Granulation Time in Fluidized Bed Granulators

Papiya Roy[1], Rajesh Khanna[1], Duvvuri Subbarao[2]*
Department of Chemical Engineering
[1] Indian Institute of Technology Delhi, New Delhi, 110016, India
[2] Universiti Teknologi PETRONAS, 31750,Tronoh, Perak, Malaysia

Abstract

Size enlargement of fine powders by wet granulation involves mixing of fine particles with a binder liquid to form larger wet granules and drying them by evaporation of the binder liquid to form larger dry granules. Identification of the time for completion of granulation process is critical as further mixing of dry granules is providing extra energy for their attrition. In a batch fluidized bed granulator, monitoring the bed pressure drop and bed temperature with time can provide information on time for completion of the granulation process. Experimental observations on granulation time and granulation size for wet granulation of urea powder in a lab-scale batch fluidized bed granulator are presented.

1. Introduction

Granulation is a process of forming coarser particles by combining finer particles. Its purpose is to improve the flow properties of particles, to modify the bulk densities and to control the dissolution rates *etc*. It is widely used in pharmaceutical, food, agrochemical, dyestuffs industries. In wet granulation process, feed particles and a liquid containing solute (binder) are well mixed to form wet granules; the liquid is then evaporated to bind feed particles together to form dry granules. Equipments such as rotary drums, rotating helical ribbons, anchor agitators with mechanical agitation or fluidized beds can be used for wet particle mixing as well as drying of wet granules. Particles in a gas-solid fluidized bed are in a state of good mixing due to the gas flowing through the fluidized bed of particles in the form of bubbles. Spraying a binder solution on to particles in a fluidized state can provide uniform wetting of the particles to generate wet granules. Also fluidized beds offer higher drying rates while maintaining bed isothermality. Process variables that affect fluidized bed granulation include liquid volume fraction, operating gas temperature and gas velocity [1—5]. Liquid volume fraction has to be within an acceptable range for successful granulation [6, 7]. Excess liquid volume fraction can lead to exponential growth of size of wet granules and eventual defluidization. Lesser liquid volume fraction can lead to poor granulation. Operating gas temperature and velocity define the fluidization behavior and rate of drying of wet granules. Identification of the endpoint of drying and hence granulation process is critical. Continuing fluidization process beyond the endpoint of granulation can cause attrition and breakage of dry product granules. Techniques such as sound and vibration analysis [3,8-10] growth kinetics of the particles [11-12] have been investigated for the detection of endpoint of granulation. Present study is an attempt to explore the possibility of utilizing the bed pressure drop and bed temperature for identifying the endpoint of granulation and study the effect of operating variables on the time of granulation and size of granules of urea powder as a model system.

2. Experimental study

Figure 1 presents a schematic diagram of the experimental setup which has been used in the present study.

