



FORMULATION OF POLYMERIC BINDER SYSTEM IN UREA FERTILIZER CONTAINING BIODEGRADABLE PVA AND STARCH

This report is submitted in accordance with requirement of the University Teknikal
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Sekian dimaklumkan. Terima kasih.

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DECLARATION

I hereby, declared this report entitled “Formulation of polymeric binder system in urea fertilizer containing biodegradable PVA and starch” is the result of my own research except as cited in references.

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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 requirement for Degree of Manufacturing Engineering (Engineering Materials) (Hons). The member of the supervisory committee are as follow:

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ABSTRAK

Tujuan kajian ini adalah untuk merangka sistem pengikat polimer dalam baja urea yang mengandungi biodegradasi polyvinyl alcohol (PVA) dan kanji. Bahan-bahan mentah telah disifatkan oleh analisis saiz zarah (PSA) dan spektroskopi penjelmaan Fourier infra-merah (FTIR). Pengikat ini telah disediakan melalui timbangan, pencampuran dan pemanasan bahan komposisi mentah menggunakan alat pemanas makmal. Sampel baja telah disediakan menggunakan fluidized bed granulator (FBG) dengan mengepam serbuk urea itu ke dalam ruang serbuk. Parameter mesin seperti pengabusan tekanan udara, kekerapan kipas, suhu masuk untuk pengeringan dan semburan dipilih berdasarkan kajian terdahulu. Jumlah pengikat dan kadar tambahan telah diselaraskan dengan sewajarnya. Selepas proses granulasi selesai, granul yang dikumpul perlu di ayak untuk anggaran saiz. Berdasarkan piawaian industri granul harus terdiri daripada 2 mm hingga 4 mm. Granul dikeringkan di dalam ketuhar selama tiga jam pada suhu 45 ° C sebelum analisis lanjut dilakukan menggunakan FTIR untuk ikatan polimer dan ujian biji kekerasan. Biodegradasi dan ujian penyerapan air telah dijalankan bagi ujian degradasi urea komposit. Ketumpatan urea komposit diukur menggunakan densimeter. Ia menunjukkan bahawa urea komposit yang mempunyai peratusan tertinggi iaitu kanji sebanyak 15% lebih bermanfaat untuk kajian ini. Analisis kerencaman oleh FTIR menunjukkan bahawa urea-PVA-kanji telah terikat secara kimia antara satu sama lain. Kemikroskopan electron imbasan (SEM) menunjukkan bahawa imej granul urea adalah terdiri daripada penumpuan granul yang lebih kecil yang dilampirkan dan terikat bersama-sama untuk membentuk butiran lebih besar.

ABSTRACT

The aims of the study are to formulate the polymeric binder system in urea fertilizer containing biodegradable polyvinyl alcohol (PVA) and starch. The raw materials were characterized by particle size analyzer (PSA) and Fourier transform infrared radiation (FTIR). The binder was prepared through weighing, mixing and heating the raw materials composition using a laboratory heating plate. Sample of fertilizer were prepared using a fluidized bed granulator (FBG) by pumping in the urea powder into its chamber. The machine parameters such as atomization air pressure, frequency of the fan, inlet temperature for drying and spraying were selected based on the previous work. The amount of binder and its addition rate were adjusted accordingly. After the granulation process finished, the collected granules were sieved and estimated the size. Based on industrial standard the granules should range from 2 mm to 4 mm. The granules were dried in the oven for three hours at 45°C before further analysis using FTIR for polymeric bonding and granule hardness test. The biodegradability and water absorption test were carried out for the degradation of composite urea test. The density of the composite urea was measured using densimeter. It showed that the composite urea contained highest percentage of starch which is 15% more beneficial for this study. The compositional analysis by FTIR showed that urea-PVA-starch were chemically bonded to each other. The scanning electron microscopy (SEM) images demonstrated that the urea granules were comprised of agglomeration of smaller granules attached and bonded together to form bigger granules.

