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Effect of Ammonium Sulfate Concentrations on Size Distribution of NPK Fertilizer Granules in a Rotating Drum Granulator

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ABSTRACT: Granulation enhances fertilizer efficiency by improving mechanical strength, nutrient uniformity, and slow-release properties. Understanding factors influencing granule size distribution is crucial for optimizing production and performance. This study investigates the effects of binder type and volume on NPK fertilizer granulation, particularly the role of ammonium sulfate (ZA). The granulation process was performed in a rotating drum granulator with varied NPK ratios (28-6-6, 20-20-8, 18-16-20, and 15-15-15) with amounts of binder (10 ml or 20 ml of 15% ZA solution or pure water). Granule sizes were analyzed using Image Pro Plus software, and Stokes' number was calculated to establish a correlation between the average granule radius and Stokes' number. Results show that ZA improved granule cohesion, yielding larger particles than water. Increasing ZA volume increased granule size but broadened distribution, whereas increasing water reduced granule size while narrowing distribution. The study demonstrated a robust correlation ($R^2 = 0.95$) between Stokes' number and the average granule radius, indicating that Stokes' number served as a generalized parameter of the granulation process for various NPK formulations and binder types.
Keywords: granulation, fertilizer, ammonium sulfate, Stokes' number

1. Introduction

Granulation involves increasing particle size to create granules with desired properties from a liquid or particulate feed. This method is widely used in the pharmaceutical, detergent, food, and fertilizer industries. In the fertilizer industry, the granulation process for compound fertilizers is developed to minimize transportation, storage, and handling costs (Yu et al., 2021). Compared to powdered forms, granular fertilizer offers improved mechanical resistance, uniform composition, and slow-release properties when used in agriculture. Higher concentrations of nutrients tend to cause more caking issues with powdered forms. However, this issue can be reduced by creating a granulate with a high mean particle size and a narrow particle size distribution (Walker et al., 2001).

Wet granulation is one of the most effective methods widely used in producing dense and uniform granules. This process involves three main steps: wetting and nucleation, growth and consolidation, and attrition and breakdown (Iveson et al., 2001; Szulc et al., 2024). Parameters such as the liquid-to-solid (L/S) ratio and binder properties are critical in controlling granule size, size distribution, and quality. Studies have shown that higher L/S ratios increase granule size but can lead to broader granule size distributions due to excessive moisture content (Dhenge et al., 2010; Meng et al., 2019). High binder viscosity can result in a high degree of granulation (Adetayo et al., 1993, 1995; Walker et al., 2000). Additionally, factors such as binder dispersion and wetting efficiency play a crucial role in defining granule formation. Effective binder dispersion ensures uniform wetting and controlled nucleation, leading to a narrow size distribution and improved mechanical properties of granules (Iveson et al., 2001; Mort & Tardos, 1999).

Innovative binder materials have been explored to optimize the granulation process further. Ammonium sulfate (ZA) has proven effective in enhancing granule strength and uniformity through its ability to form crystalline bonds with fertilizer components (Xue et al., 2017). Similarly, tapioca starch has been shown to improve granule layering (Handayani et al., 2023), while biochar and nanocytosan have emerged as promising materials for improving granule stability, nutrient retention, and environmental sustainability (Amelia et al., 2020; Sastra et al., 2023; C. Wang et al., 2022). Despite these advancements, ammonium sulfate remains a widely used binder in fertilizer granulation due to its ability to improve mechanical strength while maintaining nutrient content (Steiger et al., 2024; G. Wang et al., 2013). However, the extent to which ZA concentration affects granule formation and its relationship with key granulation parameters remains unclear.

While previous studies have explored alternative binders like tapioca starch and biochar, limited research has focused on quantifying the effects of ZA concentration on

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granule size and its relationship with Stokes' number. Stokes' number is a fundamental parameter in granulation mechanics that describes the balance between particle inertia and viscous dissipation during collision events, directly influencing granule formation dynamics. Although Stokes' number has been widely applied in the pharmaceutical industries (Bouwman et al., 2006), its role in optimizing fertilizer granulation remains underexplored. Understanding its relationship with ZA-induced granule growth can provide valuable insights into process control and efficiency.

