International Journal of Chemical Engineering and Applications, Vol. 1, No. 3, October 2010
ISSN: 2010-0221

Granule Growth Mechanism Studies in a Fluidized Bed Granulation

K.S.K. Rao Patnaik, *Member, IACSIT* and K. Sriharsha

Abstract—**The process of granulation is widely used in manufacturing particulate solid materials in order to produce a desired size or shape, especially when some particular physical properties are required. It can be used to manufacture product with improved qualities such as more uniform structure, non dusting, non caking and more resistance to mechanical shocks and protective coatings. It has been used on an industrial scale in the manufacture of many products including inorganic salts, fertilizers, pharmaceuticals, food, ceramic, steel, water purification, mineral beneficiation, and powder metallurgy. It is evident from the literature, the mechanism of granulation is complex. It depends on type of material being granulated and also by the method of running the process .It is essential to carry out laboratory scale experiments with the materials to be used before starting the design of any industrial scale granulator. The experimental setup consists of a main glass fluidization column having an ID of 10.16cm and 24cm height. This glass column is placed over a mild steel column of the same diameter and height 98cm.The binder solution was fed through a 2mm stainless steel tube and the compressed air from a 6.35mm tube at the tip of the atomizer. The binder solution was atomized due to the impingement of the air jet. The present investigation is to study the concept of granule formation by individual drops and to quantify the relationship between drop size and granule size by direct measurement. It has been shown that the mechanism and kinetics of granule growth in fluidized bed granulation process and important conclusions are presented in the paper.**

Index Terms—**Fluidized bed granulation, binder solution, pneumatic twin-fluid atomizer, growth mechanism.**

I. Introduction

Granulation may be taken to mean the product of particles of processed materials which in themselves consist of agglomerates of smaller particles adhering to each other.The process of granulation (or agglomeration) is widely used in manufacturing particulate solid materials in order to produce a desired shape or size. The subject agglomeration describes the forming of a physically larger body from a number of smaller bodies i.e., particles in identifiable form.

Particle enlargement may directly make powdered product particles, such that the larger mass becomes a permanent entity but still retains the original more useful or it may improve their further processing. Granulation is beneficially employed to impart desirable flow properties to reduce dusting hazards and losses, to facilitate the recovery of fines, to prevent the segregation of components, to secure controlled porosities, to densify, to promote fluidization and to create definite sizes and shapes. Among many different techniques the fluidized bed granulation process has attracted interest in recent years. A wide range of applications of fluidized bed granulation has been reported, including the pharmaceutical, food, fertilizer, ceramic, steel, water purification, mineral benefication and powder metallurgy. Granulation of inorganic salts, the calcination of radioactive wastes and for effective eff1uent treatment to fight against pollution. One of the main advantages of this manufacturing process is many different operations such as granulation, mixing, evaporation, drying, crystallization, cooling and classification are performed within one system. As a modern consideration, the utilization of low grade iron-ores depend in large measures on processes for making a suitable blast-furnace raw material from the fine-sized concentrate produced by ore dressing methods. To be consistent with these furnace requirements, the fine material must be agglomerated into a relatively coarse product. Granules with a narrow size distribution have been formed in a fluidized bed by adding a series of precisely sized drops of binder solution. Waldie, B (1980) studied the concept of granule formation by Individual drops and to quantify the relationship between drop size and granule size by direct measurement. Resulting size data together with that from previous binder distribution and spray drop-size measurements provide a wide ranging correlation between granule size and drop size of the form.

$$D_g \alpha \ D_d^n \quad (1)$$

Where D_g and D_d are granule diameter in μm and drop diameter in μm, respectively

For lactose and ballotini particles under specified conditions, values of n were 0.80 and 0.85 respectively. The correlation applied over a wide size range, granules from 150 to 5000 μm and drops from 35 to 3000 μm diameter.

