

**RESEARCH PAPER**

## **Optimization for Detection of On-Site Phosphate Contamination Qualitatively**

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### **Abstract**

Water pollution is a serious case when the amount of phosphate exceeds the safe level. This phenomenon will lead to eutrophication thus affecting the organisms under the water surface. Therefore, the water sample at the site area need to be tested directly so that contamination can be detected shortly. The objective of this study is to design a test kit for detection of phosphate content in water with minimum usage of reagents and water sample. Different volumes of ammonium molybdate reagent (0.5 and 1 mL) and stannous chloride (1 and 2 drops) were tested on different concentrations of phosphate. Reagents was then tested on different volume of water samples (25, 20, 15, 10 and 5 mL) and within 5 minutes, color changes were documented. It was found that the optimize reagents volume for phosphate contamination identification were 0.5 ml and 1 drop of molybdate reagent and stannous chloride, respectively. Minimum volume of water samples enough to react with reagents was at 15 ml. A phosphate test kit were then designed to test on-site phosphate contamination qualitatively. Then, the kit reading accuracy test was held on three different water samples. Quantitative analysis was also done by reading the contamination through UV-Vis spectrophotometer. It was found that there were only slightly different in the amount of phosphate content through qualitative and quantitative analysis.

**Keywords:** Phosphate test kit; Convenient; Accurate

### **Abstrak**

Pencemaran air adalah kes yang serius apabila jumlah fosfat melebihi paras yang selamat. Fenomena ini akan membawa kepada eutrophication sehingga mempengaruhi organisma di bawah permukaan air. Oleh itu, sampel air di kawasan tapak perlu diuji secara langsung supaya pencemaran dapat dikesan dengan segera. Objektif kajian ini adalah untuk merekabentuk kit ujian untuk mengesan kandungan fosfat dalam air dengan meminimumkan penggunaan reagen dan sampel air. Isipadu reagen yang diuji adalah ammonium molybdate (0.5 dan 1 mL) dan stannous chloride (1 dan 2 titis) pada kepekatan fosfat yang berlainan. Isipadu reagen yang telah dikenalpasti kemudian diuji pada jumlah sampel air yang berbeza (25, 20, 15, 10 dan 5 mL) dan perubahan warna telah didokumenkan. Telah dikenalpasti bahawa isipadu reagen bagi pengenalpastian pencemaran fosfat, masing-masing adalah 0.5 ml dan 1 titis reagen untuk molybdate dan stannous chloride. Manakala, jumlah minimum sampel air yang diperlukan untuk ujian ini adalah sebanyak 15 ml. Kit ujian fosfat kemudiannya direka khas dan ketepatan bacaan kit telah diuji dengan melakukan ujian kontaminasi fosfat kualitatif di kawasan kolam atau sungai secara terus pada tiga sampel air yang berbeza. Analisis kuantitatif juga dilakukan dengan membaca pencemaran melalui spektrofotometer UV-Vis. Didapati hanya sedikit perbezaan dalam jumlah kandungan fosfat melalui kedua-dua analisis.

**Kata kunci:** Kit ujian fosfat; Mudah; Tepat

## **INTRODUCTION**

Eutrophication was recognized as a pollution in European and North American lakes and reservoirs in the mid-20th century. In Malaysia, several lakes and reservoirs were eutrophic, meaning they were nutrient-rich, experiencing algae blooms, and they were likely to exhibit poor water quality (Zati et al. 2017). It may not look dangerous, but eutrophication is a harmful environmental process, especially when it is empowered by human activities, which is called cultural eutrophication and the amount of phosphate exceed the safe level. Eutrophication affecting the organisms under the water surface and cause death in humans and animals even at the least concentration when ingested in drinking water (Murakami et al. 2001). Moreover, eutrophication decreases the fitness for use of the water for water sports (swimming, boating and fishing) and the existence of dense algal growth areas can inhibit or prevent access to waterways. Thus, this study came out with one alternative solution to detect level of phosphate in affected water with minimum usage of reagents. The objective if this study is to design a phosphate test kit at a cheaper price so that the test is feasible during lab test in school or field sampling.

