CHARACTERIZATION OF STRENGTH AND STRUCTURE OF GRANULAR AND PRILLED UREA FERTILIZER

By

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(Matrix no.: 12200)

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Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

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TRONOH, PERAK, MALAYSIA
September 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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STEWARD BABA ANAK GAIT

ABSTRACT

Solid urea fertilizers can be produced in two forms: granules and prills. The study of comparison between granules and prills is nearly non-existence and hence it becomes the ultimate goal of this project. Due to different physical structures, granular and prilled fertilizers are distinguishable in terms of their structural strength and properties while their chemical properties remain similar. In Malaysia, there are currently two operating plants producing granular urea, namely Asean Bintulu Fertilizer (ABF) and PETRONAS Fertilizer Kedah (PFK). Comparison of products from these two companies was being investigated. The scope of work includes size distribution, surface morphology, dynamic strength, solubility and moisture content. More than 50% of ABF and PFK granules have the size range around 3.00mm. Prills have an average size of 1.66mm. SEM images of granules exhibited the aggregation of crystals to form a single urea particle while prills appear as one unit. The sticking out structure of granules makes them tend to stick to one another while prills have good flowablity. Naturally, granules have greater strength due to the stacking of crystals which prevent instant damage to the urea. This justification was verified as more fine particles are produced from prills when being milled which defined the idea that prills are easily crushed. For a fixed mass of urea fertilizers, prills dissolve faster in water due to larger surface area. Granular urea fertilizers have higher amount of moisture content compared to prills which causes it have higher tendency to form cakes during storage due to the cohesive nature of water. In short, granular urea fertilizers are relatively better for its superiority compared to prills.

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ABBREVIATIONS AND NOMENCLATURES

ABF	Asean Bintulu Fertilizer
FESEM	Field Emission Scanning Electron Microscope
FIAM	Fertilizer Industry Association of Malaysia
FYP	Final Year Project
LFSU	Lab Facilities and Safety Unit
NAP	National Agricultural Policy
NPK	Nitrogen-Phosphorus-Potassium
PFK	PETRONAS Fertilizer Kedah
RPM	Revolutions per minute
STR	Staged Nutrient Release
UAN	Urea ammonium nutrient
UTP	Universiti Teknologi PETRONAS
VP	Variable Pressure

CHAPTER 1
INTRODUCTION

1.1 BACKGROUND OF STUDY

Malaysia agriculture is one of the main drives of economy in the country for being one of the leading producer of palm oil in the world and the third largest producer of natural rubber. Agriculture was given serious consideration by the government after economic crisis hit in 1997 with the objective to reduce food importation bill. The Third National Agricultural Policy or NAP3 was formulated for 1998-2010 for the strategic agricultural development master plan. It aimed to achieve food self-sufficiency and to develop in an efficient and competitive manner. The challenge is to increase exports and reduce imports of agricultural commodities. This has made agriculture sector to progress rapidly over the years. In other words, this expands the need of cultivation which triggers the urge for more efficient usage of agricultural inputs. For instance, fertilizers have to be used to extensively to sustain the production of high yield crop and ultimate profitability. A study done by Stewart, Dibb, Johnston and Smyth in 2005 shows that 40%-60% of crop yield in the world is due to the use of commercial fertilizer. In Europe, the fertilizer market is expected to grow to 15.3 billion by 2018. With a total land area of 327, 733 km^2, Malaysia tropical soil tends to be highly leached and infertile and hence, the application of fertilizers is very essential. Oil palm is the largest consumer of fertilizer in Malaysia due to its abundant plantations. Fertilizer Industry Association of Malaysia (FIAM) was established in the year 1987 to regulate the use of fertilizer. (Food and Agriculture Organization of the United Nations, 2004)

Fertilizer is naturally or synthetically produced chemical compound to supply essential nutrients into the soil for plant growth. Fertilizer can be inorganic or inorganic depends on the source. Organic fertilizer originates from plant or animal matter. It takes months or years for this type of fertilizer to release nutrients into the soil. Organic fertilizer breaks down slowly into complex structures (humus) which builds the soil structure and moisture and nutrient-retaining and cap. Comparatively, organic fertilizer provides lower concentration than inorganic fertilizer which helps to avoid over fertilization. Despite the fact that using organic fertilizer supports the idea of going green, it does have its disadvantages such as problem of economic collection, treatment, transportation and distribution. Chemically synthesized inorganic fertilizer only widely developed during Industrial Revolution and was further expanded due to the pre-industrial British Agricultural Revolution and the industrial Green Revolution of the 20th century. The use of inorganic fertilizer is significant as nearly half of the world population in the world is fed because of its use. In Malaysia, the main fertilizers used are urea, ammonium sulphate, calcium ammonium nitrate, phosphate rock, super phosphates, ammonium phosphate, potassium chloride, potassium sulphate and nitrogen-phosphorus-potassium (NPK), nitrogen-phosphorus (NP) and phosphorus-potassium (PK) compound fertilizers. (Food and Agriculture Organization of the United Nations, 2004)

The common macronutrients provided by the fertilizer include nitrogen, phosphorus, potassium, calcium, magnesium and sulfur. These are consumed in large quantities as they constituent about 0.15% to 6.0% of plant tissue. Micronutrients such as boron, chlorine, copper, iron, manganese, molybdenum and zinc occupy the plant tissue only up to 0.4%. Carbon, hydrogen and oxygen are the major parts of the plant are provided by water and carbon dioxide. Plants can only absorb the required nutrients in easily dissolved chemical compounds. Nitrogen is usually absorbed in the form of ammonia and nitrates. Phosphorus and potassium are taken up by the plant as phosphoric acid and potassium chloride respectively. (Otero, Vitoria & Canals, 2005)

Fertilizers are in several forms. There are five main physical forms of solid fertilizers and two types of liquid fertilizer. The end product of solid fertilizer production can be further processed into granules, prills, compact, pellet and powder. Regardless of different in mechanical properties, they still supply similar nutrients. Liquid fertilizers can be either solution such as urea ammonium nutrient (UAN) where soluble nutrient sources are dissolved or suspension by keeping finely divided nutrient particle suspended in water. Liquid fertilizer has immediate effect as it is readily absorbed by the soil and can be used in wide coverage. For solid fertilizer, it will need to dissolve beforehand just by the use of water. If the soil is too enriched with nutrients, this will cause "burning" of the plant. Technology manages to produce fertilizer spikes and tabs which are slow-release fertilizer. This is done by polymer coating. This gives the fertilizer a "true time-release" or "Staged Nutrient Release" (SNR) fertilizer nutrient. 'Multicote" is a process applying layers of low-cost fatty acids with a paraffin topcoat. Coating does not have adverse effect on urea fertilizer properties. (Juneno, 2012)

