UNIVERSITI PUTRA MALAYSIA

# *COMPACTION, STRENGTH AND DISSOLUTION CHARACTERISTICS OF UREA 46% N TABLETS*

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FK 2014 89

# COMPACTION, STRENGTH AND DISSOLUTION CHARACTERISTICS OF UREA 46% N TABLETS

By

INTAN SORAYA BINTI SHAMSUDIN

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

**August 2014**

**DEDICATION**

*This thesis is especially dedicated to my parents;*

*Haji Shamsudin Abu Bakar*
*Hajah Khatijah Abdul Kadir*

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

**COMPACTION, STRENGTH AND DISSOLUTION CHARACTERISTICS OF UREA 46% N TABLETS**

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**INTAN SORAYA BINTI SHAMSUDIN**

**August 2014**

**Chairman: Mohd Shamsul Anuar, PhD**

**Faculty: Engineering**

Lab scale urea tablets are formed through direct compaction by uniaxial die compaction method. The main objectives of this study are 1) to investigate the effect of particle size on the compaction properties and characteristics of urea tablets, 2) to evaluate the influence of die wall lubrication on the compaction properties and characteristics of urea tablets and 3) to determine the influence of admixed lubrication on the compaction properties and characteristics of urea tablets.

The particle sizes were varying in the form of granules and powders for urea granules tablets (TG tablets) and urea powder tablets (TP tablets). The lubricants; magnesium stearate (MgSt) and stearic acid were lubricated on the surfaces of the die wall and admixed in the formulation at compositions of 1%, 3%, 5% and 10% w/w. The compaction process was conducted at five applied pressures ranging between 37.67 MPa and 188.35 MPa using a universal testing machine. The compaction properties, namely plastic work, elastic work, friction work and maximum ejection pressure were analyzed from the force-displacement profile of the compaction process. Characteristics of the urea tablets were tested for the compressive strength and ammonium ion release through dissolution test.

From the different particle sizes experimental results obtained, it was found that TG tablets deformed more by fragmenting deformation under applied pressure according to the high plastic work and low compressive strength. TP tablets deformed more by plastic deformation under applied pressure based on the high plastic work and high compressive strength. The TG tablets had lower ammonium ion release than urea granules at all applied pressures. They also had lower ammonium ion release than TP tablets at almost all applied pressures except 75.34 MPa. The results of die wall

lubrication have shown the tablets die wall lubricated with stearic acid had lower plastic work and maximum ejection pressure. However, they had higher elastic work and friction work. They produced high strength tablets and lower ammonium ion release than urea granules and other tablets when formed at 75.34 and 113 MPa.

From the admixed lubrication results, most of the tablets had good compaction properties, possessed high compressive strength and release lower ammonium ion when formed at applied pressures from 113 to 188.35 MPa. The amount of lubricant used was less than 1% w/w and stearic acid as the choice of the lubricant. As a conclusion, this preliminary study provides information concerning the tablets forming ability, choosing the suitable form of materials and the influence of lubricant through die wall and admixed lubrication in direct compaction of urea.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
Sebagai memenuhi keperluan untuk ijazah Master Sains

**SIFAT-SIFAT PADATAN, KEKUATAN DAN KETERLARUTAN TABLET UREA 46% N**

Oleh

**INTAN SORAYA BINTI SHAMSUDIN**

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Tablet urea berskala makmal telah dibuat menggunakan pemadatan langsung melalui kaedah pemadatan acuan satu arah. Objektif utama kajian ini adalah: 1) untuk menyelidiki kesan saiz zarah ke atas ciri-ciri padatan dan sifat tablet urea, 2) untuk menilai pengaruh pelinciran secara dinding acuan ke atas ciri-ciri padatan dan sifat tablet urea dan 3) untuk menentukan pengaruh pelinciran secara percampuran ke atas ciri-ciri padatan dan sifat tablet urea.

Saiz zarah telah dipelbagaikan dalam bentuk butir dan serbuk untuk tablet butir urea (tablet TG) dan tablet serbuk urea (tablet TP). Pelincir; magnesium stearate (MgSt) and stearik asid telah dilincirkan pada permukaan dinding acuan dan dicampur dalam formulasi pada komposisi 1%, 3%, 5% dan 10% (berdasarkan berat). Proses pemadatan dijalankan di lima tekanan gunaan antara 37.67 MPa hingga 188.35 MPa menggunakan mesin ujian universal. Ciri-ciri padatan iaitu tenaga kerja plastik, tenaga kerja elastik, tenaga kerja geseran dan tekanan pengeluaran maksimum telah dianalisis dari profil tekanan-sesaran proses pemadatan. Sifat tablet urea telah diuji untuk kekuatan mampatan dan pembebasan ion ammonium melalui ujian keterlarutan.