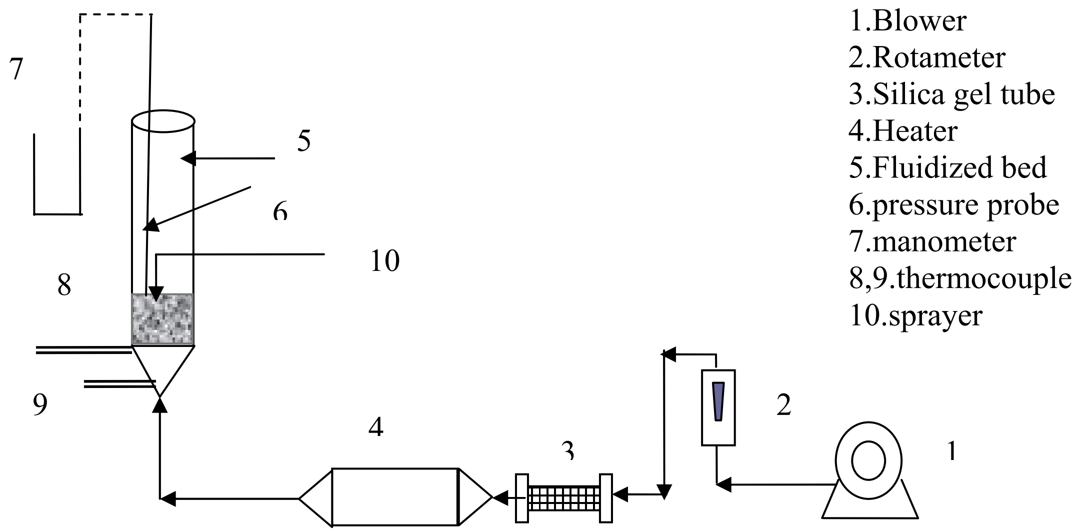

Figure 1: Schematic diagram of the experimental set up

Air flow from the blower (1) is metered (2), dried over silica gel (3) and heated to the required temperature as it flows over an electrical heater assembly (4) before it enters the fluidized bed granulator (5). The fluidized bed granulator is a 2.54 cm diameter glass column of height 15 cm fitted with a porous plate distributor. A pressure probe (6) is connected to water manometer (7) to measure bed pressure drop. Two thermocouples (8) and (9) are provided to monitor temperature of inlet gas and fluidized bed respectfully. Graded urea particles were used for fluidization and granulation experiments. A sprayer (10) is used to spray a predetermined quantity of the binder liquid (saturated solution of urea) on to the bed of particles from the top over a short period. Experiments were performed at various combinations of the following operating parameters

- Initial particle size - 327.5, 390 and 512.5 micron
- Operating velocity - 65.8, 98.8 and 131.7 cm/s
- Inlet air temperature - 30°C, 37°C and 50° C
- Amount of binder - 8.7%, 15.8% and 26.8%

(100*weight of solution /weight of bed particles)

Urea particles of a given cut size were fluidized with hot inlet air. After the bed got heated to the inlet air temperature, binder solution was sprayed onto the top of the bed. Binder solution was added in small quantities at a time for a short period. (There is an upper limit to addition of binder solution per weight of bed at which the bed can get defluidized and was experimentally observed to be around 30.5%). As liquid and the bed particles get well mixed, wet granules form and grow while getting dried. Consequently, inlet air velocity was chosen sufficiently high to keep the bed of wet granules fluidized.

3. Identification of End point of granulation

Addition of binder solution at the start of granulation process increases the bed weight and corresponding bed pressure drop. As liquid evaporates due to drying, bed pressure drop decreases and bed temperature reduces to approach wet bulb temperature. Towards completion of liquid evaporation by drying and granulation, bed pressure drop reduces to the initial value. Bed temperature reduces to a minimum and then increases it equals the inlet gas temperature. Typical bed pressure drop and temperature traces with time during the period of granulation are shown in fig. 2a and fig. 2b for the experiment performed at a gas velocity of 131.7 cm/s with initial particle size of 327.5 micron, inlet gas temperature of 50 ^oC, moisture content of 8.7% . From fig.2a on bed pressure drop, it can be seen granulation was complete in 20 minutes.

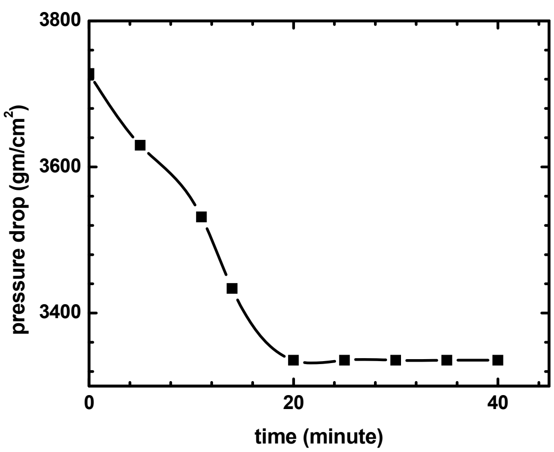

Figure 2a: Pressure drop as a function of time. The parameters are initial particle size: 327.5 micron, operating gas velocity: 131.7cm/s, moisture content: 8.7% and inlet gas temperature: 50°C.

