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The 7th World Congress on Particle Technology (WCPT7)

## Urea Finishing Process: Prilling versus Granulation

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### Abstract

Solid urea is the largest nitrogen fertilizer product which is produced in two forms of granules and prills. Although the chemical properties of both prills and granules remain similar, their different physical and mechanical properties are distinguishable and make them suitable for different application either as fertilizer or raw materials for chemical industry. The objective of this work is to analyse physical and mechanical properties of urea granules produced in two different plants in Malaysia using fluidized bed process and compare them with the imported urea prills to the country; hence make a process-product relationship for urea finishing processes. Results of size distribution of the samples show that the most of the granules fall in the size range between 2.40 and 3.50 mm, whereas the prills size is around 1.60 mm. Strength measurement using side crushing test also shows that the prills with the average failure load of 3.80 N remain significantly weaker than the granules with failure load of 10-17 N. Strength distribution of the particles also shows that a more uniform strength distribution is observed for the prills than the granules. It is concluded that the urea prilling process is the finishing process which produces the weaker and the more uniform size and strength of the particles than the fluidized-bed granulation process.

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*Keywords:* Urea; Prills; Granules; Prilling; Strength

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### 1. INTRODUCTION

The global population growth over the past four decades has caused rapid growth of agricultural activities which in turn has increased the demand of fertilizer worldwide. In particular nitrogen fertilizers are the most common types used in the world of agriculture [1, 2]. Nitrogen can be found in many different forms and from a number of sources as listed in Table 1. [3] Among these sources urea tends to be the dominant nitrogen fertilizer used in agriculture. [4,

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5] The phenomenal rise in the production of urea over the past few decades without any serious competitors reflects its essential role in the world of agriculture and other various sectors [5-10].

Urea is considered as an organic compound due to its carbon content. It is also known as carbonyldiamide as two amide groups are attached to the sides of a carbonyl functional group, with the chemical formula being  $\text{CO}(\text{NH}_2)_2$ . Urea can be found naturally in the urine of mammals or produced artificially through the synthesis reaction of carbon dioxide with ammonia at a pressure as high as 21 MPa and a temperature of 180 °C. For commercial use, urea is mainly produced in a solid form, either as prills or granules depending on the finishing process being used. For this either a prilling tower or a granulator is used at the finishing stage [11].

In prilling technique, the concentrated urea melt is fed to a perforated rotating bucket/shower-type spray head located at the top of the prilling tower. The bucket is rotated at high speed and sprays urea melt in the form of droplets. Liquid droplets are solidified and cooled 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 [11].

In granulation technique, a rotating high-velocity air stream is established in the central agglomeration tube. Particles are picked up at the base of the tube and accelerated by the air stream. The particles come into contact with the fluid droplets produced from the spray nozzle at the base of the tube. The relative velocity of particles, air and fluid droplets are high so wetting is efficient and drying begins almost immediately. During granulation, urea melt is sprayed onto the circulating recycled prills (or seed particles) while air passes through the granulator and solidifies the melt deposited on the seed material. In some cases, urea melt is conditioned prior to spraying. This can enhance the storage and handling characteristics of the granular urea [12, 13].

In recent years, there has been an increasing amount of literature on the modelling and simulation of urea granulation processes. [11-15]. The characterisation of solid particles is of significant importance to investigate the properties of materials and further of their performance in all aspect of usage. The motivation of this work was to understand the technical reason behind importing urea fertilizer in the form of prills to Malaysia, while the produced granules is more than the demand of this this country, hence is exported. Therefore, it was aimed to evaluate the relationship between different urea finishing processes and the corresponding physical strength of the product. This makes it essential to assess the mechanical strength of both prilled and granulated urea fertilizers obtained from different plants in Malaysia. According to their manufacture, it is expected that the granules have a greater mechanical strength than the prills, so that a strength gradient exist in accordance with the radius of the grain [16]. The objective of this study was the direct determination of the strength profile of both granulated and prilled urea samples. In addition, the thermal characteristics of the respective urea samples were compared to assess the differences in their thermal behaviour.

