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Assessment of Phosphorus Sorption and Desorption
Characteristics of Tropical Acid Soil Applied with
Paddy Husk Compost

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DECLARATION

I hereby declare that the work embodied to this Report is the result of the original research and has not been submitted for a higher degree to any universities or institutions.

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I certify that that the Report of this final year project entitled “Assessment of Phosphorus Sorption and Desorption Characteristics of Tropical Acids Soil Applied With Paddy Husk Compost” by UMI ZAHRAH BINTI NANYAN, matric number F15A0240 has been examined and all the correction recommended by examiners have been done for the degree of Bachelor of Applied Science (Agrotechnology) with Honours, Faculty of Agro-Based Industry, Universiti Malaysia Kelantan.

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Assessment of Phosphorus Adsorption and Desorption Characteristics of Tropical Acids Soil Applied With Paddy Husk Compost

ABSTRACT

Phosphorus (P) is an essential component of adenosine triphosphate (ATP), which is involved in most biochemical processes in plants and enables them to extract nutrients from the soil. It plays an important role for root growth, it is often major element in starter fertilizers, those applied at planting. Applications of organic amendment is highly recommended in reduce phosphorus sorption and improve the Phosphorus availability in the soil. Thus, the objective of this study are to determine the changes in the soil P sorption and desorption upon application of paddy husk compost and the soil maximum P buffering capacity of the soil upon applied of paddy husk compost. Paddy husk compost was used and the sorption data were fitted to the Langmuir Equation. The results proved that at initial P concentration of $< 20 \text{ mg L}^{-1}$, sorbed P increased with the amendment of paddy husk compost while more Phosphorus desorbed with increasing P application rates (20 to 30 mg L^{-1}). The decreases in P sorption in this study was due to the precipitation of exchangeable aluminium and iron at the highly negatively charged humic substances functional group surfaces of the organic amendment. The sorption of phosphorus was enhanced by increase of soil pH due to application of organic amendments. Besides, amending of paddy husk compost showed high buffering capacity and increased the availability of phosphorus in acid soil used in this study. The finding shows suggests that the application of paddy husk compost can enhanced the soil phosphorus availability by changing the soil sorption and desorption capacity and pH buffering capacity.

Keywords: *Phosphorus, paddy husk, sorption, desorption, pH buffering capacity*

**Penilaian Penyerapan dan Desorpsi Fosforus untuk Jenis Tanah Asid Tropika
Dengan Menggunakan Kompos Sekam Padi**

ABSTRAK

Fosforus (P) adalah komponen utama adenosine trifosfat (ATP) yang terlibat dalam proses biokimia pada tumbuh-tumbuhan dan membolehkan ia mengekstrak nutrien dari tanah. P memainkan peranan penting untuk pertumbuhan akar dan elemen utama dalam baja pemula yang diguna pakai untuk penanaman. Penggunaan organik amat disyorkan untuk mengurangkan penyerapan dan meningkatkan ketersediaan fosforus di dalam tanah. Objektif kajian ini adalah menentukan perubahan dalam penyerapan dan desorpsi fosforus tanah dan untuk menentukan kapasiti bufer pH yang maksimum pada penggunaan kompos sekam padi. Hasil membuktikan bahawa pada kepekatan awal P <20 mg L⁻¹, serapan fosforus meningkat dengan pindaan kompos sekam padi manakala pada kenaikan kadar aplikasi fosforus (20 hingga 30 mg L⁻¹) lebih banyak fosforus diserap ke dalam tanah. Penurunan dalam penyerapan fosforus dalam kajian ini adalah disebabkan oleh mendakan pertukaran aluminium dan besi yang boleh pada bahan-bahan humik yang sangat negatif yang dikenakan ke atas permukaan kumpulan berfungsi bagi pindaan organik. Penyerapan fosforus dipertingkatkan dengan peningkatan pH tanah yang disebabkan oleh pindaan organik. Dalam kajian ini, pindaan kompos sekam padi telah menunjukkan kapasiti bufer yang tinggi dan ketersediaan fosforus dalam tanah yang berasid. Hasil kajian menunjukkan bahawa penggunaan kompos sekam padi dapat meningkatkan ketersediaan fosforus tanah dengan mengubah kapasiti penyerapan dan desorpsi tanah serta kapasiti bufer pH.

Kata kunci: Fosforus, sekam padi, penyerapan, desorpsi, kapasiti bufer pH

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LIST OF ABBREVIATIONS AND SYMBOLS

P	Phosphorus
KH_2PO_4	<i>Potassium dihydrogen phosphate</i>
Al	Aluminium
Fe	Iron
H_2PO_4^-	Dihydrogen phosphate
N	Nitrogen
K	Potassium
Mn	Manganese
Ca	Calcium
Zn	Zinc
NaOH	Sodium hydroxide
HCl	Hydrochloric acid
H_2SO_4	Sulphuric acid
KCl	Potassium Chloride
Cl^-	Chloride ion
AAS	Atomic Absorption Spectroscopy
UV-VIS	Ultraviolet and Visible Spectroscopy
ANOVA	Analysis of variance

CHAPTER 1

INTRODUCTION

1.1 Research Background

Phosphorus (P) is an important element that is classified as macronutrient as it is needed by the plant in large amount. Phosphorus is one of three nutrients usually added to soil as fertilizer and plays an important role in plants to store and transfer energy generated by photosynthesis for use in growth and reproduction processes. According to United States Department of Agriculture (2001), P is released faster when the soil is well aerated (higher oxygen levels) and much slower on saturated wet soils. Sufficient P levels encourage strong growth and shoot, advance early maturity, improve water efficiency and grain yield. Thus, P deficiency stunts vegetative growth and grain yield. P availability can be controlled by three primary factors: soil pH, amount of organic matter, and proper placement of fertilizer phosphorus used by the crops (California Fertilizer Association, 1995).

Most P is stored in organic and fixed form. According to Edward (2002), only a small amount of phosphate ion is in the solution and usable by plants, and the uptake of P depends on a number of soil conditions. As plants remove phosphate from solution, mineral and organic phosphate become soluble *via* mineralization and immobilization.

The main factor in losing P in tropical acid soils is because of soluble inorganic P is fixed by aluminium (Al) and iron (Fe). Organic amendments can be used to overcome P fixation in acid soils and increased soil pH compared with inorganic amendments. Moreover, organic amendments increased soil pH and reduced exchangeable acidity, exchangeable aluminium, and exchangeable iron (Ch'ng, Ahmed, & Nik Muhammed, 2014). As a result, P availability increased and improve nutrient uptake in soil.

Sorption of P can be measured to determine the potential of P fixation, or at the other extreme of soil P saturation, estimate the risk of P loss. Phosphorus sorption can be measured in two ways; exposing soil to a high P spike (single-point sorption) or determining a sorption isotherm by exposing a series of soil samples to different concentration of P (Essington,2004). In Guinea, sorption isotherm was used to estimate P application to highly fixing soil based on calculated P fixation for the upper plow horizon (Nwoke et al,2003).

Paddy husks are defined as organic matter which is produced of by-products from harvesting and processing of agricultural crops. According to Salman Zafar (2015), paddy husk residues are usually available in relatively large quantities at the processing site and may be used as captive energy source. Most of the husk from the milling is either burnt or disposed as waste in open fields and a small amount is used as fuel for boilers, electricity generation, bulking agents for composting of animal manure.

Compost is known as the mixture of decaying organic substances as dead leaves that is added to soil to improve its quality. The compost may be used as a soil amendment. The benefit of compost application as the organic amendment are reducing the weight and volume of organic material, making it easier to handle and to kill harmful organisms. Besides, it reduces the C: N ratio of organic materials and eliminating the problem of nitrogen tie-up when compost is added to soil. This application which shows high capacity of organic amendment is

able to improve the physical condition and support the growth of beneficial living organism in the soil (Edward, 2002). Furthermore, the application of compost has beneficial effect on soils and also plants which can improved water holding capacity, plant water availability, increased soil structural stability, soil N and P content and microbial activity (Verma, 2013).