From fig.2b on bed temperature with time, temperature reached a minimum at around 10 minutes and then increased to equal the inlet temperature by 40 minutes. At a time of 20 minutes (corresponding to the completion of granulation time recognized from bed pressure drop measurements), an inflection point can be recognized on the rising section of temperature trace. Beyond the inflection point, evaporation of moisture from the bed is negligible and bed temperature increased due to heating by incoming gas.

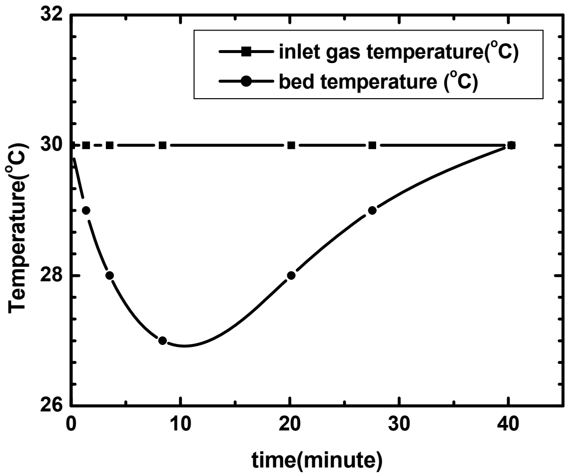

Figure 2b: Bed temperature as a function of time. The parameters are initial particle size: 327.5 micron, operating gas velocity: 131.7cm/s, moisture content: 8.7% and inlet gas temperature: 50°C.

The initial decrease in the bed temperature compared to inlet gas temperature is due to rapid evaporative cooling. As the fraction of moisture in the bed decreases, the effect of evaporative cooling diminishes and the bed temperature rises to reach the inlet gas temperature due to heating by the incoming gas. Slope of the temperature change with time increases during the period of evaporative cooling followed by a decrease during the period of heating. The transition can be identified by locating the inflection point (dT/dt is maximum or d^2T/dt^2 is zero). In the present work, end of granulation process is identified by these two techniques.

Granulation Time:

Typical experimental observations on the effect of moisture content, gas velocity and inlet gas temperature on time of granulation are presented in Fig.3 a,b,c.

Fig. 3a presents the effect of moisture content on granulation time at an operating velocity of 65.8 cm/s and inlet air temperature of 30°C with initial particle size as a parameter. Granulation times are more for higher moisture contents. It is interesting to note that granulation of smaller particles need longer granulation times. Fig. 3b shows the effect of operating gas velocity on granulation time with moisture content as a parameter and inlet gas temperature of 30°C. Higher operating gas velocity will lead to faster evaporation and lesser granulation time. Fig. 3c shows the effect of inlet gas temperature on granulation time at 8.7% moisture content and gas velocity of 65.8 cm/s with initial particle size as a parameter. At higher inlet gas temperatures, moisture evaporates at a higher rate and hence, the time required for granulation is less.

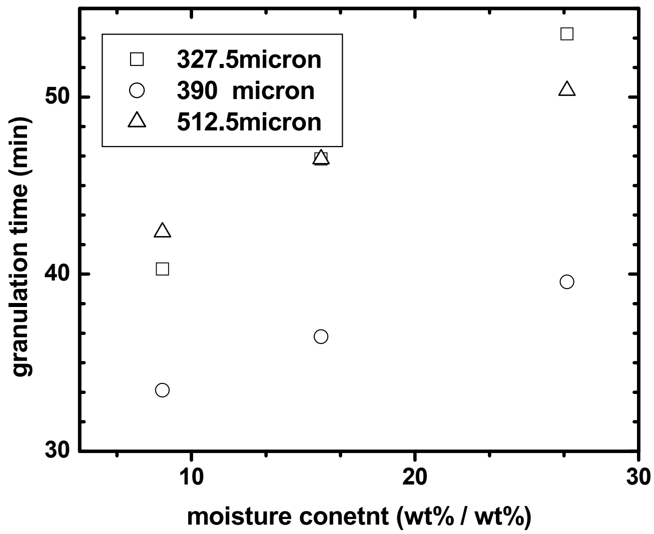

Figure 3.a: Effect of moisture content on granulation time for different initial particle sizes. The parameters are operating gas velocity: 65.8cm/s and inlet air temperature 30°C.

