

**SYNTHESIS AND CHARACTERIZATION OF  
2,4-DICHLOROPHENOXYACETIC ACID INTERLEAVED WITH  
CALCIUM-ALUMINIUM LAYERED DOUBLE HYDROXIDE BY  
COMPARISON OF CO-PRECIPITATION AND ION-EXCHANGE  
METHOD**

**FATIN HAIZIRA BINTI MOHD HAYAZI**

**Final Year Project Report Submitted in  
Partial Fulfilment of the Requirements for the  
Degree of Bachelor of Sciences (Hons.) Chemistry  
in the Faculty of Applied Sciences  
Universiti Teknologi MARA**

**JANUARY 2019**

This Final Year Project Reported entitled “**Synthesis and Characterization of 2,4-Dichlorophenoxyacetic Acid Interleaved with Calcium-Aluminium Layered Double Hydroxide by Comparison of Co-Precipitation and Ion-Exchange Method**” was submitted by Fatin Haizira binti Mohd Hayazi, in partial fulfilment of the requirements for the Degree of Bachelor of Science (Hons.) Chemistry, in the Faculty of Applied Science, and was approved by

---

Ts Dr Tuan Sheikh Ahmad Izaddin bin Sheikh Mohd Ghazali  
Supervisor  
B. Sc. (Hons.) Chemistry  
Faculty of Applied Sciences  
Universiti Teknologi MARA  
72000 Kuala Pilah  
Negeri Sembilan

---

Nurul Huda Abdul Halim  
Project Coordinator  
B. Sc. (Hons.) Chemistry  
Faculty of Applied Sciences  
Universiti Teknologi MARA  
72000 Kuala Pilah  
Negeri Sembilan

---

Mazni Musa  
Head of Programme  
B. Sc. (Hons.) Chemistry  
Faculty of Applied Sciences  
Universiti Teknologi MARA  
72000 Kuala Pilah  
Negeri Sembilan

Date: \_\_\_\_\_

## TABLE OF CONTENTS

	<b>Page</b>
<b>ACKNOWLEDGEMENTS</b>	<b>iii</b>
<b>TABLE OF CONTENTS</b>	<b>iv</b>
<b>LIST OF TABLES</b>	<b>vi</b>
<b>LIST OF FIGURES</b>	<b>vii</b>
<b>LIST OF ABBREVIATIONS</b>	<b>ix</b>
<b>ABSTRACT</b>	<b>xi</b>
<b>ABSTRAK</b>	<b>3</b>
<b>CHAPTER 1 INTRODUCTION</b>	<b>4</b>
1.1 Background of study	4
1.2 Problem Statement	10
1.3 Significance of study	12
1.4 Objectives of study	14
<b>CHAPTER 2 LITERATURE REVIEW</b>	<b>15</b>
2.1 Background of Layered Double Hydroxide	15
2.2 Structure of Layered Double Hydroxide	15
2.3 Interleaving Process of Layered Double Hydroxide	17
2.4 Preparation of Layered Double Hydroxide	18
2.5 2,4-Dichlorophenoxyacetic Acid	21
2.6 Application of Layered Double Hydroxide	22
<b>CHAPTER 3 METHODOLOGY</b>	<b>26</b>
3.1 Material and Chemical	26
3.2 Apparatus	26
3.3 Synthesis of Calcium-Aluminium Layered Double Hydroxide	26
3.4 Synthesis of 2,4-D-Ca-Al-LDH by Co-Precipitation Method	28
3.5 Synthesis of 2,4-D-Ca-Al-LDH by Ion-Exchange Method	29
3.6 Characterization of 2,4-D-Ca-Al-LDH	30
<b>CHAPTER 4 RESULTS AND DISCUSSION</b>	<b>33</b>
4.1 Powder X-Ray Diffraction Analysis (PXRD)	33

4.2 Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR)	42
<b>CHAPTER 5 CONCLUSION AND RECOMMENDATIONS</b>	<b>50</b>
5.1 Conclusion	50
5.2 Recommendations	51
<b>CITED REFERENCES</b>	<b>52</b>
<b>APPENDICES</b>	<b>59</b>
<b>CURRICULUM VITAE</b>	<b>78</b>

## ABSTRACT

### SYNTHESIS AND CHARACTERIZATION OF 2,4-DICHLOROPHENOXYACETIC ACID INTERLEAVED WITH CALCIUM ALUMINIUM LAYERED DOUBLE HYDROXIDE

By using co-precipitation method, the herbicide named 2,4-dichlorophenoxyacetic acid or known as 2,4-D was successfully interleaved with Ca-Al layered double hydroxide forming nanocomposite of 2,4-D-Ca-Al-LDH. There were two methodologies used which were co-precipitation method and ion-exchange method. Co-precipitation method has shown a successfully interleaved of the anion with the host compared to the ion-exchange method. The interleaved of 2,4-D with Ca-Al LDH happened at 0.05 M and can be confirmed by Power X-Ray Diffraction (PXRD) and Attenuated Total Resonance Fourier Transform Infrared (ATR FTIR). From the PXRD results, the successful intercalation happened when the basal spacing increased from 8.67 Å to 17.59 Å and a new sharp and intense peak was formed due to the insertion of guest anion which was 2,4-D into the interlayer region of Ca-Al LDH. Results from PXRD was supported by FTIR spectrum, where the nitrate peak that was at 1350.01 cm<sup>-1</sup> was disappeared that proved the intercalation was successfully achieved. The peak at 1643.17 cm<sup>-1</sup> to 1625.73 cm<sup>-1</sup> indicated the presence of C=O stretching and a new peak value due to the asymmetric stretching vibration of COO<sup>-</sup> after interleaving process of 2,4-D into the inter gallery of Ca-Al LDH. From this research, the 2,4-D herbicide has been successfully interleaved into the interlayer region Ca-Al LDH using co-precipitation method at 0.05 M which is the optimum concentration for the successful intercalation of 2,4-dichlorophenoxyacetic acid into Ca-Al LDH.