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UV-VIS spectrophotometric determination of Nickel (II) using 3, 5-dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone (DMHBIH) from polluted water of Bagmati River, Kathmandu, Nepal

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Abstract
The Bagmati River is being polluted excessively in such a manner that if the trend continues then obviously the Bagmati will be dead for all phenomenon. Similarly Bagmati is also more or less contaminated with the heavy metals such nickel, lead, mercury, cadmium and chromium. The contamination may be due to the waste disposed near the river side, sewerage discharge and industrial waste water effluents and river bank encroachment. Nickel (II) is a metal ion appearing together in a wide variety of samples of the food industry, in the manufacture of alloys, the production of heat-resistant steels and is present in small amounts in most soils, plants and in drainage water. Colorimetry is defined as the method of trace analysis that involves absorption of radiations having frequencies in the visible spectrum. This is done using a Spectrophotometer that transmits radiation from a source through a sample cell and into a detector which measures the absorbance of the sample. The wavelength of radiation absorbed depends on the identity and amount of substance present in the sample. The relationship between these factors is called the Beer-Lambert law, the Bouger-Beer law or more simply, Beer's law. Thus, in this experiment, UV-Vis Spectrophotometeric determination of Nickel (II) with 3, 5-Dimethoxy-4-hydroxybenzaldehyde isonicotinoyl hydrazone (DMHBIH) reagent was proposed in basic buffer solution (pH 9.0). Samples were collected from Bagmati river of Kathmandu and the maximum absorbance was observed in the pH range 8.5-9.5. The molar absorptivity and Sandell's sensitivity of Nickel (II) complex with DMHBIH at λ max 386 nm was found to be 1.82 x 10^4 L $mol^{-1}cm^{-1}$ and 0.0082 $\mu g/cm^2$. Beer's law validity range varies from 0.234 to 2.94 µg /ml. Nickel (II) forms 1:1 complex with DMHBIH and stability constant of Nickel (II) complex was $9.4X10^6$. The first order derivative amplitude was measured by the peak height method at λ max 440 nm. The second order derivative amplitude was measured by the peak height method at λ max 470 nm. The developed spectrophotometric method was applied for the determination of Nickel (II) in alloy samples.

Keywords: Bagmati river, Nepal, Nickel (II), 3, 5-Dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone (DMHBIH).

1. Introduction
UV–Vis spectrophotometry is the most common technique used for nickel (II) determination owing to its simplicity and low cost. Accordingly, the present study reports a facile procedure to determine micro levels of nickel (II) in polluted water collected from Bagmati river of Kathmandu, Nepal. The Bagmati River is a major river in Nepal which originates in the central northern mountains and runs through urban areas of Kathmandu valley. Bagmati River has its' origin in Bagdwar from the Southern slope of Shivapurilekh, north of Kathmandu basin at an altitude of about 2650m and flows straight to south-west cutting Mahabharat range [1]. The Bagmati is an important tributary of the Ganges and has a catchments area of 3710 km2 in Nepal. The river rises in the Kathmandu Valley, which comprises just 15% of the area of the Bagmati Basin in Nepal. The basin can be divided into three parts: the Upper Bagmati Basin (662 km^2), the Middle Bagmati Basin and the Lower Bagmati Basin. The drainage area of the Bagmati as far downstream as Bhandarikharka (downstream of Chobhar) is 662 km^2 [2]. Bagmati River inside the Kathmandu Valley has been heavily polluted due to increasing rapid population growth; unplanned expansion of Kathmandu city and haphazard waste disposal into the river [3]. The river is also used for discharging of untreated sewage and industrial effluents

into them and the place for dumping of solid wastes and direct sewerage discharge into the river have been the major problem in the management of Bagmati River. So far many organizations are working to prevent pollution of Bagmati River but, conditions remains as it is the same or even worse [4].

