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Optimization spectrophotometric determination of phosphor in soil and compost mixture

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ABSTRACT

Determination of phosphor in soil and compost mixtures by spectrophotometric method has been optimized. The method in this study was based on the formation of molybdenum blue complex by ammonium heptamolybdate as complexing and potassium antimonyltartarate to increase the intensity of the blue color. The optimum condition were found at solution pH 2, the wavelength 725 nm, complex stability for 10 minutes. Standard calibration curve obtained from the regression equation $y = 0.349x + 0.006$, with R^2 0.995 for $n = 3$. Phosphor content in the mixtures of soil containing 0.3648 % phosphor and compost that made from green yard waste with EM4 as activator was evaluated by varying mixture composition and incubation time. The highest phosphor content obtained 0.4165% in the composition of soil : compost 0.4 : 0.6 w/w with incubation time for 45 days.

Keywords: Compost, phosphor, soil, spectrophotometric method

INTRODUCTION

The use of inorganic fertilizer/chemical fertilizers continuously without control will hardened soil structure and land damaged [1]. Rehabilitation by adding organic matter to the soil was very efficient to recover land damaged [2].

Compost can be given as the resource of additional nutrients to restore soil fertility. It should be given in sufficient quantities, so that plants can grow better. Compost is like multivitamins for the soil and plants will improve soil physical and chemical properties. Hard soil will become more loose, poor soil will be fertile, acid soils will become more neutral. Some element that is contained by the compost is nitrogen, phosphorus, calcium, potassium and magnesium [3].

The use of organic materials is currently getting a lot of attention from various circles. According to Singh. (1993), organic matter can increase the cation exchange capacity, the ability of nutrients and water, the activity of microorganisms, as well as a variety of other soil properties. Organic acids can effectively decrease the toxicity of Al to form a complex with Al, thereby increasing the availability of P, which is one of the important nutrient in plant growth [4-6].

From previous research compost was made from green yard waste with EM4 as activator and the content of macro nutrients (N, P, K) was analyzed [7]. However, the content of phosphor in the mixture of soil and compost is not reported yet, in this study optimization spectrophotometric determination of phosphor in soil and compost mixtures was evaluated. Determination of phosphor by spectrophotometric method was based on the formation of molybdenum blue complex by ammonium heptamolybdate as complexing and potassium antimonyltartarate to increase the intensity of the blue color.

EXPERIMENTAL SECTION**Equipment and Materials**

The equipments were used in this study: a set of UV-Vis spectrophotometer (Genesys 20, Thermo Scientific), analytical balance (Kern & Sohn GmbH), pH meter (Lovibond Senso Direct), hot plate and glassware. While the materials were: soil taken from its own grounds, compost from green waste yard, nitric acid (HNO₃) p.a, hydrogen peroxide (H₂O₂) p.a, sulfuric acid (H₂SO₄) p.a, potassium dihydrogen phosphate (KH₂PO₄), ammonium tetrahydrate heptamolybdate ((NH₄)₆Mo₇O₂₄·4H₂O), antimonytartarate potassium (K(SBO)C₄H₄O₆·½H₂O), ascorbic acid (C₆H₈O₆), hydrochloric acid (HCl) 0.01 M, nitric acid (HNO₃) 0.1 M, filter paper, and distilled water.

Reagents Preparation

Nitric Acid 0.1 M, Chloride Acid Solution 0.01 M, Phosphorus Standard Solution 1000 mg / L, 10 mg / L were prepared by dilution and complexing reagent was prepared by weighing as much as 12 g Ammonium Heptamolybdate, add 50 mL of H₂SO₄ p.a and 0.2270 g of potassium antimonytartarate then diluted in 500 mL volumetric flask with distilled water and homogenized. As much as 250 mL of this solution was added 1.0600 g of ascorbic acid incorporated into the 1000 mL volumetric flask, then diluted with distilled water and homogenized [8]

Sample Preparation**Preparation of Compost from Organic Yard Waste**

Compost was prepared from organic yard waste such as dried leaves and fresh leaves that have been separated from a tree branch. The leaves were cut with a smaller size and homogenized, 50 kg of organic yard waste was poor with water until the leaves were wet. About 50 mL EM4 and 50 mL palm sugar solution (1 kg in 500 mL) were added and stirred, put into the composter, closed and stirred once a week until the compost produced. After harvest, sifted compost and compost obtained smooth.