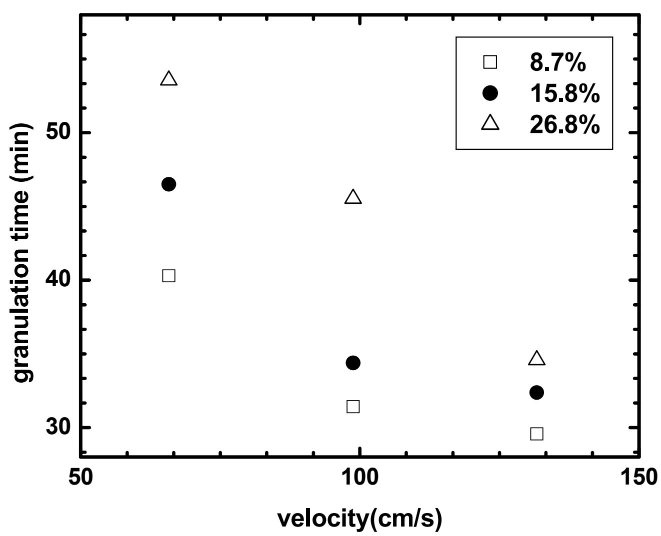

Figure 3.b: Effect of operating gas velocity on granulation time for different moisture contents. The parameters are initial particle size 327.5 micron and inlet air temperature 30°C.

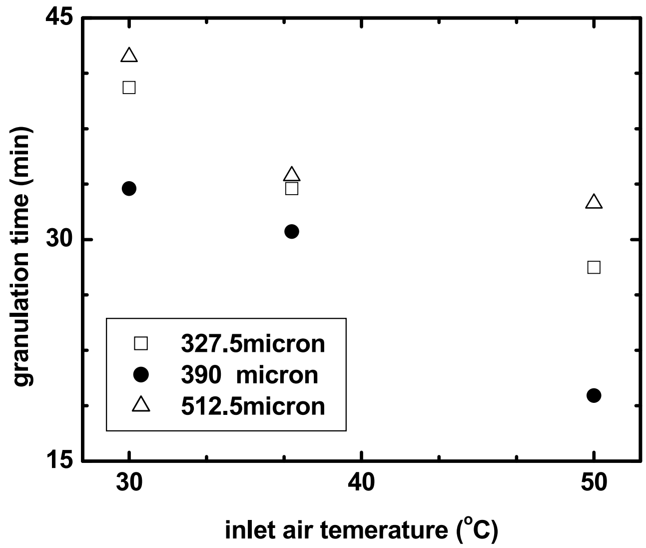

Figure 3.c: Effect of inlet gas temperature on granulation time for different initial particle sizes. The parameters are operating gas velocity 65.8cm/s and moisture content 8.7%.

Model for granulation time:

Wet granulation process involves mixing of liquid binder with particles to form wet granules and their drying to form dry granules. Drying step is much slower than mixing step and, hence time required for granulation is essentially time needed for drying. Drying rate depends on rate of heat transfer between gas and granules. Due to good granule mixing, temperature of bulk of the bed is uniform and heat transfer from gas to granules takes place over a thin layer ΔX very near the grid. Then, time for granulation can be expressed as

$$t_g \cong t_d = \frac{\text{Heat required to evaporate Moisture Content in the bed}}{\text{Heat Transfer from gas to bed}}$$

$$= \sum_{t_d} \left[\frac{\Delta\left(\frac{MC}{100}\right) AL(1-\varepsilon)\rho_p \lambda}{A u_o \rho_g c_{pg} \left(T_{gi} - T_b\right)} \right] \quad \textbf{(1)}$$

Temperature difference between inlet gas temperature and the bed increases from zero initially and then decreases to zero. To estimate time for drying this equation needs to be integrated over small time intervals with appropriate temperature difference. As a first guess, it is proposed that this equation be approximated as

$$t_g \simeq \frac{\left(\frac{MC}{100}\right) AL(1-\varepsilon)\rho_p \lambda}{Au_o \rho_g c_{pg}\left(T_{gi} - \frac{T_{b\min}}{2}\right)} \qquad \textbf{(2)}$$

As a first approximation, data on time of granulation is presented as a function of the ratio of moisture content to gas velocity in fig.4.

