



UV-VIS spectrophotometric determination of magnesium after complexing with 8-hydroxy quinoline in sodium dodecyl sulphate micellar medium

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Abstract

In this study, an alternative UV-VIS spectrophotometric method for determination of magnesium was proposed. The method is based on complexation with 8-hydroxyquinoline (oxin) in sodium dodecyl sulphate (SDS) micelle medium, the absorbance of the product was recorded at 390 nm. pH of the solution, the concentration of ligand and surfactant were critical parameters, which affect the absorbance measurements, and optimised. The molar absorptivity coefficient is $5 \times 10^2 \text{ Lmol}^{-1}\text{cm}^{-1}$, the Sandell's sensitivity is $0,052 \mu\text{gcm}^{-2}$, detection limit is $0,3 \text{ mgL}^{-1}$, quantification limit is $1,1 \text{ mgL}^{-1}$ and linear working range is $2-8 \text{ mgL}^{-1}$ at pH 12 and 390 nm. The proposed method was applied for determination of magnesium to some drug and mineral water samples, nearly good accuracy and reproducible values were obtained percent as recovery and relative standard deviation.

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1. Introduction

Magnesium determination areas are great variety. These areas can be classified as water, industry, clinic, foods. Magnesium determinations were made by gravimetric, volumetric, atomic spectrophotometric, UV-VIS spectrophotometric, fluorimetric, electroanalytical and ion chromatographic method. Atomic absorption spectrophotometric (AAS) pathway is the best method for metal ions determination. However, because this instrument is expensive, not every routine analysis is available. The UV-VIS spectrophotometric methods are the most common. The conventional UV-VIS spectrophotometric determination of metal ions is based on the complexation of the metal ion with a ligand. UV-VIS spectrophotometric methods are divided into three in terms of the micro environment of the species where the signal is measured. These are: 1) Aqueous media 2) Organic solvent media (with liquid-liquid extraction) 3) Micellar media methods. Information about these studies were given in Table 1, Table 2 and Table 3.

While the direct aqueous medium method is more advantageous, the liquid-liquid extraction method has been developed in cases where the metal complex does

not dissolve in water or good peaks are obtained. One way for the same situations is to measure the signal in micelle. The micelle medium of a surfactant whose suitability is determined experimentally leads to improvement. The reason for this improvement can be explained as follows. The analyte species or its derivation is attached by micelle (held by the micelle), which changes the spectroscopic property of the different surrounding species by affecting the orbital energy levels, resulting in a better peak. The micelle media methods also have the following advantages: 1) Environmentalist; surfactants undergo biological degradation very quickly compared to organic solvents. 2) Cost of analysis is lower; in one assay, the surfactants are used in very small amounts relative to organic solvents. 3) The method is shorter and easier since no separation is required [1].

In this study, an alternative method for determination of magnesium in micelle medium is proposed. For mycelium, In this study, SDS as a surfactant and oxin as a ligand are used for the determination of Mg. In the literature, no studies have found these three together. With the realization of this study, we will present an alternative method of magnesium determination which can meet the demands of institutions that do not have AAS device because they do not have budgets.

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Table 1. UV-VIS Spectrophotometric determination of magnesium in aqueous medium

Ligant	pH	λ_{\max} (nm)	Dedection limit (mg L ⁻¹)	$\varepsilon \times 10^{-3}$ M ⁻¹ cm ⁻¹	Linear working range (mg L ⁻¹)	References
Eriochrom Black T	9.5-11.7	630	-	-	-	[2]
Chlorophospho-nazo III	7	699	-	-	-	[3]
Chromotrope 2R	10.8	570	-	-	-	[4]
Titan Yellow*	>12	545	-	2.8	-	[5]
Acid Chrome Black Special*	10	540	-	33	-	[5]
Calmagite	alkaline	520	-	-	0,0-36	[6]
1,8- dihydroxy- anthraquinone	-	510	-	-	0.25 - 2.00	[7]
Xylidyl Blue-1	alkaline	524	-	22	0-4	[8]
Terpiridine	3	668	-	-	-	[9]
Furfurin	9.5	540	0,075	9,2	0.8 – 4.3	[10]
CPC	10.5	570	0.24	-	0-20	[11]
Acid Chrome Blue K	-	540	-	9.3	0-2	[12]
Bromopyrogallo Red	9.4	RS- CWT**	-	-	0.2-3,2	[13]