Affordable phosphate test kit is convenient for agriculture, landscaping purpose and even domestic use. Other benefits of using this test kit are water quality can often be improved, can control the water nutrients and greatly improved water clarity. The development of this test kit is very encourage able as the aims were for detection of phosphate content in the water with minimum usage of reagents and water sample. The water at the site area can be tested directly and contamination can be detected shortly. Thus, the product is environmental friendly and not harmful to mankind.

Phosphate test kit made is very cost effective, cheap and affordable. The product is lightweight and compact which suitable for field trip. The kit able to operate a qualitative test kit under simple instructions. The product is durable towards minor physical impact. It is cost effective and environmental friendly as minimum usage of reagents required compared to the existing commercialized test kits that are expensive and acidic which need a proper storage.

## **MATERIALS AND METHODS**

### **Obtaining natural water sample**

500 mL of water samples were taken from the surfaces of Lata Kinjang waterfall, Temoh pond and Zeta pond (UiTM Tapah). The water samples were kept into opaque plastic bottles and transferred to laboratory for further investigation.

### **Preparation of standard solution**

Standard solution of phosphate was prepared at different concentration (0.02, 0.05, 0.1, 0.2, 1.0 and 2.0 mg/L).

### **Experimenting based on standardized experiment**

25 mL of the water sample was poured in an Erlenmeyer flask to be analyzed. 1.0 mL of ammonium molybdate solution was poured into the flask and swirled. Next, 2 drops of stannous chloride solution were added into the flask. A blue color solution appeared in 5 minutes. These steps were repeated for each concentration of standard and blank. The spectrophotometer was set to wavelength of 650 nm. Blank solution was used to set it to be read as zero absorbance. The absorbance of the standard solutions was measured using 650 nm wavelength. The whole steps were repeated for natural water sample obtained.

### **Graphing standard curve**

A standard curve was made using the absorbance value obtained using spectrophotometer. From the concentration and the absorbance of the standard solutions, a plot of absorbance versus concentration was made using Microsoft Excel. The standard curve display the standard equation of  $y = mx + c$  and regression coefficient ( $R^2$ ).

### **Optimizing volume of stannous chloride and ammonium molybdate**

25 ml of water sample was tested using different amount of stannous chloride (1 and 2 drops) mixed with different volume of ammonium molybdate reagent (0.5 and 1 ml).

### **Optimizing volume of water sample**

Optimized volume of stannous chloride (1 drop) and ammonium molybdate (0.5 ml) were tested on different volumes of water samples (25, 20, 15, 10 and 5 mL). Color changes were recorded after 5 minutes.

### **Designing test kit**

A test kit was designed containing stannous chloride, ammonium molybdate, color indicator, dropper, syringe and mixing bottle. It then was ready for qualitative analysis of phosphate contamination.

### **Testing the test kit accuracy**

Phosphate concentration of natural water samples were tested according to optimized volume (15 ml of water sample, 1 drop of stannous chloride and 0.5 ml of ammonium molybdate) and then further analyzed quantitatively and qualitatively. Comparisons were made to confirm significant of using color technique for phosphate concentration in water sample. For quantitative analysis, water samples and reagents were analyzed by using UV- Vis spectrophotometer. The absorbance value or optical density reading then were used to calculate the phosphate concentration from standard curve plotted in graph (Figure 1a).













