ISSN: 2312-8135 | Print ISSN: 1992-0652

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The Blue Molybdenum Reaction for the Determination of Phosphate in Natural Water and Detergent Samples

Vian S. Ismail

Faculty of Science, Soran University, vian.esmaeil@soran.edu.iq, Soran, Erbil, Kurdistan, Iraq.
***Corresponding author email**: vian.esmaeil@soran.edu.iq; mobile: 07504605501

تفاعل الموليبدينوم الأزرق لتقدير الفوسفات في عينات المياه الطبيعية والمنظفات

فيان صديق إسماعيل

كلية العلوم، جامعة سوران، vian.esmaeil@soran.edu.iq، سوران، اربيل، كردستان، العراق .

Accepted: **16 /6 /2023** **Published:** **30 /9 /2023**

ABSTRACT

Background:

The ideal reaction conditions for the quantitative detection of phosphate in various natural water and detergent samples were carefully researched in order to produce and preserve the colored complex product. The blue complex was identified using a simple and accurate UV-VIS spectrophotometer with a maximum wavelength of 870 nm.

Even though phosphate is an essential ingredient for the development of aquatic species, it is important to know exactly how much is present because an excess amount of phosphate can eutrophic water. Since detergents are one of the sources of phosphate in natural water, determining the concentration of phosphate in detergent samples is also essential.

Materials and Methods:

The method is based on the addition of sodium molybdate, which causes the interaction of orthophosphate with molybdate to produce an intense, stable, and water-soluble phosphomolybdate blue complex. Then, this combination is reduced using hydrazine hydrate in a sulfuric acid solution.

Results:

With a correlation coefficient of 0.9944, the system complies with Lambert-Beer's law at 870 nm in a concentration range of (0.05-9) ppm. Molar absorptivity was determined to be 12.16×10^3 L mol^{-1} cm^{-1} and Sandell's sensitivity was found to be 0.0156 g cm^{-2}. Water samples were collected from Warte, Choman, Jundeyan, and Bexal in Kurdistan's vicinity of Soran. Further, detergent samples that were obtained from the market and their phosphate level were examined. The Bexal waterfall had the lowest concentration of phosphate, 0.18 ppm, while the Warte and Jundeyan water samples had the highest concentrations, 3.31 ppm, and 3.04 ppm, respectively. Moreover, similar phosphate concentrations in the two detergent samples were discovered to be at 20.43 and 24.76 ppm.

Conclusion:

The molybdenum blue reaction has been successfully employed to provide a sensitive and quick measuring method to quantify the phosphate content in various water and detergent samples using UV-VIS spectrophotometry by applying the right reaction conditions and reagent quantities.

Keywords:

Phosphate, water, detergent, phosphomolybdate, and UV-VIS spectrophotometer

INTRODUCTION

All living things, including people, animals, and plants, require phosphorus as a vital nutrient. It ranks as the 20th most plentiful element in the solar system and the 11th most prevalent element in the crust of the planet.[1-5] Orthophosphate ($H_xPO_4^{(3-x)-}$), condensed phosphate as (pyro-, meta-, and other polyphosphates), and organically bound phosphates are the three kinds of phosphate. These phosphate classes can be found in three different forms: dissolved, particulate, and biological. Dissolved phosphate is the main phosphorus type found in freshwater and wastewater. A crucial indicator of water quality among them is orthophosphate; it has undergone significant laboratory characterization.[4-6] As a result of polyphosphates' fragility, orthophosphate in water is likewise the most stable form and is produced from it. Many factors affect the phosphorus levels in water bodies. To name but a few: location, density, level of agricultural and industrial activity nearby, rock type, the topography of rainfall pattern, climate, frequency of sampling, biological activities in soil, atmospheric deposition, chemical weathering of bedrock, flow rate, proximity to surface water, topsoil type, and depth.[1]