Thus, this study aims to investigate the impact of binder amount and NPK composition on granule size and distribution, with a particular focus on the role of ZA. This research specifically examines how different concentrations of ZA influence the granulation process and whether Stokes' number can serve as a predictive tool for granule size control. The study also investigated the relationship between Stokes' number and granule size, which can be used to optimize the granulation process and improve the efficiency and performance of NPK fertilizer in agricultural applications. By addressing this gap, the study contributes to a better understanding of ZA's role in granulation kinetics and fertilizer quality control, offering a novel perspective on granule size prediction based on process parameters. The findings of this study can assist in optimizing binder selection and granulation conditions, ultimately improving the formulation and performance of granular fertilizers for agricultural use.

2. Materials and Methods

The materials used in this work were diammonium phosphate (DAP) (98%, Sigma-Aldrich), potassium chloride (KCl) (99%, Sigma-Aldrich), Zwavelzure ammonium (ZA) (99%, Sigma-Aldrich), and urea supplied by PT Pupuk Kujang Cikampek (Indonesia).

The granulator used in this study had a drum length of 750 mm and an inner diameter of 240 mm. It operated at a rotation speed of 15 rpm. The drum was designed as a cylindrical structure with two supporting discs, each with a diameter of 250 mm, positioned on its sides to maintain movement stability. The granulator was driven by a belt system connected to an electric motor. The drum was positioned horizontally with a 0-degree inclination to ensure uniform granulation.

2.1. Experimental Procedure

NPK granular fertilizer was prepared by weighing 300 g of fertilizer material, consisting of urea, diammonium phosphate (DAP), and potassium chloride (KCl), with NPK mass ratio variations of 28-6-6, 20-20-8, 18-16-20, and 15-15-15 (used as a control). These ratios were selected to explore the effects of different nutrient compositions on granulation performance, considering variations in nutrient requirements across different agricultural applications. The materials were ground using a mortar and manually blended for 10 minutes before granulation. The homogeneity of the mixture was checked visually by ensuring a uniform color distribution. The mixtures were then moistened evenly with either 10 ml or 20 ml of the binder solution (15% ammonium sulfate (ZA) or water), corresponding to approximately 3% and 6% of the total granule mass, respectively. These binder concentrations align with recommended ranges for effective granulation, as they ensure adequate adhesion while maintaining granule integrity (Richard H. Van de Walle & David Smith, 1992). A 15% ZA solution was chosen for its ability to enhance adhesion during granulation while contributing to the nutrient composition of the final product. The two binder volumes were selected to represent low and high moisture conditions, ensuring sufficient adhesion for granule formation without leading to excessive agglomeration.

To ensure adequate wetting, the granulator motor was activated, and a small amount of binder solution was sprayed evenly on the inner wall of the granulator. The granulation process was conducted for 30 minutes, and the resulting granular fertilizer was dried in an oven until the water content reached 2%. Each sample was labeled according to its NPK ratio, binder type, and volume used. For instance, "28-6-6 W20" represents an NPK ratio of 28-6-6 with 20 ml of water as the binder, whereas "20-20-8 ZA10" represents an NPK ratio of 20-20-8 with 10 ml of 15% ZA solution.

2.2. Analysis Method

2.2.1. Granule Size Measurement

Granule size was determined through photo analysis of sample particles using Image Pro Plus software. Images of the granules were captured with a calibrated reference scale embedded in the image to ensure measurement accuracy. Each photograph contained 200-300 randomly selected granules per batch, which were analyzed to measure particle diameters.

The boundaries of the granules were manually outlined using the circle selection tool, with the measurement parameter set to radius. The raw values obtained were initially recorded in pixels and later converted into actual size (mm) using a conversion factor derived from the reference scale in the image. The conversion factor was determined by measuring a reference object of known size within the image, ensuring accuracy in the size estimation. The estimated measurement error margin was ±0.5 mm, accounting for potential variations due to image resolution and manual selection precision.

The average granule radius was calculated using Equation (1):

$$\bar{d}_p = \frac{\sum d_{p,i}}{n} \tag{1}$$

where:

$\bar{d}_p$ is the average granule diameter,

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$d_{p,i}$ is the diameter of an individual particle,

n is the total number of particles measured.