Table 1. Forms of nitrogen and their potential sources. [3]

Forms of nitrogen	Source
Organic nitrogen	Animal manure, compost and plant residues
Urea	Commercial fertilizer and fresh manure
Ammonium ( $\text{NH}_4^+$ )	Chemical fertilizers such as ammonium nitrate and ammonium sulfate, fresh manure and breakdown of organic matter into the soil
Nitrate ( $\text{NO}_3^-$ )	Chemical fertilizer such as ammonium nitrate and potassium nitrate
Nitrogen gas ( $\text{N}_2$ )	About 80% of air within soil spaces

## 2. EXPERIMENTAL

### 2.1. Materials

Fresh samples of urea fertilizer were obtained for performing tests. Granulated urea product was obtained from two different plants in Malaysia, that for confidentiality purposes they are named as sample 1 and 2, while prilled urea product was obtained from a commercial supplier.

### 2.2. Sieve analysis

To observe the size distribution of granulated and prilled urea products, sieve analysis was carried out by sifting them through several sieves, progressively more fine meshed. A series of 13 BS standard sieves was used during the analysis and vibrated by Endecotts D450 digital sieve shaker for 20 minutes. About 110 g of each urea sample was sifted with sieves of the following successive opening sizes; 4.75, 4.00, 3.35, 2.80, 2.36, 2.00, 1.70, 1.40, 1.18, 1.00, 0.850, 0.71 and 0.600 mm.

### 2.3. Strength measurement

All urea samples were tested experimentally to observe their breakage or failure behavior under compressive loads. The test was carried out using an Instron mechanical testing machine with a maximum load of 100 N and a crosshead speed of 0.05 mm min<sup>-1</sup>. About 30 to 50 granules were tested for each sample for the purpose of reliability checking. During the test, each granule was selected randomly from its container so some granules had bigger size than others for the same kind. The granules were mounted on the anvil of the instrument in a random orientation. The software of the Instron equipment (Bluehill) was used for the surveillance of the failure load of the grains. The test was also done, but without the samples. Thus, just the bottom section of the loading tool was loaded on the anvil to determine the machine extension. The sample size was computed by subtracting the original extension (penetration) from the machine extension. The apparent strength was calculated by dividing the failure load (force) applied per cross-sectional area perpendicular to the force

### 2.4. Thermal analysis

Differential scanning calorimetry (DSC) was performed using Mettler Toledo differential scanning calorimeter. Urea samples of approximately 5 mg were analysed in vented aluminium pans under dry nitrogen purge (at 50 mL min<sup>-1</sup>) over the temperature range of 0 to 350 °C at a heating rate of 5 °C min<sup>-1</sup>. The DSC instrument was calibrated with an aluminum pan before experiments.

Thermogravimetric analysis (TGA) was performed using Mettler Toledo thermogravimetric analyzer. Urea samples of approximately 10 mg were analysed over the temperature range of 25 to 750 °C with nitrogen as a purge gas flowing at 200 ml min<sup>-1</sup>.

## 3. RESULTS AND DISCUSSION

### 3.1. Sample size distribution

Figure 1 shows the size distribution of all three urea samples obtained from sifting. As shown in Figure 1, in general the granulated urea samples (1 and 2) are larger and have a fairly broader size distribution than the prilled urea sample. The latter result suggests that samples 1 and 2 have less dimensional consistency than prills. It can be seen that the granules of sample 2 are slightly larger than that of sample 1 with the size difference being about

0.25 mm. About 55% (by weight) of sample 2 granules are of 3.00 mm size and about 45 % (by weight) of sample 1 and prills are 2.80 mm and 1.65 mm, respectively. Table 2 provides the mass-mean diameter of all three samples. These values were calculated assuming that urea granules were spherical.

