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SPECTROPHOTOMETRIC DETERMINATION OF PHOSPHATES IN DETERGENT SAMPLES AVAILABLE IN TANSEN, PALPA

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Abstract

Phosphate is primarily used as a builder in detergents, which helps soften hard water, thereby increasing the cleansing power of detergents. Extensive discharge of phosphate from detergents leads to eutrophication of surface water bodies. This work aims to assess the amount of phosphate present in detergent samples available in Tansen, Palpa, and compare its concentration between local and foreign detergents. Phosphate in detergent samples can be determined by the molybdenum blue phosphorus method using a spectrophotometer. The experimental process relies on forming a phosphomolybdate blue complex with ammonium molybdate and the subsequent reduction of the complex by hydrazine hydrate in an aqueous sulphuric acid medium. The system obeys Beer-Lambert's law at 830 nm in the concentration range of 2-16 ppm. In this study, the phosphate concentration in local detergents (Dhoni, Light, V-Series, Nirma, Upahar, Patanjali, Rin Shakti and Dr Jhatka) varied from 15.54-33.03 ppm, and that of foreign detergents (Wheel, Super Check, Surf Excel, Tide and Ariel) ranged from 23.81- 32.65 ppm. The amount of phosphate was compared between local and foreign detergent samples using the student's t-test, which revealed no significant difference between them. The results obtained from the study disclosed that the detergents under investigation are within the permissible limits of phosphate. However, non- phosphate-based builders can be used to discourage the use of phosphate in detergents.

***Keywords*:** Chelating agent, Eutrophication, Molybdenum blue phosphorus method, Spectroscopy.

Introduction

Phosphorous is one of the abundant elements found on earth's crust in the form of phosphate, which plays a crucial role in the biochemical process. Phosphates are used in detergents as builders or chelating agents in the form of sodium tripolyphosphate or sodium pyrophosphate, which remove Calcium and Magnesium ions present in hard water, thereby making it soft and increasing the efficiency of surfactants (Hossain et al., 2017). A large amount of phosphate is passed into water bodies by household waste discharge, detergents, sugar industries, and fertilizers which flourishaquaticvegetation and phytoplankton. This accelerates the natural aging of lakes which is called eutrophication. Consequently, dissolved oxygen in water decreases and kills aquatic life, disordering the aquatic ecosystem (Kundu et al., 2015; Oliveira & Machado, 2013). The significant contribution to soluble phosphorous in municipal

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wastewater is household detergents (Audu, A.A.& Yusuf, 2015). Thus, it is mandatory to assess the amount of phosphates in detergents. Many methods for the determination of phosphate have been recorded, like titrimetry, HPLC, complexometry, atomic absorption spectroscopy, flow injection analysis, and spectrophotometry (Oladeji et al., 2016; Shyla et al., 2011). However, the spectrophotometric molybdenum blue method is the most common, efficient, and economical. This method involves forming molybdophosphoric acid from orthophosphate and the excess of the molybdate in the acidic medium, which is then reduced to give molybdenum blue. The phosphate concentration in the detergent sample is directly proportional to the intensity of blue colour developed, which can be measured spectrophotometrically at the wavelength of maximum absorbance; λMax(Adelowo & Agele, 2016). Different types of reducing agents have been reported in literature like sodium sulphide (Agbazue et al., 2015), thiourea (Shyla et al., 2011), sodium thiosulphate (Nalumansi et al., 2020), hydrazine hydrate (Pradhan &Pokhrel, 2013), and so on. This research aims to assess the amount of phosphate in detergent samples of local and foreign brands and observe if there is a significant difference between them.

Materials and methods

Instruments

Scanning Visible Spectrophotometer Model EI-2306, an Electronic Balance (Four Digits), MuffleFurnace Model- Vivo India, and Water Bath (Grant OLS-200) were used.

Preparation of standard solution

All the solutions were prepared using distilled water.

