

Improving Potassium (K) Retention in Acidic Soil by

Amending Muriate of Potash Fertilizer with Zeolite

by

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THESIS 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.

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Meningkatkan Pengekalan Kalium (K) dalam Tanah Berasid dengan Meminda Muriat Baja Kalium dengan Zeolit

ABSTRAK

Kalium (K) ialah makronutrien penting untuk tumbuhan yang mengambil bahagian dalam banyak proses fisiologi. Kalium mudah hilang semasa proses larut lesap dan berkemungkinan besar akan terlarut lesap dan oleh itu hilang dari sistem tanah. Selain itu, larut lesap baja K juga berlaku dalam tanah berasid. Penyusupan ke dalam tanah mengakibatkan penambahan yang rendah dan kehilangan K tinggi, yang menunjukkan kekurangan umum di kawasan berpenduduk padat. Kebanyakan K adalah mudah alih dalam tanah dan kebanyakannya hilang dari tanah dengan larut lesap, menjejaskan kecekapan baja yang digunakan. Matlamat projek ini adalah untuk (1) Mensampel dan mencirikan sifat fizikokimia terpilih bagi sampel tanah untuk digunakan dalam larut lesap makmal kajian K (2) Mencirikan sifat kimia terpilih bagi zeolit (3) Untuk menentukan kesan pindaan MOP dengan zeolit pada kadar yang berbeza kepada pengekalan K dalam tanah dan sifat kimia tanah yang terpilih. Zeolit yang digunakan dalam kajian ini adalah dalam bentuk serbuk. Satu eksperimen larut lesap untuk menilai kesan baja K yang dipinda dengan kadar zeolit yang berbeza terhadap larut lesap K dan pengekalan dalam tanah telah dijalankan selama 30 hari. Air larut lesap dikumpul setiap 3 hari dan dianalisis untuk kandungan K. Sampel tanah dikumpul selepas kajian larut lesap berhenti untuk menentukan jumlah K yang tersimpan di dalam tanah selepas larut lesap. Rawatan dengan zeolit dapat mengurangkan larut lesap K daripada tanah. Kadar penggunaan 20t/ha zeolit didapati memberikan pengekalan K yang terbaik di dalam tanah. Rawatan dengan aplikasi zeolit juga meningkatkan pH tanah kepada hampir neutral. Ini menunjukkan potensi penggunaan zeolit dalam meminda sifat kimia tanah.

Kata kunci: Kalium (K), zeolit, larut lesap, pengekalan, baja bukan organik

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

ABSTRACT

Potassium (K) is an essential macronutrient for plants that participate in many physiological processes. Potassium is easily lost during the leaching process and very likely to be leached out and therefore run off from the soil system. In addition, the leaching of K fertilizer also occurs in acid soils. Infiltration into the soil results in low replenishment and high K loss, which indicates a general deficiency in densely populated areas. Most of K is mobile in soils and mostly lost from the soil by leaching, affecting the efficiency of the fertilizers applied. The aim of this project was to (1) Sample and characterize selected physicochemical properties of soil sample to be used in laboratory leaching of K study (2) To characterize the selected chemical properties of zeolite (3) To determine the effects of amending MOP with zeolite at different rates on K retention in soil and selected chemical properties of soil. The zeolite that was used in this study was in powder form. A leaching experiment to assess the effect of K fertilizer amended with different rates of zeolite 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 samples were collected after the leaching study stopped to determine the amount of K retained in the soil after leaching. Treatments with zeolite were able to reduce the K leaching from the soil. Application rate of 20t/hazeolite was found to provide the best retention of K in the soil. The treatments with zeolite application also improved the soil pH to near neutral. This indicates the potential of zeolite application in amending the soil chemical properties.

Keywords: Potassium (K), zeolite, leaching, retention, inorganic fertilizer

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LIST OF ABBREVIATION AND SYMBOLS

S	Sulfur
Ν	Nitrogen
Р	Phosphorus
К	Potassium
ОМ	Organic matter
С	Carbon
Ca	Calcium
Mg	Magnesium
Na	Sodium
NaOH	Sodium hydroxide
HCl	Hydrochloric acid
H2SO4	Sulphuric acid
С6Н8О6	Ascorbic acid
рН	Potential of hydrogen
EC	Electrical conductivity
МОР	Muriate of potash
NADPH	Nicotinamide adenine dinucleotide phosphate
K ₂ O	Potash
KCL	Potash muriate
K ₂ SO ₄	Sulfate of potash
KNO3	Potash nitrate
NH ₄ +	Ammonium

NO ₃ ⁻	Nitrate	
		Food and Agriculture Organization of United
FAO		Nation
MARDI		Malaysian Agricultural Research and Development
		Institute
SPSS		Statistical package for social science
ANOVA		Analysis of variance

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The most common inorganic cation in crops is potassium (K) which consists of up to 10% of the dry weight of the plant (Broadley et al. 2004; Watanabe et al. 2007). The K+ is found in high concentrations in growth tissues and organs reproductive, implying that it is necessary for metabolism of cell and the extension of growth. The potassium was needed for the function of many enzymes which those involved in metabolism of energy, synthesis of protein and transportation of solute (Leigh and Wyn Jones 1984; Mengel et al. 2001; Amtmann; Troufflard & Armengaud, 2008; Britto and Kronzucker 2008). Potassium plays a significant role in metabolism of plant, development and adaptation of stress. However, potassium is not like other macronutrients like nitrogen, phosphorus and sulfur because potassium does not absorb into organic matter, but it remains in ionic form (K+) throughout the "life" of the plant. For several aspects of food production, K in appropriate quantities is needed. Potassium is an essential content which not only for maximum yield, but also for improving the quality of nutritional in the crop and dairy products (by animal feed). For mechanical stabilization of fruits and cereal crops, it requires a high tissue K content during storage and in the field. The appreciation in the importance of potassium for taste is very less especially for tea and wine. An increase in crop resistance to biotics (pathogens and pests) and factors against abiotic stress (salinity and drought) is associated with a high supply of K (Amtmann, Troufflard & Armengaud, 2008; Shabala and Cuin, 2008).

