



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Physical Characterization of Urea-Lignin Fertilizer Granule

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

by

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Manufacturing Process). The members of the supervisory committee are as follow:

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(Principal Supervisor)

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ABSTRAK

Kajian ini adalah untuk menjalankan kajian mikrostruktur baja urea mengikat granul menggunakan lignin dengan berat yang berbeza. Mikroskop Elektron Pengimbasan (SEM) akan dikenakan sebagai peralatan analisis yang utama. SEM adalah sejenis mikroskop electron yang memaparkan imej sampel menerusi proses imbasan menggunakan alur elektron yang bertenaga tinggi. Skop kajian akan merangkumi urea sintetik dan lignin yang boleh didapati secara komersil. Granul dihasilkan menggunakan 'extruder'. Lignin diperolehi daripada sumber yang boleh diperbaharui terutamanya seperti pokok-pokok, tumbuhan-tumbuhan, dan tanaman pertanian. Lignin adalah bebas toksik dan ciri-ciri ini menjadikan ia satu produk yang sangat serba boleh untuk kegunaan dalam industri dan industri pemprosesan makanan. Lignin adalah pengikat yang sangat berkesan dan mesra ekonomi yang bertindak sebagai ejen yang mengikat atau sebagai gam kepada pelet baja.

ABSTRACT

This research is to conduct microstructure study on extruded urea fertilizer granule bind with lignin of various weight concentrations. Scanning Electron Microscope (SEM) will be used as the main analysis equipment. SEM is a type of electron microscope that images a sample by scanning it with a high energy beam of electron. The scope of study will cover on synthetic urea and lignin which are commercially available. The granule is produced using extruder. Lignins are derived from an abundant and renewable resource notably trees, plants, and agricultural crops. Lignin is nontoxic and these characteristics of it make it an extremely versatile product for use in industrial and food processing industries. Lignin is a very effective and economical adhesive acting as a binding agent or as glue to the pellets of the fertilizers.

DEDICATION

To my beloved parents who have always encouraged me to do my best and supported me wholeheartedly in everything I pursued.

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TABLE OF CONTENT

Abstrak	i
Abstract	ii
Dedication	iii
Acknowledgement	iv
Table of Content	v
List of Tables	vi
List of Figures	vii
List Abbreviations, Symbols and Nomenclature	viii
CHAPTER 1 : INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement	2
1.3 Objective	3
1.4 Scope of the Project	3
CHAPTER 2 : LITERATURE REVIEW	4
2.1 Urea Fertilizer	4
2.2 Fluidized Bed Granulator	6
2.3 Scanning Electron Microscope (SEM)	9
2.4 Lignin	11
2.5 Starch	12
2.6 Granulation Process	13
2.6.1 Excipients	14
2.6.2 Density	15
2.6.3 Dry Granulation	16
2.6.4 Wet Granulation	18

CHAPTER 3 : METHODOLOGY	19
3.1 Introduction	19
3.2 Raw Material	22
3.2.1 Synthetic Urea	22
3.2.2 Lignin	23
3.2.3 Soy Bagasse	24
3.2.4 Wheat Flour	24
3.3 Mixing Process of Material	25
3.3.1 Sample Composition	25
3.3.2 Samples Preparation	26
3.3.3 Mixing Temperature	27
3.4 Machine and Equipment	28
3.4.1 Extruder	28
3.4.1.1 Extruder Parts	29
3.4.2 Scanning Electron Microscope	31
CHAPTER 4 : RESULT AND DISCUSSION	34
4.1 Results of Experiment	34
4.1.1 Sample Obtained	35
4.1.2 SEM Observation	37
4.1.2.1 Raw Materials	38
4.2.2.2 Samples	40
4.2 Discussion	45
CHAPTER 5 : CONCLUSION AND FUTURE WORK	47
5.1 Conclusion	47
5.2 Recommendation	48
REFERENCES	49

LIST OF TABLES

2.1	Apparatus, process and product variables influencing fluidized bed granulation	8
3.1	Samples prepared	25
3.2	Temperature setting for each sample	27
3.3	Extruder parts	29

LIST OF FIGURES

2.1	Fluidized Bed Granulator	7
2.2	Schematic of the Chilsonator roller compaction process	16
3.1	Flow chart of the project	19
3.2	SEM block diagram	20
3.3	Pure Urea (CH ₄ N ₂ O)	22
3.4	Lignin	23
3.5	Soy Bagasse	24
3.6	Wheat Flour	24
3.7	Extruder	28
3.8	Scanning Electron Microscope	31
4.1	Sample 1	35
4.2	Sample 2	35
4.3	Sample 3	36
4.4	Sample 4	36
4.5	Sample 5	37
4.6	SEM micrographs of lignin	38
4.7	SEM micrographs of soy bagasse	38
4.8	SEM micrographs of wheat flour	39
4.9	SEM micrographs of urea	39
4.10	SEM micrographs of sample 1	40
4.11	SEM micrographs of sample 2	41
4.12	SEM micrographs of sample 3	42
4.13	SEM micrographs of sample 4	43
4.14	SEM micrographs of sample 5	44