The term "metal" designates an element which is a good conductor of electricity and whose electric resistance is directly proportional to the absolute temperature [5]. In addition to this distinctive characteristic, metals share several other typical physical properties such as high thermal conductivity, high density, malleability and ductility the ability to be drawn into sheets and wires. Viewed from the standpoint of environmental pollution, metals are classified into three categories: (1) noncritical, (2) toxic, but very insoluble or very rare and (3) very toxic and relatively accessible [6]. Copper, zinc and lead come under the third category of pollutants [7]. Heavy metals are found naturally in the earth, and become concentrated as a result of human activities. Common sources are mining and industrial wastes; vehicle emissions; lead-acid batteries; fertilizers; paints; treated woods; aging water supply infrastructure; and micro plastics floating in the world's oceans [8]. Nickel is biologically important, being an essential trace element in human diet. Since nickel is extensively used in the preparation of alloys and catalysts, its determination may be considered as interesting research activity. The determination of nickel in various samples in which it is found at low levels requires the use of sensitive and selective procedures. The obvious reasons of determining nickel by spectrophotometric method are due to its experimental simplicity, rapidity and the wide applicability of the procedure. Thus, an experiment was conducted to determine heavy metal as Nickel (II) extracted from polluted water from Bagmati River, Kathmandu, Nepal using UV-VIS spectrophotometer with 3, 5-dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone (DMHBIH) reagent. Many different organic compounds have been used as spectrophotometric reagents for the determination of nickel (II) in aqueous solutions [9].

2. Experimental

2.1 Apparatus

Spectrophotometric measurements were made in a Schimadzu 160A microcomputer based UV-Visible spectrophotometer equipped with 1cm quartz cells, an ELICO LI-120 digital pH meter for pH adjustments and Sartorius electronic balance was used for weighing.

2.2 Reagents

All reagents used were of A.R grade unless otherwise stated. All solutions were prepared with distilled water. The standard Ni (II) solution (0.1 M) was prepared by dissolving 8.28 g of Nickel nitrate ($Ni(NO_3)_2$, LR SD fine) in distilled water in a 250-ml standard flask. The reagent 3, 5-dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone (DMHBIH) (Figure 1) was synthesized by refluxing equimolar amounts of 3, 5-dimethoxy-4-hydroxy benzaldehyde and isonicotinoyl hydrazine. In a 250 ml round bottomed flask hot ethanolic solution of 3, 5-dimethoxy-4- hydroxyl benzaldehyde(1.82g, 0.01 mole) and hot ethanolic solution of isonicotinoylhydrazine (1.3615g, 0.01mole) were mixed and refluxed using water condenser for 3 hours. On cooling the reaction mixture, a yellow coloured product separated out, which was collected by filtration and washed with double distilled water. The resulting hydrazone was recrystallized using 50% ethanol (yield, 76%, mp 221^{O}C). Buffer solutions were prepared by 1M Hydrochloric acid -1M Sodium acetate (pH 0.5-3.5); 0.2 M Acetic acid -0.2 M Sodium acetate (pH 4.0-7.0); 2M Ammonium chloride -2M Ammonium hydroxide (pH 7.5-12.0). The reagent solution (0.01 M) was prepared by dissolving 0.3022 g of DMHBIH in 100 ml of Dimethylformamide (DMF). The reagent solution was stable for 48 hours. The molar absorptivity and Sandell's sensitivity of Ni (II) at λ max 386 nm was found to be 1.82×10^4 L mole^{-1}.cm-1 and 0.013 µg/cm^2. Beer's law validity range 0.414-10.36 µg/ml. Ni (II) forms 1:1 complex with DMHBIH and stability constant of the complex was 8.99×10^6. The reagent gives bright yellow coloured water soluble complex in alkaline buffer solution and the maximum absorbance was observed at pH 9.0.

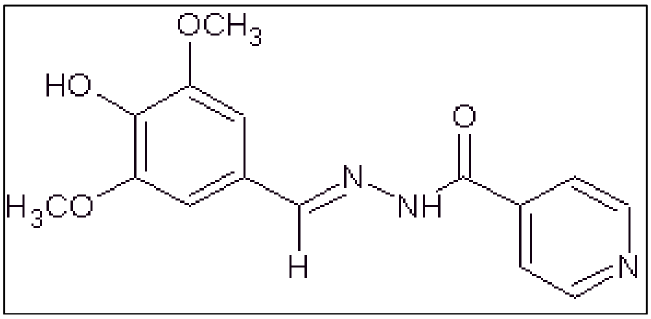

Fig 1: Structure of 3, 5-Dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone (DMHBIH).