In agriculture, the use of potassium chloride (muriate of potash, MOP) is the most common chemical source of K used in potassium fertilizers because of the high K content value of 60%, low price, wide availability and ease of integration into fertilizer production of 95% (Johnston, 2003). Muriate of potash (MOP) is the most popular potassium fertilizer (Khan, 2014). The MOP is characterized as a K fertilizer that is easily soluble in soil moisture. In addition, MOP is also one of the main nutrients which for plants and there are various types of salt which contain K content in a form that is easily absorbed by plants (Econ, 2019). Therefore, potash fertilizers have become an increasingly important input for the growing population demand for food and fibre.

Since MOP is the country's primary source of fertilizer K, the particle size of MOP appears to play a significant role in modifying the behavior of fertilizer on field crops by influencing its rate of dissolution and leaching, according to sporadic studies conducted under controlled conditions (Pritchett and Nolan 1960, Miwa and Kurihara 1977). 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 (Kayser, 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). MOP fertilizer having largest particle size showed invariably low yield response compared with the control in different crops. Hence, the K balances rate requires an accurate determination of the K leaching loss (Askegaard, 2004). According to study by from Malavolta (1985) soil type could affects K leaching as the final amount of K lost depends on the availablity of K in soil, which is related to the soil texture.

According to Coombs et al. (1997), crystalline materials with a clinging tetrahedra system are zeolite minerals because each one contains four atoms of oxygen which surround the cation. The water and cations might reversibly extract or substituted by other cations in the composition of natural zeolite (Rehakova et al., 2004). The channel dimensions specify any ionic species such as maximum size that reached the zeolite pores. As a consequence, the pores of several zeolites are not cylindrical (Beitollah and Sadr, 2009). Since zeolite has special cation exchange, molecular sieve and absorption properties, it can be used in agriculture (Glisic and Milosevic, 2008; Hecl and Toth, 2009). The use of zeolites as fertilizers, stabilizers and chelating agents can be used as it is believed zeolites have the ability to lose and gain water in reverse without changing the crystal structure. Zeolite can be used in agriculture due to its high porosity, high cation exchange capacity, and selectivity to ammonium and potassium cations. They can be used as nutrient carriers and nutrient release carriers. However, in agriculture, zeolite is mainly used to capture, store and slowly release nitrogen.



1.2 Problem Statement

There are several research and studies that have been carried out to reduce the leaching of muriate of potash (MOP) by excess application of MOP in order to saturate the K in soil. Usage of a few types of micronutrients such as copper and zinc were also reported. However, there is a lack of research on the application of MOP fertilizer with zeolite to reduce the leaching of K.

1.3 Objectives

- 1. To sample and characterize selected physicochemical properties of soil sample to be used in laboratory for K leaching study.
- 2. To characterize the selected chemical properties of zeolite.
- 3. To determine the effects of amending MOP with zeolite at different rates on K retention in soil and selected chemical properties of soil.

1.4 Scope of study

The scope of study is to assess the effect of amending MOP with zeolite at different rates on reducing K leaching in soil and improve the selected chemical properties of soil.

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1.5 Significance of Study

Since the beginning of 2007, the price of fertilisers has fluctuated significantly. The cost of K in the form of improve the MOP has remained high. Potash supply is expected to meet demand, which is expected to rise at a rate of 2.4 percent per year, and according to the United Nations Food and Agriculture Organization (FAO, 2008). The proposed approval in help to reduce the amount MOP fertilizer used in agriculture thus reducing their import to Malaysia. It is also may contribute to the increase in crop production in the country and reduce the cost of production by solving the issue of K leaching in soil.

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CHAPTER 2

LITERATURE REVIEW

2.1 Potassium

Potassium is a macronutrient and an important component of plant growth and reproduction. Potassium is found in plants in higher concentrations than any other nutrient except nitrogen. Despite the fact that plants are capable of extracting significant amounts of potassium from soils, potassium deficiency in soils is not as common as nitrogen deficiency. Potassium might be dismissed at a rate that is effectively determined by the plant, since many soils have a large potassium reserve. In this regard, potassium's behaviour differs from that of nitrogen, since the maximum rate of nitrogen withdrawal from the soil is entirely beyond the plant's control (Black, 1957).

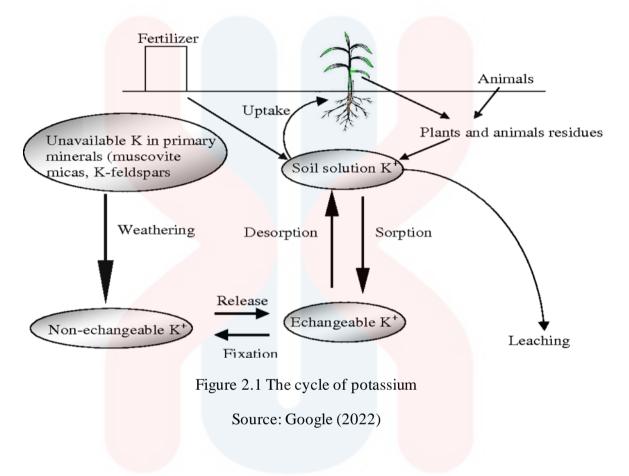
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2.1.1 Introduction of Potassium (K)

Potassium is one of the most important elements for life on earth. It is needed in large quantities by all plants, animals and even on the soil. Generally, potassium is one of the major nutrients which required by all plants. 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. According to McAfee (2008), feldspar and mica were the most significant soil components of potassium which account for 90% to 98% of the total.

