

Improving Potassium (K) Retention in Acidic Soil by Amending Muriate of Potash Fertilizer with Rice Straw Biochar

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A thesis submitted in fulfilment of the requirements for the

degree of Bachelor of Applied Science (Agrotechnology) with

Honours

Faculty of Agro Based Industry

Universiti Malaysia Kelantan

2022

DECLARATION

I hereby declare that the work embodied in here is the result of my own research except for the excerpt as cited in the references. FIAT

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ACKNOWLEDGMENTS

Alhamdulillah, all praise to Allah for His strength and grace in completing this thesis. First of all, I would like to thank my supervisor, Dr Ch'ng Huck Ywih for providing me with the right guidance, in-depth advice and warmth throughout my research. His help, support and suggestions throughout the trial and lab work has contributed to the success of the research.

In addition, I would also like to thank the Dean, Deputy Dean and all the faculty members of the Faculty of Agriculture Industry (FIAT) who participated in providing assistance and support to my post-graduate affairs. My appreciation is also given to all lab assistants and staff.

Sincerely thank all my friends for their concern, moral support and spiritual encouragement during the research. I am so grateful for the people who matter most to me, my beloved parents; Mr. Mohd Zamri Bin Khamis and Mrs. Wan Nilam Binti Wan Ibrahim, and to my family for endless love, encouragement, support and prayers in every form for so long. Without them, this work would not have been possible. Finally, I also thank those who indirectly contributed to this research. All your kindness and effort are greatly appreciated.



Meningkatkan Pengekalan Kalium (K) dalam Tanah Berasid dengan Meminda Muriate Baja Kalium dengan Biochar Jerami Padi.

ABSTRAK

Kalium (K) dalam tanah asid tropika terdedah kepada larut lesap yang meluas semasa hujan lebat disebabkan oleh mobiliti K yang tinggi dalam tanah. Oleh kerana kapasiti pengekalan yang rendah dan ketersediaan K dalam tanah, petani cenderung untuk menggunakan baja K yang berlebihan untuk mencapai hasil yang tinggi dan menepukan K dalam tanah. Objektif kajian ini adalah (1) Untuk mengambil sampel dan mencirikan sifat fizik-kimia terpilih bagi sampel tanah yang akan digunakan dalam larut lesap makmal kajian Kalium (K) (2) Mencirikan sifat kimia terpilih biochar jerami padi (3) Untuk tentukan kesan pindaan MOP dengan biochar jerami padi pada kadar yang berbeza terhadap pengekalan K dalam tanah dan sifat kimia tanah yang terpilih. Satu eksperimen larut lesap untuk menilai kesan baja K yang diubah pada kadar biochar jerami padi yang berbeza ke at<mark>as larut les</mark>ap K dan pengekalannya di dalam t<mark>anah, telah</mark> dijalankan selama 30 hari. Air larut resap dikumpul setiap 3 hari dan dianalisis untuk kandungan K. Sampel tanah dikumpul selepas kajian larut lesap dihentikan untuk penentuan jumlah K yang tersimpan di dalam tanah selepas proses larut lesap berlaku. Rawatan dengan aplikasi biochar jerami padi didapati dapat meningkatkan pengekalan K dalam tanah. Kadar penggunaan 15-20 t/ha biochar jerami padi didapati memberikan pengekalan K yang lebih baik di dalam tanah. Rawatan dengan penggunaan jerami padi juga turut menurunkan kerasidan dengan meningkatkan nilai pH tanah untuk menjadi neutral. Ini menunjukkan potensi biochar jerami padi dalam mengubah sifat kimia tanah berasid dan mengurangkan larut lesap K tanah selepas penggunaan baja MOP.

Kata kunci: Biochar jerami padi, baja bukan organik, Kalium (K⁺), larut lesap.

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ABSTRACT

Potassium (K) in the tropical acid soil is prone to extensive leaching during intense rainfall due to the high mobility of K in soil. Due to the low retention capacity and K availability in the soil, farmers tend to apply excessive K fertilizer to attain higher yield and saturate K in the soil. The objective of this study were (1) To sample and characterize selected physic-chemical properties of soil sample to be used in laboratory leaching of Potassium (K) study (2) To characterize the selected chemical properties of rice straw biochar (3) To determine the effect of amending MOP with rice straw biochar at different rates on K retention in soil and selected chemical properties of soil. A leaching experiment to assess the effect of K fertilizer amended with difference rates of rice straw biochar on the K leaching and retention in soil, was carried out for 30 days. Leachate was collected every 3 days and analysed for K content. Soil sample were collected after the leaching study stopped to determine the amount of K retained in the soil after leaching. Treatments with rice straw biochar application were able to improve the K retention in the soil. Application rate of 15-20 t/ha of rice straw biochar was found to provided better retention K in the soil. The treatment with rice straw application also decreased to the soil acidic by raising the soil pH to clear neutral. The indicates the potential of rice straw biochar in amending the chemical properties of acidic soil and reduce the K leaching for the soil after application of MOP fertilizer.

Keywords: Rice straw biochar, inorganic fertilizer, Potassium (K⁺), leaching.