Soil acidification rates requires knowledge of pH buffering capacity. The importance of increasing the pH value of buffering capacity at low pH has been recognised in modelling studies of pH change. It is estimated that pH buffering capacity represents to mineral phase, organic matter content and varies with the Al^{3+} concentration in soil solution that takes into account increasing in pH buffer capacity (Nelson & Su, 2010).

1.2 Problem Statement

The effect of paddy husk compost applied on the soil P sorption and desorption has rarely been reported. Understanding the fundamental aspects of the reaction between compost and soil, as well as the mechanism of the increase in soil P availability is important.

1.1 Hypothesis

1)

Null Hypothesis, H_0 : The degree of soil P sorption and desorption remain unchanged upon application of paddy husk compost

Alternative Hypothesis, H_1 : The degree of soil P sorption and desorption change upon application of paddy husk compost.

2)

Null Hypothesis, H_0 : The soil maximum P buffering capacity remain unchanged upon application of paddy husk compost.

Alternative Hypothesis, H_1 : The soil maximum P buffering capacity change upon application of paddy husk compost.

1.2 Research Questions

Can the application of paddy husk compost change the degree of P sorption and desorption in the soil? Can application of the paddy husk compost influence the soil maximum P buffering capacity of the soil upon applied of paddy husk compost.

1.3 Objectives

The objectives of this studies are:

1. To sample and characterize all the soil samples used in this study.
2. To determine the changes in the soil P sorption and desorption upon application of paddy husk compost.
3. To determine the soil of maximum P buffering capacity upon application of paddy husk compost.

1.4 Scopes of Studies

The study is conducted to determine the way to minimize the sorption of P that applied with the paddy husk compost.

1.7 Significance of Study

This study can help to provide fundamental understanding on the mechanism of P sorption and desorption using organic amendment such as compost.

CHAPTER 2

LITERATURE REVIEW

The study of P has always been intensive as it is an essential plant nutrient. The availability and the nutrient imbalance in the root zone can give impact to the plant growth. It is also crucial to optimize agronomic production while minimizing eutrophication and other environmental hazards.

2.1 Phosphorus cycle

Phosphorus is a limiting nutrient for terrestrial productivity and plays a key role in the net carbon uptake in terrestrial ecosystems. The availability of new P in ecosystems is restricted by the rate of the release of this element during soil weathering. Because of the limitation of P availability, P is generally recycled to various extents in ecosystems depending on climate, soil type, and ecosystem level (Filippelli et al. 2006). Figure 1 shows the pathways of P form by which P may be taken up by plants or leave the site as P runoff or leaching.

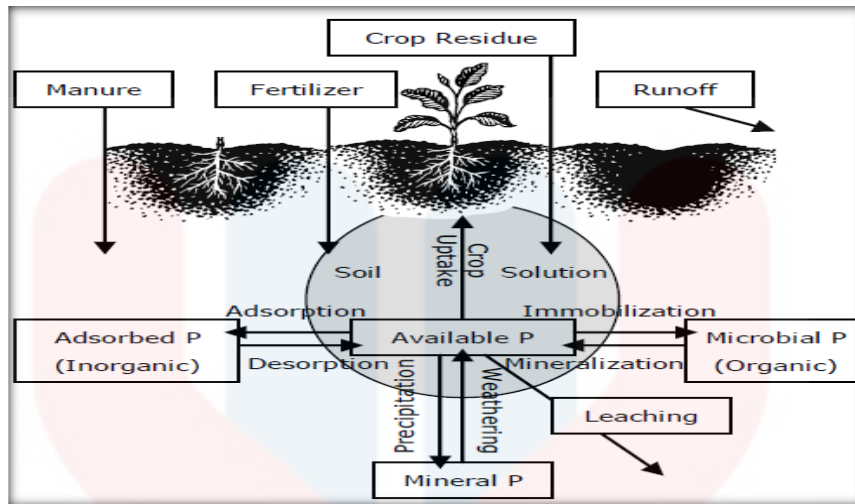


Figure 1: Simplified P cycle

Source: (Fact & Series, n.d.)

The general processes of P are: weathering and precipitation, mineralization and immobilization, and adsorption and desorption. The process of weathering, mineralization, and desorption will increase the plant available P while immobilization, precipitation, and adsorption decrease plant available P.

Mineralization is a biological process in which soil microbes transform organic forms of P in plant residues or organic soil amendments into plant-available P. Phosphorus can be attached to soil particles such as clay or certain minerals containing iron (Fe) or aluminium (Al). Phosphorus can release from these soil particles, supplying P to the soil solution through a process called desorption. Finally, solid rocks can be a source of P as they break down into soil over a long period of time by a process called weathering. Simultaneously, remineralisation may occur during the incubation period due to the recycling of microbial P as a result of mortality and predation of

microorganisms and signifies mineralization of the newly synthesized organic P, (Randhawa et al., 2005).

2.1.1 Importance of P

Phosphorus is one of three major elements (N, P, and K) that are required for plants and livestock (Syers et al. 2008; Tiessen 2008). It is important for The quantities of P in soil are generally small, and this often limits plant growth. The lack of phosphorus limits the development of leaf and surface area and leaf number. Besides, it also can reduce the quality of fruits and vegetables. A recent study on plant species diversity in Eurasian wetlands and grasslands (Fujita et al. 2014) showed that plants in P- deficient communities had shorter flowering periods and lesser seed production, and therefore, endangered species were more frequent in P-deficient environments.

2.1.2 Present concerns with Phosphorus

2.1. 2. 1 Weathering

The main source of P is found in rocks. The first step of the P cycle involves the extraction of P from the rocks by weathering. Weather events, such as rain and other sources of erosion, result in phosphorus being washed into the soil.

Soil is provided by the weathering of minerals like the apatite, which are calcium phosphate minerals. As apatite weathers, it releases anions that can be used by plants. These anions are primary orthophosphate (H_2PO_4^-) and a secondary orthophosphate (HPO_4^{2-}). The rain and the weathering process will cause removal inorganic phosphates is then distributed in soil and water. The plants take up the inorganic phosphates from the soil and utilized by animals that consume that plants (Pagliari, Kaiser, Rosen, & Lamb, 2009). Therefore, P can become unavailable through precipitation, if inorganic P reacts with dissolved Fe, Al, Mn (in acid soils), or Ca (in alkaline soils) to form phosphate minerals. The conservation of organic to inorganic phosphate is known as mineralization while the reverse reaction of mineralization is immobilization.

2.1.2.2 Mineralization and Immobilization

Mineralization of organic matter results in the slow release of P to the soil solution during the growing season, thus make it available for plant uptake. The mineralization is the process of microbial conversion of organic P to inorganic resulting the forming of plant available P known as orthophosphate. This process reduces the need for fertilizer applications and the risk of runoff and leaching. Moreover, immobilisation occurs when plant available P are consumed by microbes, turning the P into organic P forms that are not available to plant (Fact & Series, n.d.). The carbon(C): P ratio determines whether there is net mineralization or net immobilization occur. Net mineralization occur when there is enough P in the soil to sustain both plants and microorganism (CTAHR. 2017).

2.2 Forms of Phosphorus in the soil

2.2.1 Solution P pool

Phosphorus exists in many different forms in the soil. Solution P pool is very small and usually contains P less than a kilogram per hectare. It is mostly inorganic P. Plants take up P primarily in the form of orthophosphate. Solution P is important because it is the pool from which the plant takes P and the only pool with measurable movements (Pagliari et al., 2009). Most P taken by plants during the growing season will move just a few millimetres or less from the ground to the roots. A growing plant would quickly reduce the P in the soluble P pool if the pool is not continuously recharged by the active and fixed pools.

2.2.2 Active P pool

Active P pool is the organic P that is attached to small particles in soil. As plants take up phosphate, the concentration of phosphate in the solution is decreased and some phosphate from the active P pool is released. This is the main source of P available for crops. The active P pool will contain inorganic phosphate that attaches to small particles in the soil. Phosphate that react with elements such as Ca or Al to form somewhat soluble solids, and organic P that is easily mineralized (Pagliari et al., 2009).

