FLUIDIZED BED GRANULATION PROCESS: EFFECT OF CHAMBER AND BINDER TEMPERATURE ON GRANULE PHYSICAL PROPERTIES

This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Manufacturing Process) (Hons.)

by

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DECLARATION

I hereby, declared this report entitled “Fluidized Bed Granulation Process: Effect of Chamber and Binder Temperature on Granule Physical Properties” is the result of my own research except as cited in references.

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Date : 31 JUNE 2017

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Manufacturing Process) (Hons). The member of the supervisory committee are as follow:

……………………….................................

(Main Supervisor**)**

……………………….................................

(Co –Supervisor)

ABSTRACT

This project paper showed through experimental research on the production of urea fertilizer by using a machine called fluidized bed granulator. This study was done by ensuring a few parameters that may influence desired urea fertilizer formed. There are three parameters used in this experiment, there are chamber temperature, binder temperature and the binder concentration required to produce the size, density and hardness of the desired urea. The optimum parameters determined by the observation of the experiments and the literature review on previous research. Additionally, the experiment will conduct appropriately with changes of the process variables such as spray rate to improve the result of previous studied. Data are collected by measuring size, hardness and density of the urea granules using several methods such as sieve test, water displacement method, and hardness test. Data are analyzed on using ANOVA analysis by Design Expert software. Applying the two level factorial design in the experiment, it is observed that the optimize value of each parameter with desirability of 0.882 and predicted hardness (2.52 kgf/granule) and density (1.2665g/cm3) could be obtained with 50 ˚C of chamber temperature, 50 ˚C for binder temperature, 55% for urea solution concentration. Most granules form meet the desired size, hardness and density.

.

ABSTRAK

Kertas projek ini menunjukkan melalui penyelidikan eksperimen kepada pengeluaran baja urea dengan menggunakan mesin yang dipanggil fluidized bed granulator. Kajian ini dijalankan dengan memastikan beberapa parameter yang boleh mempengaruhi perjalanan baja urea terbentuk. Terdapat tiga parameter yang digunakan dalam eksperimen ini, terdapat kebuk suhu, suhu pengikat dan kepekatan pengikat yang diperlukan untuk menghasilkan saiz, kepadatan dan kekerasan urea yang dikehendaki. Parameter optimum ditentukan melalui pemerhatian daripada eksperimen dan kajian literatur pada penyelidikan sebelumnya. Selain itu, eksperimen akan menjalankan dengan sewajarnya dengan perubahan pembolehubah proses seperti kadar semburan untuk meningkatkan hasil daripada sebelum dikaji. Data dikumpulkan dengan mengukur saiz, kekerasan dan kepadatan granul urea menggunakan beberapa kaedah seperti ujian saringan, kaedah sesaran air, dan ujian kekerasan. Data dianalisis pada menggunakan analisis ANOVA oleh perisian Design Expert. Menggunakan dua tahap reka bentuk faktorial dalam eksperimen, didapati bahawa nilai mengoptimumkan setiap parameter dengan kebaikan 0.882 dan kekerasan meramalkan (2.52 kgf / biji) dan ketumpatan (1.2665g / cm^3) boleh diperolehi dengan 50˚ C kebuk suhu, 50˚ C untuk suhu pengikat, 55% untuk kepekatan larutan urea. Kebanyakan butiran yang terhasil memenuhi saiz yang dikehendaki, kekerasan dan kepadatan.

DEDICATION

Only
My appreciated father,
my beloved mother,
my adored sisters and brothers,
for giving me moral support and money
Thank You So Much

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Here I want to thanks and appreciation to my supervisor, Mr Mohd Fairuz Bin Dimin as provide tutoring and guidance to me until the bachelor project is completed.

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LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

ANOVA	-	Analysis of Variance
DX	-	Design Expert
Kgf/granule	-	kilogram force per granule
CT	-	Chamber temperature
BT	-	Binder temperature
BC	-	Binder concentration

CHAPTER 1

INTRODUCTION

This chapter introduced about the problem statements, objectives and scope of research. The chapter will describe the project statement that need to be achieve the goal and the problems in determine the data and result.

