



UNIVERSITI PUTRA MALAYSIA

***CHARACTERIZATION AND CONTROLLED RELEASE
STUDIES OF 4-(2,4 DICHLOROPHENOXY) BUTYRATE
AND 2-(3-CHLOROPHENOXY) PROPIONATE
NANOHYBRIDS***

NOR SHAZLIRAH SHAZLYN BINTI ABDUL RAHMAN

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NANOHYBRIDS**

By

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

CHARACTERIZATION AND CONTROLLED RELEASE STUDIES OF 4-(2,4-DICHLOROPHENOXY) BUTYRATE AND 2-(3-CHLOROPHENOXY) PROPIONATE NANOHYBRIDS

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June 2012.

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Controlled release formulations were developed by intercalation of organic anions, namely 4-(2,4-dichlorophenoxy)butyrate (DPBA) and/or 2-(3-chlorophenoxy)propionate (CPPA) into the inorganic Zn-Al layered double hydroxide (ZAL) and zinc layered single hydroxide (ZLH) interlayers. Both anions, DPBA and CPPA are herbicides, a group of herbicides which function by mimicking the action of auxins and plant growth hormones. In this study, the nanohybrid compounds were synthesized by direct co-precipitation and anion exchange method for ZAL nanohybrids while direct reaction of zinc oxide with both anions under aqueous solution was applied for the formation of ZLH nanohybrids. Simultaneous intercalation of the dual herbicides was confirmed by the expansion of the interlayer spacing from 8.9 Å in the ZAL to 25.1 Å and 26.9 Å for the nanohybrid prepared by direct and ion-exchange method while ZLH shows higher basal spacing up to 28.7 Å due to higher charge density which can accommodate more guest anions into the ZLH interlayers compared to ZAL. Generally, the loading percentage obtained from simultaneous equation derived from UV-Vis spectrophotometer data for DPBA was found

to be higher than CPPA. Result shows, nanohybrid synthesized by co-precipitation method contain of 2.5 % CPPA + 41.4 % DPBA while the percentage loading of the nanohybrid synthesized by anion-exchange method is 8.6 % CPPA + 53.9 % DPBA. Dual intercalation of both herbicides anions into ZLH resulted in percentage loading of 25.3% CPPA + 78.4 % DPBA. The high content of DPBA indicates that it is the dominant species and preferentially intercalated into the host interlayers than CPPA. In addition, DPBA anion has two chlorine atoms attached to the benzene ring therefore interacts more strongly with the positive charged layers. The simultaneous release profiles of both anions from their respective nanohybrids into sodium carbonate aqueous solution were best described by the pseudo-second order kinetic. This demonstrates the potential application of layered materials as hosts especially for the preparation of a controlled release formulation of the two herbicides simultaneously. The bulky anionic size, orientation and interaction with the positively charged inorganic interlayer could affect the degree of intercalation as well as the release of these herbicides from the host interlamellae.

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

PENCIRIAN DAN KAJIAN LEPASAN TERKAWAL 4-(2,4 DIKLOROFENOSIL) BUTIRIK DAN 2-(3-KLOROFENOSIL) PROPIONIK NANOHIRID

Oleh

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Formulasi lapisan terkawal telah digunakan bagi interkalasi dua anion organik, iaitu 4-(2,4-diklorofenosa)butirika (DPBA) dan 2-(3-klorofenosa)propionika (CPPA) ke dalam lapisan organik hidroksida lapis berganda-zink-alumina (ZAL) dan hidroksida lapis zink (ZLH). Kedua-dua anion, DPBA dan CPPA adalah racun herba yang berfungsi sebagai secara meniru tindakan auxina dan hormon pertumbuhan. Dalam kajian ini, sebatian nanohibrid telah disintesis segera pemadaman langsung dengan kaedah pertukaran anion untuk ZAL nanohibrid, manakala zink oksida bertindak balas langsung dengan kedua-dua anion dalam larutan akueus yang digunakan untuk pembentukan ZLH nanohibrid. Interkalasi serentak dua racun herba ini disahkan oleh pengembangan jarak lapisan daripada 8.9 Å bagi ZAL kepada 25.1 Å dan 26.9 Å bagi nanohibrid yang disediakan oleh kaedah langsung dan pertukaran ion, manakala ZLH menunjukkan pengembangan jarak yang lebih tinggi sehingga 28.7Å disebabkan oleh ketumpatan caj yang lebih tinggi dan boleh menampung lebih banyak anion tetamu didalam lapisan ZLH berbanding ZAL. Secara umumnya, peratusan muatan yang diperolehi daripada persamaan serentak melalui

