SENSITIVE AND HIGHLY SELECTIVE SPECTROPHOTOMETRY METHOD FOR DETERMINATION OF NICKEL

Nilima Das, Dr. Seema Agrawal
Department of Chemistry, Dr. A.P.J. Abdul Kalam University, Indore, M.P., India

ABSTRACT :
The precise measurement of trace metal ions is crucial to many fields, including analytical chemistry, environmental monitoring, and industrial processes. Conventional spectrophotometric analysis has limitations that can be circumvented by using nickel (II) ion complexes with an appropriate complexing agent. The use of nicotinohydroxamic acid (NHA) in a phosphate-borax buffer media at pH 9.0 was investigated as a potential spectrophotometric technique for assessing nickel (II). With a molar absorptivity of 1.37 104 L mol-1 cm-1 at 530 nm, Beer's law absorption spectra were seen for the sensitized compound throughout a nickel (II) concentration range of 0.43-8.56 g mL-1. Nickel and N-hydroxyacetic acid made up around half of the molar ratio in the sensitive combination. Scientists looked at the potential impact of different ions.

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1. INTRODUCTION

The detection of trace elements is essential in many scientific and industrial disciplines. Nickel is a standout among these elements because of its numerous important roles in sectors as varied as metallurgy, electronics, environmental monitoring, and even biology. Precision in measuring even tiny quantities of nickel is necessary to safeguard product quality, environmental safety, and human health. Spectrophotometry has become an essential tool in this setting as a result of its precision and consistency in determining nickel content. In addition to its application in measuring nickel concentrations, spectrophotometry provides insight into the samples' composition, structure, and behavior by using the electromagnetic light's interaction with the material.

Spectrophotometric measurement of nickel is based on the interaction of light and matter, specifically the absorption of light by nickel ions in a sample solution. The theoretical basis for this method is the Beer-Lambert equation, a staple of spectroscopy. According to this law, absorbance is directly related to the product of the concentration of the absorbing species and the time it takes for light to travel through the solution. Creating a calibration curve using the established relationship between absorbance and concentration aids in nickel testing. This method takes use of the fact that nickel ions absorb light at certain wavelengths when their electrons are stimulated to higher energy levels. The quantity of light transmitted through a substance reduces as a consequence of absorption, and this loss is proportional to the nickel concentration of the substance according to the Beer-Lambert equation.

A spectrophotometric investigation relies heavily on the experimental design and equipment utilized in the study. An ordinary spectrophotometer will include a light source, a monochromator, a sample holder, a photodetector, and a data processing device. Since many electronic transitions occur in the ultraviolet (UV) and visible (Vis) regions of the electromagnetic spectrum, a UV-Vis

spectrophotometer is often used for nickel analysis. The chemical containing nickel is dissolved in a selected solvent to ensure that the sample is homogeneous and completely dissolves. The prepared solution is placed in the sample container and is then exposed to a monochromatic light beam. Light transmission is measured by the spectrophotometer's photodetector, and light absorption is calculated by the instrument's CPU. The absorbance of a sample is compared to the absorbance of a series of standard solutions whose nickel contents are known, yielding a calibration curve. Using this curve as a benchmark, the nickel concentration of samples that have not been analyzed may be calculated.

The spectrophotometric determination of nickel has several applications in industry and academia. Nickel concentrations may be measured in environmental samples such water, soil, and air with the use of spectrophotometers. Human activities, such as mining and automobile emissions, are major contributors to nickel pollution. Accurate nickel detection and quantification is crucial for conducting reliable environmental risk assessments and creating effective mitigation solutions. In the metallurgical industry, spectrophotometry is used for quality control throughout the production of alloys to ensure that the desired nickel concentration is maintained. It is used in the pharmaceutical business for researching nickel-containing drug formulations, which is essential for staying in compliance with regulations and keeping patients safe. The presence of nickel in enzymes and proteins demonstrates its biological importance and emphasizes the need of accurate nickel quantification in biochemical research.

In addition to its useful applications, spectrophotometric nickel determination is significant because it elucidates the properties and interactions of individual elements. Understanding the intricate electronic structure of nickel ions is crucial to studying their coordination chemistry and chemical reactivity. Spectrophotometric instruments are becoming more precise, sensitive, and automated as a result of technological advancement, leading to a wider range of applications in both academic and industrial settings. Knowledge gained from spectrophotometric studies expedites discoveries in several scientific disciplines.