Dari keputusan eksperimen saiz zarah berbeza yang diperolehi, didapati bahawa tablet TG lebih banyak berubah bentuk melalui perubahan bentuk penyerpihan di bawah tekanan gunaan menurut tenaga kerja plastik tinggi dan kekuatan mampatan rendah. Tablet TP lebih banyak berubah bentuk melalui perubahan bentuk plastik di bawah tekanan gunaan berdasarkan tenaga kerja plastik tinggi dan kekuatan mampatan tinggi. Tablet TG mempunyai pembebasan ion ammonium yang lebih rendah daripada butiran urea di semua tekanan gunaan. Mereka juga mempunyai pembebasan ion ammonium yang lebih rendah daripada TP tablet di hampir semua

tekanan gunaan kecuali 75.34 MPa. Keputusan pelinciran secara dinding acuan menunjukkan tablet yang dilincir dinding dengan stearik asid mempunyai tenaga kerja plastik dan tekanan pengeluaran maksimum yang rendah. Bagaimanapun, mereka mempunyai tenaga kerja elastik dan tenaga kerja geseran yang lebih tinggi. Mereka menghasilkan tablet kekuatan tinggi dan pembebasan ion ammonium rendah berbanding butiran urea dan tablet lain apabila dibentuk di 75.34 dan 113 MPa.

Dari keputusan pelinciran secara percampuran, kebanyakan tablet mempunyai ciri-ciri pemadatan yang baik, kekuatan mampatan tinggi dan membebaskan ion ammonium lebih rendah apabila dibentuk di tekanan gunaan dari 113 kepada 188.35 MPa. Selain itu, jumlah pelincir digunakan yang disarankan adalah kurang daripada 1% (berdasarkan berat) dan stearik asid sebagai pelincir pilihan. Sebagai kesimpulan, kajian permulaan ini menyediakan maklumat berkenaan keupayaan tablet dibentuk, pemilihan bentuk bahan yang sesuai dan pengaruh pelincir melalui pelinciran secara dinding acuan dan pelinciran secara percampuran dalam pemadatan langsung urea.

# ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to my supervisor, Dr. Mohd Shamsul Anuar for his expertise, understanding, patience and encouragement throughout my research project. My deep appreciations are extended to my master committee members, Assoc. Prof. Dr. Yus Aniza Yusof and Assoc. Prof. Dr. Ahmad Husni Mohd. Hanif for their assistances and advices.

I wish to express my thankful to laboratory technicians from Department of process and Food Engineering and Department of Mechanical and Manufacturing, especially Mr. Wildan Ilyas, Pn. Siti Hajar Zakaria and Mr. Rahman for their cooperation and help in allowing me to use Universal Instron machine and others equipments for my research project. I wish to thank Mr. Azmi from Institute Biosains, UPM for helping me to do Scanning Electron Microscopy (SEM) test. A special thanks to Universiti Putra Malaysia Research University Grant project number 05-02-12-1921RU and Graduate Research Fellowship (GRF) of Universiti Putra Malaysia for the financial support.

An appreciation to my beloved family especially my parents, Haji Shamsudin Abu Bakar and Hajah Khatijah Abdul Kadir for their support, encouragement and understanding throughout my project. A special thank to my friends; Muhammad Hazwan, Iryani Ismail, Mohammed Yusuf, Nor Zulaila, Amnani Shamjuddin and Faiqa Shazea for helping and supporting me during times of difficulty and hardship. Lastly, I would like to thank all people who were involved directly or indirectly in helping and guidance me during my project.

I certify that a Thesis Examination Committee has met on 15 August 2014 to conduct the final examination of Intan Soraya binti Shamsudin on her thesis entitled "Compaction, Strength and Dissolution Characteristics of Urea 46% N Tablets" 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.

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**Declaration by Members of Supervisory Committee**

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in Rule 41 in Rules 2003 (Revision 2012-2013) were adhered to.

