

Performance of spouted bed on drying of foliar fertilizer: analysis of yield and powder retention

Performance do leito de jorro na secagem de fertilizante foliar: análise do rendimento e da retenção de pó

Ana Carolina Ribeiro Stoppe¹; Mário Sérgio da Luz²; José Luiz Vieira Neto³; Kássia Graciele Santos⁴

¹ Master's in Technological Innovation, Federal University of Triângulo Mineiro, Uberaba-MG, Brazil. Orcid: 0000-0002-4744-0738. E-mail: <u>anacarolina_stoppe@hotmail.com</u>

² Professor of the Professional Master's Program in Technological Innovation, Federal University of Triângulo Mineiro, Uberaba-MG, Brazil. Orcid: 0000-0003-1226-9480. E-mail: <u>mario.luz@uftm.edu.br</u>

³ Professor of the Undergraduate Course in Chemical Engineering, Federal University of Triângulo Mineiro, Uberaba-MG, Brazil. Orcid: 0000-0003-0736-3974. E-mail: <u>iose.neto@uftm.edu.br</u>

⁴ Professor of the Professional Master's Program in Technological Innovation, Federal University of Triângulo Mineiro, Uberaba-MG, Brazil. Orcid: 0000-0001-7452-6900. E-mail: <u>kassia.santos@uftm.edu.br</u>

ABSTRACT: Foliar fertilization is one of the most important management techniques used to improve crop productivity and quality. To increase the efficiency of fertilization, the fertilizer must be highly soluble. The drying of paste and solutions in spouted bed can change the powder structure, making it more soluble. However, depending on the operational conditions, the powder yield produced in this operation may be low. As such, the goal of this research was to investigate the impact of operational conditions on powder yield and powder losses caused by adhesion on the walls of the bed and cyclone, on particle surface, on the exhaust tube, filter, and filter overflow. To accomplish this, a 2³ factorial design was used. The results of powder yield (between 2.3 and 26.6%.) were like those obtained in other spouted bed applications, but lower when compared to spray-dryer. The higher powder retention occurred on the surface of inert particle and at the bed wall. A high level of instability was observed during spouting, which may have increased the powder adherence at the equipment's walls. The powder obtained after drying was solubilized about 5.9 times fast than the original fertilizer, demonstrating that the technique is promising if the bed stability is maintained.

Keywords: Foliar nutrition. Spouting. Microgranulation. Agroindustry.

RESUMO: A fertilização foliar é uma das principais técnicas de manejo utilizadas para melhorar a produtividade e qualidade das culturas. Para que a fertilização seja mais eficiente, é necessário que o fertilizante seja altamente solúvel. A secagem de pastas e soluções em leito de jorro é capaz de alterar a estrutura do pó a fim de torná-lo mais solúvel. No entanto, o rendimento de pó produzido nesta operação pode ser baixo, a depender das condições operacionais. Assim, este trabalho teve como objetivo estudar a influência das condições operacionais sobre o rendimento de pó e as perdas de pó que ocorrem por adesão nas paredes do leito, do ciclone, tubulação de exaustão, filtro e overflow do filtro. Para tal, utilizou-se um planejamento fatorial 23. Os resultados de rendimento de pó (entre 2.3 e 26.6%.) foram similares a obtidos em outras aplicações do leito de jorro, mas baixos comparados com o rendimento em spray-dryer. A maior retenção de pó ocorreu por adesão nas paredes do equipamento. O pó obtido apresentou tempo de solubilidade 5,9 vezes menor que o material in natura, mostrando que a técnica é promissora, desde que a estabilidade do leito seja mantida.

Palavras-chave: Nutrição foliar. Jorro. Microgranulação. Agroindústria.

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INTRODUCTION

Foliar fertilization is important because it is a quick and effective way to deliver nutrition directly to a plant's leaves. This technique aids plant development by delivering nutrients directly to the leaves, where they are more quickly absorbed. So, the foliar fertilizer needs to be highly water soluble to be absorbed directly by the leaves, providing instant nutrition to the plant. Foliar fertilizer can help a plant stay healthy and vigorous, and it can also help increase crop yields. Foliar fertilizer can also help plants resist diseases, improve water retention, provide essential micronutrients, and provide additional protection from extreme temperatures.

Foliar fertilizers vary in solubility depending on their composition. Generally, foliar fertilizers are composed of inorganic salts, organic acids, and/or chelated minerals. Most inorganic salt fertilizers are highly soluble, while organic acids and chelated minerals tend to be less soluble.

Some foliar fertilizers are sold in powders, which decreases transportation expenses. As a result, the farmer must solubilize them in water and pulverize them on the crops. In the field, fertilizers and pesticides are regularly combined, which can diminish solubility and clog spray nozzles (ALBERTO et al., 2022). Efforts to make foliar fertilizers more soluble may increase value to the product while lowering operational expenses in this context.

The high solubility of foliar fertilizers in powder provides benefits such as the ability to apply lesser amounts of solution, resulting in cost savings for the customer, as well as the prevention of nozzle and equipment clogging concerns (ALBERTO et al., 2022).

