### **UNIVERSITI TEKNOLOGI MARA**

# SYNTHESIS, CHARACTERIZATION AND CONTROL RELEASE PROPERTIES OF SINGLE METAL ZINC LAYERED HYDROXIDE-2-NAPTHOXYACETIC ACID (BNOA) NANOCOMPOSITE

### HASNATUL FITRIAH BINTI ABD RAHIM

Thesis submitted in fulfilment of the requirements for the degree of **Master of Science** (Applied Chemistry)

**Faculty of Applied Sciences** 

March 2024

#### ABSTRACT

The utilization of nanocarriers in the development of environmentally friendly herbicides has led to the creation of controlled release formulations. These formulations offer precise concentration and timed-release properties. To achieve this, zinc chloride (ZnCl<sub>2</sub>) and zinc nitrate hexahydrate (Zn (NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O) were used as precursors to synthesize zinc layered hydroxide-chloride (ZLC) or zinc layered hydroxide-nitrate (ZLN). These compounds were then intercalated with 2-napthoxyacetic acid (BNOA), an herbicide, resulting in nanocomposites called Zinc layered hydroxide-2napthoxyacetic acid (ZLCB) and (ZLNB) through ion exchange. The formation of these nanocomposites was analysed using Powder X-Ray Diffraction (PXRD), which revealed an increase in the basal spacing from 7.9Å to 27.3Å in ZLCB and from 9.8Å to 28.2Å in ZLNB, indicating successful intercalation of the anions into the interlayer of the host material. Fourier Transform Infrared Spectroscopy (FTIR) confirmed the presence of BNOA anions within the interlayers of ZLCB and ZLNB by detecting the formation of COO<sup>-</sup> ions (carboxylate ions) a new peak at approximately 1611 cm<sup>-1</sup> and 1616 cm<sup>-1</sup> respectively. The surface area of ZLCB and ZLNB also increased from 6.61  $m^2g^{-1}$  to 27.47  $m^2g^{-1}$  and from 9.16  $m^2g^{-1}$  to 28.08  $m^2g^{-1}$  respectively, indicating the successful insertion of BNOA into the interlayers of ZLC and ZLN. Additionally, the loading percentage of BNOA, estimated using a CHNS analyser, was found to be 83.9% for ZLCB and 41.80% for ZLNB. The controlled release properties demonstrated that the release of BNOA in various aqueous solutions followed the order of  $Na_3PO_4 >$  $Na_2SO_4 > NaCl$  and confirmed pseudo-second order kinetic models for both nanocomposites.

### ACKNOWLEDGEMENT

In the name of Allah, the Most Gracious and Merciful,

First and foremost, I would like to express my deepest gratitude to Assoc. Prof. Dr. ChM. Hamizah Mohd Zaki for her guidance, assistance, encouragement, and unwavering support. Without her, this thesis would not have been possible. Throughout the years, I have gained a profound understanding of the project through her teachings and guidance, and, most importantly, I have been inspired by her exemplary leadership and wisdom. I am equally grateful to Assoc. Prof. Dr. Siti Halimah Sarijo for her kind assistance, wise advice, and constant encouragement and support. It has been an honour for me to collaborate with both during these remarkable years.

I would also like to extend a special appreciation to my beloved parents, Abd Rahim Husain, and , for their unwavering faith in me throughout this journey. Additionally, I am grateful to my dear siblings, Muhammad Hakimi Abd Rahim, and Hasnatul Asyiqin Abd Rahim, for their constant support. Furthermore, I would like to express my heartfelt gratitude to my dear friends, Nur Asyiqin Azman, Nur Syuhada Mohd Haeizar, and Mohamad Nor Amirul Azhar Kamis, who have always stood by my side throughout this journey, providing words of encouragement and helping me in numerous ways.

Lastly, I would like to thank Universiti Teknologi MARA for granting me the opportunity to pursue my studies and for providing the LESTARI grants that have supported me throughout this journey.

## TABLE OF CONTENTS

CONFIRMATION BY PANEL OF EXAMINERS			ii				
AUTHOR'S DECLARATION ABSTRACT ACKNOWLEDGEMENT TABLE OF CONTENTS LIST OF TABLES LIST OF FIGURES LIST OF SYMBOLS LIST OF ABBREVIATIONS			iii iv v vi ix x xii xiii				
				CHAPTER 1 INTRODUCTION			14
				1.1	Resear	rch Background	14
				1.2	Problem Statement		16
				1.3	Objective of Study		17
				1.4	Signif	icance of Study	17
CHAPTER 2 LITERATURE REVIEW			19				
2.1	Agrochemicals Technology		19				
2.2	Plant Growth Regulator		20				
2.3	Auxin	Auxins					
	2.3.1	Natural and Synthetic Auxins	24				
	2.3.2	2-napthoxyacetic Acid (BNOA)	27				
2.4	Single	Metal Layered Hydroxide (SMLH)	28				
2.5	Structure of Single Metal Layered Hydroxide (SMLH)		29				
	2.5.1	The value of x	31				
	2.5.2	The nature of $M^{2+}$ and $M^{3+}$	31				
	2.5.3	Types of anions	32				
2.6	Intercalation of Single Metal Layered Hydroxides (SMLH) with Guest						
	Anions		33				

## CHAPTER 1 INTRODUCTION

#### 1.1 Research Background

The agriculture sector faces significant challenges in optimizing production while maintaining environmental sustainability. Excessive and uninformed use of fertilizers and pesticides has resulted in residues and toxins contaminating soil, water sources, and the air. This has led to increased costs for fertilization, irrigation, and energy, as well as negative impacts on agricultural land worldwide. However, nanotechnology provides an innovative and resourceful platform to enhance the efficiency of agricultural materials and address environment issues (Mao et al., 2022).

Linear expansion of food demand over time due to population growth requires enhanced agricultural productivity, including the development of improved pesticides, herbicides, and plant hormones. With that, plant growth regulators (PGRs), such as Auxins, are used to modify plant growth and optimize agricultural practices. PGRs like acetic acid (IAA), indole-3-butyric acid (IBA), 4-chloro-2-methylphenoxyacetic acid (MCPA), 2,4-dichlorophenoxyacetic acid (2,4-D), and 2-naphthoxyacetic acid (BNOA) have been commonly used in the agrochemical industry (Sparks & Lorsbach, 2017).

Nano fertilizers, as alternatives to conventional fertilizers, can reduce nutrient accumulation in the soil, minimize eutrophication, and prevent pollution of drinking water by enabling controlled release of nutrients in required amounts. Layered Double Hydroxide (LDH) or Single Metal Layered Hydroxide (SMLH) are promising options for the sustainable and efficient release of fertilizers (Grover et al., 2022; Johnston et al., 2021). Nonetheless, further research is needed to explore the interactions between these layered nanomaterials and different functional classes of PGRs to enhance controlled release formulation.

In this study, Zinc Layered Hydroxide-Chloride (ZLC) and Zinc Layered Hydroxide-Nitrate (ZLN) will be utilized to intercalate selected Auxins as guest anions. The intercalation of Auxins, 2-naphthoxyacetic acid (BNOA) with SMLH offers significant advantages for controlled release formulations in agriculture. Firstly, this process enhances the stability and protection of auxins, ensuring it prolonged activity and reducing degradation. The layered structure of zinc hydroxide provides a physical