1.2 PROBLEM STATEMENT

Malaysia produces only granular urea fertilizers via two operating plants, Asean Bintulu Fertilizers (ABF) in Bintulu, Sarawak and PETRONAS Fertilizers Kedah (PFK) in Gurun, Kedah. Prilled urea fertilizers are also available for sale in Malaysia but they are imported from other countries such as China and Europe. The current literature is mostly on the characterization on non-urea granules and study on prilled urea fertilizer is nearly non-existent. Above all, direct comparison between granular (specifically ABF's and PFK's) and prilled urea fertilizers is absent. It is essential to identify the key properties of these urea fertilizers for future reference in the local industry.

1.3 OBJECTIVE AND SCOPE OF STUDY

In Malaysia agriculture, the utilization of granulated fertilizer is more popular than that of prilled ones. This project comes with the following objectives:

- To characterise prilled and granular urea fertilizers
- To compare the different mechanical properties of prilled and granular urea fertilizer.

The objectives are the ultimate goal at the end of this project accomplishment to identify the differences of prilled and granular urea fertilizers. The scope of this project is to be conducted within Universiti Teknologi PETRONAS (UTP) environment and loosely attached to Malaysian agriculture application. The work includes:

- To determine the size distribution of the urea fertilizer
- To study the surface morphology
- To perform dynamic test to test the strength of the urea fertilizer
- To study the solubility
- To determine the moisture content

All the urea samples are expected to be obtained from local manufacturing plants such as PETRONAS Fertilizer Kedah (PFK) and Asean Bintulu Fertilizer (ABF). The project limits to study the effect of technology and size on the mechanical properties and the characterization of prilled and urea fertilizer as clearly stated in the objectives.

1.4 RELEVANCY OF THE PROJECT

The project is not closely-related and in-line with the syllabus of chemical engineering program of UTP which are structured based on oil and gas industry. Nevertheless, the application of chemical analysis is being utilized extensively in this project in studying the properties of the urea fertilizers. The knowledge of using the equipment in obtaining the necessary data is much related to the course Chemical Analysis which is taught in the final year. From another perspective, this project is relevant to the current scenario in Malaysia in which agriculture is given emphasis due to enforcement of NP3. Application of fertilizer is established throughout Malaysia and the outcome of this final year project can be used as reference for related study in the area.

1.5 FEASIBILITY OF THE PROJECT

This project is expected to be accomplished within a period of two semesters which commences on first week of May semester itself (21st May, 2012). It is sufficient to have the project done within the stipulated time considering the campus has adequate facilities to perform some of the tests in house whereas the other tasks might permit the need to outsource. It is realistic to have the tasks done in September semester. Towards the end of the semester, project dissertation should have been done which includes the final analysis and conclusion. Objectives of the project shall be met prior to the accomplishment.

CHAPTER 2
LITERATURE REVIEW

In Malaysian agriculture, the use of potassium fertilizer is more extensive than any other fertilizers for palm oil plantations. (Food and Agriculture Organizations of the United Nations, 2004) Nonetheless, nitrogenous fertilizer is fairly favourable for domestic use in maintaining lawns and gardens. Nitrogen can be added to the soil in many forms (Refer to Table 2.1). Fertilizers are easily available and hence, civilians tend to overfertilize their gardens which results in "burning" of the plants and damage the leaves due to high mineral salt concentration in the soil. Fertilizers high in nitrogen discourage flowering and favor leafy growth. Excess nitrogen may be leached into the groundwater in the form of nitrate, a common pollutant. (Andrews, 1998)

Table 2.1: Forms of nitrogen and their sources (Andrews, 1998)

Forms of nitrogen	**Source**
Organic nitrogen	• Animal manure • Compost • Plant residues
Urea	• Commercial fertilizer • Fresh manure
Ammonium (NH_4^+)	• Chemical fertilizers such as ammonium nitrate and ammonium sulfate • Fresh manure • Breakdown of organic matter into the soil
Nitrate (NO_3^-)	• Chemical fertilizer such as ammonium nitrate and potassium nitrate
Nitrogen gas (N_2)	• About 80% of air within soil spaces

Typical production of nitrogenous fertilizer imposes several environmental issues such as air emissions, wastewater, hazardous materials, wastes, and explosion. Greenhouse gases such as carbon dioxide and nitrous oxide are the common gases and in some cases, particulates are emitted from prilling. Wastewater and liquid effluents include process wastewater discharges from ammonia, urea, nitric acid, ammonium nitrate and calcium ammonium nitrate plants. The most common hazardous wastes produced are spent catalysts after their replacement in gas desulphurization, ammonia plants and nitric acid plants. Not to mention the main product, ammonia can be hazardous itself if not being handled properly. Common causes of fire and explosions in nitrogenous facilities include accidental release of synthetic gas in ammonia plants or combustion of ammonium nitrate, an oxidizing agent in the ammonia plant. (Ivorychem, 2012)

Urea is commonly used nitrogenous fertilizer in agriculture. It is naturally produced in the urine of mammals with the chemical formula of $CO(NH_2)$. Urea is considered organic due to its carbon content. For commercial use, the synthesis of urea involves the combination of ammonia at pressure as high as 21MPa pressure and operating temperature of 177^oC under the following reactions:

$$CO_z + 2NH_3 \rightarrow CO(NH_2)_2 + H_2O$$

The water is removed by dehydration. (Gilgames, 2001) The first step is fast and exothermic and essentially goes to completion under reaction conditions industrially. The dehydration is slower and endothermic and does not go to completion. The main product will be further processed for its desirable use, either solid prilled or granular product. With the prilling technique, the concentrated urea melt is fed to a rotating bucket/shower-type spray head located at the top of prilling tower. Liquid droplets are formed which solidify and cool on free fall through the tower against a forced or natural up-draft of ambient air. The product is removed from the tower base to a conveyor belt. Cooling to ambient temperature and screening may be used before the product is transferred to storage. With granulation, a less concentrated urea feedstock is used. The lower concentration allows the hydration to be eliminated and simplifies the process condensate treatment. This process involves the spraying of the melt onto recycled seed particles or prills circulating in the granulator. Air passing through the granulator solidifies the melt deposited on the seed material. Conditioning the melt prior to spraying can enhance the storage/handling characteristics of the granular urea. Urea fertilizer is also available coated slow release pellets. It can be used as strong nitrogen component to solid combination fertilizers such as urea phosphate.