1.1 Research Background

For several decades, the pharmaceutical industry has used fluid-bed granulation to improve powder properties such as flowability and compressibility for downstream processing. Instead of reducing the particle size, the process propose at particle growth. Granulation is defined as a particle size enlargement process where the small powder particles are gathered into bigger, permanent structures in which the original particles can be distinguished (Burggraeve et al. 2013). The agglomeration of material used in this study, urea powder made through the physical interaction using fluidized bed top spray granulator in the presence of droplets of liquid binder. Here, the temperature in the fluidized bed are important parameters that affect the whole process. Previous research state that the distribution of the gas humidity and temperature is an important factor influencing the process of coating particles in a fluidized bed (Maronga & Wnukowski 1998).

The aim of the project is to identify, analyze and correlate the chamber and binder temperature on the formation of urea fertilizer granules using the fluidized bed granulator. The project will investigate whether the temperature of the chamber and binder will influence the quality such as size, density and hardness of the urea granules formed. Randomized Regular Two Level Factorial is the selected method in this project that is useful for the modelling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize the parameter used. This study should foresee the optimum process parameter to obtain the desired urea granule.

1.2 Problem Statement

The case study main focus is to determine the correlation of chamber and binder temperature on the granule physical properties to produce urea granules good in size, density and hardness. Temperature is basically use in the chamber to control the level of humidity and the mass of granulated urea formation (Rostam & Said 2014). Base on the discussion in Fertilizer Manual (International Fertilizer Developement Centre (IFDC) 1998), the physical properties for each kind of traits which is size, density and hardness have their own standard that need to follow. In order to obtain good physical properties and achieve the standard in the production of urea granule, optimum parameter needed to be used which is chamber and binder temperature. The fluidized bed granulator play an important role in the industry such as food and pharmaceutical industry. Due to the technology absence in production of urea in Malaysia, the standard traits of urea granule was not fully achieved by the industry. Therefore, it is crucial that any parameter that contributing the quality of finished granules must be in control which are fan speed, binder feed rate and air pressure. The experimental session of the study will investigate the effect of chamber and binder temperature to obtain optimal size, density and hardness that will overcome the caking or formation of coherent mass problem.

Urea is the material that widely use in agricultural in the form of powder to be apply to crops. The powder or urea fertilizer will use as material of binder inserted in the chamber and study the efficiency with using same material of the binder compared to former research that use lactose as binder additives. Thus, the temperature increasing steadily, while the moisture in the chamber is high that can effect on the formation of urea granules during granulation. The slowly increasing of temperature will effected moisture in the bed where high moisture give low in solidified process of urea formation.

1.3 Objectives

Objectives of this study are:

I. To design an experiment using Regular Two Level Factorial Design for chamber and binder temperature against urea granule size, density and hardness produced using fluidized bed granulator.

II. To analysis the relationship between the process parameter which is chamber and binder temperature against the granule size, density and hardness.

III. To determine the degree of correlation between the process parameter which is chamber and binder temperature against the granule size, density and hardness.

1.4 Scopes of the Research

The fluidized bed granulator is Fluid bed Processor (FLP) Mini Granulator with maximum of 500 g capacity. Furthermore, this type of granulator is the most suitable application for fertilizer granule. Urea seed obtained from crushed and ground urea fertilizer granules with industrial blender. The binder solution is from urea granules dilute with distil water at various concentration. The input parameter of this case study is the chamber and binder temperature. Optimization of the chamber and binder temperature is done by using Regular Two Level Factorial Design. The Design Expert is the statistical software package that will help to perform the Design of Experiment (DOE) in this study. There are many types of parameter that will affect the physical properties of the granule used such as binder and spray pressure. Besides that, the other parameter are declare as constant parameter such as fan speed, air pressure, nozzle height and spray rate. For this case study, the physical properties that will be analyse are granule size, density and hardness.

CHAPTER 2

LITERATURE REVIEW

This chapter specified the review from previous research that is related to this final year project. There are previous research on the process formation of urea granules which are granulation and agglomeration. Other than that, this chapter also discussed the process parameter and variables effect on granule physical properties with using FLP Mini Granulator.

2.1 Urea Granules

Urea is produced from ammonia and carbon dioxide that are fed into the reactor at high temperature and pressure. It is also known as carbamide and has the highest nitrogen content among the solid fertilizers in common use. Furthermore, the properties of urea that is odourless solid, colourless and high soluble in water that makes it is widely use in the agricultural industry. The chemical formula of this organic compound is CO $(NH_2)_2$.