LIST OF ABBREVIATION, SYMBOLS AND NOMENCLATURE

HFS	-	Horizontal Feed Screw
NH^3	-	Ammonia
NH_4^+	-	Ammonium
SEM	-	Scanning Electron Microscope
VPS	-	Vertical Pre-compression Screw

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Fertilizer is a material that is added to the soil to supply one or more elements required for plant growth and productiveness. One of the most popular types of fertilizer is urea fertilizer. The three major elements of urea fertilizers are nitrogen, potassium and phosphorus. Fertilizers will enhance the natural fertility of the soil as well as replacing the chemical elements that had been taken from the soil. Lignins derive from abundant and renewable resource: trees, plants, and agricultural crops. Because lignins are very complex natural polymers with many random couplings, the exact chemical structure is not well known. Lignin is a nontoxic and an extremely versatile product for industrial usage and food processing industries (Kirchner and Elisabeth, 1997).

1.2 Problem Statement

Urea is a white crystalline solid containing 46% nitrogen, is widely used in the agricultural industry as an animal feed additive and fertilizer. Urea is very soluble in water and hence requires better packaging quality. Urea is not as stable as other solid nitrogenous fertilizers. Urea decomposes even at room temperatures, particularly in a humid atmosphere releasing ammonia and carbon dioxide. The formation of ammonia and carbon dioxide results in serious loss. By adding lignin, the goodness in the lignin can be transferred to the fertilizer itself. Lignin is also a natural glue hence will be very suitable to act as the binder to the granule of the fertilizer. Lignin is in abundant and can easily be found so the use of lignin can help the manufacturers of the fertilizer to bind the fertilizer. In order to know the grain shapes, SEM can be used.

1.3 Objective

The goals of this project are:

- (a) To conduct microstructure study on urea fertilizer granule bind with lignin using various weight concentration.
- (b) To study whether lignin can be used as a good binder to the fertilizer.

1.4 Scope of the Project

The scopes of the project are:

- (a) Microstructure study of urea fertilizer granule bind with lignin using Scanning Electron Microscope (SEM).
- (b) Granule evaluated is produced using extruder.

CHAPTER 2

LITERATURE REVIEW

2.1 Urea Fertilizer

Urea is a very commonly found element in fertilizer. It consists of such great importance to the agriculture industry as a nitrogen-rich fertilizers that will enhance the natural fertility of the soil. In detail, it will actually replace the chemical elements taken from the soil by harvesting, grazing, leaching or erosion. Urea is commonly sold for fertilizer in 2 - 4 mm diameter granules. These granules are formed by spraying molten urea onto seed granules that are supported on a bed of air. Among the advantages of urea fertilizer includes costs which are cheap and it is immediately available due to the abundance of it in market. Although urea is naturally obtained, all urea fertilizers in market today are produced or synthesized in factories. On a commercial basis, urea is available as prills or as a granulated fertilizer (Copplestone and Kirk, n.d).

Commercially, fertilizer urea can be purchased as prills or as a granulated material. In the past, it was usually produced by dropping liquid urea from a "prilling tower" while drying the product. The prills formed a smaller and softer substance than other materials commonly used in fertilizer blends. Today, though, considerable urea is manufactured as granules. Granules are larger, harder, and more resistant to moisture. As a result, granulated urea has become a more suitable material for fertilizer blends (Curtis J. Overdahl, Rehm and Meredith, 1991).

Urea fertilizer does possess some advantages such as it can be applied to soil as a solid or solution or to certain crops as a foliar spray. It also involves little or no fire or explosion hazard. Urea's high analysis, 46% N, helps reduce handling, storage and transportation costs over other dry N forms. The urea manufacture releases few pollutants to the environment. When properly applied, it will results in crop yield increases equal to other forms of nitrogen. This advantages of urea makes it a manufacturers favourites. (Overdahl, Rehm and Meredith, 1991).