Final product of urea comes in different forms but they are of the same chemical composition. Both have minimum nitrogen content of 46% and melting point of 132^oC. With pure white appearance, they contain 160 ppm of free ammonia. Prilled urea is the finest presentation and has the quickest release time. Granular urea fertilizer is coarser and takes a longer time to break down. The comparison of prilled and granular urea fertilizer can be seen in Table 2.2.

Table 2.2: Comparison of prilled and granular urea fertilizer (SABIC, 2012)

Prilled Urea	**Aspects**	**Granular Urea**
0.3% max	Moisture	0.5% max
1% max	Biuret	1.4% max
-	Formaldehyde (HCHO)	0.45% min
1 – 4mm 90 – 94% min	Granulation	2 – 4 mm 90 – 94 % min
1-2.8 mm 90% min	Particle Size	2-4 mm 90%min > 4mm 7% max +2.8mm .60% min

Biuret is a chemical compound that produced when two molecules of urea condense It is a common contaminant that disrupts the purity of urea fertilizer. It inhibits plant growth and in high content, it can be toxic.

Urea is used as nitrogen fertilizer worldwide. This is essentially because of its 46% nitrogen content which is the highest among other nitrogen fertilizers. Urea is readily soluble in water but the application in liquid is not a common practice. In the soil, urea is converted to ammonium ions by a series of enzyme reactions. Under normal condition, the ammonium ions are absorbed by the soil where they become attached to the negatively charged soil particles and the nitrogen is then made available for the plant to take up. Urea-derived ammonium ions act in the same way like from any other nitrogenous fertilizer. The process of breaking down into ammonium ions from urea usually takes one week.

Compared to other nitrogenous fertilizer, urea has its own benefits. Urea can be applied as a solid or solution as a foliar spray. This permits the versatility of using urea everywhere from aerial plantations to the farm spreading by hand. Urea does not have any fire or explosion hazard. It is less corrosive to the equipment. Urea is considered to be a low cost nitrogen fertilizer form. The high nitrogen content reduces handling, storage and transportation cost over other dry nitrogen form. It is safer to ship and can be stored and distributed through conventional systems. On the other hand, the process of manufacturing urea releases few pollutants into the environment. Provided the urea is properly applied, it works the same as any other nitrogenous fertilizer. Urea can be used in all crops. It can readily dissolve into the soil due to its high solubility in water. Urea is considered to be mobile as it is a neutral molecule. It is not charged and can rapidly move into the soil followed by rain or irrigation where it is converted to ammonic nitrogen and attached to the soil particle and hence, prevent leaching loss. (Ivorychem, 2012)

CHAPTER 3
METHODOLOGY

3.1 RESEARCH METHODOLOGY

There are a number of aspects to look into before starting the investigation on the differences of prilled and granular urea fertilizer. A problem statement needs to be identified as this will be the ultimate motivation to propel this project towards its completion. When objectives set, a series of activities or tasks can be proposed and performed to solve the problem. For this project, the major task involved is to characterize the urea fertilizer of different forms and determine the physical and mechanical properties. It is understood that chemical composition for both forms should be similar to each other. Main emphasis shall be put to study the mechanical properties of the fertilizer. The results will be further analysed and discussed in reaching the final conclusion of the project.

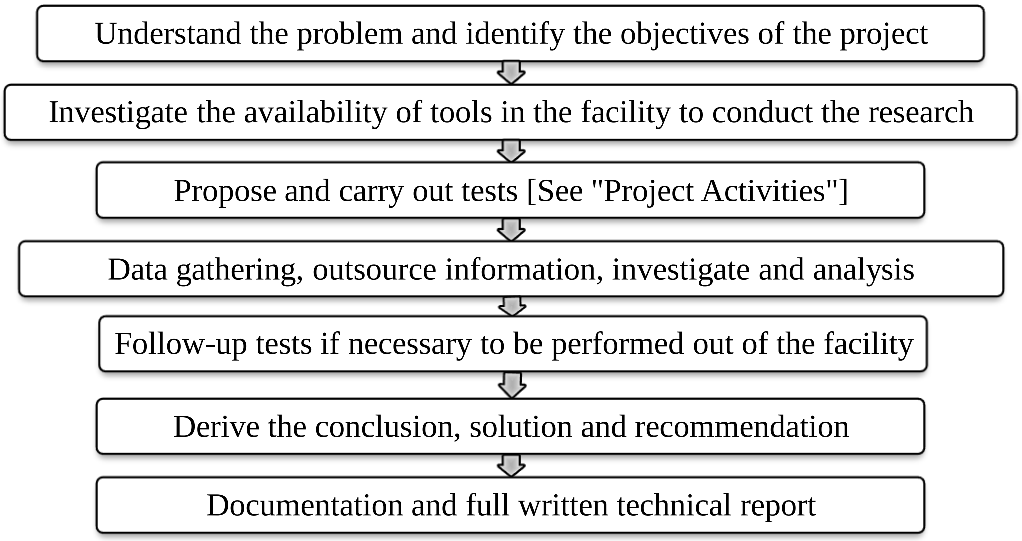


Figure 3.1: Flow process of research methodology

3.2 PROJECT ACTIVITIES

Fresh samples of granular urea fertilizers were obtained from ABF and PFK. Approximately five hundred grams of granular fertilizers were used throughout the project for sufficient characterization. Comparison of PFK's and ABF's fertilizers was performed due to the possible different technologies in producing the granular fertilizers. Prilled fertilizer was purchased from Shenyang Jin Cheng Ji Rui Commercial Trade Co.,Ltd, China. General characterization was deduced from

ABF's and PFK's granular urea fertilizers and subsequently, the results were compared with the characteristics of prilled urea fertilizers. Labeling of each urea is shown as in Table 3.2.

Table 3.2 Sample labeling

Sample 01	ABF Granular Fertilizer
Sample 02	PFK Granular Fertilizer
Sample 03	Prilled Fertilizer

All samples were firstly sieved in obtaining the size distribution of the urea fertilizers and afterwards, average diameter size for each sample was determined. The characteristics of the fertilizer were then analyzed based on the respective sizes range for reasonable comparison. Internal and external structures of granules and prills were examined as well to study the correlation on the properties. The following tests describe the procedures in details.