Spray dryers, lyophilization, fluidized bed dryers, and spouted bed (SB) dryers are all standard methods for drying pastes and liquids (MUJUMDAR, 2014), also used for particle coating (SOUZA et al., 2019).

A spouted bed dryer operating with inert particles is commonly used for drying pastes and suspensions for many applications, like pharmaceuticals, chemicals, and food industries. During the drying of pastes in SB dryer, the inert particles are placed on the top of the spouted bed and the paste is evenly distributed over them by atomization or dripping. As the paste dries, it is drawn down the spout by an air flow generated by a fan. The inert particles act as suspension support and help to promote even drying of the paste, as well as providing some degree of heat transfer from the air to paste (ALMEIDA; ROCHA, 2002).

Powder retention occurs when some of the powder remains inside the spouted bed after the drying process has been completed and on the walls of cyclone. This can reduce the powder recovery efficiency of the dryer and can also reduce the quality of the finished product. The adhesion forces can cause some of the powder to stick to the walls of the spouted bed. This can result in the powder not being removed from the spouted bed during the drying process, leading to a low powder recovery efficiency (SOUZA; OLIVEIRA, 2004). The adhesion forces can be reduced by using a higher air flow rate, which can help to reduce the amount of time that the powder is exposed to the walls of the spouted bed. In addition, lower temperatures could benefit the drying of organic material, avoiding the glass transition temperature of the material can help to reduce the adhesion forces (SOUZA; OLIVEIRA, 2004).

Some researchers looked at the effect of operating circumstances on the powder retention on spouted bed dryers with inert particles. Barros, Ferreira and Freire (2019) dried a CaCO₃ suspension in SB dryer. Depending on the drying settings, they recovered about



11 to 53% of solids at cyclone underflow, 2 to 8% in the filter, and 23 to 34% of particles were retained inside the drying vessel.

The operational conditions can interfere in the spouted bed dryer performance. Intermittent feeding has been shown to improve the drying rate of spouted beds and reduce the amount of powder retention on the bed wall (DANTAS et al., 2018). It also increases the turbulence in the bed, which helps to reduce the amount of powder retention on the bed wall. The atomizer position plays a significant role in the performance of spouted bed drying and it can influence the powder retention and drying rate. So, the atomizer position should be adjusted based on the specific design of the spouted bed and the current application (SOUZA; OLIVEIRA, 2004).

In this context, the aim of this work was to investigate the main causes of powder retention in spouted bed drying of a commercial foliar fertilizer. For that, the foliar fertilizer was characterized, as well the inert particles. The fluid dynamics of spouted bed was investigated experimentally and using correlation of literature, for different initial weight of inert particles. The drying was performed to evaluate the effect of feeding time; intermittent suspension feeding time and feed atomizer position on the powder production and powder losses at the equipment walls and particle surface. So, the statistical analysis was done to identify the main mechanisms of powder retention that can jeopardize the powder production efficiency.

METHODS AND MATERIALS

Particulate Materials

The commercial powder foliar fertilizer (PFF) employed in this investigation was a micronutrient source with an acidity of 2.80 to 3.80 and a composition of 16.5% S, 0.50% B, 1.00% Cu, 25.0% Mn, and 4.00% Zn, the same fertilizer dried by Alberto et al. (2022) using spray dryer technique.

The inert particles of interest in this study were low-density polyethylene pellets (LDPE), a material that was selected due to some of its characteristics. As polyethylene is a light material, it requires a smaller amount of energy to perform the necessary fluid dynamics of the equipment, in addition to having a slightly porous surface, which will facilitate the detachment of the dry powder that will cover the particle.

To estimate whether a solution or paste will efficiently wet the surface of a particle, it is necessary to account for the contact angle between them. In studies carried out by Rocha, Donida and Marques (2009) it was found that in spouted bed dryers using aggregates, contact angles greater than 70° do not contribute to the coating of the particle. This happens because the adhesion forces of the polymeric film on the surface of the inert solid are not strong enough, tending towards drying and granulation, with the fine powder being eluted by the process.

To verify the contact angle of the foliar fertilizer suspension and the LDPE surface, 40 photos of different drops of the 0.02 g.mL⁻¹ solution (concentration recommended by the manufacturer for use in agriculture) were taken on a smooth LDPE surface. Then, the photos taken with a cell phone were processed online on a website called the Online Transfer (GINIFABI, 2021). On this site it is possible to add any type of photograph and calculate angles on the images. Next, an average was calculated to find the average value of the angles. The measured contact angle was 76.22°, showing and adequate wettability. So,



because it is above 70°C, it is regarded suitable for bed drying granulation (ROCHA; DONIDA; MARQUES, 2009).

As inert particles, we employed low-density polyethylene pellets (LDPE) in this investigation. **Table 1** shows the physical and morphological properties of polyethylene obtained by the analysis of particles image using ImageJ software (BONTEMPO; CASTEJON; SANTOS, 2020). The results were similar to those reported by Coutinho et al. (2003). Picnometry tests revealed that the average diameter of the LDPE particles was 4.1 mm, and the apparent density was 878 kg/m³.