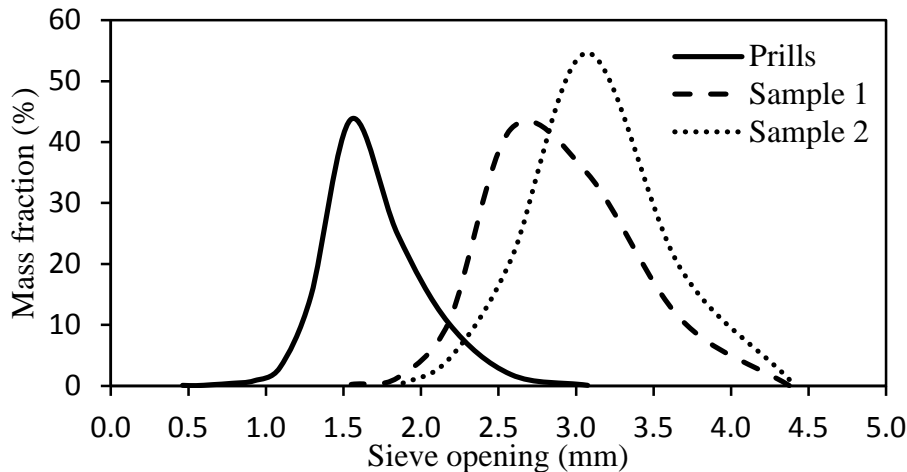


Figure 1. Size distribution of urea samples.

Table 2. Mass-mean diameter of urea samples.

Sample	Mass mean diameter (mm)	Standard deviation (mm)
Prills	1.65	0.10
Sample 1	2.82	0.19
Sample 2	3.06	0.17

The size distribution of granules can have several impacts on physical properties of urea. For example prilled urea takes less time to dissolve in water than the granulated urea as of its small size. While prilled urea has a high dimensional consistency (Figure 1), granulated urea is easier to handle and store.

### 3.2. Strength characterisation

From a naked eye observation both prilled and granulated urea samples were of a fairly uniform spherical shape. The frequency-failure load data for granule failure are shown in Figure 2. The data relate to the frequency of granule crushing at a particular compression force (failure load). The results show that prilled urea demonstrated the most uniform strength (failure load) distribution, while the other two failure load distributions are broader and slightly tailed. This phenomenon indicates that both granulated samples have a lower dimensional consistency than the prilled urea. The apparent difference simply reflects the different internal structure of granules.

Figure 3 shows the results of strength measurement for different granule sizes (diameters) of each urea sample. It can be seen that, the apparent granule strength of all urea samples decreases as size of the granule increases (with some exception). In general, most of the fine sample granules reached the load failure quicker than the coarse granules. The results show that for sample 1, the granule strength varies between 1.02 MPa and 3.38 MPa for a granule size of 2.30 mm and 2.56 mm, respectively. The results illustrate that sample 2 has the coarsest granules among the other two samples. The failure strength of sample 2 varies from 4.35 MPa to 1.82 MPa depending on the size of the granules. The results show that prilled urea has a failure load ranging from 1.68 MPa to 2.55 MPa, depending on the size of the granules. The average apparent strength of all three samples is presented in Table 3. In

comparison, the sample 2 granules showed the highest stability (2.87 MPa) on average compared to the granules of sample 1 (2.47 MPa) and prills (2.0 MPa). Prilled urea demonstrated the lowest stability because of its small sample size. All the results were obtained from a typical behaviour of the load-extension relationship. However, the determination of the failure load can become complicated when the latter behaviour is untypical or in another word, when some of the graphs show unusual behaviour. The main reason of such trend when the behaviour is untypical is due to the angular or sub angular internal structure of some granules.

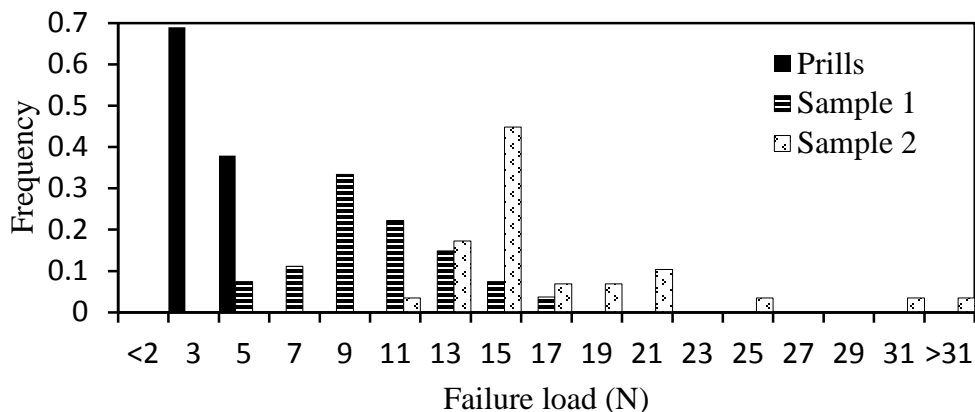


Figure 2. Failure load distribution of urea samples.