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LIST OF ABBREVIATION AND SYMBOLS

К	Potassium	
N	Nitrogen	
Р	Phosphorus	
ОМ	Organic Matter	
С	Carbon	
Ca	Calcium	
Mg	Magnesium	
Na	Sodium	
NaOH	Sodium hydroxide	
H2SO4	Sulphuric acid	
HCI	Hydrochloric acid	
pH	Potential of hydrogen	
EC	Electrical Conductivity	
МОР	Muriate of Potash	
ATP	Adenosine triphosphate	
FAO	Food and Agriculture Organization of United Nation	
MARDI	Malaysian Agricultural Research and Development	
	Institute	
SPSS	Statistical Package for Social Science	
ANOVA	Analysis of Variance	

CHAPTER 1

INTRODUCTION

1.1 Research Background

1.1.1 Background

Potassium (K) is an important group of macronutrients for plant growth and development. K is involved in numerous physiologies in living plant cells such as electrical neutralization, enzyme activation, and membrane potential maintenance (Wang, & Wu, 2017). Moreover, in the soil is divided into three different categories. The first category of K is dissolved in water, the second K is absorbed into clay particles and organic matter and the third is K trapped in the crystal structure of mica and feldspar. Dissolved potassium is the only direct fraction available to plants (Rawat, Sanwal & Saxena, 2016). Besides, K also functions as a physiology associated with biotic and abiotic resistance such as disease, pest drought and salinity in the soil (Wang et al., 2013).

In addition, K is a very important cation in all organisms which affects the production of plants and ecosystems.

Muriate of potash (MOP) is the most popular potassium fertilizer (Meena 2016). The MOP is a fertilizer containing potassium chloride, where can applied directly to the soil. The MOP is characterized as a K fertilizer that is easily soluble in soil moisture. In addition, MOP is an important plant nutrient a and variety of salts containing K in a form that plants can easily absorb. Therefore, potash fertilizers have become an increasingly important input for the growing population demand for food and fibre.

Potassium leaching rates usually depend on the amount of K that can be exchanged in the soil, largely reflecting the level of input K and the excess K produced (Muller & Isselstein, 2007). Besides, the leaching K rate can also be explained by crop yield and nutrient balance in productive sandy soils and that the leaching K level can be controlled by nutrient input. However, soil concentrations of these nutrients have different concentrations ranging from 0.04% to 3.0% (Sparks, 2000). The maximum particle size (MOPA) fertilizer MOP has always shown a lower yield response compared to controls from various crops. Hence, the K balances rate requires an accurate determination of the K leaching loss (Askegaard, Eriksen & Johnston, 2004). According to a study by (Malavolta, 1985), soil type has an impact on K leaching. Because the amount of K lost in the end is determined by the available K in the soil which is influenced by the soil texture. Study by Cao et al. (2018), showed that biochar is a substance that could reduce fertilizer leaching.

Biochar is basically charcoal used to isolate carbon and improve soil fertility. The function of biochar depends on the pyrolysis temperature and can increase with the increase of temperature from 500 to 600 °C (Andrenelli et al., 2016). Biochar is a porous carbon solid comprises of the thermochemical conversion of organic matter in an oxygen-

deficient environment with physicochemical features that make it appropriate for safe and long-term carbon storage. Next, various types of biochar can be found which it may contain animal waste, plant waste and sewage that being used to provide useful biochar (Ahmad et al., 2014). Hence, there are many agricultural wastes that use low cost raw materials to produce biochar. Biochar benefits the environment through amending potential strategy for nutrient pollution, improved soil properties, climate change mitigation, waste management, and energy production (Lehmann and Joseph, 2009). Rice straw as a most important crop leftovers show its potential to fix heavy metals from water waste (Jusoh, Manaf & Latiff, 2013). Then, biochar can increase nutrient availability, carbon absorption and soil fertility (Tian et al., 2016). The importance of biochar in agriculture also leads to a steady increase in production and has many benefits to the environment for a long period of time. Moreover, application in biochar can increase the crop yield by 10% by increasing nutrient retention and soil pH (Jeffery et al., 2016).

1.1.2 Problem statement

Although there is several research and studies that have been carried out to reduce 1.5 MOP by using excess application in order to saturate the K in soil. Usage of a few types of micronutrient such as copper and zinc were also reported. However, there is a lack of research on amending MOP fertilizer with rice straw biochar to reduce the leaching of K being conducted.

1.1.3 Research question

Would amending MOP with rice straw biochar will reduce the leaching of K from the soil and thus improve the K retention in the soil?

1.1.4 Objective

The objectives of this study were to:

- 1) To sample and characterize selected physic-chemical properties of soil sample to be used in laboratory leaching of Potassium (K) study.
- 2) To characterize the selected chemical properties of rice straw biochar.
- 3) To determine the effect of amending MOP with rice straw biochar at different rates on K retention in soil and selected chemical properties of soil.

1.1.5 Scope of study

This study focuses on assessing the effect of amending MOP with rice straw biochar at different rates on reducing K, leading and improving the selected chemical properties of an acidic soil.

1.1.6 Significance of study

The world potash market is characterized by a limited number of producers supplying high concentration products in all country. The cost of K in the form of improve the MOP has remained high. To meet the rising demand for plant nutrition, the global production of potassium fertilizer has increased dramatically in the last century. However, other factors such as changing input prices can affect fertilizer prices. The approval will help to reduce the amount of MOP fertilizer used and improving its usage in Malaysia. It may also improve the crop production in the country by solving the serious K leaching problem in soil.

CHAPTER 2

LITERATURE REVIEW

2.1 Potassium

Generally, potassium is the major nutrients required by the plant's growth and reproduction. Potassium is thought to come from the disintegration of potassium-bearing minerals in nature. Soil potassium is stated to exit in non-accessible and readily available forms to crops, depending on their availability to plants. Though the amount of potassium in mineral form is estimated to range from 5000 to 25000 ppm, the amount readily available to crops only ranges from 1 to 10 ppm (Tisdale, Nelson & Beaton, 1985).