The Geldart classification allows particles to be classified based on the kind of fluidization they cause with the gas, considering the difference between particle and air densities (ρ_p - ρ) in g/cm³ and the particle diameter d_p, in µm. According to the Geldart classification, the LDPE particles can be classified as a D group, large particles with the ability to spout (GELDART, 1986).

Propriety	Mean value ± deviation
Projected area diameter - da (cm)	0.4704 ± 0.0111
Volumetric diameter - d _v (cm)	0.4105 ± 0.0010
Sfericity – φ	0.9469 ± 0.0145
Particle density – ps (g/cm3)	0.8994 ± 0.0061
Bed voidage - ε	0.3943 ± 0.0007

 Table 1. Inert particle proprieties (PEBD)

Experimental unit

Figure 1 (a) presents the main elements of the spouted bed system, as well as the discrimination of the process variables and bed geometry. The drying tests were carried out on a stainless-steel cone-cylindrical spouted bed with a 40° internal angle. The cylindrical section and bottom inlet orifice had dimensions of 0.12 m and 0.02 m, respectively.

The air was supplied by an air blower (4 hp). A 4000 W resistance regulated by a PID controller heated the air. The air flow rate was measured using an orifice flow meter, and the pressure drop across the orifice was monitored using differential pressure transducers. A pressure transducer (Series MS Magnesense® from Dwyer Instruments) was used to monitor the pressure drop through the particle bed, and the signal was sent to a microcomputer through an A/D data acquisition card (National Instruments DAQ - USB 6001) and processed using LabVIEW software.

The temperature was monitored in different positions to obtain T0, T1, T2, and T3, described at **Figure 1**. The mean interest point was the bed center (T1), which corresponds to the region with the most effective fluid-particle interaction. At this position, the initial temperature should be around 100°C. According to Alberto et al. (2022), drying at 100°C can remove water from crystalline compounds, modifying the morphology of the product to amorphous to increase its solubility. However, as the inert material used deforms with heat, temperatures above 110°C should be avoided.

The fertilizer solution was sprayed with a dual-fluid atomizing nozzle, one linked to the peristaltic pump and the other to a 1 bar air compressor hose. The dried fertilizer was elutriated and collected in a 0.105 m cylindrical Lapple cyclone. To gather tiny particulate matter, we install a bag filter (200 mesh) at the cyclone overflow.



Figure 1. Experimental Unit scheme: a) Drying system. Caption: (1) Spouted bed; (2;3;4;5) Type K thermocouples; (6) Bed pressure gauge; (7) Solution feed; (8) Cyclone (Lapple); (9) exhaust pipe from bed to cyclone. (b) Internal view of the spouted bed with the atomizer at the two heights used during the tests, H1 and H2. All measurements are in millimeters



Fluid dynamic of spouted bed with inert particles

The fluid dynamic of the spouted bed was evaluated by the characteristic curves of pressure drop versus air flow rate, performed at different loads of inert solids: 0.2 kg, 0.3 kg, 0.4 kg, 0.5 kg, 1.0 kg, and 1.5 kg. These curves were analyzed to obtain the maximum pressure drop (ΔP_{Max}), pressure drop at minimum spouting (ΔP_{ms}), and minimum spout velocity (U_{ms}), which were also compared with correlations in the literature, according to **Table 2**. The empirical correlations were calculated using the Archimedes number (Ar) and/or the minimum spouted Reynolds number (Re_{ms}) and often include the interaction of bed geometric factors as well as the properties of inert particles, drying fluid, and system operational conditions (SANTOS et al. 2019).

Drying experiments

All drying experiments were performed using about 1 kg of LDPE particle, an air supply flow of 120 m³/h (about 1.5x Q_{ms}), a feeding flow of 19 mL/min; atomizer nozzle pressure of 1 bar and a drying air temperature at 100 °C.

The tests were designed according to a 2^3 factorial design to evaluate the effect of three factors over the responses: feeding time (*t_F: 10 and 30 s*); intermittent suspension feeding time (*t_i: 60 and 300 s*) and feed atomizer position (*H_a: 0.4 and 0.68 m*).



Multiple regression was used to evaluate the whole set of experimental data, assessing the impact of each independent variable on the responses: powder yield (Y [%]) and mass fraction of powder retention at different places: adhered on particle surface (R1), at bed wall (R2), at cyclone wall (R3), at the exhaustion pipe (R4), at the bag filter (R5) and at overflow of filter (R6). The Student's t-test was used to determine the significance of regression parameters (p-level<0.1).