Table 2. 1: Properties of urea (Fertilizer Manual IFDC 1998)

Chemical formula	CH_4N_2O

Molecular weight	60.06
Nitrogen content %	46.6
Specific gravity	1.335
Melting point ˚C	132.7
Colour	White
Density g/cm³	1.32
Urea	99.0 % min
Water	0.5 % max

2.1.1 Urea Application

Urea or carbamide that containing 46 % nitrogen with a white crystalline solid appearance is widely used in the agricultural sector both as a fertilizer and animal feed additive. As for the fertilizer, when the urea is applied to the soil, the area around it becomes a region of high pH and ammonia concentration. Poor seed germination and early seedling growth caused by the free ammonia that has formed are the major limitation for wheat production. Wan et al. (2016) showed in an empirical study that when ammonia volatilization level near zero or under zero urea application, germination (%) was high (>88%), and no significant differences among the wheat varieties.

For the seedling growth, it is stated that under a higher N application rate, ammonia volatilization has been significantly increased and therefore subdued seedling growth. This study focus attention on the capability of the selection of varieties with high tolerance to ammonia toxicity in enhancing seed germination and uniform seedling foundation through a breeding programme (Wan et al. 2016).

In the field of agriculture, the former researcher determined effect of different spacing and urea application rates on fruit nutrient composition, growth and yield of tomato in derived savanna vegetation of Nigeria. The plant height of tomato is increase by urea rate and row spacing for both locations Ejiba and Kabba. There has been a significant increase in plant height, stem girth, branches and number of leaves as the urea application rate increased. The suitable or most recommended of urea fertilizer application rate for the local farmer is at 54.8 kg per hectare for a profitable production of tomato (Saleem et al. 2013).

Besides that, in the human respiratory disease studies, urea has been applied to serve as sample volume marker since it can uninhibitedly diffuse through most body compartments and is less influence by disease states (Wang et al. 2016).

2.1.2 Urea Preparation

From the study, Fertilizers Europe (2000) stated that the production of urea involve the combination of two compound that is ammonia and carbon dioxide to form the salt formed of ammonium carbamate. The preparation of urea granule in fluid bed granulator consist of at least one inlet for fluidization air, a distribution plate above the fluid bed and sprayers that are installed in distribution plate, where the urea melt is sprayed on or over the urea particles in the granulator chamber and the particles are kept in motion by the fluidization air. This invention pointed out the fluidization air contains very finely atomized water that will make the water can evaporate very rapidly and achieve the desired cooling by evaporation of water without the large amount of fine dust that ensure the urea recovered from off gas and to protect the environment (Mutsers et al. 2010).

The preparation of urea formaldehyde resin/reactive kaolinite composites has been prepared by in situ polymerization to improve the properties. Numerous study have been done to improve the mechanical properties and thermal stability. This study show that the thermal stability has been much well than the pure urea-formaldehyde resin. The addition of reactive kaolinite has been improve the water resistance of the composites and decreased the formaldehyde emissions (S. Chen et al. 2016).

2.1.3 Urea Synthesis

The essential process in the synthesis of urea, created in 1922 which is called Bosch-Meiser urea prepare after its pioneers. The process comprises of two fundamental equilibrium reactions, with deficient conversion of the reactants. For the first, carbamate formation is the quick exothermic response of liquid ammonia with gaseous carbon dioxide (CO_2) at high temperature and pressure to form ammonium carbamate ($H_2N\text{-}COONH_4$).

$2NH_3 + CO_2 \rightleftharpoons H_2N\text{-}COONH_4$ Equation 2.1

The conversion of urea or for the second reaction that is occur in the slower endothermic of decomposition of ammonium carbamate that will turn into water and urea.

$H_2N\text{-}COONH_4 \rightleftharpoons (NH_2)_2CO + H_2O$ Equation 2.2

The second reaction occur as indicated by Le Chatelier's principle, for most of the chemical equilibrium the second reaction is driving by the reaction heat from the first reaction. The general conversion of urea formation and carbon dioxide is exothermic (Meessen 2010). The synthesis of urea granule has been done as shown in Figure 2.1.