2.2.3 Fixed P pool

Fixed P pool of phosphate contains inorganic phosphate compounds that are very insoluble and resistant to mineralization by soil microorganisms. Phosphate in fixed P pool may remain in soils for years without being consumed by plants in the soil. The inorganic phosphate compounds are more crystalline in their structure and less soluble than P compound available in active P pool (Pagliari et al., 2009).

2.3 Factors affecting P in soil

2.3.1 Temperature

Temperature can affect mineralization rate of P from the decomposition of organic matter. Organic matter decomposed and released the P faster in warm humid climates rather than in cool dry climates. Furthermore, P is released faster when soil is aerated (higher oxygen levels) compared to the saturated wet soils. Changes in temperature can affect P in soil as it can affect the P cycling by altered the chemical reaction kinetics and the rate of processes. High temperatures encourage organic matter decomposition. But when temperatures are too high or too low, they can restrict P uptake by the plant (Whitehead and Crossman, 2012).

2.3.2 Soil pH, type and texture

Soil type is generally determined by the texture of the soil, which is a measure proportions of the following three particles. Firstly, sand (classified as having a particle size between 0.05 mm and 2.0 mm), second, silt (0.05 mm to 0.002 mm and third, is clay <0.002 mm). Besides, pH and soil mineralogy, soil texture also can influence the P availability. For example, low-permeability in the soil profile can prevent the downward movement of P (Allen et al. 2006). It is because although P is generally considered less mobile than N, but P translocation can be significance even in fine-textured soils.

2.4 Phosphorus Sorption and Desorption

2.4.1 Sorption and Desorption

The whole reaction, adsorption plus penetration, is called sorption. Penetration of adsorbed P into the soil particles continues even in dry soil, albeit more slowly. When P is added to soils, P sorption will be occurring. Sorption of P refers to a rapid surface reaction and a slow reaction P on solid phase (mineral soil and organic compounds). It was initially continued by the rapid exothermic ligand exchange reaction that occurred with a reactive surface group (Bolland, Allen, & Barrow, 2003). Moreover, the sorption process is controlled by P concentration in the solution (intensity) and solid phase capability to add P into solution (capacity). When inorganic P is added to soil, these

reactions continue until a new equilibrium is reached. However, desorption is the release of adsorbed P from its bound state into the soil (Hongthanat, 2010).

2.4.2 Factors affecting Adsorption and Desorption of P

The adsorption of P is influenced by many factors that affect the charge on the surfaces of soil constituents and the form of phosphate in soil solution. The effect of soil pH result in adsorption or desorption of hydrogen ion at the surface for phosphate (Bolland et al., 2003). At lower pH, the adsorbed of hydrogen ion tend to have more positive charge on the surface of soil constituent. Conversely, at higher pH, desorption of hydrogen ion will make the charge more negative. Thus, phosphate ion is negatively charged and only can react with such surfaces if the affinity and concentration are large enough. The effect of the soil pH depends on the ion in the solution. Moreover, if the calcium ion dominates, make it easy for phosphate ion to react with negatives surfaces and decrease in P sorption with increasing pH.

2.4.3 Sorption and desorption isotherm

Phosphorus sorption and desorption isotherms have been widely used to characterize the P status and to establish phosphorus requirement in the soil (Anghinoni, Baligar, & Wright, 1996). These isotherms are used to determine the relative importance of the soil parameters in P retention and supply to plant roots. Phosphorus sorption studies

were conducted according to the standardized P sorption procedure recommended by the SERA-IEG 17 group (Graetz and Nair, 2009). One way to determine the soil capacity of P sorption is by developing a P sorption isotherm. Moreover, P sorption isotherms have been used to obtain information about the capacity and intensity aspects of P availability. Besides, desorption process in nutrient uptake models is determined by P sorption and desorption isotherm. An isotherm describes the amount of P on the solid phase or at equilibrium solution.

2.4.3.1 Langmuir, Freundlich

Langmuir isotherm

Langmuir equation was used to determine P sorption maxima (S_{max}) and bonding energies constant (k).

The Langmuir Equation can be written as:

$$C / S = C / S_{max} + 1 / K S_{max} \tag{2.1}$$

C = concentration of P in solution after 24-h equilibration, mg L⁻¹,

S = the amount of P sorbed on solid phase, mg kg⁻¹,

S_{max} = maximum P sorption capacity of soil, mg kg⁻¹

k = a constant related to the bonding energy, $L\ mg^{-1}$.

When C/S (y-axis) is plotted against C (x-axis), a line can be fit to the points by linear regression (Figure 1.2). The slope of the line is equal to $1/S_{max}$, Since the intercept is equal to $1/(kS_{max})$, S_{max} is substituted into this equation to solve for k .

Freundlich isotherm

Freundlich isotherm is commonly used to describe the sorption characteristics for the heterogeneous surface. The Freundlich adsorption coefficient or K_F are calculated as below:

$$A = K_F C^N \quad (2.2)$$

The Freundlich equation becomes a linear relationship when $N = 1$. Subsequently, the Freundlich equation (2.2) can be log-linearized and equation (2.3) can be obtained. Plotting of $\log C$ on the x-axis and $\log A$ on the y-axis showed a best-fit straight line with a slope of N , and $\log K_F$ is its intercept. The log linearized form of the equation is:

$$\text{Log}(A) = \text{Log}(K_F) + N \text{Log}(C) \quad (2.3)$$

2.5 Agricultural waste

2.5.1 Paddy husk

Rice (*Oryza sativa*) is grown on every continent except Antarctica and the extent of paddy cultivation covers about 1 percent of the earth's surface. More than half of the world's population depends on rice as a staple food and it ranks second to wheat in terms of cultivation area and production

Paddy husk is a by-product of rice processing, which can be used for compost production. The availability of paddy husk is mainly depend on the paddy production and the fraction of husk in paddy (Rodrigo & Perera, 2011). According to national news agency (BERNAMA, 2013), there are 200,000 ha idle land in Malaysia used for paddy plantation and thus will increase the paddy production into 30%.

Paddy husk is paddy residue preparation consist of rice milling and transportation for rice husk system boundaries. Rice husk lifecycle involves four main processes, which are paddy production, rice milling, transportation and electricity generation. Totally the effect resulted after paddy residue in power generation are much better compared to conventional type power generation (Shafie 2015).

2.6 Composting

Composts or other organic amendments can be applied to overcome P deficiency. However, appropriate management strategies must be implemented to increase P availability to plant roots, which must contact available P for uptake to occur. There are improvements to P availability that can be achieved through the use of organic matter like residues. The applications of plant biomass from species like tithonia (*Tithonia diversifolia*) can produce increases in soil available P and maize yields comparable to inorganic P inputs (Jama et al. 2000; Nziguheba et al. 1998).

The compost used as a source of organic matter consists of partly separated and subject to organic waste fermentation. The application of compost as a soil amendment in soil had resulted in the decrease of P fixation and increase P availability (Essoka, 2014). With the use of compost, the organic material lost from the soil in various ways is once again given to the soil and thereby losing the nutrients. Compost give beneficial effect as it can be improving and sustaining the soil quality and reduces the use of fertilizer and pesticide.

2.7 Mechanism of compost in influencing the degree of soil P sorption and desorption

There are many potential mechanisms by which compost can influencing the degree of soil P sorption and desorption (Guppy et al. 2005; Iyamuremye and Dick 1966; Oberson et al. 2006, 2011): (1) inducing ligand exchange (i.e., organic sorbates competing with P

for mineral binding sites); (2) buffering soil acidity ; (3) providing a large sink for exchangeable P and (4) stimulating biological cycling of P, chiefly via microbial drivers.

2.7.1 Ligand exchange

Guppy et al. (2005) argue that at realistic application rates, organic matter inputs such as manure increase available soil P due to the addition of P in organic matter rather than organomineral interaction such as ligand exchange. For example, mesocosm studies typically use low- weight organic acids as model organic matter in concentration of 3-5 of orders of magnitude greater in field experiments. Although, it may effectively compete with sorption sites, it can also increase the sorption of P to its chelated metal cations.