2.1.1 Introduction of potassium (K)

One of the most important elements for life on Earth is potassium (K). It is crucial for flora and fauna as well as soil need. Minerals like feldspars, particularly orthoclase (pink granite, Mohs hardness, and moonstone), and micas are the main sources of potassium in the soil which release this element in the course of their weathering. Potassium is made available to plants through this process. Furthermore, potassium can be present in the soil in three different ways in the exchangeable ion K in the soil solution, absorbed or released from the surfaces of clay particles and in organic matter (Hillel, 2008). However, some layered-aluminosilicate clay minerals, may be able to adsorb potassium not only on their exterior surfaces but also within their layered crystal lattices immobilized or fixing potassium ions.

2.1.2 Potassium cycle in soil

Potassium is a macronutrient that plants absorb in significant amounts (Sparks and Huang, 1985). The minerals feldspars and micas contain most of the K. Exchange is defined as the equilibrium maintained between soil solution K and exchangeable K throughout the fixation and release process. Instead of microbial activity, soil cation exchange and mineral weathering possess its action in the soil. As shown in the K cycle (Figure 2.1.2) K is in perpetual transformation. When K leaves the soil system, it does not form gases that could be lost to the atmosphere, and it does not cause any off-site environmental issues. A lot of plant functions, such as carbohydrate metabolism, enzyme activation, osmotic control, and protein synthesis, need potassium (Rath & Rousk, 2015). Despite the fact that K plays a variety of roles in plant and animal nutrition, it is not found in the structures of organic compounds. Instead, potassium stays in solution in the cells as an ionic form or serves as a cellular enzyme activator. Photosynthesis, fixation, starch production, and sugar transmission always required by potassium. Furthermore, K absorption by plants is usually lower than K released from the exchange pool. Thus, the K concentration in certain soils is a factor that limits plant growth. (Nieves-Cordones et al., 2016).







2.1.3 Classification of potassium

Potassium (K) is one of 17 nutrient groups needed by plants to develop and reproduction (Mikkelsen & Roberts, 2021). Nitrogen. Phosphorus, and potassium it is a macronutrient (K). The potash is defined as K₂O and is used to represent the concentration of potassium containing fertilisers such as muriate potash (KCI), potassium sulphate (K₂SO₄), potassium double sulphate and magnesium (K₂SO₄ 2MgSO₄), and potassium nitrate (KNO₃). Potassium is the eighth most common element on Earth comprise about 2.1% of the Earth's crust but it is a highly reactive element and does not occur freely in nature. Sir Humphry Davy extracted metallic potassium from molten caustic potash (KOH) for the first time in 1807. The minerals sylvine (KCl), carnallite (KCl, MgCl₂.6H₂O), langbeinite (K₂Mg₂(SO₄) ₃), and polyhalite (K₂Ca₂Mg (SO₄)₄.2H₂O) all contain potassium. Minerals are frequently found in old lakes and on the seabed. Potassium nitrate (KNO₃) known as saltpetre or nitre is a mineral that used in fertilizers and other products (Joshi et al., 2014).

2.1.4 Importance of potassium in soil plant system

Potassium forms many important compounds. Potassium (K) is an application plays key part in improving yield of crop. Besides, potassium is one of the main nutrients needed by all crops for growth. The good potassium nutrition is vital to consistently improve crop productivity. Potassium required for plant growth and is involved in a number of metabolic activities, including protein synthesis, photosynthesis, enzyme activation, water and nutrient transport through the xylem, and starch synthesis (Meena et al., 2016). Potassium often aids in the adaptation of plants to winter hardiness, fungal diseases, and insect pests. Potassium is also important because it helps in the control of stomatal opening and closing.

Stomata serve as gas exchanger allow to help in maintaining a healthy water balance as potassium ion pumped for supporting this process. Adequate potassium (K) content is also can increase phenol concentration, which plays important role in crop resilience (Prasad, 2010). Plants with slight of contain potassium can affect growth roots, grow slowly, produce small seeds, and provide less yields. Thus, the concentrations of soluble potassium in soil are usually low, and more than 90% of potassium in the soil exits in the form of insoluble silicate (Meena et al., 2016).

2.1.5 Problem of potassium fertilizer

The problem of potassium fertilizer often happen is caused due to a leaching loss. Most of potassium is a mobile ion in soils and mostly lost from the soil by leaching, affecting the efficiency of the fertilizers applied (Quemener, 1986). However, leaching losses can be described when K input exceeds soil retention capacity and crop demand, in the presence of drainage (Goulding et. al., 2021). Besides, potassium can also be lost in surface runoff. Generally, losses in surface runoff are a low, and dependent on rainfall intensity and K inputs (Alfaro et al., 2008).



2.2 Muriate of Potash (MOP)

Muriate of potash is known as potash, or potassium chloride which is commonly used to replace potassium removed from the soil and leaching. Potash refers to a wide range of ore-bearing rocks, ores, and processed products that all contain potassium in a water-soluble form (Warren, 2016). Besides, MOP (potassium chloride) contains 60% K₂O. Gypsum, anhydrite, polyhalite, kieserite, kainite, carnallite, and bischofite was found in the potash deposits rich in MgSO₄ accessories.

2.2.1 Production of MOP as fertilizer in agriculture industry

Muriate of potash is a fertilizer production is very important for crops. Fertilizer containing potassium is used in the production of fruits and vegetables (Prakash & Verma, 2016). There are over 90% of global potash production used for plant nutrition. This is because, (MOP) fertilizer has many benefits for use on crops. Besides, (MOP) is the most commonly used source of potash for farm use and is also a major source of potash ingredient for compound fertilizers containing potassium.