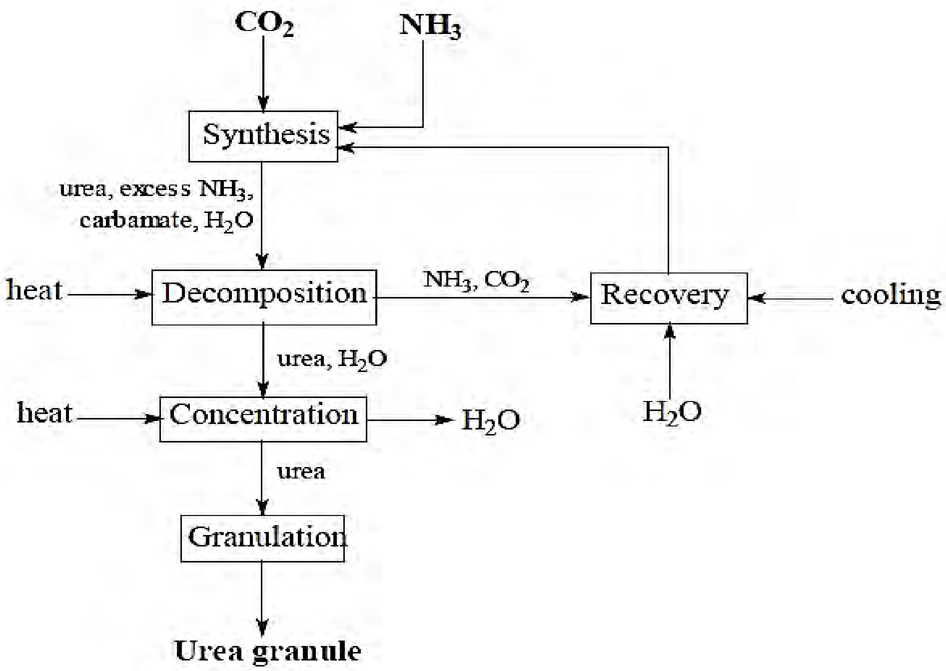

Figure 2. 1: Schematic representation of urea synthesis (Taranaki 1998)

From the previous study, it is indicated that in an open system the series of ureas and amides could not be synthesized by catalytic dehydrogenation coupling but via catalytic dehydrogenation of volatile alcohols and amines that were received in moderate to high yields.

Table 2. 2: Direct synthesis of symmetrical ureas from methanol and amines in a Pd-Ag membrane reactor (T. Chen et al. 2016)

Entry	T ℃	Methanol (mmol)	Amine (mmol)	Yield %
1	180	5	Butyl amine (5)	38
2	180	2.5	Butyl amine (5)	65
3	180	1.25	Butyl amine (5)	83
4	180	2.5	Butyl amine (5)	70
5	140	2.5	Butyl amine (5)	67
6	140	2.5	Butyl amine (5)	78
7	140	2.5	Propyl amine (5)	70
8	140	2.5	Propyl amine (5)	78
9	140	2.5	Hexyl amine (5)	65
10	140	2.5	Benzyl amine (5)	62

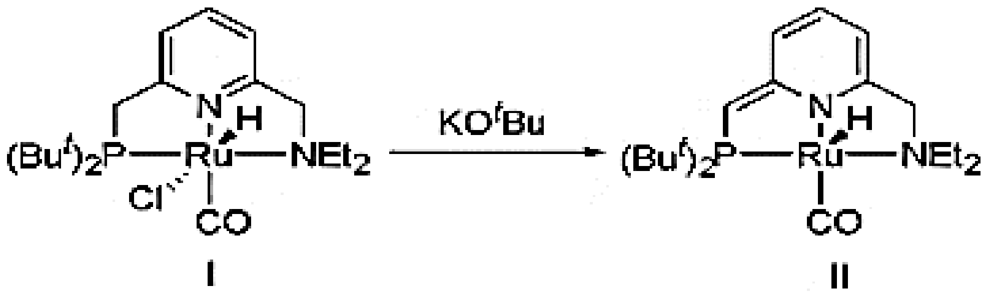

Figure 2. 2: The Milstein catalysts (T. Chen et al. 2016)

When the toluene solution of Milstein catalyst with (1:1 ratio) of methanol and butylamine has been heated at 180˚C for 12 hour, 38% yield of 1, 3-dibutylurea has been obtained at Table 2.2 (entry 1). The figure 2.2 shows the formation of Milstein catalyst. After that, the yield increased to 65% when the ratio of methanol to butyl amine changed to 1:2 (entry 2). The effect of temperature is clearly observed after the reaction temperature lowered to 140˚ C, the yield is slightly increased to 67%. It is indicating that there is no impermeable gases in the residual when net inner pressure decreased to zero after cooling the system to room temperature. From this study, it is stated that a progression urea and amides could be acquire in average to good yields (T. Chen et al. 2016).