2. REVIEW OF LITERATURE

Raafid, Esraa et al., (2020) Spectrophotometers provide for a quick and simple method of determining the amount of Ni (II) present in alloy samples. First, the ligand known as 1-((4-(1-(2-hydroxyphenylimino) ethyl)phenyl)diazenyl)naphthalene-2-ol (HPEDN) was manufactured by combining p-aminoacetophenone and 2-naphtol in a reaction. After that, the azo-schiff reaction was produced by reacting the product of the previous reaction with 2-aminonaphtol. The optimal conditions for complex formation were found to be pH=9, 25°C, and 15 minutes. The molar absorptivity for a Nickel(II) concentration range of 1.1-7.1 g/mL was determined to be 0.2648 x 104 L1cm1 under ideal conditions. This was the value that was found. It was found that a concentration of ligand of 2 x 10-4 M was the most effective one. The LOD and LOQ for concentrations were both established at the same value of 1.3082 g.mL1. The stoichiometric ratio of the Ni:HPEDN chelate is 1:2. This particular use of the spectrophotometric method is notable for its speed, precision, and sensitivity. Data from flame atomic absorption spectrometry were verified by comparing them to data that had been published in the past.

Tariq, Zianab& Adnan, Shaimaa (2019) Micelle-mediated extraction was utilized in order to achieve a higher level of preconcentration of the chemical reagent 2-(2-bromophenyl)imino)methyl)-4- (5,6-dimethylpyridin-2-yl)phenol in preparation for nickel analysis.In order to dissolve the chelating agent known as 2-(2-bromophenyl)imino)methyl)-4- (5,6-dimethylpyridin-2-yl)diazenyl)phenol, a solution of the nonionic surfactant known as Triton X-114 was used. We used UV-Vis spectrophotometry with a maximum wavelength of 650 nm in order to get an accurate reading of the analyte concentration. Micelle mediation extraction was used to

remove Ni(II) ions from environmental samples. The optimal extraction conditions (pH, surfactant and reagent concentrations, incubation time, and temperature, among other things) were determined through classical optimization, and the analysis of variance was utilized to statistically evaluate the calibration plot.

Kumar, B. Natesh et al., (2016) Two imine ligands, namely (E)-N1-(2-hydroxy-5-nitrobenzylidene) isonicotinoylhydrazone and 2-(4-fluoro benzylideneamino) benzenethiol, are put into the system in order to make the detection of nickel(II) both quick and simple. At pH 4.0 and 4.7, a reaction takes place in which nickel(II) interacts with the ligands in a stoichiometric ratio of 1:1 to produce red and light purple complexes. It may be concluded that Beer's law was adhered to by the complexes since they maintained a correlation value of 0.9996 across a concentration range of 0.8-20.0 g L-1. Sandell's sensitivity was 0.98 ng cm-2 for the red complex, and 0.91 ng cm-2 for the light purple complex. However, the light purple complex was more sensitive than the red complex. It was determined that the absorption ranged from 5.1 104 to 6.3 104 mol1 cm1. It was discovered that the concentration of nickel(II) varied from 0.89 ng L-1 to 0.82 ng L-1. This technique was put to use in order to get precise readings of nickel (II) concentrations in a wide range of fluids and soils. For the purpose of evaluating the in vitro antibacterial properties of the complexes, Bacillus subtilis, Staphylococcus aureus, Escherichia coli, and Pseudomonas aeruginosa were used as test organisms. For the purpose of determining the complexes' binding affinities, band gap calculations based on density functional theory were utilized.

Mandhare, Datta & Barhate, Vasant (2015) An efficient and straightforward spectrophotometric method for assessing Nickel (II) has been developed by the utilization of the Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol (HPIMNP). From an aqueous solution with a pH ranging from 7.8 to 8.2, Ni (II) may be extracted into n-butyl alcohol with a quantitative yield of 99.92% by using HPIMNP in the presence of 2 ml of an ammonium chloride solution with a concentration of 5 M. At a wavelength of 480 nm, the n-butyl alcohol extract possesses its maximum penetrating power (max penetrating power). Beer's rule is valid at concentrations ranging from 0.2 to 2.0 g/ml, and this is the range that it applies to. The sandell sensitivity of nickel-HPIMNP is 0.067 g.cm-2, and its molar absorptivity is 882.35 L mole-1 cm-1. With the use of Job's continuous variation and the Mole ratio technique, we were able to conclude that the extracted species are made up of Ni and HPIMNP in a ratio of 1:1. Researchers have looked into a variety of ionic interferences and the effects such interferences have. The Ni (II) content of a number of different alloy samples was determined by following the indicated procedure.