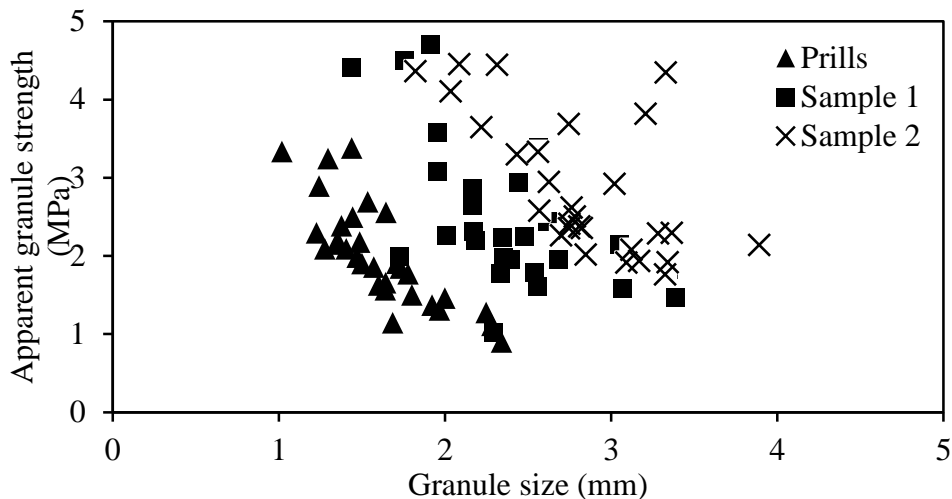


Figure 3. Failure load distribution of urea samples.

Table 3. Average strength of urea samples.

Sample	Average apparent strength (MPa)	Standard deviation (MPa)	Coefficient of variation
Prills	2.0	0.10	0.32
Sample 1	2.47	0.19	0.38
Sample 2	2.87	0.17	0.31

### 3.3. Thermal characterisation

Figure 4 shows the TGA data plot for all three urea samples. In the TGA data plot three major stages of mass loss are present, each of which are associated primarily with different pyrolysis reactions of urea. Several attempts have been made to investigate the thermal decomposition of urea [17-18]. Schaber *et al.* [17] performed a similar series of experiments to analyse the evolved gases and residue obtained from the thermal decomposition of urea. In their study, Schaber *et al.* found four stages of mass loss out of which the first three were the major ones. According to their findings, the loss observed between 160 and 250 °C (Figure 4) is associated mainly with urea vaporization and decomposition. At early stages of decomposition, ammonia and cyanic acid begin to evolve from the melt. These gases then react with intact urea to produce biuret, which tends to be the main product of urea decomposition. The second major loss begins in earnest between 230 and 360 °C (Figure 4) is associated with biuret decomposition. At this stage cyanuric acid and ammeline are formed. The final major loss tends to be more gradual and occurs in earnest between 360 and 450 °C (Figure 4). At this stage, residual cyanuric acid and ammeline continue to decompose to produce ammeline. According to Schaber *et al.* the complete elimination of ammeline and ammeline can only be achieved at temperatures in excess of 700 °C.

As shown in Figure 4, the TGA data plot of prilled urea has an extra stage of mass loss at 100 °C compared to the other two samples. Three major stages of mass loss are observed for urea samples 1 and 2 and four stages are observed for urea prills. Table 2 provides the percentage mass loss for all of the respective stages. As can be seen from the table, the TGA traces of mass loss for all three urea samples are very similar with an exception at 100 °C that is due to the evaporation of water (3.5 % by weight) from urea prills. The granules of prilled urea are smaller in size leaving more surface area to uptake water and probably providing a more porous structure to transfer water. In fact, there might also have been traces of water in samples 1 and 2, but not enough to be detected by the TGA instrument.

Figure 5 shows the DSC thermal curves of all three urea samples. In all DCS curves two distinct thermal events are visible, the melting of urea at approximately 133 °C and decomposition of urea between 160 to 250 °C. As shown in Figure 5, the DSC traces of all three urea samples are very similar indicating that they all have similar thermal characteristics regardless of their structure.

