FLUIDIZED BED GRANULATION PROCESS : STUDY ON AIRFLOW EFFECT ON UREA GRANULE PHYSICAL PROPERTIES

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

by

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DECLARATION

I hereby, declared this report entitled "Fluidized Bed Granulation Process : Study on Airflow Effect on Urea Granule Physical Properties" is the results of my own research except as cited in reference.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering (Manufacturing Process) (Hons.).

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ABSTRAK

Granulasi lapisan terbendalir merupakan teknik menggumpal yang telah banyak digunakan dalam industri baja, makanan dan perubatan. Walaubagaimanapun, pembentukan ketulan besar mudah berlaku kerana terdapat banyak parameter yang perlu dikawal dan ini akan menyebabkan kegagalan dalam proses. Oleh itu, kajian ini yang mengaplikasikan teknik lapisan terbendalir dengan zarah urea dan pengikat urea yang tulen sebagai bahan kajian adalah penting. Ini adalah untuk menentukan kesan aliran udara yang berbeza dan pelbagai proses parameter seperti halaju angin, tekanan semburan pengikat dan masa beg goncang terhadap ciri-ciri bijian yang dihasilkan. Sebanyak tiga puluh eksperimen yang pelbagai keadaan proses telah direka dengan bantuan Response Surface Methodology (RSM) dalam perisian Design Expert untuk mengenal pasti taburan saiz bijian, ketumpatan bijian dan kekerasan bijian dalam setiap eksperimen. Ciri-ciri bijian ini akan diuji dengan cara tapisan, densimeter dan alat menguji kekerasan mudah alih. Data yang diperolehi akan dimasukkan ke dalam perisian untuk analisis dengan menggunakan analisis varians (ANOVA) dan pada masa yang sama, keadaan proses yang optimum untuk mendapatkan ciri-ciri bijian yang diingini akan ditentukan. Dalam keadaan proses yang sama, aliran udara siklon adalah faktor paling penting untuk menghasilkan bijian dengan saiz dan ketumpatan yang diperlukan jika dibandingkan dengan aliran udara biasa. Daripada keputusan ANOVA, tekanan semburan pengikat dan masa beg goncang memberi kesan yang kuat terhadap kekerasan bijian untuk kedua-dua jenis aliran udara. Ramalan nilai optimum untuk aliran udara siklon dan biasa membuktikan bahawa bijian yang mempunyai ketumpatan dan kekerasan diingini boleh dihasilkan apabila kelajuan angin, tekanan semburan pengikat dan masa beg goncang diset pada 28 m/s, 0.31 MPa and 60 s masing-masing untuk aliran udara siklon manakala aliran udara biasa adalah pada 25 m/s, 0.20 MPa and 15 s secara masing-masing.

ABSTRACT

Fluidized bed granulation is a granulation technique that widely applied in fertilizer, food and pharmaceutical industry. The complexity and multiphase of this process caused failure such as lump formation was easy to occur. Hence, this study which dealt with fluidized bed granulation of urea seed particles by pure binder solution of urea was conducted which aimed to determine the effect of different airflow patterns and process conditions like wind velocity, binder spray pressure and bag shake duration on the physical properties of granules produced. Thirty run of experiments with different setting of process condition that designed using Response Surface Methodology of Central Composite Design in Design Expert software were carried out to find the distribution of granule size, density and hardness. Properties of granules were tested by using sieving, densimeter and portable hardness tester respectively. The data collected was transferred to the software to analyze results using analysis of variance (ANOVA) and determined the optimum parameters to obtain granules with desire properties. It was observed that spiral airflow was the most important factor to produce granules with required size and density under similar process conditions if compared with normal airflow. From ANOVA, binder spray pressure and bag shake duration showed the strongest influence on hardness of granules on both types of airflow patterns. Optimization studies for spiral and normal airflow proved that granules with desire density and hardness can be produced at middle level of wind velocity and binder spray pressure together with high level bag shake duration. For spiral airflow, the optimum value for wind velocity, binder spray pressure and bag shake duration was 28 m/s, 0.31 MPa and 60 s respectively; for normal airflow, it was 25 m/s, 0.20 MPa and 15 s respectively.

