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Improvement in the Mechanical Strength of Compacted Urea Fertilizer Tablets through Die Wall Lubrication

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ABSTRACT

This research was conducted to investigate the compaction performance and mechanical strength of compacted urea fertilizer in unlubricated and lubricated die systems. The ground urea 46% N fertilizer was compacted in a 13 mm flat-face cylindrical die set in both unlubricated and lubricated die systems with vegetable fatty acids and magnesium stearate as lubricants at various compaction stresses to produce urea fertilizer tablets. In conclusion, a lubricated die system reduces the frictional effects during the production of urea fertilizer tablets and also produces a mechanically stronger urea fertilizer tablet than those produced in an unlubricated die system. In addition, the vegetable fatty acids and magnesium stearate lubricants are found to improve the compaction performance of urea fertilizer tablet as well as its mechanical strength.

Keywords: Ejection, fertilizer, lubricant, mechanical strength, uniaxial die compaction, urea

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INTRODUCTION

The development of slow release agricultural based-fertilizer is important in order to retain the concentration of the nutrient, mainly nitrogen within the plant roots for a desired length of time (Tzika et al., 2003). Slow release fertilizer can be categorized into organic fertilizer and water-soluble inorganic fertilizer. While these types of fertilizers have been widely used as slow

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release fertilizers, they possess different characteristics. The living micro presence in the organic fertilizer makes it unstable and the solubility has been limited by the low absorption of water into the fertilizer (Tzika et al., 2003). Whereas, the chemical compounds present in the water-soluble inorganic fertilizer makes it stable and the solubility is controlled by coating the fertilizer with a hydrophobic polymer (Tzika et al., 2003).

The lubrication of compact water-soluble inorganic fertilizers is one of the ways to produce slow release urea fertilizers by improving its physical appearance and integrity to form a coherent tablet. Recently, the compaction of urea into tablet form has shown promising characteristics as a controlled release vehicle for ammonium ions but large frictional effects were observed during the compaction of the urea powders into tablet form (Shamsudin, Anuar, Yusof, Mohd Hanif, & Tahir, 2014).

Urea fertilizer or scientifically known as carbamide; is a white crystalline water-soluble compound containing 46% nitrogen (N) (Watson, Stevens, Garrett, & McMurray, 1990; Jones, Koenig, Ellsworth, Brown, & Jackson, 2007). Its importance as a fertilizer has grown progressively and represents 40% of world N consumption (Watson et al., 1990). It is produced from a variety of manufacturing processes such as prilling, pan and drum granulation and fluidized bed granulation, which eventually forming small urea pellets size range from 1 to 4 mm each (Jones et al., 2007). Urea fertilizer is highly soluble in water and tends to volatile into ammonia gas (Jones et al., 2007) if it is handled improperly during broadcasting on the soil. In this research, the urea was manufactured by Petronas Fertilizer (Kedah) Sdn. Bhd., Malaysia in granular form and having 46% nitrogen, 1% biuret and 0.5% moisture by its maximum weight.

An established conventional die wall lubricant; magnesium stearate is often employed as lubricant in pharmaceutical industry due to its lubricating properties; forming a low shear strength between the die wall and the compact. Thereby, it facilitates the uniform transmission of stresses within the compact and thus, reduces the frictional effects that occurred between the die wall and the compact during the ejection process (Briscoe & Rough, 1998). Thus, the lubrication is important to prevent the wall friction from hindering the force transmissions and producing unwanted density gradients within the formed powder compact (Briscoe & Evans, 1991). Apart from that, the lubricant is able to form a hydrophobic film around the powders and thus, negatively affect the tablet crushing strength, disintegration time, friability and dissolution (Sheikh-Salem & Fell, 1981; Bolhuis, Lerk, Zijlstra, & Boer, 1975; Levy & Gumtow, 1963). Another recent research (Ariyasu, Hattori, & Otsuka, 2016) suggested some mechanisms on how the magnesium stearate delayed the tablet dissolution. The established magnesium stearate lubricant used in this current work consists of 3.8 – 5.0 % magnesium. Other new viable lubricants to be used during the compaction process are continuously researched and developed such as the work evaluating the use of sucrose fatty acids esters (Nakamura et al., 2017). In this current work, another type of lubricant, a vegetable based fatty acids lubricant or its commercially name; TriStar 149 derived from 0.1% Lauric and Myristic, 49.1% Palmitic, 50.2 % Stearic and 0.5% Arachidic fatty acid is also evaluated and used as a comparison to the established magnesium stearate lubricant.