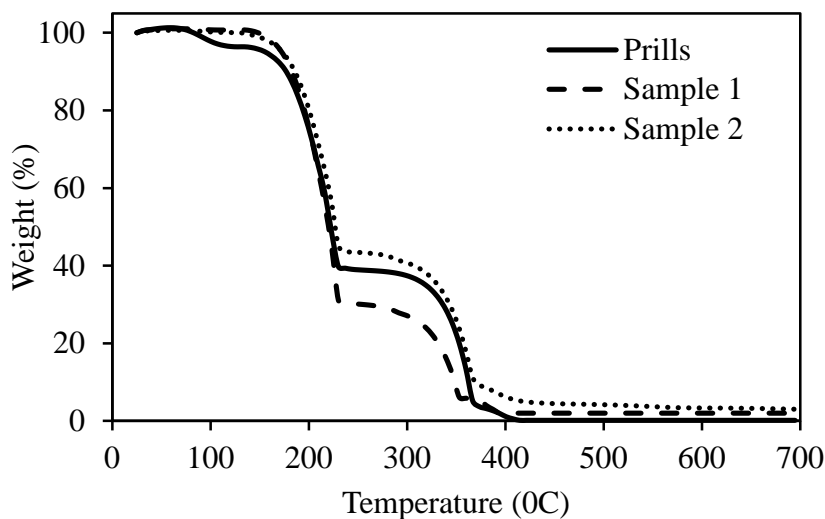


Figure 4. TGA curves of urea samples.

Table 2. Percentage mass loss of urea samples at each stage of the TGA.

TGA stages of mass loss	Percentage weight loss of urea		
	Prills	Sample 1	Sample 2
1 <sup>st</sup>	3.5	58.6	57.7
2 <sup>nd</sup>	56.4	34.2	35.5
3 <sup>rd</sup>	32.8	7.3	6.7
4 <sup>th</sup>	7.3	0	0

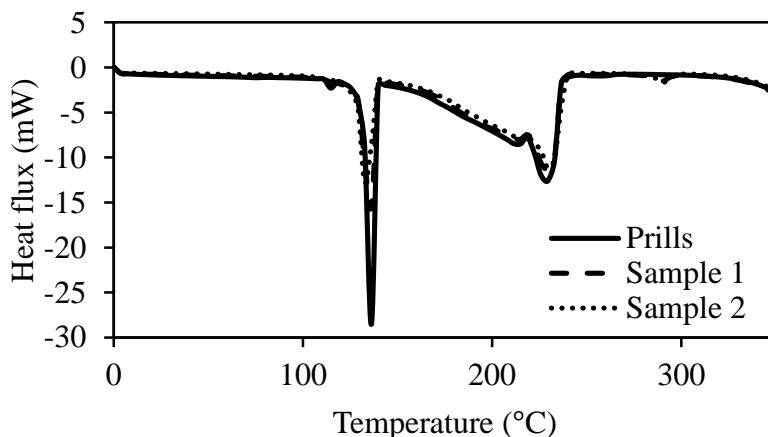


Figure 5. DSC curves of urea samples.

#### 4. Conclusions

Urea is the dominant form of fertilizer which has been used at an increasing rate in the world of agriculture. The physical structure of urea fertilizer is the key factor to its mechanical and physical properties such as flowability and avoidance of caking, while thermal and chemical properties are unlikely to be affected. The TGA and DSC results confirm that both prilled and granulated urea samples are of a very similar composition and thermal characteristics (melting point). The only key difference between them is the internal structure of granules which is the fundamental key to their mechanical properties. The strength measurement showed that samples of granulated urea have a greater strength as their prominent size and internal structure, while prills performed low strength having the small size. Taken together, these findings suggest a role for urea finishing processes in affecting mechanical and physical properties of the final product. One of the more significant findings to emerge from the TGA data plot is that prills showed a mass loss of about 3.5 % due to the evaporation of water. Whereas, the latter loss was absent in the TGA data plot of both granulated samples. This might be explained by the fact that urea prills have larger pores and a higher surface area which allow for uptake of a larger total amount of water. However, with a small sample size, caution must be applied, as the findings might not be transferable to the actual loss of water from the urea prills. Much work still needs to be accomplished with a more detailed analysis, such as Karl Fischer titration to aid a more precise determination of the moisture and water content of both prilled and granulated urea.



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