## **RESULTS AND DISCUSSION**

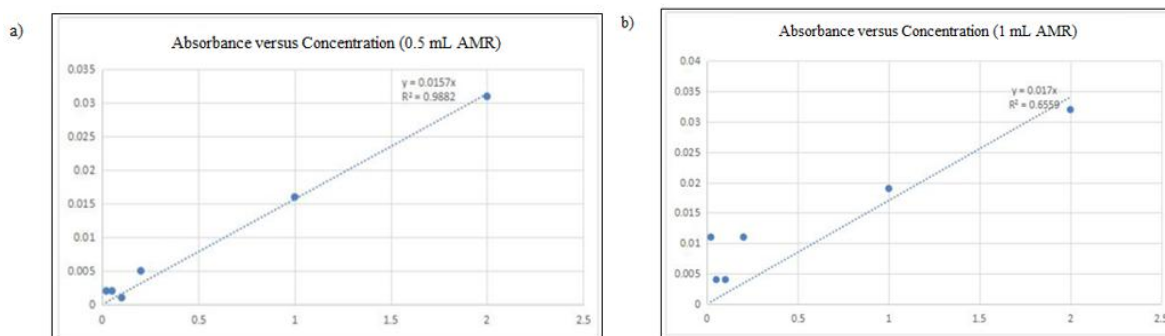
### **Optimization volume of stannous chloride and ammonium molybdate**

According to Uraisin *et. al*, (2016) phosphomolybdenum blue and silicomolybdenum blue is the products yield from the reaction of molybdate ions with stannous chloride in acidic medium. For optimization of stannous chloride, 1 ml of ammonium molybdate with phosphate solution at different concentration (0.02, 0.05, 0.1, 0.2, 1.0, and 2.0 mg/L) were mixed with 1 drop of stannous chloride. The color changes were observed and documented in Table 1. Same procedure then were repeated with 2 drops of stannous chloride.

Blue color were identified in high concentration of phosphate (2.0 mg/L). Optimization were done and based on the results obtained, it was qualitatively analyzed that different volume of stannous chloride (1 and 2 drops) did not shows any significant difference in the blue color changes, hence 1 drop of stannous chloride identified as the optimized volume.

**Table 1.** Color changes of added in different concentrations of phosphate and stannous chloride

Conc. of phosphate (mg/L)	1 drop of SnCl <sub>2</sub>	2 drops of SnCl <sub>2</sub>	Conc. of phosphate (mg/L)	1 drop of SnCl <sub>2</sub>	2 drops of SnCl <sub>2</sub>
0.02			0.2		
0.05			1.0		
0.1			2.0		



**Figure 1.** Absorbance value for different concentrations of ammonium molybdate (a) 0.5 mL (b) 1.0 mL

Optimization volume of ammonium molybdate were done at different volumes (0.5 mL and 1 mL). The reagent was tested using optimized stannous chloride (1 drop) with different concentrations of phosphate. Based on Figure 1, different volume of ammonium molybdate at every phosphate concentrations resulted no significant effect on the graph curves. Ammonium molybdate at 0.5 ml and 1 ml given slope reading at  $y=0.0157x$  and  $y=0.017x$  respectively with slight different of 0.002. Thus, minimum amount of ammonium molybdate (0.5 ml) was enough to detect phosphate at each concentration.

### Optimization volume of water sample

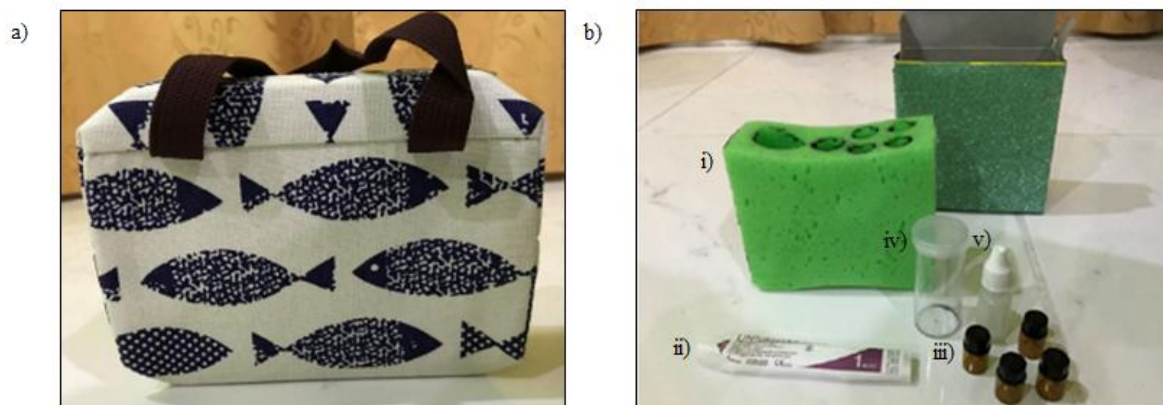
Six different volume of water sample (1, 5, 10, 15, 20 and 25 mL) were used in optimization of water sample. Different volume of water samples was mixed with optimized stannous chloride (1 drop) and optimized ammonium molybdate reagent (0.5 mL). According to Table 2, result showed that 15 mL of water sample provide the most substantial color changes. Using greater volume of water sample (>15 mL) is not suitable as no color changes can be observed.

