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Novel and Easy Spectroscopic Method for Cadmium Estimation in Aqueous Samples

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A simple and an accurate method has been developed for the estimation of cadmium spectroscopically in water and soil systems. The method involves chelate bond formation of Dimethylglyoxime ligand with Cadmium under feasible and basic medium environment. The method shows 1:2 formation bond in between Cadmium and Dimethylglyoximine. The optimal condition for the formation of bonding between Cadmium and DMG and consequent determination of cadmium was established. The applicability of Beer-lambert law was found in between 0.03 to 0.900 µg/ml. The molar extinction coefficient of Cd (DMG) complex was found to be 1.5 × 105 mol^{-1} cm^{-1}. Finally, the method was compared with the already available methods and comparative results shows that the study carried out in the said research seems better and economically feasible.

Keywords: Cadmium, spectroscopy, aqueous, Jobs method, Mole ratio.

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Introduction

Traces of cadmium are important in industry, as a toxicant, as a biological non-essential, as an environmental contaminant, as a hazard at work, and more (1). It is a highly poisonous metal that has caused several fatalities (2). The development of a technique for the trace and ultra-trace measurement of this dangerous metal is crucial due to the growing environmental contamination caused by cadmium-based businesses and the usage of fossil fuels. One of the most effective instruments in chemical analysis is spectrophotometry, which is basically a trace-analysis method. Dimethylglyoxime has not yet been employed for the spectrophotometric measurement of cadmium, however it has been documented as a spectrophotometric reagent for nickel and copper (3). Its application in a very sensitive and specific spectrophotometric approach for determining cadmium traces is described in this study. In terms of sensitivity, selectivity, range of determination, simplicity, speed, pH/acidity range, thermal stability, accuracy, precision, and ease of use, the approach has clear advantages over current methods (4, 5).

I. Methodology

Estimation of copper: All reagents were purchased from Naizak Saudi Arabia. All the solution (stock and standard) were prepared freshly. Under recommended/established conditions, Cd (II) metal was estimated spectroscopically using UV-VIS Spectrophotometer. An Elico pH meter was used for the pH adjustments.

Cadmium Chloride solution: 0.1 g of cadmium chloride was dissolved in 10 ml of deionized water.

Dimethylglyoxime solution: 0.1 g of DMG dissolved in 10 ml of ethanol.

Buffer Solution: (Citric Acid Bis-Tris propane) Mix 1.0 M Citric acid and 1.0 M BIS-TRIS propane to produce the desired buffer pH.

λ_{max} determination of a Cd (DMG) complex solution: The procedure described by Lee et al. [6] was modified and applied for (λ_{max}), determination. 3 ml of metal solution, 3 ml of DMG solution 5 ml of buffer and 9 ml of deionized water was taken accurately in 20 ml volumetric

flask. The mixture was kept at room temperature for about 20 minutes to make the components to react and then optical density was measured in the range from 300 to 850nm in a time gape of 10 and 20 minutes. In between absorbance and wave-length a graph was plotted and from the graph λ_{max} was determined as shown in figure 1. The λ_{max} value and the maximum colour of the complex was observed at 534 nm. The colour formation of the complex was instantaneous and was stable up to 48 hours. There seems to be no effect of the reactants with change in their concentration ratio.

pH effect on (λ_{max}) of a solution: In a set of 10 Volumetric flasks (20ml) marked from 1 to 10, the cadmium (II) solution was transferred in a ratio 1 to 10 ml. To each volumetric flask, 5 ml of DMG solution and 3 ml of buffer solution was added. The volume of volumetric flask was made up to 20 ml with deionized water. The complete set of volumetric flasks were kept at room temperature for about 15 minutes. Then the absorbance was recorded for each volumetric flask solution at 534 nm (λ_{max}).

pH effect: The pH effect was observed for the formation of Cd (II) (DMG) to find out the optimum pH for complex stability in which 1 ml of cadmium and 2 ml of DMG was taken and then the effect of pH was measured from 4 to 11 using Citric Acid Bis-Tris propane buffer solution. The volume of each solution was kept 10.0 ml with double distilled water. The graph was plotted between pH and absorbance as shown in figure 2. As per the graph the highest colour development occurs from pH 4 to 11. There had not been found any change in colour below 4 or above 11 pH. Also, the complex provides highest absorbance value at 9.5 pH suggesting that the system favors basic medium for its preparation and for the stoichiometry of the complex.

