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COMPACTION OF UREA 46% NITROGEN BY UNIAXIAL DIE COMPACTION

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ABSTRACT

The compaction process of urea 46% nitrogen fertilizer in powder and granular form using uniaxial die compaction technique could be characterized by evaluating their compressibility, compactibility and tabletability characteristics. The results were analyzed in terms of the porosity, compaction pressure, tensile strength and disintegration. Results demonstrated that the urea powder tablet significantly became more compressible at 150.7 MPa and 188.3 MPa than the granule tablet. The compactability result shows that the powder tablet has strong bonding and thus, it has higher tensile strength in comparison to the urea granule tablet. For the quality assessment of the tablets, it was observed that powder tablet has significantly low disintegration rate than granule tablet only at compaction pressure of 150.7 MPa. Indeed, it can be conclude that compaction of urea fertilizer in granule and powder form needs further improvement with special focus on the disintegration and dissolution characteristics.

Keywords: *compressibility, compactibility, tabletability, pressure, tensile strength*

INTRODUCTION

Compaction of urea 46% nitrogen fertilizer

In agricultural mainly fertilizer industry, application of slow release fertilizer has become demanding due to its beneficial like slow releasing the urea, minimize the ammonia leaching and the chemical stability. Recently, research on slow release fertilizers has been performed by coating the fertilizer with waxes, polymers, and sulphur [1]. However, it consumes high production cost of coating materials, processes and less efficient. The concept of improving the physical appearance of the urea fertilizer by size enlargement process; compaction is believed could retain its mechanical integrity for a desired time and thus, slow releasing the urea. The compaction process is one of the size enlargement processes in which the smaller particles are assembled together to form a larger single coherent mass without eliminating the original particles [2]. It has become important in various fields; powder metallurgy, ceramic industry, explosive filling, catalyst manufacture and pharmaceutical tableting [3] and practiced for the past few years in pharmaceutical, agricultural, food industries and other industries including minerals, metallurgical and ceramics [2]. Therefore, this research was mainly focus on the formation of urea tablets, manufactured from the easily available granule form and from the ground urea powder. The characteristics of the compaction process in which the formed urea tablets are assessed are; compressibility, compactibility and tabletability [4].

1. Compressibility: relationship between porosity and compaction pressure
2. Compactibility: relationship between tensile strength and the porosity.
3. Tabletability: relationship between tensile strength and compaction pressure.

MATERIALS AND METHOD

Powder preparation

In this study, urea fertilizer; white crystalline solid containing 46% nitrogen has been used due to its highest source of nitrogen content, simplest form of fertilizer, abundant and inexpensive. Urea 46% nitrogen in the form of granules were manufactured and purchased from Petronas Fertilizer (Kedah) Sdn. Bhd., Malaysia. The ground urea powder, obtained from grinding the urea granules, has poor flowability according to its Carr Index and Hausner's Ratio assessment (Table 1).

Fig. 1: Urea fertilizer in granule form

Fig. 2: Urea fertilizer in powder form

Table 1: Physical properties of materials used in this work

Material properties	Urea 46 % nitrogen
Moisture content (%)	3.94
True density (kgm^{-3})	1341
Bulk density (kgm^{-3})	470
Tapped density (kgm^{-3})	783
Carr index, CI (%)	40
Hausner's ratio, HR	1.67
Flowability	poor flow
Median particle size, d_{50}(μm)	248
Particle size, d_{90}(μm)	850
Particle size, d_{10} (μm)	12

Compaction process

Compaction process by uniaxial die compaction method was performed using the Instron Universal Testing 5566 machine (Canton MA, USA) in room temperature 20 -25°C. Figure 3 shows the compaction cycle during the compaction process. There are three main stages involved; filling the die with an accurate weight of powder (die filling stage), tablet formation by compressing and compacting the powder (loading stage), removal of the upper punch from the die (unloading stage) and finally ejection of the formed tablet from the die by the force applied from the upper punch (ejection stage). Compaction pressures ranges from 37.7 MPa to 188.3 MPa were used to compact 1.0 g ± 0.02g of urea powder and granule inside 13mm stainless steel cylindrical uniaxial die (Specac, UK) as shown in Figure 4. The loading and unloading processes were conducted at constant speeds of 0.1 mm/s and 0.0167 mm/s [5]. The applied force and displacement were recorded automatically by computer software called Bluehill software (Canton MA, USA). The tablet was ejected out from die with a constant speed of 0.083 mm/s. After ejection, the urea compact was stored at least 24 hours prior to mechanical test for allowing full elastic recovery of the tablet [6]. The height of the tablet was measured using a digital Vernier Caliper (Mitutoyo, Japan).