2.1.2 Potassium Cycle

Based on soil type, the form contributes for 90% to 98% of total soil of K. The amount of K is found in the mineral's feldspar and mica. Besides, the use of K in crystalline and insoluble form unable to be applied to plants. The climate of mineral or break down, over time, releasing K into the environment. Some of K moves to the slowly accessible pool during minerals season. Some people even go to the nearest pool (see Figure 1). The K content that has been dissolved in groundwater allows the plant to easily absorb. However, the K content has been attached to the minerals of clay which when the concentration of K in the groundwater decreases can release more K into the soil solution. Many plant functions, such as metabolism of carbohydrate, activation of enzyme, osmotic control, and protein synthesis, need potassium (Chesworth,2008). Plant growth gains more from K attached to clay mineral exchange sites than from K trapped within clay mineral layers.



2.1.3 Classification of Potassium

One of 17 groups of nutrients is potassium (K) which required for plant growth and reproduction (Robert, 2004). In addition, nitrogen (N) and phosphorus (P) were classified as a macronutrient. Potassium inhibits nicotinamide adenine dinucleotide phosphate (NADPH) oxidases thus retaining the transport of photosynthetic electron. However, Potassium deficiency may reduce photosynthetic CO2 fixation and assimilate transport and utilization (Ahmad, 2012). Potash is also known as K₂O which can be used to express the content of various fertilizers containing potassium, such as potash muriate (KCI), sulfate of

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potash (K₂SO₄), double sulfate of potash and magnesium (K₂SO₄ 2MgSO₄), and potash nitrate (KNO₃). Then, potassium is the eighth most abundant element on earth which comprises about 2.1% of the earth's crust in fact it is also one of the most reactive elements and has never been found free in nature. In 1807, Sir Humphry Davy first isolated Metallic potassium through the electrolysis of molten caustic potash (KOH). Besides that, minerals such as sylvite (KCl), carnalite (KCl, MgCl2,6H2O), langbeinite (K2Mg2(SO4)3) and polyhylite (K2Ca2Mg(SO4) 4,2H2O) are believed to be obtained in potassium content. Typically, such minerals are believed to be frequently found in ancient lakes and seabeds. In addition, potassium nitrate (KNO3) is also known as saltpeter or nitre which can be used in fertilizers, match heads and pyrotechnics.

2.1.4 Importance of Potassium in Soil Plant System

The major nutrient element is potassium which found in soil that plants need in larger amounts. A very important nutrient is potassium (K+) which is important for plant development and even potassium also plays an important role in the regulation of plant production which involves osmoregulation, plant-water relationship and balance of internal cation/anion. Therefore, potassium also helps to improve early growth by reducing the growth of new tissues and cell expansion. Potassium is also necessary for plant ontogeny, as well as for improving plant quality and oil content. As a result, a lot of potassium is needed to keep plants healthy, but it is often skipped in favour of many crop production systems use nitrogen and phosphorus (N and P). Among those involved are parasites, drought, pests, cold, salinity, frost and water stagnation (Wang et al. 2013). Potassium application, on the other hand, may help plants resist insects and diseases.

2.1.5 **Problem** of Potassium Fertilizer

The problem of potassium fertilizer often happen is high cost needed due to 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). In addition, the lack of nutrients in the soil is affected by a variety of causes and as a result, the plant suffers from deficiency of nutrient. Furthermore, root growth is slowed, and K absorption is decreased in poorly drained soils due to the presence of large amounts of reduced substances. There are various possibilities to the cause of potassium deficiency. Among them are insufficient of fertilization, excess table salt i.e. sodium in the root area, unfavorable structure of soil such as sandy soil and the development of degradation zones around the roots (Kayser and Isselstein 2005; Moody and Bell, 2006; Andrist Rangel et al. 2007).

2.2 Muriate of Potash (MOP)

In potassium fertilizers, the most common chemical source of K was used in agriculture is potassium chloride (muriate of potash, MOP) because of it has high potassium content (60%), low price, widespread availability and ease with which it can be incorporated into fertilizer production (95%) (Johnston, 2003). Khan et al., (2014) initially declared and questioned the use of potassium chloride (MOP) as a primary

potassium fertilizer around the world, as well as a number of soil testing methods for plant accessible potassium.

2.2.1 **Production of MOP as Fertilizer in Agriculture Industry**

Muriate of potash is a fertilizer production is very important for crops. Potash fertilizer is used for fruit and vegetables production (Timms, 2005). Potassium chloride (KCl), recognized as the muriate of potash (MOP), is the most commonly used source of potassium for food crops. We'll use the abbreviation 'KCl' and reserve the word 'potash' for K2O, which is commonly used in fertilizer usage statistics. Only the cation K+ in KCl is typically thought of as a significant plant nutrient; the corresponding anion Cl- is generally thought to be undesirable but inevitable. Cl, from the other side, is now regarded as a necessary micronutrient for optimal growth (Fixen, 1993). Both K and Cl are the most essential inorganic osmotic active substances in plant cells and tissues, as well as the key ions involved in charge neutralization (Clarkson and Hanson, 1980).

2.2.2 Introduction and Properties of MOP

MOP has the physical properties of a white crystalline salt in its pure form. The colour of K minerals varies from white to red, depending on the impurities present and the refining methods used. At 30 °C, it dissolves 37 grams per 100 grams of water. Muriate of potash is evaporated of natural potash are a typical part of a series of brine evaporation, crystallizing at the higher concentration, either at the surface primary salts or in the shallow subsurface secondary salts (Cendon, 2003). The crystalline MOP has a specific gravity of 1.98 and is not quite hygroscopic. It flows easily and does not mold. MOP's chemical properties include 58% K_2O by weight and about 47% chloride. MOP has a neutral reaction and does not contain acidity or alkalinity when being applied to the soil.

2.2.3 Uses of MOP

Potash is used as a MOP fertilizer as well as a component of complex fertilizers. All complicated vegetation use Muriate of potash because the supply of potash nutrient. Usually, muriate of potash fertilizer is suitable for use on any type of plant. Sulphate of potash, which is also imported, accounts for a small portion of the country's total nutrient consumption (Kinekar, 2011). Farmers must supplement soil reserves with potash fertilizer whenever the soils are unable to provide the K needed for optimal yields.