The presence of phosphates in surface and groundwater bodies at naturally occurring levels has no negative effects on the environment, human health, or animal health. However, very high levels of phosphates have varied negative effects on nature depending on the issue. The Environmental Protection Agency (EPA) suggested that the maximum phosphate concentrations in streams where phosphates immediately enter natural water and streams where phosphates do not directly enter natural water be 0.05 and 0.1 mg/L, respectively, to regulate the eutrophication process.[1, 7] The process of eutrophication has taken place as a result of the excessive phosphate levels. This process has increased algae growth, thus reducing dissolved oxygen levels, creating dead zones, and killing fish.[1-5, 7-9] Moreover, an abundance of algae can obstruct pipes, restrict recreational opportunities, produce offensive scents, and even pose some health risks. Both artificial and natural sources can contribute to the contamination of surface and groundwater. A few examples of natural sources are atmospheric deposition, mineral and rock weathering, runoff, sedimentation, and natural breakdown of rocks and minerals. Fertilizer, wastewater, and rotting system currents are examples of anthropogenic sources, together with animal waste, detergents, industrial waste, phosphate mining, drinking water treatment, forest fires, and synthetic material development surface.[1, 7]

Detergent is one of the main sources of phosphate in natural water. Surfactants, builders, and additional components including brighteners, fragrances, anti-redeposition agents, and occasionally enzymes make up laundry detergents[7, 10]. The two types of phosphates, sodium tripolyphosphates, and sodium/potassium pyrophosphate, have also been added to detergents as builders. The washing performance was improved because these phosphates can bind a variety of metal ions found in natural water, including calcium, magnesium, iron, and manganese. [4, 7, 10]

A regular phosphate study is required due to an increase in phosphate discharge into natural water and the harm it poses to the aquaculture industry, environmental sciences, agriculture, and medicine. Due to the major impact of phosphate level on water quality, numerous analytical techniques were necessary to determine the amount of phosphate in various samples.

Colorimetry[11, 12], potentiometry[13], fluorimetry[14], phosphorescence spectroscopy[15], electrochemical sensing[16, 17], ion chromatography[18], HPLC[19], bio-sensing[20], and spectrophotometric methods[1-4, 7, 10, 21-24] are a few examples of these techniques.

The majority of the analytical techniques for calculating the amount of phosphate listed above require the use of highly accurate equipment for auxiliary detection together with complicated calibration and/or preprocessing procedures. As a result, the spectrophotometric method, which relies on the blue molybdenum principle, is the technique that is most frequently utilized.[1-5, 7, 10] Because of its benefits such as simplicity, usability, and affordability, this popular approach is excellent for routine phosphate analysis. In this approach, the amount of phosphate is determined via the interaction of orthophosphate with sodium molybdate in an acidic aqueous solution, followed by its reduction by a variety of reductants. Various reducing agents have been used and documented in the literature, including thiourea[23], sodium sulfide[1, 10], hydrazine[3, 4], and ascorbic acid[2, 7]. But throughout this study, hydrazine hydrate was employed as a potent reducing agent to produce results that were accurate, sensitive, and quick for the phosphate routine assay. The objective of this study is to employ the blue molybdenum reaction to assess the amount of phosphate in samples of detergent and natural water after optimizing the experimental conditions.

MATERIALS AND METHODS

Instrumentation

The UV-VIS spectrophotometer, Model EMCLAB instruments-Germany V-1100 digital spectrophotometer with 1 cm glass square cells, was used to perform the spectral measurements. The applicable spectrophotometer operates in the (325-1000 nm) wavelength range.

Preparation of Standard Solutions

All of the compounds were analytical reagent grade, and all necessary solutions were made using distilled water. Every glass and piece of plastic utensil was regularly washed and rinsed with distilled water. These are the steps taken in the production of the reagents for PO_4^{3-} determination:

-Preparation of phosphate stock solution:

0.025g of dipotassium hydrogen phosphate (K_2HPO_4) has been transferred into a 100mL beaker and dissolved in distilled water. The solution has then been transferred into a 500 mL volumetric flask and diluted to the mark with distilled water. The concentration of the final solution was 500 ppm. The working phosphate solutions were prepared by further dilution.

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- Preparation of Working Solution of Sodium Molybdate (2.5% w/v):

2.5g sodium molybdate reagent was diluted and made up to the mark in a 100 mL volumetric flask.

- Preparation of sulfuric acid solution (10N)

28mL of concentrated sulfuric acid solution was diluted and made up to the mark in a 100mL volumetric flask.

- Preparation of hydrazine hydrate solution (0.5M)

2.44mL of concentrated hydrazine hydrate was diluted and made up to the mark in a 100mL volumetric flask.