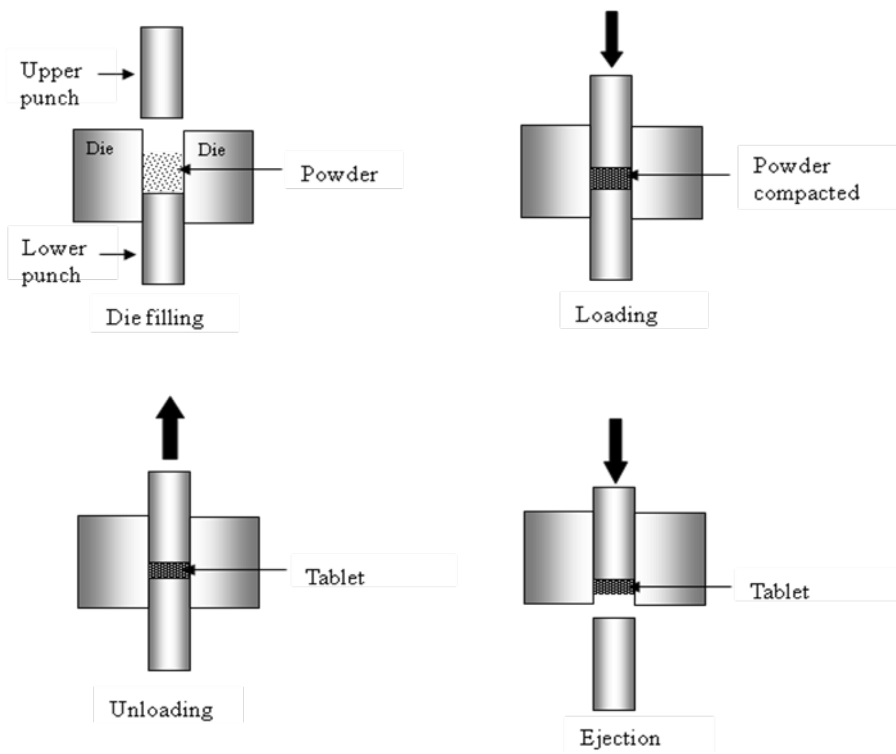

Fig. 3: Compaction cycle [7].

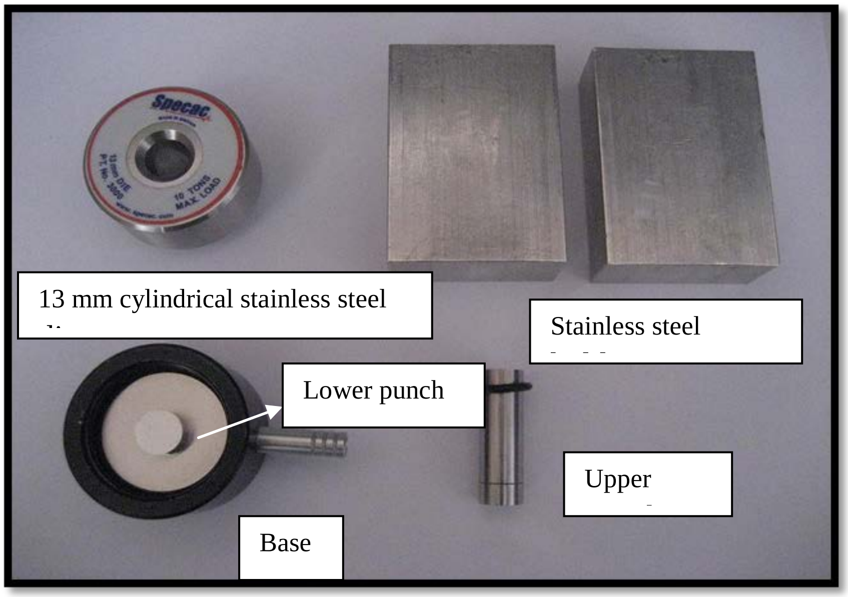

Fig. 4: 13 mm stainless steel cylindrical uniaxial die

Tablet strength test: Diametrical compression test (Brazilian test)

