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Fluid Dynamic and Polymeric Coating of Forest Seeds of *Senna macranthera* (Collad.) Irwin et Barn in Conical Spouted Bed

Marina Pozitano[1]*, Sandra Cristina dos Santos Rocha[2]

[1,2] School of Chemical Engineering, Laboratory of Fluid Dynamics and Drying, University of Campinas-UNICAMP
Albert Einstein Avenue, 500, 13083-852, Campinas, State of São Paulo, Brazil
mpozitano@feq.unicamp.br

Senna macranthera (Collad.) Irwin et Barn trees have been used in urban forestry because of their small size and beauty and in reforestation, due to their rapid development. Aiming to preserve the seeds of *S. macranthera*, a polymeric film coating of the seeds is proposed in this work. To analyze the coating process a fluid dynamic study of the particles in a conical spouted bed was performed before the seeds covering in the same equipment. Fluid dynamic was analyzed with loads of 150, 250, 450, 750 and 1000 grams and, through the analysis of bed pressure drop versus gas flow curves, were obtained the parameters of maximum pressure drop, stable spout pressure drop and minimum spouting velocity. It was verified that for loads of 250 and 450 grams, the seeds showed a wide range of stable spouting regime with good circulation of particles resulting in good fluid-solid contact. As a result of the fluid dynamic analysis, the load of 450 grams was chosen to perform the coating process. Operating conditions were determined to obtain an adequate coverage for the seeds: atomization pressure of 115 kPa, flow rate of the coating suspension of 2ml/min and air inlet temperature of 70 °C. For these conditions, the efficiency of the process was 63 % and the average growth of seeds was 24.8 %.

1. Introduction

Seed coating has been used in species of vegetables, flowers, grass, fodder and some forest (Oliveira et al., 2003). Coating of this type of particle improve storage conditions, facilitates labor and use of agricultural implements, beyond presenting the advantage of enabling the joint use of nutrients, fungicides, insecticides, herbicides and beneficial microorganisms embedded in the coating suspension (Silva and Nakagawa, 1998 and Santos et al., 2000).

The physical characteristics of *S.macranthera* seeds, such as diameter and density indicate the spouted bed as a way of efficient gas-solid contact providing stable dynamic regime. The study of the coating process of *S.macranthera* seeds in spouted bed is an initiative to improve the conditions of storage, protection and preservation of the species. Thus, the objective of this work was to analyze the fluid dynamic of the seeds in conical spouted bed for five different loads of seeds and to define the

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formulation of a coating suspension and operating conditions in order to obtain a homogeneous and uniform coating, with good process efficiency.

1.1 Spouted bed equipment

The equipment used in the seeds coating process is illustrated in Figure 1.

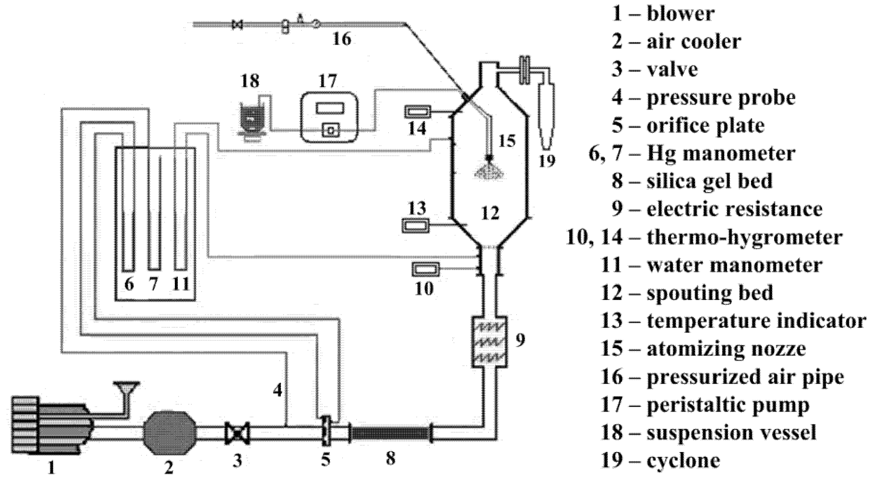

Figure 1: Spouted bed equipment.

The spouted bed (g) is built in acrylic with cone-cylindrical geometry and the column of the bed has a diameter of 20 cm and 50 cm high. Its conical base has a 45° angle with an internal height of 7 cm and an entrance hole diameter of 5 cm. In this equipment it is possible to fix the flow of the inlet air for the spout and the atomizing air pressure and also to control the temperature of the inlet air and the flow of the coating suspension. Air flow should be enough to launch particles upwards in a movement similar to a source, then returned to the peripheral region of the bed resulting in a cyclic movement of the particles inside the bed.