2.7.2 Buffering soil acidity

Phosphorus availability can be achieved through the application of organic amendment to the soil. Furthermore, ameliorating P fixation with organic matter is thought to reflect diseases in binding sites and/or exchangeable acidity as a result of organic matter complexation of exchangeable Al^{3+} and consumption of H^+ via decomposition processes of organic matter buffering (Stewart, 2016).

2.7.3 Large sink for exchangeable P

Sink- based soil tests measure P available from the soil to a passive sink, such as a plant root idealized by a positively charged surface such as iron- and/ or aluminium-impregnated or anion exchange surface in the form of membranes or resins. Analogous to the roots, as P is removed from the soil solution by adsorption to a sink, P not in the soil solution (e.g., weakly sorbed to mineral or organic matter) replenished the soil solution P by equilibrium processes (Qian & Schoenau, 2002). Because they measure exchangeable P, sink methods are considered to be a more realistic indication of soil P available for crop uptake.

2.7.4 Biological cycling of P

Biological cycling of P can play a significant role in provisioning plant-available P, in particular for weathered soils with high potential for fixation. Organic amendments can affect biological P cycling through microbial biomass P (MBP) and enzymes that hydrolyze organic P. An excellent overview of soil phosphates and their role in biological P cycling to increase its crop availability is provided by Nannipieri et al. (2011). The addition of compost produced from a mixture of coffee pulp, manure, gypsum, and PR to an Oxisol increased potential activities of soil enzymes with increased compost addition (10-80 g kg⁻¹ soil) after 28 days, including both acid and alkaline phosphomonoesterases, in tandem with increased microbial respiration (Oliveira and Ferreira, 2014).

CHAPTER 3

METHODOLOGY

3.1 Soil Sampling

The uncultivated soil in University Malaysia Kelantan Jeli Campus area was sampled at 0-20 cm using soil auger. After that, the soil samples were bulked together. The soil samples were air-dried. After drying, the soil samples were crushed manually using pestle and mortar and sieved through 0.2 mm. The sieved soil sample was kept in tight plastic bags.

3.2 Soil Analysis

3.2.1 Bulk Density Determination

The bulk densities at soil depth 0-2 cm were determined by coring method (Dixon & Wsniewski, 1995). The soils with coring were weighed and oven-dried for 24

hours at 150⁰ C. After 24 hours, coring with soil were weighed. The bulk densities of the soil were calculated using the formula described by Dixon & Wsniewski,1995 as follows:

$$\text{Bulk density (g cm}^{-3}\text{)} = \text{dry soil weighted (g)} / \text{soil volume (cm}^3\text{)} \quad (3.1)$$

3.2.2 Soil pH and Soil Electrical Conductivity (EC)

The pH of the soil sample was determined by Potentiometric method. It was measured to determine the degree of acidity or alkalinity in soil suspended in water. The soil pH and EC was determined by using pH meter and EC rate, respectively. It was done by using a ratio of 1:2:5 (soil and distilled water suspension) by using digital pH and EC meter. A 12.5 mL of distilled water was added with 5 g of air-dry soil into the beaker. The procedures were repeated for 3 samples. Next, the samples were shaken for 15 minutes at 180 rpm by using a shaker. After that, the samples were left for 24 hours before using a digital pH meter for pH determination and EC meter for EC determination.

3.2.3 Soil Texture Determination

The soil texture was determined by using hydrometer (Bouyoucos, 1962). A 50 g of soil sample was placed in blender cup. After that, 4 M of NaOH was added into the blender cup to adjust the soil pH to the pH of 10. Distilled water was filled into the blender cup within 10 cm of the top rim. Then, blender cup was placed on stirring machine and mixed for 15 minutes. After 15 minutes, the soil suspension was transferred into 1 L of measuring cylinder. Distilled water was added into measuring cylinder up to a volume of 1130 ml. The soil suspension was stirred by using the stirring rod. After 40 seconds, hydrometer was placed into the suspension. The reading on the hydrometer stem was recorded. Next, the hydrometer was removed from the measuring cylinder. The soil suspension was stirred and second reading of the hydrometer was recorded. The third reading was stirred and recorded after 2 hours. The calculations for soil texture was based on the formula as shown below:

$$\text{Percentage of silt + clay + sand} = 100\%$$

For 40 seconds reading:

$$\text{Percentage of silt + clay} = (a/50) \times 100\% = w$$

$$\text{Percentage of sand} = (100-w) \% = x$$

After 2 hours reading:

$$\text{Percentage of clay} = (b/50) \times 100\% = y$$

$$\text{By different: Percentage of silt} = w - y = z \tag{3.2}$$

3.2.3 Soil Total Organic Matter and Total C Determination

The soil total organic matter and total C was determined using the dry combustion method (Tan, 2015). Air-dry sample was placed in the oven and stored at 60 °C for 24 hours using the dry combustion method (Tan, 2005). The initial weight of the crucible was taken after the samples were cooled down. After that, the crucible with 5 g of the soil sample was weighed. The soil samples were ashed at the 300 °C to in the muffle furnace for 8 hours with temperature 550 °C. Lastly, the samples were cooling down and the weight of the samples after ash was recorded. The total organic matter and C are shown below:

Calculation of Total Organic Matter:

$$\frac{\text{Initial Weight of soil sample (g)} - \text{final weight of soil sample (g)}}{\text{Initial weight of soil sample (g)}} \times 100\% \quad (3.3)$$

Calculation of Total C:

$$\frac{\text{Initial Weight of soil sample (g)} - \text{final weight of soil sample (g)}}{\text{Initial weight of soil sample (g)}} \times 0.58 \quad (3.4)$$

3.2.5 Soil Exchangeable Acidity and Al

The soil exchangeable acidity and Al were determined using titration method (Rowel, 1994). A 10 g of soil and 30 mL of 1 M KCl was placed into a beaker and left for

24 hours. After 24 hours, the sample was filtered with Whatman Filter Paper No. 2 into the 100 mL volumetric flask and the volume was made up. Next, 50 mL of the soil extract was pipetted into 250 mL of conical flask. Then, 5 drops of phenolphthalein was added as indicator. The solution was titrated against 0.01 M NaOH until pink colour was developed. The soil exchangeable acidity was measured. The solution was titrated until it become colourless. The soil exchangeable of Al was measured. The soil exchangeable acidity and soil exchangeable of Al were calculated by using the following calculation:

$$\text{Exchangeable acidity (cmol kg}^{-1}\text{)} = \frac{[0.2 \times \text{Titrate volume of } 0.01 \text{ M NaOH} \times 10]}{\text{soil mass (g)}} \quad (3.5)$$

$$\text{Exchangeable Al (cmol kg}^{-1}\text{)} = \frac{[0.2 \times \text{Titrate volume of } 0.01 \text{ M HCl} \times 10]}{\text{soil mass (g)}} \quad (3.6)$$

3.2.6 Soil Extractable of K, Ca, Mg, Na, Cu, Zn, and Fe Determination

Soil extractable K, Ca, Mg, Na, Cu, Zn, and Fe were extracted using the Mehlich No. 1 Double Acid Method (Mehlich,1953). A 5 g of soil sample was weighed and placed into a 50 mL beaker. After that, a 25 mL of the extraction reagent was added and the solution was shaken for 10 minutes on a reciprocal shaker. Then, the supernatant was filtered into another beaker using Whatman Filter Paper No. 2 the extract collected. An Atomic Absorption Spectrometer (AAS) was calibrated and aspirated into the AAS and the reading was recorded. The soil exchangeable cation was calculated using the equation by Tan (2005) as shown below:

$$\text{Soil exchangeable cation (ppm)} = \text{AAS reading (ppm)} \times \frac{\text{Volume of extractant (mL)}}{\text{Weight of soil sample (g)}} \quad (3.7)$$

3.2.7 Soil Available P Determination

The soil available P was extracted using Mehlich No. 1 Double Acid Method (Mehlich, 1953). Firstly, 4 mL of concentrated HCL and 0.7 mL of concentrated H₂SO₄ were pipetted into a 1000 mL volumetric flask. A 5 g of sample was weighed and placed into a 50 mL beaker. Next, a 25 mL of the extraction reagent was added and the solution was shaken for 10 minutes on reciprocal shaker. After that, the supernatant was filtered into plastic vials using Whatman Filter Paper No. 2 and the P extract was collected. The solution was analysed by the molybdenum blue method (Murphy & Riley, 1962). The blue colour was analysed by UV spectrophotometer (Thermo Scientific Genesys 20) at 882 nm wavelength.