Preparation of Composition Variation of Soil and Compost Mixture

Soil and compost were mixed with variation of the composition of the soil : compost (1:0; 0.8:0.2; 0.6:0.4; 0.4:0.6; 0.2:0.8; 0:1) in a plastic cup with a total weight of 50 g. The sample was incubated with the variation of time (0; 15; 30; 45; 60 and 75) days and watering every morning.

Sample Destruction

All samples were destructed by inserted each sample 0.5 g into the Kjeldahl flask. Then added 5 mL of HNO₃ p.a, 0.5 mL of H₂O₂ and allowed 30 minutes to react completely and then heated with a heating mantle until the yellow runs and the solution became clear, let it cool and filtered to obtain the extract clear. Then adjusted the pH to 2 by using a solution of NaOH or HCl, and then diluted to the appropriate volume of 50 mL with 0.01 M HCl.

Determination of Water Content in Soil and Compost Mixture

Water contained in soil and compost mixture were determined by gravimetric method. The sample was weighed 1.0002 g at vaporizer cup. Then heated in an oven for ± 2 hours at temperature 105^o C until constant weight was found.

Optimum Condition Determination of Phosphorus by Spectrophotometric Method

Optimum conditions of Phosphorus determination by spectrophotometric method were evaluated by determining maximum wavelength, effect of pH solution, effect of time, effect of potassium antimonytartarate concentration and effect of ammonium heptamolybdate concentration on molybdenum blue complex stability.

Determination of Phosphorus Content in Soil and Compost Mixture**Preparation of Standard Phosphorus Calibration Curve**

Phosphorus standard solution was prepared with concentrations (0; 0.1; 0.2; 0.3; 0.4; 0.5; 1.0 and 2.0) mg/L at pH 2. Each standard solution of 1 mL was added with 9 mL of complexing reagents contained ammonium heptamolybdate and potassium antimonytartarate 0.0194 M and 0.0014 M. Then left for 10 minutes and absorbance was measured by UV-Vis spectrophotometer at a wavelength of 725 nm.

Determination of phosphorus in Samples

Optimum condition was applied to determine phosphorus content in soil and compost mixture. Each of sample destruction were diluted 25 times and 1 mL of solution was placed in tube and added 9 mL of complexing reagent, left for 10 minutes to perform stable complex. The absorbance was measured by UV-Vis spectrophotometer at a wavelength of 725 nm.

RESULTS AND DISCUSSION

Maximum Absorption

The absorbance of phosphomolybdate was measured at range of wavelength 670-750 nm by UV-Vis spectrophotometer, Figure 1 showed that the maximum wavelength at 725 nm.

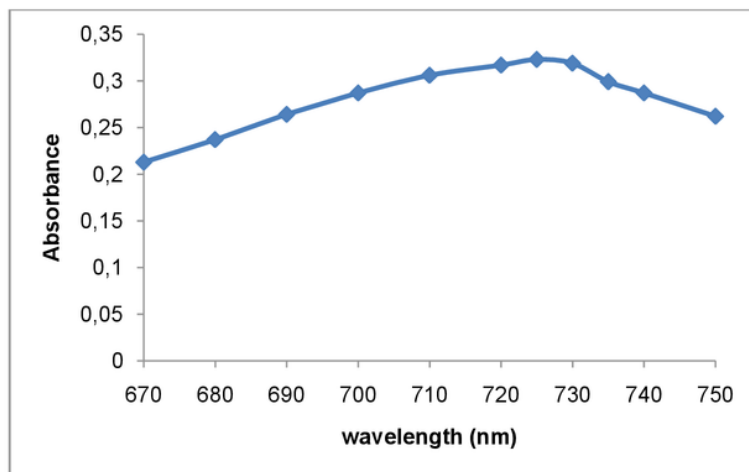


Figure 1. Absorption curve of phosphomolybdate complex solution at a wavelength of 670-750 nm. 1 mL of phosphate standard solution of 10 mg/L, 9 mL complexing reagent

Absorbance value at a wavelength of 670-725 nm increased. But with increasing wavelength above 725 nm the absorbance decreased. So from the above data shows that the maximum wavelength obtained at a wavelength of 725 nm which is evidenced from the highest absorbance value.