* photometric

** Continuous wavelenght transforms

Table 2. UV-VIS spectrophotometric determination of magnesium with liquid-liquid extraction

Ligant	Solvent	pH	λ_{\max} (nm)	Dedectio n limit (mgL ⁻¹)	$\varepsilon \times 10^{-3}$ M ⁻¹ cm ⁻¹	Linear working range mgL ⁻¹)	References
8-hydroxyquinoline*	Cloroform	11.2-11.3	380	-	5.6	-	[5]
PTTHA	Cloroform	9.5	590	-	2.8	-	[14]

* Photometric

Table 3. UV-VIS spectrophotometric determination of magnesium in micellar medium

Ligant	Surfactan	pH	λ_{\max} (nm)	Dedection limit (mgL ⁻¹)	$\epsilon \times 10^{-3}$ M ⁻¹ cm	Linear working range (mgL ⁻¹)	References
Calmagite	Empigen BB	11.5	655	-	-	-	[15]
Xylidyl Blue -1	Brij 35	alkaline	505	-	-	0.0- 48	[16]
Bromopyrogallo l Red	Tween 80	10	570	0.03	-	0.5-3.5	[17]
Eriochrome Black T	CTAB	9.5	640	0.012	8.9	0.05-1.2	[18]

2. Materials and Methods

2.1. Tools and Equipment

UV-VIS Spectrophotometer: UV-1800 UV-VIS Spectrometer and UNICAM UV-VIS Spectrometer
UV 2 pH meter: Adwa AD8000.

2.2. Reagents

Solids of all reagents are of analytical purity. The solutions were prepared with double distilled water.

1) 375 ppm Oxin solution (pH 12): 0.0940 g of solid auxin was removed and transferred to a 250 mL beaker. It was diluted close to the adjustment line and the pH was 12. A 250 mL flask was then transferred to the flask and raised to the volume adjustment line with water. It was observed that the solution exceeding one week after preparation should not be used.

2) Sodiumdodecylsulfate (SDS): 5% (w / v). 5.0 g of SDS solid were dissolved in water and the volume was completed to 100 mL. To use this solution should not exceed a week.

3) pH 12 Glycine Buffer: Firstly, the following two solutions were prepared: 3-1) (0.2 M NH₂CH₂COOH + 0.2 M NaCl) solution: 1.5014 g of glycolic acid and 1.1688 g of NaCl were dissolved in a small amount of water in the same beaker and 100 mL of flask. the volume to 100 mL. 3-2) 0.2 M NaOH solution: 0.8 g of sodium hydroxide was dissolved in some pure water and then made up to 100 mL with volume of water. 54.6 mL of the second solution, 45.6 mL of the first solution were taken and mixed [4]. Alternatively, at various concentrations NaOH is added drop by drop to the first solution (prior to completion to 100 mL) until the pH is adjusted to pH 12.0 at a well-adjusted pH meter, then added to 100 mL with water.

4) 1000 mgL⁻¹ Mg (II) standard: 10,1411g (MgSO₄ 27H₂O solid was weighed and dissolved in water, was transferred to a 1 L balloon joje and the volume was completed with water to liter.

5) 100 mgL⁻¹ Mg (II) standard: It was prepared by diluting ten times before the previous one.

2.3. Recommended Method

A series of balloon joje of 10 mL each are collected. These are made in two groups, one for calibration curve and the other for samples. To each of the flasks in both groups were added 4 mL of oxin (375 mgL⁻¹), 1.5 mL of SDS (5% w / v), 1 mL of pH 12 glycine buffer, with the following four solution sequences, volumes and concentrations. For calibration, 200 - 400 - 600 - 800 μ L of 100 mgL⁻¹ Mg (II) is added sequentially and each amount is parallel. 400 μ L of the prepared sample is added to each of the balloons taken for the sample (at least three) and pre-added to the reagent. The solution levels are completed with double distilled water to the marking line, sealed and mixed.