DEDICATION

Only

my beloved father, Loh Yong Hwa

my appreciated mother, Chong Yoke Gee

my adored sister and brother, Shea Ting, Chun Kong and Shea Jing

for giving me moral support, financial, cooperation, encouragement and understandings

Thank You So Much & Love You All Forever

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LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
AWS	-	Airtight Wind Shield
CCD	-	Central Composite Design
D_{50}	-	Geometric Mean Diameter
DOE	-	Design of Experiment
FAO	-	Food and Agriculture Organization of the United Nations
PCs	-	Personal Computers
PLCs	-	Programmable Logic Controllers
RSM	-	Response Surface Methodology

LIST OF SYMBOLS

°C	-	Degree Celsius
µm	-	Micrometre
g	-	gram
g/cm^3	-	Gram Per Cubic Centimetre
g/min	-	Gram Per Minute
kg/cm^2	-	Kilogram Per Square Centimetre
m/s	-	Meter Per Second
ml	-	Millilitre
ml/min	-	Millilitres Per Minute
mm	-	Millimetre
MPa	-	Mega Pascal
N	-	Newton
rpm	-	Revolution Per Minute
s	-	Second
w/v%	-	Weight Per Volume Percentage
w/w%	-	Weight Per Weight Percentage
wt %	-	Weight Percentage

CHAPTER 1

INTRODUCTION

This chapter will introduce about the problem statement, objectives and scope of study.

1.1 Research Background

The development of fluidized bed granulation was begun when Wurster reported his fluid bed experiments were success to prepare the compressed tablets using air suspension in 1960. Fluidization is a process which subjects a granular material in solid state to behave like a fluid. While, granulation is an enlargement process that gathers fine particles into a larger mass of aggregates by spraying binder solution to the dry powder bed (Roy *et al.*, 2009b).

Granulation process can be categorized into two types, wet granulation and dry granulation. With the used of liquid binder, the granulation is classified as a wet process. The binder solution is sprayed onto the solid particles to agglomerate them and the formed granules are then going through the drying process to extract the excess solvent to form permanent bonds between particles (Villa *et al.*, 2016). Additives such as starch can add into the binder solution to speed up the processing time and improves the properties of the granules formed

Fluidized bed granulation can be classified into three different types which are top spray, bottom spray and tangential spray. This depends on the spray nozzle location either on top, bottom or side of the column in chamber. However, only the top spray fluidized bed granulation is studied in this research. Each of this technique has its own function in granulating the seeds. For instance, top spray and tangential spray of fluid bed are commonly applied in granulation process while the fluid bed bottom spray is normally applied in industry of pharmaceutical to make film coating and layering with superior properties (Srivastava & Mishra, 2010). The top spray fluidized bed granulation has been applied in a lot of different processing industries such as detergent, agrochemical, food industries and pharmaceutical due to its versatility. Mixing, granulating and drying the materials all can be done in the same apparatus. This can save the process time, labour cost and reduce material handlings.

The raw material used is commercial urea fertilizer granules with chemical formula $CO(NH_2)_2$. It is an odourless, colourless and non-toxic dry organic compound with minimum about 46% of nitrogen content based on the data from Petronas Chemical Fertilizer. Pure binder solution without any additives is applied in this study although the addition of this granulating aids can improve the crystallisation process and provide a greater hardness value to granules of fertilizer. This is due to the addition of additives will increase the cost of production of the fertilizer.

The main focus of this study is to identify the effect of the normal and spiral airflow pattern and optimize the value of input parameters such as wind velocity, spray pressure and bag shake duration to obtain the required physical properties of the urea granules formed such as the size, density and hardness. A lot of previous study needs to be referred to determine the suitable range of input values for each parameter. However, it is still lacking of information about the influence of different air flow pattern with the input parameters especially the bag shake duration to the physical properties of granules formed.

The findings of the above study is important because it can help to save a lot of processing time without compromising the quality of granules formed in the granulator. Occurrence of caking and formation of lumps also will be reduced by optimizing the parameters in the process.