2.3 Leaching of MOP

The leaching factor of (MOP) is a potassium to the types of soil. By the leaching process, the potassium (K) might be easily lost. This is due to the soil solution K has a high chance of leaching and thus loss from the soil system (Swarnima, 2016). Normally, soil type and quantity of rainfall must be taken into account when making management decisions on the leaching losses. Besides that, the leaching of potash also include in

acidic soils. On the other hand, sporadic experiments were conducted under controlled conditions, with dissolution and leaching rates being monitored (Pritchett and Nolan, 1960; Miwa and Kurihara, 1977). In most soils, all the potassium of annual plants can be applied during planting. In light-textured soils, especially in tropical regions, rainfall after planting may cause a lot of rainfall, leading to a lot of leaching after planting.

2.3.1 Common Management of Reducing MOP Loss via Leaching

The basic principle of soil management is to ensure that plants get enough essential nutrients to produce the required yield (Brady, 2002). Essential nutrients are usually applied together with chemical fertilizers to increase yields. Continuous use of excessive chemical fertilizers will lead to the loss of nutrients in the soil. In turn, this will lead to pollution, which is not beneficial to the growth of crops. Sandy soils can be reduced by limiting the soil pH between 6.2 to 6.5. However, the application of high levels of limestone to low-potassium soils can lead to potassium deficiency of plants growing in the soils.

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The terms in Greek are zeo (boil) and lithos (light) are combined to form the word zeolite which means stone. According to Coombs et al. (1997), a zeolite mineral also known as a substance of crystalline where with a system of linked tetrahedra, each of which contains four oxygen atoms surrounding a cation. The crystals of zeolite minerals are extremely diverse in appearance, size, and crystal symmetry. Most of them are in the monoclinic system, although in addition to the triclinic system, other crystal systems are also represented. Twins are common, which makes the crystal more symmetrical than the actual crystal. For example, twins almost always have Tilbit, even in rectangular clear water. Place one of these crossed extreme bass on the stage of a petrographic microscope, and the extinction pattern will immediately identify the twins.

2.4.1 Introduction and Properties of Zeolite

Collectors seek zeolites since they are usually well crystallized and come in a variety of forms. They're often found in the same environment as other common minerals including datolite, prehnite, apophyllite, amethyst, and other quartz varieties. Zeolites are a particular category of solid substances that have a wide range of uses in manufacturing processes and pollution control. Water and cations may be reversibly extracted or substituted by other cations in the composition of natural zeolite (Rehakova et al., 2004). They can be used in both proactive and reactive decontamination techniques, as well as to clean up different types of chemical spills.

2.4.2 Types of Zeolite

The natural minerals like zeolites are mined all over the world. However, the amount of zeolites used in industrial applications are synthesized. Consequently, the pores of several zeolites are not cylindrical (Beitollah and Sadr, 2009). All zeolites are created equal while developing applications. There are almost 50 kinds of zeolites with different physical and chemical properties (clinoptilolite, chabazite, pyroxene, mordenite, etc.). The main variations are due to the structure of crystal and composition of chemical. The density of particle, selectivity of cation, size of molecular pore, and strength are several of the characteristics that vary based on the zeolite. It is important to understand the form of zeolite being used in order to ensure that it is suitable for one's needs. There are various naturally occurring and synthetic zeolites, each with a specific structure.

2.4.3 Uses of Zeolite

Researcher and mineralogist Mumpton, F. (1999) called natural zeolites as "magic rocks" because they have a wide range of uses, including: agronomic methods for soil improvement, dietary supplements for animal nutrition, use of pesticides and Pesticides are used for crop protection, substrates of hydroponic (Zeoponic), used for plants that can be grow in space flight, and even used to successfully apply healing incisions and wounds and anti-cancer drugs. Zeolite minerals which like other clay minerals, have appealing properties of physical and chemical that enable them to be used as dust applications in Integrated Pest Management (IPM) protocols and organic agriculture to combat biological control pests. Their capability of adsorption, cation-exchange, dehydration-rehydration, and properties of catalysis make them ideal for use in chemistry, biotechnology, control of pollution, pesticides, and medicine. Zeolite is used as a soil acid corrector, a donor of macro and trace elements, a source of maintaining seed quality and germination, a carrier of pesticide active ingredients, biologically active substances, reaction filters, filtering and rheological additives (Adamovich, 2011). As a result, they contribute to agricultural production and have a significant impact on food quality.

2.4.4 Potential of amending MOP with zeolite in Reducing MOP Loss via Leaching

The sandy soil is used to decrease the muriate of potash loss by leaching (Jeffery, 2011). K is considered as the most easily leached cation which due to its displacement to the solution of soil and to its percolation, especially in sandy soils. The changing of physical properties in soil such as infiltration and conductivity of hydraulic, is a technique that influences the movement of water and nutrients in the soil, especially in light soils (Mamedov et al., 2016; Wangetal, 2016a). Low permeability and water retention potential. Another challenge for improving crop productivity is to improve the soil's ability to store nutrients, the most important of which are nitrogen (N), phosphorus (P), sulfur (S) and potassium (K). Higher crop yields can result from the existence of more nutrients in the root zone. As a

result, in order to increase growing rates, the problematic soils must be treated in order to improve irrigation and fertilization performance (Wang et al. 2016b).



CHAPTER 3

METHODOLOGY

3.1 Preparation and Soil Sampling

The samples of soil were taken at 0 to 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 sieved to pass through a 2-mm sieve for 18 pots, respectively for laboratory analysis.

