Refereed Proceedings

The 12th International Conference on Fluidization - New Horizons in Fluidization Engineering

Engineering Conferences International *Year* 2007

Experimental Validation of Macro- and Micro-Level Scaling Laws in Small- and Medium-Scale Top-Spray Fluidised Bed Coaters

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EXPERIMENTAL VALIDATION OF MACRO- AND MICRO-LEVEL SCALING LAWS IN SMALL- AND MEDIUM-SCALE TOP-SPRAY FLUIDISED BED COATERS

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ABSTRACT

Top-spray fluid bed coating experiments of 500 g sodium sulphate cores (180 – 300 µm) have been conducted in a small-scale single-spray nozzle GEA Strea-1 fluid bed. The process has been scaled-up to a bed load of 4 kilos in a single-spray nozzle Niro MP-1 fluid bed testing different scaling laws for comparison. Results indicate clearly that up-scaling using a drying force parameter (combining bed temperature with bed relative humidity) as well as a droplet related relative parameter is a promising alternative to common fluid bed scaling principles.

INTRODUCTION

In the production of solid enzyme products, coating of the enzyme formulation onto inactive filler cores in fluid beds is a common choice. The desired product is thereby a product consisting of unagglomerated individual carrier particles each coated homogeneously with a layer of enzyme. If formulation or process conditions are incorrectly chosen either excessive agglomeration or excessive spray drying of the feed will happen. In both cases a poor product quality is achieved. Often product and process properties are optimised in small- and medium-scale fluid beds and then transferred to large production-scale. The scale-up of a fluid bed granulation process requires decisions to be made at many levels. Scaling decisions must be closely related to a large number of parameters including: fixed parameters, parameters related to the starting material and the type of fluid bed, input parameters, operating conditions including spraying and mixing conditions as well as processing time etc. With such a variety of interlinked parameters and properties combined with a general lack of fundamental understanding of the granulation process, it is obvious that the scaling of a fluid bed granulation process is a difficult task.

Currently, scaling is still more of an art rather than science being a mix of physics, mathematics, experience, common sense and qualified guesses. Previously suggested scaling laws, adapted from the fluid bed combustion theory, focus on hydrodynamic similarity across scale as suggested e.g. by Glicksmann et al. (1993) and Horio et al. (1986). Although validated by the before mentioned authors and others in fluid catalytic cracking regenerators and gas-liquid bubbling fluid beds,

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these scaling principles may however not readily be used with fluid bed coating experiments due the presence of the liquid phase in the fluid bed coating systems. Instead other scaling principles have been proposed, as reviewed by Hede (2006), although little experimental validation of these scaling principles has been reported in literature so far.

The present paper seeks to test selected previously suggested scaling principles by providing experimental evidence for the ranking of different scaling principles and parameters. Coating experiments are performed in small-scale top-spray fluid bed (GEA Aeromatic-Fielder Strea-1) and the resulting granule product properties are compared to coated granules produced in a medium-scale fluid bed (Niro MP1). Based on experimental data, a number of the most promising scaling principles are analysed, validated and ranked according to their relevance and applicability.

EXPERIMENTAL AND MATERIALS

Fluid beds in two different scales were used for scale-up experiments. The aim was to conduct a number of well-defined fluid bed coating experiments in the small scale bed and then try to scale-up the process with respect to the particle size fractions and agglomeration tendency of sodium sulphate cores coated with aqueous solutions of crushed sodium sulphate using dextrin as binder material.

In the effort to fix as many parameters as possible across scale, the two fluid bed set-ups were carefully build up as similar as possible. Both were top-sprayed fluid beds each having a single two-fluid nozzle. In both cases, an outlet nozzle diameter of 1.2 mm was used. Likewise was the coating liquid feed in each case led from the external heated reservoir at a constant heating temperature of 60 °C to the nozzle through an adjustable electrical peristaltic tube pump. The fluidisation velocity in m/s was likewise identical across scale. A sketch of the general set-up in each of the three fluid bed systems may be seen from figure 1.

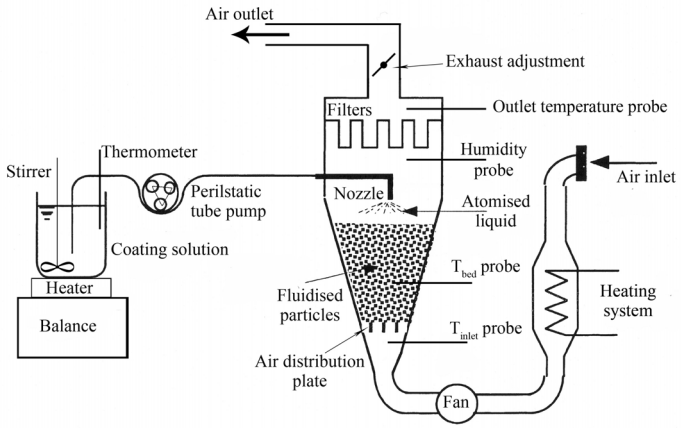

Figure 1: Sketch of the general top-spray set-up used in both fluid bed scales (Hede, 2005).