3.2.1 Sieve Analysis

The purpose of sieve analysis is to obtain size distribution of the urea fertilizers. A sample of about 150 g for each type of fertilizer was sieved. A series of 8 BS standard sieves (4.75mm, 3.35mm, 2.80mm, 2.36mm, 2.000, 1.18mm, 0.710mm, and 0.60mm) were used for the analysis and vibrated by D450 Digital sieve shaker (Endecotts, England) for 5 minutes. The selected amplitude selected was 0.70mm.

The average diameter of urea fertilizers were calculated based on method proposed by Allen (1990). The mean diameter was calculated based on the following equation:

$$d_w = \sum_{i=1}^{n-1} \left[\frac{w_i d_i}{w_t}\right] \quad (1)$$

Where

d_i = mean diameter of collecting sieve and the one above

n = number of sieves including bottom plate

w_i = mass of granules/prills remain on each sieve

w_t = total sieve mass

3.2.2 Surface Morphology

Single particle of size about 2.00mm for each sample were examined using Zeiss SUPRA-55 VP Field Emission Scanning Electron Microscope (FESEM) to study the surface morphology. Four magnifications of 35x, 100x, 500x and 2000x were employed to study all the samples. SEM was operated using voltage of 20.00kV.

3.2.3 Dynamic Strength Test

Ball milling was performed on all the samples where 30g of urea fertilizers were run using 300g of stainless steel milling media. The strength of each sample was correlated to the amount of powder formed after each run. There was no current literature used ball milling on urea fertilizers and hence, it was necessary to run few runs in determining optimum operating conditions to rationally compare the strength of the urea samples. It came down to two selected operating conditions which are shown in the table 3.2.3.

Table 3.2.3 Operating conditions of ball milling

Mode	Revolutions per minute (rpm)	Period (minutes)
Intense	80	30
Mild	30	10

For intense mode, it was done started from granules expecting the granules would crush at the end of the run where prills would crush totally. On the other hand, prills were first to be tested for the mild mode expecting they would crush partially where granules would stay intact. The outcome of milling were analysed qualitatively and quantitatively. The crushed urea fertilizers were sieved using 212 µm tray. Urea fertilizers that were filtered out of this tray were considered fine particles and this amount corresponded to the strength of urea.

3.2.4 Solubility Test

10g of urea samples were immersed in 100ml of water to investigate the solubility of urea in the water. All three samples were tested under room temperature and little swirling action was added to speed up the reaction. Amount of time needed for the first sample to dissolve was recorded in identifying the urea which has the fastest solubility in water.

3.2.5 Moisture Content

Approximate 20g of urea fertilizers were put into the oven under temperature of 100^{o}C for 45 minutes. The mass of samples before and after drying were measured.

The difference in the mass was assumed to be the amount of water in the water. Moisture content of urea fertilizers was calculated using the following formula:

$$Moisture\ content = \frac{final\ \ mass - initial\ \ mass}{initial\ \ mass} \times 100\% \qquad (2)$$

3.3 KEY MILESTONE

Table 3.3: Overall FYP key milestone

Week	Date	Activities/Remarks
FINAL YEAR PROJECT I		
1	21^{st} – 27^{th} May	Meeting FYPI co-ordinator and supervisor
2	28^{th} May – 3^{rd} June	Confirmation of title
3	4^{th} – 10^{th} June	Literature Search and LFSU Briefing
4	11^{th} – 17^{th} June	Preliminary research work
5	18^{th} – 24^{th} June	
6	25^{th} June – 1^{st} July	Submission of extended proposal
7	2^{nd} – 8^{th} July	Preparation for proposal defense
8	9^{th} – 15^{th} July	Proposal defense
9	16^{th} – 22^{nd} July	Preliminary investigative work and analysis
10	23^{rd} – 29^{th} July	
11	30^{th} July – 5^{th} August	
12	6^{th} – 12^{th} August	
13	13^{th} – 19^{th} August	
14	20^{th} – 24^{th} August	Submission of interim report
	25^{th} – 29^{th} August	STUDY WEEK
	30^{th} August – 9^{th} September	FINAL EXAMINATIONS WEEK
	10^{th} – 17^{th} September	SEMESTER BREAK
FINAL YEAR PROJECT II		
1	18^{th} – 23^{rd} September	Meeting FYPII co-ordinator and briefing
2	24^{th} – 30^{th} September	Research/Experiment continues
3	1^{st} – 7^{th} October	
4	8^{th} – 14^{th} October	
5	15^{th} – 21^{st} October	Adjunct lecture
6	22^{nd} – 28^{th} October	Briefing on writing thesis
7	29^{th} October – 4^{th} November	
8	5^{th} – 11^{th} November	Submission of Progress Report
9	12^{th} – 18^{th} November	
10	19^{th} – 25^{th} November	
11	26^{th} November – 2^{nd} December	
12	3^{rd} – 9^{th} December	Preparation for dissertation and viva

3.4 GANTT CHART

Final Year Project I

Timeline (Week)	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
First meeting with co-ordinator and supervisor														
Regular meeting with supervisor														
Confirmation of project title														
Literature search and LFSU briefing														
Preliminary research work														
Methodology/Project activities proposal														
Submission of extended proposal														
Proposal defense														
Investigate the availability of lab equipment														
Identify the possibility of outsourcing														
Requesting for samples														
Preliminary investigative work and analysis														
Submission of draft interim report														
Submission of interim report														

Final Year Project II

Timeline (Week)	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
First meeting with co-ordinator and supervisor														
Regular meeting with supervisor														
Research Work continues/Experiment begins														
Submission of Progress Report														
Experiment continues														
Data Analysis														
Pre-SEDEX														
Submission of Draft Report														
Submission of Technical Report														
Submission of Dissertation														
Oral Presentation (VIVA)														