Table 2. Empirical correlations to estimate maximum pressure drop, air velocity and pressure drop at minimum spouting condition

Referências	Correlations		Boundary Conditions
Mukhlenov; Gorshtein (1965)	$(\text{Re})_{\text{ms}} = 3.32 \text{ Ar}^{0.33} \left(\frac{\text{H}_0}{\text{D}_0}\right) \text{tg} \left(\frac{\text{Y}}{2}\right)^{0.55}$	(1)	$D_0=10.3-12.9$ mm; $H_0=3-15$ cm; $\gamma=12-60^\circ$; $d_p=0.5-2.50$ mm; $\rho_p=1000-2360$ kg/m ³ ; $D_c=5$ cm.
	$\frac{-\Delta P_{\rm ms}}{H_0 \rho_p g} = 7.68 \left[tg\left(\frac{\gamma}{2}\right) \right]^{-0.2} ({\rm Re})_{\rm ms}^{-0.2} \left(\frac{H_0}{D_0}\right)^{-0.53}$	(2)	, , , , , , , , , , , , , , , , , , ,
Tsvik et al. (1967)	$(\text{Re})_{\text{ms}} = 0.4 \text{ Ar}^{0.52} \left(\frac{\text{H}_0}{\text{D}_0}\right)^{1.24} \text{tg} \left(\frac{\text{Y}}{2}\right)^{0.42}$	(3)	$\begin{array}{l} D_0{=}2{-}4\ cm;\ H_0{=}10{-}50\ cm;\\ H_0/D_0\ {=}1.6{-}8.7;\ \gamma{=}20{-}50^\circ;\\ d_p{=}1.5{-}4.0\ mm;\\ \rho_p\ {=}1650{-}1700\ kg/m^3. \end{array}$
Olazar et al. (1992) Olazar et al. (1993)	$\frac{-\Delta P_{\text{max}}}{(1-2)^{-0.11}} = 1.2 \text{tg} \left(\frac{\gamma}{2}\right)^{-0.11} (\text{Re})_{\text{max}}^{-0.06} \left(\frac{H_0}{2}\right)^{0.08}$	(4)	D ₀ =3-6 cm; H ₀ =3.6-6.1 cm; v=28-45°; d _n =1-2.5 mm;
	$H_0\rho_p(1-\varepsilon_0)g = (2/\sqrt{ms} (D_0))$	(5)	$\rho_p = 240-3520 \text{ kg/m}^3.$
	$\frac{-\Delta P_{\rm ms}}{-\Delta P_{\rm max}} = 1 + 0.116 \left(\frac{H_0}{D_0}\right)^{-1} \text{ tg} \left(\frac{\gamma}{2}\right)^{-0.3} \text{ Ar}^{0.0125}$		
Pallai; Nemeth	$-\Delta P_{max} = H_0 \left(\rho_p - \rho \right) (1 - \epsilon) g$	(6)	D _c = 10 a 30 cm.
(1909)	$\frac{-\Delta P_{ms}}{-\Delta P_{max}} = 0.8 - 0.01 \frac{D_c}{D_0}$	(7)	
Sampaio Correlation (Almeida; Rocha, 2002)	$-\Delta P_{\rm ms} = \frac{2}{3} \rho_{\rm p} \ g \ H_0$	(8)	D_0 =15cm; H ₀ =3-15 cm; γ = 60°; d _p =4-6 mm; ρ_p =1100-1190 kg/m ³ .
Saldarriaga et al.	$\frac{-\Delta P_{\text{máx}}}{2} = 1.20 \text{ tg} \left(\frac{\gamma}{2}\right)^{-0.11} (\text{Re}) -0.06 \left(\frac{H_0}{2}\right)^{0.11}$	(0)	$0.83 < H_0/D_0 < 50;$
(2013)	$H_0 \rho_p (1-\varepsilon_0) g$ $(1-\varepsilon_0) g (D_0)$	(3)	$50 < (\text{Re})_{\text{ms}} < 4900.$
Reynolds Number (Re)	$(Re)_{ms} = \frac{U_{ms} \rho d_{\rho}}{\mu}$	(10)	
Arquimedes Number (Ar)	$Ar = \frac{\rho d_p^3 (\rho_p - \rho)}{\mu^2}$	(11)	
Fonte: Santos et al. (20	119)		

Fonte: Santos et al. (2019).

The experimental procedure of drying consisted of heating the air up to the specified temperature. After that, paste injection started. About of 100 mL of PFF aqueous solution at a concentration of 0.5 kg/L was fed according to the feeding time and intermittent suspension feeding time, defined by the experimental design. The fluid was then evaporated to produce the dried powder foliar fertilizer (D-PFF).

The powder yield was determined according to Eq. (12), as the ratio of the mass of dry powder collected in the underflow of cyclone (m) and the initial mass of fertilizer in the feed solution (m_0) .

$$Y\% = \frac{m}{m_0}.100$$
 (12)

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RESULTS AND DISCUSSION

Fluid dynamic of spouted bed with inert particles

Figure 2 illustrates the spouted bed characteristic curves, where the pressure drop is a function of air flow supply, for different initial weight of inert solids. Through these curves, the condition of flow and pressure at the minimum spout was identified.

