



UNIVERSITI PUTRA MALAYSIA

***ZINC-LAYERED HYDROXIDE WITH NITRATE AND PHOSPHATE AS
COUNTER ANIONS AND ITS EFFECT ON GROWTH PERFORMANCE
OF KELAMPAYAN SEEDLINGS [Neolamarckia cadamba (Roxb.)
Bossert]***

NOR FARHANA BINTI KHADIRAN

ITMA 2021 11



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By

NOR FARHANA BINTI KHADIRAN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of Master of Science**

February 2021

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

ZINC-LAYERED HYDROXIDE WITH NITRATE AND PHOSPHATE AS ANIONS AND THEIR EFFECT ON THE GROWTH PERFORMANCE OF KELAMPAYAN SEEDLINGS [*Neolamarckia cadamba* (Roxb.) Bosser]

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February 2021

Chair : Prof. Mohd Zobir Hussein, PhD
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Currently, layered hydroxide is subjected to intense research as this type of 2-dimensional (2D) nanomaterial can be used in various aspects of agriculture, particularly as a slow-release matrix of nutrients for plant growth. In this work, zinc layered hydroxide was synthesized by co-precipitation and anion exchange method which produces zinc layered hydroxide nitrate (ZLHN) and zinc layered hydroxide phosphate (ZLHP) nanofertilizer, respectively to evaluate the effect of nitrate and phosphate sorption efficiency towards plant growth. ZLHN and ZLHP showed the formation of a new peak of the basal interlayer spacing of 9.57 Å and 6.78 Å in the powder X-ray diffraction (PXRD) pattern, respectively. The FTIR, thermal, elemental, surface area and morphology analysis supported the formation of ZLHN and ZLHP nanofertilizer. The synthesized ZLH fertilizer was also tested for release kinetic studies while the bioassay was performed on Kelampayan (*Neolamarckia cadamba*) seedlings. The controlled release studies of ZLHN and ZLHP were carried out in deionized water, 0.005M sodium carbonate and soil media solution at pH 6.5. The highest percentage release was recorded to be 63.87% of nitrate in 0.005M sodium carbonate solution and 6.4% of phosphate in deionized water while the lower percentage release was recorded at 29.61% of nitrate and 0.21% of phosphate in soil media solution at 96 hours, respectively. The release profile was best fitted to the pseudo-second-order model with R² values of 0.99 and 0.97 for ZLHN and ZLHP nanohybrid, respectively. The ZLH nanofertilizer were found to be non-toxic to plant, where all the green bean seeds were successfully germinated in the experiment. The percentage of seed germination can be arranged in order of ZLH nanofertilizer > commercial fertilizer > primary material treatment. Besides, after sixteen weeks of treatment of ZLH nanofertilizer toward the growth of kelampayan seedlings showed good performance and significant difference in diameter and height compared to other treatments. At the end of the study, the nutrient plant uptake was also determined and showed higher uptake in the leaves of Kelampayan treated with ZLH nanofertilizer and they are significantly higher compared to the other treatments. In conclusion, this study suggests that the development of ZLH nanofertilizer has the potential to be used as fertilizer nanodelivery systems.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

ZINK HIDROKSIDA BERLAPIS DENGAN NITRAT DAN FOSFAT SEBAGAI ANION PENGIMBANG DAN KESANNYA TERHADAP PRESTASI TUMBESARAN ANAK POKOK KELAMPAYAN
[*Neolamarckia cadamba* (Roxb.) Bosser]

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Pada masa kini, hidroksida berlapis dikaji secara intensif kerana bahan nano daripada jenis 2-dimensi (2D) ini dapat digunakan dalam pelbagai aspek pertanian, terutamanya sebagai matrik pelepasan nutrisi perlahan untuk pertumbuhan tanaman. Dalam kajian ini, hidroksida berlapis zink telah disintesis dengan keadah pemendakan bersama dan pertukaran anion untuk menghasilkan baja nano zink berlapis hidroksida nitrat (ZLHN) dan zink berlapis hidroksida fosfat (ZLHP), masing-masing digunakan untuk menilai kesan kecekapan penyerapan nitrat dan fosfat terhadap pertumbuhan tanaman. ZLHN dan ZLHP menunjukkan pembentukan puncak baru dengan jarak antara lapisan masing-masing ialah 9.57 Å dan 6.78 Å dalam corak pembelauan sinar-X mereka. Analisis FTIR, terma, kandungan organik-tak organik, luas permukaan dan morfologi menyokong penghasilan baja nano ZLHN dan ZLHP. Baja ZLH yang disintesis juga telah diuji untuk mengkaji pelepasan kinetik nutrisi manakala biocerakinan dilakukan pada anak benih Kelampayan (*Neolamarckia cadamba*). Kajian pelepasan terkawal ZLHN dan ZLHP dilakukan dalam air deionisasi, larutan natrium karbonat 0.005 M dan larutan media tanah pada pH 6.5. Pelepasan peratusan tertinggi dicatatkan sebagai 63.87% nitrat dalam larutan natrium karbonat 0.005 M dan 6.4% fosfat dalam air deionisasi sementara pelepasan peratusan yang lebih rendah dicatat pada 29.61% nitrat dan 0.21% fosfat dalam larutan media tanah pada 96 jam. Profil pelepasan paling sesuai ditunjukkan pada model kinetik order pseudo-kedua dengan nilai R^2 0.99 dan 0.97 masing-masing untuk "nanohybrid" ZLHN dan ZLHP. Nano-baja ZLH didapati tidak beracun terhadap pokok, di mana semua biji kacang hijau berjaya bercambah dalam proses penyemaian. Peratusan percambahan biji boleh disusun mengikut urutan baja nano ZLH>baja komersial>bahan utama. Selain itu, setelah enam belas minggu rawatan baja nano ZLH terhadap pertumbuhan anak benih kelampayan, data menunjukkan prestasi yang baik dan perbezaan yang ketara dari segi ketinggian dan ukur lilit batang berbanding rawatan lain.