This model appears most appropriate for a granule formed by engulfment or particles by a single drop rather than by a succession of agglomeration events involving several drops. Bin, A et. al (1985) studied the batch or semi batch processes of granulation in a fluidized bed, particals are agglomerate and cluster. It is demonstrated that such processes can be used to determine kinetic data of granule growth. This effect has been taken into account in a simplified treatment of the experimental data leading to estimation of the linear growth rate of the granules. If the mass increase of the particles plays

K.S.K.Rao Patnaik is with the Department of Chemical Engineering, University College of Technology (A), Osmania University, Hyderabad – 500 007, India(Fax No: 91- 40 – 27098472, email: ksk_raopatnaik@yahoo.co.in).

K. Sriharsha is with the Department of Mechanical Engineering, CBIT, Osmaina University, Gandipet, Hyderabad--500 075, India.

a dominant role, the mass averaged size of the product particles should be used as a representative granule size. The number

N of particles (granules) in the bed was evaluated using the relationship

$$N = \frac{6}{\pi \rho_s} \Sigma \left[\frac{\Delta m_i}{(d_i)^3} \right] \qquad (2)$$

Where ρs, Δm_i and d_i are the density of non-porous particles, g/cc, total mass of the bed, g, i th fraction and mean diameter of the particles, cm, i th fraction respectively.

Which depends on the granules being spherical and non-porous.

Iveson,S.M et.al (2001) reviewed their work on Nucleation mechanism, growth techniques and breakage phenomena in agitated wet granulation processes.

Hapgood,K.P and Rhodes.M (2008) have studied the size enlargement in granulation

Processes.Hapgood,K.P et.al (2008) also described the granulation rate calculations very effectively.

II. Problem Formulation

The solids may be processed under wet or dry conditions. Wet granulation generally comprises the use of a binder to form moish granules after dried and finally screened to produce the required size distribution. The binders are materials selected for their ability to form strong efficacy at high rates. For some industrial applications the binders may be mixed with coloring matters. In food technologysome flavoring ingredients are added with lactose binders. Granulation is also used as one of the method for increasing the effectiveness of a tabletting process in Parma world. If provides for highly efficient heat and mass transfer rates. The lactose, kaolin, dicalcium phosphate materials are widely used as diluents in tabletting operations in Parmaceutical operations.

A. Objectivities and aim

Following the problem formulation the objectivities of the work are:

1. Effect of fluid temperature
2. Effect of binder addition rate
3. Effect of time of granulation
4. Effect of size of feed material
5. Effect of granulation mechanism/ Efficiency η_g and Elutriation index I_e

III. Experimental

A. Experimental Setup

A schematic diagram of the experimental setup for the study of fluidized bed granulation using pneumatic twin fluid atomizer is shown in Fig. 1.