DEDICATION

My beloved father, Othaman b. Latif

My appreciated mother, Rohana bt Dollah

My respected supervisor, Dr. Toibah bt Abdul Rahim

My beloved co-supervisor, Assc. Prof. Dr. Azizah bt Shaaban

My adored siblings, sisters and brothers

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LIST OF ABBREVIATIONS

| | | |
|--|---|---|
| BSFBG | - | Bottom Spray of Fluidized Bed Granulation |
| C | - | Carbon |
| CRF | - | Control Release Fertilizer |
| CO(NH ₂) ₂ | - | Carbamide |
| FBG | - | Fluid Bed Granulation |
| FTIR | - | Fourier Transform Infrared Spectroscopy |
| H | - | Hydrogen |
| K | - | Potassium |
| KNO ₃ | - | Saltpetre |
| O | - | Oxygen |
| OH | - | Hydroxyl |
| N-H | - | Nitrogen Hydrogen |
| C-H | - | Carbon Hydrogen |
| C-C | - | Carbon Carbon |
| MSDS | - | Material Safety Data Sheet |
| N | - | Nitrogen |
| NaNO ₃ | - | Soda Nitrate |
| NH ₂ CONH ₂ | - | Urea |
| NH ₂ CONHCH ₂ OH | - | Urea Formalaldehyde |

| | | |
|--------|---|--|
| HPMC | - | Hydroxypropyl Methylcellulose |
| P | - | Phosphorus |
| pH | - | Potential of Hydrogen |
| PSA | - | Particle Size Analyzer |
| PVP | - | Polyvinylpyrrolidone |
| PVA | - | Polyvinyl Alcohol |
| SEC | - | Simple Event Correlator |
| SEM | - | Scanning Electron Microscopy |
| TSFBG | - | Top Spray Fluidized Bed Granulation |
| TGSFBG | - | Tangential Spray Fluidized Bed Granulation |

LIST OF SYMBOLS

| | | |
|--------------------|---|------------------------------|
| w/v% | - | Weight Per Volume percentage |
| °C | - | Degree Celsius |
| kgf | - | Kilogram Force |
| MPa | - | Mega Pascal |
| % | - | Percentage |
| mm | - | Millimetre |
| Mn | - | Average of Molecular Weight |
| Mw | - | Molecular Weight |
| g/cm ³ | - | Gram per cubic centimetre |
| µm | - | Micronmetre |
| mm s ⁻¹ | - | Millimetre per second |
| c | - | Solution Concentration |
| [η] | - | Intrinsic Viscosity |
| g | - | gram |
| kV | - | kilovolt |
| Hz | - | Hertz |
| W ₁ | - | Initial Weight |
| W ₂ | - | Final Weight |

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Maxwell (2006) stated that in the late 1990's, the granules has been preferred more than prills or crystal by the fertilizer industry. In spite of the fact that the compound properties of both prills and granules stay comparable, their diverse physical and mechanical properties are distinguishable and make them reasonable for various application either as fertilizer or crude materials for agricultural industry. Solid manure might be finely pounded, granulated, blended and prepared into uniform prills which come in three sorts for example plain, which is the most normally utilized. Second, Pelleted or coated, which are uniform and more expensive. The other type is next is slow release which is covered with one of the few substances that cause the nutrients to be discharged over a period of time, usually from three to nine months. Prills such as urea fertilizer are small, easy to handle, lightweight, and more readily accept the moisture.

In the prilling system, the concentrated urea dissolve is bolstered to a punctured turning bucket/shower-top spray head situated at the highest point of prilling tower. The bucket turned at rapid and spray the urea liquefy as droplets. Liquid droplet is solidified and cooled on free fall through the tower against a constrained or regular updraft of surrounding air. The product is removed from the tower base to a conveyor line. Cooling and filtering to the surrounding air may be used before the product is transferred to storage (Alamdari et al., 2000). In this recent years, extensive fertilizer is produced as granules. It is because the quality of prills is significantly lesser than granules, the primary contrast being the lower

quality is smaller size of prills. Granules form into a larger, harder, long storage life and more resistance to moisture. Nowadays, granules have become a choice for the user as a fertilizer.

Over the last decades, fluidized bed granulation (FBG) knew as one of the techniques for granulation process in pharmaceuticals and other industrial operations. This technique usually used for wet granulation that required the occurrence of liquid binder to essential particles to produce the agglomeration of granules. FBG additionally forms a method that can reduce material handling and abbreviates shorten time process compare with another wet granulation process (Srivasta and Mishra, 2010). There are a couple of method under this technique that applicable for wet granulation process, for instance, Top Spray, Bottom Spray, and Tangential Spray Fluid Bed Granulation process. In pharmaceuticals industries, the formulation of paracetamol has been included with a liquid polymer binder for example polyvinylpyrrolidone (PVP) (Kadajji and Betageri, 2011). The polymeric binder helped this process to enhance powder properties such as flowability and compressibility for downstream processing.