Pada akhir kajian, pengambilan nutrisi oleh anak benih juga telah di analisis dan menunjukkan pengambilan yang lebih tinggi pada daun kelampayan yang dirawat dengan baja nano ZLH dan jauh lebih tinggi dibandingkan dengan rawatan lain. Kesimpulannya, kajian ini menunjukkan bahawa penghasilan baja nano ZLH berpotensi untuk digunakan sebagai sistem penyampaian baja nano.



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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

LMH	Layered metal hydroxide
LDH	Layered double hydroxide
ZLH	Zinc layered hydroxide
ZLHN	Zinc layered hydroxide nitrate
ZLHP	Zinc layered hydroxide phosphate
ANOVA	Analysis of variance
2D	2 dimension
PXRD	Powder X-Ray diffraction
FTIR	Fourier transform infrared spectroscopy
TGA/DSC	Thermogravimetric and differential scanning calorimetry
FESEM	Field emission scanning electron microscope
CRBD	Complete randomized block design

CHAPTER 1

INTRODUCTION

1.1 Background of study

Lately, nanotechnology becomes one of the active fields of research (Moore, 2006; Sergeev, et al., 2008) due to its promising in opening a huge number of opportunity in many fields like medicine (Hesse, et al., 2012; Kura, et al., 2013), pharmaceuticals (Silva, et al., 2019), electronics (Chen, et al., 2013; Maduraiveeran, et al., 2017) and agriculture (Zulfiqar, et al., 2019; Raliya, et al., 2018). The term “Nanotechnology” has been defined by Norio Taniguchi from the Science University of Tokyo in 1974 (Qureshi, et al., 2018) which studied manipulating the atomic and molecular size. Nanotechnology involved the design, synthesis and properties determination and the application of structures, devices and systems that will be controlled by the size and shape at the nanometer scale (Madhuri, et al., 2012), where the size of nanoparticles is in the range between 1-100 nm (Thakkar, et al., 2010; Deepa, et al., 2016). It has a high surface area (Khan, et al., 2019) and unique properties in optical, physical and biological properties (Rawtani, et al., 2018; Zhang, et al., 2017) which promising significant contribution to agricultural problems such as controlled delivery of fertilizer (Ghormade, et al., 2011), etc.

Plants need enough water and nutrients to live, stand with their species and biodiversity (Songkhum, et al., 2018) also increase crop productivity (Wissuwa, 2003). Most of the plants faced with wide challenges such as water availability, multi-nutrient deficiency (Godfray, et al., 2010) and low nutrient use efficiency (NUE) (Marchiol, 2018) due to the properties of soil, leaching and characteristics of fertilizer (Baligar, et al., 2015). Based on the data compiled by the Food and Agriculture Organization of the United Nations (FAO), the reduction of water and nutrients uptake would affect the production of agricultural products to meet the needs of the world due to the increasing population (York, 2017).

In the past decades, chemical fertilizer containing multi-nutrients such as N, P, K which were needed by the plants were used, and played an important role in increasing crop yield and maintaining enough food supplies (Chaudhary, et al., 2017; Meena, et al., 2016). Unfortunately, it created a negative impact on agro-ecosystem (Ding, et al., 2017), due to the high nutrient release into the ground and topwater, which also causes the emission of gases into the atmosphere (Drechsel, et al., 2015). These phenomena would cause the appearance of many problems including atmospheric (Zahoor, et al., 2014) and groundwater pollution (Datta, et al., 1997), soil acidification, eutrophication and loss of biodiversity (Mahanty, et al., 2017). Therefore, to overcome these limiting factors, a new strategy of nanotechnology attracts more attention, creating new nano-fertilizer which were used to increase the efficiency of nutrients uptake by plants and decreasing the loss of nutrients to the environment. The nano-fertilizer also provide control and slow release of nutrient (Jyothi, et al., 2017) allowing lower used of dosage (Preetha, et al., 2017).

