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Phosphate determination in sugarcane juices, water and detergent by spectrophotometric from Bhavnagar region

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ABSTRACT

spectrophotometric method is developed for the determination of phosphate in sugar cane juice, water and detergent samples. The method is based on the formation of phosphomolybdate with added molybdate followed by its reduction with sodium sulphide in aqueous sulfuric acid medium. The system obeys Lambert-Beer's law at 715 nm in the concentration range 0.3-12.24 ppm. Molar absorptivity, correlation coefficient and Sandell's sensitivity values were found to be $6.1 \times 10^3 \text{ mol}^{-1} \text{ cm}^{-1}$, -1 0.999 and $0.0156 \text{ mg cm}^{-2}$ respectively. The results obtained were reproducible with acceptable standard deviation 3.7% and relative error 3.4%.

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KEYWORDS

Phosphate;
Water;
Detergents;
Spectrophotometry.

INTRODUCTION

Phosphorus is the eleventh most abundant element on the surface of the earth and is most commonly found as phosphate. It plays an important role in biochemical processes and is a key factor in the eutrophication of surface water^[1] Increased phosphate concentrations are linked with increasing rates of plant growth^[2]. The analytical chemistry of phosphorus is very important in many fields, for example, medical and clinical science, agriculture, metallurgy and environmental science^[3]. Moreover, in recent years large quantities of phosphate have been used in beverages^[4], detergents^[5], fertilizers^[6] and also in sugar industries^[7-9].

The literature^[7-9] gives the importance of phosphate during the sugarcane juice clarification leading to cane sugar manufacture. In this context, the chemi-

cal action of phosphate during clarification of sugarcane juice assumes significant role particularly when the juice is deficient in natural phosphate content in the range of 300-350 ppm as P_2O_5 which is essential for good clarification. In case of less phosphate content in sugarcane juice, soluble phosphate addition is the only alternative to achieve the target value by adding orthophosphoric acid, single super phosphate or triple super phosphate. Therefore, it becomes very important to determine the amount of phosphate in sugarcane juice. As a consequence, various phosphate determination procedures have been reported^[10-22] which include for example, titrimetry^[10], complexogravimetry^[11], colorimetry^[12,13], atomic absorption spectroscopy^[14], flow injection analysis^[15], HPLC^[16] and spectrophotometry methods^[17-22]. Among such methods spectrophotometry involving

molybdovanadate^[23] and ammonium molybdate^[16] are most commonly used^[16]. In ammonium molybdate spectrophotometric method, different reductants have been employed such as tin(II) chloride^[23,25], ascorbic acid^[26] and 1-amino-2-naphthol-4-sulfonic acid^[27]. Some of these methods also involve complicated and expensive equipments^[16] and need extraction procedure^[20,23] and such techniques are usually not available in common laboratories. Though there are large number of methods^[10-22] available for the determination of phosphate but such methods used in sugar cane juice for phosphate determination are unread, instead only a few methods^[8,9,23,25] have been used. Therefore, it becomes very important^[7,8] to determine the phosphate in sugar cane juice^[7] and sodium sulphide is found to be a very effective reducing agent^[28] but not commonly used for phosphate determination^[28]. Considering the importance of phosphate and its determination in sugar cane juice an attempt is made here to develop the simple spectrophotometric method which is based on the formation of phosphomolybdate with added molybdate^[16,24] followed by its reduction with sodium sulphide^[28] in aqueous acidic medium.

EXPERIMENTAL

Elico spectrophotometer model SL 171 (Hyderabad, India) with 1 cm matched quartz cells, Sartorius digital balance readable 0.0001g were used.

Reagents

All the chemical reagents used were of analytical grade and the water used was distilled water. Weighed amount of ammonium molybdate, 1.7081 g was dissolved in about 150 mL of warm water^[27]; slightly milky solution obtained was cooled to room temperature. It was then transferred into a 250 mL volumetric flask and diluted to the mark with water. 0.05g of sodium sulphide was transferred into a clean 100 mL beaker. It was dissolved in about 50 mL of water and then the solution was transferred into a 100 mL volumetric flask. The beaker was washed 3-4 times with water and washings were also transferred into the flask and the solution was diluted to the mark with water. The solution was standardized iodometrically^[28] before use. 0.1145 g of disodium

hydrogen phosphate was transferred into a 150 mL beaker^[29]. It was dissolved in water, then; the solution was transferred into a 250 mL volumetric flask and diluted to mark with water. The working solutions were prepared by diluting 5 mL of this solution to 50 mL with water. Sulfuric acid (0.25N) was prepared by diluting the concentrated sulfuric acid ($\approx 36N$) with water.