Urea is made from ammonia and carbon dioxide. The ammonia and carbon dioxide are fed into the reactor at high pressure and temperature, and the urea is formed through reaction of ammonia and carbon dioxide. The use of urea in single line urea granulation plants nowadays have increased rapidly every year. With increasing environmental awareness more and more attention is paid to the ammonia emissions from such plants. Due to the size of the plants there is a release of a considerable amount of ammonia from a single source or location, which raises concerns for the neighbours of such installations. On the other hand urea producers want to benefit from the economics of scale and are looking for ever increasing single line capacities for which they want to obtain operating permissions from regulatory authorities. Most of the ammonia is used on site in the production of urea (Potthoff, 2002).

The efficiency of urea in fertilizer can be altered by the use of slow release systems, chemical additives (e.g. acidifying agents, soluble salts), by altering granule size or soil incorporation. Most of these strategies have proved useful techniques to reduce ammonia emissions from urea fertilizer under specific circumstances, but their adoption into more general farming practice is unlikely to be practical, at present. (Watson, 2000)

2.2 Fluidized Bed Granulator

A fluidized bed is a bed of solid particles with a stream of air or gas passing upward through the particles at a rate great enough to set them in motion. As the air travels through the particle bed, it imparts unique properties to the bed. A granulator which receives the seed granules at one end and discharges enlarged granules at the other as molten urea is sprayed through nozzles. Fluidized bed granulator main function is to do the granulation process. Granulation process by this granulator is important to prevent segregation of the constituents of the powder mix. Segregation is due primarily to differences in the size or density of the components of the mix, the smaller and/or denser particles concentrating at the base of a container with the larger and/or less dense ones above them. An ideal granulation will contain all the constituents of the mix in the correct proportion in each granule, and segregation of the ingredients will not occur. It is also important to control the particle size distribution of the granules because, although the individual components may not segregate, if there is a wide size distribution the granules themselves may segregate. This machine is also important in order to compact the mix. Some powders are difficult to compact even if a readily compactable adhesive is included in the mix, but granules of the same formulation are often more easily compacted and produce stronger tablets. This is associated with the distribution of the adhesive within the granule and is a function of the method employed to produce the granule. Often solute migration occurring during the post-granulation drying stage results in a binder-rich outer layer to the granules. This in turn leads to direct binder bonding, which assists the consolidation of weakly bonding materials (Summers and Aulton, n.d.).

In fluidized bed granulator, the powder particles are fluidized in a stream of air, but in addition granulation fluid is sprayed from a nozzle on to the bed of powders. Heated and filtered air is blown or sucked through the bed of unmixed powders to fluidize the particles and mix the powders; fluidization is actually a very efficient mixing process. Granulating fluid is pumped from a reservoir through a spray nozzle positioned over the

bed of particles. The fluid causes the primary powder particles to adhere when the droplets and powders collide. Escape of material from the granulation chamber is prevented by exhaust filters, which are periodically agitated to reintroduce the collected material into the fluidized bed. Sufficient liquid is sprayed to produce granules of the required size, at which point the spray is turned off but the fluidizing air continued. The wet granules are then dried in the heated fluidizing airstream. Fluidized bed granulation has many advantages over conventional wet massing. All the granulation processes, which require separate equipment in the conventional method, are performed in one unit, saving labour costs, transfer losses and time. Another advantage is that the process can be automated once the conditions affecting the granulation have been optimized (Summers and Aulton, n.d.).