3.2 Soil Analysis

The sample of soil were analysed for soil texture, soil pH, total organic matter, total carbon, total nitrogen, electrical conductivity of soil (EC), cation exchange capacity of soil (CEC), exchangeable K and available P. According to Peech (1965), the pH of soil

was measured in ratio of 1: 10 (soil: water) by using a digital pH meter. Hydrometer method was used to determine the soil texture (Jones, 2001) while loss-on ignition method will be used to measure organic matter content (OM) and total organic carbon (TOC) (Tan, 2005). The Kjeldahl method was applied in order to determination the total N (Bremner, 1965). Besides, double acid method it was described by Mehlich (1953) will be used to extract soil available P and exchangeable K. Afterwards, Laquartwin (K) meter method was used to analysed determination of Potassium in soil leaching. While soil available P were measured by using molybdenumblue method (Murphy and Riley, 1962). The developing of blue colour was analysed by UV-Vis spectrometer at 882 nm wavelengths spectrometer (Thermo Scientific Genesys 20, USA). The method described by Cotteine (1980), which an ammonium acetate leaching method were applied to measure the cation exchange capacity of soil (CEC). The exchangeable NH4+ and available NO3- will be extracted by using a method described by Keeney and Nelson (1982), after the ions were determined by using steam distillation (Tan, 2005). The detailed procedures of each analysis were described as follow.

3.2.1 Soil pH and Electrical Conductivity Determination

To determine the soil pH and EC was used soil water method. 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 50 ml of distilled water was added together with 5 g of air-dried into 100 ml conical flask. The samples were shaken at 180 rpm using a shaker for 15 minutes. After that, the samples were left overnight for 24 hours before using a digital pH meter for pH determination. Then, were filtered the suspension to pass through Whatman filter paper No. 2 and use the filtrate to measure the electrical conductivity by using EC meter.

3.2.2 Soil Texture Determination

According to Bouyoucos, (1962), the soil texture determination was carried out by using hydrometer method. In this method, the texture of the soil was determination 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, 10 mL sodium metaphosphate and 100 mL water were added before it was be blended continuously for 15 minutes. After 15 minutes, the soil suspension it was transferred into a 1000 mL measuring cylinder without leaving any soil left. Next, the distilled water was added until the volume in the cylinder reaches 1130 mL. Then, stir it by using stirring rod to ensure thorough mixing of sediments in the suspension. A hydrometer was placed carefully into a suspension and exactly after 40 seconds after the stirring has stopped, and the reading of the meniscus on the hydrometer stem was recorded. The hydrometer was rinsed and cleaned dry. After that, the suspension was stirred on more time and was repeated the analysis of the 40 seconds. The hydrometer was rinsed and cleaned dry once again. Thoroughly stir the suspension again and the third hydrometer reading was be taken after 2 hours of settling time by following the previous reading method. 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.

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The basic principle of the calculations is:

% sand +% silt +% clay = 100% (x + y) / 2 = a40s reading : % (silt + clay) = (a/50) × 100 = b % sand = 100 - b = c 2 hours reading: % clay = (x/50) × 100 = y % 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. The sample was cooled down using desiccator. First, the initial weight of the crucible was taken. Then, the weight of the crucible was weighed with the 5 g of the soil sample. Afterward, the sample was ashed at 300 °C in the muffle furnace for an hour and temperature was increased 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 OM and C was calculated using the following calculation (Tan, 2003).

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

3.2.4 Cation Exchange Capacity Determination

The cation exchange capacity of soil (CEC) determined by using ammonium acetate (leaching method) (Hailegnaw, 2019). A 10 g of soil sample was weighed and placed into a 250 ml conical flask. Then, a 100 mL of 1M ammonium acetate (NH4OAc) was added into soil and was shaken at 180 rpm for about 5 hours. After that, the soil solution was filtered to pass through Whatman filer paper No. 2, and then the filtrate was discarded. Next, the soil was washed with 20 ml of 95% ethanol and was filtered again, then ethanol was discarded after collection. Later, the soil was filtered with 100 ml of 0.1 M potassium sulphate (K2SO4). The filtered was collected and make the volume of volumetric flask up to 100 ml. Afterward, pipette 10 ml of the sample into distillation apparatus. Then, 10 ml of 40% sodium hydroxide (NaOH) was added into the same beaker. The sample was distilled and the distillate was collected in 10 ml of 2% boric acid indicator solution. During the distillation process, the reddish-purple colour of boric acid was changed to green colour. When twice of the original volume (20 mL) was obtained, the 100 mL conical flask containing the distillate was removed from the distillation apparatus. After that, the distillate was titrated against 0.01 M HCl until the green colour changed to reddish purple. This gives the CEC in cmol kg-1 and the CEC of the soil was calculated by given formula:

Cation exchange capacity of soil (cmol kg⁻¹)

10

3.2.5 Nitrogen Determination

The nitrogen content in soil were determined by using Kjeidahl method (Kjeltec 8200). A 5 g of air-dried was weighed and were placed it into digestion tube. After that, a 5 ml of sulphuric acid (H2SO4 tube) was added and then, followed by 1 g Kjeldahl catalyst tablet into the digestion tube. The digestion was arranged in Kjeldahl digestion block and was digested for 3 hours at 400 °C until the sample become colourless. The sample was cooled down for 15 minutes before adding 30 ml of distilled water. Then, the sample was transferred to 100 ml conical flask after cooling down, and make the volume up to 100 ml. Next, pipette the 10 mL extract into distillation apparatus, and followed by 10 mL of 40% NaOH. Distilled the sample for 3 minutes and collect it in 10 mL of 25% boric acid indicator solution. Then, the reddish-purple colour of boric acid was changed to green colour. After that, the sample was titrated against 0.01 MH2SO4 till the colour changed from green to reddish purple. The calculation for percentage of N in soil was as follows:

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

Where,

V = Volume of $0.01 \text{ M} \text{ H}_2 \text{SO}_4$ or $\text{H}_2 \text{SO}_4$ titrated for the sample (mL)

B = Blank titration volume (mL)

 $M = Molarity of H_2SO_4$ solution

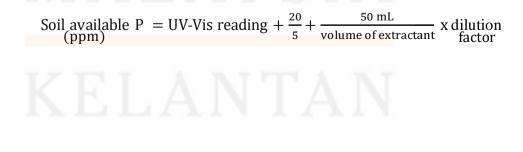
14.01 =Atomic weight for N

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 were extracted by Mehlich No. 1 Double Acid Method (Mehlic, 1953). A 5 g of soil sample was placed into the 250 ml conical flask. Then, a 20 ml double acid (0.05 M HCl + 0.025 M H2SO4) was added into the sample and the solution was shaken it at 180 rpm for 10 minutes. After that, the supernatant was filtered into another beaker using Whatman filter paper No. 2. Next, 8 ml of reagent B was pipette into 50 ml volumetric flask. After that, 0.1 to 5 ml soil extract was added slowly while observe the light blue colour development. Then, distilled water was added until to 50 mL volume once the solution colour was changed to blue. Subsequently, the sample was pipetted into cuvette. UV-Vis reading at wavelength of 882 was recorded and subjected for calculation as follow to obtain the soil available P.