2.2.2 Uses of MOP

Potash is most usually used to provide soluble potassium which is one of the three basic plant nutrients. Muriate of potash is a fertilizer widely used to cultivate pastures, sugarcane, fruit trees, vegetables, and other farm crops. Usually, MOP fertilizer is suitable for use on any type of plant. The use of (MOP) fertilizer encourages root growth and can increase the strength of the stem. In addition, the chloride content of MOP also helps disease resistance and improves flower yield and fruit quality (Ebert, 2009). Potassium in (MOP) fertilisers is critical for encouraging nitrogen fixation in legume crops as well as increasing the size, colour, and sugar content of products like fruits.

2.3 Leaching of MOP

The leaching factor of (MOP) is a potassium to the types of soil. The leaching process can easily lose potassium (K). Because the soil solution K has a significant risk of leaching and thus loss from the soil system, (Bhattarai & Swarnima, 2016). Normally, soil type and quantity of rainfall must be taken into account when making management decisions on the leaching losses. Besides, the leaching of potash also in acidic soils. Potassium deficiency is widespread in intensively cropped areas, with low replenishment and high losses due to leaching. Potassium reduction is becoming more common in the soil. Potassium deficit is one of the main constraints to crop production as well as the cause of MOP leaching due to unbalanced fertilization (Meena et al., 2016).

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2.4 Common management of reducing MOP loss via leaching

A fundamental principle of soil management is to provide necessary nutrients to plants in sufficient quantities to achieve desired crop productivity. To reduce the MOP loss via leaching with using the sandy soil (Jeffery et al., 2011). Due to its displacement to the soil solution and percolation especially in sandy soils potassium is thought to be the most easily leached cation. This is because, sandy soils can be reduced by limiting the soil to a pH of 6.2 to 6.5. However, the application of high levels of limestone to lowpotassium soils can lead to potassium deficiency of plants growing in the soils.

2.5 Biochar

Biochar is a carbon-rich organic material is also a by-product of high-temperature and low-oxygen pyrolysis. Biochar is produced by a pyrolysis process naturally that use completely or almost completely heats biomass such as wood, fertilizers and leaves without presence of oxygen to produce gas and oil as a product. The quantity of these materials produced are influenced by the processing conditions (Zhang et al., 2020). Furthermore, biochar has the potential to supply environmentally farm based renewable energy. The quality of biochar is heavily relied on several factors such as metals, soil type, pyrolysis raw materials and the quantity of biochar used in the soil.



2.5.1 Introduction and properties of biochar

Biochar is a charcoal-like substance that is used in the soil to trap carbon and improve soil characteristics. Biochar is a term that is commonly used to refer to various pyrolysis co-products such as syngas (Fryda & Visser 2015). Biochar is also crucial for the development of sustainable agricultural systems (Placek et al., 2017). Use of modified biochar is regarded as a viable approach to achieve high yields without damaging the natural ecosystem and has some positives impacts.

Rice straw biomass was used to produce biochar at 600°C for 3 hours by a recently reported method (Guo and Chen 2014). Regardless of pyrolysis temperature or residence duration rice straw biochar is often alkaline. This is because, alkaline biochar can be used as a soil amendment for neutralizing acidity, improving soil fertility and sequestering C in acidic soils. The biochar become blended uniformly and characterized for particular surface, production yield, pH, and pore width which have been observed with the aid of using the protocol of (Chintala et al., 2013). In addition, rice straw biochar was used to improve deposits contaminated with heavy metals. Within the ash part of biochar contain elements such as carbon, hydrogen, sulphur, oxygen, nitrogen, as well as minerals. Biochar properties are divided into three, among them physical, biological and chemical.

The physical properties of biochar directly and indirectly affect the soil system. Soil has unique physical properties that depend on the properties of minerals, organic matter their relative quantities, the relationship between minerals and organic matter. When biochar is present in soil mixtures, it has a major impact on the system's physical qualities, influencing depth, texture, structure, porosity, and consistency via modifying surface area, distribution, and density.

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From a chemical point of view, biochar reduces the acidity of the soil by raising the pH and helping the soil retain nutrients and fertilizers. (Lehmann and Rondon. 2006). Besides, it also helps to prevent drainage and leaching of manure by reducing fertilizer usage and reducing agricultural pollution in the surrounding area (Cao et al., 2018). In terms of biological properties, biochar serves to increase the respiration of soil microorganisms by creating space for soil microorganisms, increasing soil biodiversity and increasing soil density. (Slapakova, Jerabkova & Tejnecky, 2018).

2.5.2 Types of biochar

Basically, type of biochar is a from charcoal given to soil and crop systems as a soil conditioner. The process of manufacturing biochar is almost the same as charcoal which is commonly used as fuel. Biochar can be made from varieties of feedstock. There are types of biomasses include agricultural wastes, rice husks, rice straw, bagasse, paper products, animal manures, and urban green waste.

Agricultural waste is defined as waste that is not desired to be produced as an agricultural product activity such as manure, silage, fertilizers, pesticides and herbicides as well as waste used from farms and poultry houses (Siedt et al., 2021). Besides that, agricultural waste can act as fertilizer as they contain nutritive minerals such as nitrogen, phosphorus, and potassium that can assist plants growth.

The type of biochar is rice husks. Rice husks biochar is the by product of rice processing and which is abundant in Asian countries. Normally, the rice husks management involves direct dumping into the soil, composting or burning them out in mass as a fuel. Furthermore, biomass with a high carbon content such as rice husk can be converted into energy-rich biochar through thermochemical treatment. The yield of biochar was computed as the mass of biochar produced from the dry mass of rice husks (Rajkovich et al., 2012).