1.2 Problem Statement

Nowadays, fluidized bed technology has found in a wide range of applications such as chemical, pharmaceutical and food industries. It plays an important role especially in powder processing and pharmaceutical process which aimed to provide a well heat and mass transfer and help in the particle formation. However, it is very difficult to operate the process of granulation in fluidized bed granulator. A lot of parameters need to be control and the interaction between the parameters has to be understood in detail, otherwise the granulation process is easy to fail and bring to the problems such as formation of lumps, caking and non-uniform structure of the granules. The conditions influence the granulation process should be optimized in order to produce the granules with required properties especially in the size, density and hardness. Low density granules are easy to dissolve in water. More storage spaces are needed for granules weak in strength due to breakage problems. The products cannot stack together in one place. Hence, optimization of process is necessary to reduce the failure products formed and this can save processing time and the production cost for related industry. The factors that need to count in this study are pattern of airflow, binder spray pressure, wind velocity and shake duration of filter bags. However, there is no similar study that relates all these factors together, so through this study, some guidelines can be provided when designing fluidized bed granulation process.

1.3 Objectives

The objectives of the study are as shown:

i. To identify the effect of different airflow pattern and process variables on the size, density and hardness of urea granules formed in a fluidized bed granulator.

ii. To design the experiment using Respond Surface Methodology (RSM).

iii. To determine the optimum input parameters for different airflow pattern to produce urea granules with optimum level of physical characteristics.

1.4 Scopes of Research

This study is conducted by using laboratory type top spray fluidized bed granulator in Ceramic Laboratory of Faculty of Manufacturing Engineering in Universiti Teknikal Malaysia Melaka. The raw material used is commercial urea fertilizer granules $CO(NH_2)_2$ which are ground into powder form by using commercial blender as the seeds and to prepare for pure urea binder solution. This research is first focused on the effect of two different airflow pattern, normal and spiral airflow on the physical properties of granules formed. There is two air distributor plates with different hole design from the granulator will be utilized to create the two different forms of airflow. The experiments are designed by using Response Surface Method (RSM) with Central Composite Design (CCD) via Design Expert Version 6.0.8. The input parameters focus on this study are wind velocity, binder spray pressure and bag shake duration. The output parameters concentrate on size, density and hardness of granules formed only. The output results are determined by using the equipment available in UTeM such as sieve shaker, electronic densimeter and portable hardness tester. The testing results are analyzed by using ANOVA and optimized to identify the optimum input parameters to produce the urea granules with optimum level of physical characteristics.

CHAPTER 2

LITERATURE REVIEW

This chapter is to introduce the theory and research about the fluidized bed granulation process that has been applied in many different industries. The granulation process, air flow pattern, input parameters (wind velocity, spray pressure, bag shake duration) and output results (size, density, hardness) on the urea granule formed will be studied by referring to previous work that done by numerous researchers.

2.1 Granulation

Granulation is a granule forming process that gathers fine particles to change into a greater mass of aggregates by spraying binder solution to the dry powder bed (Roy *et al.*, 2009b). The three main elements that needed to produce well granules in granulation are initial seeds, good mixing of particles and binder solution (Cotabarren *et al.*, 2015). Granulation process is able to produce the granules with desired properties especially in obtaining better shape and size distribution and moreover can easier their handling for many different field of industries such as pharmaceutical, fertilizer, chemical, food industries and so on. However, the problems encountered for each field are totally different although the granulation theories applied are similar. For example, the production rate for pharmaceutical industries are made in kg/day while for detergent and fertilizer industries, the production rate is measured in tons/h (Mort, 2005).

The granulation process can be classified into wet or dry method based on the binder nature. Binder solution is needed for wet granulation but not necessary for dry granulation. Hence, when the binder solution is applied in the granulation process either by top/bottom-spraying or tangential spraying into the powder bed and moistens the particles, it is considered as wet granulation. Before the granulation process is conducted, mechanical agitation of the seeds is necessary. This aimed at reducing its particle size and for easier the binder distribution (Veliz Moraga *et al.*, 2015). The collision of the wet particles creates liquid bridges between them and the particles will start to combine and nucleate the granules. The agglomerates are then created by converting the liquid bridges to solid bridges through the evaporating of binding agent by hot air (Ziyani & Fatah, 2014).