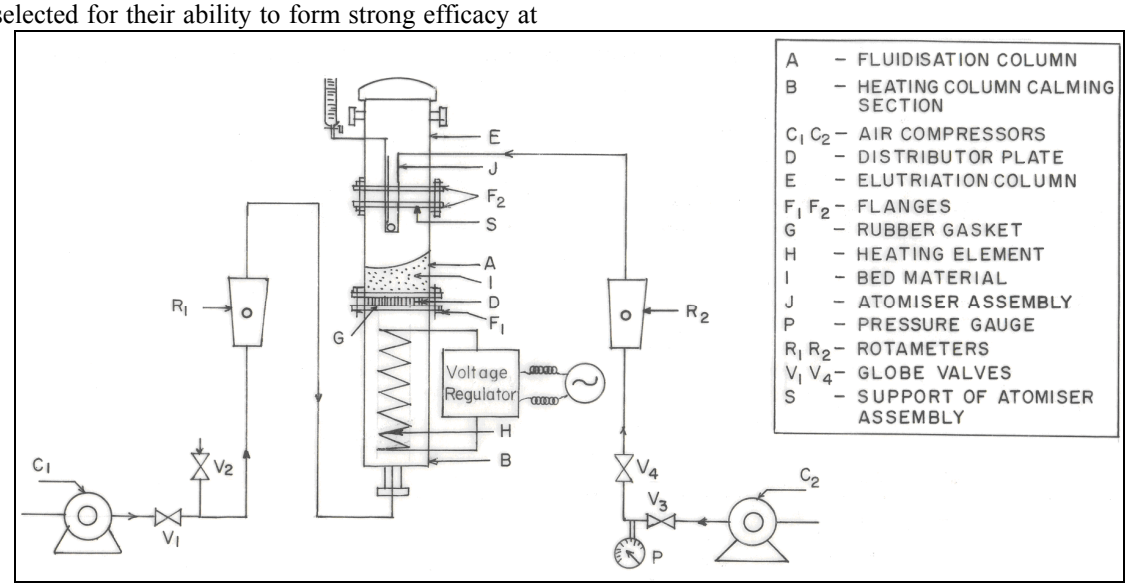

Fig.1: A schematic diagram of the experimental setup

The experimental set up consists of a main fluidization column A, made up of glass having an inside diameter of 10.16cm and 24cm height. This glass column was placed at the top of another mild steel column B of the same internal diameter as the glass column but provided with a flange F_1. The mild steel column is 98 cm high. The bottom position of the column, a heater H is provided while the top position T acts as a calming section for the fluidized bed. The glass column A is also provided with moulded flanges F_2 and rubber gaskets. The distributor plate D was consisting of a 0.3175 cm thick mild steel plate with holes of 0.3175 cm

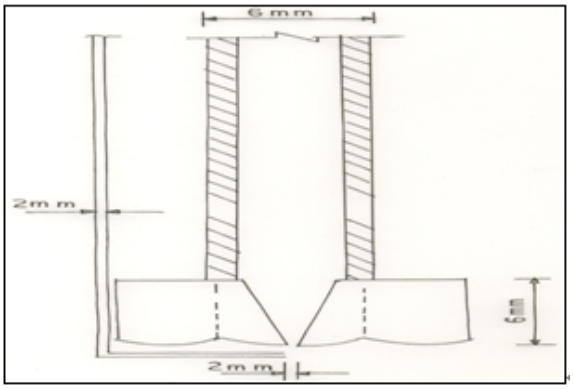

Fig.2: Pneumatic twin fluid atomiser

Punched through it and in all 29 holes were punched. The

number was arrived at, providing a minimum pressure drop of 35 cm of water across the distributor plate D. The fine particles were prevented from dropping through by covering the distributor plate with a 100 mesh brass screen. The heating of the fluidizing medium (air) was done by electrical means of nichrome wire wound on an asbestos support. The fluidizing medium, air was fed by a reciprocating compressor through the heating section B into the glass column A via the distributor D. In order to prevent heat losses through the walls of the column B was lagged by winding with 0.635 cm asbestos rope, over which a layer of asbestos and magnesia coating was applied.

The binder solution was fed from a burette to keep of the flow rate. The binder solution enters the 2 mm tube as shown in Fig.2 and the compressed air fed into the 0.635 cm tube at the tip of the atomiser J. The binder solution was atomised due to the impingement of the air jet. A pressure gauge P was provided in the airline. The whole atomiser assembly J fixed into the glass column A such that the spray umbrellas reached the top of the fluidized bed without coming into contact directly with the wall of the glass column. Initial bed height was always kept constant in all the experimental runs.

B. *Procedure*

The compressed air was fed into the fluidized bed column A through the heating section B and through the distributor D. The dimmer stats were adjusted to get the required temperature in the bed. The fluidizing air flow rate was adjusted such that a predetermined bed height was achieved. The flow rate was maintained constant throughout the batch experimental run. The binder solution was atomized by pneumatic twin-fluid atomizer. The feed rate of the binder solution and the flow rate of compressed air to the atomiser were adjusted in such a way that an optimum spray was obtained by the material. The binder solution added for the experimental run was fixed at either 0.5% or 1% of total material taken, i.e., for 800 g of material the binder amount added was 4 or 8 g respectively. This binder amount was fixed with water to make a solution of 5% concentration and fed to the atomiser J. The product was withdrawn by opening the flanges F1 and F2 holding the glass column A, distributor D and the mild steel column B.