2.2.2. Statistical Analysis

The analysis of variance (ANOVA) was conducted using Minitab software with the General Linear Model (GLM) to evaluate the effects of different factors on granule size. The response variable analyzed was granule size, while the factors included NPK type, binder type, and binder volume. To assess both the main effects and interactions between factors, the model was set to include interactions up to the third-order. This allowed for a comprehensive evaluation of individual factor effects as well as two-way and three-way interactions, providing insights into the combined influence of these parameters on granule size.

2.2.3. Stokes' Number and Size Distribution Analysis

The fundamental mechanism of granule formation involves the forces of two-particle collisions. The viscous Stokes' number is the ratio of the relative kinetic energy between colliding particles to the viscous dissipation of the pendular bond. Stokes' number for drum granulation follows Equation (2) (Adetayo et al., 1993).

$$St_v = \frac{8\,\rho_g\,r\,\omega\,R}{9\,\mu} \tag{2}$$

where:

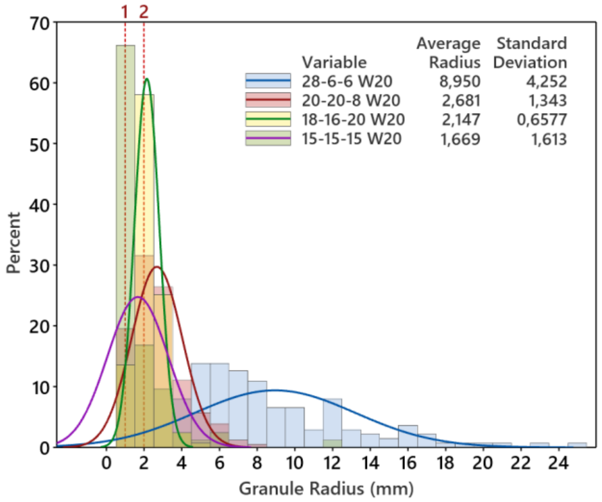
ρ_g is the granule density (kg/m^3),
r is the adequate granule size (m),
ω is the granulator speed (/s),
R is the granulator radius (m),
μ is the binder viscosity (kg/m/s).

The correlation between granule size and Stokes' number was plotted on a graph and can then be used to estimate the granule size obtained at a given operating condition.

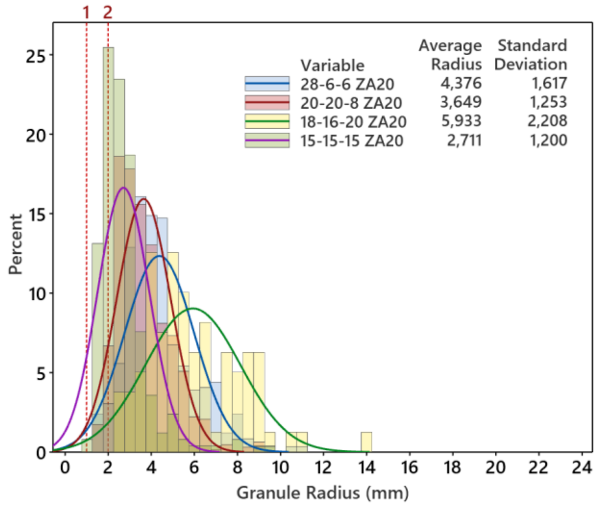
3. Results and Discussion

3.1. Effect of Binder Type and NPK Composition on Granule Size

Key factors, such as binder type, urea content, and the liquid-to-solid ratio, influenced granule size and distribution. Figure 1 shows that using pure water as a binder (Figure 1a) in NPK fertilizer granulation resulted in smaller granules on average than ZA as a binder (Figure 1b), except in an NPK ratio of 28-6-6. This variation was mainly due to the binding mechanisms involved during the granulation process. Specifically, while water provided temporary cohesion through capillary forces, the subsequent evaporation of water resulted in a structural weakness of the granules, making them susceptible to disintegration (Khalil Abu Rabeah et al., 2020). In contrast, the use of ZA as a binder was likely to increase particle cohesion and stability, resulting in larger granules and a more consistent size distribution compared to pure water as a binder. This result aligned with that ammonium sulfate increased the interaction between materials and their viscosity, increasing the granulation rate (Xue et al., 2017).