Rao, M. et al., (2015) The element nickel (II) may now be quantified through the use of a spectrophotometric method that is not only simple but also extremely accurate. In aqueous solution with a pH ranging from 3.0 to 11.0, the reagent 2-amino acetophenone isonicotinoyl hydrazone, also known as 2-AAINH, may form a brilliantly yellow combination with nickel (II). The molecule has a Sandell's sensitivity of 0.56 x 10-2 g cm-2, with a maximum absorbance of 470 nm, a molar absorptivity of 1.05 x 104 L mo1-1cm-1, and a molar absorptivity of 1.05 x 104 L mo1-1cm-1. Beer's rule states that concentrations of Ni (II) should fall between 0.29 to 6.16 g mL-1. The detection of nickel (II) can also be accomplished by the use of second-order derivative spectrophotometry. The possibility that numerous different ionic species can interact with one another is now the subject of investigation. Testing of this kind might be beneficial for analyzing samples such as drinkable water, metals such as aluminum and steel, and oils such as groundnut, amongst other things.

Abbas, Esraa et al., (2013)The reaction of imidazole with sulfadiazine, which served as the azo reagent, resulted in the development of a novel chromogenic reagent. This allowed for the creation of an exceedingly sensitive spectrophotometric technique for detecting

nickel (II). When combined in water with a pH of 7, the reagent and nickel(II) produce a complex that is dark brown in color. Fourier transform infrared (FT-IR), ultraviolet-visible nuclear magnetic resonance (1HNMR), and carbon-13 nuclear magnetic resonance (13CNMR) spectroscopies were used to identify the reagent and its complex. The formula and charge of the produced complex were determined using molar ratio and molar conductivity measurements. According to the Sandell colorimetric technique, the chemical has a sensitivity of 0.0177 g/cm2 and has a maximum absorbance at 486 nm (with a molar absorptivity of 0.3299 x 104 L.mo1-1.cm-1). This is based on its molar absorptivity.The linearity of the approach is demonstrated by a correlation coefficient of 0.9995 and a detection limit of 0.1927 ng/mL-1. The range of Ni (II) values that are covered by this analysis is from 0.5 to 7.6 ng/mL. The relative standard deviation of the method was calculated to have a value of 0.39221, while its recovery was determined to be 98.7%, and its error was found to be 1.3%. The intricacy of the system indicated that the M:L ratio should be 1:2. It was used to measure Ni(II) in alloy, and it was chosen because it is a sensitive, accurate, and quick spectrophotometric approach. It was determined that these findings and the findings acquired using flame atomic absorption spectrometry are consistent with one another.

3. RESEARCH METHODOLOGY

Apparatus

All absorbance readings were taken using a Systonics UV-VIS spectrophotometer that had matching glass cells of 1 centimeter in size. A digital pH meter (ELICO LI- 120) equipped with a glass-calomel electrode was used to determine the pH values and make the necessary adjustments.

Reagents

The experiment was carried out with water that had been through two distillation processes and reagents that were of an analytical reagent grade only. A nicotinohydroxamic acid (NHA) reagent solution with a concentration of 0.001M was produced by the use of the procedure that has been disclosed. After first being created from a stock solution of Ni (II) (3.6 x 103M) in bidistilled water, the metal solution was then standardized via the use of conventional methods, and lastly, it was diluted to the appropriate concentration. To make the buffer solution, the appropriate amounts of borax and potassium hydrogen phosphate were combined with a 10% weight-per-volume (w/v) solution of sodium hydrogen carbonate and a 1% solution of Triton X-100 dissolved in distilled water. The interference of different ions was studied by using a solution of alkali metal salts at a concentration of 5% (w/v) and a solution of other metal salts at a concentration of 0.2% (w/v).

Procedure

For the purpose of measuring all absorbance values, a Systonics UV-VIS spectrophotometer equipped with 1 cm matched glass cells was utilized. With the assistance of a digital pH meter (ELICO LI- 120) and a glass-calomel electrode, we determined the pH values and then performed the necessary adjustments.

4. DATA ANALYSIS AND INTERPRETATION

Absorption spectra

A spectrum absorption was seen for both an empty reagent vial and the grass-green Ni (II)-NHA complex when Triton X-100 was present. When the surfactant is present, as shown in Figure 1, the complex absorbs very strongly at the wavelength that corresponds to its greatest level of absorption. This is the rationale behind why the analytical wavelength for detecting metal ions was decided to be 530 nm.