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# TABLE OF CONTENTS

## LIST OF TABLES

## LIST OF FIGURES

## LIST OF ABBREVIATIONS

| | |
|---|---|
| TG tablets | Urea granules tablets |
| TP tablets | Urea powder tablets |
| SEM | Scanning electron microscopy |
| MgSt | Magnesium Stearate |
| USG | Urea super granules |
| LUG | Large urea granules |
| UAS | Urea-ammonium sulphate |
| EMS | Electron Microscope |
| PU | Polymer urea |
| PEG | Polyethylene glycol |
| $\sigma_y$ | Vertical stress component during diametrical compression test |
| $\sigma_x$ | Horizontal stress component during diametrical compression test |
| N | Nitrogen |
| KCl | Potassium chloride |
| PG | Phosphogypsum |
| DAP | Diammonium phosphate |
| $ZnSO_4$ | Zinc sulphate |
| $(NH_2)_2CO$ | Urea |
| $(NH_4)_2CO_3$ | Ammonium carbonate |
| $HCO_3^-$ | Bicarbonate anion or hydrogen carbonate ion |
| $NO_3^-$ | Nitrate |
| $NH_{3\,(g)}$ | Ammonia gas |
| $NH_4^+$ | Ammonium ion |

| | |
|---|---|
| $N_{2(g)}$ | Nitrogen gas |
| $OH^{-}$ | Hydroxide anion |
| $H^{+}$ | Hydron |
| $NH_4OH$ | Ammonium hydroxide |
| $Mg^{2+}$ | magnesium cation |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background

Nitrogen (N) fertilizers are widely applied for the purpose of giving additional N nutrient for plant growth and maintaining the soil fertility (Pietsch, 2005). It is one of common factors that limit the yield of agriculture production as the nutrient within the plant roots cannot be retained for a desired period of time (Li et al., 2008; Ni et al., 2009). The application of N fertilizers in rice fields has been commonly adopted to improve the N availability and high rice grain yields. Among the N fertilizers, urea is the most widely used one applied to rice fields because it is the cheapest form of granular chemical N fertilizer containing high N content (~46%) (Trenkel, 1997; Zheng et al., 2009). Its importance as fertilizer has grown progressively and represents about 50% of the world’s N fertilizers (Baligar et al., 2001). Thus, it is economical to produce, ease of handling, transport and available throughout the world (Mohd Ibrahim et al., 2014).

The utilization efficiency or plant uptake of N from urea is generally very low e.g. 35-60% due to its water soluble property and the common practice of the farmers e.g. applied the urea to the rice soil surface soon after its application (Conrad, 2002; Cai et al., 2007; Ji et al., 2014; Prasad et al., 1971; Al-Zahrani, 1999; Ibrahim and Jibril, 2005; Li et al., 2008). Estimate of N of the applied urea fertilizer lost to the environment has been reported to be about 40-70% by which it causes economic, resources losses and serious environmental pollution such as surface runoff, nitrate leaching, urea hydrolysis and ammonia volatilization (Mikkelsen et al. , 1978; Craswell et al., 1981; Flinn and O'Brien, 1982; Flinn et al., 1984; Fan et al., 2004; Quaggio et al., 2005; Dobermann, 2005; Cantarella, 2007; Al-Zahrani, 2000; Jones et al., 2007; Dave et al., 1999; Guo et al., 2005; Liu et al., 2007).

Ammonia volatilization is the loss of N to the atmosphere through conversion of the ammonium to ammonia. Ammonium is the availability source of N for plant life while ammonia is not considered as the plant available by which its losses through urea hydrolysis may reach 80% of the total N applied under field conditions (Gould et al., 1986). In the field of urea fertilizer industry, the application urea method e.g. deep, nest or band placements and urea modification through agglomeration technology are implemented for regulating the N losses and improves the N efficiency of urea. Table 1.1 listed the two of the most commonly groups in agglomeration technology namely agitation agglomeration by granulation process and pressure agglomeration by compaction process and their descriptions.