Absorbance measurements of the solutions in the flasks at 390 nm are started without passing over the last addition. Absorbance measurements should be completed in less than 20 minutes, the first reading and the last reading should be between 10 and 15 minutes at most.

2.4. Preparation of samples

2.4.1. Preparation of magnesium sulfate ampoule sample 330 μL was taken from the original sample and diluted to 1 L.

2.4.2. Preparation of Magnesie Calsinee Saba sample

Some solid powder was transferred to a vessel and left in the etuv at 120 ° C. 0.600 g of this was weighed. 25 mL of 1 M HNO_3 was dissolved. 6 M NaOH was added to make the pH slightly basic. The solution was transferred to a 100 mL flask and the volume was completed to the marking line with double distilled water (Solution A). 2000 μL of this was transferred to a 50 mL flask and diluted to this volume (Solution B).

2.4.3. Preparation of Magnesie Calsinee Saba sample

One film tablet was taken and weighed. 31 mL of 1 M HNO_3 was added to the beaker and stirred for a long time. Filtered. It was made slightly basic so that the pH was between 8-9. The volume was made up to 250 mL with water (Solution A). 6,850 mL (6.0 mL + 850 μL) was taken and diluted to 100 mL (Solution B).

2.4.4. Preparation of magvital drug sample

One bowl was taken and the whole powder was transferred and weighed. Water was added to the beaker and mixed. Waited until the bubbles disappear in the water. To this solution, pH 3.4, 1 M HNO_3 was added dropwise to a pH of about 2 and heated. 6 M NaOH was added to make the pH slightly basic. The volume was completed to 250 mL (Solution A). 6.850 mL (6 mL + 850 μL) was taken and diluted to 100 mL (Solution B).

2.4.5. Preparation of water samples

The water sample mineral water was transferred to a beaker after the bottle was opened and waited until the exit of the gas bubbles was completed. The post is the same for both. Filtered. It was then taken up to a certain volume and acidified and heated, then made slightly basic to a pH of from about 8-9 to the same as that of the Mg standard. The solution is transferred to a flask which its volume was pre-determined, then is diluted.

3. Results and Discussion

3.1. Effect of SDS micelle on the spectrum of Mg-oxin complex

Critical micelle concentration (cmc) value of SDS is given as 8.1×10^{-3} M (0.23% (w / V) [18]. The volume to be added at least is 0.0162 mL, since the value added here is much larger than this value, the measuring medium will certainly be micelle.

When the spectra of the oxin buffer (glycine pH12) Mg st mixture were taken in one medium without SDS and the other with SDS micelle medium, respectively, Figure 1 and Figure 2 were obtained. As shown in the second figure, an analytical peak was obtained in SDS micelle medium.

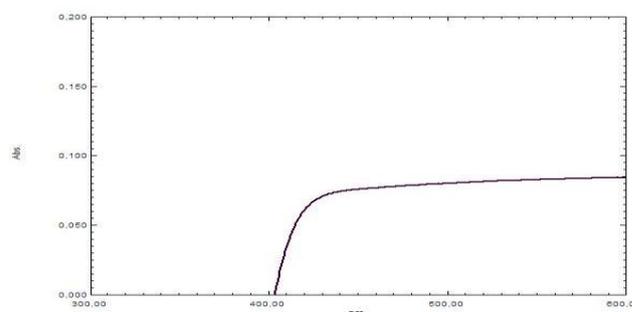


Figure 1. Spectrum of Mg-oxin complex in the absence of surfactant. 10 mL total volume Reactive additives in: 3 mL oxin (saturated in pure water), 1 mL buffer (pH 12) and 0.5 mL 100 ppm Mg (II)

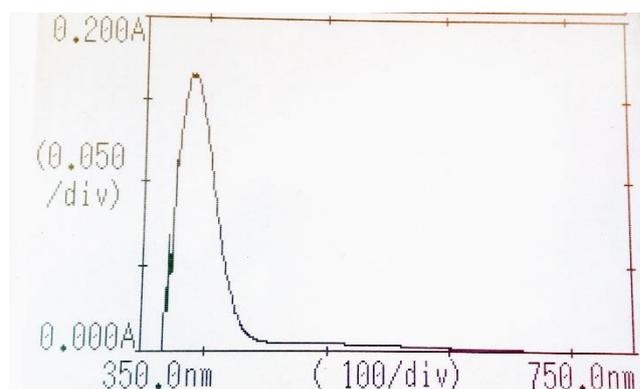


Figure 2. Spectrum of Mg-oxin complex in SDS micelle medium. Media composition: [Oxin] = 150 mgL^{-1} , [SDS] = 0.026 M (0.75% (w / v), pH = 11.5 -12, [Mg (II)] = 8 mgL^{-1}