3.2.8 Soil Total N

The soil total N was determined by Kjeldhal Method (Bremner, 1965) and was divided into 3 parts, digestion, distillation and titration. For digestion, 0.5 g for soil were weighed and added into digestion tubes. Next, 5 ml of concentrated sulphuric acid (H₂SO₄) followed by 1g of Kjedaahl catalyst were added into the tubes. Then, the samples

were shaken and calibrated for 30 minutes. After that, the samples were heated at 400°C for 1 hour. The samples were allowed to cool down. A 30 ml of distilled water was added to the samples and the volume was made up to 100 ml when the solution was cooled. For distillation, 10 ml of sample from the digestion part were added in distillation tube. Then, 10 ml of 40% NaOH was added into the same tube and was distilled for 50 seconds. After that, the distillate was collected with another flask that contained 10 ml of 2% boric acid-indicator solution. The flask that contained 10 ml of 2% boric acid-indicator solution were distilled until it reached 20 ml. For titration, the 20 ml solution from the digestion part was titrated against 0.01 M sulphuric acid (H₂SO₄) until the colour change from green to purple.

3.3 Compost Production

The paddy husk composts were prepared from the mixture of paddy husks, goat manure, molasses as well as chicken feeds. The paddy husks were taken from a paddy field at Pasir Puteh, Kelantan, Malaysia. These paddy husks were later bulked, air-dried, and shredded. The sampling of goat manure was acquired from a dairy farm located in Kemahang, Kelantan. The composting process was carried out in an open space of research area in Universiti Malaysia Kelantan, Jeli Campus, Malaysia. The 3 composting containers with 435 mm (height) x 425 mm (base) were prepared for composting purposes. The 12 holes were made with a hole size 0.5 cm diameter, respectively. The paddy husk compost was made by the mixture of 80% shredded paddy husk + 10% of goat manure slurry + 5% of chicken feed + 5% of molasses in composting container.

The preparation of paddy husk compost was referred on the formulation ratio done by Ch'ng et al. (2013). The composting processes were repeated for 3 replications and it took 60 days for maturation.

3.3.1 Compost characterisation

The paddy husk compost was analysed for pH, EC, total organic matter, total C, total N, P, K, Ca, Mg, Na, Zn, Cu, and Fe. The procedure for analysing PH, EC, total organic matter, and total C are similar to the procedures described in the previous sections.

3.3.2 Determination of Total P and K in compost

Total P and K in the compost were determine by using the single dry ashing (Jones, Uwah, Iren, & 1991). Firstly, 1 g of ground and dry sample was weighed and placed into crucible. The sample was placed and ashed in a muffle furnace at 300 °C for one hour. After one hour, the temperature of the muffle furnace was increased up to 520 °C and ashed it for another 5 hours. The sample was cooled. After that, a few drops of distilled water was added and 2 mL of concentrated HCl were added to the sample. The sample was evaporated to dryness in the fume chamber by using a hot plate. Next, 10 mL of 20% HNO₃ was added to the sample and allowed it to heat for further one hour. The

sample was filtered to pass through Whatman Filter paper No. 2 into 100 mL volumetric flask and the volume was made up. For the K determination, the sample was transferred into AAS and the absorbance reading was recorded. The molybdenum blue method (Murphy & Riley, 1962) was used to determine the total P in the compost. The blue colour was analysed using UV spectrophotometer at 882 nm wavelength.

3.4 Soil P sorption and Desorption Determination

The soil P sorption and desorption were carried out in the Physics laboratory at University Malaysia Kelantan. The experimental design used in this study was completely randomized design (CRD) with three replications. The treatments evaluated in this study are as follows:

Table 3.1 List of treatment of P sorption and desorption

Treatment	Description
T0	Soil only
T1	Soil + 5t ha ⁻¹ compost
T2	Soil + 10t ha ⁻¹ compost
T3	Soil + 15 t ha ⁻¹ compost
T4	Soil + 20t ha ⁻¹ compost

P sorption determination was based on the standard procedure described by Graetz Nair (2009) with some modification on the concentration of KH_2PO_4 used. The samples contained 2 g of soil only or samples of soil with organic amendment and weighed into 100 mL centrifuge bottles. After that, 20 mL of 0.001 M KCl containing of 0, 5, 10, 15, and 20, 25 and 30 mg L^{-1} P as KH_2PO_4 was added into each of centrifuge bottles. The sample was replicated for three times. Then, the sample were shaken for 24 hours at 180 rpm for 10 minutes. The supernatant with P were collected and determined by using the molybdenum blue method (Murphy & Riley, 1962). The sorbed P was determined as the difference between initial amount of P added and the amount in the equilibrium solution. The P sorption data was fitted in either Langmuir because of the degree of fitness into the isotherm. After P sorption analysis, the soil cake was allowed to air dry for 24 hours and the soil cake in centrifuge bottle were dissolved using Vortex and the samples were shaken at 180 rpm for 10 min. A total of 20 ml of 1M NH_4Cl was added after 24 hours and the soil cake in centrifuge bottle were dissolved using Vortex and the samples were shaken at 180 rpm for 10 min. Then, the samples were centrifuged at 10,000 rpm for 10 min for P desorption determination. The supernatant with P was collected and determined using the molybdenum method (Murphy & Riley, 1962).

3.5 Compost pH Buffering Capacity Determination

Compost pH buffering capacity was determined by using the method described by Costello and Sullivan (2014). The experiment was carried out after the soil P sorption and desorption experiment. A 5 g of compost was weighed separately into 100 ml beaker

by using digital weighing balance. After that, 0, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 ml of 0.25 M H₂SO₄ was added with sufficient water to make up the total liquid addition to 50 ml (1: 10, compost: water). Then, the samples were stirred for 10 second after acid addition and then stirred again for another 10 second for pH measurement using a digital pH meter after 72 hours (Ch'ng, Ahmed, & Majid, 2016). The compost pH buffering capacity or the quantity of acidity needed to reduce pH by one unit was calculated as the negative reciprocal of the slope of the linear regression, soil or soil mixture pH (y-axis) versus acid addition (X- axis).

Compost pH buffering capacity (mol H⁺ kg⁻¹ soil) = -(1/slope)

Where the slope is equal to the linear regression line for each sample.

3.6 Data Analysis

The comparison of the treatment was measure using the Turkey's test while analysis of variance (ANOVA) was used to detect the treatment effects. The Statistical Analysis System (SAS) also was used to analyse the data collected in this research.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characteristic of soil

The selected physico-chemical properties of Rengam series soil (Typic Paleudult, clayey, kaolinitic, isohyperthermic) soil are used in Table 4.1. The soil bulk density was 0.32 g cm^{-3} (Table 4.1). However, the bulk density obtained in this study was relatively lower compared to the soil bulk density of secondary forest in the study of Ch'ng et al. (2015). The low soil bulk density of this study was influenced by the soil aggregates, porosity and other soil constituents that might be causing to be less compact and loose. Besides, the lower value of bulk density of this study was also due to high organic matter content which can also result in low bulk density in the soil. The soil texture of the soil samples was a sandy clay loam. Sandy clay loam soils have a high concentration of sand that gives a very gritty texture. Furthermore, the soil was acidic with pH value of 5.80 and had lower concentration of available P (1.62 ppm). The low concentration of P was due to maximum sorption of P occurs in the acidic soil (Simonsson et al., 2018).