II. Job's method of continuous variation

The Job's method was employed for the stoichiometric ratio determination of Cd (DMG) complex [7], in which a series of solution mixture with variable mole fractions of Cd (II) and DMG were prepared. To each solution mixture 2 ml of buffer solution was added and the total volume was made up to 20 ml with distilled water. All the volumetric flasks were mixed well and the optical density was measured at 535 nm. As per the graph plotted between corrected absorbance and mole fraction it

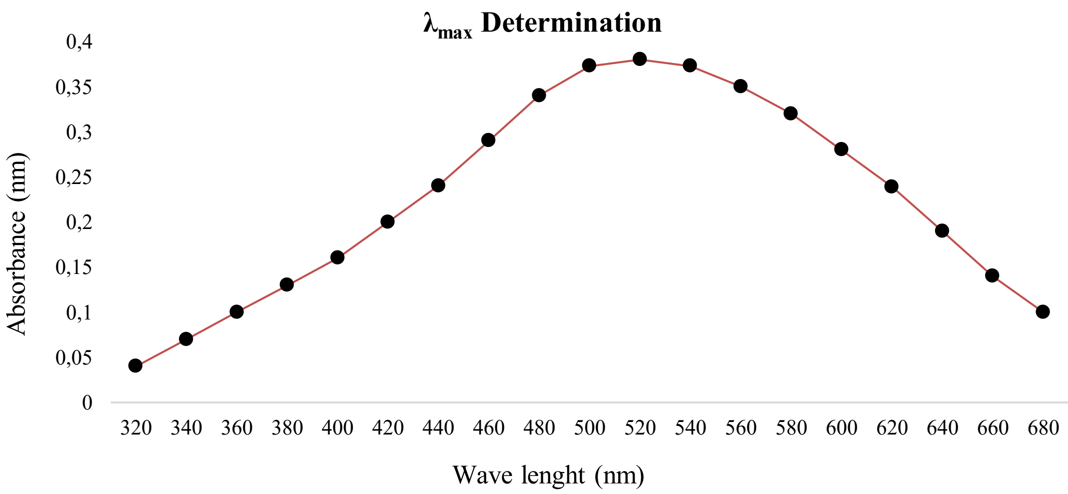

Fig. 1. λ_{max} determination of complex.

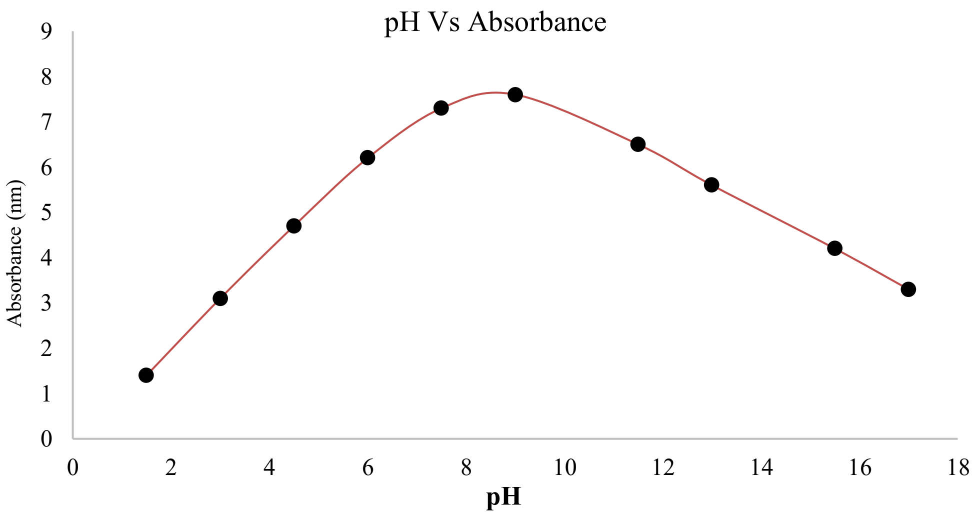

Fig. 2. pH Variation with absorbance.

had been found that 2 moles of cadmium react with 4 moles of dimethylglyoximine mentioning showing the compositions of the complex as 1:2 in Cu-dimethylglyoximine complex (table 1, figure 3).

Table 1.
Metal and ligand Concentration values**,** Mole fraction and Corrected absorbance.

S. No	Metal	Ligand	Mole Fraction	Corrected Absorbance
1	1	9	0.1	0.245
2	2	8	0.2	0.275
3	3	7	0.3	0.310
4	4	6	0.4	0.350
5	5	5	0.5	0.377
6	6	4	0.6	0.351
7	7	3	0.7	0.325
8	8	2	0.8	0.291
9	9	1	0.9	0.267
10	10	0	1.0	0.235

III. Mole ratio method

The stoichiometry of the Cd (DMG) coordination compound was determined as per the study by Nardo and Dawson [8] with little modifications as applicable for weak complexes like Cd (DMG). To 2 ml of prepared cadmium solution, 4 ml of buffer and 4 ml of dimethylglyoximine solution was added with continuous shaking. The optical density of the complex was observed at 534 nm. The experiment was repeated with different volumes of the Cd and DMG ranging from 2 ml to 10 ml and visa-versa with 2ml intervention as shown in table 3. A graph was plotted between volume of Cadmium and ligand and per the results obtained shows 1 mole of metal reacts with 2 moles of dimethylglyoximine (Figures 4 and 5).