3.5 TOOLS

Table 3.5: Tools used in the project

SIEVE SHAKER	
Model	Endecotts D450 Digital Sieve Shaker 
Description	It is a totally operator controlled. It is ideal for sieves up 18” in diameter. It is a powerful test sieve shaker designed to offer outstanding control feature. It is fitted with a unique clamping system which ensures those sieves are held firmly without over tightening and allows them to be quickly removed and replaced. The shaker is powered by an electromagnetic drive which has no rotating parts to wear making it virtually maintenance free and extremely quiet in operation.
FIELD EMISSION SCANNING ELECTRON MICROSCOPE	
Model	Zeiss SUPRA-55 VP Field Emission Scanning Electron Microscope 
Description	Excellent imaging properties combined with analytical capabilities makes this high end FE-SEM suitable for a wide range of applications in materials science, life science and semiconductor technology. The large specimen chamber for the integration of optional detectors and accessories enables the user to configure the SUPRA for specific applications without sacrificing productivity or efficiency. The unique variable pressure (VP) capability of SUPRA enables examination of non-conducting specimens without time consuming preparation

SOLID HANDLING STUDY EQUIPMENT	
Model	SOLTEQ Model BP 102 
Description	This model has been designed to introduce students of Chemical Engineering to a single but different aspect solids behaviour. The unit mainly consists of a sieve shaker, a cylindrical hopper, a horizontal cylinder, a mixing vessel, a ball mill and cyclone and pneumatic conveying system. Each item can be run independently over a series of laboratory periods.  The stainless steel ball mill, cylindrical in shape, is charged with grinding material to be used to study comminution history of a batch of granular solids. The charge and rotational speed may be varied by controlling the speed of the motor. The capacity is 5 litres. The milling media available are stainless steel and zygonite balls.

OVEN	
Model	Universal oven Model UF350plus 
Description	The universally applicable oven is the classic appliance for temperature control in science, research and material tests in industry. The technologically perfected masterpiece made of high-quality; hygienic, easy-to-clean stainless steel leaves nothing to be desired in terms of ventilation and control technology, over temperature protection and precisely tuned heating technology.

LAB APPARATUS

		
Evaporating Dishes	**Conical Flask**	**Weighing Balance**

CHAPTER 4

RESULTS & DISCUSSION

4.1 SIEVE ANALYSIS

4.1.1a Sample 01: ABF Granular Urea Fertilizer

Table 4.1.1 shows the raw sieving result of ABF granular urea fertilizer and the size distribution is clearly shown in Figure 4.1.1.

Table 4.1.1 Sieve result for ABF granular urea fertilizer

Sieve opening (mm)	Mass of urea (g)	Cumulative mass (g)	Mass percent (%)	Cumulative mass percent (%)
4.75	0	0	0	0
4.00	1.22	1.22	0.80	0.80
3.35	18.07	19.29	11.81	12.61
2.80	58.00	77.29	37.91	50.52
2.36	55.90	133.19	36.54	87.06
2.00	14.90	148.09	9.74	96.80
1.18	4.90	152.99	3.20	100
0.71	0	152.99	0	100

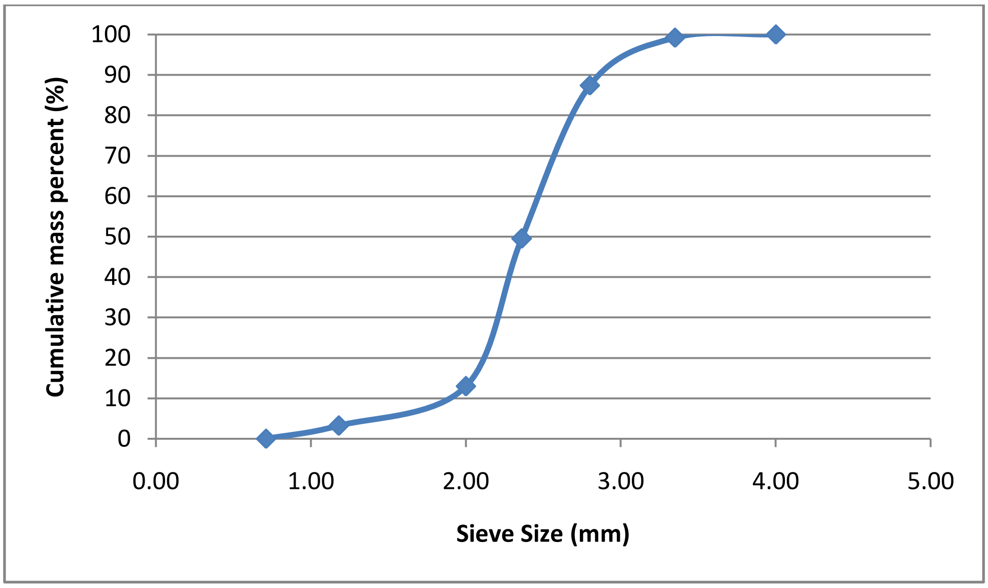


Figure 4.1.1 Size distribution of ABF granular urea fertilizer

Using the formula, the average diameter of ABF granule is 2.84 mm.

4.1.1b Sample 02: PFK Granular Urea Fertilizer

Table 4.1.2 shows the raw sieving result of ABF granular urea fertilizer and the size distribution is clearly shown in Figure 4.1.2.

Table 4.1.2 Sieve result for PFK granular urea fertilizer

Sieve opening (mm)	Mass of urea (g)	Cumulative mass (g)	Mass percent (%)	Cumulative mass percent (%)
4.75	0	0	0	0
4.00	1.23	1.23	0.81	0.81
3.35	49.67	50.90	32.59	33.40
2.80	74.02	124.92	48.56	81.96
2.36	20.90	145.82	13.71	95.67
2.00	5.50	151.32	3.61	99.28
1.18	1.10	152.42	0.72	100
0.71	0	152.42	0	100