The small-scale fluid bed was a modified GEA Aeromatic-Fielder Strea-1 top-spray fluidised bed (see figure 2) with a stainless steel fluidising chamber of 12 L allowing a particle bed load of 500 g to be fluidised. Modification to the original vessel design made it possible to insert a digital thermometer into the fluidisation chamber wall

thereby being able to measure the bed temperature between the bottom air distribution plate and the nozzle outlet. In addition, an external humidity measurement apparatus (Testo 645 Thermohygrometer) was inserted above the nozzle allowing the relative humidity in the fluidisation chamber to be measured. Custom-made reusable stainless steel filters were inserted in the top of the fluidisation chamber in order to prevent particles and spray-dried coating droplets from being exhausted. The back-flush option in the Strea-1 set-up was switched on allowing the filters to be flushed every five seconds.

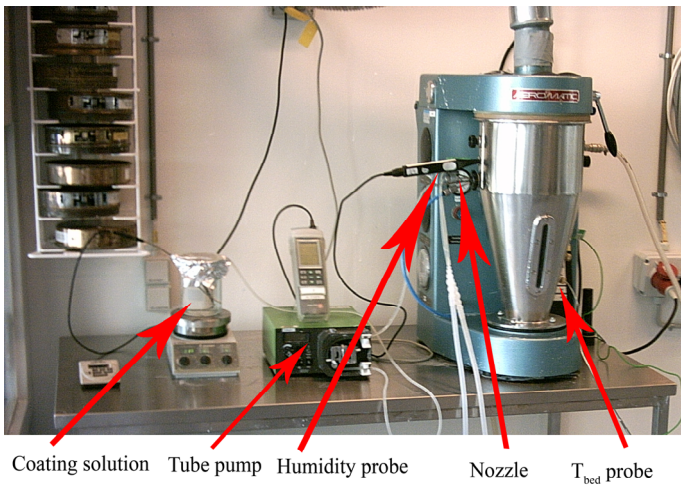

Figure 2: Picture of the small-scale Strea-1 set-up (Hede, 2005).

As medium-scale top-spray fluid bed a standard Niro-Aeromatic Multiprocessor type MP-1 was used with a stainless steel fluidising chamber of 16 L allowing a particle bed load of 4000 g, thereby being eight times larger in capacity in respect to the Strea-1 set-up. A picture of the MP-1 set-up can be seen in figure 3. As for the Strea-1 set-up, it was possible to measure the bed temperature between the bottom air distribution plate and the nozzle outlet. The same external humidity measurement apparatus (Testo 645 Thermohygrometer) was inserted above the nozzle allowing the relative humidity in the fluidisation chamber to be measured at a location identical to the Strea-1 set-up. The back-flush option in the MP-1 set-up was switched on allowing the filters to be flushed every five seconds exactly as it was the choice in the small-scale set-up.

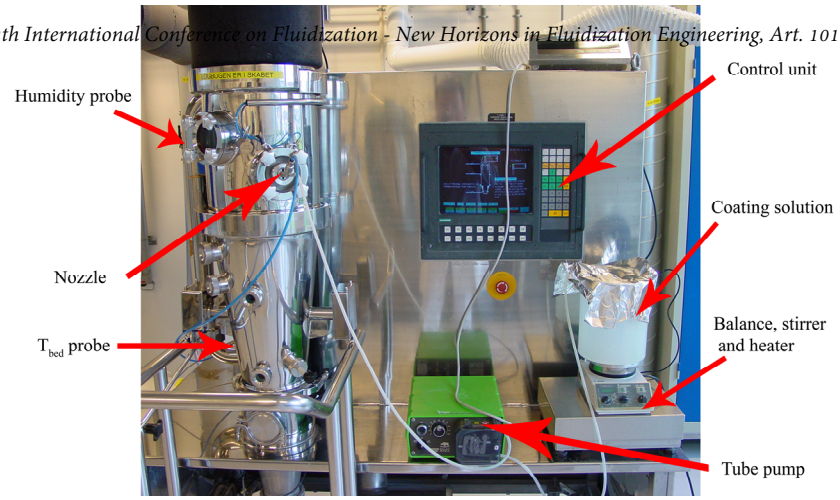

Figure 3: Picture of the medium-scale MP-1 set-up.