The combination of nanotechnology and fertilizer, the so-called nano-fertilizer has changed the world of fertilizer in agriculture practices by creating new opportunities for advancing the nutrient uptake of plants. Nano-fertilizers is a product made using nanoparticles to improve the efficiency of nutrient uptake. The nano-fertilizer can be classified into nanoscale fertilizer (nanoparticles containing nutrients), nanoscale additives (traditional fertilizer with nanoscale additives and nanoscale coating (traditional fertilizer coated with nanoparticles) (Mikkelsen, 2018). These nano-fertilizer creating more penetration capacity, high surface area and high efficiency and eco-friendly (Qureshi, et al., 2018).

Layered metal hydroxide is the latest and advanced way of creating a nanofertilizer that can slowly supply nutrients to crops. Layered metal hydroxide is classified into two; layered double hydroxide (LDH) (Halajnia, et al., 2013) and layered hydroxide salts (LHs). Nowadays, the layered double hydroxide in the world of fertilizer (nitrogen, phosphate and potassium) were exploited followed by slow and controlled release properties (Novillo, et al., 2014) as a nanolayered-hosts, but in this study, the layered hydroxide salt (zinc layered hydroxide) was used as a host, and also was introduced as one of the alternatives to solve the problems faced by the conventional fertilizer.

1.2 Problem statement

Fertilizer is a chemical compound that is applied and used to help improve the performance of plant and fruit growth. Fertilizer is usually applied either through the soil or by foliar feeding which will help the root and leaves nutrient uptake, respectively. Population growth in the coming years will give a direct impact on the world of agriculture. Agriculture is an important source of nutrients for humans, like plants. Fertilizers are formulated in appropriate concentrations and involving the combinations of three main nutrients which are nitrogen which promotes leaf growth and forms protein and chlorophyll, phosphorus which contributes to roots and fruit development and potassium contributes to stem and root growth and synthesis of protein for various crop and produce good growing conditions. (Corradini, et al., 2010). However, the fertilizer applied in the plant will lose about 40-70% nitrogen, 80-90% phosphorus and 50-7-% of potassium to the environment and cannot easily be absorbed by plants. This phenomenon will leave very serious environmental pollution problems (Corradini, et al., 2010). The growth of the plants and their quality usually depend on the fertilizer quality and amount of water used (Liu, et al., 2008). Based on that, various methods which are involving the latest technology with innovative techniques for increasing crop yield by using environmentally clean chemistry which is also known by several authors as “green chemistry” (Praveen, et al., 2012).

Nowadays, the highest attention from researchers has been paid to layered hydroxide salt (LHS) and some studies have been demonstrated the use of these nanomaterials as the host matrices for storage and slow release of biologically active products which are also known as a guest (Benicio, et al., 2015). This is because of the good properties of the LHS as the host, and it is biodegradable and easy to prepare (Mohsin, et al., 2013). In addition, the host is commonly used for control and slow-release study in many applications. The slow-release properties are important in maintaining nutrient

availability by releasing critical nutrients for effective plant growth gradually (Madzokere, et al., 2020). The main reason for the intercalation activity of the guest anions into the interlayer galleries of layered hydroxide is to minimize the exposure of the guest anions which act as nutrients into the soil media. Here the work is focused on a review of layered metal hydroxide (LMH) such as zinc layered hydroxide which is acts as nanomaterials with potential use for agriculture for plant nutrition. To date, there is no intercalation of nitrate and phosphate into zinc layered hydroxide has been reported. The slow-release study was done in order to understand the release behavior of nitrate and phosphate in different media. The slow release of nitrate and phosphate is hoped to be effective and environmentally friendly.

1.3 Significant of the study

This research is focused on the synthesis of zinc-layered hydroxide, where nitrate and phosphate are used as the counter anions using zinc nitrate hexahydrate, $Zn(NO_3)_2 \cdot 6H_2O$ and potassium dihydrogen phosphate, KH_2PO_4 as the starting salts. The use of zinc-layered hydroxide as eco-friendly, low nutrients consumption, prolongs the release of the active agent at an appropriate rate and in the low-cost production process (Ahmed, et al., 2016). In planting application, the ZLH nanofertilizer has the potential for reducing the release amount of nutrients presented in nanofertilizer making the nanofertilizer more available to nanoscale kelampayan tree pores resulting in the high nutrient use efficiency (Dwevedi, et al., 2016).

1.4 Objectives

The objectives of this study are outlined as listed below:

- 1) To synthesis zinc layered hydroxide (ZLH) nanohybrid which is used as a host to containing two plant nutrients; nitrogen and phosphorous and deliver them for better plant uptake.
- 2) To characterize the physico-chemical properties of zinc layered hydroxide fertilizer nanohybrid and study the release behavior of the Nitrogen and Phosphorous from the synthesized nanohybrid.
- 3) To evaluate the suitability of this new fertilizer formulation toward plant growth and nutrient plant uptake.

1.5 Limitations

Although this research has reached its aims, there were some notable limitations:

- 1) The sample powder of ZLHN and ZLHP (ZLH nanofertilizer) were produced in small volume for each synthesis.
- 2) Each synthesized sample powder of ZLH nanofertilizer goes to XRD analysis for passing some criteria before applied to Kelampayan seedlings.
- 3) The Kelampayan seedlings growth study in a short duration due to the limited time to use the facilities of FRIM's nursery which followed the date of the project study.

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