IJCPE
Iraqi Journal of Chemical and Petroleum Engineering

Iraqi Journal of Chemical and Petroleum Engineering
Vol.11 No.4 (December 2010)1- 11
ISSN: 12010-4884

University of Baghdad
College of Engineering

DETERMINATION OF THE OPTIMUM OPERATING CONDITIONS IN THE GRANULATION OF GAMMA ALUMINA CATALYST SUPPORT

Alaa Dhari Jawad , Ibrahim Richeh , and Ahmed Saleh

Chemical Engineering Department- College of Chemical and Petroleum Engineering- Al-Baath University- Syria

ABSTRACT

Granulation Technique for Gamma Alumina Catalyst Support was employed in inclined disk granulator (IDG), rotary drum granulator (RD) and extrusion – spheronization equipments .Product with wide size range can be produced with only few parameters like rpm of equipment, ratio of binder and angle of inclination. The investigation was conducted for determination the optimum operating conditions in the three above different granulation equipments.

Results reveal that the optimum operating conditions to get maximum granulation occurred at (speed: 31rpm , Inclination:42^0 , binder ratio:225,300%) for the IDG,(speed: 68rpm , Inclination: 12.5^0 , binder ratio: 300%) for the RD and (speed:1200rpm , time of rotation: 1-2min)for the Caleva spheronizer used in the extrusion spheronization method. These results are compatible with similar works on granulation of different materials [1, 2, and 3].

Keywords: Spheronization, Inclined Disk Granulator, Rotary drum, extrusion – spheronization, Gamma Alumina, Catalyst Support, shaping of catalyst.

INTRODUCTION

In chemical industry, alumina agglomrtates are often used as catalyst supports [4].The use of gamma Al_2O_3 as a catalyst support requires a shaping operation adapted to the type of process: moving bed or fixed bed .For a moving bed, it is necessary to prepare 1 to 4mm diameter beads to facilitate circulation and

to limit the mechanical abrasion of the catalyst. For the fixed bed, the support can be in the form of either beads or cylindrical extrudate 1-4 mm in diameter. The catalyst support is shaped essentially by three methods: granulation, drop coagulation, and extrusion [5].

Forming or shaping of supports and catalysts is an important step in the preparation procedure of a commercial catalyst. The catalyst must have a prescribed shape and size for the given chemical reactor. The shape and size is determined by the application of the catalyst and the type of the reactor in which it will be used. For a fixed bed reactor it is very important that the pressure drop over the bed is as low as possible. This condition is usually fulfilled by using pellets, extrudate or spheres with a diameter greater than 3 mm. [6, 7].

Granulation is the generic term used for particle agglomeration processes, in which fine powdery solids are agglomerated together with a liquid /melt binder to form larger aggregates. Depending on the process requirements, a rotating drum, fluidized bed or high shear mixer granulator may be used for granulation by agitation [8].This paper is concerened with granulation in inclined disk granulator, Rotary Drum granulator and extrusion – Spheronizaton equipments.

Drum granulation is a particle size enlargement process often obtained by spraying a liquid binder or slurry onto fine particles as they are agitated in a rotary drum. A typical continuous granulation circuit consisting of a drum granulator, rotary dryer ,screen classifier, crusher and a number of conveyors [9].

Extrusion and spheronization is currently one of the techniques used to produce pharmaceutical pellets. The preparation of spherical granules or pellets by extrusion and spheronization is now a more established method because of its advantages over the other methods [10, 11].

In principle the basic machine consists of a round disc with rotating drive shaft, spinning at high speed at the bottom of a cylindrical bowl. The spinning friction plate has a carefully designed groove pattern to the base. This is most often crosshatched, but several sizes and other types are available. These discs are designed to increase the friction with the product [12].

As processing continues the shape of the pieces gradually changes as shown ion fig. 2 below [12]. When the particles have reached the desired shape then spheroids can be removed.

Disk granulators

It consists of a tilted rotating disc with a rim to hold the tumbling granules. Powder feed are continuously fed to the disc, typically at the edge of the rotating granular bed. Liquid binder is added through a series of single fluid nozzles distributed across the face of the bed. The critical speed is speed at which a granule is just held stationery on the rim of the disc by centripetal forces alone.

$$N_o = (g \sin \beta / 2\pi^2 D)^{1/2} \qquad 1\text{-}1$$

Discs are typically operated at between 50 and 75% of critical speed with

the angle between 45 and 55^0.If the speed is too low, the particle mass will slide

against the disc instead of tumbling [3, 14].