Next, type of biochar is rice straw. Pyrolysis of rice straw to produce biochar for soil amendment is a way to improve soil fertility and extend carbon storage. These properties indicate that rice straw-derived biochar may be used as fertilizers and soil amendment (Wu et al., 2012). Furthermore, rice straw derived biochar also contained turbostratic crystallites at 400°C, and showed a high level of aromatization at 500°C. Thus, the higher charring temperature will increase the aromaticity of biochar, and might include its recalcitrance.

2.5.3 Uses of biochar

Nowadays, the use of biochar in the soil is very helpful in improving soil quality and stimulating plant growth. The use of Biochar is to produce energy, and also the potential for increase the productivity of agricultural land. Besides that, biochar can also improve contaminated soils, and possible mitigation effects on climate change. Biochar may be effective for carbon sequestration, nutrient leaching losses, and lowering greenhouse gas emissions (Siedt et al., 2021). The operating temperature of the process has a major influence on the quality of the biochar produced. Because of its carbon and nutritional content of biochar produced at moderate temperatures is appropriate for agricultural usage. While the higher temperature, can increase its porosity and thus increase its effectiveness in absorption contaminants found in the soil (Agrafioti, 2013). Furthermore, the use of biochar in agriculture can increase the efficiency of nitrogen use by increasing the chemicals in the soil properties. Biochar in agrochemicals can assist reduce phosphate and nitrate by binding to them. Besides, use of biochar can reduce pollution of streams, groundwater, and resolving major problems hindering intensive agriculture (Capodaglio, 2017).

2.6 Potential of amending MOP with biochar in reducing MOP loss via leaching.

Most of the application the efficiency of biochar depends heavily on surface area and ion exchange capacity. These properties depend on the biochar source material, pyrolysis temperature, and heating rate. One of the characteristics of these biochar is the presence of both positive and negative charge groups on their surfaces area. Although the biochar surface area plays a role in dye adsorption surface area charge effects are very clearly (Nanda, 2015). Furthermore, biochar produced at higher temperatures contains more inorganic components and has lower acidic functions suggesting that it could be applied to neutralise acidic soil. The pH of biochar regulates the sorption of organic particles onto its surface, determining its ionic strength. Hence, the sorption potential of biochar for methyl violet increased significantly when the pH was raised from 7.7 to 8.7. According to Moon et al. (2013), biochar amendment to lead-contaminated soil increased the soil pH thereby inducing negative charge on biochar surface and favouring the sorption of lead. Other than that, biochar surfaces are usually charged negative which facilitates the electrostatic attraction towards positively charged cationic organic compounds. **CHAPTER 3**

METHODOLOGY

3.1 Preparation and soil sampling

The soil samples were taken at 0-30 cm from an uncultivated land in Agro Techno Park of University Malaysia Kelantan Jeli Campus. A total of 8 sacks of soil samples were taken within a 20m x 20m randomly. The soil was flattened in a big tray and the soil was air-dried for 2 or 3 days. Furthermore, the soil sample was crushed using mortar and pestle. Then, the soil was placed in sieve to pass through a 2-mm sieve for 18 vases, respectively for laboratory analysis.



3.2 Soil analysis

The soil sample was analysed for soil texture, soil pH, total organic matter, total carbon, total nitrogen, soil electrical conductivity (EC), soil cation exchange capacity (CEC), exchangeable K and available P. According the research article (Peech, 1965) soil pH were measured in ratio of 1: 10 (soil: water) with application a digital pH meter. The soil texture was dictated using the hydrometer technique (Jones, 1991). While loss-on ignition method was used to measure organic matter content (OM) and total organic carbon (Tan, 2003). The Kjeldahl method was applied in order to determination the total N (Bremner, 1965). In addition, Mehlich (1953) double acid method was used extracting soil available P and exchangeable K. Afterwards, an Atomic Absorption Spectrophotometer (AAS) was used to determine those cations (Analyst 800, Perkin Elmer, Norwalk, USA). The molybdenum blue method was used to determine the amount of available P in the soil (Murphy and Riley, 1962). A UV-Vis spectrometer was used to examine the development of blue colour at 882 nm wavelengths spectrometer (Thermo Scientific Genesys 20, USA). The ammonium acetate leaching method was applied as describeb by Cotteine (1980) in order to measure soil cation exchange capacity (CEC). The exchangeable NH_4^+ and available NO_3^- was extracted by using a method described by Keeney and Nelson (1982), after the ions was determined via steam distillation (Tan, 2005). The following are the detailed processes for each analysis:



3.2.1 Soil pH and Electrical Conductivity determination (EC)

The soil water method was used to determine the soil pH and EC. Digital pH and EC meter were used to measure soil pH and EC, a ration of 1:10:5 (soil and distilled water suspension) was used in this method (Peech, 1965). A 100 mL conical flask was filled with 50 mL distilled water and 5 g of dried soil. The samples were shaken at 180 rpm using a shaker for 15 minutes. After that, the samples were then allowed to rest for 24 hours before being analysed for pH using a digital pH metre. The suspension was then filtered through Whatman filter paper No. 2 before being utilised to determine the electrical conductivity using an EC metre.