**Table 1.1: Commonly processes of agglomeration technology in urea fertilizer industry.**

| Process | Type of fertilizer | Description | References |
|---|---|---|---|
| **Granulation** | Granulated and coated urea fertilizers | The urea granules were granulated and spray coating with coating materials, binding agents, chemicals or additives as the purpose to coat and increase the size.<br>Produced in a fluidized bed granulator, pan granulator, drum granulator, fluidized bed coater, pan coater and rotary drum coater | Du et al. (2006)<br>Anggoro (2011)<br>Lan et al. (2011) |
| **Compaction** | Compressed/compacted fertilizers | The urea granules were milled and dry mixed with binding agents or additives.<br>The mixtures were mechanically compressed/ compacted in a compression device such as laboratory pressing machine, hydraulic pressure machine or hot press in a compression moulding machine.<br>Compaction parameters such as different pressures and mass of compositions influenced the quality of urea compacts. | Savant and Stangel (1990).<br>Rahman et al. (1996).<br>Purakayastha and Katyal (1998). |

## 1.2 Problem statement

Extensively studied in literatures have been reported the granulation process for development of coated urea fertilizers. Most of the studies emphasized the implementation of original industrial grade urea granules and several of coating materials to regulate the N losses. The coated urea fertilizers are prepared by granulating and coating the conventional granules urea fertilizers with various materials (Mingchu et al., 1994; Choi and Meisen, 1997; Ayub et al., 2001; Vashishtha et al., 2010; Mulder et al., 2011; Wang et al., 2012).

Few of the previous studied have reported the compaction of urea focusing the effects of compaction parameters and incorporating additives on the urea performance (Mikkelsen et al., 1978; De Datta et al., 1983; Yadvinder-Singh et al., 1994; Rahman et al., 1996; Purakayastha and Katyal, 1998; Savant and Stangel, 1990; Savant and Stangel, 1998; Sheena Shareena, 2011). Thus, this required more study on the compaction process mainly the factors that govern to the successful formation of the compacted urea know as urea tablets. These factors; the different form of urea, properties of the materials that used to be formed into urea tablets and the process parameters involved during the compaction process are associated with the properties of the final urea tablets.

Apart from that, little information is known about the properties of urea under the influence of an applied pressure. It is important to identify the drawbacks in the production of the urea tablets by analyzing the compaction properties such as elasticity, plasticity, bonding, and brittleness (Antikainen and Yliruusi, 2003; Wu et al., 2005; Amidon et al., 2009). This information is essential for predicting any occurrence of a urea tablet failure, the way it may manifest or its tablet-forming ability (Ragnarsson and Sjögren, 1983; Alderborn, 2007; Han et al., 2008).

In addition, there has been no study on the compaction properties of urea fertilizer in the presence of lubricant. The use of lubricant in the formation of urea tablets is crucial for improving the compaction process and the properties of the urea tablets which are dependent upon the properties of the lubricants (York, 1980; Sinka et al., 2007; Ahmat et al, 2011). For that reasons, two of the most commonly used lubricants in their category and undergo plastic deformation (Ebba et al., 2001; Wang et al., 2010); magnesium stearate (MgSt) and stearic acid are utilised in the formation of urea tablets for comparison purposes.

## 1.3 Scope and objectives

The scope of this study is to form experimental urea tablets for deep placement through direct compaction by laboratory scale uniaxial die compaction method. It is conducted as the purpose to understand the compaction properties, characteristics of the urea tablets and factors influencing the compaction process such as different size of urea, applied pressures, lubricants and compositions of lubricants. The objectives of this study are:

1) To investigate the effect of particle size on the compaction properties and characteristics of urea tablets.

2) To evaluate the influence of die wall lubrication on the compaction properties and characteristics of urea tablets.

3) To determine the influence of admixed lubrication on the compaction properties and characteristics of urea tablets.

## 1.4 Outline of the Thesis

**Chapter 1** introduces the urea fertilizer which is the main material, the production of slow or controlled released urea fertilizer by wet granulation and compaction processes as the methods to overcome the N loss and the objectives of the research. **Chapter 2** briefly introduces the direct compaction by uniaxial die compaction method, stages in the compaction process and characteristics of materials. Factors that influence the compaction process such as applied pressure, particle size, types of lubricant and compositions of lubricant are also discussed. Two different tests to characterize the urea tablets are explained briefly. **Chapter 3** thoroughly describes the process flow of the works, preparation of the materials, the compaction process and the tablet testing such as compressive strength and ammonium ion release through dissolution test. The analyses of the physical properties of the materials are also included. In **Chapter 4**, the physical properties of the materials are introduced and discussed. This chapter continue with the compaction process by discussing the effect of different particle size in the formation of urea granules tablets (TG tablets) and urea powder tablets (TP tablets). The effect of lubrication through die wall and admixed lubrication are also discussed. At the end of this thesis, **Chapter 5** presents the conclusions of the works in this study as well as recommendations for future works.

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