The soil organic matter and soil total C of the soil sample collected from this study were 5.50% and 3.19%, respectively. The value of soil organic matter and soil total C reported in this study was relatively lower compared to the data reported by Ch'ng et al. (2016). The soil fertility is mainly determined by the management of soil organic matter including soil microbial activity, soil nutrient management and also soil organic C. The exchangeable K, Ca, Mg, Na, Fe, Cu, and Zn were 387.00, 881.33, 5217.33, 3131.47, 559.60, 0.68, 44.71 and 3.83 ppm respectively. The concentration of Al and Fe were highly relative due to low soil pH and the ions occupied most of cation exchange site (Table 4.1). The high concentration of Al³⁺ gave potential to P fixation due to dissolvment of P anions in the soil. In acidic soil, Al results to toxicity to plants and deficiency of nutrient such as P, Ca, and Mg.

Table 4.1. Selected physico-chemical properties of Rengam series soil.

Property	Value obtained
Bulk density (g cm ⁻³)	0.32
Soil texture	Sand: 54%
	Clay: 24%
	Silt: 22%
	=> Sandy clay loam (<i>Typic Paleudult</i> , clayey, kaolinitic, isohyperthermic)
pH (Water)	5.80
Electrical conductivity (dSm ⁻¹)	0.03
Total organic matter (%)	5.50
Total C (%)	3.19
Available P (ppm)	1.62
Total N (%)	0.01
Exchangeable acidity (cmol _c kg ⁻¹)	0.73
Exchangeable Al (cmol _c kg ⁻¹)	3.83
Exchangeable K (ppm)	387.00
Exchangeable Ca (ppm)	881.33
Exchangeable Mg (ppm)	5,217.33
Exchangeable Na (ppm)	3,131.47
Exchangeable Fe (ppm)	559.60
Exchangeable Cu (ppm)	0.68
Exchangeable Zn (ppm)	44.71

4.2 Characteristic of paddy husk compost

The selected chemical properties of the paddy husk compost are shown in Table 4.2. The C/N and C/P ratio of paddy husk compost were 16.66 and 72.73, respectively. These ratios suggest net mineralization of the compost (Ch'ng et al., 2016; Ch'ng, 2018). The paddy husk compost also has relatively high concentration of exchangeable cations especially K (8.97%), Ca (1.03%), Mg (0.55%) and Na (34.60%) (Table 4.2). The neutral pH of the compost was 7.62. Thus, the application of compost can increase the soil pH and improved the P availability in the soil.

Table 4.2. Selected chemical properties of the paddy husk compost

Property	Rice husk compost
pH	7.62
Electrical conductivity (dSm ⁻¹)	1.85
Total organic matter (%)	79.00
Total carbon (%)	45.82
Total nitrogen (%)	2.75
Total phosphorus (%)	0.63
C/N ratio	16.66
C/P ratio	72.73
Total K ⁺ (%)	8.97
Total Ca ²⁺ (%)	1.03
Total Mg ²⁺ (%)	0.55
Total Na ²⁺ (%)	34.60
Total Zn ²⁺ (µg/g)	211.00
Total Cu ²⁺ (µg/g)	6.40
Total Fe ²⁺ (µg/g)	3,006.00
Humic acid (HA) (%)	2.80
Ash content (%)	10.36

4.3 Sorption and Desorption

Figure 4.1 shows soil P sorption data of each sample described by the Langmuir equation ($R^2 \geq 0.20$). Based on Figure 4.1, the P sorption isotherm curves shows that the amount of P sorbed into soil amendment increased by the initial P concentrations in all samples with or without organic amendments treatments (T0-T4). From this study, at lower concentration of P ($< 20 \text{ mg L}^{-1}$), the sorbed P was significantly different with the organic amendments (T1, T2 and T3). However, when the P concentration increased ($> 20 \text{ mg/L}$), the sorbed P remain constant and was less sensitives (Ch'ng et al., 2016) to the paddy husk compost. P sorption capacity of soils were depend on type of organic amendment applied and the available soil type (Yu et al., 2013). This study suggests that the influences of paddy husk compost on P sorption is more pronounced at low P loadings. Furthermore, the reaction of P sorption to the organic amendment seem highly depend on the initial level of soil acidity (Ch'ng et al., 2016). As shown in Figure 4.1, the adsorbed P decreased when paddy husk compost (T4) was applied, thus causing increased in soil pH. However, the sorption increased at the treatment of paddy husk compost in (T3).

The Langmuir sorption maximum (S_m) significantly decreased from 0.39 mg kg^{-1} (T3) to -2.83 mg kg^{-1} (Table 4.3) when paddy husk compost added with soil in various ratio. The decrease in P sorption is more pronounced at higher rate of compost application. Thus, P sorption decreased due to the precipitation of Al and Fe at the highly negative charged cause soil pH increased. Soil pH significantly influenced the values of the Langmuir equation's parameters. A related study has revealed that the sorption of P decrease due to precipitation of exchangeable of Al and extractable of Fe at the highly negatively charged humic substances functional group surfaces of the organic

amendments used in this study (Ch'ng et al., 2016). Upon application of organic amendment causes the soil pH increases. Due to higher pH, the soil surfaces become high in negative charged as anion repulsion increased thus, the P sorption decreased (Murphy and Steven, 2010). HPO_4^{2-} is anion that dominant in higher pH and very favour to be attracted by soil colloids (Haynes et al., 2000).

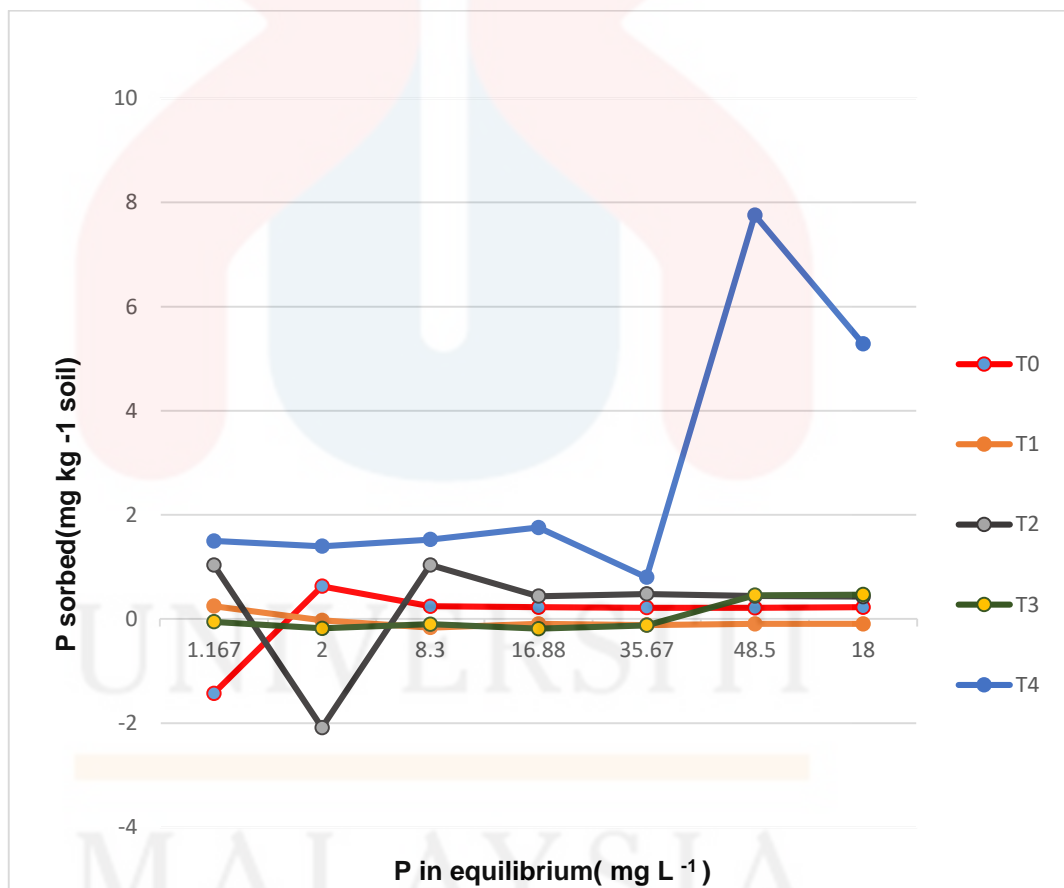


Figure 4.1: Effects of treatments on the soil phosphorus isotherms

Based on the sorption isotherm curves, it shows that the paddy husk compost ratios are influenced the sorption of P in the amended soil. The Langmuir sorption maximum (S_m) and maximum buffering capacity of soil for P increased as compost content in the soil increased. This is because the organic matter, CEC and the availability of P in the paddy husk compost were high (Table 4.2). This study shows that the CEC of the compost is higher than the anion exchange capacity (Mukherjee, Zimmerman & Harris, 2011). This due to the increase surface negatively charged by soil organic matter and humus in compost. Besides, the compost surfaces are characterized by negatively charged functional groups which attract cation instead of anion such as PO_4^{3-} in the applied of soil.