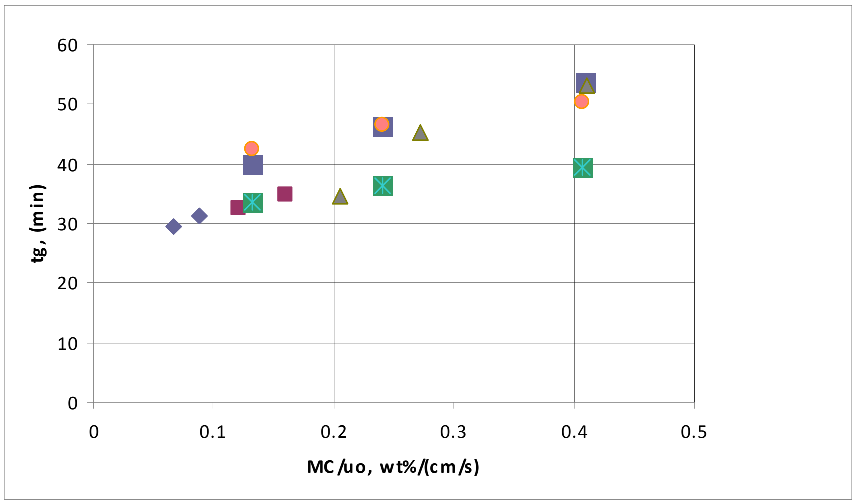

Fig.4. Correlation for time of granulation as a function of the ratio of moisture content to gas velocity.

This model needs to be improved considering the heat transfer process between the gas and the granules to explain the effect of initial particles size on granulation time.

4. Size of Granules

Figure 4 presents the variation of the ratio of the average final granule size to the average initial particle size as a function of operating parameters.

Fig. 4a presents the effect of initial particle size on the ratio of granule size to initial particle size at an operating velocity of 65.8 cm/s and inlet air temperature of 30^{o}C with moisture content as a parameter. The ratio increases with moisture content for all the particle sizes. The ratio is lower for larger initial particle size.

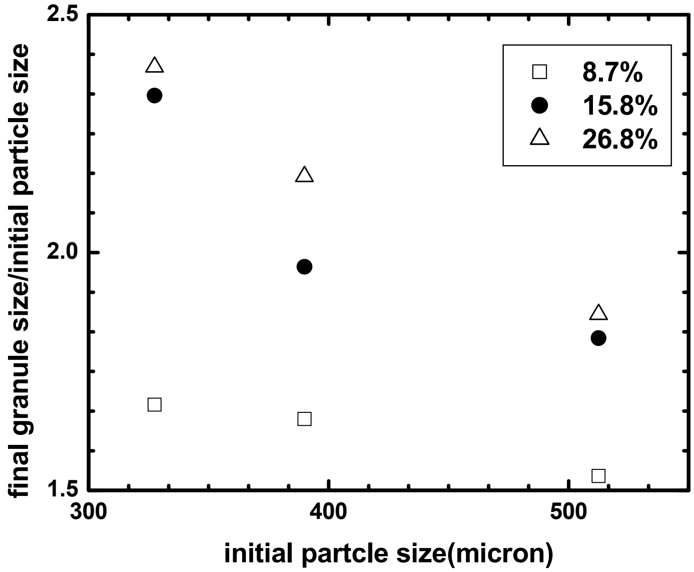

Figure 4a: Effect of initial particle size on the ratio of average final granule size and initial particle size. The parameters are operating velocity: 65.8cm/s and inlet air temperature: 30^{o}C.

Fig. 4b shows the effect of operating gas velocity on the ratio at 8.7% moisture content and inlet gas temperature of 30^{o}C with initial particle size as a parameter. Higher operating gas velocity resulted in lower ratio of granule size to initial particle size. The ratio is higher for smaller initial particles.