Sample Preparation

Preparation of Water Samples

Spectrophotometric analysis for the blue molybdenum complex was looked at to undertake phosphate analysis in various sources of water in the Soran region. Many locations in the Soran region, including Warte, Choman, Jundian, and Bexal, were used to collect the water samples. To get rid of the insoluble particles, Whatmann-40 filter paper was used to filter all of the samples. Several types of phosphate, including orthophosphate, condensed phosphate (pyro-, meta-, and poly-), and phosphorus bonded to organic materials, were present in the filtrate. All of these phosphate forms may exist in soluble form or suspension. However, after acidifying (2N H_2SO_4) and then heating for about 30 minutes, all forms of condensed phosphate were hydrolyzed to orthophosphate.[3] As a result, only orthophosphate has been measured using the blue molybdenum method for the duration of this study.

Preparation of Detergent Samples

Commercial detergent samples weighing 5.0 grams, including samples of the SARA and BRIDGE brands, were placed in a crucible and burned fully over a benzene burner. The collected ash was placed in a 250mL beaker, and 50mL of distilled water was added to dissolve it. Thereafter, hydrogen sulfide, nitrite, and other gases were released from this solution by acidifying it with sulfuric acid and heating it on a hot plate for about 10 minutes.[3] The filtrate was then put into a 100mL volumetric flask and filled to the line with distilled water before being utilized for the phosphate assay after being passed through a Whatmann-40 filter paper.

Sample Preparation Protocol for Phosphate Analysis

The method outlined below has been suggested for determining how much orthophosphate is present in various samples:

a) To a 10mL volumetric flask, add 2mL of sample, 2mL of 2.5% sodium molybdate (w/v), and 1mL of 10N sulfuric acid. The reaction mixture should then be shaken.
b) Add 1mL of a 0.5M solution of hydrazine hydrate. Add distilled water to get the volume up to the required level.
c) Leave the solution alone for at least ten minutes to allow for the best color development.
d) At 870 nm, measure the absorbance.
e) Determine the phosphate concentration in ppm (mg/L) using the calibration curve.
f) If the absorbance starts to exceed the acceptable range, dilute the sample solution and proceed as described above.

RESULTS AND DISCUSSION

Principle of the method

The blue molybdenum reaction has been extensively researched for its use in phosphate level detection. Equations 1 and 2 describe the two steps of this reaction[5, 25]: first, the creation of a Keggin ion around the analyte anion; and second, the reduction of this hetero-polyacid to yield a product with a deep blue hue. It is interesting to note that orthophosphate (PO_4^{3-}) forms Keggin ions with the formula $[Xn^{+}Mo_{12}O_{40}]^{(8-n)-}$, where X is the heteroatom, just as the other tetrahedral anion of the kind XO_4.

$$PO_4^{3-} + 12MoO_4^{2-} + 27H^{+} \longrightarrow H_3PO_4(MoO_3)_{12} + 12H_2O \qquad (1)$$

$$H_3PMo(VI)_{12}O_{40} + \text{Reductant} \longrightarrow [H_4PMo(VI)_8Mo(V)_4O_{40}]^{3-} \qquad (2)$$

The blue molybdenum reaction is usually carried out in an aqueous solution in the presence of a Mo(VI) source and a reducing reagent in a strongly acid medium. The formation of the hetero polyacid and its reduction is mainly controlled by the concentration of the acid. The best pH for determining orthophosphates is pH 0-1 to get the best color intensity.[25] Various reaction conditions and reducing agents have been reported in the molybdenum reaction to give a reduced product. These products have been examined by using various analysis methods.

Absorption Spectra

The absorption spectrum of the blue phosphor molybdenum product in an acidic media has a maximum absorption at 870 nm (λ_{max}=870nm). As illustrated in Figure 1, this was produced by graphing the absorbance on the x-axis versus wavelength on the y-axis within the range of (800-900)nm.

