International Journal of Engineering Research
Volume No.3, Issue No.3, pp : 182-185
ISSN:2319-6890)(online),2347-5013(print)
01 March 2014

Studies on Parameter Optimization for Particle Growthin a Fluidized BedGranulator: Interval Halving Method

Abanti Sahoo, Lisa Sahoo

Chemical Engg. Dept., N. I. T.Rourkela-769008 India

E-Mail: asahu@nitrkl.ac.in, India

Abstract :Effects of various process parameters such as amount of liquid binder, temperature, gas flow rate and weight of bed materials on the growth of particles in a Fluidized Bed Granulator are analysed. A comparative study is carried out for two bed materials (i.e. TiO_2 and $CaCO_3$) through experimentations. The result i.e. particle size of fluidized bed materials is correlated with the various process parameters. These correlations are observed to be satisfactory with both exponential and power trends but higher R^2 value is observed with the exponential trend. The calculated values of the percentage of particle growth thus obtained through these developed correlations are compared with the experimental values for both the bed materials which show good agreements with each other. The process parameters are also optimised using Interval Halving method which indicates improvement in the performance of the fluidized bed granulator. MATLAB coding is also developed for the optimization of these correlations. Neither any local maximum nor any local minimum is observed with any of the system parameters thereby implying further the better applicability of these developed correlations over a wide range of parameters.

1. Introduction

Granulation is a size enlargement process by which fine powders are agglomerated into large agglomerates. Granulation improves flowability and appearance which finds great application in pharmaceutical industries. It improves handling of powder which is difficult to handle because of their cohesiveness and low flowability[1]. The control of fluidized bed granulation process is very much difficult as it involves wetting, drying and mixing of particles simultaneously. Therefore it is necessary to understand the mechanism of particle growth and to study the effect of various parameters on the growth rate of particles in a fluidized bed. Also optimisation of the parameters plays an important role in order to make the system an economical and efficient one. Among the various granulation techniques, fluidized bed granulation (FBG) is considered to be one of the most widely used methods. Fluidized bed granulation (FBG) has many advantages over conventional methods. The conventional granulation methods require separate equipment. But FBG is performed in one unit which saves energy cost,labour cost, transfer losses and time. Another advantage is that once the conditions affecting the granulation have been optimized the process can be automated.Granulation technology has a wide spectrum of applications ranging from pharmaceutical to food, fertilizer, and detergent to mineral, ceramic, waste processing and advanced materials. In the present study Interval Halving method has been used forthe optimisation of parameters in FBG process because of its several advantages over other methods.Many studies have been carried out for better understanding of thesystem parameters and their effects on the process. The bed moisture content and the droplet size are two important elements in FBG.

1.1 Optimisation

Various classical optimization techniques are there based on what is to be optimised. On the basis of process parameters, optimization method is classified into two principal categories such as (a) Single-Variable Optimization and (b) Multi-Variable Optimization. Single Variable optimisation can be carried out by any of three methods namely, Bracketing methods, Region-Elimination methods and Gradient-based methods. Interval halving method is one of three sub categories of Region-Elimination methods.Because of complex nature of fluidized bed granulation and complexity of multivariable effects, single variable optimisation technique has been used in the present work for parameter optimization. In this work Interval Halving method has been adopted for optimisation of process parameters. The minimum of a function is found in two phases. First, a crude technique is used to find a lower and an upper bound of the minimum. Thereafter, a more sophisticated method is used to search within these limits and find the optimal solution with the desired accuracy. Before optimization of process parameters it is essential to express the output as a function of input parameters. Therefore attempt has been made to correlate the output of granulation process to all the system parameters in the present work by regression analysis.