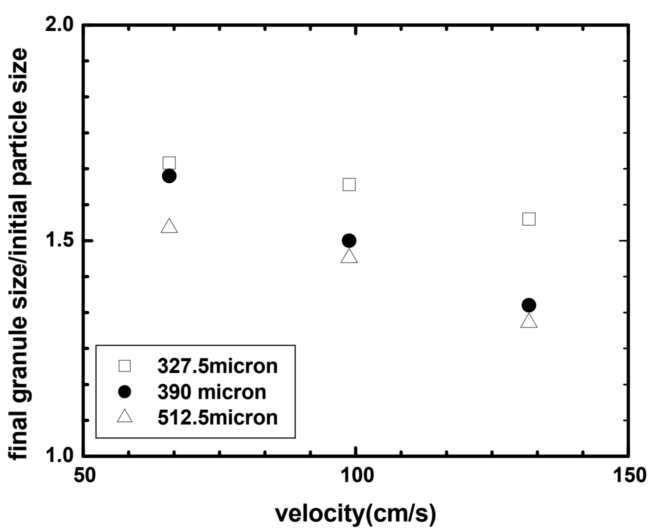

Figure 4b: Effect of operating velocity on the ratio of average final granule size and initial particle size. The parameters are inlet gas temperature: 30 °C and moisture content 8.7%.

Fig. 4c shows the effect of inlet gas temperature on the ratio for moisture content of 26.8% and initial particle size of 327.5 microns with gas velocity as a parameter. The ratio increases with increase in inlet gas temperature.

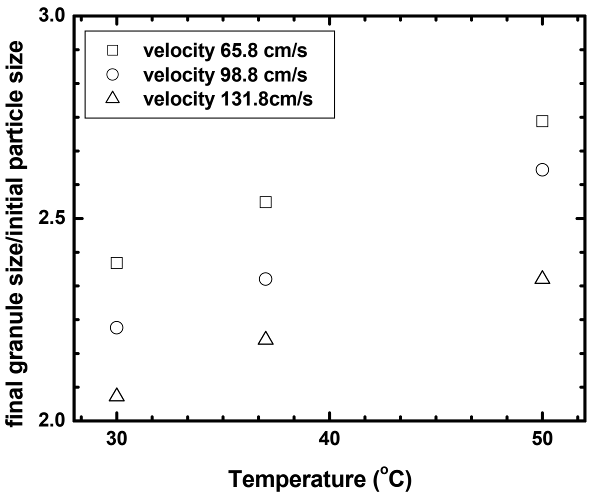

Figure 4c: Effect of inlet gas temperature on the ratio of average final granule size and initial particle size. The parameters are initial particle size = 327.5 micron and moisture content 26.8%.

These observations are in line with the bulk of information in the literature.

Model for size of Granules:

Wet granules form due to particle mixing with binder solution. The particles are held together by cohesive capillary forces due to the presence of liquid in between the particles. In fluidized bed granulators, the wet granules are suspended by gas drag force against gravitational force. The resulting shear determines the size of wet granules. Hydraulic capillary force which keeps the granule cohesive is

$$F_{ca} = \pi D_h \sigma \frac{MC}{100} \qquad \textbf{(3)}$$

Diameter of capillaries in the granule, considering that a granule is similar to a packed bed, can be obtained as

$$D_h = 4\frac{Volume\ of\ voids\ in\ the\ granule}{surface\ area\ of\ particles\ in\ the\ granule} = 4\frac{\pi D_{pi}^{\ 3}}{6}\frac{\varepsilon_g}{\left(1-\varepsilon_g\right)}\frac{1}{\pi D_{pi}^{\ 2}}$$
$$= \frac{2}{3}\frac{\varepsilon_g}{\left(1-\varepsilon_g\right)}D_{pi} \qquad \textbf{(4)}$$

