

**UNIVERSITI TEKNOLOGI MARA**

**SYNTHESIS, CHARACTERIZATION  
AND SUSTAINED RELEASE  
STUDIES OF UV ABSORBER  
INTERCALATED INTO LAYERED  
METAL HYDROXIDES**

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**MSc**

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## AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.


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## ABSTRACT

Synthesis, characterization and sustained release studies of ultraviolet (UV) absorber intercalated into layered metal hydroxides aims were to synthesize cinnamic acid into LDH by using co-precipitation method and intercalation of salicylic into LDH by anion exchange method. Co-precipitation (direct method) was used for encapsulation of salicylic and cinnamic acid between the galleries of Zinc Layered Hydroxide (ZLH). Various concentrations were used ranging from 0.03 M, 0.05 M, 0.07 M, 0.1M, 0.2 M, 0.25 M, 0.35 M, 0.4 M 0.6 M to 0.8 M for cinnamic acid (CA) and salicylic acid (SA) and the pH  $7 \pm 0.5$  was kept constant throughout the synthesis process. Release behaviours were determined using 0.05 M sodium chloride (NaCl), sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) and sodium phosphate ( $\text{Na}_3\text{PO}_4$ ) as aqueous media for 60000 minutes or until reach an equilibrium. The release profile was fitted into three types of kinetic models such as Zeroth Order, First Order and Pseudo-Second Order. The Powder X-Ray Diffraction (PXRD) data shows a well phase with high crystallinity of layered double hydroxide-cinnamic (LDCA), layered double hydroxide-salicylic (LDSA), zinc layered hydroxide-cinnamic (ZLCA) and zinc layered hydroxide-salicylic (ZLSA) intercalated compound. Increase in basal spacing from 9.0 Å of LDH-host to 17.7 Å for LDCA and 16.3 Å for LDSA. Disappearance of peak ranging between  $2\theta = 30^\circ$  to  $60^\circ$  with presence of new peak at 24.4 Å for ZLCA and 16.15Å for ZLSA deduce successful intercalation. The results were supported with the data obtained from the whole analysis using FTIR ASAP, ICP-OES, FESEM, TGA-DTG, UV-Vis andCHNS. All the nanocomposites that were synthesized observed to have Type IV which were mesoporous type material with calculated percentage loading of 23.57%, 24.40%, 40.07% and 35.35% for LDCA, LDSA, ZLCA and ZLSA respectively. Encapsulation of cinnamic acid and salicylic acid into the interlayer galleries of LDH and ZLH able to control the release of anion from its interlayer region. The data obtained from UV-Vis shows the release profile of cinnamic and salicylic acid were in the order of  $\text{Na}_3\text{PO}_4 > \text{Na}_2\text{CO}_3 > \text{NaCl}$  with percentage release of 89%, 63%, 43% for LDCA, 89%, 70%, 40% in LDSA, with 95%, 80%, 65% for ZLCA and 80%, 69%, 45% for ZLSA respectively. Pseudo-Second Order were the best kinetic model with  $R^2 > 0.8$  explaining the release of cinnamic acid and salicylic acid from the LDH and ZLH interlayer region. This study shows that LDH and ZLH can be used as the matrix for the sustained release formulation ultraviolet absorber of trans-cinnamic acid and salicylic acid compound.

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## TABLE OF CONTENT

	<b>Page</b>
<b>CONFIRMATION BY PANEL OF EXAMINERS</b>	<b>ii</b>
<b>AUTHOR'S DECLARATION</b>	<b>iii</b>
<b>ABSTRACT</b>	<b>iv</b>
<b>ACKNOWLEDGEMENT</b>	<b>v</b>
<b>TABLE OF CONTENT</b>	<b>vi</b>
<b>LIST OF TABLES</b>	<b>x</b>
<b>LIST OF FIGURES</b>	<b>xi</b>
<b>LIST OF SYMBOLS</b>	<b>xiv</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xv</b>
<b>LIST OF CHEMICAL FORMULAS</b>	<b>xvii</b>
<b>CHAPTER ONE INTRODUCTION</b>	<b>1</b>
1.1 Background of Study	1
1.1.1 Nanocomposites	1
1.1.2 Layered Compound	2
1.2 Ultraviolet radiation	3
1.3 Ultraviolet Absorber	5
1.3.1 Inorganic Particulate	5
1.3.2 Organic absorber	5
1.4 Sustained Release	6
1.5 Problem Statement	6
1.6 The Significance of Study	8
1.7 Objectives	9
1.8 Scope and Limitations	10
<b>CHAPTER TWO LITERATURE REVIEW</b>	<b>11</b>
2.1 Introduction	11
2.1.1 Brucite Mg(OH) <sub>2</sub>	11
2.2 Layered Metal Hydroxide	13