Tablet strength was analyzed by investigate their tensile strength during the diametrical compression test or "Brazilian disc test" using the Instron Universal Testing 5566 machine (Canton MA, USA). This test was conducted by placing the tablet between two flat plates and fractured diametrically with a constant speed of 0.0116 mm/s [5]. Then, the tensile strength is calculated from the diametrical breaking force where it required tensile force of the tablet [8]. For a cylindrical flat faced tablet, the tensile strength (σ) can be calculated based on the following equation [8]

$$\sigma_t = \frac{2P}{\pi DT} \tag{1}$$

where σ_t = the tensile strength

P = the tensile force

D = tablet diameter

T = tablet thickness

Modification of disintegration test

The disintegration test for the tablets was performed in 500 ml deionized distill water at ambient temperature; 27° C ± 0.5° C using a Pharma Test Semi-Automated Dissolution Test Instrument Type: PT-DT8 (Pharma Test Appparatebau GmbH, Germany). The modification of disintegration test was made according to the solubility of standard urea granule. The time for the tablets to disintegrate was recorded using stop watch. The disintegration test was conducted in four replicates.

Statistical analysis

The experimental data are presented as mean and standard error of n= 4 replicates (mean ± SE). Statistically analysis was performed using Minitab 16 Statistical Software. The data were analyzed by a one-way analysis of variance (ANOVA) and followed by Tukey's test for multiple comparisons. The differences were defined as significantly different at $p < 0.05$.

RESULTS AND DISCUSSION

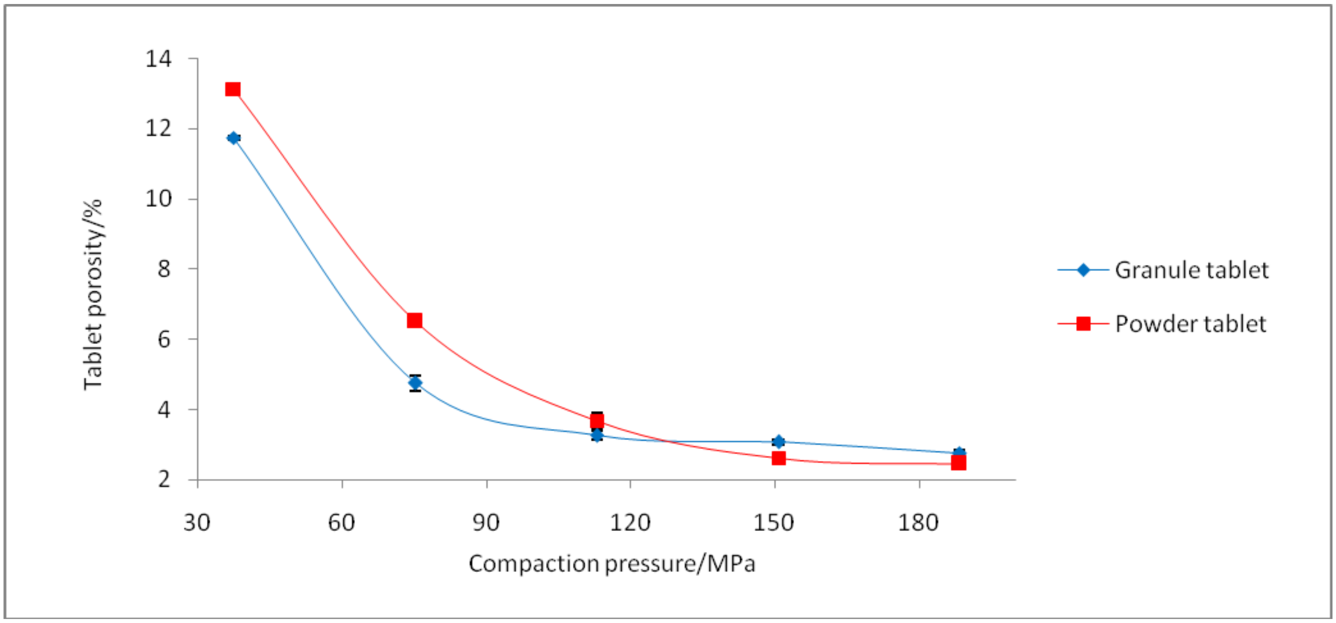

Fig. 5: Compressibility of urea powder and granule at various compaction pressures. (Standard errors are indicated by error bars)