Figure 2. Characteristic curves of the spouted bed operating with: (a) 0.2 kg, (b) 0.5 kg, (c) 1 kg and (d) 1.5 kg



It is clear how the increase in inert load affects the operating factors of the bed. The maximum pressure drop and minimum spout velocity rise as the number of inert particles inside the bed increases. Santos et al. (2015) presented similar results, showing that this behavior is reasonable since an increase in inert mass causes more resistance to air flow, resulting in a rise in pressure drop and minimum spout velocity.

Figure 2 (a) shows the curve with a load of 0.2 kg; it can be observed that this quantity did not match with the trip in turns 1 and 2, showing bed instability. The similar behavior can be seen in the other curves with initial solid weight between 0.2 and 0.5 kg. The characteristic curves demonstrated outstanding stability with weights of 1 kg (c) and 1.5 kg (d).



Bacelos and Freire (2006) observed that the increasing of particle bed's height could broaden the range of stable spouting regimes in the bed. This is because the cyclic spouting motion requires a minimum particle height to be attained.

Perazzini et al. (2015) statistically evaluated the load of inert particles as a variable for the rate of water evaporation in a spouted bed, and she discovered that high values of this variable contributed to the system's best working state.

Based on this background and experimentally testing bed stability at different weights, the 1 kg mass was chosen as the quantity utilized in the drying tests, as it has a significant surface area and would require less energy consumption to carry out the dryings than the 1.5 kg mass.

Table 3 shows the experimental values of maximum pressure drop; pressure and air velocity at minimum spouting condition, while **Figure 3** compares these results with those predicted by correlations in **Table 2**.

Table 3. Experimental measurement of maximum pressure drop $(-\Delta P_{max})$, velocity (U_{ms}) and pressure drop $(-\Delta P_{ms})$ at minimum spouting condition, for different initial solid weight at spouted bed

Initial solid weight (g)	Particle Superficial Area (cm ²)	-ΔP _{max} (Pa)	-ΔP _{ms} (Pa)	U _{ms} (m.s ⁻¹)
200	1067.64	676.64	193	6.43
300	1601.46	1010.06	285	7.60
400	2135.29	931.61	387	8.18
500	2669.11	1372.89	485	8.77
1000	5338.22	2422.18	817	11.46
1500	8007.32	2863.42	1195	13.56

Figure 3 (a) depicts the ratio of projected minimum spout velocities as a function of empirically determined values. The correlation that came closest to the observed values was that of Mukhlenov and Gorshtein (1965). This prediction takes into count the ratio of the static bed's height by the size of the bed's inlet diameter (H_0/D_0), and their boundary conditions do not present limits for the values resulting from this relation. Tsvik et al. (1967) limit the values for H_0/D_0 in their boundary conditions to the point when the final two points are beyond the limitations for this factor and away from the experimental points. Olazar et al. (1992) limit the height range for the valid static spouted bed H_0 to 3.6 - 6.1 cm. which has an influence on the prediction of the minimum spouting velocity for the equipment in this investigation. because H_0 values range from 10 to 30.5 cm.

Figure 3(b) shows a comparison of estimated and experimental values of minimum spout pressure drop. Pallai Nemeth (1969) had the closest correlation to this condition. Since the equation of this correlation presents considerable interference from the geometry of the spout since the only requirement of contour (Dc between 10 and 30 cm) is valid for the analyzed bed. The equipment geometry differs from most of the boundary conditions of the other correlations. Which explains the larger difference between anticipated and actual values.

Figure 3 (c) depicts a comparison of three literature correlations and experimental data for the maximum bed pressure decrease. The results obtained by the correlation of Saldarriaga et a. (2013) were in agreement with the experimental data; once the bed



geometry obeys two boundary requirements of this correlation (the Re_{ms} value for 50 to 4900 and H_0/D_0 between 0.83 and 50).

Figure 3. Comparison between experimental data and those estimated by correlations for (a) minimum spout velocity; (b) minimum spout pressure drop and (c) maximum pressure drop



Mapping of powder losses during drying

The drying tests consisted of eight tests following the 2³ experimental design, according to **Table 4**. The powder production efficiency of fertilizer in spouted bed dryers ranged between 2.3 and 26.6%. In comparison to the spray dryer (67-82%) results reported by Alberto et al. (2022), we see a decreased powder production of foliar fertilizer.

The powder yield (Y) was reduced by the increment of intermittent suspension feeding time (x2); while high values of atomizer height (x3) favored the powder yield. The interaction (x2x3) was also significant, according to the Student's t-test at **Table 5**.

We noticed a high solid retention at the spouted bed wall and cyclone wall, as well as on the particle's surface, at the end of the experimental testing. The powder recovery was



compromised by the instability of the spouting regime. As a result, we believe that modifying the shape of the spouted bed bottom may boost the bed's fluid dynamic stability, which will have a direct influence on the powder yield by reducing its adherence to the bed walls.