Recommended procedure

A series of 10 mL volumetric flasks were arranged. To each flask 0.5 mL of 0.387M ammonium molybdate, 3mL of 0.25N sulfuric acid and aliquots of disodium hydrogen phosphate corresponding to 0.3-12.24 ppm (0.1, .2, 0.3, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, and 4.0 mL) were added. Finally, to each flask, 1 mL of 2.0833x10⁻³ M sodium sulphide solution was added. Then, each solution was let at room temperature for about 20 min. The absorbances of solutions were measured at 715 nm against water.

Sugarcane juice sample preparation

Sugarcanes were collected from the sugar cane fields situated around Mysore. Sugar cane juice was extracted from such sugar canes by laboratory crusher^[8]; the obtained juice was collected and stored in a separate reagent bottle. The same juice of 5 mL of that was diluted to 100 mL with water and filtered to separate any suspended particles^[25].

Detergent sample preparation

5g washing powder of Indian origin was placed in an oven for about half an hour at about 350 OC, the obtained ash was taken in a 150mL beaker. It was dissolved in about 50 mL water and the solution was made acidic by adding dilute sulfuric acid. It was heated on water bath for about 10 min. to expel the hydrogen sulphide, nitrite *etc.* It was filtered and the filtrate was transferred into a 100 mL volumetric flask and diluted to the mark with water, later it was used for its phosphate analysis.

Water sample

Water sample was collected from Gaurishanker Lake which is located in Bhavnagar city. Sample was filtered through a cellulose membrane filter, the filtrate containing orthophosphate, condensed phosphate

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(Pyro, Meta and poly phosphate) and organically bound phosphorous, all of which may be present in soluble form and in suspension. As is well known that only orthophosphate can be determined directly by phosphomolybdate procedures, pyro, tripoly and poly phosphate are completely hydrolyzed to orthophosphate by acidification and heating (30 min.) and it was used for its phosphate analysis.

RESULTS AND DISCUSSION

The developed method is based on the formation of phosphomolybdate^[11,17,23,25] due to the reaction between molybdate and phosphate followed by its reduction with sodium sulphide in aqueous sulfuric acid medium. Under optimized experimental condition, with fixed concentration of molybdate and reducing agent, the colour intensity was found to be proportional to the amount of phosphate present in disodium hydrogen phosphate, sugarcane juices, water and detergent samples. The reaction conditions as well as the various experimental parameters affecting the development and stability of the coloured complex were carefully investigated and optimized for quantitative determination of phosphate in various samples analyzed. The experimental variables such as concentration of ammonium molybdate, concentration of sulfuric acid, concentration of sodium sulphide, order of addition of the reagents and also colour stability of the complex were optimized for the effective determination of phosphate. The stability constant and composition of the system were also determined and values were found to be 1.992×10^6 L mol⁻¹ and 1:1 with respect to molybdate and phosphate. For a comparison of the results, phosphate present with those various samples was also determined separately following the official method^[25]. The results obtained for phosphate both by the proposed and official method^[25] are shown in TABLE 1 and 2 which account for the complimenting nature of the results from both the methods. Therefore, the proposed method could be either a substitute to an official method^[25] or it could also be an independent method for the determination phosphate in the sugarcane juice samples.

TABLE 1 : Phosphate determination from sugar cane juices

Sample	Phosphate mg/l	Phosphate mg/l
	Developed method	Official method
Sugarcane juice-1	160	165
Sugarcane juice-2	180	175
Water sample-1	35	40
Water sample-2	20	25

TABLE 2 : Phosphate determination from detergent power

Company	Phosphate (%)	Phosphate (%)
	Developed method	Official method
A	18	19
B	28	27.5
C	18.5	19