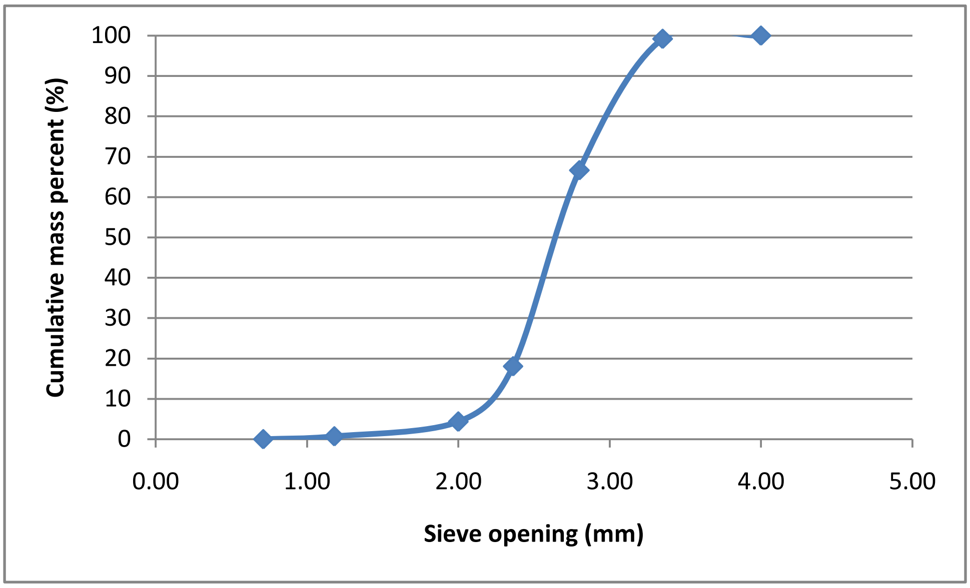


Figure 4.1.2 Size distribution of PFK granular urea fertilizer

Average diameter of PFK granule is 3.17 mm.

4.1.1c Sample 03: Prilled Urea Fertilizer

Table 4.1.3 shows the raw sieving result of ABF granular urea fertilizer and the size distribution is clearly shown in Figure 4.1.3.

Table 4.1.3 Sieve result for prilled urea fertilizer

Sieve opening (mm)	Mass of urea (g)	Cumulative mass (g)	Mass percent (%)	Cumulative mass percent (%)
2.80	0	0	0	0
2.36	2.40	2.40	1.58	1.58
2.00	18.60	21.00	12.28	13.86
1.18	126.70	147.70	83.63	97.49
0.71	3.80	151.50	2.51	100
0.60	0	152.10	0	0

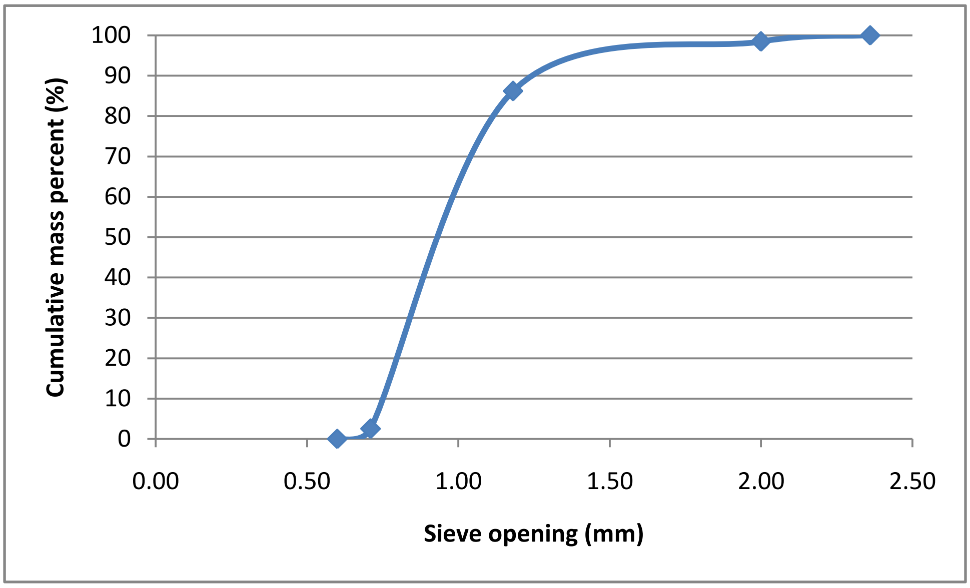


Figure 4.1.3 Size distribution of prilled urea fertilizer

Average diameter of prill is 1.66 mm.

4.1.2 Discussion

From visual examination, both granules and prills are generally spherical. Figure 4.1.4 shows that granules have larger size or diameter than that of prills. Out of the two granules, PFK's is slightly larger, with the difference of 0.33 mm. More than 50 percent of PFK granules are of the size 2.8 mm. For ABF, more than half of the granules are of the size 2.36 mm. Most of the prills sizes are around 1.18mm. Different in sizes for the same mass of urea fertilizers lead to different total surface area. This parameter affects the solubility of urea fertilizer where this property is very important in agriculture. Urea fertilizers need to be dissolved in water first before readily absorbed by the plant. The outcome of solubility test is explained in the later section.