3.2. pH effect

To determine the optimum pH, the following was followed: A series of 10 mL flasks were taken. To each flask 3 mL of saturated oxin, 2 mL of SDS (5% w / v), 1 mL of buffer (pH 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12), 200

μL 100 mgL^{-1} Mg (II) standard was added. The solution volumes were made up to 10 mL with double distilled water. Blank solution was taken as reference; this was not prepared for all solutions, but individually prepared at each pH.

The spectra of each solution were taken separately. Peaks were not obtained from solutions of pH 2-8, Turbidity was observed at pH 8. Analytical peaks were obtained from pH 9 and above. Figure 3 was obtained when the absorbances measured at the wavelength peaking at each pH were plotted against pH. As shown in the graph, the absorbance at pH 12 is maximum. 12 was chosen as the optimum pH.

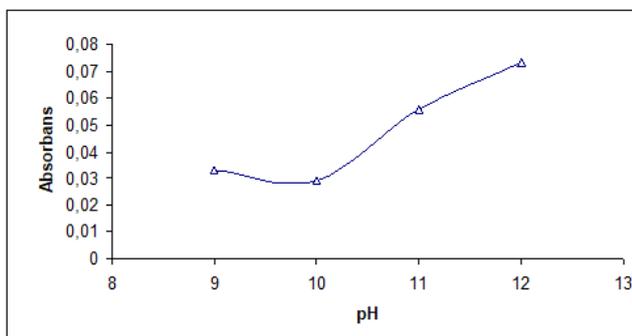


Figure 3. Effect of pH on absorbance. Media content: Oxin: Saturated diluted (in pure water) 3/10 after addition, SDS: 1% w / v (after addition), The pH values shown are those of the added buffers (initial), [Mg (II)] = $2 \mu\text{gL}^{-1}$ (after addition)

3.3. Effect of oxin concentration on absorbance

To determine the optimum oxin concentration, the reagents were added with the oxin volume variable and the others constant: The oxin reagent volume was predicted to be 4 mL before determining the optimum concentration. 1000 ppm oxin (0.01 M NaOH) solution in different volumes of 0.5, 1.0, 1.5, 2.0 mL was transferred to the balloons. 3.5-2 mL of double distilled water was added, each having a total volume of 4 mL. To each was added 2 mL of 5% SDS and 1 mL of buffer (pH12), 200 μL of 100 ppm Mg (II). The solution volumes were completed to 10 mL. Each volume was made in parallel. A different blank solution was used for each oxin concentration. The absorbances of the solutions were measured at peak wavelength. The mean of the parallel ones were taken. Figure 4 was obtained when the absorbance versus oxin volume was plotted.

According to the graph, the 1.5 mL volume is the optimal volume, which corresponds to a final concentration of 150 ppm after this dilution (completion to 10 mL). If the volume of oxin is taken

to 4 mL, the initial concentration corresponds to 375 ppm.

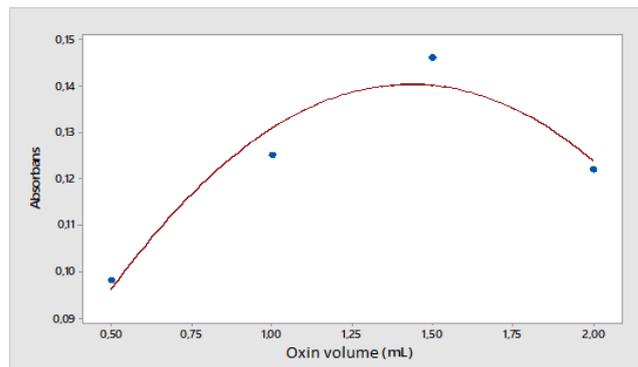


Figure 4. Effect of oxin concentration on absorbance. Solution additives in a volume of 10 mL: 1000 mgL^{-1} oxin (at 0.01 M NaOH) variable, 2 mL SDS (5%), 1 mL buffer (pH12 glycine), 200 μL 100 ppm Mg (II) standard