Compressibility is the ability of the powder to reduce its porosity as a result of the applied compaction pressure [4]. It plays an important role in determining the tablet strength at low compaction pressures. The more compressible the powder, the stronger the resultant tablets [10]. It can be represented by plotting the graph of compaction pressure and porosity as shown in Figure 5. Generally, the porosity in both urea powder and granule tablets decreases with increasing compaction pressure (Figure 5). At the compaction pressure of 113 MPa, the tablet porosity difference between granule and powder tablets is insignificantly different ($p>0.05$). However, the powder tablets at 150.7 MPa and 188.3 MPa show significantly more compressible than granule tablet ($p<0.05$). This significant effect is probably due to the formation of larger bonding area within the powder tablet which causes them to pack closely together and reduces the porosity.

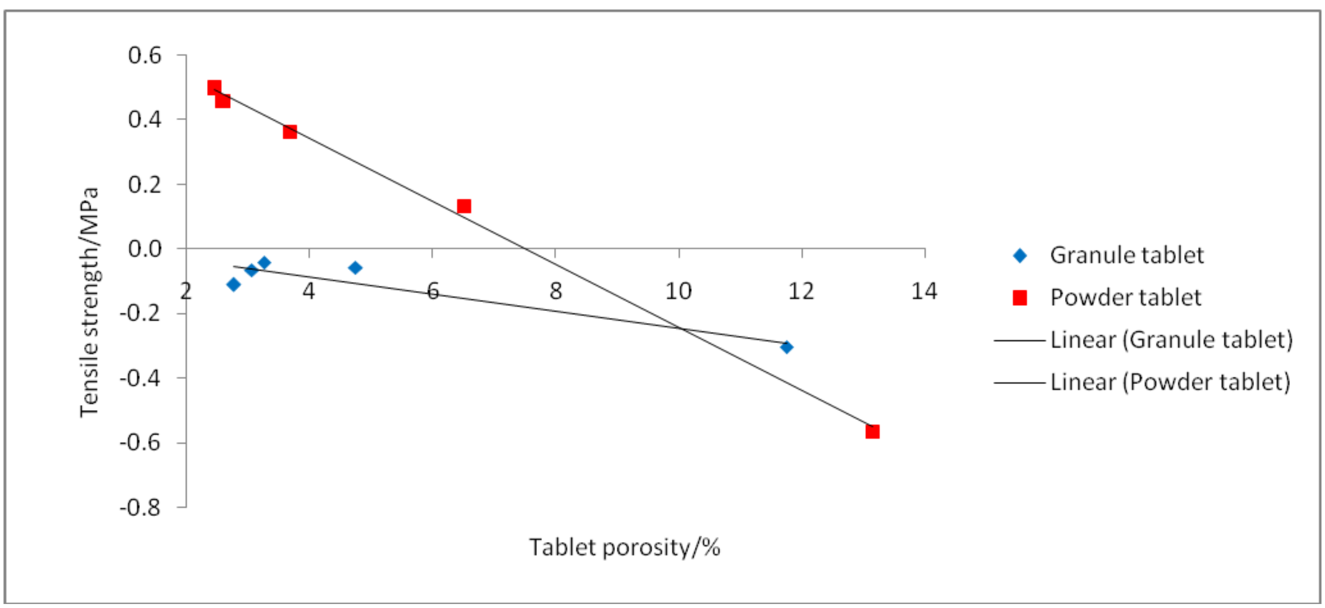

Fig. 6: Compactibility of urea in powder and granule form at various compaction pressures. (Standard errors are indicated by error bars).

Compactibility is the ability of the powdered to produce compacts under the effect of reduction porosity. This can be represented by plotting the logarithm of tablet tensile strength against the tablet porosity (Figure 6). This relationship can be expressed by the following equation [9]:

$Ln = Ln\sigma_0 - b\varepsilon$

Ln = the tensile strength

$Ln\sigma_0$ = the tensile strength at zero porosity. Tensile strength at zero porosity, $Ln\sigma_0$, was obtained by fitting the equation to the data followed by extrapolation

b = constant related to the pore distribution in the tablets

ε = the tablet porosity

Table 2: Compactibility data for urea granule tablet and powder tablet

	Tensile strength at zero porosity, $Ln\sigma_0$	Pore distribution in tablet, *b*	Coefficient of determination, R^2
Granule tablet	0.0199	0.0264	0.8626
Powder tablet	0.7322	0.0977	0.9976

The higher value obtained from pore distribution in tablet, *b* indicates highest yield stresses or anisotropic deformation [10]. The lowest value of tensile strength at zero porosity, σ_0 obtained from granule tablet indicates that the tablet forming weakest bonding during the compaction process and hence, it has the highest adsorbing capacity [11].