Therefore, it is the aim of this work to assess the lubricant performance in the compaction of urea powder into tablet form using magnesium stearate and vegetable fatty acids in reducing the frictional effects during the urea fertilizer tablet production and improving the urea fertilizer tablet mechanical integrity.

MATERIALS AND METHODS

Powder Preparation

Urea 46% N in granular form was manufactured and purchased from Petronas Fertilizer (Kedah) Sdn. Bhd., Malaysia. To obtain urea powder in laboratory, 100 g urea granules were ground for 1 minute using an electric grinder (model RT-02A grinder, Taiwan) in order to obtain urea powder with a relatively uniform mean particle size distribution as described in Table 1.

The lubricants; magnesium stearate was purchased from Acros Organic, Belgium while the vegetable fatty acids lubricant was provided by Hemo Asia Sdn Bhd, Malaysia. All the lubricants were used as received.

Physical Properties of Powders

The moisture content of the powders was obtained by using the oven method. 5 g of each powder was dried in an oven dryer (Jeio Tech, Korea) at 105 ºC for 1 day. The true density of the powder was determined by using an automatic helium AccuPyc II 1340 Gas Pycnometer (Micromeritics Instrument Corp., USA). The bulk density was measured by pouring 25 g ± 0.01 g powder into 100 ml graduated cylinder and slightly tapped twice to avoid the powder from sticking onto the wall and bottom of the cylinder. The bulk density can be calculated by dividing the mass of the powder by the volume occupied in the cylinder (Kumar & Kothari, 1999). Tapped density was obtained using GeoPyc 1360 Tap Density Analyzer (Micromeritics Instrument Corp., USA).

Formation of Urea Fertilizer Tablet

1.0 g ± 0.01 g of the urea powder was compacted inside a 13mm evacuable pellet die (Specac, UK) set according to laboratory uniaxial die compaction method (Figure 1). The urea fertilizer tablet was formed in a room temperature between 20-25ºC using a universal testing machine (model 5566, Instron, Canton MA, USA). The process involved three main consecutive stages; filling of die with the accurate powder (die filling stage), the application of stress onto the powder bed and forming a tablet (loading stage), the removal of stress (unloading stage) and finally the produced tablet was ejected from the die (ejection stage).

In this study, various compaction stresses ranging from 37.7 MPa to 188.3 MPa were used for comparison purposes. For the assessment of the lubrication performance, the die wall, upper and lower punch surfaces were lubricated with magnesium stearate or vegetable fatty acids. Ethanol was utilized prior to each compression to remove any sticking powder on the die wall. The loading and unloading processes were conducted at a constant rate of 0.1 mm/s and 0.0167 mm/s (Mohammed et al., 2005). The applied force and displacement were recorded automatically by a compatible computer software called Bluehill (Canton MA, USA). Then, the produced urea fertilizer tablet was ejected from the die at a constant ejection rate of 0.083 mm/s. The data of the force-displacement during ejection was again recorded automatically by Bluehill software (Canton Ma, USA). The work of deformation (plastic work during loading-unloading and ejection work during ejection) during the compaction process was calculated based upon the recorded force and displacement data.

The physical testing of the urea fertilizer tablet was performed 24 hours after ejection (Krycer et al., 1982; Mollan & Celik, 1996). The physical measurements and tests performed were: weight by electronic mass balance (OHAUS, Switzerland); tablet thickness and diameter by vernier caliper (Mitutoyo, Japan).

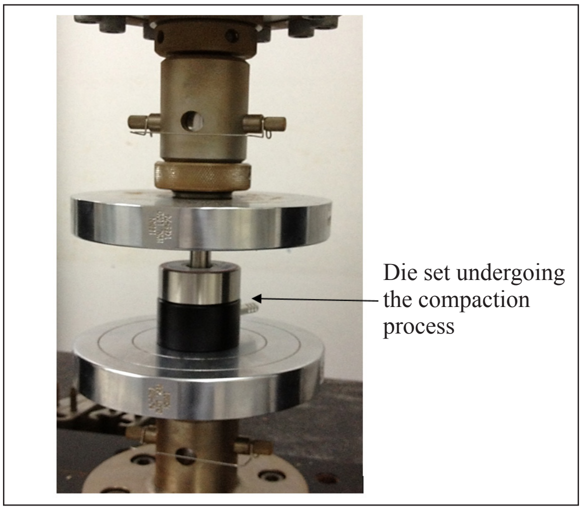

Figure 1. The laboratory uniaxial die compaction process using a universal testing machine