In modern theories of granulation, Iveson *et al.* (2001) indicated that it was more easier to view the granulation as three key mechanisms if compared with traditional description which in terms of a number of different mechanisms. The three key mechanisms as shown in Figure 2.1 are:

i. Powder wetting and nucleation

   Binding agent will spray onto the bed and wet the particles. This makes a well distribution between nuclei granules. Granulation will start with the adhesion between particles due to liquid bridges and the particles then combine to form pendular state. Transition from pendular to capillary state if the agitation is continued.

ii. Granule growth and consolidation

   Granules will collide with other particles to form larger granules in coalescence. If the granule collides with the wall of the equipment's chamber, the granule will consolidate. It releases the air trapped inside the granule and reduces the size of granule.

iii. Granule attrition and breakage

   Wet and dried granules break during the granulation process in the granulator. Breakage of wet granule will influence the size of granule formed while attrition of dried granule will produces the dust to the process.

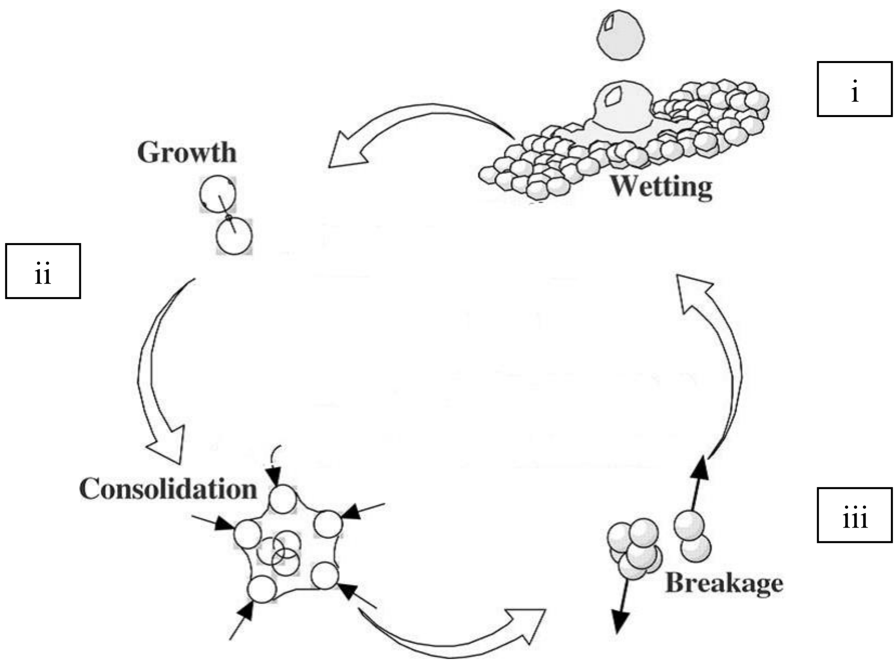

Figure 2.1: Three key mechanisms of granulation. Modified from (Mehta *et al.*, 2005).

2.2 Fluidization

The operation that delivers the air or liquid upward and pass through the packed bed of particles to make the solids to behave like fluid is known as fluidization. Fluidization is normally applied in chemical/physical-based operations. In chemical operations, it is used to know the solid-gas reaction; in physical operations, it can be used for transportation, heating and mixing of fine powder, combustion of coal and so on.

The changes in the parameters such as the gas velocity, size and density of particles will change the behaviour of fluidized bed. According to Smith (1980), the bed considered as fluidized only if the gas velocity was reached the incipient fluidization point. It is the point at which the pulling force was equal or greater than the force of gravitation. Hence, we can divide the fluidized bed into different kinds of fluidization regimes based on the gas velocity, for instance fixed bed, minimum fluidization, bubbling bed, slugging bed, turbulent bed and pneumatic transport. The schematic representation about this six different regimes as presented in Figure 2.2.

In fixed bed, the pulling force is too weak to lift the bed, particles only vibrate but continue to remain as the same level with the bed. When the gas velocity is increased to minimum fluidized velocity, the pulling force is balanced with the particles weight, the bed becomes fluidized and this starting the minimum fluidization. If the gas velocity is further increased, bubbles will form and the bubbling fluidized bed occurred. Hence, any addition of gas travels through the bed will become bubbles beyond the minimum fluidization velocity. Formation of slugging bed is determined when the size of bubbles become almost equal to the diameter of bed. The rising bubbles at this stage will combine and grow. The slugging bed is then transitioned into turbulent bed in which the voids of gas are no longer the same shape and size bubbles. The fluidized bed is finally becomes an entrained bed with increasing the gas velocity to superficial. Pneumatic transport of solids are formed (Cocco *et al.*, 2014; Kruggel-Emden and Vollmari, 2016).