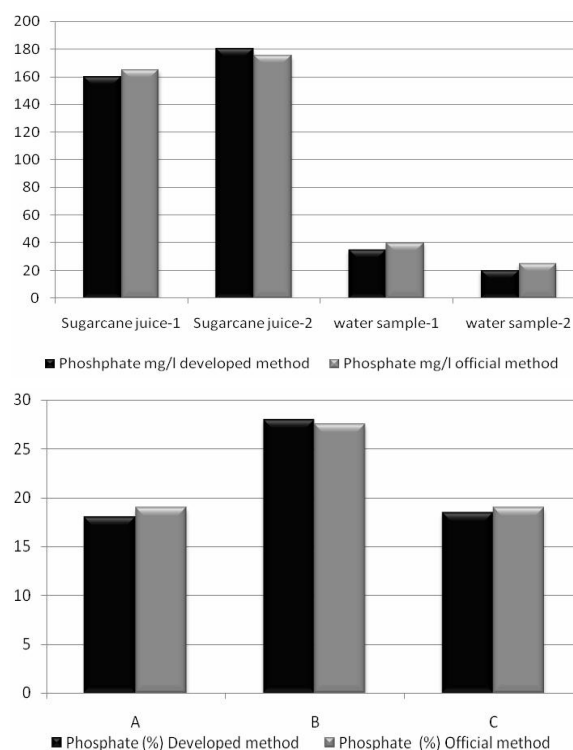


Figure 1 : Comparison between official method and developed method

Optimization

Effect of concentration of ammonium molybdate

First 5.528×10^{-3} M ammonium molybdate solution was prepared^[29,31]. Three series, each series having three labeled 10 mL volumetric flasks were arranged. To each flask of the first series 0.1 mL of 5.528×10^{-3} M molybdate solution was added. To each flask of the second series 0.3 mL of 5.528×10^{-3} M molybdate solution was added. To each flask of the third series 0.5

mL of 5.528×10^{-3} M molybdate solution was added. To the flasks of each series, measured but various volumes (0.2, 0.5, 1.0 mL) of phosphate (30 ppm) solution, 1 mL of 2.0833×10^{-3} M sodium sulphide solutions were added. All the solutions were diluted to the mark with water. The absorbance of the solutions were measured at 715 nm. The first series solutions containing 0.1 mL of molybdate showed absorbance 0.01, 0.02, and 0.05 for 0.2, 0.5, 1.0 mL of phosphate. It yielded a straight line, but ran almost parallel to x-axis, indicating low sensitivity. A second series of solutions containing 0.3 mL of molybdate solutions showed absorbance value 0.02, 0.03 and 0.05 for 0.2, 0.5 and 1.0 mL of phosphate respectively. This also yielded a straight line but indicated low sensitivity when the absorbance is plotted against concentration of phosphate. A third series of solutions containing 0.5 mL of ammonium molybdate solutions showed absorbance values of 0.07, 0.09, and 0.15 for 0.2, 0.5 and 1.0 mL of phosphate respectively. The absorbance values were found to increase with concentration of phosphate and the colour of the solutions were stable up to 2h. Therefore, 0.5 mL of ammonium molybdate was more preferred than its volume used in the earlier two sets solutions for the determination of phosphate.

Effect of concentration of sulfuric acid

The effect of concentration of sulfuric acid on absorbance was investigated to achieve high absorbance via taking various volumes (1-3 mL) of 0.25 N of sulfuric acid, 0.5 mL of ammonium molybdate, 1 mL of phosphate (30 ppm) and 1 mL of 2.0833×10^{-3} M sodium sulphide solutions were added to the 10 mL volumetric flasks. Then, the solutions were diluted to the mark with water. Absorbance of each solution was measured at 715 nm. The absorbance of the solutions were 0.02, 0.05 and 0.15 respectively for 1, 2 and 3 mL of 0.25 N sulfuric acid. The absorbance values indicated that the solution containing 3 mL of 0.25 N sulfuric acid appeared to be more sensitive. Therefore, it was used throughout the experiment to determine the phosphate.

Effect of foreign ions

The effect of foreign ions were examined by using 3 ppm of phosphate and the results obtained are shown in TABLE 3.

TABLE 3 : Effect of foreign ions on the determination of phosphate

ION	Salt added	Tolerance limit in ppm
Na	NaCl	100
K	KCl	180
Mg	MgSO ₄	350
Fe	Fe(NH ₄) ₂ (SO ₄) ₂	15
Ni	NiSO ₄	15
Cu	CuSO ₄	10
Cd	CdSO ₄	170
Zr	ZrOCl ₂	10

CONCLUSIONS

The proposed method is working on simple and straight forward principles of the reduction of phosphomolybdate by sodium sulphide leading to molybdenum blue which was monitored at, max 715 nm. The developed method found to be having the following advantages over the reported methods. (i) It is more sensitive when compared to the reported method^[17]. (ii) The preparation of reducing agent in this method is very simple when compared to that in the official method. (iii) It neither involves extraction nor sophisticated instruments like HPLC, flow injection spectrophotometer and atomic spectroscopy, nor requires strict control of pH or temperature or heat treatment. As the method is observed to be working satisfactorily for the determination of phosphate in various samples giving the results which are not only comparable with the results of phosphate determined separately from an official method and also reproducible as revealed by the values of statistical parameters like standard deviation, 3.7% and relative error, 3.4%. Therefore, the method could be employed for the determination of phosphate as an independent or a complementary one to the official method.

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