Strength of Urea Fertilizer Tablet

The strength of the urea fertilizer tablet was measured by applying a compressive stress, which is also known as the Indirect Tensile test or the Brazilian test. The tablet was placed between a pair of flat platens. A compressive stress was applied to the tablet at a constant rate of 0.0116 mm/s (Mohammed et al., 2005]. In this study, only tablets that failed through diametrical fracture or split into two halves with the fracture plane running through the center of the compact, indicating ideal tensile failure (Hiestand & Smith, 1984) were accepted. The tensile strength (σ) of the compact can be calculated using the following equation (Fell & Newton, 1970):

$$\sigma = \frac{2P}{DH} \tag{1}$$

where P, D and H are the maximum force recorded before fracture during the Brazillian test, diameter and thickness of the tablet. The tensile strength of the tablet was determined in four replicates using a universal testing machine (model 5566, Instron, Canton MA, USA).

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 $p< 0.05$.

Theoretical Background

Work of Deformation: Plastic Deformation. The compaction process in lubricated and unlubricated dies needs an energy to deform the powder. The work or energy used to compact the urea powder in lubricated and unlubricated die to produce the urea fertilizer tablets can be calculated from the upper punch forces and displacements (Ragnarsson & Sjogren, 1983). It is used to deform the particles both elastically or plastically, interparticle friction, friction with the die wall, to create new surfaces by fragmentation and forming the interparticles bonding (Alderborn, 2007). In this work, only the plastic work is calculated from the force-displacement data obtained during the loading-unloading stage of the compaction process.

Work of Die Wall Friction. The last stage in the compaction process; ejection stage is an important stage which determines the quality of the tablets once they come out from the die. The mechanism of the ejection process which involved friction with die wall could produce work of die wall friction (Sugimori & Mori, 1989). The work of die wall friction could be calculated by integrating the area under the curve of the ejection force and displacement data obtained during the ejection process.

RESULT AND DISCUSSION

Physical Properties of the Raw Materials

Table 1
Physical properties of materials used in this work

Material properties	Urea 46 % N	Magnesium stearate	Vegetable fatty acids
Moisture content (%)	3.94	1.65	0.09
True density (kgm^{-3})	1341	1088	985
Bulk density (kgm^{-3})	470	250	310
Tapped density (kgm^{-3})	783	483	474

Plastic Work

A common derived parameter to reflect particle deformation during the compaction process to form the urea fertilizer tablets is by quantifying its plastic work formation. From Figure 2, the plastic work increases as the compaction stress increases for both lubricated and unlubricated die systems. An increase in the plastic work indicates that the compaction stress has been transferred from the upper punch to the powder respectively. Apparently, urea fertilizer tablets in lubricated die produced high plastic work than those in unlubricated die at various compaction stresses, demonstrating that the lubricant facilitates the uniform transmission of compaction stress and hence, forming a strong coherent urea fertilizer tablet.

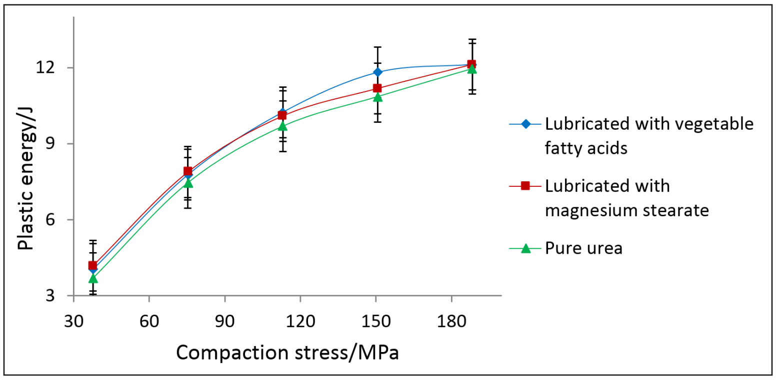

Figure 2. Plastic work of urea fertilizer tablets in lubricated and unlubricated dies (standard errors are indicated by error bars)