**Table 2.** Blue color intensity in different volume of water sample

Concentration of phosphate (mg/L)	Volume of water sample (mL)					
	25	20	15	10	5	1
0.02						
0.05						
0.10						
0.20						
1.00						
2.00						

**Test Kit**

Resulted volume of reagents to quantitatively analyze phosphate content through color changes were identified and a test kit was designed to make it easy to be used in field. Kit containing with color indicator (Table 3), four small bottles of 3 mL ammonium molybdate reagent and a syringe. A bottle (dropper like) containing stannous chloride ( $\text{SnCl}_2$ ) were also provided together with 15 ml water sample bottle (Figure 2). The color indicator shows the intensity of phosphate concentration in water sample after added with optimized reagent. The test kit will be very helpful to detect qualitatively the phosphate contamination on site in easy way.















**Figure 2.** Sample of a) test kit bag b) the components inside test kit bag i) sponge ii) 1 mL syringe; iii) ammonium molybdate iv) sample bottles and v) stannous chloride.



**Test on different water samples quantitatively and qualitatively**

Water samples from several locations were tested qualitatively using the designed on-site phosphate test kit. The results obtained from the analysis was tabulated in Table 4.

**Table 3.** Color indicator for qualitative analysis of phosphate concentration

Phosphate concentration (mg/L)	Color intensity	Phosphate concentration (mg/L)	Color intensity
0.02		1.5	
0.05		2.0	
0.1		2.5	
0.2		3.0	
0.5		4.0	
1.0		5.0	

**Table 4.** Comparison of phosphate detection through qualitative and quantitative analysis.

Water samples	Color	Concentration of phosphate (mg/L)	
		Result according to kit color indicator (Qualitative)	Quantitative
Lata Kinjang waterfall		0.05 mg/L Not contaminated	$0.04 \pm 0.02$ mg/L
Temoh (large pond)		0.5 mg/L Not contaminated	$0.81 \pm 0.18$ mg/L

UiTM Tapah, Perak  
(small pond)



1.5 mg/L  
Contaminated and exceed safe  
level

$1.8 \pm 0.07$  mg/L

Safe level = less than 1.0 mg/L

Lata kinjang waterfall and Temoh pond showed the tested sample remain colorless after being mixed with reagent. According to the color indicator, the water samples collected were not contaminated with phosphate as it place in range of 0.05 to 0.5 mg/L of phosphate concentration. For the third water sample from pond in UiTM Tapah (Zeta pond) the tested water sample color had changed from colorless to blue indicates presence of phosphate in the water sample. The Zeta pond phosphate concentration was recorded at 1.5 mg/L by referring to the kit color indicator. This concentration was describe as a contaminated concentration as a safe level of phosphate content in water must be less than 1.0 mg/L. According to quantitative analysis in Table 4, the concentration of phosphate shows only slight different with the qualitative result analysis. This prove that this phosphate kit qualitative analysis were reliable to be used in sampling site.

## CONCLUSION

Based on the result obtained, 0.5 ml of ammonium molybdate, 1 drop of stannous chloride and 15 ml of water sample were identified as the optimized volume for detection of phosphate using the test kit. This qualitative analysis test kit was reliable to be used at sampling site as it gives slightly similar results compared with the quantitative analysis using UV-vis spectrophotometer.

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