Table 4.3: Phosphorus sorption parameters of the isotherms describe by Langmuir equation

Treatment	Estimated by Langmuir Equation			
	KL (L mg ⁻¹)	Sm (mg kg ⁻¹)	Maximum P Buffering Capacity (L kg ⁻¹)	R ²
T0	-0.75	0.21	-0.16	0.15
T1	-0.83	-0.09	0.073	0.13
T2	-0.92	0.39	-0.36	0.042
T3	-0.06	0.39	-0.02	0.084
T4	-0.07	0.42	0.18	0.27

As shown in Figure 4.2, the P desorbed increased when applied with paddy husk compost at T3 and T4. This is because of the significant differences in binding energy as the values (K_L) decreased from -0.748 L mg^{-1} in the soil only (T0) to $-0.0651 \text{ L mg}^{-1}$ in T4 (Table 4.3). It is suggested that only those ion compete with phosphate ion at ligand exchange sites may resulting in decreased P adsorption (Shailaja & Sahrawat, 1990). The decreased in binding energy related to the increased in pH due to the application of organic amendment and resulted higher P desorption (Ch'ng et al., 2016). Figure 4.2 shows that the concentration of P at 30 ppm are more effective in P desorption. The sorbed of P are significantly increase as the concentration of P increase.

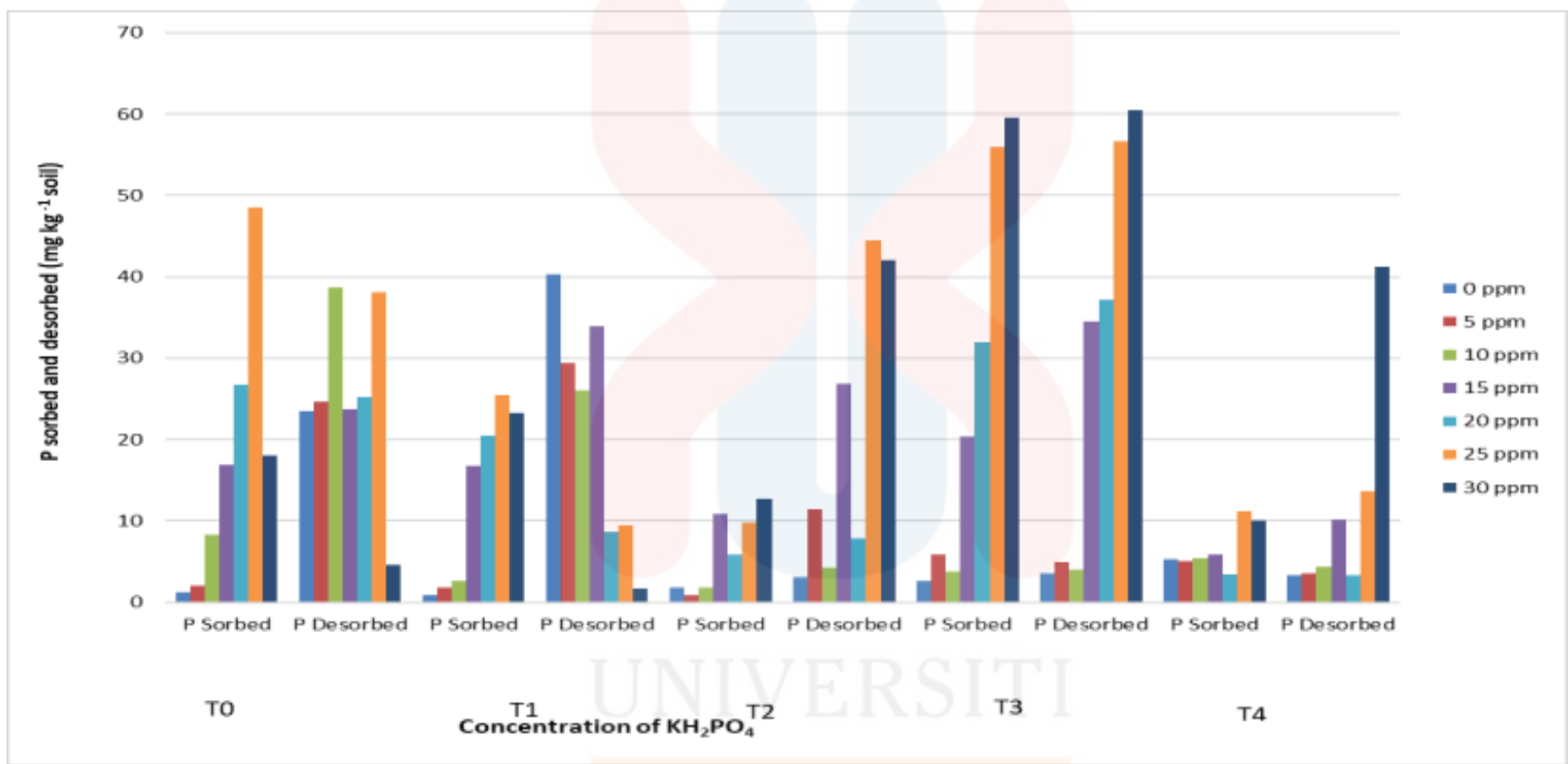


Figure 4.2 Effects of treatments on the amount of P sorbed and desorbed on the soils

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4.4 Compost Buffering Capacity

The response of paddy husk compost to acidification is depend on the compost pH buffering capacity which give ability for the compost to change the pH (Costello, Sullivan, 2014). The response of paddy husk compost pH to dilute acid addition was fit with linear regression function (Figure 4.3). The linear regression function shows that paddy husk compost had high buffering capacity. High pH buffering capacities indicates relatively high contents of cation (Ca, Mg, and K) concentrations. High pH buffering capacities can minimize the increasing pH compost that will reduce the availability of P due to increase of Al and Fe concentration (Ch'ng et al., 2016). Moreover, acidic soil with amended compost significantly increase the soil pH and soil EC because organic matter had large amount of negative charge which can give strong reaction with basic organic molecules (Latifah, Ahmed, & Majid, 2017).