Table 4. Experimental factorial design and the main responses: powder yield (Y [%]) and mass fraction of powder retention at different places: adhered on particle surface (R1), at bed wall (R2), at cyclone wall (R3), at the exhaustion pipe (R4), at the bag filter (R5) and at overflow of filter (R6)

	Factors			Responses						
Exp.	t _{F [s]}	t _{l [s]}	H _A [m]	V [0/]	Mass fraction of solid retention					
	(X ₁)	(X ₂)	(X ₃)	r [%]	R1	R2	R3	R4	R5	R6
1	10 (-1)	60 (-1)	0.40 (-1)	8.9	0.435	0.120	0.125	0.075	0.061	0.095
2	30 (+1)	60 (-1)	0.40 (-1)	7.6	0.315	0.160	0.040	0.035	0.065	0.309
3	10 (-1)	300 (+1)	0.40 (-1)	2.9	0.505	0.115	0.080	0.050	0.042	0.179
4	30 (+1)	300 (+1)	0.40 (-1)	2.3	0.505	0.185	0.155	0.055	0.040	0.039
5	10 (-1)	60 (-1)	0.68 (+1)	18.2	0.075	0.315	0.135	0.115	0.044	0.134
6	30 (+1)	60 (-1)	0.68 (+1)	25.2	0.103	0.420	0.060	0.110	0.055	0.001
7	10 (-1)	300 (+1)	0.68 (+1)	6.6	0.090	0.430	0.220	0.080	0.020	0.094
8	30 (+1)	300 (+1)	0.68 (+1)	4.9	0.105	0.440	0.160	0.095	0.040	0.111
9*	30 (+1)	60 (-1)	0.68 (+1)	26.63	-	-	-	-	-	-

* Test 9 is a duplicate of test 6, in which the mass fraction retained in each region of the system was not accounted for, however the yield of test 9 was the highest obtained during the study.

Table 5. Estimated Effects by Student's t-test for the responses: powder yield (Y [%]) and mass fraction of powder retention at: particle surface (R1), at bed wall (R2) and at the bag filter (R5)

Responses	Factor	Effect	Deviation	p-level
	mean	0.100	0.010	0.000
V (B2 0.020)	x2	-0.116	0.020	0.002
$f(R^2 = 0.939)$	x3	0.091	0.020	0.006
	x2x3	-0.060	0.020	0.030
P_1 particle surface $P_2^2 = 0.007$	mean	0.267	0.023	0.000
RT – particle sufface R =0.907	x3	-0.347	0.045	0.000
	mean	0.273	0.013	0.000
R2- bed wall (R ² =0.950)	x1	0.056	0.027	0.089
	x3	0.256	0.027	0.000
	mean	0.046	0.001	0.000
	x1	0.008	0.002	0.038
R5- Bag Filter (R ² =0.977)	x2	-0.021	0.002	0.003
	x3	-0.012	0.002	0.013
	x1x3	0.007	0.002	0.052

Table 4 also presents the mass fractions of fertilizer adhered in each region of the drying system that were not collected in the underflow of the cyclone, contributing to the



reduction of the process yield. **Table 5** displays the statistical analysis of the effects of the three factors over all responses obtained, with a significance p-level of 10%.

The mass fraction of the fertilizer adhered on the surface of the inert particles (R1) was influenced by the height of the atomizer (x_3), as shown at **Figure 4a**. This effect had a negative sign, so the lower the H_A from the bottom, the closer the solution was sprayed onto the particles, causing a powder retention on particle surface.

Figure 4. Response surface showing the influence of feeding time (x1), intermittent suspension feeding time (x2) and the feed atomizer position (x3): (a) retained the surface of LDPE particles (R1); (b) collected at filter bag (R5)



Figure 5 shows the mass fraction of fertilizer retained on the bed wall (R2). Statistical analysis of **Table 5** shows that the factor that most influenced the fraction of mass lost due to adhesion to the bed wall was the height of the atomizer (x3). The effect with a positive sign indicates that the greater distance between the atomizer and the bottom of the bed, where the inert material is located, caused the sprayed mixture to move away from the inert particles. Thus, this reduced the solid-liquid contact between the inert particles and the sprayed fertilizer, causing part of the fertilizer to deposit on the bed wall, drying and adhering to the region.

The feeding time (x1) also had a positive effect, because as tF increases, a greater volume of atomized solution accumulates inside the bed, meaning that not all the material has managed to be transferred to the surface of the particle. inert in time, drying and adhering to the bed region. Thus, the wet inert particles collide with the walls of the bed, forming a film of fertilizer on the walls. As a result, the more fertilizer adhered to the wall, the lower the yield. Also, the temperature used may be insufficient to dry all wet material at higher feed times.

Rosa (2010) observed a similar behavior, in which atomization heights far from the bottom led to loss of suspension, as contact with hot air dries the solution before it interacts with the inert particles. This causes the solution to be dried by elutriation and not by the mechanism of interest in this work, the spouled bed with the inert particles.

Machado et al. (2015) also found that tests with low yields, in long intermittence times, led to significant amounts of material retained on the bed wall and on the surface of the equipment.



Figure 5. Image in different bed positions of mass fractions retained on the wall



The fertilizer fractions retained at the exhaustion pipe (R4) and overflow of bag filter (R6) were not affected by the factors, but represented 7.3% and 12% of solid retention, respectively.