3.2.2 Soil texture determination

The soil texture determination was carried out by using hydrometer method following Bouyoucos (1962). In this method, the texture of the soil was determined by measuring total sand (2.0 - 0.05 mm), silt (0.05 - 0.002 mm), and clay (<0.002 mm). A 50 g of soil was placed in a blender cup. Then, after 15 minutes of continuous blended, 10 mL sodium metaphosphate and 100 mL water were added. The soil suspension was transferred into a 1000 mL measuring cylinder after 15 minutes without leaving any soil behind. Next, the distilled water was added until the volume in the cylinder reached 1000 mL. Then, it was stirred by using stirring rod to ensure thorough mixing of sediments in the suspension. A hydrometer was properly placed in a suspension. After 40 seconds of stirring, the reading of the meniscus on the hydrometer stem was recorded. After each use, the hydrometer was cleaned and dried. After that, the suspension was stirred for 40

seconds and the reading was recorded. The hydrometer was cleaned and dried. After 2 hours of settling time, the suspension was stirred again, and the third hydrometer reading was taken using the same procedure as before. The amount of silt and clay respectively represents by the given formula where x represents first reading, y for second reading and z for third reading. The calculations are based on the following basic principle:

Percentage of sand + silt + clay = 100%

(x + y) / 2 = a

For 40 second reading:

Percentage of silt+ clay = $(a/50) \times 100 = b$

Percentage of sand = 100 - b = c

After 2 hours reading:

Percentage of clay = $(x/50) \times 100 = y$

By difference: Percentage of silt = 100 - c - y

3.2.3 Soil Total Organic Matter and Total Organic Carbon (C) determination

To determine the total organic matter and total carbon in this study, dry combustion method (loss on ignition method) was used (Tan, 2003). The air-dried sample was placed in an oven and was left for 24 hours at 60 °C. Desiccator was used to cool down the sample. The crucible's initial weight was measured first. The crucible's weight was then measured to the 5 g of soil sample. Afterward, the sample was ashed at 300 °C in the muffle furnace for an hour and temperature will increase to 550 °C. The ashing process was continued for another 8 hours. Lastly, the sample was allowed to cool before

inspection. The weight of the sample in porcelain crucible after ash was calculated. The total organic matter (OM) and C was calculated using the following calculation (Tan, 2003).

Organic matter (%) = $\frac{\text{(Initial weight of sample and crucible (g) - Final weight of sample and crucible (g))}{\text{Initial weight of sample (g)}} \times 100$

Total Carbon = % Organic matter x 0.58

3.2.4 Cation Exchange Capacity (CEC) determination

The cation exchange capacity (CEC) of the soil was dictated by using ammonium acetate (leaching method) (Hailegnaw et al., 2019). A 10 g of soil sample was weighed and placed in a 250 ml conical flask. The mixture was stirred at 180 rpm for roughly 5 hours before being added with 100 mL of 1 M ammonium acetate (NH4OAc). The solution was then filtered using Whatman No. 2 filter paper and the filtrate was discarded. The soil was washed with 20 ml of 95% ethanol, then filtered again and discarded after collection finish. Besides, the soil was filtered with 100 ml of 0.1 M potassium sulphate (K₂SO₄), then collected and make the volume of volumetric flask up until 100 ml. Following that, a pipette was used to place 10 ml of the sample into the distillation apparatus. Then, in the same beaker, 10 mL of 40% sodium hydroxide (NaOH) was added. The sample was distilled, and the distillate was collected in a 10 mL boric acid indicator solution containing 2% boric acid. During the distillation process, boric acid has

a reddish-purple colour was changed to green colour. The 100 mL conical flask containing the distillate was removed from the distiller after obtaining twice the original capacity (20 mL). The distillate was then titrated against 0.01 MHCl until the green colour to reddish purple. This resulted in a CEC of cmol kg1 and the soil CEC was determined using the following equation:

Cation exchange capacity of soil (cmol kg-1)

 $= \frac{[\text{Titrate value (mL)} \times \text{ concentration of acid used } \times 100 \times 10 \times 1000]}{10}$

3.2.5 Nitrogen determination

The Kjeidahl method was used to determine the nitrogen content in soil (Kjeltec 8200). Weighing 5 g of soil air-dried and placing it in a digestion tube. After that, 5 mL sulphuric acid (H₂SO₄ tube) was added to the digestion tube, followed by 1 g Kjeldahl catalyst tablet digestion was set up in a Kjeldahl digestion block and the sample was digested for 3 hours at 400°C until it become colourless. After 15 minutes of cooling, 30 mL of distilled water was added to the sample. After cooling, the soil sample was transferred to 100ml conical flaks, and the volume was increased to 100ml. 10 mL was pipetted and extracted into distillation apparatus under the observation of 10 mL of 40% NaOH. The sample was distilled for 3 minutes before being collected in 10 mL of a boric acid indicator solution containing 25% boric acid. The colour of boric acid was changed from reddish-purple to green. The sample was then titrated against 0.01 M H₂SO₄ until it

changed colour from green to reddish purple. The percentage of nitrogen in the soil was calculated as follows:

N (%) = [(V-B) × M × R × 14.01 / Wt × 1000] × 100

Where,

V = Volume of 0.01 M H_2SO_4 or H_2SO_4 titrated for the sample (mL)

B = Blank titration volume (mL)

 $M = Molarity of H_2SO_4 solution$

14.01 = Atomic weight for N

- R = Ratio between total volume of the extract and the extract volume used for distillation
- Wt. = Weight of the sample (g)

3.2.6 Phosphorus determination

The soil available P was extracted by Mehlich No. 1 Double Acid Method (Mehlic, 1953). 250 ml conical flask was filled with a 5 g of soil sample. The solution was shaken for 10 minutes at 180 rpm and 20 ml double acid (0.05 M HCl + 0.025 M H₂SO₄) was added. After that, the supernatant was filtered into another beaker using Whatman filter paper No. 2. Next, 8 ml of reagent B were pipetted into a 50 ml volumetric flask. Then, slowly add 0.1 to 5 mL soil extract while observing the light blue colour development. Distilled water was added until 50 mL volume once the solution colour was changed into blue. The sample was then pipetted into a cuvette. To calculate the soil

available P, a UV-Vis reading at wavelength of 882 was obtained and submitted to the following calculation.