At low compaction stresses ranging from 37.7 MPa to 113.0 MPa, the urea fertilizer tablet in vegetable fatty acids and magnesium stearate lubricated dies showed statistically no significant different ($p>0.05$). However, both the lubricants differ significantly with unlubricated urea tablet ($p<0.05$). It seems that the lubricants do not show any significant difference at low compaction stresses. As the compaction stress increased, the plastic work for both lubricants increased gradually and the plastic works for the vegetable fatty acids lubricant urea fertililzer tablets were observed to become more constant at 150.7 MPa and 188.3 MPa, in comparison to the magnesium stearate lubricant which exhibited increasing plastic work in the same region. This indicates that vegetable fatty acids lubricant urea fertilizer tablets need approximately a low compaction stress; 150.7 MPa to achieve the optimum plastic work in comparison to the magnesium stearate lubricant urea fertilizer tablets and unlubricated urea fertilizer tablets. Overall, the magnitudes of the final plastic works obtained at the highest compaction stress used in this work (at 188.3 MPa) were similar for both the lubricants and higher in comparison to the unlubricated system.

Ejection Stress and Ejection Work

A higher compaction stress generates a higher residual die wall stresses and frictional stresses at the die walls prior to ejection (Sugimori & Mori, 1989; Briscoe & Rough, 1998). During the ejection, these stresses have to be overcome (Briscoe & Rough, 1998). Unlubricated urea fertilizer tablet exhibited the highest maximum ejection stress in comparison to the urea fertilizer tablet lubricated with vegetable fatty acids and magnesium stearate. The magnesium stearate lubricant acted as a lubricant to reduce the friction between die wall and urea particles as the tablet was formed and ejected. Also, the vegetable fatty acids lubricant had same performance as the magnesium stearate where no significant differences ($p>0.05$) were found for all the compaction stresses.

The tablet-die wall frictional effect was investigated by ejecting the urea fertililzer tablet in lubricated and unlubricated dies at various compaction stresses. The maximum ejection stress of urea fertilizer tablet in lubricated and unlubricated dies is plotted as a function of compaction stress as shown in Figure 3. As the compaction stress increased, the maximum ejection stress increased.

The ejection work against various compaction stresses was plotted in Figure 4. The ejection work is calculated from the force-displacement data obtained during the ejection of the urea fertilizer tablet from the die cavity. It gives a first-order estimation of the tablet

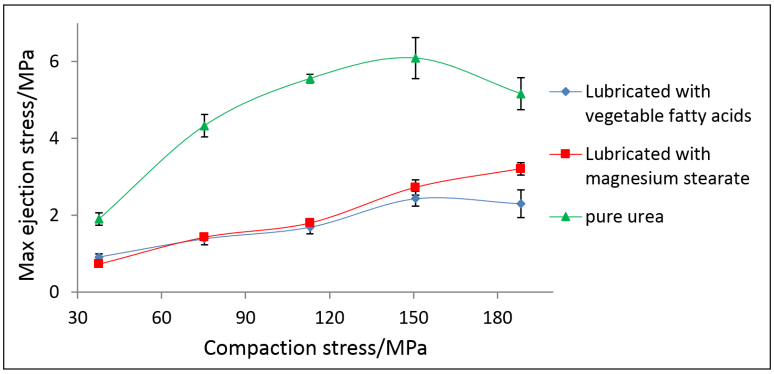

Figure 3. Maximum ejection stress of urea compact in lubricated and unlubricated dies (standard errors are indicated by error bars)

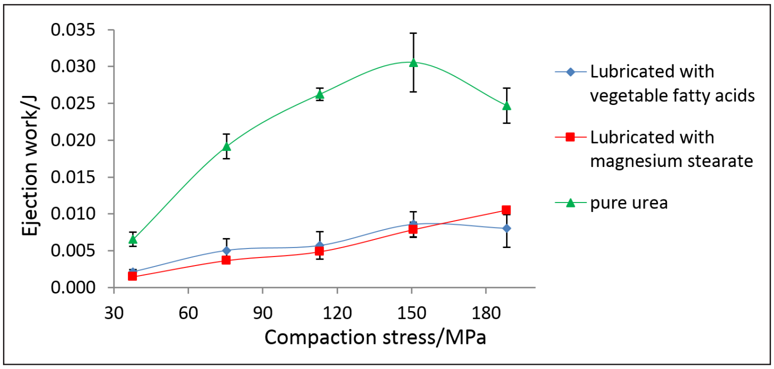

Figure 4. The ejection work of urea fertilizer tablet in lubricated and unlubricated dies (standard errors are indicated by error bars)