The transition from fixed to spouted bed can be verified by the curves of pressure drop versus fluid velocity. With increasing fluid flow, the pressure drop increases until the point of maximum pressure drop, ΔP_m, and at this point, the corresponding surface velocity is called U_m. For air velocities above this point, there is an increase in an internal spout height and a decrease in the bed pressure drop until installation of the spouted regime (at U_{jm}), when the pressure drop of the bed remains practically stable (ΔP_s) with increase air flow (Mathur and Epstein, 1974).

Among the applications of the spouted bed technology are drying and coating of chemicals, pharmaceuticals and agricultural products and the treatment of sludge wastes (San Jose et al., 2010a, b).

1.2 Senna macranthera

The *S. macranthera* (Collad.) Irwin et Barn. is a pioneer plant that belongs to the Leguminosae – Caesalpinioideae family, its seeds are smaller than 1 cm (Lorenzi, 1998) and according to the Geldart (1993) classification of particles, these seeds belong to group D, that characterizes particles of high density with good movement and solid-fluid contact in a spouted bed.

S. macranthera seeds have tegument dormancy which hinders the absorption of water and gases needed for germination (Carvalho and Nakagawa, 2000). The tegument dormancy can be broken by immersion of the seeds in concentrated sulfuric acid. Floriano (2004) recommends an immersion time in acid of 50 minutes while Santarem and Aquila (1995) suggest 15 minutes to break dormancy of this species.

2. Material and Methods

Fluid dynamic was analyzed with loads of 150, 250, 450, 750 and 1000 grams and, through the curves of bed pressure drop versus gas flow, the following parameters were obtained: maximum pressure drop (ΔP_m), stable spout pressure drop (ΔP_s) and minimum spouting velocity (U_{jm}). Experimental values of ΔP_m, ΔP_s and U_{jm}, were compared with theoretical values obtained using empirical equations of Pallai and Nemeth (1969) (ΔP_m and ΔP_s) and Mathur and Gishler (1965) (U_{jm}).

Formulation of the coating suspension was developed based on the Eudragit FS 30D® component. This polymer has good dissolution rate and absorption of water at neutral pH values, allowing the absorption of water by the seeds when sown. Considering 100 g of suspension, the following composition was developed: Eudragit FS 30D® – 30.6 g; talc – 4.6 g; triethyl citrate – 0.45 g; pink pigment – 0.15 g; magnesium stearate – 2.25 g; titanium dioxide – 1.35 g and water – 60.60 g.

Due to the tegument dormancy, the seeds were chemically scarified by immersion in concentrated sulfuric acid for 20 min before coating. Then they were washed in water and dried in the spouted bed until the establishment of particles circulation.

Atomization pressure (P_{at}), flow of the suspension (Q_{susp}) and air inlet temperature (T_{ar}) were adjusted to avoid excessive elutriation of the coating suspension and seeds, keeping the spouting stable.

Experimental growth of particles (δ_{exp}) was determined by difference between the mass of coated seeds (m_{rec}) and initial mass (m_0), divided by its initial mass (m_0). The theoretical growth (δ_t) was obtained by Equation 1, where: ρ_{susp} is the density of the suspension; CS is the solids concentration in suspension and t is the total time of atomization.

$$\delta_t = \frac{Q_{susp} \cdot \rho_{susp} \cdot CS \cdot t}{m_0} \qquad (1)$$

Finally, the process efficiency was determined as the ratio of the experimental to the theoretical growth.

3. Results and Discussion

The comparison between experimental and theoretical fluid dynamic parameters is shown in Table 1.

Table 1: Experimental and calculated empirical values for U_{jm}, ΔP_m and ΔP_s

Loads (g)		150	250	450	750	1000
Bed height fixed (cm)		2.6	3.5	4.5	6.7	7
U_{jm} (m/s)	theoretical	0.25	0.29	0.33	0.40	0.41
	experimental	0.10	0.16	0.23	0.27	0.28
	relative error	153.2	87.48	43.0	51.4	49.3
ΔP_m (Pa)	theoretical	295.9	384.2	538.1	564.5	711.8
	experimental	336.7	392.3	454.5	847.0	1017.9
	relative error	12.1	2.1	18.4	33.4	30.1
ΔP_s (Pa)	theoretical	224.9	292.0	409.0	429.1	541.0
	experimental	176.4	255. 7	339.2	467.2	334.0
	relative error	27.5	14.2	20.6	8.2	62.0

The reading of the experimental value of U_{jm} is made in the fluid dynamic curve at the point where the spouting regime collapses but, due to the vagueness and the difficulty in visualizing this point in the graphs obtained, U_{jm} presented high relative errors. Moreover, the three parameters analyzed are related with the geometry of the bed and particles characteristics and, the relative errors were also due to the seeds ample size distribution having diameters varying from 2.2 mm to 4.8 mm. And, the flattened and non-circular shape of seeds, difficulted the movement of the particles favoring the elevation of the relative errors, since the empirical relations were determined for spherical shaped particles of small range of sizes.