The range of parameters covered in the present investigation are presented in Table.1

IV. Results and Discussion

In the present investigation studies on granulation process in a fluidized bed have been conducted and an attempt was made to determine the effect of various fundamental aspects viz., fluid temperature, binder addition rate, time of granulation and size of feed material.

The efficiency of granulation was calculated as

$$\eta_g = \frac{W_i - W_f}{W_i} \times 100 \quad (3)$$

Whereas the elutriation efficiency was evaluated from

$$\eta_E = \frac{W_a}{W_c} \times 100 \quad (4)$$

The elutriation in dex (I_e) is considered as a measure of the intensity of attrition within the bed charge and is estimated as the ratio of diameters of particles collected in the cyclone separator to the smallest particles in the initial bed charge i.e.,

$$I_e = \frac{D_{p,\ Cyclone}}{D_{p,\ bed}} \quad (5)$$

TABLE .1 Materials used & Scope of experiments

Name of the variable	Range
Material	Lime stone powder, Active
Fluid bed weight	700 — 800 g
Size of particles	0.503, 0.314, 0.178 mm
Fluid temperature	50,53,55,58,60,63,65,70,75,80 ^{0}C
Binder	Dextrin Powder
Solvent	Water
Amount of Binder	0.5&1% of total bed weight

A coulter counter was used for measuring the particle diameter for the elutriated fines separated in the cyclone.

η_g = granulation efficiency, %

W_1 = initial weight of charge, mg

W_f= final weight of a size, mg

W_a= weight of fines collected, mg

W_c= original weight of charge, mg

η_E= Elutriation efficiency, %

I_e= Elutriation Index

D_p= particle diameter, μm

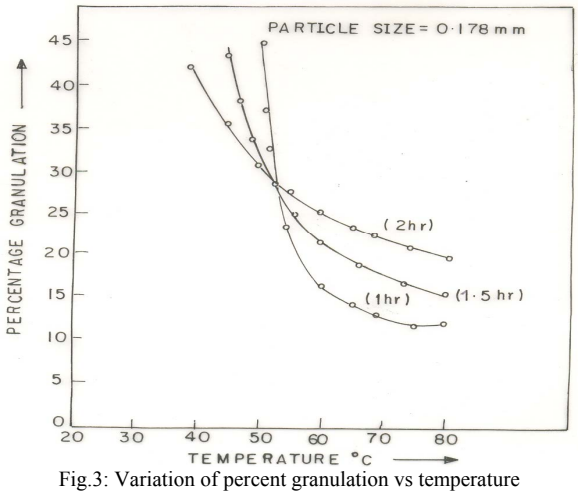

Fig.3: Variation of percent granulation vs temperature

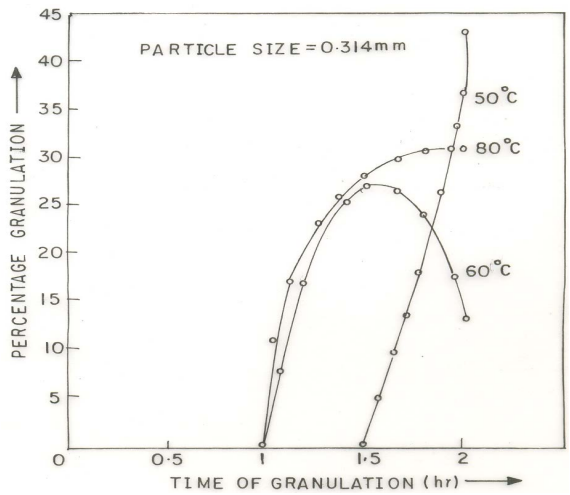

Fig 4: Variation of percent granulation of vs time of granulatio

In Figure. 3 shows the percent granulation is plotted against temperature. It can be observed that, with increase in temperature the percent granulation may decrease. The parameter being the time of granulation. Similar trends were also observed with 0.5 & 1 % binder concentration. This behaviour can be attributed to two reasons. The ratio of binder solution to bed material is fixed, keeping the total amount of binder constant, the time is varied from 1 hour to 2 hours. At higher times of granulation, the binder rate dropped which resulted in insufficient binder amount for the growth of particles. Besides, due to evaporation of solvent in the binder solution could have been more rapid, hence sufficient amount of binder might not have reached the particles to aid in the growth mechanism. This behaviour is clearly observed with average particles diameter of 0.178 mm.