(a)

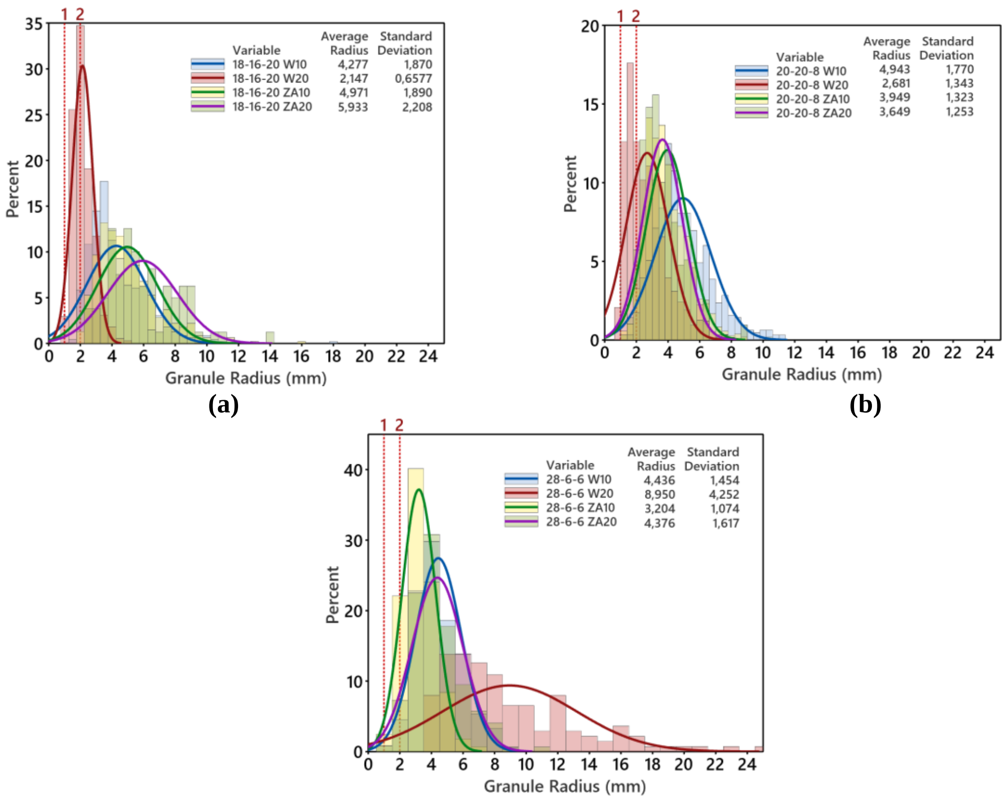


(b)

Figure 1. Granule size distribution of NPK formulations (18-16-20, 20-20-8, and 28-6-6) with (a) 20 ml water and (b) 20 ml of 15% ammonium sulfate (ZA). Granules with ZA tend to be larger and have a broader size distribution than those with water. The red markers at 1 mm and 2 mm represent the typical optimal granule radius range for efficient nutrient release.

As shown in Figure 2, increasing the binder volume from 10 ml to 20 ml had opposite effects for water-based and ZA-based granulation. For water as a binder (Figure 2a and Figure 2b), increasing the binder volume from 10 ml to 20 ml resulted in smaller granule sizes. However, the size distribution became narrower, indicating that although the granules were smaller, they were more uniform in size. This trend suggests that excessive water may have inhibited granule growth, likely due to over-wetting, which prevented stable agglomerates from forming and led to disintegration into smaller particles. In contrast, in the 28-6-6 composition (Figure 2c), higher amounts of urea resulted in larger granule sizes due to its ability to absorb more water. However, this also resulted in a broader size distribution due to uneven wetting and local agglomeration during the granulation process. This observation is consistent with prior research, which indicated that an increase in water content during the granulation process not only enhanced larger particles but also contributed to a wider size distribution (Dhenge et al., 2010; Walker et al., 2000). A broad granule size distribution can influence fertilizer efficiency, as finer granules tend to dissolve more rapidly, potentially altering nutrient release rates and soil absorption. For optimal performance, fertilizer granules are typically maintained within a size range of 2–4 mm to ensure uniform nutrient distribution and minimize losses (Kasmadi et al., 2019; G. Wang et al., 2013).