Among all the masses analyzed, fluid dynamic graph for 450 grams seeds showed the profile more similar to those found in the literature for stable spouted beds (Figure 2).

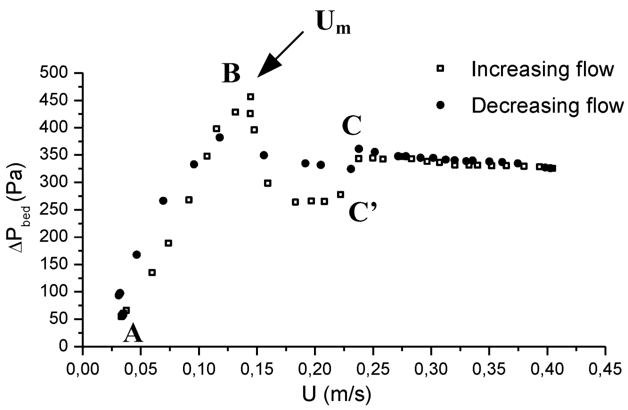

Figure 2: Fluid dynamic curve for 450 grams of seeds.

The graph shows a good range of stable spout velocities, demonstrating that the load 450 grams is suitable for coating the seeds of *S. macranthera* under stable spout regime (above points C).
In the trajectory from A to B , the particles remained in fixed bed with the gas passing between the bed empty spaces. The point B, at the apex, corresponds to ΔP_m (454.5 Pa) and its corresponding superficial velocity U_m (0.14 m/s). Movement of stable spouting began in C, and from this point, an almost stable pressure drop is observed (ΔP_s 339.2 Pa). Point C' corresponds to U_{jm} (0.23 m/s) and it is obtained for the decreasing flow curve where a stable pressure drop is no more observed (spout collapse).
Preliminary experiments of coating showed that the best values of P_{at}, T_{ar} and Q_{susp} for a better maintenance of the movement of particles in the spouted bed were 115 kPa, 70 ºC e 2 ml/min, respectively. Spouting air temperatures above 70 ºC caused elutriation of the coating suspension and atomization pressures above 115 kPa damaged the movement of stable spouting.
The physical characterization of the coating suspension resulted in CS equal to 0.2 kg/kg and ρ_{susp} of 1032.8 ± 0.004 kg/m³. The total time of suspension atomization to obtain a homogeneous film with complete coverage of the seeds was 382 minutes. Experimental and theoretical growth and process efficiency obtained for the operating conditions above especified were 24.8 %, 39.4 % and 63 %, respectively.
Images of the surface and radial cut of the seeds in natura, scarified and coated were captured in a scanning electron microscope (SEM), Leo 440i model, Oxford, England (Figure 3).

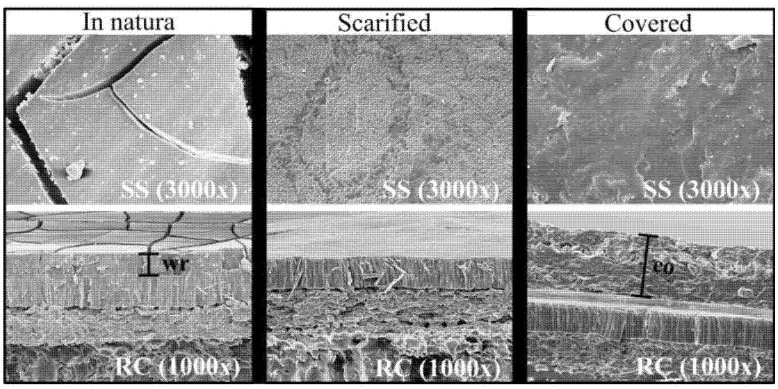

Figure 3: SEM Micrographs of the seeds in natura, scarified and covered (SS - seeds surface; RC - radial cut; wr - wax removed in the chemical scarification; co - coating layer).

In Figure 3, the images of the surface were captured with optical zoom of 3000 times while the images of the radial cut were extended in 1000 times. It is possible to verify the formation of a homogeneous coating film around the seeds and that film thickness was even greater than the thickness of wax removed. Therefore, it is expected that the coating film provides greater protection to the seeds, which is being tested by storage analyses.

4. Conclusions

The fluid dynamic curve for a load of 450 grams of seeds had a profile typical of a spouted bed with a wide range of stable spout regime.

The spouted bed showed to be adequate for coating of the seeds of *Senna macranthera*, resulting in relative growth of around 25 %. Taken into account the lack of information and scarcity of the product, the process efficiency of 63 % was considered satisfactory.

SEM Micrographs showed that 20 minutes of immersion in sulfuric acid were sufficient to remove the wax layer of the seed and for the coated seeds, homogeneous coating was observed.

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