Effect of Time on the Stability of Molybdenum Blue Complex

Determination of the effect of time on the stability of the complex carried out on the variation of time (0; 5; 10; 15; 20; 25; 30; 35; 40; 45; 50) minutes and the results can be seen in Figure 4.2.

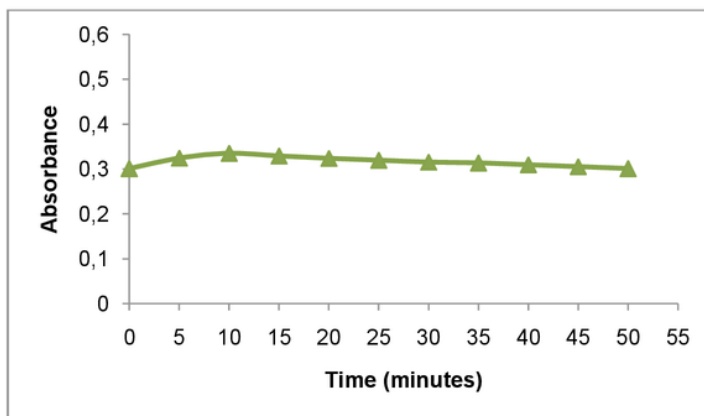


Figure 2. Curve of time effect on the Stability of Molybdenum Blue Complex.

Description: 1 mL of phosphate standard solution of 10 mg/L, 9 mL complexing reagent (ammonium heptamolybdate 0.0097 M and 0.0007 M potassium antimonyltartrate). Wavelength 725 nm.

Figure 2 indicated that the absorbance from 0-10 minutes increased slightly and after that it tend to stable. From the curve can be concluded that the stability of phosphomolybdate complex was high and can be measured at long range of time.

Effect of Potassium Antimonyltartarate Concentration on the Stability of Molybdenum Blue Complex

The complexing agent used was a mixture of ammonium heptamolybdate 0.0097 M with potassium antimonyltartarate (0.0007 ; 0.0014 ; 0.0027 M) were measured with a UV- Vis spectrophotometer at a wavelength of 725 nm .

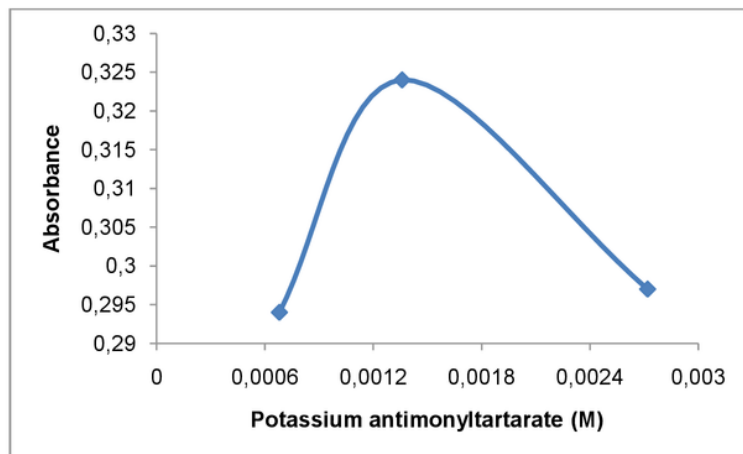


Figure 3. Curve of Effect Potassium Antimonyltartarate Concentration on the Stability of Molybdenum Blue Complex.

Description: 1 mL of phosphate standard solution of 10 mg/L, 9 mL of complexing reagents ammonium heptamolybdate (0.0097 M) and potassium antimonyltartarate (0.0007 ; 0.0014 ; 0.0027 M), for 10 minutes at wavelength 725 nm.

From the variation of the concentration of potassium antimonyltartarate, at a concentration of 0.0014 M has the highest absorbance value which indicated that the stable molybdenum blue complex was performed and gave the maximum absorbance.

Effect of Ammonium Concentration on the Stability of Molybdenum Blue Complex.