3.2.7 Soil Exchangeable K Determination

The soil exchangeable K were extracted by using Mehlich No. 1 Double Acid Method (Mehlich, 1953). A 10 g of soil sample was placed into the 250 ml conical flask. Then, a 40 ml double acid 0.05 M HC1 + 0.025 M H2SO4) was added and the solution was shaken for 15 minutes. After that, the supernatant was filtered using Whatman Filter Paper No.2, and the extract was aspirated into AAS and the reading was recorded. The soil exchangeable cation will be calculated using the equation below:

Soil exchangeable cation (ppm)

= ASS reading (ppm) + $\frac{\text{Volume of extractant (mL)}}{\text{Weight of soil sample (g)}}$

3.3 Characterization of Zeolite

The zeolite that was used in this study was in powder form. The pH of zeolite was determined in a ratio of 1: 2 soil:distilled water suspension or 1 M KCl solution by using a glass electrode (Peech, 1965). Next, the CEC of zeolite was measured by using the CsCl method which derived by Ming and Dixon (1986) and the exchangeable Mg, Ca, and K of the zeolite were extracted also by using the method of Ming and Dixon (1986). Thus, their concentrations were determine as previously outlined. The MOP and zeolite were put into a plastic vial and shaken for 30 minutes at 190 rpm to ensure a uniform mix.

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). The chemical fertilizers and organic amendments were mixed thoroughly with soil prior to the leaching experiment. 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 based on the standard recommendation for maize cultivation (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 was used. The treatments were arranged in a completely randomized design with three replications (Table 3.1). Treatments evaluated in this leaching experiment was summarized as follows:



Table 3.1: Treatments of the incubation study	Table 3.	1:	Treatments	of	the	incu	bation	study
-----------------------------------------------	----------	----	------------	----	-----	------	--------	-------

Treatments	Descriptions
TO	Soil only
T1	Soil + 2.5g MOP
T2	Soil + 2.5g MOP + 1.5g Zeolite
T3	Soil + 2.5g MOP + 3.0g Zeolite
T4	Soil + 2.5 g MOP + 4.5 g Zeolite
Т5	Soil + 2.5 g MOP + 6.0 g Zeolite

3.5 Post-Treatment Soil Analysis

Water was watered to every pot with soil such that leachates through leaching were collected to determine the K lost from the soil. The volume of water applied was based on rainy days over 30 days. A 5-year rainfall data was obtained from the Malaysian Meteorological Department from which average amount of rainfall per month were applied. Leachates was collected and analysed every 3 days for K and pH. The soil samples were collected at 60 days of leaching. The soil samples were air-dried and analysed for pH, exchangeable acidity, total exchangeable K by using standard procedures outlined previously in Chapter 3.



3.6 Statistical Analysis

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

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CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Results and Discussions

Table 4.1 shows the selected physico-chemical properties of soil. The soil texture was sandy loam with the pH value of 4.0. Based on Table 4.1, the EC for the soil was 13.33 μ S/cm where the soil organic matter, soil total C and soil total N were 5.87%, 3.4% and 0.18%, respectively (Tan, 2003). Plus, the presence of available P in the soil was 4.5 ppm and the exchangeable K was 6.67 ppm.

ΜΔΙΔΥ	SIA
Property	Value obtained
Soil textural class	Sandy loam
рН	4
Electrical conductivity (µS/cm)	13.33
Soil organic matter (%)	5.87

Table 4.1 Selected physico-chemical properties of soil

Soil total C (%)	3.4
Soil total N (%)	0.18
Available P (ppm)	4.5
Exchangeable K (ppm)	6.67

The selected chemical properties of zeolite are shown in Table 4.2. The pH of the zeolite was 6.80 while the CEC was 100.33 cmol₄ kg⁻¹. This indicates that the zeolite possessed a large surface area for cation exchange properties. Besides, the total N, Ca, Mg, K, NH₄+ and NO₃⁻⁻ also presence in this soil which were 1.18 mg kg⁻¹, 18400 mg kg⁻¹, 11200 mg kg⁻¹, 14850 mg kg⁻¹, 12.60 mg kg⁻¹ and 11.58 mg kg⁻¹, respectively (Hailegnaw, 2019).

Property	Value obtained
pH	6.8
CEC (cmol, kg ⁻¹)	100.33
Total N (%)	1.18
Ca (mg kg ⁻¹)	18,400
Mg (mg kg ⁻¹)	11,200
K (mg kg ⁻¹)	14,850
$NH_{4}+(mg kg^{-1})$	12.6
NO_3 (mg kg ⁻¹)	11.58

Table 4.2 Selected chemical properties of zeolite

The K content is one of the most abundant nutrients required by all the plants. The results of K availability in leachate with different rates of zeolite were obtained. A total

of 10 batches of leachates of were tested in 30th days as shown in Figure 4.1. There was a reduction trend of K content in all treatments over 30 days of leaching. Figure 4.1 shows the K content ranged from 5.00 ppm to 9033.33 ppm. Starting day 3 of leaching, K lost in T1 (soil with MOP only) were high compared with other treatments. This indicates that zeolite had high K sorption compared to soil with MOP only which reduces the K leaching over 30 days in the leaching experiment (Swarnima, 2016).