3.4. Effect of SDS concentration on absorbance

In order to determine the optimum SDS concentration, the reagents were added at this time with the variable SDS volume being variable and the others constant: 4 mL 375 ppm oxin (pH 12), 1-3 mL 5% SDS in different volumes, 1 mL pH 12 glycine buffer, 300 μL 100 ppm Mg standard was added. The solution volumes were completed to 10 mL. One blank was prepared for all as a reference. Figure 5 was obtained when the absorbance values measured at the peak giving wavelength were plotted against the added SDS volume.

There seems to be much deviation between the absorbances values in the graph. This is because the absorbance axis was scaled at very narrow intervals. In fact, there is not much deviation in absorbance. 1.5 mL was taken as the appropriate SDS concentration.

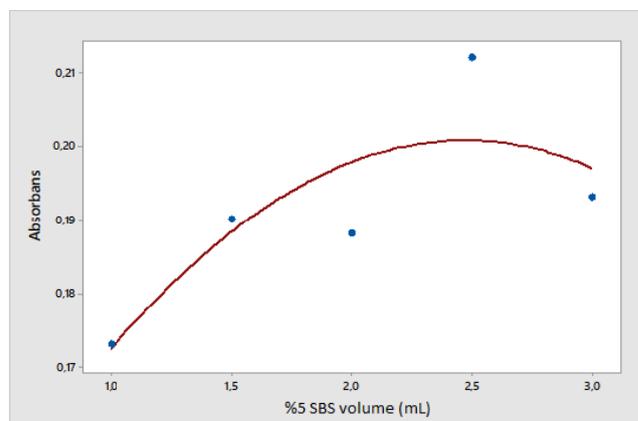


Figure 5. Effect of SDS concentration on absorbance. Additives in a volume of 10 mL : 4000 μL 375 mgL^{-1} oxin ;, SDS: different, 1000 μL Buffer (pH12 glycine), 100 mgL^{-1} , 300 μL , Mg (II) standard

3.5. Optimum wavelength

The peak wavelength varies according to the pH of the medium, and the lower the wavelength, the lower the wavelength. The pH in Figure 3 showing the effect of absorbance on the pH is not the pH of the medium, but the pH of the buffers used before addition. After addition, the pH drops slightly below that of the added buffer. As the ambient pH is lower than 12, the peak wavelength shifts from 392 to 365 nm. The amount of shear varies according to the concentration and pH of oxin used. Oxin has the lowest solubility in pure water and is about 45 mgL^{-1} . The saturated auxin solution in pure water is slightly acidic. The solubility of oxin increases at high pH. When saturated oxin (in pure water) is used, the pH decreases further. In experiments using this solution, peak wavelength was observed below 390 nm and absorbances were measured accordingly. When the oxin solution was adjusted to a pH of 12 and had a higher and optimal concentration, it was observed that the decrease in pH was less than 12 and the peak wavelength was 390 nm or close. This value corresponding to the optimal pH as the optimal wavelength was taken.

One way to reduce the pH drop after buffer addition is to increase the buffer capacity. In subsequent experiments, the buffer components were prepared in such a way that the concentrations of glycine and NaOH were doubled (the preparation given in Section 2.2 was this).

3.6. Stability and optimum reading time

When the balloons were last added, the levels were equalized and the absorbance signals were monitored after closing the mouths, the signals could not remain stable for a long time. As can be seen in Figure 6, in the spectrum band, there was a rise in ground (noise) over time, with higher concentrations. The spectra in the figure were taken approximately 30 min after the preparation of the solutions and starting at the lowest concentration (2 mgL^{-1}). Others were taken at about 5-10 minute intervals, with a higher concentration later. After 45 minutes, peak λ_{max} values also changed and the peak feature disappeared and turbidity occurred in the solution. The cloudiness was not observed in the empty trial solution; this shows us that clouding is not caused by coagulation of micelles.