Determination of the maximum concentration of ammonium heptamolybdate to perform the stability of molybdenum blue complex with phosphate by using various of concentration of ammonium heptamolybdate (0.0097; 0.0121; 0.0146; 0.0170; 0.0194; 0.0218 M) with potassium antimonyltartarate 0.0014 M as complexing reagent, which is measured with UV - Vis spectrophotometer at a wavelength of 725 nm. Result can be seen in Figure 4 .

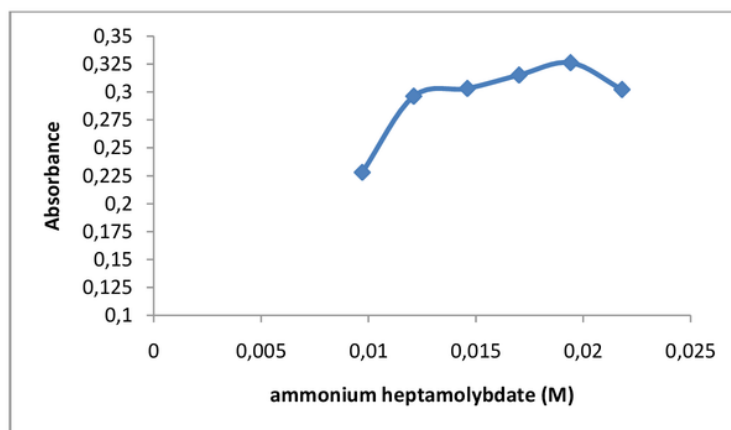


Figure 4. Curve of Ammonium Heptamolybdate Concentration effect on the Stability of Molybdenum Blue Complex.

Description: 1 mL of phosphate standard solution of 10 mg/L, 9 mL of complexing with the concentration of complexing reagents (ammonium heptamolybdate 0.0097; 0.0121; 0.0146; 0.0170; 0.0194; 0.0218 M) and potassium antimonyltartarate 0.0014 M), for 10 minutes at wavelength 725 nm.

From Figure 4. The highest absorbance was obtained at a concentration of 0.0194 M. At that concentration, ammonium heptamolybdate performed a stable complex. This is evidenced from the molybdenum blue color is formed so clear. On the other hand at concentration below 0.0194 M the complex was uncompletely performed, while the concentration of 0.0218 M the suspension was performed that interfered in the measurement. Therefore heptamolybdate ammonium concentration used was 0.0194 M, which reached maximum absorption.

Effect of pH on the Stability of Molybdenum Blue Complex.

The effect of pH to performed the stable complex of molybdenum blue was determined by varying the pH 2; 3; 4; 5 and 6 of standard solution, from that variation only solution with pH 2 produced molybdenum blue color, whereas at pH 3; 4; 5 and 6 did not form complex. The blue color indicated that fosfomolybdate and well known as molybdenum blue complex.

To make sure the optimum pH that produced stable complex, a series of phosphate solution with pH 1.5; 2; 2.5 and 3 reacted with complexing reagent and the results can be seen in Figure 5.

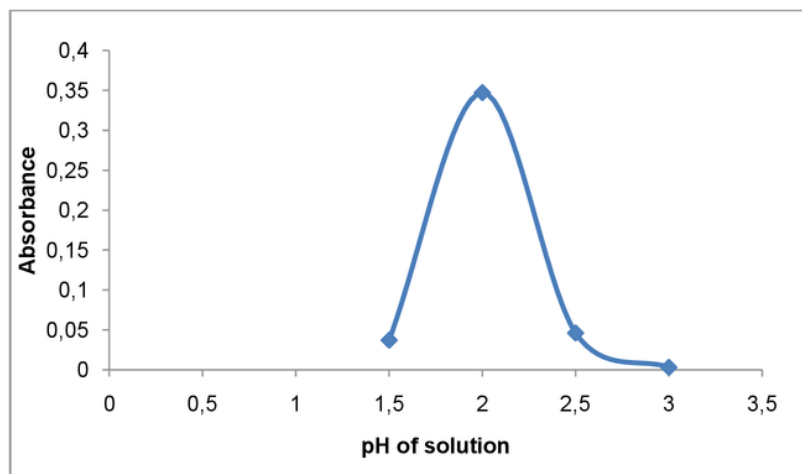


Figure 5. Curve of pH Effect on the Stability of Molybdenum Blue Complex.