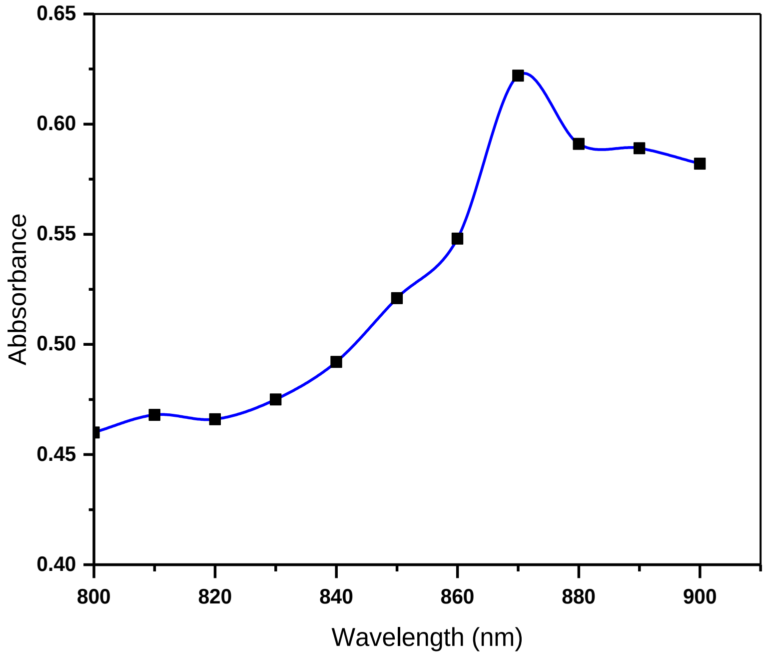

Figure 1: Absorption spectrum of 5ppm orthophosphate measured against blank solution

Optimization conditions for the analysis

Several experimental parameters, such as the quantity of sodium molybdate, sulfuric acid, hydrazine hydrate, and also color stability during various incubation times of the complex, have been optimized to detect the phosphate level quantitatively.

i- The effect of sulfuric acid concentration

The rate at which the reaction proceeds is greatly influenced by the acid content, especially how intense the phosphor molybdenum blue complex is. For high absorbance measurement, several amounts (0.1-2mL) of 10N sulfuric acid have been investigated. A final volume of 10mL has been created by combining these various volumes with 2mL of sodium molybdate (2.5% w/v), 1mL of phosphate solution (50ppm), and 1mL of hydrazine hydrate (0.5M). The solution mixture was then left for 10min for the best color detection. This process is dependent on the stability of the produced compound, the temperature, and the freshness of the chemicals used.

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When the absorbance of each generated solution is measured at 870 nm and compared, it has been found that the solution contains 1mL of sulfuric acid (10N), which is equal to 0.4N sulfuric acid; i.e., it has a higher sensitivity (Figure 2).

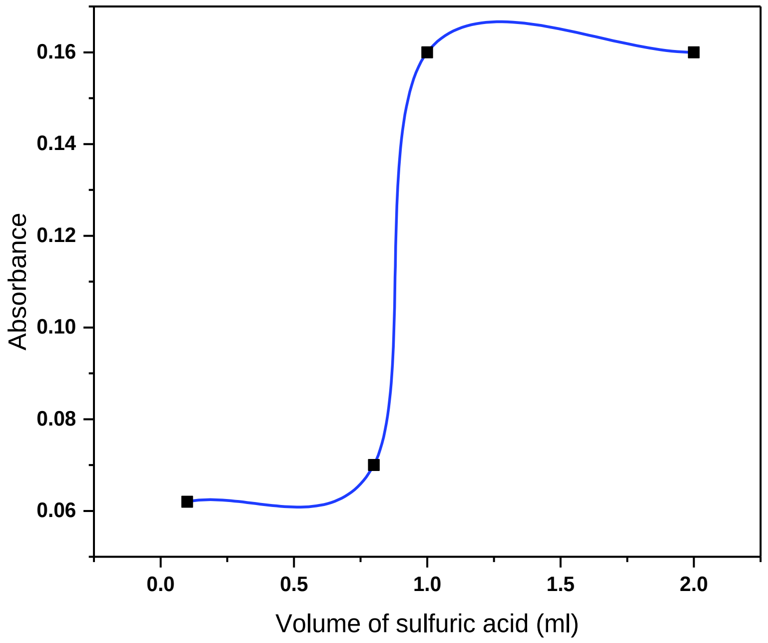

Figure 2: The effect of the amount of sulfuric acid (10N)

ii- The Effect of Sodium Molybdate Concentration

The quantity of sodium molybdate has an impact on the sensitivity of the analysis as well. This was investigated using various volumes (0.5–3)mL of sodium molybdate (2.5% w/v) with the addition of 1mL sulfuric acid (10N), 1mL phosphate solution (50ppm), and 1mL hydrazine hydrate (0.5M) in the final volume of 10mL. The results of the reaction mixture being incubated for 10 minutes revealed that 2mL of sodium molybdate (2.5% w/v) produced the greatest absorbance. This shows how much of this reagent is necessary to produce a stable phosphomolybdate complex (Figure 3).