The purpose of this work is to determine the optimum operating conditions in the granulation (spheronization) of the gamma alumina catalyst support by using three spheronization methods(inclined disk granulator, rotary drum and extrusion – spheronization) .

EXPERIMENTAL WORK

Materials

Gamma alumina powder supplied Suzhou Yuguang Lighting Materials Co. Ltd. From China was used with the properties shown in table 1 .The XRD chart for it is shown in Fig. 1.

Table 1 Properties of the Gamma alumina powder

No.	Specification	Value
1	Name	High purity Alumina powder
2	Type	Gamma-Al_2O_3
3	Purity%	99.99
4	Particle size,μm	0.2
5	Loose density, g/cm^3	0.16
6	BET,m^2/g	120
7	K,ppm	30
8	Na,ppm	18
9	Fe,ppm	10
10	Si,ppm	20
11	Mg,ppm	10
12	Mn,ppm	9
13	Ti,ppm	10
14	Cr,ppm	10
15	Cr,ppm	10
16	Zn,ppm	10

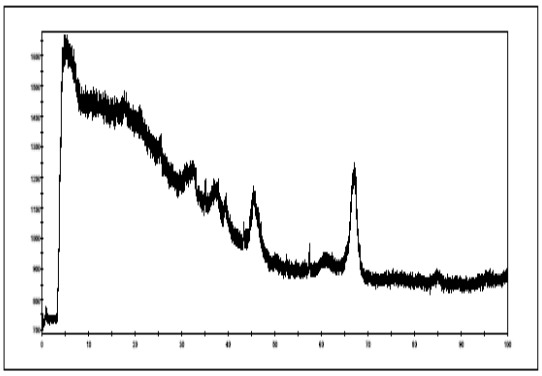


Figure 1 XRD chart for the Gamma alumina powder used

Commercial Sodium silicate solution was used as a binder, its specific gravity is 1.500 and its concentration is 44%, and used widely in detergent industries in Syria.

Equipments

Inclined Disk Granulator (Pan Granulator)

In our work, an equipment was designed to fix pan granulator of dimension, D: 27cm, height of the wall of the pan (H):5 cm .The equipment was provided with invertor (Delta 0.75hp) to supply variable speed (0-300 rpm) ,also it is provided with facility to make inclination angle in the range of (0-90^0), the motor is of type (50Hz 230/400V /Y,0.25KW,1.71/0.99A

, 1365 rpm).

Rotary Drum Granulator

The rotary drum of diameter 17 cm, H: 14cm and has opening of D: 6cm was made and fixed on the same device designed for the IDG described above.

Caleva spheronizer MBS

A spheronizer device manufactured from Caleva process solution Ltd. company , UK, which has the capacity of loading 120gm extrudate for spheronization was used in the extrusion –spheronization method to get spheres of gamma alumina catalyst support.

Design of Experiments

The response surface method from the Minitab 15 software was used to get the table of design for the determination of the optimum conditions in the granulation of gamma alumina powder in the IDG and RD operation which include the affecting parameters (inclination angle, speed of rotation, binder ratio) [15] .The minimum and maximum limits for the above variables which was given to the Minitab software were determined as shown in table 2.

Table 2 Minimum and Maximum values to the variables studied

S	Variable	Devise	Min.	Max.
1	Inclination angle	IDG RD	45^0 2^0	55^0 10^0
2	Rotation speed	IDG RD	30 27	52 66
3	Binder ratio%	IDG RD	150 150	225 225

The minimum and maximum speed for the IDG were determined from us by calculating the critical speed of the disk used (D 27 cm) as shown in Eq. 1and then operating in the range of 0.5-0.7n_c , the minimum and maximum angle was determined according the information in the literature[14]. The minimum and maximum binder percentage were chosen according to reference [15]

Table 3 and table 4 shows the design of experiments to the above three parameters on the IDG and RD operation to find the optimum conditions of operation to get the maximum granulation to the gamma alumina support spheres obtained.