stored elastic energy (Anuar & Briscoe, 2009). In Figure 4, the ejection work was found to increase with increasing compaction stress in both in unlubricated and lubricated die systems. The unlubricated die systems possessed higher ejection works in comparison to the lubricated system, which could be explained by observing their maximum ejection stress in Figure 3. Hence, as a first-order approximation, the urea fertilizer tablets formed in a lubricated die system have lower stored elastic energies in comparison to the tablets formed in the unlubricated die. Both lubricated die with vegetable fatty acids and magnesium stearate showed no significant different (p>0.05) for ejection work at all compaction stresses.

Mechanical Strength

Figure 5 shows the tensile strengths of urea fertilizer tablets in lubricated and unlubricated dies increase in proportion to the compaction stresses. It also can be observed that the tensile strength of the urea fertilizer tablet formed in a lubricated die shows a significant difference (p<0.05) in comparison to those formed in an unlubricated die. Apart from that, the lubrication of the urea fertilizer tablet with magnesium stearate and vegetable fatty acids gives significant difference (p<0.05) at all compaction stresses.

This compaction behaviour can be assumed to be due to the increase in the inter-particulate bonding due to a higher contact area between the particles during the plastic deformation (Shamsudin et al., 2010) thus, enhancing the mechanical strength of the tablet. This is supported by data shown earlier in Figure 2, where the increase in the plastic work is observed with the increase in the compaction stress. Therefore, a high tensile strength is required for crack propagation in lubricated urea fertilizer tablet due to the increase in the inter-particulate bonding within the tablet (Figure 5). Based upon the increase in the plastic deformation (Figure 2) and the resulting mechanical strength (Figure 5) obtained in

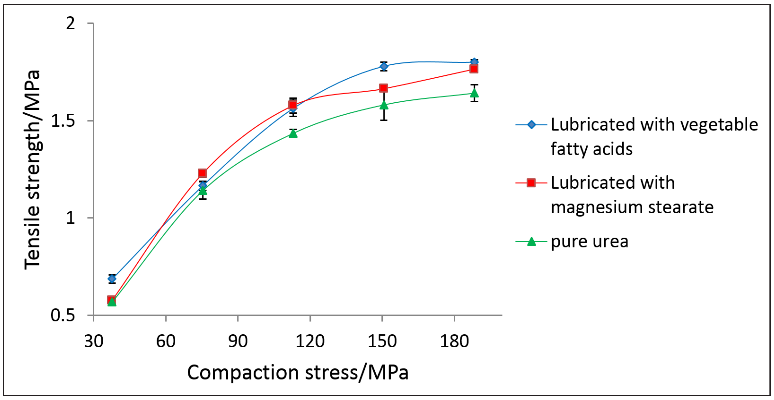

Figure 5. The tensile strength of urea compact in lubricated and unlubricated dies (standard errors are indicated by error bars)

a lubricated die system, these give further indication that the formed urea fertilizer tablets in the lubricated die system have relatively lower stored elastic energies in line with the low values of the ejection works (Figure 4).

CONCLUSION

The effect of die wall lubrication in the compaction of urea 46% N powder has been investigated experimentally using the uniaxial die compaction method. As observed, the urea powder can be characterized as having poor flow powder based upon its Carr Index and Hausner Ratio values. During the compaction, the lubricated urea tablets with vegetable fatty acids and magnesium stearate experienced high plastic deformation. The lubricants improve the ejection process by reducing the frictional effects, where the unlubricated system requires high ejection stress and ejection work compared to the lubricated systems. For the diametrical strength assessment, the lubricated die system produces urea fertilizer tablets that are relatively stronger than those produced in the unlubricated die system. Vegetable fatty acids lubricated die system produces urea fertilizer tablets exhibiting the highest mechanical strength for the range of compaction stresses utilized in this work. Unlubricated die system produces urea fertilizer tablets having low tensile strengths. It can be concluded that a lubricated die system produces mechanically stronger urea fertilizer tablets and the performances of both the vegetable fatty acids and magnesium stearate lubricants are dependent upon the compaction stress.

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REFERENCES

Alderborn, G. (2013). Tablet and compaction. In M. Aulton & K. Taylor (Eds.), *Aulton's Pharmaceutics, The design and Manufacture of Medicines, Fourth edition* (pp. 505-549). Edinburgh: Elsevier.