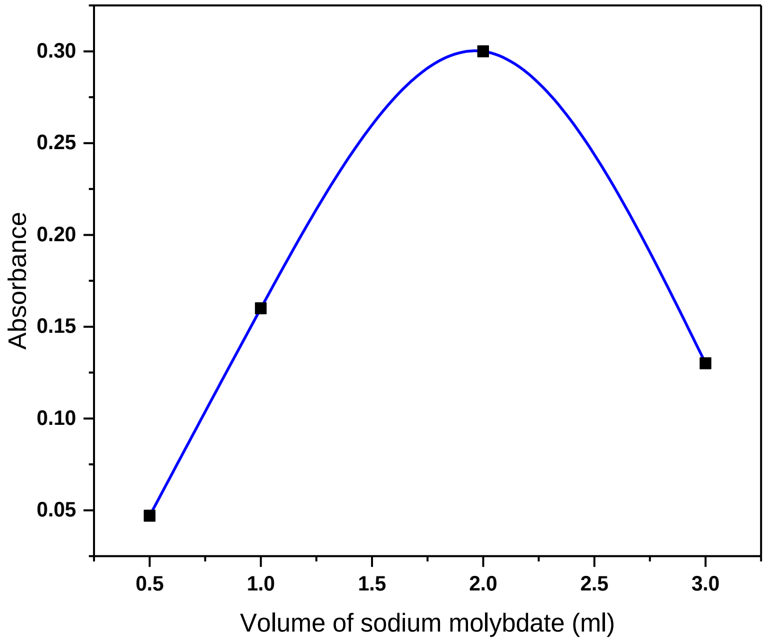

Figure 3: The effect of the amount of sodium molybdate (2.5% w/v)

iii- The effect of hydrazine hydrate concentration

Several quantities (0.05–2mL) of the hydrazine hydrate-reducing agent (0.5M) have been investigated. Phosphomolybdic acid has been reduced by this reagent to produce phosphor molybdenum blue. Sodium molybdate (2.5% w/v), sulfuric acid (10N), and phosphate solution combined with varying amounts of hydrazine hydrate (50ppm). The findings (Figure 4) demonstrated that as hydrazine hydrate concentration is increased, the absorbance rises to 1mL before remaining unchanged. As a result, for this investigation, 1mL of hydrazine hydrate (0.5M) has been determined to be the ideal volume.

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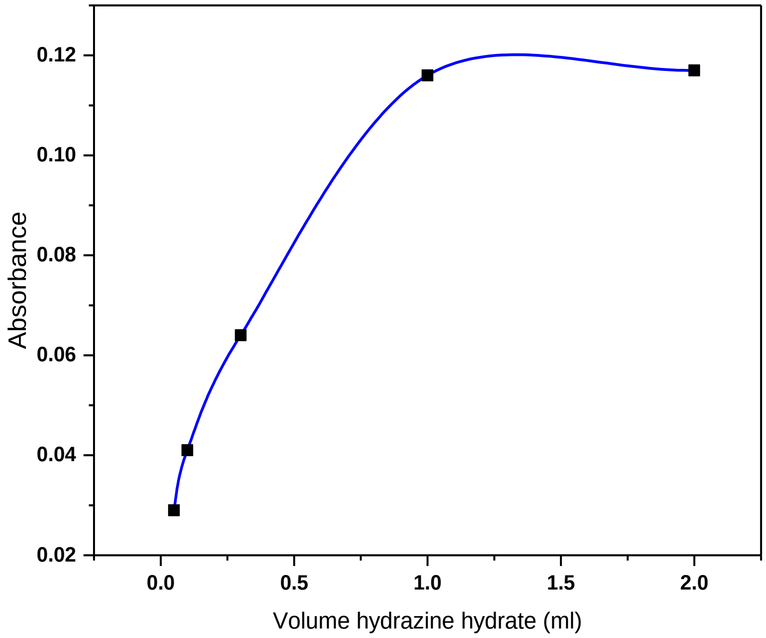