Ground rise was not seen until the first 20 minutes. It is insignificant for the first 30 minutes. The start of the absorbance measurements of the solutions should not

exceed 20 minutes after the final solution component has been added and the levels have been equalized and the mouth closed, the measurement should be completed in 10, maximum 15 minutes.

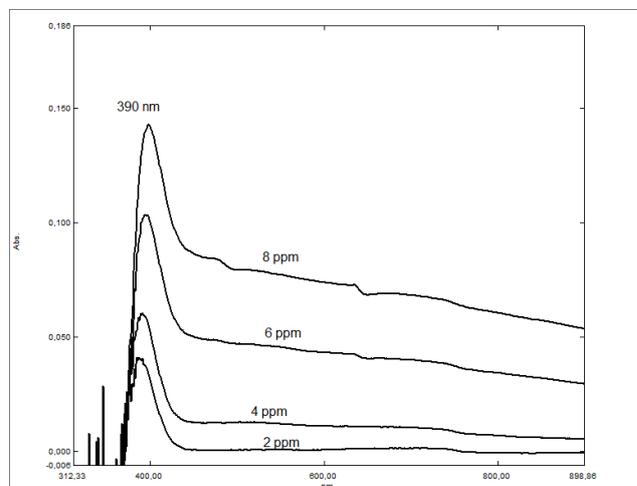


Figure 6. UV-VIS absorption spectrum according to the method and its variation over time. Media composition; [oxin] = 150 mgL^{-1} , [SDS] = 0.026 M (0.75% (w/v), pH = 12, [Mg(II)] values indicated on the curve in the graph) Spectra began to be taken about 30 minutes after the last reagent was added, this process continued to be more concentrated later.

Table 4. Optimum values of critical factors in the measurement medium

Parametre	pH	SDS, % (w/v)	Oksin, mgL^{-1}	λ_{max} , nm	Time
Value	12	0,75	150	390	20 dak*

* This is the time after which the last reagent is added and the moment of equalizing the levels is taken as the starting point.

3.7. Interferences

Species that have been investigated are generally found in the measurement environment. These are sodium, potassium, calcium, chloride, carbonate, bicarbonate, sulfate ions. No interference effects of sulfate, carbonate bicarbonate and nitrate were observed. Other results was given in Table 5. As the tolerance limit, the species/analyte (w/w) ratio or the corresponding concentration that changed the absorbance by $\pm 5\%$ was accepted.

Observations up to 0.01 M to determine whether NaCl is interfering. No interference was observed. Precipitation was observed when KCl was 50 mgL^{-1} in the measuring medium. Observation of this turbidity even in the blank solution indicates that the turbidity is due to the cloud point state or peptidization or coagulation; it can also be said that KCl interference is

independent of Mg (II). The KCl value of 50 mgL⁻¹ corresponds to a concentration of 23.54 mgL⁻¹ K⁺ if calculated. Considering that the concentration in the original sample is at least 10-fold diluted to the final medium, the K⁺ ion concentration in the original sample is at least 235 mgL⁻¹. This value is above the K⁺ levels found in water and drug samples. As a result, it can be said that K⁺ and Na⁺ will not be at the level that will cause effect in the samples where the method is applied.

Since heavy metal ions can complex with oxin such as Mg, interference is expected when these ions are present. The concentrations of metal ions other than Ca(II), Mg (II) in water and drug are at ppb levels. Moreover, the method is much lower than they are subjected to dilution when applied, so that their interference is out of the question.

When the Ca / Mg ratio was above 2/1, it was observed that the absorbance increase exceeded $\pm 5\%$. This ratio was taken as the tolerance limit for Ca (II). Masking with tartrate, citrate and EDTA was used to remove Ca (II) interference, but none of them yielded a positive and consistent result.

Table 5. Tolerance limits of some species (which changes the absorbance by $\pm 5\%$) species / analyte ratios), (ppm / ppm)

Species	Tolerance limit	[Sp] / [Mg] (ppm/ppm)
NaCl	>0,01 M (>580 ppm)	> 145
KCl	50 ppm*	(Mg independent)
Ca(II)	-	2/1

*Coagulation limit

3.8. Calibration curve parameters

Table 7. Application of the proposed method to some water samples

Sample	Known conc., mgL ⁻¹	Ca/Mg	Found conc., mgL ⁻¹	% R	% RSD
Mineral water samples 1	317 ^b	0.13 ^c	332.8 \pm 34.8 ^a	105	11
Mineral water samples 2	108 ^b	2.2 ^c	136.8 \pm 6.8 ^a	126	5

^a n=5, % 90 convedence level, t= 2,13,

^bThe value given on the label.