Anuar, M. S., & Briscoe, B. J. (2009). The elastic relaxation of starch tablets during ejection. *Powder Technology, 195*(2), 96-104.

Ariyasu, A., Hattori, Y., & Otsuka, M. (2016). Delay effect of magnesium stearate on tablet dissolution in acidic medium. *International Journal of Pharmaceutics, 511*(2), 757-764.

Bolhuis, G. K., Lerk, C. F., Zijlstra, H. T., & de Boer, A. H. (1975). Film formation by magnesium stearate during mixing and its effect on tableting. *Pharmaceutisch Weekblad, 110*, 317-325.

Briscoe, B. J., & Evans, P. D. (1991). Wall friction in the compaction of agglomerated ceramic powders. *Powder Technology, 65*(1-3), 7-20.

Briscoe, B. J., & Rough, S. L. (1998). The effects of wall friction on the ejection of pressed ceramic parts. *Powder Technology, 99*(3), 228-233.

Fell, J. T., & Newton, J. M. (1970). Determination of the tensile strength of tablets by the diametrical compression test. *Journal of Pharmaceutical Science, 59*(5), 688-691.

Hiestand, H. E. N., & Smith, D. P. (1984). Indices of Tableting Performance. *Powder Technology, 38*(2), 145-159.

Jones, C., Koenig, R., Ellsworth, J., Brown, B., & Jackson, G. (2007). *Management of Urea Fertilizer to Minimize Volatilization*. Montana: Montana State University Extension Service.

Krycer, I., Pope, D. G., & Hersey, J. A. (1982). An evaluation of the techniques employed investigate powder compaction behaviour. *International Journal of Pharmaceutics, 12*(2-3), 113-134.

Kumar, V., & Kothari, S. H. (1999). Effect of compressional force on the crystallinity of directly compressible cellulose excipients. *International Journal of Pharmaceutics, 177*(2), 173-182.

Levy, G., & Gumtow, R. H. (1963). Effect of certain tablet formulation factors on dissolution rate of the active ingredient III tablet lubricants. *Journal of Pharmaceutical Science, 52*(11), 1139-1144.

Mohammed, H., Briscoe, B. J., & Pitt, K. G. (2005). The interrelationship between compaction behaviour and the mechanical strength of pure pharmaceutical tablets. *Chemical Engineering Science, 60*(14), 3941-3947.

Mollan, M. J., & Celik, M. (1996). The effects of lubrication on the compaction and post-compaction properties of directly compressible maltodextrins. *International Journal of Pharmaceutics, 144*(1), 1-9.

Nakamura, S., Ishii, N., Nakashima, N., Sakamoto, T., & Yuasa, H. (2017). Evaluation of sucrose fatty acid esters as lubricants in tablet manufacturing. *Chemical and Pharmaceutical Bulletin, 65*(5), 432-441.

Ragnarsson, G., & Sjogren, J. (1983). Work of friction and network during compaction. *Journal of Pharmacy and Pharmacology, 35*(4), 201-204.

Shamsudin, I. S., Anuar, M. S., & Tahir, S. M. (2010). Compaction of Sweet Potato (*Ipomoea Batatas* L.) and *Stevia Rebaudiana* Food Powders. *Particulate Science and Technology, 30*(2), 136-144.

Shamsudin, I. S., Anuar, M. S., Yusof, Y. A., Mohd Hanif, A. H., & Tahir, S. M. (2014). Effect of particle size on direct compaction of urea fertilizer. *Particulate Science and Technology, 32*(6), 544-553.

Sheikh-Salem, M., & Fell, J. T. (1981). The influence of magnesium stearate on time dependent strength changes in tablets. *Drug Development and Industrial Pharmacy, 7*(6), 669-674.

Sugimori, K., & Mori, S. (1989). Characterization of die wall pressure to predict capping of flat- or convex-faced drug tablets of various sizes. *Powder Technology*, 58(4), 259-264.

Tzika, M., Alexandridou, S., & Kiparissides, C. (2003). Evaluation of the morphological release characteristics of coated fertilizer granules produced in a Wurster fluidized bed. *Powder Technology, 132*(1), 16-24

Watson, C. J., Stevens, R. J., Garrett, M. K., & McMurray, C. H. (1990). Efficiency and future potential of urea for temperate grassland. *Fertilizer Research, 26*(1-3), 341-357.