1.2 Optimisation technique:

Exponential regression uses Excel's tool for regression analysis which determines the slope and intercept of straight

International Journal of Engineering Research
Volume No.3, Issue No.3, pp : 182-185
ISSN:2319-6890)(online),2347-5013(print)
01 March 2014

line data. An exponential equation has the general form of $y = Ae^{BX}$ where, A, B are overall coefficient and exponent coefficient. X is the independent variable.In the present work the fraction of particle growth (dependent variable) has been correlated against the product of number of independent parameters by exponential regression analysis as mentioned below.

For exponential regression:

$$y = A\left[e^{m_1 W} e^{m_2 F} e^{m_3 T} e^{m_4 V}\right] = A\left[e^{(m_1 W + m_2 F + m_3 T + m_4 V)}\right] \quad (1)$$

Where, m_1, m_2, m_3, m_4 are individual parameter coefficients.The overall independent variable for the granulation process is defined as follows.

$$X = [m_1 W + m_2 F + m_3 T + m_4 V]$$

Taking logarithm on both sides, Eq.-(1) can be written as

$$\ln y = \ln A + BX \quad (2)$$

For Dimensional Analysis (power form of regression)

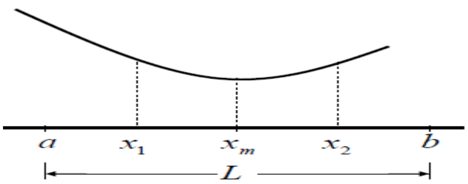
$$y = K\left[W^{a_1} F^{a_2} T^{a_3} V^{a_4}\right]^n \quad (3)$$

Where, a_1, a_2, a_3, a_4 are individual parameter exponents. K and n are overall coefficient and exponent of the correlation.

Once the minimum point is bracketed, a more sophisticated algorithm needs to be used to improve the accuracy of the solution. In this section, three algorithms that primarily work with the principle of region elimination are described. Depending on the function values evaluated at two points and assuming that the function is unimodal in the chosen search space, it can be concluded that the desired minimum cannot lie in some portion of the search space. Internal Halving is a sub type of this method.

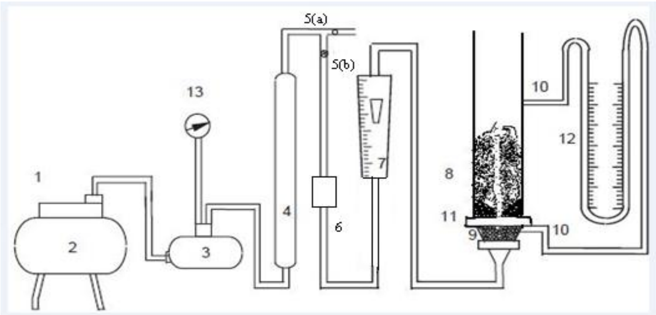
1.3Interval halving method

In this method, function values at three different points are considered. Three points divide the search space into four regions. Fundamental region elimination rule is used to eliminate a portion of the search space based on function values at the three chosen points. Three points (x_1, x_2 and x_m) chosen in the interval (a, b) are all equidistant from each other and equidistant from the boundaries by the same amount as shown below.Two of the function values are compared at a time and some region is eliminated.



2. Experimental

The schematic diagram of the experimental set-up is shown in Fig.-1.The experimental set up consists of a fluidizer of 10cm diameter and 100cm height. Fluidizer is made up of transparent perspex material. A rotameter and a manometer are connected to the fluidizer. A compressor and a hot air generator are also there to supply hot air at the set temperature. Titanium dioxide powder (average size 25µm and density 0.77gm/cc) and Calcium carbonate powder (average size 15µm and density 0.7gm/cc) are taken separately as bed materials for the experimentations. Sucrose solution is taken as the binder for both the bed materials. A known weight of sample was taken in the fluidizing column. Temperature of the hot air generator was set at a particular value. Compressed air was allowed to pass through the hot air generator to the fluidizer till steady fluidization of bed materials is achieved. Flow rate is then measured from the rotameter. Binder solution prepared at a known concentration is added for granulation of the bed materials. The same procedure was repeated several times by varying each of the parameters (viz. the volume of binder, the amount of the bed materials, temperature of inlet air and flow rate of the fluidizing air) under investigation as mentioned in Table-1. Again the whole procedure was repeated for the second sample.