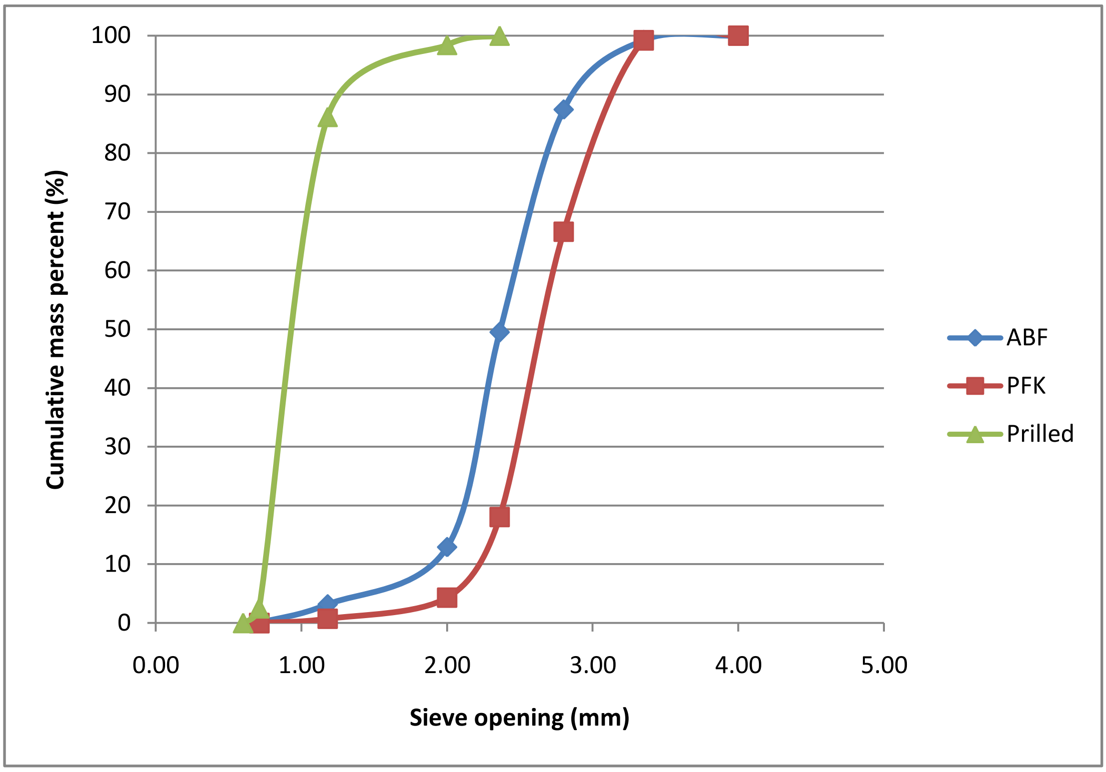


Figure 4.1.4 Size distribution comparison of all three urea fertilizers

4.2 SCANNING ELECTRON MISCROSCOPE IMAGES

4.2.1 Result

Figure 4.2.1 shows SEM images of all three samples in four magnifications: 35x, 100x, 500x and 2000x.

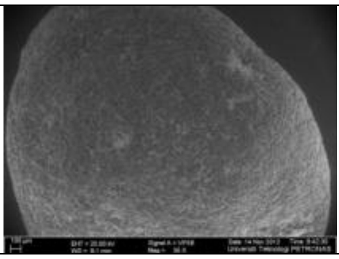
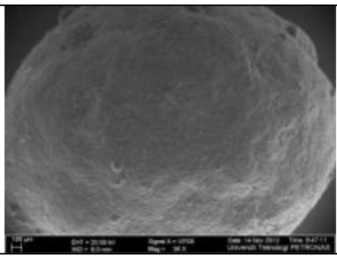
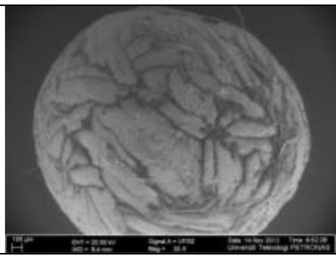
ABF Granules	PFK Granules	Prills
		

Figure 4.2.1a: SEM images of all three samples in 35x

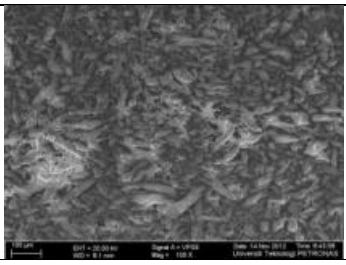
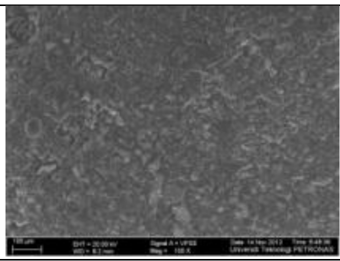
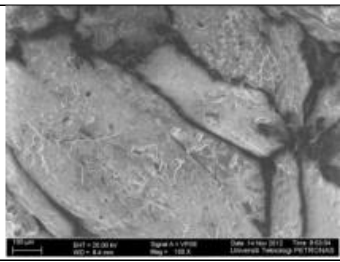
ABF Granules	PFK Granules	Prills
		

Figure 4.2.1b: SEM images of all three samples in 100x

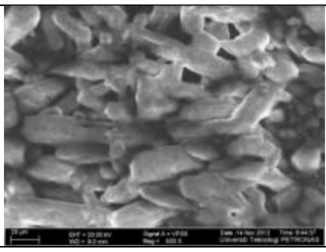
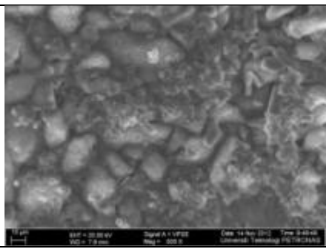
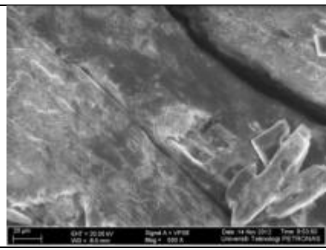
ABF Granules	PFK Granules	Prills
		

Figure 4.2.1c: SEM images of all three samples in 500x

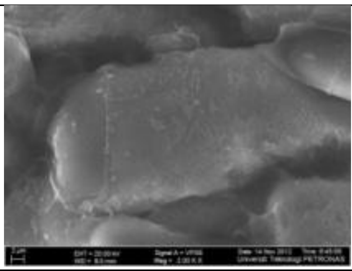
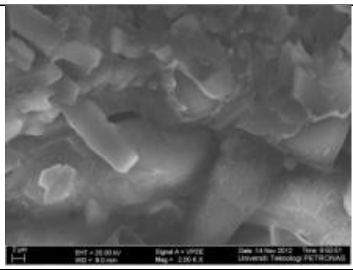
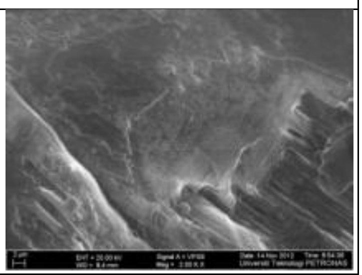
ABF Granules	PFK Granules	Prills
		

Figure 4.2.1d: SEM images of all three samples in 2000x

4.2.2 Discussion

Figure 4.2.1a shows that the shapes of granular and prilled urea fertilizers are nearly spherical. In term of size, granules appear larger than prills.

From the examination of granules at 100x (Figure 4.2.1b), it can be seen that for a single particle of urea granule comprises many crystals that aggregate. ABF and PFK granules seem more compacted than prills. Due to these structures, this makes granules to be relatively stronger than prills. This is because as granules receive impacts externally, the crystals will break down successively which prevents complete or instant damage to the particle. On the other hand, prill is made up of a single unit and hence, once it is broken, the whole structure breaks apart.

Under the same magnification of 500x (Figure 4.2.1c), it can be seen that ABF's granules are more porous than PFK granules. Porosity is relative to the strength of the urea fertilizers. The empty space within the urea fertilizers gives way to the particles to break down when urea fertilizers are being crushed. Also, the arrangement of crystals in ABF's granules is more compact than PFK's. Reasonably, this makes ABF's granules to be weaker than PFK's and yet still stronger than prills.