In Figures. 4 & 5 show the percent granulation is plotted against time of granulation, the parameter being the fluid temperature. At higher temperatures, the percent granulation in general shows an increasing trend and then decreasing behaviour with the increase in time of granulation. The decrease in growth of particles with increase in time can be due to inadequate amounts of binder input to the system.

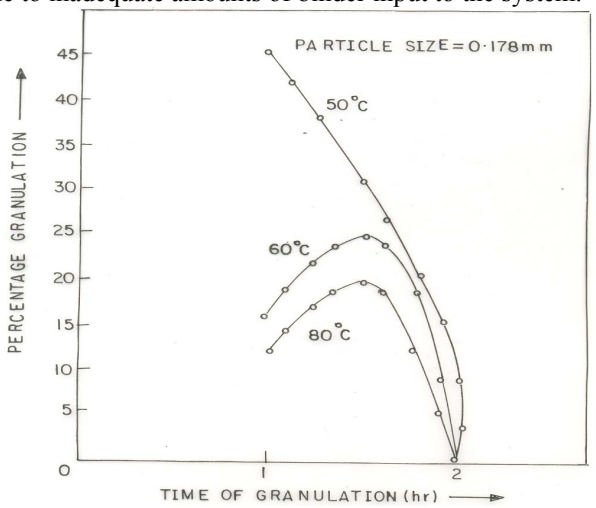

Fig.5: Variation of percent granulation vs time of granulation

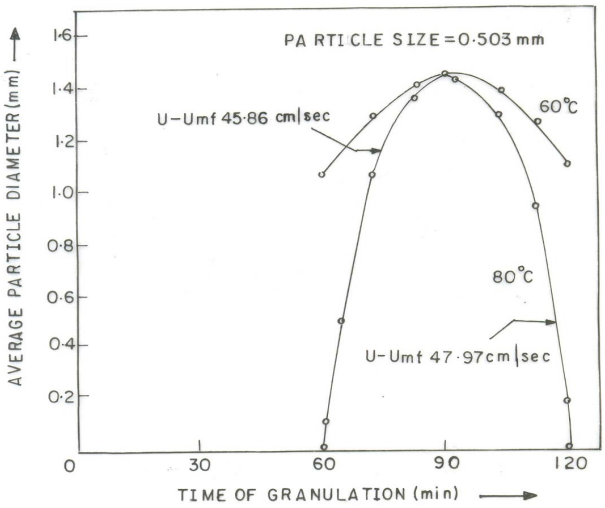

Fig.6: Variation of average particle diameter vs time of granulation

In Figures. 6, 7 & 8 show the average particle diameter of the product is plotted against time of granulation for difference in fluid temperatures. For 0.503 mm average particle diameter, keeping the time of granulation constant, at higher fluid temperature resulted in smaller product size, because of rapid evaporation of the binder solvent, lowering the chances of binding. For 0.314 mm average particle diameter, spraying 0.5% binder solution, no granulation was observed at higher temperatures. This might be due to insufficient binder rate, but at lower temperature 50°C, granulation was observed. Spraying 1% binder amount, the particle diameter showed an Increasing pattern with time of granulation for all temperatures. Thus shows that binder rate was sufficient for the particles to grow. When 0.178 mm average particle diameter was used as bed material, the product diameter showed a decreasing pattern with increase in time of granulation, and binder was insufficient. This was observed in case of 0.5% binder amount when 1% binder amount was sprayed. It showed an increasing pattern at lower temperatures but at high temperatures a decreasing trend. We can conclude as at higher temperature, higher binder rates are required compared to binder amount at lower temperatures.