For ZA as a binder (Figure 2a and Figure 2c), increasing the binder volume from 10 ml to 20 ml led to an increase in granule size, but at the same time, it widened the size distribution. This suggests that higher ZA volume facilitated granule growth, possibly by increasing adhesion and promoting larger particle agglomeration. However, the broader distribution indicates that some granules may have continued to grow while others remained smaller, leading to variability in granule size. Interestingly, in Figure 2b (20-20-8 composition), the effect of increasing ZA volume was minimal, implying that the interaction between binder type, binder volume, and NPK composition significantly influences granulation behavior.



(c)

Figure 2. Granule size distribution of NPK formulations (a) 18-16-20, (b) 20-20-8, and (c) 28-6-6 with varying binder volumes: 10 ml water, 20 ml water, 10 ml 15% ammonium sulfate (ZA), and 20 ml 15% ZA. Increasing the binder volume influenced granule size differently depending on the binder type. The red markers at 1 mm and 2 mm indicate the typical optimal granule radius range for efficient nutrient release.

3.2. Statistical Analysis of Granule Size

The analysis of variance (ANOVA) results indicates that the individual effects of NPK Type ($P = 0.087$), Binder Type ($P = 0.600$), and Binder Volume ($P = 0.456$) on granule size are not statistically significant, suggesting that these factors alone do not strongly influence the granulation process. However, significant interactions were observed among these factors, highlighting the importance of their combined effects.

As summarized in Table 1, the interaction between NPK Type and Binder Type ($P < 0.001$) suggests that the impact of the binder type (ZA or water) on granule size is dependent on the specific NPK formulation used. Similarly, the interaction between NPK Type and Binder Volume ($P < 0.001$) indicates that the effect of binder volume on granule size varies according to the NPK composition. Additionally, the Binder Type and Binder Volume interaction ($P = 0.003$) reveals that the amount of binder applied influences granule formation differently depending on the binder type. Notably,

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the three-way interaction (NPK Type * Binder Type * Binder Volume, P < 0.001) is highly significant, implying that granule size is shaped by a complex relationship among these three factors rather than being driven by any single factor alone.

Table 1. ANOVA Results for the Effects of NPK Type, Binder Type, and Binder Volume on Granule Size

Source	DF	F-Value	P-Value
NPK Type * Binder Type	2	116.44	<0.001
NPK Type * Binder Volume	2	127.14	<0.001
Binder Type * Binder Volume	1	8.68	0.003
NPK Type * Binder Type * Binder Volume	2	61.00	<0.001

3.3. Correlation Between Stokes' Number and Granule Size

Stokes' number was calculated for each batch of granule samples using Equation (2). The relationship between Stokes' number and the average granule radius was analyzed, as shown in Figure 3. The results indicate a general correlation between these parameters across all experimental variants, which included different binder types, volumes, and NPK formulations.

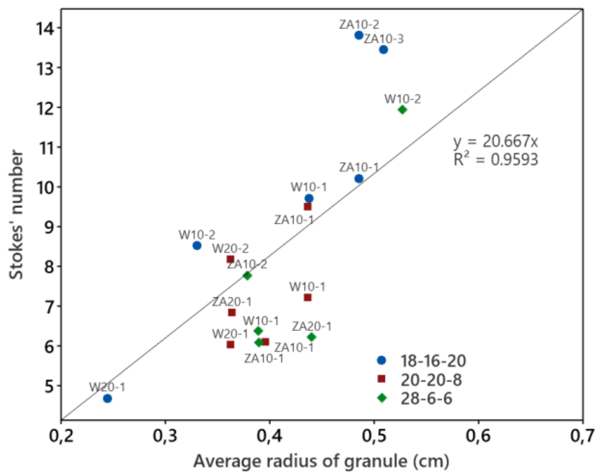

Figure 3. Correlation between the average granule radius and Stokes' number in various conditions

However, slight deviations were observed, suggesting that variations in binder dispersion, granulator dynamics, or particle interaction mechanisms may have influenced granule formation. Factors such as granulator rotation speed and binder spraying uniformity may have caused localized inconsistencies in granule size distribution. Despite these deviations, the overall trend supports using Stokes' number as a valuable tool for optimizing granulation conditions.