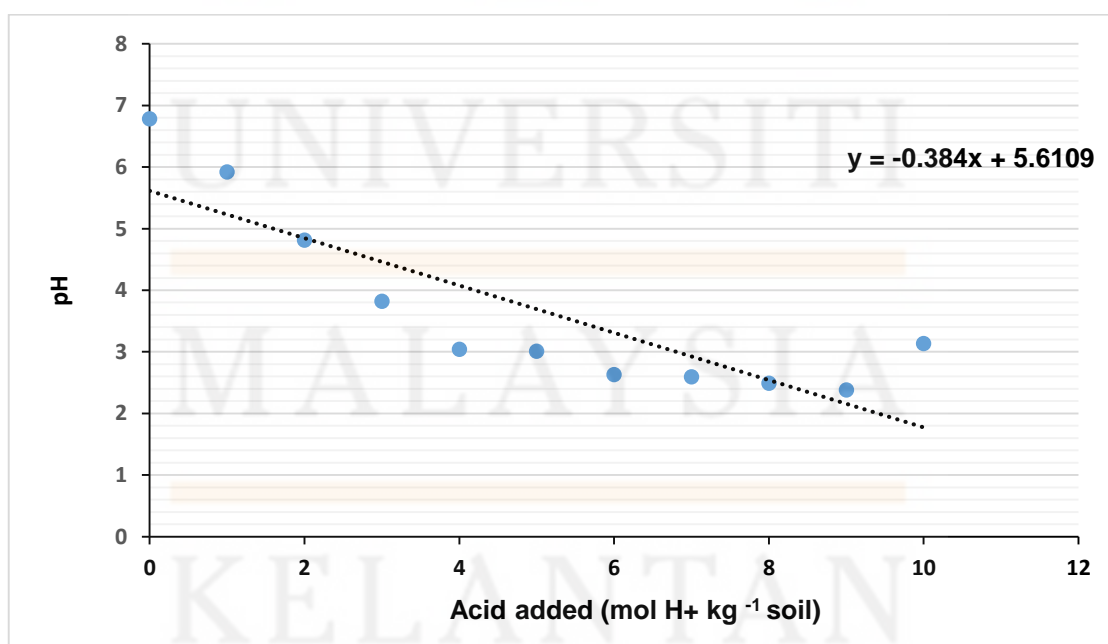


Figure 4.3: compost pH buffering capacity of paddy husk compost

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

This study provided information about the assessment of phosphorus sorption and desorption characteristics of tropical acid soil applied with paddy husk compost at the Universiti Malaysia Kelantan at Jeli Campus Area. As the results shown, the application of paddy husk compost improved the availability of phosphorus by changing the soil sorption and desorption capacity, and pH buffering capacity. From the results, initial P concentration of $< 20 \text{ mg L}^{-1}$, sorbed P increased with the amendment of paddy husk compost while more Phosphorus desorbed with increasing P application rates (20 to 30 mg L^{-1}). The decreases in P sorption in this study was due to the exchangeable of Aluminium and extractable iron at the highly negatively charged humic substances functional group surfaces of the organic amendment. The sorption of phosphorus was enhanced by increase of soil pH due to application of organic amendments. In this study, it is important to understand the fundamental aspect of the reaction between compost and soil as well as the mechanism of the increases in soil P availability. In the future study, it is recommended that to use paddy husk ashed instead of using paddy husk compost as organic amendment in this study.

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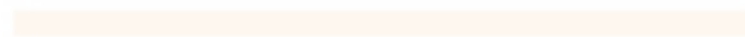
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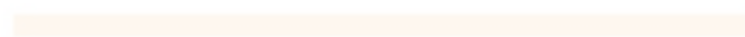
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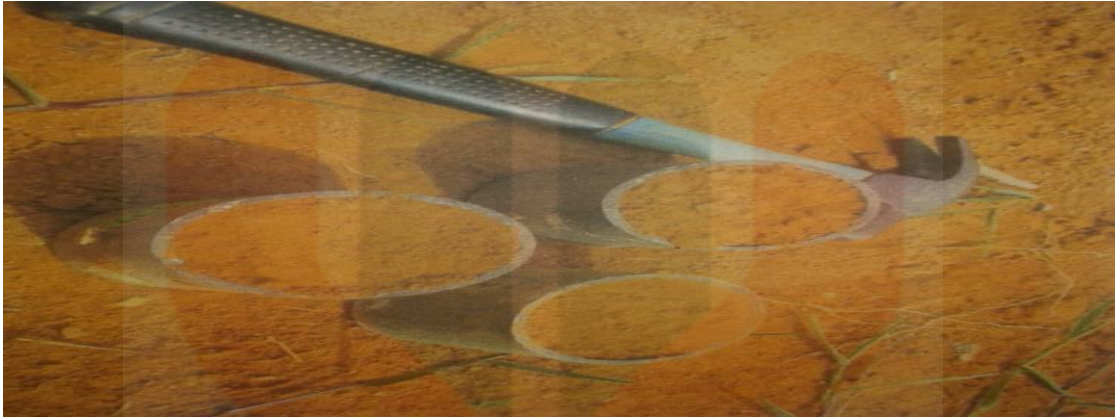
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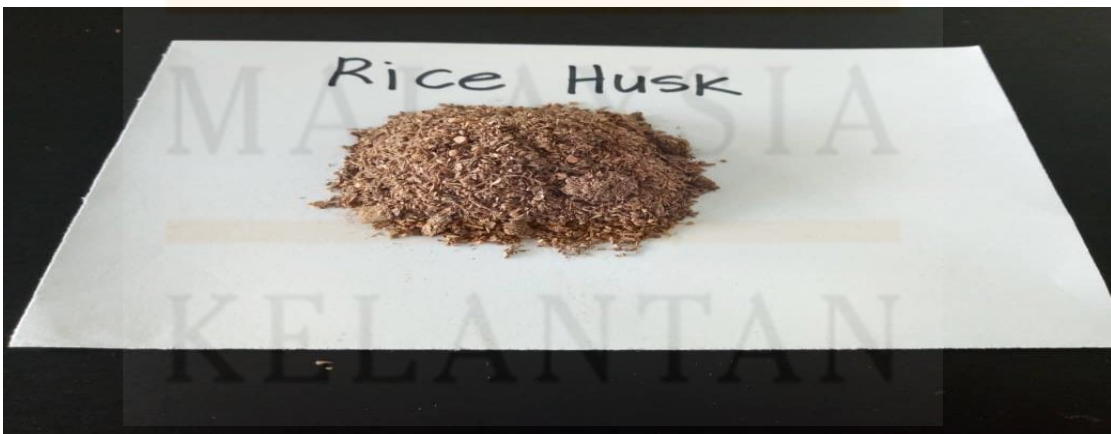
APPENDICES



Appendix A: The appendix A shows soil with coring for soil analysis laboratory.



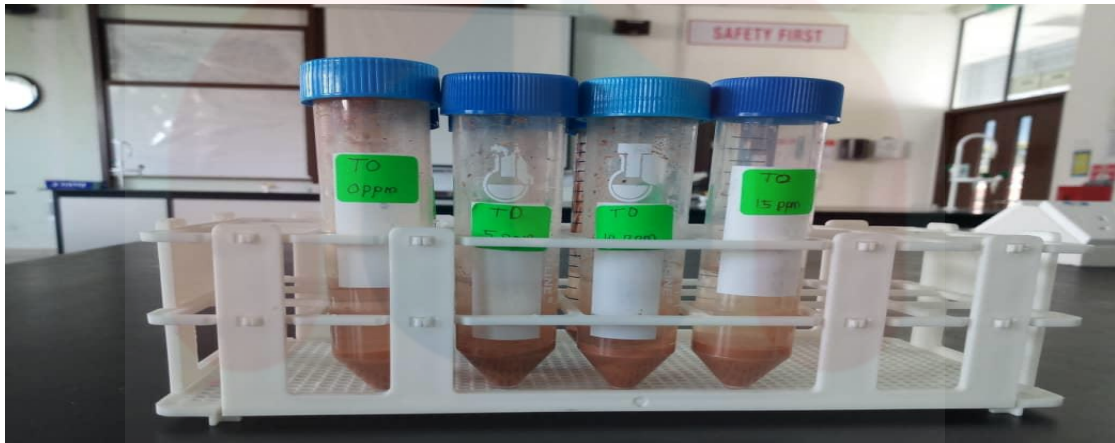
Appendix B: The appendix B shows oven-dried soil bulk for determination of bulk density.



Appendix C: The appendix C shows paddy husk compost.



Appendix D: The appendix D shows the preparation of soil with compost.



Appendix E: The appendix E shows samples of soil with organic amendment.



Appendix F: The appendix F shows supernatant with P that were collected in determination of soil sorption and desorption of P.