1.Compressor, 2. Receiver, 3.Constant pressure tank, 4.Silica gel tower, 5(a).By-pass valve, 5(b).Line valve, 6.Hot air generator, 7. Rotameter, 8.Fluidizer, 9.Calming section, 10.Pressure tappings. 11. Distributor, 12. Manometer. 13. Pressure gage

Figure-1: Schematic diagram of the experimental set up.

Table -1: Scope of Experiment

Sl No.	for TiO_2				for $CaCo_3$			
	W, g	F, Nm^3/h	T, °C	V, ml	W, g	F, Nm^3/h	T, °C	V, ml
1	25	32	50	2	50	26	50	3
2	40	32	50	2	50	26	50	5
3	60	32	50	2	50	26	50	7
4	80	32	50	2	50	26	50	10
5	25	32	50	2	30	26	50	5
6	25	60	50	2	40	26	50	5
7	25	70	50	2	45	26	50	5
8	25	85	50	2	50	26	50	5
9	25	32	50	2	40	26	50	5
10	25	32	60	2	40	24	50	5
11	25	32	70	2	40	22	50	5
12	25	32	80	2	40	20	50	5
13	25	32	50	2	40	26	50	5
14	25	32	50	5	40	26	55	5
15	25	32	50	10	40	26	60	5
16	25	32	50	15	40	26	65	5

3. Results and discussion

Fractional growth is measured by taking the ratio of final to initial size of each granule.The particle growth has been correlated against different system parameters. The variations of particle growth against all the parameters are found to fit both exponential and power trends for both the samples. It is observed that R^2- values for both the samples i.e. TiO_2 and $CaCo_3$ are higher for exponential trend in comparison with the power trend. Therefore the correlation plots for these samples in exponential trends have been shown in Fig.-2 and Fig.-3 respectively.The effects of different parameters namely weight of the sample, flow rate of air, temperature and volume of binder on particle growth were studied. It is observed that particle growth decreases as weight of bed materials increases. The reason may be due to the presence of more materials with the same amount of binder. In other words the binder amount might not be sufficient to bind more particles. The ratio between the binder volume and the number of particles decreases leading to a lower growth rate and smaller agglomerates.Itis alsofound that smaller size granules are formed on increasing the air flow rate. This may be due to the fact that higher fluid velocity results in collisions among the particles and thus the large agglomerates are reduced to small size granules.Increase in inlet air temperature seems to decrease the granule size. The reason may be attributed to the evaporation of the sprayed liquid(binder) at higher temperature. As a result less binder is mixed with the bed materials leading to small sized granules.The effect of the amount of the binder on the growth of the particles is also studied. It is observed that more the amount of binder more is the particle growthresulting in higher granule size. On adding more binder, more materials agglomerate and thus the granule size is increased.The calculated values of particle growth obtained through these developed correlations have also been compared with the experimental values which show a good agreement in most of the cases. The comparison plots are shown in Fig.-4.The standard deviation and mean deviation for both the samples with both exponential curves and power curves are calculated and listed in Table-2.

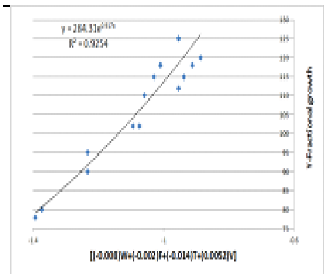


Figure-2: Correlation plot for TiO_2

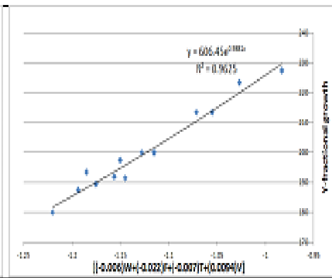


Figure-3 : Correlation plot for $CaCO_3$

Table-2: Comparison of calculated values of particle growth against the experimentally observed values