With magnification of 2000x (Figure 4.2.1d), prills appear to have smoother surface than granules. The aggregation of crystals in granules makes them have uneven surface. Physically, as the urea fertilizers are made to flow within the sample holders, prills have better flowability than granules. The sticking out structure makes the granules tends to lump to one another. This is very fundamental property where it affects the caking for urea storage.

Generally, SEM images provide physical justifications for key properties of urea fertilizers. Some of these properties are verified in the next few results sections.

4.3 BALL MILLING (DYNAMIC STRENGTH TEST)

4.3.1a Visual Examination

Figure 4.3.1a shows he granules crushed a little at the end of milling process where prills became powder completely with no trace of prills at all.

ABF granules	PFK granules	Prills
Operating conditions: 80 rpm, 30 minutes		
		

Figure 4.3.1a Physical result for intense mode

Figure 4.3.1b shows the granules remain completely intact where prills are partially crushed into powder.

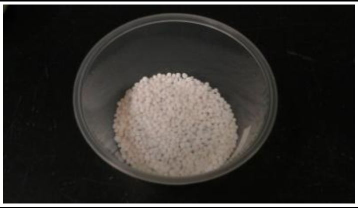
ABF granules	PFK granules	Prills
Operating conditions: 40 rpm,10 minutes		
		

Figure 4.3.1b Physical result for mild mode

4.3.1b Quantitative Result

Fine particles were defined of powder size less than 212 µm. The mass of powder was determined after sieving the milling result using single tray. The percentage of fine particle produced is as shown in Table 4.3.1b.

Table 4.3.1 Percentage of fine particles after milling

ABF granules	PFK granules	Prills
Operating conditions: 80 rpm, 30 minutes		
9.73%	1.42%	11.60%
Operating conditions: 40 rpm, 10 minutes		
0%	0%	6.39%

4.3.2 Discussion

Strength of the urea fertilizers are relatively based on the fine particles formed from milling process. The powder formed from milling was sieved and the size is generally above 212 µm. Any collected particle which has size less than 212 µm is considered to be fine particles. More fine particles were collected from milled prills which means they were more fragile than granules. The reasons were well-explained in the justification of SEM images. The aggregation of crystals in granules makes it stronger than prills. The porosity of ABF granules makes it less strength than PFK's and this can be seen as more fine particles were produced when ABF granules were milled under intense conditions. In the ascending order, the strength increases from prills, ABF granules and PFK's granules have the higher strength.

4.4 SOLUBILITY TEST

4.4.1 Result

Time taken for urea fertilizers to be completely dissolved in water is shown in Figure 4.4.

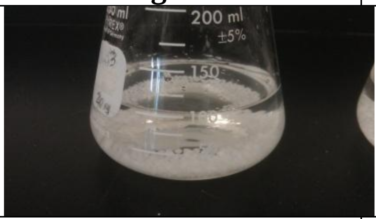
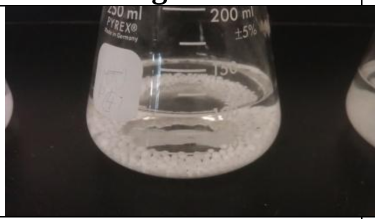
ABF granules	PFK granules	Prills
		
15 minutes	11 minutes	9 minutes

Figure 4.4: Time taken for urea fertilizers dissolve in water

4.4.2 Discussion

It took shorter time for prills to dissolve in the water than granules which makes prills have higher solubility than granules. For fixed mass of urea fertilizers, this is relatively reasonable due to larger surface area of prills than granules. The large surface area gives more rooms for urea fertilizers to diffuse into the water. Solubility is very essential property of urea fertilizers in agriculture. Urea fertilizers need to dissolve sufficiently to be absorbed by plant. Meanwhile, if urea fertilizers dissolve too fast in the soil, the soils can be over fertilized and cause burning in crops.

4.5 MOISTURE CONTENT

4.5.1a Visual Examination

Physical condition of urea fertilizers after drying is shown in Figure 4.5.1a. At the end of drying process, granules stick to one another as it was not easy to remove the dried urea fertilizers from the evaporating dishes. Prills were still able to flow easily.

ABF granules	PFK granules	Prills
		

Figure 4.5.1a Physical result after drying

4.5.1b Quantitative Result

Difference in mass is calculated using equation (2) and the moisture content is shown in Table 4.5.1b.

Table 4.5.1 Moisture content of urea fertilizers

ABF granules	PFK granules	Prills
0.18%	0.11%	0.05%

4.5.2 Discussion

Moisture content of urea fertilizers determines the quality of urea fertilizers. On the other hand, it is also to abide the legal and labeling requirements. Granules have generally higher moisture content than prills. The moisture content has directly relationship to the usual caking issue for urea storage. The higher the content of water inside urea fertilizers, it tends to form lump or caking. It is due to cohesive property of water and hence when two particles of moisture content come in contact with one another, they will stick together. In other words, granules have higher tendency to form cakes than prills during storage.

CHAPTER 5
CONCLUSION & RECOMMENDATIONS

Physical structures of urea fertilizers hold the fundamental key to mechanical properties. Chemical properties of all urea fertilizers are theoretically the same due to the same chemical composition in which the chemical formula is $CO(NH_2)_2$. Their nitrogen content is 46% and appears as white crystalline substance. Urea is highly soluble in water.

The key difference of granules and prills is the aggregation of crystals of large size granules. Other than being larger, granules have greater physical strength meanwhile prills faster solubility in water. The high moisture content in granular urea fertilizers cause them to form caking easier than that prills during storage. After considering all the characteristics from tests done in this project, granules are relatively more superior to prills. This shall explain the reasons the wider application of granular urea fertilizers.

The ultimate objectives of this project are achieved where characterization and successively, the comparison of granular and prilled urea fertilizers were accomplished throughout this project.

Recommendations

Further tests can be done to characterize urea fertilizers. For instance, porosity of urea fertilizers is important which correlates to the physical strength. Evaporation method (drying method) is not the best way to measure the moisture content due to the low percentage. Karl-Fisher method is the best universally accept in determining the amount of water inside the urea fertilizers. Internal structures of urea fertilizers can be further analyzed using X-Ray scan to have greater justifications for overall mechanical properties of the urea fertilizers. Due to unavailability of equipment, the strength of urea fertilizers were tested and determined relatively to the physical outcome of dynamic test performed which relied on the fine particles parameter. More accurate result can be obtained through the use microhardness tester where the machine can provide instant numerical result.

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