IV. Beer- Lambert law and Determination of unknown Metal Concentration

The set of 20 ml volumetric flasks were numbered from 1 to 10, and in each volumetric flask 6 ml of DMG, 3 ml of buffer solution and different concentrations of cadmium was added successively as per the number till the volume was made up to 20ml with deionized water. All the volumetric flasks were kept at room temperature for 10 minutes and the optical density was observed at 535 nm. A straight-line graph was obtained between concentration (µg/ml) and absorbance from which the concentration of unknown samples was determined (table 2).

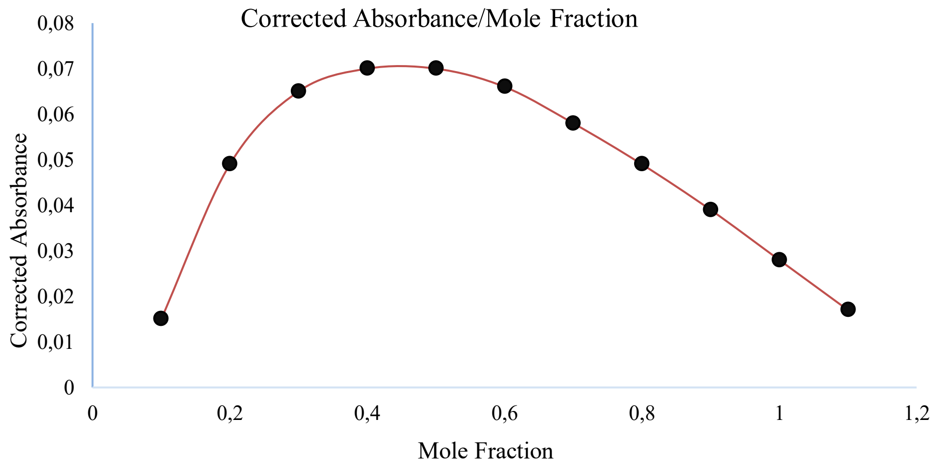

Fig. 3. Job's method for concentration determination.

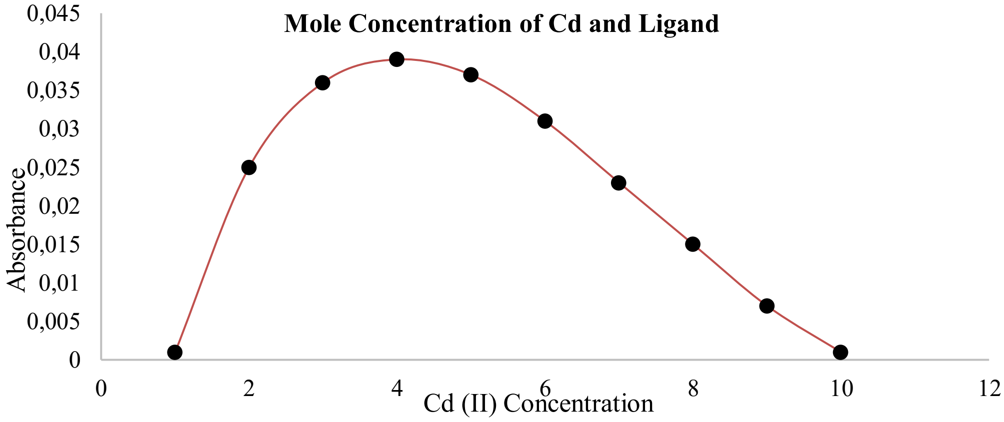

Fig. 4. Mole Ratio method for concentration determination.

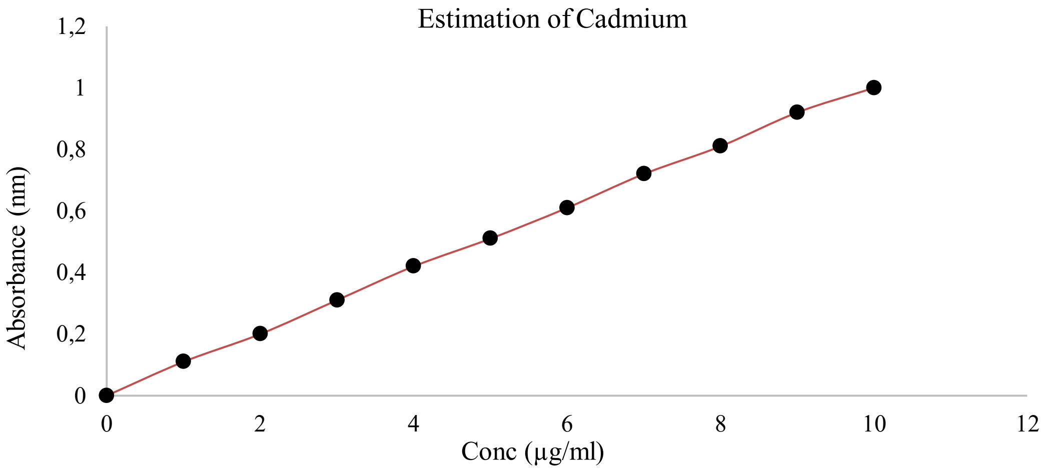

Fig. 5. Line Graph for determination of unknown Cd in a sample.