Deviation	For TiO_2		For $CaCO_3$	
	Exponential form correlation	Power form expression	Exponential form correlation	Power form expression
Std. deviation, %	4.174	4.502	1.252	1.483
Mean deviation, %	-0.085	-0.084	-0.004	-0.016

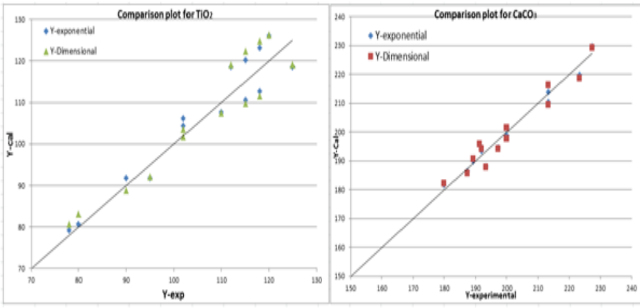


Figure-4 : Comparison plot for calculated values of particle growth with the experimentally observed values

The particle growths for the two samples are compared with each other which shows that the effects are higher in magnitude for $CaCO_3$ implying the better particle growthwith $CaCO_3$. The developed correlations in exponential and power forms for both the samples are as follows.

3.1 Correlations in Exponential Forms

For TiO_2

$$Y = 284.3 \times e^{0.917 \times [-0.008W - 0.002F - 0.014T + 0.0052V]}$$

For $CaCO_3$

$$Y = 606.45 \times e^{0.9881 \times [-0.006W - 0.022F - 0.007T + 0.0094V]}$$

(4-b)

3.2 Dimensional Analysis (Correlations in Power forms)

For TiO_2 :

$$Y = 9812.5 \times \left[(W)^{-0.394}(F)^{-0.123}(T)^{-0.901}(V)^{0.0341}\right]^{0.8491} \quad (4\text{-c})$$

For $CaCO_3$:

$$Y = 10170 \times \left[(W)^{-0.244}(F)^{-0.487}(T)^{-0.379}(V)^{0.0515}\right]^{1.009} \quad (4\text{-d})$$

3.3 Parameter Optimisation (Internal Halving method):

Considering equation-(4-a) for sample analysis, the parameters of thecorrelation have been tried to be optimized using single variable method to get maximum particle growth. In this method one variable is varied at a time and keeping other variables constant. Therefore theoptimisation of process variables has been carried out one by one.

<u>Volume of the binder</u>

With the volume of the binder parameter selected for optimization, the values of other variables, such as weight of bed materials, air flow rate and inlet air temperature are substituted inEquation-(4-a). The resulting expression can be written as follows.

$$Y = 284.3 \times e^{0.917 \times [(25)\times(-0.008)+(32)\times(-.002)+(50)\times(-0.014)+(V)\times(0.0052)]}$$

$$\Rightarrow Y = 284.3 \times e^{0.917 \times (-0.964 + 0.0052 \times V)}$$

$$\Rightarrow Y = 284.3 \times e^{-0.884 + (4.77 \times 10^{-3})V} \quad (5)$$

Similarly different expressions are obtained with different parameters.

<u>Inlet air temperature (T) :</u>

With this parameter, the values of binder volume,fluidizing air flow rate (F) and amount of bed material (W) are substituted in Equation-(4-a). The resulting expression is written as

$$Y = 284.3 \times e^{-0.23+(-0.012)T} \quad (6)$$

Air Flow rate (F):

With this parameter, the values of binder volume, fluidizing air temperature (T) and amount of bed material (W) are substituted in Equation-(4-a). The resulting expression is written as

$$Y = 284.3 \times e^{-0.82+(-1.834\times10^{-3})F} \quad (7)$$

Weight of bed material (W) :

With this parameter, the values of binder volume (V), fluidizing air temperature (T) and air flow rate (F) are substituted in Equation-(4-a). The resulting expression is written as

$$Y = 284.3 \times e^{-0.75+(-7.34\times10^{-3})W} \quad (8)$$

Thus the multivariable equation, Eq.-(4-a) is converted into a number of single variable equations so that the single variable optimization technique can be followed. The defining or objective function only changes with different single variable equations such as, equation (6), (7) and (8).With the help of MATLAB coding the process parameters were subjected to optimisation using Interval Halving method. It is observed that there is neither a local maximum nor a local minimum with any of the above mentioned process variables.

