Figure 3). It was also observed that equilibrium time above 1 minute did not affect the complexation and extraction of the complex. Thus further study of complexation was carried out by using 3 minutes as an equilibrium period.

Calibration curve:

A calibration graph of Ni (II) was prepared by complexing varying amount of Ni (II) in the range 0µg to 160µg with 14 ml 10^{-2} M HHMCP in chloroform. Plot of absorbance versus concentration of Ni (II) gave a straight line indicating that that Beer's range up to 100µg of Ni (II) at 495 nm. (Figure 4)

Mole ratio method:

Mole Ratio Method is used to determine the composition of the complex. Complexation was carried out by treating equimolar solutions of Ni (II) and HHMCP. Plot of absorbance versus mole ratio gave two lines intercepting each other at mole fraction 1. This indicates metal to ligand ratio 1:1. (Figure 5)

Job's continuous variation method:

Job's Continuous Variation Method is also used to determine the composition of the complex. Complexation was carried out by treating equimolar solutions of Ni (II) and HHMCP For complexation of Ni (II) varying moles of Ni (II) were treated with varying moles of HHMCP in chloroform to obtain mole fraction 0.1 to 1.0. Plot of absorbance versus mole fraction also suggest metal to ligand ratio 1:1. (Figure 6)

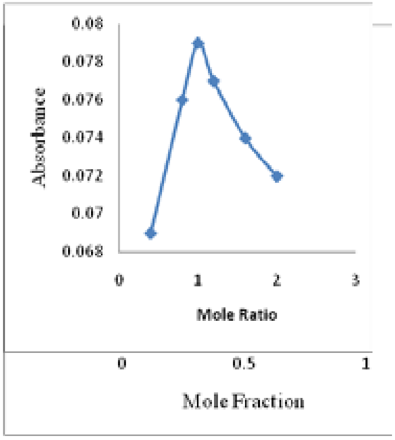

Figure 6: Job's Continuous Variation

Method

Effect of foreign ions:

Under the optimum conditions the effect of various cations and anions on the extraction and spectrophotometric determination of 100µg Ni (II) was studied by adding known amount of foreign ion in interest to Ni (II) aqueous solution before adjusting the required pH. Complexation was carried out as per the method mentioned above.(Table 1)

In case of intensive interference of some foreign ions the test was repeated with successively smaller amount of the same foreign ion. The tolerance for the added foreign ion was decided as the largest amount that give error less than 2 % in the extractive determination of Ni (II) at 495 nm

Table 1: Effect of Foreign Ions on Extraction of Ni (II)

Foreign Ion added	Amount Tolerated in μg
Sn (II)	20
Ru (II)	20
Mn (II)	10
Fe (II)	15
Co (II)	15
Cu (II)	15
Zn (II)	10
Cd (II)	10
Cr (VI)	30
Pd (II)	25

Applications:

To study the analytical applicability of the proposed method, it was applied for separation and spectrophotometric determination of Ni (II) from real samples such as Ni (II) from Raney Nickel catalyst, monel metal etc. (Table 2). The results were compared with those obtained using the traditional methods. As seen, the results of two different methods are in satisfactory agreement.

Table 2: Determination of Ni (II) from real Samples

Samples	Ni (II) content (%)	
	Certified Value	From Complexation with HHMCP
Nickel Aluminum Alloy Powder	50	50
Monel Metal	67	66.4
Cupronickel	25	24.7

Conclusions

An extractive spectrophotometric method was developed for estimation of Nickel (II). 2-[(E)-N-(2—{[2-[(E)-[(2-hydroxyphenyl) methylildene] amino] phenyl} (methyl) amino} phenyl) carboximidoyl] phenol (HHMCP) was synthesized[18] and successfully used for quantitative extraction of Nickel (II) at pH 7.0. Since the equilibration time is very less, the method is very quick. The method is applicable for determination of Nickel (II) from alloys and catalys

REFERENCE

1. Merian, E.; Anke, M.; Stoppler, M. Elements and Their Compounds in the Environment, Vol. 2, Wiley, VCH: Weinheim; 2004. | 2. Wand, K. Nickel Trace Elements in Life Science, Chinese Measurement Press: Peking; 1991. | 3. Zerner, B. Bioorg. Chem.1991, 19, 116. | 4. Templeton, D. Biological Monitoring of Chemical Exposure in the Workplace, Word ealth Organization: Geneva; 1990. | 5. Franson, M.A.H. Standard Methods for Examination of Water and Waste Water, American Publication Health Association; Washington, D.C., USA; 1995. | 6. Safavi A, Abdollahi H, Nezhad MRH, Kamali R., Spectrochim Acta A, 2004; 60: 2897. | 7. Bezerra MA, Conceição ALB, Ferreira SLC., Anal Bioanal Chem, 2004; 378: 798. | 8. Manzoori JL, Karim-Nezhad G., Anal Chim Acta, 2004; 521: 173. | 9. Sun Z, Liang P, Ding Q, Cao J., J Hazard Mater, 2006; 137:943. | 10. Shemirani F, Jamali MR, Kozani RR, Salavati-Niasari M., Sep Sci Technol, 2006; 41: 3065. | 11. B. Patel, Nitin Kumar and K. K. Desai, Asian J. Chem., 15(2), 751-754 (2003). | 12. K. Zarei, M. Atabati and Z. Malekshabani., Anal. Chim. Acta.556(1), 247-54 (2006). | 13. A. P. Kumar, P. R. Reddy and V. K. Reddy, J. Autom. Methods. Manag. Chem., 48768 (2007). | 14. M. K. Naik and N. V. Thakkar, Indian J. Chem., 34A, 410-411 (1995). | 15. G. A. Shar and G. A. Soomro, The Nucleus, 41(1-4), 77-82 (2004). | 16. X. Fan, G. Zhang and C. Zhu, Analyst, 123, 109-112 (1998). | 17. N. Hokoufi, F. Shemirani and F. Memarzeadeh, Anal. Chim. Acta., 601(2), 204-211 (2007). | 18. Ghanasham B. Sathe, Vikas V. Vaidya, Ravindra G. Deshmukh, Maharudra B. Kekare, Vikas S. Kulkarni , Atul C. Chaskar, Journal of Applicable Chemistry, 2013, 2 (3): 433-437 |