For the mass fraction of fertilizer collected in the bag filter (R5) after the cyclone overflow, the most significant factor was the t_i , as can be seen in **Table 3**. The interaction of x1x3 can be visualized by the response surface in **Figure 5b**. The t_i has a negative influence on the powder collected at the filter, showing that longer intermittence times resulted in the creation of bigger particles, which are often gathered at the cyclone underflow and are not pulled to the filter. The height of the atomizer also had a negative effect, in that a greater distance between the atomizer and bottom causes the creation of bigger particles, resulting in a lesser quantity of dust trapped in the filter.

Characterization of foliar fertilizer

Figure 6 shows the image of the dried fertilizer powder, with a magnification of 1600x. It is possible to observe the increase in the size of the particles, as well as their shape.



Figure 6. Image of fertilizers with 1600x zoom after drying

Using Equation 13, it was possible to calculate the Sauter diameter of the fertilizer, that indicates the particle diameter at which the volume per surface ratio is the same for all particles present in a sample. The mean diameter of Sauter increased after drying, changing from 0.086 mm in natura to 0.093 mm after the process.

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$$\overline{\mathsf{D}} = \frac{1}{\sum_{i=1}^{\Delta x_i}}$$

where Xi is the mass fraction retained in the opening of the sieve n, and D is the average diameter between sieve opening n and sieve opening n-1.

The solubility test was performed before and after the fertilizer went through the drying process. The fertilizer solubility time was measured by analyzing the color of the solution during its solubilization in water, under agitation. At first the solution is cloudy, as can be seen in Figure 7(a), over time it was possible to notice the formation of the vortex, as the solution became translucent, which allowed the identification of the solubilization time, in the **Figure 7(b)**.

The in natura fertilizer presented a solubilization time of 61 seconds, and this value dropped to 19 seconds after the drying process with spouted bed and inert particles. This reduction in time points to a 69% improvement in the solubility of the foliar fertilizer.

Figure 7. Foliar fertilizer (a) before and (b) after solubilization



Alberto et al. (2022) performed the drying of the same fertilizer in natura used in this study, the method used was through spray dryer equipment. The author found a solubility of the product after the spray dryer treatment, between 11 and 31 seconds, a range that

coincides with that found in the final product in the spouted bed.

CONCLUSIONS

Spouted bed dryers are an important tool in the pharmaceutical, chemical and food industries. Understanding how adhesion and inert particles can affect powder retention. and using strategies to reduce dust retention. the efficiency of the spouted bed dryer can be improved. Using cyclones. powder that is trapped in the spouted bed can also be recovered, thus reducing the amount of powder trapped and improving the quality of the finished product.

Through the characteristic curves, it was possible to choose a load of 1 kg of aggregates, as it presents the best fluid dynamic stability for the geometries of the spouted bed used. The correlations predicted in the literature approached the observed values when the geometry and operation ranges were within the stipulated boundary conditions. A mass balance was performed between regions along the equipment to map the lost fertilizer fractions, these data were statistically analyzed, reaching the conclusion that the height of the atomizer was the variable that influenced all regions, but the flashing time had greater

(13)



effect on fractions adhered to the filter and bed wall. Finally, the solubility test carried out with the fertilizer after the drying process obtained an improvement of 69%.

If you are looking for a way to reduce the amount of dust retention in spouted bed dryers. then understand the effect of adhesion and inert particles. and using the strategies outlined in this article. can help you achieve this.

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REFERENCES

ALBERTO, L.; LUZ, M. S.; DOS SANTOS, K. G.; OKURA, M. H. Enhanced solubility of foliar fertilizer via spray dryer: Process analysis and productivity optimization by response surface methodology. **Ciência e Agrotecnologia**, v. 46, e002422, 2022. DOI: <u>https://doi.org/10.1590/1413-7054202246002422</u>

ALMEIDA, C.; ROCHA, S. C. S. Fluidodinâmica de sementes de brócolis em leito fluidizado e leito de jorro. **Scientia Agricola**, v. 59, n. 4, p. 645-652, 2002. DOI: <u>https://doi.org/10.1590/S0103-90162002000400004</u>

BACELOS, M. S.; FREIRE, J. T. Stability of Spouting Regimes in Conical Spouted Beds with Inert Particle Mixtures. **Industrial & Engineering Chemistry Research**, v. 45, n. 2, p. 808–817, 2006. DOI: <u>https://doi.org/10.1021/ie050633s</u>

BARROS, J. P. A. A.; FERREIRA, M. C., FREIRE, J. T. Spouted bed drying on inert particles: Evaluation of particle size distribution of recovered, accumulated and elutriated powders. **Drying Technology**, v. 38, n. 13, p. 1709-1720, 2019. DOI: https://doi.org/10.1080/07373937.2019.1656644

BONTEMPO, L. H. S.; CASTEJON, L. V., SANTOS, K. G. Drying of Tangerine peel: kinetics and performance of a convective solar dryer. **Research, Society and Development**, v. 9, n. 6, e44963458, 2020. DOI: <u>https://doi.org/10.33448/rsd-v9i6.3458</u>

DANTAS, T. N. P. Influência das propriedades físicas de graviola e aditivos na secagem em leito de jorro com alimentação intermitente. 2018. 121 p. Tese (Doutorado em Engenharia Química) - Centro de Tecnologia, Universidade Federal do Rio Grande do Norte, Natal, 2018.