Figure 4: The effect of the amount of hydrazine hydrate (0.5M)

iv- The effect of time

Since the stability of the complex is time-dependent, the stability of the blue complex was tested within 60 minutes. The findings revealed that (Figure 5) up to 10 minutes of incubation, there is a sudden increase in absorbance, followed by a gradual increase. This demonstrates the blue phosphor molybdenum complex for up to 60 minutes and even longer. Hence, the ideal color development is established in less than 10 minutes.

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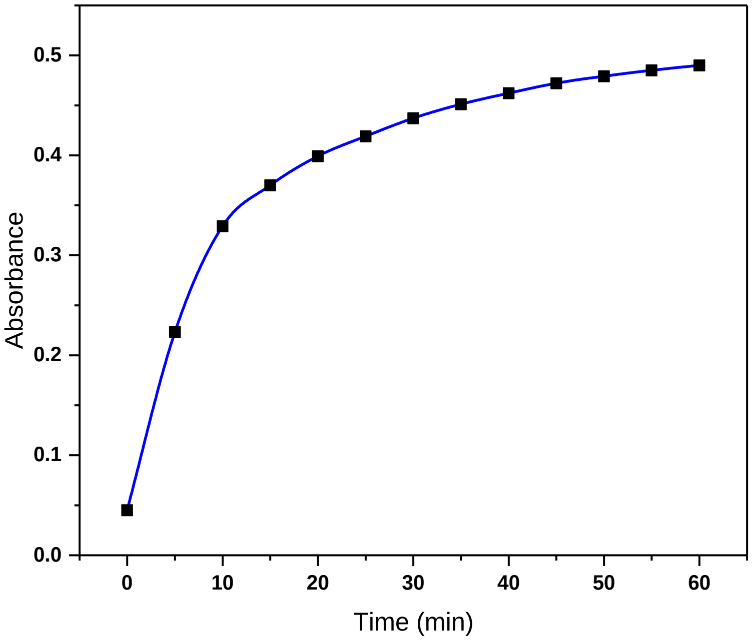

Figure 5: The effect of time on the stability of the complex

v- The effect of an order of addition

According to the data, adding the reactants in a certain order had no bearing on how the reaction progressed or how much absorbance was produced.[3, 4] The recommended method; however, has been used throughout this analysis to ensure consistency in the order of reagent addition, as follows: various concentrations of phosphate solution have been mixed with 2mL of sodium molybdate (2.5% w/v), 1mL of sulfuric acid (10N), and 1mL of hydrazine hydrate (0.5M). The combination has been allowed for 10 minutes to allow for the most color development before the absorbance at 870 nm is measured.

Calibration curve

Confirming the linear relationship between the absorbance and the amount of phosphate is a crucial stage in the spectrophotometric analysis procedure. A calibration curve can be seen in Figure 6 by plotting the absorbance against the phosphate solution concentration (ppm) at the wavelength of 870 nm. At the concentration range of (0.05-9)ppm, Lambert-Beer's law is observed. To get the concentration into the measurable range, the sample needs to be diluted above 9 ppm. Statistical properties for the suggested technique are described in Table 1.