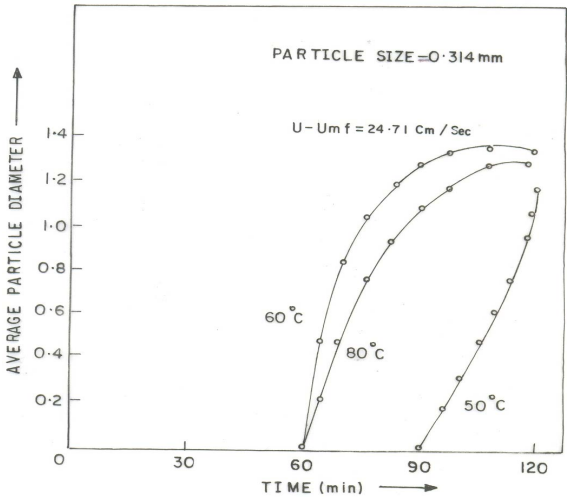

Fig.7: Variation of particle diameter vs time of granulation

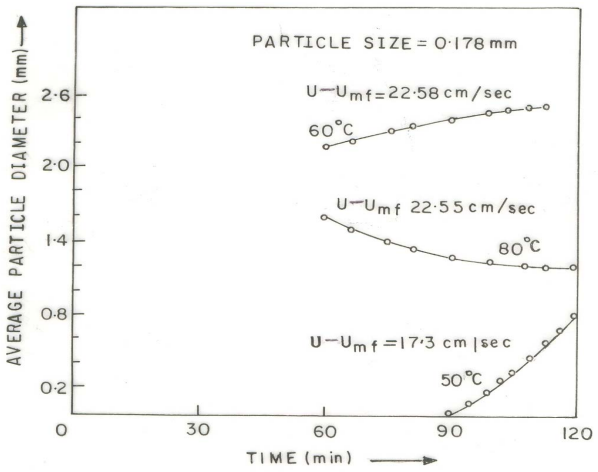

Fig.8: Variation of average particle diameter vs time of granulation

The efficiency of granulation for natural phosphate ore is higher and reaches a maximum earlier and shows a rapid derioration as compared to other materials. Pure materials lactose, Di Calcium Phoshate have an interval of time over which the granulation efficiency is nearly constant. After this time interval, granules deteriorate to attain stability at some what lower efficiency of granulation. Purified materials, containing somewhat higher percentages of impurities than pure materials kaolin, behave differently, the particles coagulate continuously with increasing granulation efficiency through the process, then nearly stabilize at some higher efficiency of granulation.

V. Conclusions

The percentage granulation shows a decreasing trend with an increase in fluid temperature. This trend is observed for both 0.5 and 1% binder amount.

At given fluid temperature, the percentage granulation shows an increasing trend and then decreasing pattern with time of granulation.

The effect them of time of granulation on the product size resulted in smaller product size at higher fluid temperatures. This reveals that at higher fluid temperature rpid evaporation of binder solvent, which lowered the chances of binding.

Bin, A et, al (1985) rightly pointed that preliminary studies in laboratory scale experiments are necessary because till date, little is known about the granulation mechanism which will be most significant during granulation of given material.

The instability of the granules formed from natural or impure materials is attributed to the weak bonding action between the particles in a grarule perhaps, due to the nonuniform distribution of forces of attraction between different species in a granule owing to the difference in their surface properties and coagulation mechanisms.

Due to the limited range of results, the authors are not able to give a proper relationship between the drop size and the granule growth on granulation processes in a fluidized bed.