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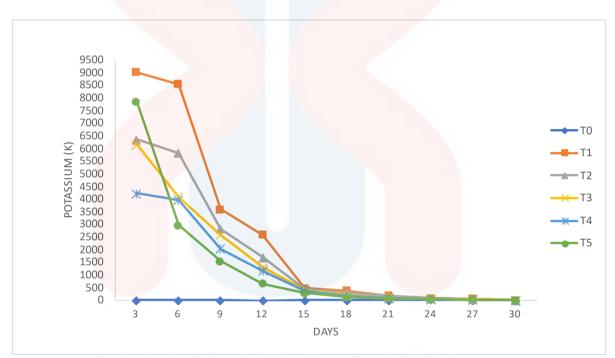


Figure 4.1 K availability in leachate over 30 days of leaching

Apart from that, soil without zeolite (T1) had the highest K leaching in day 3 and days 6 compared the treatments with zeolite (T2-T5). This is because, soil solution K has a high chance of leaching and thus loss from the soil system (Swarnima, 2016). There is low replenish and high loss of K by leaching in acidic soil especially in the highly weathered soils in the tropics.

Besides, the soil with 6.0 g of zeolite (T5) in days 30 obtained the lowest concentration of K. This might be due to the crystals of zeolite minerals that enables the chelation of K on the crystalline structure of zeolite that improved K retention (Coombs et al., 1997).

Figure 4.2 demonstrates the sum of K content of soil after 30 days of leaching. The accumulation of K in leachate was reduced significancy where the rate of zeolite was increased (T4 and T5). Among the treatments, the results in Figure 4.2 showed the K content of soil ranging from 6.97 ppm to 2498.47 ppm. The leached K content in T1 was the highest at 2498.47 ppm (Rehakova et al., 2004).

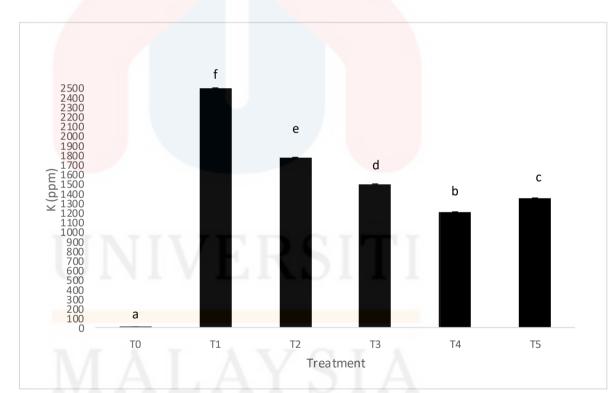


Figure 4.2 Accumulation of K in leachate throught 30 days of leaching

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

The result shows that the accumulation of K in leachate significantly decrease in soil with MOP and zeolite (T2, T3, T4 and T5) than in soil with MOP without zeolite (T1) and soil alone (T0) at 30 days. The lowest concentration of accumulation of K in leachate in T2, T3, T4 and T5 were partly due to increase in the rate of the zeolite as maintain of the nutrient of K in soil. The large surface and crystalline structure of the zeolite were able retch the positively charged K ions and decrease the K leaching. Besides, the crystalline structure is usually full of O – and COO – functional groups which facilitates the chelation process towards the positively charged cations such as K. This observation could be one of the reasons why the T1 was the highest of the other treatment in accumulation of K in leachate due to the presence of soil with MOP without zeolite. This is supported by Swarnima, (2016) other researchers who discovered the K content can be easily lost by the leaching process from the soil system.

Figure 4.3 illustrates the pH value in soil after 30 days of leaching between different treatments. The treatments of MOP amended with zeolite (T2-T5) showed significant high soil pH compared to soil only (T0). The soil pH was increased basically due to the initial pH of the zeolite itself which was 6.80 (Table 4.2). Besides, the increased soil pH was also due to the precipitation of Ca by the zeolite. The higher buffering capacity of the zeolite also explains the increase of soil pH in these treatments with zeolite. Besides, the contents of inherent of Ca and Mg in zeolite might have contributed to the increase in the soil exchangeable of Ca and Mg contents that able to increase the soil pH (Rehakova et al., 2004).

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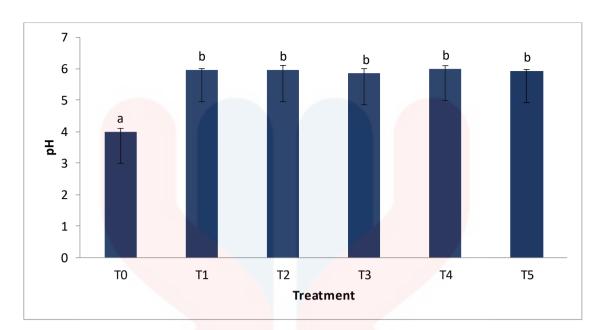


Figure 4.3 pH in soil after 30 days of leaching

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

In a related study, Prasad and Foster claimed that processes of acidification can be balanced by maintaining or enhancing the pH through MOP regular use (Meena, 2015). This explains the no significance difference in the soil pH between T1 (soil and MOP only) and treatments with zeolite (T2-T5).

Figure 4.4 demonstrates the EC in soil after 30 days of leaching. Based on all the treatments, the results in Figure 4.4 shown that the EC in soil after leaching ranging from 10.00 to 33.33. The EC in T3 gave the highest value which is 33.33 and the lowest value in T0 which is 10.00. Therefore, there were significant differences (p<0.05) in terms of EC at all treatments (Jeffery, 2011).



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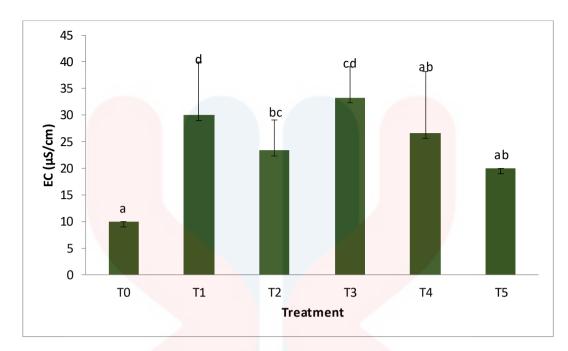


Figure 4.4 EC in soil after 30 days of leaching

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

The K concentration retained in soil after leaching in different treatments is shown in Figure 4.5. Based on Figure 4.5, treatments with the highest rate of zeolite application (T5) were able to retain the higher K concentration in the soil. This was due to high rate of zeolite application which had a higher K sorption and surface area. This enables the adsorption and chelation of K on the zeolite more effective. Meanwhile, T1 (soil and MOP only) had significantly lowest K retention because K is naturally mobile in the soil and the highly weathered acidic soil with low clay content reduced the adsorption of the K on the clay surface that prevent the K for leaching (Swarnima, 2016).