This capillary force needs to be in equilibrium with drag force because of gas flow through the bed

$$\frac{2}{3}\frac{\varepsilon_g}{\left(1-\varepsilon_g\right)}\pi D_{pi}\sigma\frac{MC}{100} = c_D\frac{\pi D_g^{\ 2}}{4}\frac{\rho u_o^{\ 2}}{2\varepsilon^2} \qquad \textbf{(5)}$$

From this ratio of wet granule size to initial fine particle size can be obtained as

$$\frac{D_g}{D_{pi}} = \left(\frac{16}{3c_D}\frac{\varepsilon_g}{1-\varepsilon_g}\right)^{1/2}\left[\frac{\sigma\left(MC/100\right)\varepsilon^2}{D_{pi}\,\rho u_o^{\,2}}\right]^{1/2} \qquad \textbf{(6)}$$

Voidage in the granules ε_g may be assumed to be around 0.5. Inter granular movement and wall friction can cause attrition and breakup. These are also manifestations of drag force exerted by gas on granules. Also, for zero moisture content, the ratio has to reduce to a value of one. In view of this eq.(6) can be adopted as

$$\frac{D_g}{D_{pi}} = 1 + k_{gs}\left[\frac{\sigma\left(MC/100\right)}{D_{pi}\,\rho u_o^{\,2}}\right]^{1/2} \qquad \textbf{(7)}$$

where k_{gs} is granular size constant.

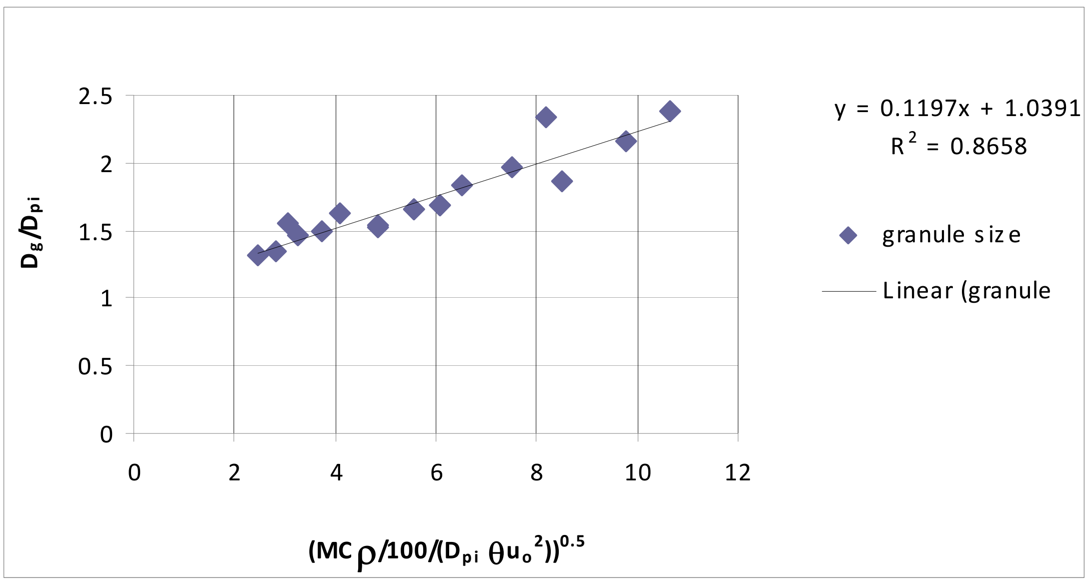

Fig.5 Comparison of the model equation (7) with experimental data.

From a comparison of this equation with experimental data as shown in the fig.5, k_{gs} is obtained as 0.1197. These are preliminary results and need to be further evaluated with a wider data base..

Conclusions.

1. Identification of the endpoint of granulation is necessary to discontinue further fluidization to avoid breakage due to attrition of the dry granules.
2. An easy method to measure the endpoint of wet granulation in fluidized beds is presented based on the bed pressure drop and temperature measurement as a function of time.
3. Bed pressure drop decreases with time to a limiting value as the liquid used for wet granulation evaporates.
4. During the wet granulation process. bed temperature decreases with time in the beginning and increases back as the evaporation of liquid is complete. The

inflexion point on the temperature- time trace in the rising temperature section corresponds to the end of evaporation and time for granulation.