A linear regression analysis with the intercept set at 0.0 was conducted to simplify the predictive model. The obtained R-squared value of 0.95 indicates a strong correlation, even though some minor deviations were observed under specific experimental conditions. This strong correlation provides a practical equation for estimating granule sizes across different binder conditions and NPK formulations, offering valuable insights for fertilizer manufacturing.

4. Conclusions

This study demonstrates that the addition of ammonium sulfate (ZA) as a binder enhances granulation efficiency, producing larger and more uniform granules across different NPK formulations. The strong correlation between Stokes' number and granule size ($R^2 = 0.95$) provides a predictive model for optimizing granulation conditions in NPK fertilizer production.

The findings contribute to improving process control in fertilizer manufacturing, allowing for better binder selection and granulation parameter adjustments to achieve desired particle size distributions. However, this study was conducted on a laboratory scale, and further validation is required in larger-scale industrial settings to assess real-world applicability. Future research should explore the effects of additional process variables, such as granulator type and operating conditions, as well as alternative binder materials, to refine granulation models and improve fertilizer quality.

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Statement

During the preparation of this work, the authors used Grammarly and ChatGPT-4o in order to improve the English language and proofread the text. After using this tool/service, the authors reviewed and edited the content as needed and took full responsibility for the publication's content.

CRediT authorship contribution statement

Meiga Putri Wahyu Hardhianti: Writing – original draft, writing – review and editing, Formal analysis, Visualization. **Ivan Sebastian**: Formal analysis, Investigation, Methodology, Visualization, Writing – review and editing. **Wiratni**: Project administration, Supervision, Conceptualization, Methodology, Validation, Writing – review and editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

References

Adetayo, A. A., Litster, J. D., & Desai, M. (1993). The Effect of Process Parameters on Drum Granulation of Fertilizers with Broad Size Distributions. *Chemical Engineering Science*, *48*(23), 3951–3961. https://doi.org/10.1016/0009-2509(93)80374-Y

Adetayo, A. A., Litster, J. D., Pratsinis, S. E., & Ennis, B. J. (1995). Population balance modelling of drum granulation of materials with wide size distribution. *Powder Technology*, *82*, 37–49. https://doi.org/10.1016/0032-5910(94)02896-V

Amelia, N., Akhwan, R., & Yuliestyan, A. (2020). Influence of Particle Size and Chemical Activation on Rice Husk Biochar as Slow Release Fertilizer. *Eksergi*, *17*(2), 73–78. https://doi.org/https://doi.org/10.31315/e.v17i2.3730

Bouwman, A. M., Visser, M. R., Meesters, G. M. H., & Frijlink, H. W. (2006). The use of Stokes deformation number as a predictive tool for material exchange behaviour of granules in the “equilibrium phase” in high shear granulation. *International Journal of Pharmaceutics*, *318*(1–2), 78–85. https://doi.org/10.1016/j.ijpharm.2006.03.038

Dhenge, R. M., Fyles, R. S., Cartwright, J. J., Doughty, D. G., Hounslow, M. J., & Salman, A. D. (2010). Twin screw wet granulation: Granule properties. *Chemical Engineering Journal*, *164*(2–3), 322–329. https://doi.org/10.1016/j.cej.2010.05.023

Handayani, D. P., Sediawan, W. B., Timotius, D., & Puspitasari, M. (2023). Distribusi Ukuran Granul dari Tepung Singkong dengan Tepung Tapioka Sebagai Pengikat pada Rotary Drum Granulator. *Eksergi*, *20*(2), 52–57. https://doi.org/https://doi.org/10.31315/e.v20i2.9170

Iveson, S. M., Litster, J. D., Hapgood, K., & Ennis, B. J. (2001). Nucleation, growth and breakage phenomena in agitated wet granulation processes: a review. *Powder Technology*, *117*, 3–39. https://doi.org/10.1016/S0032-5910(01)00313-8