Soil available P (ppm) = UV-Vis reading + $\frac{20}{5}$ + $\frac{50 \text{ mL}}{\text{Volume of extractant}}$ x dilution factor

3.2.7 Soil Exchangeable K determination

The Mehlich No. 1 Double Acid Method was used to extract the soil exchangeable K (Mehlich, 1953. 250 ml conical flask was filled with a 10 g soil sample. Then, 40 ml double acid 0.05 M HCl + 0.025 M H₂SO₄) was added after that, the solution was shaken for 15 minutes. After that, the supernatant was then filtered using Whatman Filter Paper No.2, and the extract was aspirated into AAS and the reading was recorded. The exchangeable soil cation was calculated using the equation below (Mehlich, 1953):

Soil exchangeable = ASS reading (ppm) + Volume of extractant (mL) cation (ppm) Weight of soil sample (g)

3.3 Characterization of Rice Straw Biochar

The biochar was obtained from the postgraduate student. Besides, the biochar was produced used a 200 L cylindrical kiln with the burning time of 4-6 hours, with temperature ranging from 300-400 °C. Then, cooled down for 12 hours. Activation of biochar was carried out by soaking the biochar with 5% chicken slurry for 7 days. Later, it was dried and stored in a big container for further use. The analysis for biochar characterization was identical to the soil characterizations discussed previously. In addition, scanning electron microscopy was used in conjunction with energy dispersive X-ray spectroscopy analysis (SEM-EDX JEOL JSM 6400) was carried out to analysed the surface morphology of rice straw biochar.

3.4 Laboratory leaching experiment

The laboratory leaching experiment was conducted in a research complex at University Malaysia Kelantan Campus Jeli. Pots with 15 cm in height, 13 cm in width, and 13 cm in diameter were filled with 600 g of soil (from 2 mm bulked Bekenu Series soil sample). Prior to the leaching experiment, the chemical fertilisers and organic amendments were thoroughly mixed with the soil. The combination of treatments was based on the treatments tested in the field trial. The rates of the P fertilizers (TSP and ERP), pineapple leaf residues, and the biochar were used in this study were 60 kg P₂O₅ ha-1, 10 t ha-1, and 20 t ha-1, respectively. These rates were followed by standard recommendation for maize planting (MARDI, 1993, Zhang et al., 2012; John et al., 2013). Based on these recommendations, 5 g of TSP, 8.24 g of ERP, 11.2 g of biochar, and 11.2

g compost per pot containing 600 g of soil were used. The treatments were prepared into three replication completely randomised design (Table 3.1). Treatments analysed in this leaching experiment was summarized as follows:

Treatments	Descriptions
ТО	Soil only
T1	Soil + 2.5gram MOP
T2	Soil + 2.5gram MOP + 1.5gram Biochar
Т3	Soil + 2.5gram MOP + 3.0gram Biochar
T4	Soil + 2.5gram MOP + 4.5gram Biochar
Т5	Soil + 2.5gram MOP + 6.0gram Biochar

Table 3.1: Treatments of the incubation study



Figure 3.1 The soil sample of leaching



3.5 Post-treatment soil analysis

Water was poured into each pot of soil and leachates were collected to determine the amount of K lost from the soil. The amount of water use was determined over a 30 days period based on rainy days. The Malaysian Meteorological Department provided five years of rainfall data which was applied to calculate the average amount of rainfall every month. Leachates were collected and analysed every 3 days for K and pH. The soil samples were collected at 30 days of leaching. The soil samples were air-dried and analysed for pH, exchangeable acidity and total exchangeable K by utilising standard procedures mentioned earlier in Chapter 3.

3.6 Statistical analysis

This experiment was arranged in a completely randomized design with three replicates. SPSS software version 24.0 (SPSS Inc, US) was applied in order to determine the statistical analysis for all the data. A one-way analysis of variance (ANOVA) was used to examine the influence of different rates of biochar addition on all treatments. The significant differences among treatments were separated by using Tukey's HSD test and consider significant at $P \leq 0.05$.



CHAPTER 4

RESULT AND DISCUSSION

4.1 Selected physico - chemical properties of soil samples

Table 4.1 shows the selected physico - chemical properties of the soil sample prior to leaching experiment. The soil was a sandy loam and had a low pH which is 4. The soil contained low available P which was 4.5. Besides, the soil also contained relative high concentration of soil organic matter which is 5.87%. In the productivity of plants growing in tropical and subtropical ecology, one of the most common limitations is P deficiency (Ramaekers, et al., 2010) where P is very strong in the soil through adsorption and rainfall, reducing plant bioavailability (Do Vale & Fritsche-Neto, 2013). Exchangeable K in the soil sample was low which is 6.67. This is very common in the tropical acid soils due to the high mobility of K in the soil and is extensively prone to leaching during intense rainfall events.

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Property	Value Obtained	
Soil Textural Class	Sandy Loam	
pH	4	
Electrical conductivity (µS/cm)	13.33	
Soi <mark>l Organic M</mark> atter (%)	5.87	
So <mark>il Total Car</mark> bon (%)	3.4	
Soil Total N (%)	0.18	
Available P (ppm)	4.5	
Exchangeable K (ppm)	6.67	

Table 4.1 Selected physico- chemical properties of soil samples.