Description: 1 mL of phosphate standard solution of 10 mg/L pH 1.5; 2; 2.5; 39 mL with a concentration of complexing reagent (ammonium heptamolybdate 0.0194 M and potassium antimonyltartrate 0.0014 M) for 10 minutes at wavelength 725 nm.

From Figure 5. the highest absorbance value was at pH 2. This is because phosphomolybdate complex formed at pH 2 with blue color. At pH 1.5 and 2.5 the blue color of the solution was not very clear, because of the fosfomolybdate complex formed was not stable. Whereas at pH 3 did not form a blue color. This proved that the complex formation of phosphomolybdate (molybdenum blue) stable at pH 2.

Phosphor Content in Soil and Compost Mixtures

Determination of phosphorus in soil and compost mixture by spectrophotometry was done under optimum conditions with concentration of complexing agent (ammonium heptamolybdate and potassium antimonyltartrate 0.0194 M and 0.0014 M), pH 2, complex stability for 10 minutes and wavelength of 725 nm. Phosphorus content in samples were calculated by using regression equation $y = 0.349x + 0.006$ with $R^2 = 0.995$.

Figure 6. showed that the phosphorus content in each of the various time 0; 15; 30; 45; 60; 75 days had high levels of phosphorus and increased significantly with the addition of time. However, the highest phosphorus levels could be seen in the composition of the soil: compost 0.4 : 0.6 v/v 0.4165 % within 45 days and decreased at 60-75 days. Higa and Kinjo (1989) [20] stated that the availability of P can be utilized effectively and efficiently by the plant for several weeks.

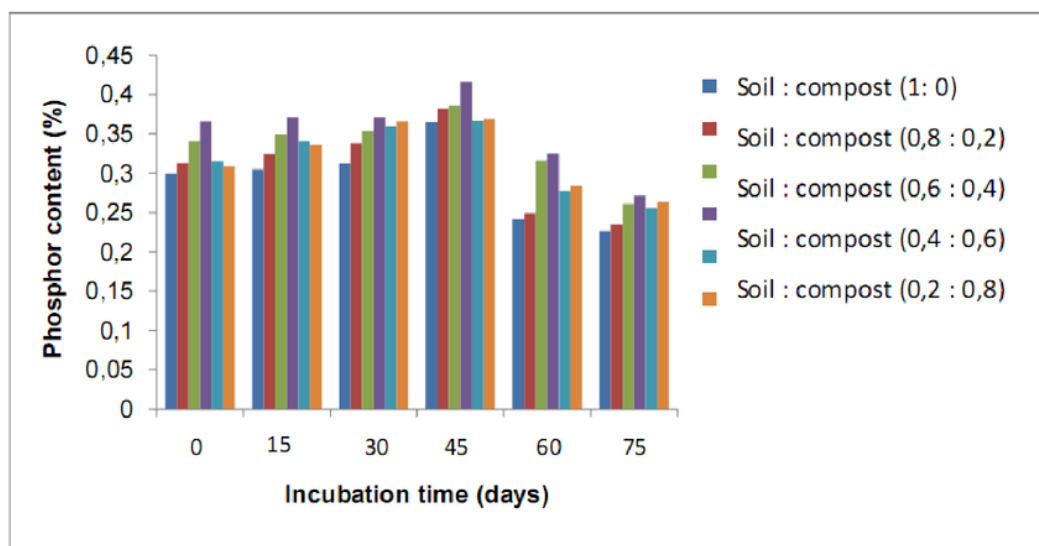


Figure 6. The content of phosphorus in the soil and compost mixture with a variety of time and composition
 Description: 1 mL of destructed samples pH 2 was added 9 mL complexing reagents (ammonium heptamolybdate and potassium antimonyltartrate 0.0194 M and 0.0014 M), complex stability 10 minutes at a wavelength of 725 nm.

CONCLUSION

Phosphorus levels in mixture of the soil containing 0.3648 % and compost containing 0.3691 % phosphorus had the highest content 0.4165 % in the composition of the soil: compost 0.4 : 0.6 w/w within 45 days incubation time. On this composition compost could increase the phosphorus content in soil about 0.05 %.

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