5. Granulation time increases with increase in moisture content of the binder solution, decreases with increase in operating velocity, decreases with increase in inlet temperature of the fluidizing air.

6. A model is presented to estimate ratio of granule size to initial particle size and the ratio is observed to be a function of Weber Number

Acknowledgements

One of the authors (Duvvuri Subbarao) wishes to record his deep sense of appreciation for the financial support by Universiti Teknologi PETRONAS.

Nomenclature

A	Area of fluidized bed
c_1	Constant
D_g	Diameter of the granule
D_h	Hydraulic diameter of voids in the granule
D_{pi}	Initial Particle Size fed to the granulator
F_{ca}	Capillary force
h	Gas to Particle Heat transfer Coefficient
k	Gas thermal conductivity
k_{gs}	Constant for granule size
L	Length of fluidized bed
MC	Moisture content per weight of bed particles in %, wt/wt %
n	Constant
T_b	Fluidized Bed temperature
T_{gi}	Inlet gas Temperature
t_d	Time required for drying the granules
t_g	Time for granulation
s_g	Surface area of a granule
v_g	Volume of a granule
u_o	Superficial gas velocity

Greek Letters

ΔX	Length of inlet gas to fluidized bed heat transfer zone
ε	Bed Voidage
ε_g	Voidage in the granules
λ	Latent heat of evaporation
μ	Viscosity of the gas
ρ	Density of gas
ρ_p	Density of partiles
σ	Surface tension

REFERENCES

1. Nienow.A.W., Smith.P.G.,Particle growth mechanisms in fluidized bed granulation-I, the effect of process variables, Chem.Engg.Sci., 38, (1983), 1223

2.R.D.Becher, E.U.Schlunder. Fluidized bed granulation the importance of drying zone for the particle growth mechanism, Chem Engg. And Proc.,37, (1998), 1-6

3.Mackaplow B.M, Lawrence A.R, Michaels, Effect of primary particle size on granule growth and endpoint determination in high shear wet granulation, Powder Technology, Volume 108, Issue 1, 1 March 2000, Pages 32-45

4. Hemati. M.,Cherif.R,Saleh.K, Pont.V, Fluidized bed coating and granulation: influence of process related variable and physicochemical properties on the growth kinetics, Powder Technology, 130, 1-3, 2003, 18

5. Rajniak P, Mancinelli C, Chern R T, Stepanek F, Farber L, Hill B T, Experimental study of wet granulation in fluidized bed: Impact of the binder properties on the granule morphology, Int. Journal of Pharmaceutics, Volume 334, Issues 1-2, 4 April 2007, 92-102

6. Miwa.A.,Yajima.T,Shigeru.I.,Prediction of suitable amount of water addition for wet granulation Int.J. of Pharm, 195, (2000),81

7. Goldszal.A, Bousquet.J, Wet agglomeration of powders: from physics toward process optimization, Powder Tech, 117, 2001, 221

8. Frake.P, Greenhalgh.D, Grierson. S. M., Hempenstall. J. M., Rudd. D. R. Process control and end-point determination of a fluid bed granulation by application of near infra-red spectroscopy, International Journal of Pharmaceutics, Volume 151, Issue 1, 12,May,1997,75-80.

9. Laicher.A., Profitlich. T, Schwitzer. K, Ahlert. D, A model signal analysis system for endpoint control during granulation, European J of Pharm Sci, 5, 1,1997, 7-14

10. Derek Daniher, Lauren Briens and Andy Tallevi, End-point detection in high-shear granulation using sound and vibration signal analysis Powder Tech, Dec 2006,

11.Simons.S.J.R, Fairbrother R.J, Direct observation of liquid binder –particle attractions: the role of wetting behavior in agglomerate growth, Powder Tech ,110, 200, 44-58

12. Findley W. P. ,Peck. G. R.,Morris K.R., Determination of fluidized bed granulation endpoint using near infrared spectroscopy and phenomenological analysis, 94, 3 , 2005.