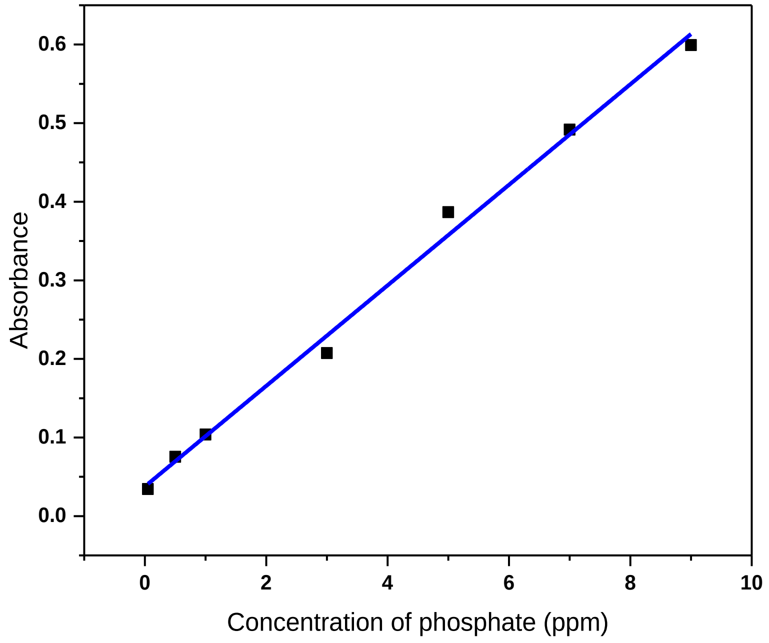

Figure 6: Calibration curve for the determination of phosphate under optimized experimental conditions at λ_{max} equal to 870nm

Table1: Analytical characteristics of the linear range for the proposed method under optimized experimental conditions at λ_{max} equal to 870nm

Parameters	***Value***
linearity range	(0.05-9) ppm
Linear regression equation	Y = 0.064x + 0.0377
Slope	0.064
Intercept	0.0377
Linearity (%R^2)	0.9944
Standard error of the slope	0.0021
Standard error of intercept	0.0104
Molar absorptivity (ε, $L.mol^{-}$, cm^{-})	$12.16x10^3$
Sandell's sensitivity ($\mu g.cm^{-2}$)	0.0156

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Quantification of the amount of phosphate in different water and detergent samples

After successfully using the suggested approach on standard phosphate, it has also been determined how much phosphate is present in various water and detergent samples.

By utilizing the adjusted reaction conditions and quantities of various reagents, the blue molybdenum method was successfully applied for the spectrophotometric measurement of phosphates in various samples. This procedure is dependent on the stability of the blue molybdenum complex, the temperature, and the freshness of the chemicals utilized. According to research, the blue phosphor molybdenum complex can form in as little as 10 minutes, as seen in Figure 5. Tables show the findings of these analyses (2 and 3). The findings have revealed that the Bexal waterfall has the lowest phosphate level at 0.18 ppm and that Warte and Jundeyan water samples have the highest levels at 3.31 ppm and 3.04 ppm, respectively. The low concentration of phosphate in the Bexal water sample, which is comparable with the reported range of the literature, (0.05-0.1)ppm for the USEPA's reported level of phosphate in natural water,[2, 7] highlights the purity of the water. However, given that phosphate is typically found in high concentrations in raw or treated sewage, agricultural drainage systems, and industrial effluents. It is further possible that the water in Warte and Jundeyan is contaminated by the phosphate discharge.

Moreover, it has been discovered that the amounts of phosphate in both detergent samples are between 20.43 and 24.76 ppm. Because they work so well as builders, phosphates are frequently employed in dry detergents as sodium tripolyphosphates. These phosphates can bind calcium, magnesium, iron, and manganese ions, which enhances the effectiveness of washing in general.[7, 10]

Table 2: The level of phosphate in different water samples

*Water samples**	*Phosphate (mg/L)*	*Phosphate (%)*
W-1	3.31	$3.3\text{x}10^{-4}$
W-2	0.73	$7.3\text{x}10^{-5}$
W-3	3.04	$3.04\text{x}10^{-4}$
W-4	0.18	$1.8\text{x}10^{-5}$

***: W-1=Warte, W-2=Choman, W-3=Jundeyan, W-4=Bexal**

Table 3: The level of phosphate in detergent powder samples

*Detergent samples **	*Phosphate (mg/L)*	*Phosphate (%)*
D-1	20.43	$2.04\text{x}10^{-3}$
D-2	24.76	$2.48\text{x}10^{-3}$

***: D-1=Sara, D-2=Bridge**

CONCLUSION

Using a UV-VIS spectrophotometer and Lambert-Beer's law, a quantitative measurement has been made to determine the amount of phosphate in a solution. To accomplish this, first, establish the calibration line by measuring the volume of a standard solution with a known concentration of phosphate using an 870nm phosphomolybdate complex absorbance measurement in an aqueous medium of sulfuric acid. With correlation values of 0.9944, it has been discovered that the linearity of the approach is predicted to be in the range of 0.05-9.0 ppm. Interestingly, the suggested procedure does not call for either expensive equipment or infrequent reagents. The process is easy, precise, accurate, and sensitive. Moreover, there is no requirement for pretreatments or precise pH control, and the chemicals used in the application are inexpensive and easily accessible in standard laboratories.