Kasmadi, K., Nugroho, B., Sutandi, A., & Anwar, S. (2019). Filter Cake Utilization as Filler of 15-15-15+5S Compound Fertilizer: Particle Size Distribution and Granule Crushing Strength Properties. *Reaktor*, *19*(4), 145–151. https://doi.org/10.14710/reaktor.19.4.145-151

Khalil Abu Rabeah, Ruben Socolovsky, Natalia Geinik, Ayoub Alhowashla, & Joseph Lati. (2020). *Binders for The granulation of Fertilizers* (Patent US20200055795A1). U.S. Patent and Trademark Office. https://patents.google.com/patent/US20200055795A1/en

Meng, W., Rao, K. S., Snee, R. D., Ramachandran, R., & Muzzio, F. J. (2019). A comprehensive analysis and optimization of continuous twin-screw granulation processes via sequential experimentation strategy. *International Journal of Pharmaceutics*, *556*, 349–362. https://doi.org/10.1016/j.ijpharm.2018.12.009

Mort, P., & Tardos, G. (1999). Scale-up of Agglomeration Processes using Transformations. *KONA*, *17*, 64–75. https://www.jstage.jst.go.jp/article/kona/17/0/17_1999013/_pdf/-char/en

Richard H. Van de Walle, & David Smith. (1992). *Binder for The Granulation of Fertilizers such as Ammonium Sulfate* (Patent US5078779A).

Sastra, R. A., Adiarto, T., Prasetyo, A. B., Darmokoesoemo, H., Indrasari, D., & Putri, M. (2023). Pemanfaatan Nanokitosan Sebagai Coating Agent dalam Pembuatan Pupuk NPK Berbasis Control. *Eksergi*, *20*(3), 195–199. https://doi.org/https://doi.org/10.31315/e.v20i3.10699

Steiger, B. G. K., Bui, N. T., Babalola, B. M., & Wilson, L. D. (2024). Sustainable agro-waste pellets as granular slow-release fertilizer carrier systems for ammonium sulfate. *RSC Sustainability*. https://doi.org/10.1039/d4su00141a

Szulc, A., Skotnicka, E., Gupta, M. K., & Królczyk, J. B. (2024). Powder agglomeration processes of bulk materials – A state of the art review on different granulation methods and applications. *Powder Technology*, *431*. https://doi.org/10.1016/j.powtec.2023.119092

Walker, G. M., Holland, C. R., Ahmad, M. N., Fox, J. N., & Kells, A. G. (2000). Drum granulation of NPK fertilizers. *Powder Technology*, *107*, 282–288. https://doi.org/10.1016/S0032-5910(99)00253-3

Walker, G. M., Holland, C. R., Ahmad, M. N., Fox, J. N., & Kells, A. G. (2001). Prediction of fertilizer granulation: Effect of binder viscosity on random coalescence model. *Industrial and Engineering Chemistry Research*, *40*(9), 2128–2133. https://doi.org/10.1021/ie000647s

Wang, C., Luo, D., Zhang, X., Huang, R., Cao, Y., Liu, G., Zhang, Y., & Wang, H. (2022). Biochar-based slow-release of fertilizers for sustainable agriculture: A mini review. *Environmental Science and Ecotechnology*, *10*. https://doi.org/10.1016/j.ese.2022.100167

Wang, G., Yang, L., Lan, R., Wang, T., & Jin, Y. (2013). Granulation by spray coating aqueous solution of ammonium sulfate to produce large spherical granules in a fluidized bed. *Particuology*, *11*(5), 483–489. https://doi.org/10.1016/j.partic.2012.10.005

Xue, B., Huang, H., Mao, M., & Liu, E. (2017). An investigation of the effect of ammonium sulfate addition on compound fertilizer granulation. *Particuology*, *31*, 54–58. https://doi.org/10.1016/j.partic.2016.04.004

Yu, Z., Zhao, J., Hua, Y., Li, X., Chen, Q., & Shen, G. (2021). Optimization of granulation process for binder-free biochar-based fertilizer from digestate and its slow-release performance. *Sustainability (Switzerland)*, *13*(15). https://doi.org/10.3390/su13158573