4. Conclusion

Granulation in fluidized bed is a complex process since a number of variables affect the process thereby affecting the particle growth. Thus selection of process parameters plays vital role in the process of granule formation. As it is observed that there is neither any local maximum nor any local minimum the developed correlations in any form (either exponential or power form) can be used successfully in industries over a wide range of parameters. Otherwise these correlations can be modified suitably for pilot plant scaled operations.

Nomenclature

W	:	Weight of particle in the bed (g)
F	:	Fluidizing air flow rate (Nm^3/h)
T	:	Temperature of fluidizing air (^oC)
V	:	Volume of binder used (ml)
Y-exp	:	Experimental Fractional growth
Y-cal	:	Calculated Fractional growth
Y	:	Fractional growth

References

i. *J.D. Litsterand B.J. Ennis, The science and engineering and granulation processes, Kluwer Academic Publishers, Dordrecht, (2004).*

ii. *H.G.Kristensen and T. Schafer, A review on pharmaceutical wet-granulation. Drug Dev. Ind. Pharm. 13, 803-872 (1987).*

iii. *W.L. Davies and W.T.Gloor, Batch Production of Pharmaceutical Granulations in a Fluidized Bed I: Effects of Process Variables on Physical Properties of Final Granulation. J. Pharm. Sci. 60, 1869-1874 (1971).*

iv. *T. Schafer and O.Worts, Control of fluidized bed granulation: effects of inlet air temperature and liquid flow rate on granule size and size distribution. Arch. Pharm. Chem. Sci. Ed. 6, 1-13 (1978a).*

v. *S.H.Schaafsma,P.VonkandN.W.F.Kossen, Fluid-bed agglomeration with a narrow droplet size distribution. Int. J. Pharm 193, 175–187 (2000).*

vi. *T. Schafer and O.Worts, Control of fluidized bed granulation: estimation of droplet size of atomized binder solutions. Arch. Pharm. Chem. Sci. Ed. 5, 178-193 (1977).*

vii. *P.Merkku,O.Antikainen and J.Yliruusi, Use of 33 Factorial Design and Multilinear Stepwise Regression Analysis in Studying the Fluidized Bed Granulation Process, Part II. Eur. J. Pharm. Biopharm. 39, 112-116 (1993).*

viii. *L.Juslin,O.Antikainen, P.MerkkuandJ.Yliruusi, Droplet size measurement: I. Effect of three independent variables on droplet size distribution and spray angle from a pneumatic nozzle. Int. J. Pharm. 123, 247-256 (1995a).*

ix. *L. Juslin, O. Antikainen, P. Merkku and J. Yliruusi, Droplet size measurement: II. Effect of three independent variables on parameters describing the droplet size distribution from a pneumatic nozzle studied by multi-linear stepwise regression analysis. Int. J. Pharm. 123, 257-264 (1995b).*

x. *M.Hemati,R.Cherif,K.Saleh and V. Pont, Fluidized bed coating and granulation: influence of process-related variables and physicochemical properties on the growth kinetics, Powder Technology 130, 18– 34 (2003).*

Figure Caption :

Fig.-1: Schematic diagram of the experimental set-up

Fig.-2: Plot of particle growth rate against the system parameters for TiO_2

Fig.-3 : Plot of particle growth rate against the system parameters for $CaCO_3$

Fig.-4 : Comparison plot for calculated values of particle growth with the experimentally observed values.