GELDART, D. Gas Fluidization Technology. John Wiley: New York, 1986.

GINIFAB. **Transferidor Online**, 2021. Available at: https://www.Ginifab/feeds/ angle_measurement/online_protractor.pt.php. Accessed on: 2023 Feb. 15.

MACHADO, I. P.; DELMIRO, T. M.; MACHADO, A. K. T.; MEDEIROS, M. F. D. Secagem em leito de jorro da mistura graviola e leite. Avaliação dos efeitos das variáveis de operação sobre parâmetros de produção, taxa de secagem e eficiência térmica. In: **XV Congresso Brasileiro de Sistemas Particulados (ENEMP)**, São Carlos, 2015.

MUJUMDAR, A. S. Handbook of Industrial Drying, 4th ed, CRC Press., 2014. 1348 p.



MUKHLENOV, I. P.; GORSHTEIN, A. E. Investigation of a spouting bed. **Zhurnal Khimicheskaya Promyhslennost**, v. 41, p. 443–446, 1965.

OLAZAR, M; SAN JOSÉ, M. J.; AQUAYO, A. T.; ARANDES, J. M.; BILBAO, J. Stable Operation Conditions for Gas-Solid Contact Regimes in Conical Spouted Beds. **Industrial & Engineering Chemistry Research**, v. 31, n. 7, p. 1784-1792, 1992.

OLAZAR, M; SAN JOSÉ, M. J.; AQUAYO, A. T.; ARANDES, J. M.; BILBAO, J. Pressure Drop in Conical Spouted Beds. **The Chemical Engineering Journal**, v. 51, p. 53-60, 1993.

PALLAI, I.; NEMETH, J. Analysis of flow forms in a spouted bed apparatus by the so-called phase diagram. In: International Congress of Chemical Engineer (CHISA), v. 3, 1969.

PERAZZINI, M. T. B.; FREIRE, F. B.; FREIRE, J. T. Influence of Bed Geometry on the Drying of Skimmed Milk in a Spouted Bed. **Adv. Chemical Eng. and Science**, v. 5, p. 447-460, 2015.

ROCHA, S. C. S.; DONIDA, M. W.; MARQUES, A. M. M. Liquid-particle surface properties on spouted bed coating and drying performance. **The Canadian Journal of Chemical Engineering**, v. 87, n. 5, p. 695-703, 2009. DOI: <u>https://doi.org/10.1002/cjce.20208</u>

ROSA, G. S. Recobrimento de uréia em leito de jorro para minimizar as perdas de nitrogênio por volatilização. 2010. 108 p. Tese (Doutorado em Engenharia Química) - Unicamp, Campinas, 2010.

SALDARRIAGA, J. F.; ATXUTEGI, A.; ALTZIBAR, H.; BILBAO, J.; OLAZAR, M. Correlations for Calculating Peak and Spouting Pressure Drops in Conical Spouted Beds of Biomass. **Journal of the Taiwan Institute of Chemical Engineers,** v. 80, p. 678–685, 2017. DOI: <u>https://doi.org/10.1016/j.jtice.2017.09.001</u>

SANTOS, K. G.; FRANCISQUETTI, M. C. C.; MALAGONI, R. A.; BARROZO, M. A. S. Fluid Dynamic Behavior in a Spouted Bed with Binary Mixtures Differing in Size. **Drying Technology**, v. 33, p. 1746-1757, 2015. DOI: <u>https://doi.org/10.1080/07373937.2015.1036284</u>

SANTOS, K. G.; SANTANA, R. C. SOUZA, D. L.; MURATA, V. V.; BARROZO, M. A. S. Spouting behavior of binary particle mixtures of different densities: Fluid dynamics and particle segregation. **Particuology**, v. 42, p. 58-66, 2019.

SOUZA, C. R. F.; OLIVEIRA, W. P. Spouted bed drying of Bauhinia forficata Link extract: effect of the position of the feed atomizer and operating conditions on equipment performance and on product properties. In: **14th International Drying Symposium**, v. B, p. 1150-1157, 2004.

SOUZA, D. L.; OLIVEIRA, A. S.; FERRATO, F. S.; TIBOLA, F. L.; FREITAS, G.; RODRIGUES, I. F. et al. Desenvolvimento e construção de leito de jorro para recobrimento de partículas de ureia. **Revista Brasileira de Ciência, Tecnologia e Inovação**, v. 4, p. 32, 2019.

TSVIK et al. The Velocity for External Spouting in The Combined Process for Production of Granulated Fertilizers. **Uzbekskii Khimichesk**, v. 11, p. 50-69, 1967.

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