4.2 Selected chemical properties of rice straw biochar

The selected chemical properties of rice straw biochar are shown in Table 4.2. Biochar is a product obtained from pyrolysis of woody materials that can provide such a crop management practice (Glasser et al., 2004). According to the study by Brusscher et al., (2010), the pH of rice straw biochar was 9.2, which indicate alkaline properties. The high pH recorded in rice straw biochar was supported by the high Ca and Mg content in the rice straw biochar. According to Wu et al., (2012) reported that rice straw derived biochar contains pH more than 9. The CEC of the biochar was high. This indicates shows that rice straw biochar high cation exchange sites for K, Ca, Mg and Na due to the O⁻ functional group located on the surface of the biochar.



Property	Value obtained
pH (water)	9.2
CEC (cmol kg ⁻¹)	75.6
Total N (%)	0.45
Available P (mg kg ⁻¹)	14.3
Exchangeable Ca (mg kg ⁻¹)	3599
Exchangeable Mg (mg kg ⁻¹)	809
Exchangeable K (mg kg ⁻¹)	12,030
Exchangeable Na (mg kg ⁻¹)	246.3

 Table 4.2 Selected chemical properties of rice straw biochar

4.3 Effect of treatment on the potassium concentration in leachate over 30 days of leaching.

Figure 4.1 show the effect of rice straw biochar on K leaching for over 30 days. There was no leaching activity of K in T0 because it only contains soil. Meanwhile, T1 contains soil with MOP and the leaching process of K started on the day three. Afterward, T2-T5 were applied with difference rates of rice straw biochar and the leaching of K were high compared to T1 on day 3. This could be due to the partial dissolution of K content in the biochar (Iqbal et al., 2019). The leaching in T2-T5 had reduced starting from days 9 onwards. Figure 4.2 shows the effect of treatment on the sum on K in the leachate after 30 days of leaching. Based on the graph, it can be observed that T1 had a high rate of K leaching compared with T0 (soil only) and treatment with biochar (T2-T5). The accumulation of K in T2-T5 at 30 days of leaching were no significant different for each

other. This indicates the effectiveness of high CEC in the biochar that capable to chelate and retain the K from leaching and release it slowly over time for plant uptake.



Figure 4.1 K concentrations in leachate over 30 days of leaching



Figure 4.2 Accumulation of K in leachate thought 30 days of leaching

Mean between columns with different letter (s) indicate significant difference between treatments by Tukey's test at $p \le 0.05$.

4.4 Effect of treatment on the soil pH, EC and exchangeable K content after 30 days of Leaching.

The treatments with mixture of chemical fertilizer and rice straw biochar (T2-T5) showed significantly higher soil pH compared to the T0 (soil only) in Figure 4.3. Several authors observed that soil applied with rice straw biochar increased the soil pH (Laird, 2008). Soil pH increased because of the precipitation of exchangeable Ca. This finding is comparable with those reported by Narambuye and Haynes (2006) and Haynes and Mokolobate (2001). The initial increase in the soil pH of biochar due to biochar in the present study is related to the initial pH at their application. The higher pH buffering capacity of the rice straw biochar (Figure 4.3) partially describes the higher pH of the soils with this biochar.

The effect of rice straw biochar on the soil EC after 30 days of leaching presented in Figure 4.4. Treatment with application of rice straw biochar managed to reduce the soil EC significantly as compared to T1(soil + MOP only). This could be due to the active cation exchange such as Na⁺ ions on the O⁻ and COO⁻ functional group located on the surface of biochar. The surface of the functional group exerted a strong affinity towards Na⁺ ions and thus reduced its concentration in the soil (Premarathna et al., 2019).





Figure 4.3 Soil pH after 30 days leaching

Mean between columns with different letter(s) indicate significant difference between treatments by Tukey's test at $p \le 0.05$



Figure 4.4 Soil EC after 30 days of leaching

Mean between columns with different letter(s) indicate significant difference between treatments by Tukey's test at $p \le 0.05$

Potassium concentration remained in soil after 30 days of leaching is shown in Figure 4.5. Based on the graph, the K concentration retained in the soil was significantly higher in the treatment with rice straw biochar (T2-T5). Higher rates of rice straw biochar application (15-20 t/ha) were found to retain significant higher K compared to low applied rates of rice straw biochar (T2 and T3). This was due to higher applied rates biochar had a higher K sorption and a large surface area. The findings of other studies stated that biochar has a special porous structure and surface functional group that adsorbs K⁺ ions effectively (Zhang, 2016). These properties indicate potential application of rice strawderived biochar as a fertilizer and soil amendment (Wu et al., 2012). Meanwhile, T1 had significant low K retention because K is absorbed in the soil and the low clay minerals in the soil used this study reduced the adsorption of K on the clay surface that prevent the K for leaching.



Figure 4.5 K concentration retained in soil after 30 days of leaching

Mean between columns with difference letter(s) indicate significant difference between treatments by Tukey's test at $p \le 0.05$

CHAPTER 5

CONCLUSION

As a conclusion, treatments with rice straw biochar application were able to improve the K retention in the soil. Application rate of 15 - 20 t/ha of rice straw biochar was found to provide better retention K in the soil. The treatment with rice straw application also decreased to the soil acidic by raising the soil pH to clear neutral. This indicates the potential of rice straw biochar in amending the chemical properties of acidic soil and reduce the K leaching for the soil after application of MOP fertilizer. This research is suggested to be future tested in the field with the presence of plants in order to assess the effect of biochar in retaining the K in soil in the natural environment.



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APPENDIX A







Figure A.2: Shaked the soil sample



Figure A.3: Filtering the sample soil sample



Figure A.4: The soil water sample