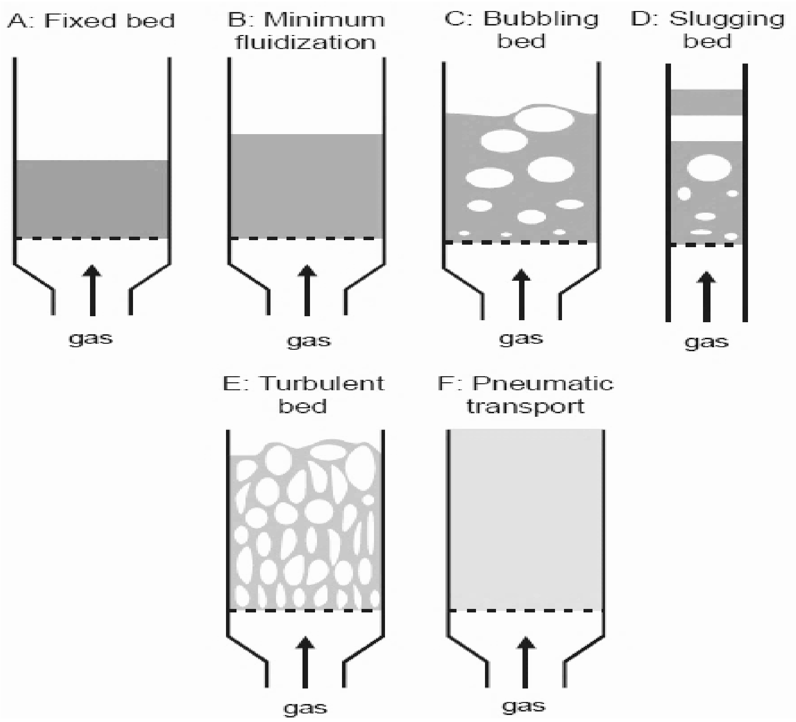

Figure 2.2: Schematic representation of fluidization regimes. Modified from(Cocco *et al*., 2014).

2.3 Fluidized Bed Granulation

Granulation process that utilized the technology of fluidized bed is known as the fluidized bed granulation. The development of fluidized bed granulation was began when Wurster reported his fluid bed experiments succeeded to prepare the coating compressed tablets using air suspension method in 1959 (Wurster, 1959). There were a lot other researchers studied in this field ever since the present of Wurster report. For instance, Rankell *et al.* (1964) has studied the effect of powder and liquid feed rates, temperature of inlet hot air and location of nozzle to properties of granule formed by applying the granule screen analysis, compressibility test and moisture content to appraise the granulated product. Furthermore, Vengateson and Mohan (2016) focused their study on the effect of binder flow rate and fluidizing air velocity to the granule size. Other process variables, such as atomizing pressure, air temperature in chamber, pattern of air flow were also studied.

Fluidized bed equipment is suitable to perform the wet granulation and it has been used in different kind of industries for more than 40 years, especially in pharmaceutical manufacturing. In fluidized bed granulation, particles are dispersed throughout the bulk of liquid which spraying from the top of the granulator. The particles are fluidized by the stream of air created by turbine fan suction and heated through air handling unit. The hot air is then blow through the distributor plate which locate at the base of vessel. The smaller the size of particles used, the better the performance of fluidized bed granulation. This statement was proved by the previous study that investigated by Roy *et al.* (2009a). It stated that if tiny solid particles are used, surface area of the particles exposed to the gas will become larger and this will easier the solid-gas heat and mass transfer.

There are a lot of advantages by using fluidized bed granulation, but also have its limitation. The advantages and disadvantages are listed down as shown in Table 2.1. Under this technique, the wetting, fluidization, granulation, coalescence and drying of granules are accomplished in the same apparatus, hence this process often known as one pot system (Jiménez *et al.*, 2006). Therefore, a good understanding about the function of fluidized bed granulator, fluidization theory, excipient interactions, parameters and variables that will affect the granule formation is important to avoid the failure of the process.