Reaction with metal ions

Table 1: Analytical Characteristics of DMHBIH

Metal ion	pH	λ max (nm)	Molar absorptivity (ε) (Lmol^{-1}cm^{-1}) x 10^4
Cu (II)	9.21	386	1.771
Pb (II)	9.17	386	1.823
Ni (II)	9.13	386	1.275

The reactions of some important metal ions were tested at different pH values. The samples were prepared in 10 ml standard volumetric flasks by adding 3 ml of buffer (pH 1.0-11), 0.5 ml of metal ion (1×10^{-3}M) and 0.5 ml of 1×10^{-2} MDMHBIH solutions. The solution mixture was diluted up to the mark with distilled water. The absorbance was measured in 200-800 nm range against reagent blank. The results are summarized in Table-1.

2.4 Direct spectrophotometry

An aliquot of the solution containing 0.414 to10.36 µg/mL of nickel (II), 3 ml of buffer solution (pH 9.0) and 0.5 ml of 1×10^{-2}M DMHBIH reagent were taken in a 10ml standard volumetric flask and the solution was diluted up to the mark with distilled water. The absorbance of the solution was recorded at 386 nm in a 1.0 cm cell against corresponding reagent blank prepared in the same way but without nickel (II) metal solution. The measured absorbance was used to compute the amount of nickel (II) from the calibration plot. Wavelength values are plotted against absorbance.

2.5 First order derivative spectrophotometry

For the above solution, first-order derivative spectrum was recorded in the wavelength range from 350-800 nm. The derivative peak height was measured by peak zero method at

440 nm. The peak height was plotted against the amount of Nickel (II) to obtain the calibration curve.

3. Results and Discussion

3,5-Dimethoxy-4-hydroxybenzaldehyde isonicotinoylhydrazone (DMHBIH) reagent is a blend of a carbonyl compound and a hydrazine. The reagent solution is stable for 48 hrs. in alkaline medium. The ligand presumably coordinates the metal ions to give a neutral water soluble complex.

3.1 Determination of Nickel (II) using DMHBIH

Nickel (II) reacts with DMHBIH in alkaline medium to give bright yellow coloured water-soluble complex. The colour reaction between Nickel (II) and DMHBIH are instantaneous even at room temperature in the pH range 8.5-9.5. The absorbance of the bright yellow coloured species remains constant for more than 2hrs. The maximum colour intensity is observed at pH 9.0.

Table 2: Physico-Chemical and Analytical Characteristics of [Nickel (II)-DMHBIH] Complex

S. N.	Characteristics	Results
1	Colour	Bright Yellow
2	λ_{max}(nm)	386
3	pH range (optimum)	8.5-9.5
4	Mole of reagent required for full colour development	10 folds
5	Molar absorptivity ($L.mol^{-1}cm^{-1}$) $x10^4$(ε)	1.82×10^4
6	Sandell's sensitivity($\mu g/cm^2$)	0.01302
7	Beer's law validity range(µg/mL)	0.414-10.36
8	Optimum concentration range(µg/mL)	0.83 to 9.32
9	Composition of complex(M:L) obtained in Job's and mole ratio methods	1:1
10	Stability constant of the complex	8.99×10^6
11	Standard deviation	0.000026
12	Relative standard deviation (%)	0.06

A 10-fold molar excess of reagent is adequate for full colour development. The order of addition of buffer solution, metal ion and reagent has no adverse effect on the absorbance. The complex formation reaction between Nickel (II) and DMHBIH has been studied in detail based on the composition of the complex as determined by using Job's and molar ratio methods. Important physico-chemical and analytical characteristics of nickel (II) and DMHBIH are summarized in Table-2.

Applications

In order to test the reliability of the proposed method it was applied to the determination of Ni (II) in tap water, ground water and river water.

Preparation and analysis of water samples

Two tap water samples, ground water and river water were collected and were analyzed after filtering through Whatman filter paper. The samples were Spiked with Ni (II) in order to validate the procedure. It can be seen that the recovery of spiked samples was good. The results indicate that the proposed method was applicable for the determination of Ni (II) in real samples.

4. Conclusion

From the above discussion, it can be concluded that DMHBIH is a potential reagent for the derivative spectrophotometric determination of Ni (II). It is very easy to synthesize DMHBIH, a novel class of reagent. The present derivative method is simple and rapid without the need for heating or extraction compared to other reagents that were used for the spectrophotometric determination of Nickel (II). The above method is comparable with other recently reported spectrophotometric methods for the determination of nickel (II) [10]

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