Table 2.
Absorbance of Cadmium-DMG complex / wavelength

Wave length (A^0)	Absorbance (nm)	Wave length (A^0)	Absorbance (nm)
320	0.041	480	0.334
340	0.072	500	0.372
360	0.142	520	0.381
380	0.133	540	0.375
400	0.164	560	0.344
420	0.191	580	0.313
440	0.235	600	0.272
460	0.281	620	0.225

V. Comparison of methods

The data obtained by the method of reference was compared with the already known methods (5, 7-Dibromo -8 - hydroxyquinoline method and 4- (o-di-azoaminophenyl arsonic acid) azobenzene method cadmium estimation were subjected to F-test (95% confidence level) to check the precision of the results. The data obtained by two known methods and by the study of reference is shown in table 5. As per the F-test analysis the average value of results in all the three cases seem to be far less than the F-distribution table values gives positive signal and suggests that the method of reference can be used precisely for the determination of Cd in samples of reference. The method was also checked by T-test evaluation related to the volumetric data about the precision of the valued results obtained with reference method makes it clear that the said estimation method for Cd can be used accurately (table 3, 4 and 5).

Table 3.
Determination of Cadmium in Unknown Water Samples

S. No	Cd (II) Conc.	Absorbance (nm)
1	0	0.015
2	1	0.112
3	2	0.215
4	3	0.321
5	4	0.414
6	5	0.513
7	6	0.580
8	7	0.612
9	8	0.680
10	9	0.720

Table 4.
Mole Ratio Method for Metal / DMG amount and their absorbances

Mixture	I	II	III	IV	V
Cd (II)	2	4	6	8	10
Dimethylglyoxime	10	8	6	4	2
Buffer Solution	8	8	8	8	8
Absorbance	0.362	0.459	0.521	0.411	0.321

Table 5.
Cadmium Determination in Various Water samples.

S. N	Water Samples	5, 7-Dibromo -8 - hydroxyquinoline method (µg/l)	4- (o-di-azoaminophenyl arsonic acid) azobenzene method (µg/l)	Concerned method (µg/l)
1	Test 1	0.17	0.19	0.21
2	Test 2	1.71	1.69	1.73
3	Test 3	1.31	1.29	1.37
4	Test 4	0.89	0.97	0.99
5	Test 5	0.75	0.86	0.81

Conclusion

In conclusion, this study shows how to use a cadmium-dimethylglyoxime (Cd-DMG) complex to determine the amounts of cadmium in soil and aqueous samples in a simple, cost-effective, and efficient spectroscopic manner. The method's viability is increased by its reliance on the creation of a simple chelate bond in a basic environment, which calls for little preparation and reasonably priced chemicals. Stability and ideal detection sensitivity are guaranteed by ideal circumstances, which include a basic pH and the determined 1:2 cadmium-to-DMG ratio. The method's strong molar extinction coefficient (1.5×10^5 mol^{-1} cm^{-1}) and consistent adherence to Beer-Lambert's law within the concentration range of 0.03 to 0.900 μg/ml confirmed its applicability for low-level cadmium detection. This approach is extremely accurate and can be used as a reliable substitute for cadmium detection in environmental investigations, according to a comparative analysis against well-established techniques employing F-test and T-test assessments.

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Новий та простий спектроскопічний метод визначення кадмію у водних зразках

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Розроблено простий та точний метод спектроскопічного визначення кадмію у водних та ґрунтових системах. Метод ґрунтується на утворенні хелатного зв'язку ліганду диметилгліоксиму з кадмієм у сприятливому основному середовищі. Встановлено утворення комплексу з співвідношенням 1:2 між кадмієм та диметилгліоксимом. Оптимальні умови для формування комплексу та подальшого визначення кадмію були визначені експериментально. Застосовність закону Бугера-Ламберта підтверджено в інтервалі концентрацій 0,03-0,900 мкг/мл. Молярний коефіцієнт екстинкції комплексу Cd(DMG) становить $1{,}5 \times 10^5$ моль$^{-1}$см$^{-1}$. Порівняння з наявними методами показало, що запропонований підхід є більш ефективним і економічно доцільним.

Ключові слова: кадмій, спектроскопія, водні розчини, метод Джобса, мольне співвідношення.