data spektrofotometer UV-Vis untuk DPBA adalah lebih tinggi daripada CPPA. Hasil kajian menunjukkan, nanohybrid yang disintesis oleh kaedah pemendakan langsung mengandungi 2.5% DPBA + CPPA 41.4% manakala pemuatan peratusan nanohybrid disintesis oleh kaedah pertukaran anion adalah 8.6% CPPA + DPBA 53.9%. Interkalasi dwi anion ke dalam ZLH memberikan peratusan muatan sebanyak 25.3% CPPA + DPBA 78.4%. Kandungan DPBA yang tinggi menunjukkan bahawa ia merupakan spesies yang dominan dan lebih cenderung diinterkelasikan ke dalam lapisan perumah berbanding CPPA. Disamping itu, anion DPBA mempunyai gelang benzena dengan 2 atom klorin yang boleh berinteraksi lebih kuat dengan lapisan yang bercas positif. Profil perlepasan serentak kedua-dua anion dari nanohybrid mereka ke dalam larutan natrium karbonat boleh dijelaskan melalui kinetik tertib pseudo-kedua. Ini menunjukkan potensi penggunaan bahan lapisan sebagai perumah terutamanya untuk penyediaan formulasi pelepasan terkawal dua racun herba secara serentak. Saiz anion, orientasi dan interaksi dengan lapisan bukan organik yang bercas positif boleh mempengaruhi tahap interkalasi serta pembebasan racun herba ini dari antara lapisan perumah.

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I certify that a Thesis Examination Committee has met on the 25 June 2012 to conduct the final examination of Nor Shazlirah Shazlyn binti Abdul Rahman on her thesis entitled “Characterization and controlled release Studies of 4-(2,4-Dichlorophenoxy) Butyrate and 2-(3-Chlorophenoxy) Propionate Nanohybrids” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master Degree of Science.

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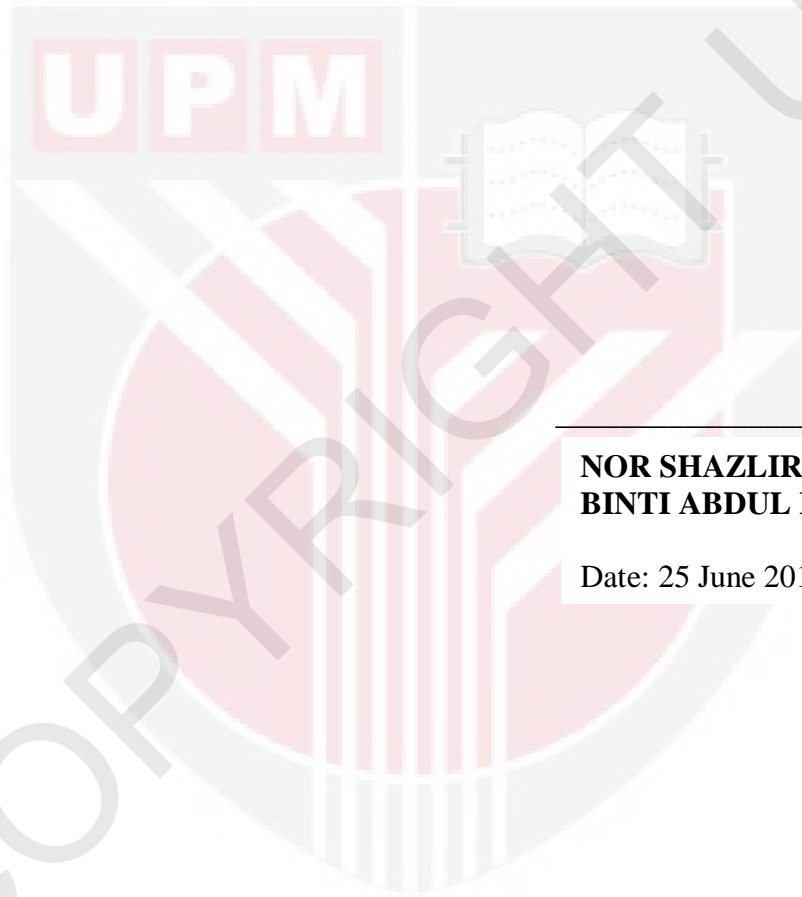
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DECLARATION

I declare that the thesis is my original work except for quotations and citations, which have been duly acknowledged. I also declare it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institutions.



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Date: 25 June 2012

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