Phosphate stock solution

0.717gm of potassium dihydrogen phosphate (KH_2PO_4) was dissolved in a 500 mL volumetric flask with distilled water, and the volume was made up to the mark by adding more distilled water. Thus, the prepared solution was exactly 1000 ppm and was diluted to get a 50 ppm solution which was used as a working phosphate solution.

Preparation of working Ammonium molybdate solution (2.5%)

50 mL of 5% ammonium molybdate solution was diluted and made up to the mark in 100 mL of the volumetric flask by adding more distilled water.

Preparation of sulphuric acid solution (10N)

27.5 ml of concentrated sulphuric acid was diluted to 100 ml in a volumetric flask.

Preparation of hydrazine hydrate (0.5M)

2.5 ml of concentrated hydrazine hydrate solution was diluted to 100 ml in a volumetric flask.

Preparation of detergent sample solution

5.0 gm of the detergent sample was accurately weighed in a crucible and was placed inside the muffle furnace for about half an hour at 330°C. Detergent ash so obtained was taken in a 250 mL beaker and dissolved in 50 mL distilled water. This solution was then acidified by adding 2-

5 ml of dilute sulphuric acid and heated on a hot plate for about 10 minutes to expel hydrogen sulphide, nitrite, etc. It was then filtered through Whatman-42 filter paper, and the filtrate was transferred into a 100 mL volumetric flask and diluted up to the mark. This solution was used for phosphate determination.

Preparation of standard solution for the Calibration curve

The linear relationship between absorbance and the concentration of phosphate solution in the calibration curve helps determine the analyte's unknown concentration. Phosphate stock solution of 50 ppm was diluted to prepare standard solutions of 2,4,6,8,10,12,14 and 16 ppm with the subsequent addition of other reagents like Ammonium molybdate, Sulphuric acid, and Hydrazine hydrate. These solutions were left in the water bath at 37℃ for about 35 minutes. After the complete development of the blue colour, the absorbance of each phosphate solution was measured, and these absorbance values were used for drawing a standard calibration curve.

Procedure for phosphate determination

The concentration of phosphate in the detergent sample solution was determined with the help of a calibration curve. 4 ml sample solution of detergent was taken in a 100 ml volumetric flask, and 8 ml of 2.5% ammonium molybdate solution was added to it followed by 2 mL of 10 N sulphuric. After complete shaking, 4 mL 0.5M hydrazine hydrate solution was added and made up to the mark. Then these flasks were kept in a water bath at 37℃ for maximum colour development for atleast 35 minutes. After the complete development of blue colour, this solution was thoroughly shaken and used for absorbance measurement using a spectrophotometer (Pradhan &Pokharel,2013).

Results and discussion

Determination of maximum absorbance

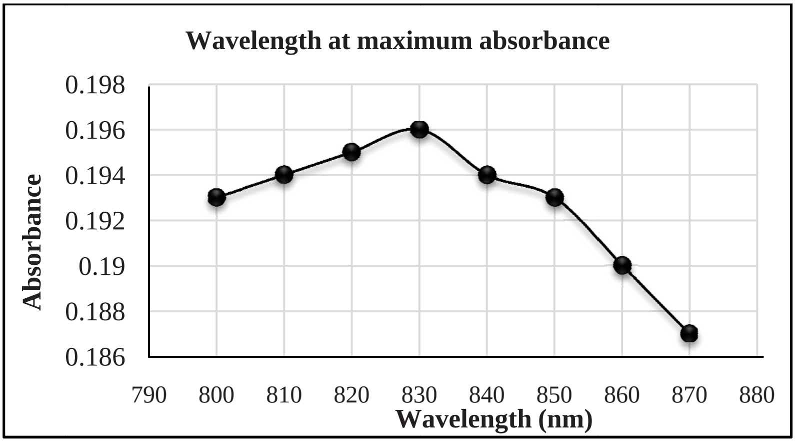
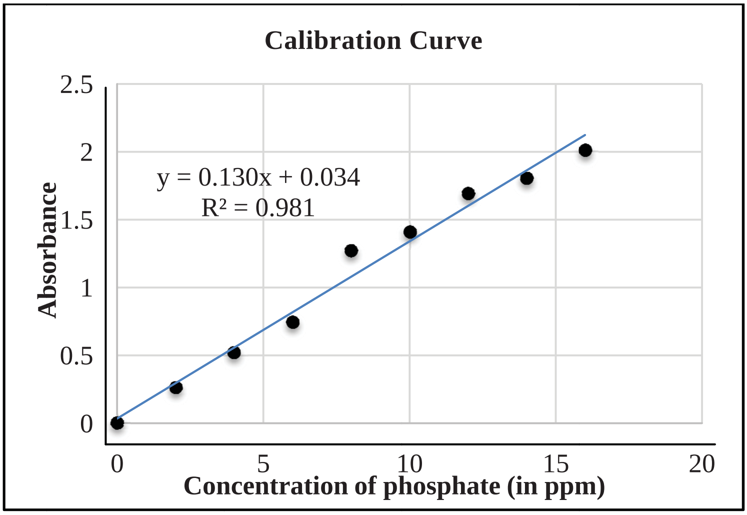