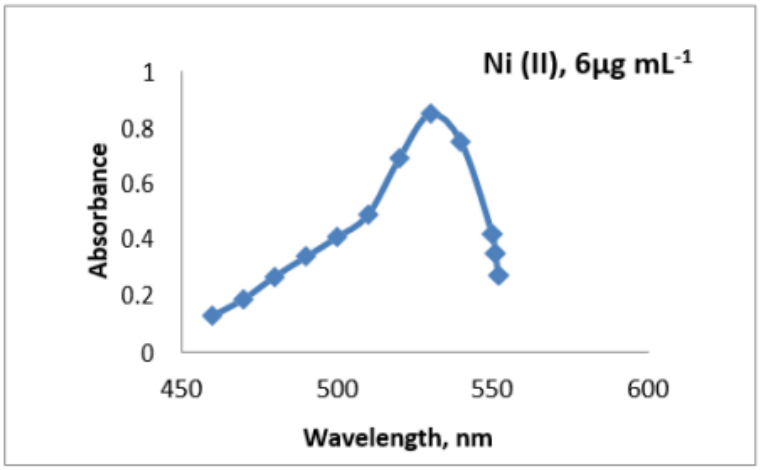

Figure 1: Absorption spectra of Ni (II) – NHA complex

Nature of surfactant

Several different types of surfactants were used in the experiment to assess the absorbance of the Ni (II)-NHA complex. Sodium lauryl sulfate (also known as SLS), cetyltrimethylammonium bromide (often known as CTAB), cetylpyridinium bromide (also known as CPB), Triton X-100, tween-80, and tween-20 are a few examples of these types of ingredients. Triton X-100 was shown to be the most effective solvent for accelerating, stabilizing, and absorbing complex formation by the group that did the research. The researchers investigated the effect on the method's sensitivity by increasing the quantity of Triton X-100 from 0.5 to 3.5 mL and from 0.5 to 3.0% (w/v), respectively. It was discovered that the ideal amount of Triton X-100 solution has an absorbency of between 1.5 and 2.0 milliliters per milliliter of liquid. Because of this, 2.0 milliliters of Triton X-100 1% was utilized in the investigation.

Statistical analysis of results

In order to determine whether or not Beer's rule is applicable to spectrophotometric measurements of Ni(II) via chelation with NHA under optimal reaction circumstances, the rule was put to the test. A linear connection was discovered to exist between the two variables when a borate buffer and NHA were utilized (Figure 2). This relationship was shown to exist throughout a concentration range of 1.71-8.56 g mL-1 for Ni(II). Table 1 displays the analytical parameters that are most significant for both Ni (II) and NHA. At an apparent molar absorptivity of 1.371 104 L mol-1cm-1, the Sandell was capable of achieving a sensitivity of 0.538 g/mL/cm2 in its measurements. If there is a high correlation coefficient and a low standard deviation in the calibration graph, then Beer's law may be utilized to do an analysis of the absorbance data.

Table 1: Analytical Parameters of [Ni(II)–NHA] complex

Characteristics	**Results**
Colour	Grass green
λ max (nm)	530
pH range (optimum)	8.5-9.5
Mole of reagent required per mole of metal ion for full colour development	10 - folds
Molar absorptivity (L mol-1 cm-1)	1.371 × 104
Sandell's sensitivity (µg/mL/cm2)	0.538

Beer's law validity range (µg/mL)	2.57-6.85
Optimum concentration range (µg/mL)	1.74 - 8.56
Composition of complex (M:L) obtained in Job's and mole ratio method	1:2
correlation coefficient (γ)	0.996
Standard deviation (%)	0.11

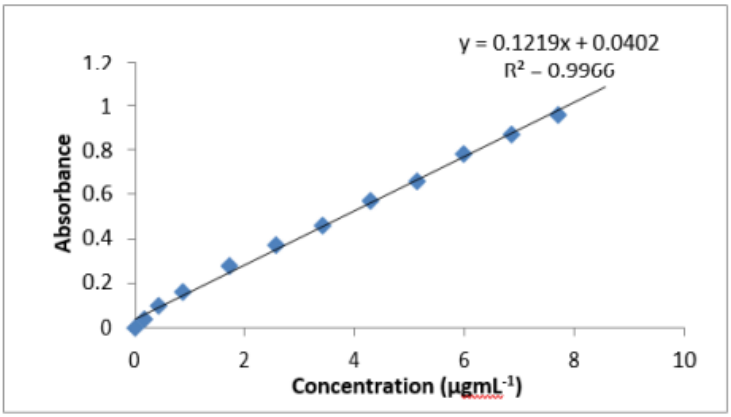

Figure 2: Applicability of Beer's Law

Job's method of continuous variation

The molecular weights of the Ni (II) and NHA solutions that were created varied, but the volumes remained the same. After shaking the test tubes, the absorbance readings at 530 nm were measured and recorded. According to the results of absorption measured against the ligand mole fraction, the complex is made up of two moles of nickel chelate and four moles of NHA.