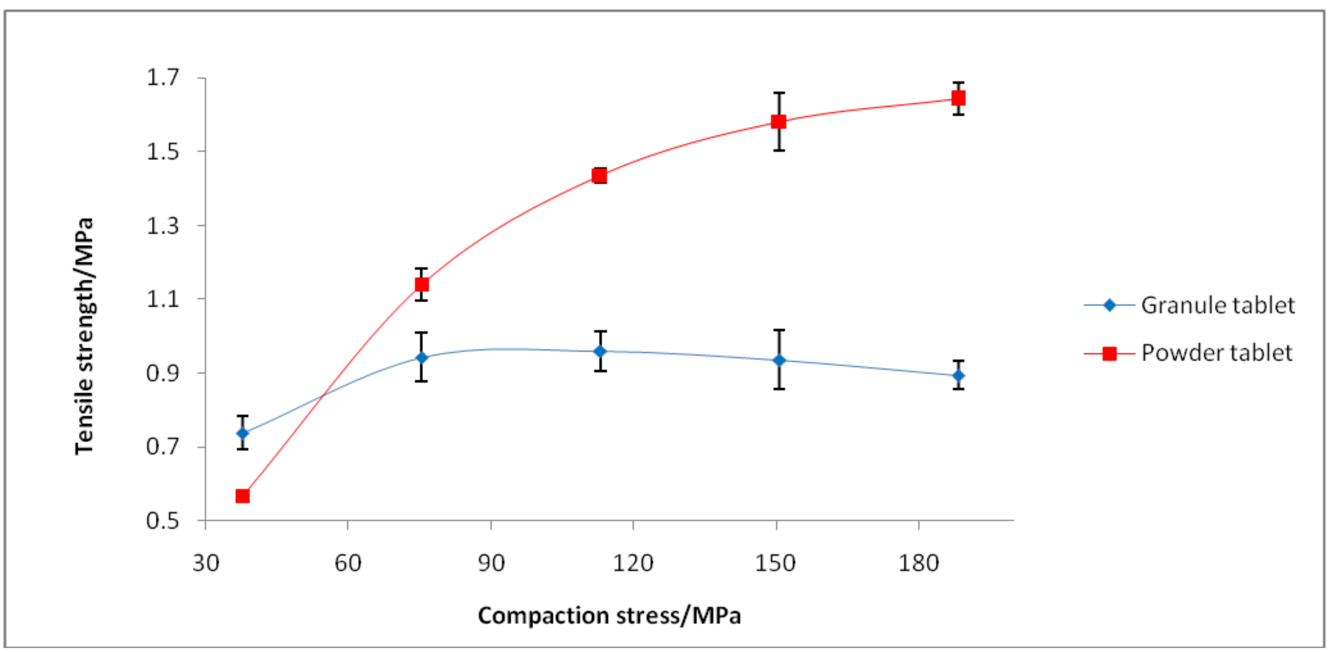

Fig. 7: Tabletability of urea in powder and granule form at various compaction pressures. (Standard errors are indicated by error bars)

Tabletability is the ability of the powder to produce tablets under the effect of compaction pressure. It can be described by analyzing the relationship between tensile strength and compaction pressure in Figure 7. From Figure 7, the tabletability for both granule and powder tablets shows a logarithmic trend as the compaction pressure increase. The tensile strength for the granule tablet increases gradually at compaction pressure of 37.7 MPa and 75.3 MPa and start reaching a plateau after 75.3 MPa which is 113 MPa until 188.3 MPa (Figure 7). Meanwhile, the tensile strength of the powder tablet show high steepness at compaction pressure 37.7 MPa and 75.3 MPa. The trend increase gradually and reached the highest tensile strength of 1.64 MPa respectively, at compaction pressure of 188.3 MPa. The higher tabletability in a powder tablet is due to the bonding strength per unit bonding area; the dominant factor which controls the tablet strength [11]. Overall, the powder tablet has higher tensile strength in comparison to the granule tablet indicating it higher mechanical strength.