Prior to coating, the sodium sulphate (180 – 300 µm) core bed load of 500 g for the Strea-1 case and 4000 g for the MP-1 case was heated until the relative humidity inside the fluidisation chamber was constant, typically ranging from 5 RH% to 8 RH% depending on weather conditions. During coating, the fluidisation velocity and fluidisation inlet air temperature were kept fixed. In addition, the bed temperature was maintained at the chosen level by adjusting only the inlet temperature of the fluidisation air. In each coating operation the aim was to coat until the bed load had increased 20 w/w%. This was done in order to make sure that a reasonable coating layer (~ 10 µm) had developed. After coating, the bed load was kept fluidised at identical fluidisation velocity and temperature conditions in order to dry the coated granules. This was done until the relative humidity inside the chamber was identical to the conditions prior to coating.

For the sieving analysis, a Retsch Sieve Shaker AS 200 control was used with a sieve stack consisting of sieves with orifices diameters of 180 µm, 212 µm, 250 µm, 300 µm, 355 µm, 425 µm, 500 µm and 600 µm. A sample of 100 g from each batch was sieved for 3 minutes with amplitude of 1.1 and the weight of each fraction was determined with the AS 200 control interface using a connected Mettler balance.

It was verified by microscope analysis of the different fractions that coated granules with diameters above 425 µm consisted of agglomerates whereas particles below this limit were primarily single coated granules. Thus, this sieve orifice diameter was set as the agglomeration limit and based on the weight of each of the fractions, an agglomeration percentage was determined for each batch according to equation 1.

$$Agglomeration\,\% = \frac{\text{coated batch weight fraction with } d_p > 425\,\mu\text{m}}{\text{total coated batch weight}} \cdot 100\,\% \qquad (1)$$

SCALING PARAMETERS TESTED

The criteria for successful scaling in the present case were to be able to repeat the level of agglomeration as well as match the particle size fractions across scale. A

number of scaling parameters and scaling laws expected to be capable of successful scaling were tested including the flux number proposed by Akkermans et al. (1998):

$$FN_m = \log\left[\frac{\rho_p \cdot U_e}{\dot{q}_{mliq}}\right] \tag{2}$$

where U_e is the excess velocity of fluidising gas, ρ_p is the particle density and $\dot{q}_{mliq}$ the is the spray mass flux being the mass flow of binder liquid divided by the spray area.

Further, the so-called relative droplet size $d_{d,rel}$, proposed by Rambali et al. (2003), was tested:

$$d_{d,rel} \sim \frac{\dot{m}_{spray}}{(\dot{m}_{nozzle\ air})^2} \tag{3}$$

which is the ratio of the nozzle spray rate $\dot{m}_{spray}$ divided by the airflow through the nozzle $\dot{m}_{nozzle\ air}$ squared.

A third parameter was tested which is the so-called *Drying Force* proposed by Hede (2005). The drying rate of the coated granules can be estimated by calculation of the Drying Force at steady state coating conditions according to the following equation:

$$\text{Drying Force} = P_{sat}\big|_{T_{bed},100\%rH} - P_{actual} \tag{4}$$

where P_{sat} is the saturated pressure at the dry bulb temperature and P_{actual} is the actual vapour pressure of the fluidisation air at the bed temperature and the bed relative humidity given as:

$$P_{actual} = \frac{\text{Bed rH\%}}{100\ \text{rH\%}} \cdot P_{sat}\big|_{T_{bed}} \tag{5}$$

RESULTS AND DISCUSSION

Results from scaling attempts with the Akkermans flux number were unsuccessful as there was neither any significant unambiguous dependence of the agglomeration tendency on the flux number nor any adequate reproduction of the particle size fractions across scale with fixed values of the flux number.

Results from experiments where the scaling parameters were the relative droplet diameter and the drying force are somewhat promising. As indicated from figure 4, showing the particle size fractions in a Rosin-Rammler plot, it may be seen how well the particle size fractions are matched across scale. This was a general trend for different tested values of the drying force and relative droplet size.