Interference analysis

The influence that other ions could have on the experimental determination of Ni (II) has been the subject of a significant amount of research. Interference was found if the absorbance of the material under investigation changed by more than 5% as a result of the presence of an ion. The determination of Ni (II) was unaffected by any of the anions, even at a concentration that was 200 times higher than normal. The majority of cations do not alter the result of the computation even when their typical concentration is increased by a factor of one hundred. The addition of phosphate, thiourea, and thicyanate resulted to a 40-fold increase in the concentration of Fe(III), as well as a 20-fold increase in the concentration of both Cu(II) and Co(II).

5. CONCLUSION

Micelles as a solubilizing medium enhance the solubility and stability of the produced complex, which increases the sensitivity and decreases the detection limits. The optimized experimental conditions provide precise and accurate quantification of Nickel (II) ions by the manipulation of pH, complexing agent concentration, and micellar concentration. Real-world samples demonstrate the method's linearity and flexibility, highlighting its use. To better understand the dynamics of trace metal ions in increasingly complex systems, our data implies that spectrophotometric detection of Nickel (II) ions in micellar medium might be an important analytical tool. More study and validation of this method is needed to fully realize its promise and enable it to be customized to handle other trace metal ions and analytical challenges.

6. REFERENCES: -

1. Raafid, Esraa & Al-Da'amy, Muneer & Kadhim, Salih. (2020). Determination and Identification of Nickel(II) Spectroscopy in Alloy Samples Using Chromogenic Reagent(HPEDN). IOP Conference Series: Materials Science

and Engineering. 871. 012025. 10.1088/1757-899X/871/1/012025.
2. Tariq, Zianab& Adnan, Shaimaa. (2019). Statistical evaluation of spectrophotometric determination of nickel(II) using Micelle-mediated extraction.. Journal of Physics: Conference Series. 1294. 052064. 10.1088/1742-6596/1294/5/052064.
3. Raghunathan, Muthuselvi. (2017). Determination of Nickel (II) by Spectrophotometry in Micellar Media. Pharmaceutical Analytical Chemistry: Open Access. 03. 10.4172/2471-2698.1000129.
4. Weldeabzgi, Azeb & Reddy, Desam &nigussiemekonnen, Kebede. (2017). Spectrophotometric Determination of Nickel (II) in Soil and Standard Alloy Samples Using 5-Methyl-2-Acetylfuran-4-Methyl-3-Thiosemicarbazone (5-MAFMT). Communications in Soil Science and Plant Analysis. 48. 10.1080/00103624.2016.1269797.
5. Kumar, B. Natesh & Kanchi, Suvardhan& Sabela, Myalowenkosi&Bisetty, Krishna & Jyothi, n v. (2016). Spectrophotometric determination of nickel (II) in waters and soils: Novel chelating agents and their biological applications supported by DFT method. Karbala International Journal of Modern Science. 2. 10.1016/j.kijoms.2016.08.003.
6. Mandhare, Datta & Barhate, Vasant. (2015). Development of extractive spectrophotometric methodfor the determination of nickel (II) with Schiff base 2-[(2-hydroxyphenylimino)methyl]-4-nitrophenol. International Journal of Current Pharmaceutical Research. 7. 1069-1073. 10.22159/ijcpr.2016v8i4.15924.
7. Rao, M. & Khan, P. & Reddy, P.. (2015). Spectrophotometric determination of nickel (II) with 2-aminoacetophenone isonicotinoylhydrazone. Der Pharmacia Lettre. 7. 281-286.
8. Rao, M. & Khan, P. & Reddy, P.. (2015). Spectrophotometric determination of nickel (II) with 2-aminoacetophenone isonicotinoylhydrazone. Der Pharmacia Lettre. 7. 281-286.
9. Zalov, Ali &Gavazov, Kiril. (2014). Extractive Spectrophotometric Determination of Nickel with 2-hydroxy-5-iodothiophenol and Diphenylguanidine. Chemistry Journal. 4. 20-25.
10. Abbas, Esraa & Kadhim, Salih & Al-Da'amy, Muneer. (2013). Determination of Ni(II) in Alloy by Spectrophotometric Method with a new Chromogenic Reagent (IDPBS). International Journal of Pharmaceutical Quality Assurance. 11. 20-22. 10.25258/ijpqa.11.1.14.