Figure 2*.A plot of absorbance against wavelength*

The absorbance of the standard phosphate solution against the reagent blank solution showed themaximum absorbance at 830 nm as shown in ***Figure 2***.

Calibration curve



The absorbance of standard phosphate solutions was measured at a wavelength of 830 nm and plotted against concentration as shown in **Error! Reference source not found.**.

Phosphate levels in detergent samples

Eight detergent samples (Dhoni, Light, V-Series, Nirma, Upahar, Patanjali, Rin Shakti and Dr Jhatka) of local brands manufactured in Nepal and 5 Indian brands (Wheel, Super Check, Surf Excel, Tide and Ariel); altogether, 13 samples were analyzed.Absorbance shown by the local and foreign detergent samples were measured at 830 nm, and the amount of phosphate in ppm and percentage are shown in ***Table 3***.

Table 3. *The concentration of phosphate in different detergent samples*

Sample no.	**Sample absorbance**	**Concentration (ppm)**	**Percentage composition of phosphate (%)**
A	0.147	24.75	0.04951
B	0.098	15.54	0.031083
C	0.139	23.25	0.046501
***Figure 3**. Standard calibration curve*			
D	0.108	17.42	0.034843
E	0.178	30.58	0.061168
F	0.191	33.03	0.066056
G	0.171	29.27	0.058535
H	0.138	23.06	0.046125
I	0.142	23.81	0.047629
J	0.186	32.09	0.064176
K	0.189	32.65	0.065304
L	0.150	25.32	0.050638
M	0.166	28.33	0.056655

In this study, some local brands of detergents contained a higher amount of phosphate than the foreign brands. Local detergent sample-F had the highest amount of phosphate, i.e. 33.03 ppm. Similarly, local detergent sampleB had the lowest amount of phosphate, i.e. 15.54 ppm. Other samples showed the amount of phosphate in the range of 15-33 ppm. This result showed that there is a higher variation in the amount of phosphate between local detergents.

Similarly, sampleK had a higher amount of phosphate among foreign detergents, i.e. 32.65 ppm, and sample-I had the lowest amount of phosphate, i.e. 25.32 ppm. This showed that there is no higher variation in phosphate between foreign detergents.

When the amount of phosphate was compared between these two brands of detergents, the assumption of normality was checked using the Shapiro-Wilk test and found the observed data sets distributed normally (p-value = 0.3564) > 0.05. For the test of equality of means, student's -t-test was conducted and obtained t = -1.364, df =11, p-value = 0.200 > 0.05 (5% level of significance), which revealed that there is no significant difference in the concentration of phosphate in local and foreign detergent samples.

The international permissible limit of phosphate in detergents is 0.5%. All local and foreign detergent samples which were used for this study contained acceptable limits of phosphate.

Conclusion

Results revealed that despite higher variation of concentration of phosphates in local brands than foreign brands, the statistical test showed no significant difference between them. The concentration of phosphate levels in the detergent samples analyzed were found to be within the international standard limit of 0.5%, which shows that these detergents are safer to use considering the phosphate concentration.

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