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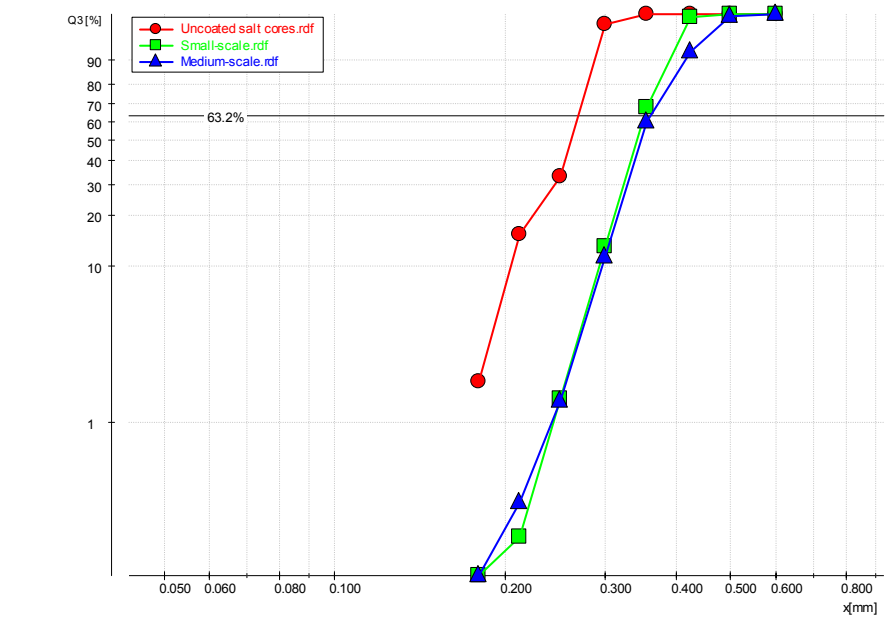

Figure 4: Example of a typical Rosin-Rammler plot indicating how well the particle size fractions were reproduced from small- to medium-scale in terms of fixed values of relative droplet size and drying force.

Keeping the drying force and the relative droplet size values constant, when transferring the coating process into the larger MP-1 scale, shows in addition to the match of size fractions remarkably good repetition of the agglomeration tendency as well as observed from four scaling attempts in figure 5. Matching the particle size fractions as well as the agglomeration tendency is not necessarily linked and the successful scaling in terms in both properties is indeed a significant achievement. Although agglomeration tendency is not the only quality parameter involved in successful scaling of fluid bed granulation processes it is a quite important parameter as agglomeration is often a significant problem in large scale fluid beds. Furthermore, previous results by e.g. Hede (2005) have indicated that optimum coating conditions leading to homogenously coated granules are close to the conditions where the particle bed starts to agglomerate. Hence it is reasonable to believe that if the tendency of agglomeration can be maintained across scale there is a good chance that other vital granules properties will be maintained as well. Further studies will have to be conducted in order to verify this but initial SEM pictures of the coated granules showed identical coating layer structure – both on the surface and in-depth - when fixing values of the relative droplet size as well as drying force.

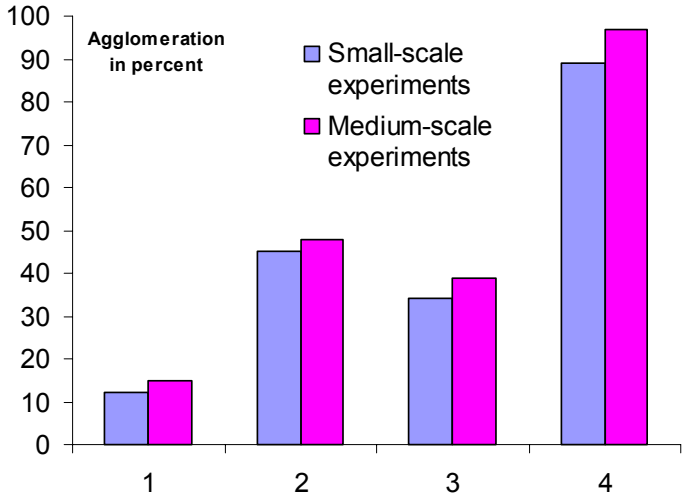

Figure 5: Comparison of agglomeration tendencies among batches produced in small-scale and in medium-scale fluid beds under different (but pair-wise identical) values of drying force and relative droplet size.

CONCLUSION

In the present paper first steps have been taken towards the testing and validation of modern scaling principles. Experimental results indicate that successful scaling may be done in terms of the relative droplet size parameter and the drying force parameter thereby having one parameter associated with the spray properties and one parameter associated with the thermodynamics in the bed during steady state coating. The scaling criteria in the present studies are agglomeration tendency during coating and matching of the particle size fractions but it is reasonable to believe that successful scaling of the agglomeration tendency is directly linked to the successful scaling of other important granule properties, as successful coating operations are known to be close to the conditions where the particle bed agglomerate. The present experimental validation of scaling laws for the scale-up of fluid bed granulators and coaters may be seen as an important first step towards the development of models and principles capable of scaling fluidised bed coating processes across scale with focus on process conditions as well as particle properties.

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