Acknowledgement

The Directors, Indian Institute of Chemical Technology and Defence Metallurgical Research Laboratories, Hyderabad have kindly permitted to use the excellent library facilities which are gratefully acknowledged with appreciation

References

[1] Bin, A., Wary, Ch.J. and Komorowski, R., Powder Techn, vol. 41, no. 1, pp.1-11, (1985).
[2] Clifford, Y.Tai, Wuand J.F. and Yishih, C., Chem. Eng of Japan, vol.13, no.5 pp 562, (1990).
[3] Feurtes, A.B. G. Marban and F. Rubiera, 1993, Trans Inst. Chem. Engrs., vol.71, Part A, pp 411-428, (1990).
[4] Hapgood, K.P and Rhodes.M, 'Size enlargement' in "Introduction to Particle Technology" 2nd edition, M.Rhodes(ed), John Wiley and Sons, (2008).
[5] Hapgood, K.P., Iveson, S.M, Liu.L & Lister, J.D, Granulation rate processes in "Handbook of granulation" A. Salman, M.Hounslow and J.P.K.Seville(eds),Elsevier science, (2008).
[6] Iveson, S.M; Lister, J.D; Hapgood, K.P, "Nucleation, growth and breakage phenomena in agitated Wet granulation processes: A review" Powder Technology, vol. 117, pp 3-39, (2001).
[7] Robinson T. and B. Waldie, Trans. Inst. Chem. Engrs., vol. 57, pp 121, (1979).
[8] Schaafsma, S.H., P. Vonk., P. Segers and N.W.Y. Kossen, 1998, Powder Technol., vol. 97, pp. 183.
[9] Stefan, H., Schaafsma and Nico, W.F.. Kossen; Marinus T. Mos., Levy Blauw and Alex C. Hoffman, , AICh E Journal, vol. 45, no. 6, pp. 1202-1210, (1990).
[10] Sugimoto, M, Yokota, N and Kauramoto, H, J. of Chem. Eng. of Japan, vol. 23, no.2, pp. 144-146., (1990).
[11] Takamikai, Takeshige Takahashi, AICh E Journal, vol. 43, no.2, pp.357-362, (1997).
[12] Waidle B. and T. Robinson, Powder Tech., vol.27, pp. 163, (1980).
[13] Waldie R., Chem.. Eng. Sci. vol. 46, no.11, pp. 2781-2785, (1991).
[14] Watano, S., N. Yehand and K. Miyanami, J. of Chem. Eng. of Japan vol. 31, no. 6 pp. 908-913,(1991).
[15] Kawakita, K and Ludde, K.H., Power Technology, 4,61(1971)
[16] Kunii, D and Levenspiel, O., Fluidization Eng, Wiley, NY (1969)
[17] Legler, B.M, Chem. Eng. Prog., 63.75 (1967)
[18] Rowe, P.N., Chem. Eng.Sci., 28,797 (1973)
[19] Smith, P.G. and Nienow, A.W., Chem Eng. Sci., 73, 950 (1982)
[20] Shanley- Wood, N.G. ed., Enlargement and Compaction of particulate solids, Butterworths, London, P.181 (1983)
[21] Bjorklund.W.J. and Offutt, G.I., A.I.Ch.E.Sym.Ser, 69,123 (1973).
[22] M.Z.Abdel – Wahhab, The behavior of fluidization as a Granulation Technique for Solids, Plenary papers complied by Sarat, Recent Advancess in Chemical Engg., pp 113-120 (1996)

Dr . K.S.K.Rao Patnaik did his Ph.D in Chemical Engineering in 1997 from Osmania University. He has published and presented 35 papers in International and National conferences and journals. Currently he is working as Professor of Chemical Engineering, University College of Technology (A) and the Dean, Faculty of Technology and also the Dean, Faculty of Pharmacy, Osmania University, Hyderabad, India. He has worked as Chairman, Board of Studies, Osmania University from 2006 to 2010. He possesses 3 Gold Medals for best research papers from Institution of Engineers (I) and also acted as panel speaker, program chair for International Conferences were held on USA and Japan. His research interest areas are the following: Advancing the Fluidization Engineering Methodology, Hydrodynamic & Heat Transfer of Three phase Sparged Reactors, Energy / Bio- Energy Conversion, Process Intensification, Plant Design & Economics and Scale-up, Nano Science and Nano Technology.