With the use of appropriate reaction conditions and reagent concentrations, the blue molybdenum method has been effectively used to measure the quantity of phosphate in various water and detergent samples using spectrophotometry. The Bexal waterfall has the lowest concentration of phosphate, 0.18 ppm, according to the results, while Warte and Jundeyan water samples have the highest concentrations, 3.31 ppm, and 3.04 ppm, respectively. Moreover, both detergent samples have similar levels of phosphate (20.43 and 24.76 ppm) in them.

Phosphate is a crucial nutrient for environmental conservation, and the results showed that this approach is effective, suited for routine phosphate measurement, and capable of quantifying phosphate concentrations exactly in both natural and manmade materials.

Acknowledgments:

The Soran University Department of Chemistry provided the available research equipment for this study, for which the author is grateful.

Conflict of interests.

There are non-conflicts of interest.

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ISSN: 2312-8135 | Print ISSN: 1992-0652

info@journalofbabylon.com | jub@itnet.uobabylon.edu.iq | www.journalofbabylon.com

الخلاصة

مقدمة:

تم البحث بعناية في ظروف التفاعل المثالية للكشف الكمي عن الفوسفات في عينات المياه الطبيعية والمنظفات المختلفة من أجل إنتاج المنتج المعقد الملون والحفاظ عليه. تم تحديد المعقد الأزرق باستخدام مقياس طيف ضوئي بسيط ودقيق للأشعة المرئية وفوق البنفسجية بطول موجي أقصى يبلغ 870 نانومتر.

على الرغم من أن الفوسفات هو عنصر أساسي لتنمية الأنواع المائية ، فمن المهم أن تعرف بالضبط كم هو موجود لأن الكمية الزائدة من الفوسفات يمكن أن تؤدي إلى تخثث المياه. نظرًا لأن المنظفات هي أحد مصادر الفوسفات في المياه الطبيعية ، فإن تحديد تركيز الفوسفات في عينات المنظفات أمر ضروري أيضًا.

طرق العمل:

تعتمد الطريقة على إضافة موليبدات الصوديوم ، الذي يتسبب في تفاعل أورثو فوسفات مع الموليبدات لإنتاج مركب أزرق فوسفوموليبدات مكثف ومستقر وقابل للذوبان في الماء. ثم يتم تقليل هذا المزيج باستخدام هيدرات الهيدرازين في محلول حامض الكبريتيك.

الاستنتاجات:

مع معامل ارتباط 0.9944 ، يتوافق النظام مع قانون لامبرت بير عند 870 نانومتر في نطاق تركيز (0.05–9) جزء في المليون(ppm). تم تحديد الامتصاصية المولية لتكون $12.16x10^{3}$ لتر مول $^{-1}$ سم $^{-1}$ وحساسية Sandell كانت 0.0156 جم سم$^{-2}$. تم جمع عينات المياه من ورتي و جومان و جنديان و بيخال في محيط سوران في كردستان. كما تم فحص عينات المنظفات التي تم الحصول عليها من السوق ومستوى الفوسفات بها. كان لشلال بيخال أقل تركيز للفوسفات، 0.18 جزء في المليون ، بينما كان أعلى تركيز لعينتي ورتي و جنديان، 3.31 جزء في المليون و 3.04 جزء في المليون، على التوالي. علاوة على ذلك، تم اكتشاف تركيزات مماثلة من الفوسفات في عينتي المنظف عند 20.43 و 24.76 جزء في المليون.

تم استخدام تفاعل الموليبدنوم الأزرق بنجاح لتوفير طريقة قياس حساسة وسريعة لتحديد محتوى الفوسفات في عينات المياه والمنظفات المختلفة باستخدام القياس الطيفي للأشعة المرئية وفوق البنفسجية من خلال تطبيق شروط التفاعل الصحيحة وكميات الكاشف.

الكلمات المفتاحية:

الفوسفات، الماء، المنظفات، الفوسفوموليبدات، مقياس الطيف الضوئي للأشعة فوق البنفسجية