^c Rate calculated from the values given on the packaging (w/w)

The parameters of a calibration curve obtained by taking the optimal values of each critical factor was given in Table 6.

Table 6. Calibration curve parameters (two repeat measurements for each concentration)

Parameter	Value
Linear regression equation (c as mgL ⁻¹)	A = 0. 0194 c - 0.014
Correlation constant / r ²	0.9964
Measurement peak wavelength λ_{max} (nm)	390
Molar absorption constant ϵ (Lmol ⁻¹ cm ⁻¹)	5 \times 10 ²
Sandell's sensitivity* ($\mu\text{g cm}^{-2}$)	0.052
Dedection limit $3\sigma/m$ ($\mu\text{g mL}^{-1}$) (n=18)	0.3
Quantization limit $10\sigma /m$ ($\mu\text{g mL}^{-1}$) (n=18)	1.1
Linear working range (mgL ⁻¹)	2 - 8
*Concentration rise which causes to an increase of 0,001 in absorbance in a spectrophotometer with 1 cm beam length [19]	

3.9. Calibration curve parameters

The accuracy and precision of the proposed method were tested by applying mineral water sample of two different brands and some Mg supplement drugs.

The composition of the mineral water sample is given on the packaging bottle. As these values were determined by the standard method in routine analysis sites, they were taken as the correct value or comparison value. The obtained data are given in Table 7.

Table 8. Application of the proposed method to some drug samples and testing the accuracy by analyte spiked method

Sample	Added Con., mgL ⁻¹	Conc. difference found, mgL ⁻¹	%R	% RSD
Mg-sulfat ampoule*	0	0.97 ± 0.49	-	11
	2	1.34 ± 0.00	67	0
	4	4.22 ± 0.16	106	0,9
	6	5.98 ± 6.18	100	23
Magnezya Calsinee**	0	2.54 ± 0.54	-	13
	2	1.86 ± 0.43	93	14
	4	3.92 ± 0.71	98	11
	6	5.05 ± 0.59	84	7
Magosit*	0	2.03 ± 1.62	-	20
	2	1.86 ± 1.59	93	19
	4	4.64 ± 1.46	116	7
	6	5.88 ± 0.16	98	0,6
Magvital*	0	0.58 ± 1.63	-	63
	4	4.23 ± 1.14	106	6
	6	4.97 ± 2.11	83	10

* n=2, % 90 convedence level, t= 6,31, \bar{x} : Mean volues

** n=3, % 90 güven düzeyi, t= 2,92

The analyte spiked method or standard addition / method was used to determine the accuracy for the drugs. For this, the following path was followed. Equal amounts (200 µL) of the prepared sample solution were added to each of a series of 10 mL each flask. On top of that, 0 - 600 µL of 100 ppm Mg (II) standard was added for each volume to be measured again. Other procedures were performed according to Section 2.3. The obtained data are given in Table 8.

When the method was applied to water samples, accuracy was positive with 5% recovery in Kızılay Erzincan mineral water sample while accuracy was lower with 126% recovery in Beypazarı mineral water sample (Table 7). This is due to the Ca / Mg ratio of 0.13 / 1 Kızılay Erzincan mineral water Ca (II) is not interference. The other is the Beypazarı mineral water with a Ca / Mg ratio of 2.2 / 1, which is above the tolerance limit. The error rate in this example is therefore higher than the previous water sample.

Accuracy values as percent recovery are mostly positive when the method is applied to magnesium supplementation drugs that do not contain Ca(II) according to the propectus information. The repeatability values are unfortunately not the same (Table 8).

4. Conclusions

As a result, the applicability of this method is limited by Ca (II) interference. The method can be applied to

magnesium-supplementing drugs which do not contain Ca (II). When applied to water samples, it may give error results due to Ca (II) interference.

Conflicts of interest

The authors state that did not have conflict of interests

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