UNIVERSITI PUTRA MALAYSIA

INTERCALATION OF UREA BETWEEN KAOLINITE NANOLAYERS FOR CONTROLLED RELEASE FERTILIZER APPLICATION

FARIBA MAHDAVI

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By

FARIBA MAHDAVI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Master of Science

May 2012

Specially dedicated to

My mother, my father and my brother Alireza the most beloved persons in my life, for their love, understanding, endless patience and encouragement when it was most needed.

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

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May 2012

Chair: Suraya Abdul Rashid, PhD

Institute: Institute of Advanced Technology

In this study the high surface area of kaolinite nanolayers was exploited as a reservoir of urea in order to minimize nitrogen leaching, to prepare controlled release fertilizer (CRF).

Urea was intercalated between kaolinite nanolayers through three different methods including ultrasonication as well as wet and dry grinding. For each technique, the effect of several parameters including urea concentration, time of process and temperature on the degree of intercalation was studied individually. The results of X-ray diffraction (XRD) showed that among these factors, urea concentration was effective and the interlayer spacing of kaolinite expanded from 7.1 Å to 10.9 Å for the highest urea concentration (urea/kaolinite=8/1). Complete intercalation which was calculated using intercalation ratio formula was achieved using dry grinding method. The morphology of kaolinite before and after intercalation was studied by using field emission scanning electron microscopy

(FESEM). Also, Fourier Transform Infrared Spectroscopy (FTIR) revealed hydrogen bonding between kaolinite and urea. The thermal behavior of intercalated kaolinite was investigated using thermogravimetric analysis (TGA) and the CHNS elemental analyzer determined that 20 wt.% urea was loaded within the kaolinite surface.

To prepare nitrogen-based CRF (NCRF), urea-intercalated kaolinite was mixed with aqueous solution of hydroxypropyl methylcellulose (HPMC) as the binder and then was granulated in three different sizes and coated with three different water-based polymer coatings including water based epoxy resin, water-based thermoplastic acrylic resin, and, water based thermosetting acrylic resin. The granules were incubated in water at room temperature for 30 days and the urea release was measured in 5 days interval using UV/Vis technique and through diacetylmonoxime (DAM) colorimetric method. The effect of several factors including binder concentration, size of granules, coating thickness and the water absorbency of coating on urea release behavior of the NCRFs was investigated. The urea release from the coated NCRFs was evaluated according to the standard declaration of CRFs and was compared with the release properties of non-coated NCRF granules, with the non-intercalated kaolinite-urea granules and with the release behavior of conventional urea granules.

From the UV/Vis results, binder concentration was not effective on the release behavior of the NCRF. On the other hand, the results showed that by increasing the thickness of coating and the size of granules the percentage of urea release decreased. The NCRFs with the largest size and the highest coating thickness

coated with the thermoplastic acrylic resin had the lowest ratio of nitrogen release among all samples. Also, the controlled release properties of the NCRFs revealed that the standard of controlled release fertilizer was conformed to.

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

INTERKALASI UREA KE DALAM LAPISAN NANO KAOLINIT UNTUK PENGGUNAAN BAJA PELEPASAN TERKAWAL

Oleh

FARIBA MAHDAVI

Mei 2012

Pengerusi: Suraya Abdul Rashid, PhD

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Kajian ini adalah berkenaan mengeksploitasikan luas permukaan lapisan nano kaolinit sebagai takungan urea untuk mengurangkan pelepasan nitrogen bagi menyediakan baja pelepasan terkawal (CRF).

Urea diinterkalasikan ke dalam kaolinit melalui tiga kaedah yang berbeza iaitu ultrasonikasi, pengisaran kering dan basah. Analisis belauan sinar-X mengesahkan pengembangan jarak lapisan antara kaolinit ialah dari 10.1 Å ke 10.9 Å dan interkalasi lengkap dicapai menggunakan kaedah pengisaran kering. Manakala, Fourier Mengubah Spektroskopi Inframerah (FTIR) mendedahkan pengikatan hidrogen antara kaolinit dan urea. Kelakuan terma bagi interkalasi kaolinit disiasat dengan menggunakan analisis termogravimetri manakala penganalisis asasi CHNS menentukan sebanyak 20 berat.% urea telah dimuatkan di permukaan kaolinit.

Untuk menyediakan nitrogen berdasarkan CRF (NCRF), serbuk urea kaolinit diadunkan dengan satu bahan tambahan dan disalutkan dengan salutan polimer. Beberapa faktor yang boleh memberi kesan terhadap kelakuan pelepasan nitrogen NCRF seperti nisbah ejen pengentalan urea, saiz butiran, ketebalan salutan dan jenis salutan telah disiasat dengan menggunakan teknik UV/Vis dan melalui kaedah kolorimetri. Pembebasan nitrogen terkawal dari NCRF bersalut telah dinilai mengikut pengisytiharan standard CRF dan telah dibandingkan pelepasan nitrogen bagi butiran NCRF yang tidak bersalut serta butiran urea kaolinit yang tidak diinterklasikan dengan kelakuan pembebasan butiran urea yang konvensional.

Keputusan spektroskopi menunjukkan nisbah ejen pengental urea bukanlah merupakan satu faktor yang berkesan terhadap pembebasan NCRF. Sebaliknya, keputusan itu menunjukkan dengan semakin bertambah ketebalan salutan dan saiz butiran, semakin pembebasan nitrogen berkurang. NCRF dengan saiz terbesar dan ketebalan salutan yang tebal disalutkan dengan damar akrilik termoset menunjukkan nisbah terendah pembebasan nitrogen bagi semua sampel. Sampel-sampel yang disediakan adalah mengikuti standard ciri-ciri pembebasan bagi European Normalization Committee.

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I certify that a Thesis Examination Committee has met on 31 May 2012 to conduct the final examination of Fariba Mahdavi on her thesis entitled **"INTERCALATION OF UREA BETWEEN KAOLINITE NANOLAYERS FOR CONTROLLED RELEASE FERTILIZER APPLICATION"** 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 of Science. Members of the Thesis Examination Committee were as follows:

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The thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of master. The members of the supervisory Committee were as follows:

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DECLARATION

I declare that the thesis is my original work expect for quotations and citations which have been duly acknowledged. I also, declare that 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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FARIBA MAHDAVI

Date: 31 May 2012

TABLE OF CONTENTS