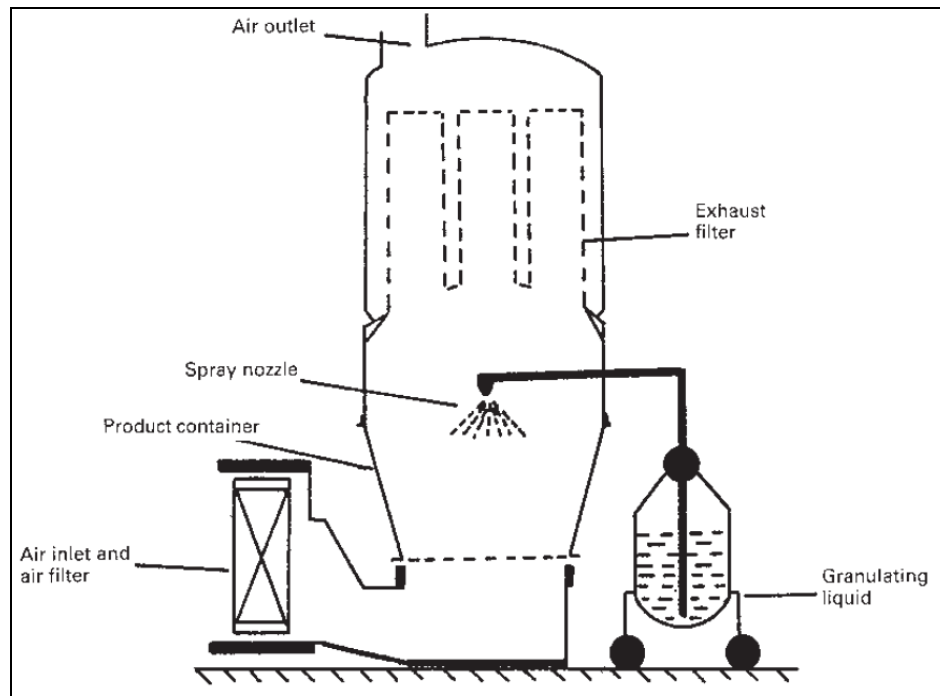


Figure 2.1: Fluidized Bed Granulator (Summers and Aulton, n.d.)

On the downside, the fluidized bed granulator is initially expensive and optimization of process parameters affecting granulation needs extensive development work, not only during initial formulation work but also during scale-up from development to production.

Table 2.1: Apparatus, process and product variables influencing Fluidized Bed Granulation (Summers and Aulton, n.d.)

(a) Apparatus parameters	(b) Process parameters	(c) Product parameters
i) Air distribution plate	i) Bed load	i) Type of binder
ii) Shape of granulator body	ii) Fluidizing air flow rate	ii) Quantity of binder
iii) Nozzle height	iii) Fluidizing air temperature	iii) Binder solvent

2.3 Scanning Electron Microscope (SEM)

Scanning Electron Microscope or SEM is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. SEM can produce very high-resolution images of a sample surface. The microscopes are great for seeing images "up close" that otherwise would be non-existent for the person viewing. SEMs are also easy to operate with the proper training. The scanning electron microscope has many advantages over traditional microscopes. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today (Schweithzer, 2003).

There is arguably no other instrument with the breadth of applications in the study of solid materials that compares with the SEM. The SEM is critical in all fields that require characterization of solid materials. While this contribution is most concerned with geological applications, it is important to note that these applications are a very small subset of the scientific and industrial applications that exist for this instrumentation. Most SEM's are comparatively easy to operate, with user-friendly "intuitive" interfaces. Many applications require minimal sample preparation. However, SEM does also possess some disadvantages and limitations such as Samples must be solid and they must fit into the microscope chamber. Maximum size in horizontal dimensions is usually on the order of 10 cm, vertical dimensions are generally much more limited and rarely exceed 40 mm. For most instruments samples must be stable in a vacuum on the order of 10^{-5} - 10^{-6} torr. Samples likely to outgas at low pressures (rocks saturated with hydrocarbons, "wet" samples such as coal, organic materials or swelling clays, and samples likely to decrepitate at low pressure) are unsuitable for examination in conventional SEM's. An electrically conductive coating must be applied to electrically

insulating samples for study in conventional SEM's, unless the instrument is capable of operation in a low vacuum mode (Swapp, 2006)

Because the SEM utilizes vacuum conditions and uses electrons to form an image, special preparations must be done to the sample. All water must be removed from the samples because the water would vaporize in the vacuum. All metals are conductive and require no preparation before being used. All non-metals need to be made conductive by covering the sample with a thin layer of conductive material. This is done by using a device called a "sputter coater." The sputter coater uses an electric field and argon gas. The sample is placed in a small chamber that is at a vacuum. Argon gas and an electric field cause an electron to be removed from the argon, making the atoms positively charged. The argon ions then become attracted to a negatively charged gold foil. The argon ions knock gold atoms from the surface of the gold foil. These gold atoms fall and settle onto the surface of the sample producing a thin gold coating (Schweithzer, 2003)

2.4 Lignin

Lignins are derived from an abundant and renewable resource such as trees, plants, and agricultural crops. Lignosulfonates in lignin are a very effective and economical adhesive, acting as a binding agent or “glue” in pellets or compressed materials. Lignin is nontoxic and an extremely versatile product for use in industrial and food processing industries. Currently, Lignin is produced mainly as a byproduct of the paper industry, separated from trees by a chemical pulping process. The physical and chemical properties differ depending on the extraction method. For example, lignosulfonates are hydrophilic and will dissolve in water. These lignins are products of sulfite pulping. Kraft lignins are hydrophobic and will not dissolve in water. Kraft lignins are obtained from the Kraft pulping process. Other technologies use an organic solvent or a high-pressure steam treatment to remove lignins from plants. The usefulness of commercial lignosulfonates products comes from their dispersing, binding, complexing and emulsifying properties. Lignosulfonates have found applications as a raw material in the production of vanillin. Vanillin is widely used as an ingredient in food flavors, in pharmaceuticals and as a fragrance in perfumes and odor-masking products (U.S. National Center for Environmental Research, n.d).