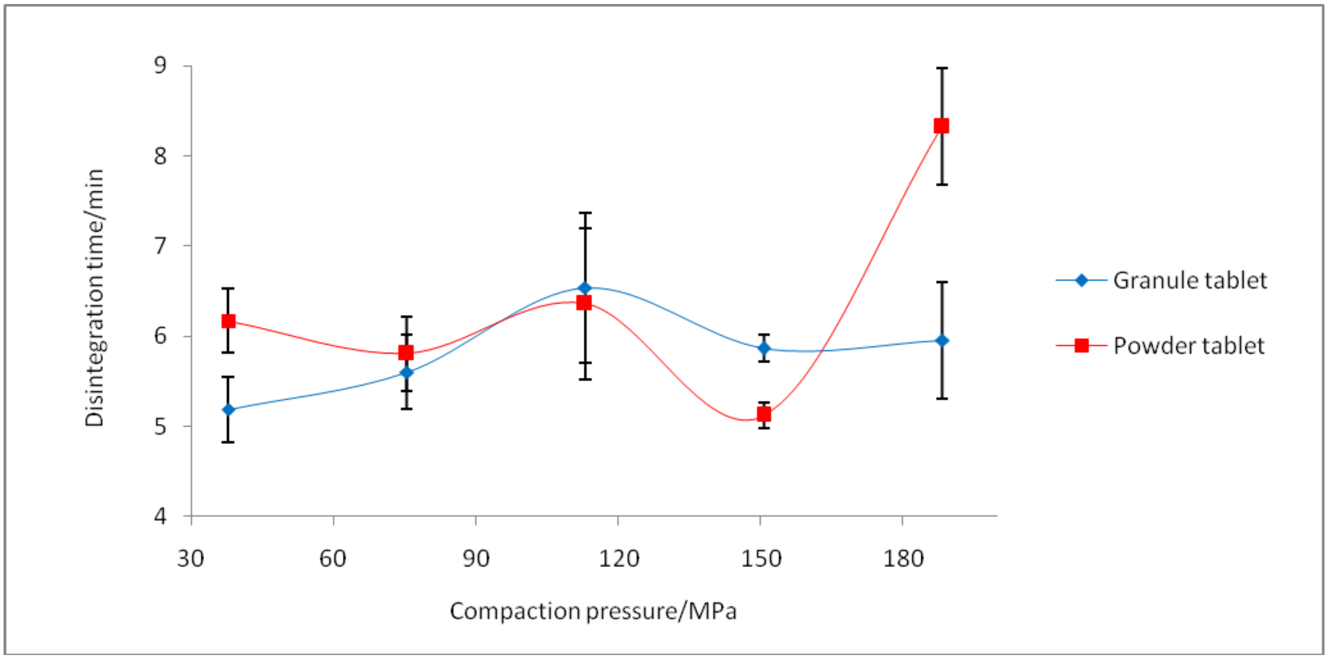

Fig. 8: Disintegration rate of urea in powder and granule form at various compaction pressures. (Standard errors are indicated by error bars)

The previous results have demonstrated the compaction characteristics of the granule and powder tablets during compaction process at various compaction pressures. Figure 8 represents the quality of the tablets in term of their disintegration rate. It can be seen there is fluctuating results obtained in Figure 8. Statistically results show that disintegration rate for granule and powder tablets have appeared significantly different only at compaction pressure of 150.7 MPa (p<0.05), respectively. The fluctuating results might be due to the non-homogeneous distribution of applied compaction pressure during the compaction process. Therefore, it reduces the ability for the inter-particulate interactions within the compacts to form a strong bonding due to inhomogeneous internal density distributions [3].

CONCLUSION

The compaction of urea granule and powder tablets was performed by uniaxial die compaction method. The results were analyzed in term of compressibility, compactibility, tabletability and disintegration characteristics. It can be conclude that the powder tablet has high reduction in porosity at 150.7 MPa and 188.3 MPa which leads to high compressibility compared to the granule tablet. The compactibility indicates that powder tablet forming strong bonding which can be observed by its high possesses tensile strength. For future study, further research on dissolution and disintegration characteristics of urea fertilizer in granule and powder form could be carry out in order to be utilizing as a controlled release fertilizer.

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