In this FBG, flowability is important properties that should be considered because it will influence the weight consistency and production of granules. Compressibility needs to form a stable intact compact mass when pressure connected. In order to form consistent and reproducible properties of granules, the powder must flow easily and uniformly into granules dies. During this two-stage process that incorporates spraying and drying, the addition of a binder liquid will be the reason to the essential particles to aggregate and form granules. Therefore, the proficiency and control of granules development during the manufacturing process are most significance to the conveyance of a high-quality final product.

1.2 PROBLEM STATEMENTS

Fertilizers come in different forms, grades and formulations. They commonly used in a form of solid granules and liquid. The chemical or urea-based type of fertilizers have higher nitrogen content which is required for plant growth. However, its application is always related to the *greenhouse effect* that warms the planet's surface, which can cause global

warming and gives negative affects to human and environmental. In order to apply urea fertilizer effectively, reformulation of the fertilizer need to be done. The use of biodegradable polymer binder that coat and bind urea particles may slow down the release of potential ammonium leaching and gaseous loss simultaneously may increase the plant growth rate and yield. Urea particles are small, irregular shape and do not flow well due to the cohesive surface characteristics. Therefore, in this study, a new formulation of binder into urea granulation process will be explored to minimize the above-mentioned problems. A good size distribution of granules and suitable binder will improve the flow and compressibility properties of the granules.

1.3 OBJECTIVES

- 1) To synthesize biodegradable urea fertilizer with PVA and Starch through granulation technique.
- 2) To examine biodegradability of the polymeric binder system.
- 3) To characterize the properties of as-synthesized urea polymeric bonding in urea fertilizer.

1.4 SCOPE

This project focused primarily on the function of biodegradable polymeric binder system for granulation process to make uniform granules that will be used as a solid fertilizer in agriculture industry. The fluidized bed granulation process was used to prepare the granules. In this work, commercially available urea powder was mixed with biodegradable binder prior the granulation process. Moreover, for biodegradable materials, PVA and starch have been selected to be used. Granules that produced via FBG have been characterized on their physical and chemical properties by using hardness test, densimeter, water absorption test, SEM and FTIR.

1.5 SIGNIFICANCE OF STUDY

This study is significant in promoting the production of granules fertilizer for reducing environmental problems. This study will also be beneficial for the farmers and other fertilizer's user when they used this new reformulation of granules fertilizer. By understanding the problems and the benefits of the fertilizer quality, the fertilizer's user will get the advantages of this reformulation. Moreover, this study will be beneficial to the agricultural industry as the production of granules fertilizer will increase due to the excellent properties of the material use for the fertilizer. It also will serve as a future reference for researcher about granulation process. In addition, this research will educate the fertilizer's user in deciding to choose whether the chemical or inorganic fertilizer is more suitable to be supplied into the soil.

1.6 CHAPTER OVERVIEW

The organization of this report is as follow; Chapter 1 provides the introduction of the research background, then followed by problem statement, objective, scopes of research and report organization. Chapter 2 comprise of literature review of previous research finding on application of starch, polyvinyl alcohol (PVA), urea fertilizer and others. Chapter 3 covers the methodology of the research which include the flowchart, raw materials, sample preparation, testing and analysis. Chapter 4 presents the comprehensive discussion on the result obtained throughout the study. Conclusion and recommendation experimental of this study is presented in Chapter 5.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Fertilizer comes in many arrangements, for example, liquid and granular. About 90% of fertilizer are applied as a granular. Fertilizer contains one or more nutrient that are needed in plants. It provides plants with all sorts of nutrients in order to help plants grow strong and healthy. A few of the main macronutrient in the fertilizer including nitrogen (N) for leaf growth, phosphorus (P) for enhance the development of roots, flower and seeds, and potassium (K) for helping the movement of water in plants (Azeem et al., 2014). Fertilizer is classified in a few ways; it is according to the nutrient in the fertilizer whether it provide a single nutrient or multi-nutrient. In addition, fertilizer can be divided into two essential group such as mineral and chemical fertilizer. Vitosh (1996) has stated that fertilizers are natural or synthetic chemical compound containing nutrients essential for the normal growth and development of plants. Generally, all carbon (C) and oxygen (O) are necessary to the plants and are provided via photosynthesis, which from carbon dioxide and oxygen gases from the atmosphere. Meanwhile, rain and ground waters are source to supply all the hydrogen (H) required for the process. A fertilizer product is a material produced by industrial processes with the specific purpose of being used as a fertilizer. Fertilizers are essential in today's agricultural system to replace the elements extracted from the soil in the form of food and another agricultural product (IFDC and UNIDO, 1998).