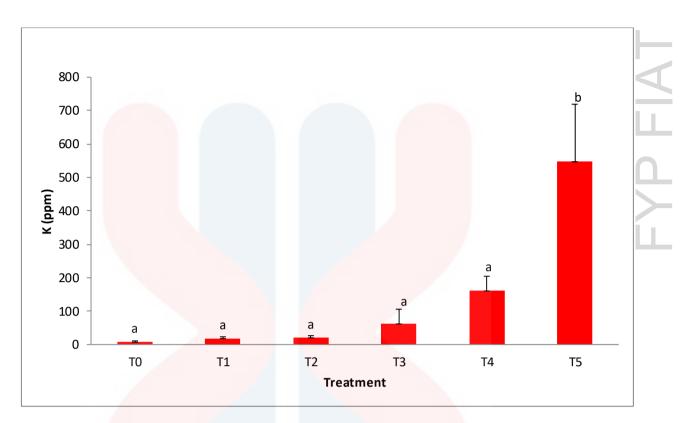


Figure 4.5 K concentration remained in soil after leaching

Mean between columns with different letter (s) indicate significant difference between treatments by Tukey's test at $p \le 0.0$

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

As a conclusion, treatments with zeolite were able to reduce the K leaching from the soil. Application rate of 20t/ha zeolite was found to provide the best retention of K in the soil. The treatments with zeolite application also improved the soil pH to near neutral. This indicates the potential of zeolite application in amending the soil chemical properties. This research is suggested to be carried out in the field with test crops to test the effect of zeolite in retaining the K in the soil in natural environment.



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APPENDIX



Figure A.1: Leaching process



Figure A.2: Filtering the sample soil sample

Table A: Descriptive accumulation ofpotassium

Descriptive Statistics							
Dependent Variable: Potassium							
Std.							
Treatment	Mean	Deviation	N				
T0	6.9667	1.01160	3				
T1	2498.4667	42.99585	3				
T2	1774.8000	10.18676	3				
T3	1497.8000	60.92151	3				
T4	1206.4333	51.80100	3				
T5	1351.2000	30.75110	3				
Total	1389.2778	768.02647	18				

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Table A.1: Tukey: Potassium

Potassium

Tukey HSD^{a,b}

		Subset					
Treatment	N	1	2	3	4	5	6
то	3	6.9667					
T4	3		1206.4333				
T5	3			1351.200	00		
Т3	3				1497.8000		
T2	3					1774.8000	
T1	3						2498.4667
Sig.		1.000	1.000	1.00	00 1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = 1548.973.

a. Uses Harmonic Mean Sample Size = 3.000.

b. Alpha = .05.

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Table B: Descriptive pH

Descriptive Statistics

Dependent Variable: pH					
TREATMENT	Mean Std. Deviation		N		
ТО	4.0000	.91652	3		
T1	5.9667	.05774	3		
Т2	5.9667	.15275	3		
ТЗ	5.8667	.15275	3		
Τ4	6.0000	.10000	3		
Т5	5.9667	.05774	3		
Total	5.6278	.81804	18		

Table B.1: Tukey: pH

рΗ

Tukey HSD ^{a,b}							
			Sub	oset			
TREATMENT	N		1		2		
Т0	3		4.0000				
Т3	3				5.8	667	
T1	3				5.9	667	
T2	3				5.9	667	
T5	3				5.9	667	
T4	3				6.0	000	
Sig.			1.000			998	

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = .151.

a. Uses Harmonic Mean Sample Size = 3.000.

b. Alpha = .05.

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Table C: Descriptive EC

Descriptive Statistics

Dependent Variable: EC						
TREATMENT	Mean Std. Deviation		N			
ТО	10.0000	.00000	3			
T1	35.0000	5.00000	3			
Т2	23.3333	5.77350	3			
ТЗ	33.3333	5.77350	3			
T4	20.0000	.00000	3			
Т5	20.0000	.00000	3			
Total	23.6111	9.36287	18			

Table C.1: Tukey: EC

EC

Tukey HSD^{a,b}

			Sub	oset	
TREATMENT	Ν	1	2	3	4
ТО	3	10.0000			
Т4	3	20.0000	20.0000		
Т5	3	20.0000	20.0000		
T2	3		23.3333	23.3333	
ТЗ	3			33.3333	33.3333
T1	3				35.0000
Sig.		.073	.894	.073	.994

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = 15.278.

a. Uses Harmonic Mean Sample Size = 3.000.

b. Alpha = .05.

Table D: Descriptive K

Dependent Variable: K

	Mean	Std. Deviation	N
0	8.0000	3.00000	3
1	18.6667	4.50925	3
2	21.0000	5.56776	3
3	62.6667	43.14317	3

4		161.3333	4	3.08519	3
5		548.0000	17	1.21916	3
Total		136.6111	20	6.31220	18
- O tai				0.01220	10
Table D.1: T	ukev• K				
Table D.I. I	uncy. IX				
		К			
		N			
Tukey HSD ^{a,b}					
			Sul	oset	
	N		Sul	oset 2	
	N		1		
0	N	3			
0	N	3	1		
1	N	3	1 8.0000 18.6667		
	N		1 8.0000		
1	N	3	1 8.0000 18.6667		

3

3

161.3333

.190

548.0000

1.000

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = 5515.667.

a. Uses Harmonic Mean Sample Size = 3.000.

b. Alpha = .05.

4

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