Table 3 Design of experiments to Inclined disc granulator (IDG)

Run 0rder	Inclination angle	rpm	Binder percentage%
1	55	35	150
2	45	48	150
3	55	35	300
4	55	35	140
5	45	41	225
6	50	48	150
7	55	35	300
8	45	48	300
9	55	41	225
10	50	41	225
11	50	48	300
12	45	41	225
13	50	41	225
14	41	41	225
15	50	41	102
16	50	41	225
17	50	52	225
18	50	41	347
19	50	30	225
20	58	41	225

Table 4 Design of experiments to Rotary drum granulator

Run 0rder	Inclination angle	rpm	Binder percentage%
1	0.5	47	225
2	6	47	225
3	6	47	347
4	6	27	225
5	6	47	225
6	6	67	225
7	12	47	225
8	6	47	102
9	6	47	225
10	6	47	225
11	2	60	150
12	2	35	150
13	10	60	300
14	10	60	150
15	6	47	225
16	10	35	300
17	2	35	300
18	6	47	225
19	10	35	150
20	2	60	300

The binder was diluted with 2 parts of distilled water and sprayed by the spray bottle. The basis for calculation the percentage of binder ratio in each batch is 20 gm of gamma alumina powder of the specifications shown above in the materials paragraph. Scraper was used by hand to scrap the powder from the surface of the IDG and RD .Spheres was collected from the IDG and the RD and then sieved to 2 and 3 mm using the ASTM standard sieves is due to the reforming catalysts specification.

Experiments were done to get the spheres of the gamma alumina catalyst support by the method of extrusion – spheronization method by using extrudate made by method of molding which composed of rubbing gamma alumina paste into the aperture of perforated steel plate. The size of granules produced is determined by the thickness of the plate and the diameter of the aperture [17].The extrudate of diameter 3mm was then loaded on the Caleva spheronizer MBS at different speeds and time of rotation to get the optimum condition which give the maximum (spheronization).

The gamma alumina paste used for making extrudate was made by mixing gamma alumina powder with the sodium silicate binder in a quantity of 60%w/w based to gamma alumina powder used and then diluted the mixture with water of quantity equal the sum of the weight of both the gamma alumina powder and the sodium silicate solution used. The speed of mixing is 800 rpm, mixing continue until getting a paste having plasticity properties.

RESULTS AND DISCUSSION

Inclined Disk Granulator

The optimum operating conditions found is as shown below:

1. Fig. 2& 3 show that the best angle of inclination is about 42^0 for the total and the 2mm size granulation.
2. Fig. 4 show the best angle of inclination to the granulation of 1mm gamma alumina is 50^0.
3. Fig. 5 & 6 show the optimum speed of rotation to get maximum total and 1 mm granules is around 31 rpm as shown in Fig. 14 and 15.
4. 4. Fig. 7 show the optimum speed of rotation to get 2 mm granules is 52rpm.

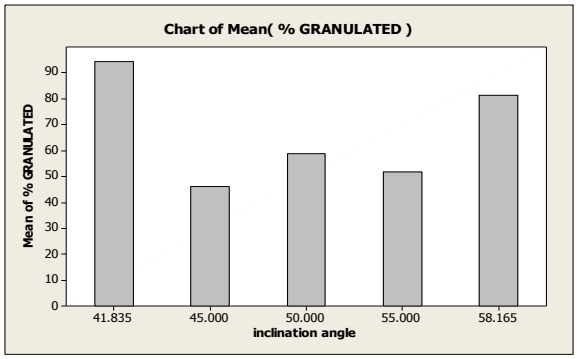


Fig. 2 The total percentage granulated with respect to angle of inclination in IDG.

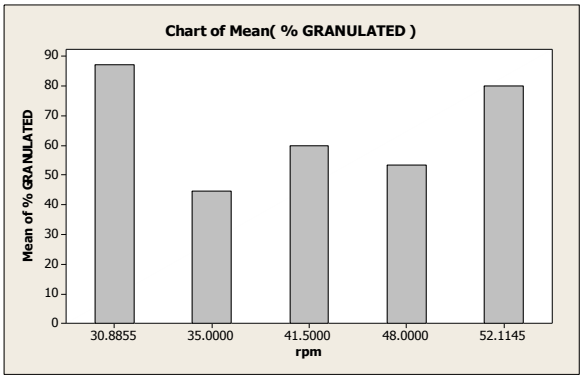


Fig. 5 The total granulated with respect to speed of rotation to IDG.

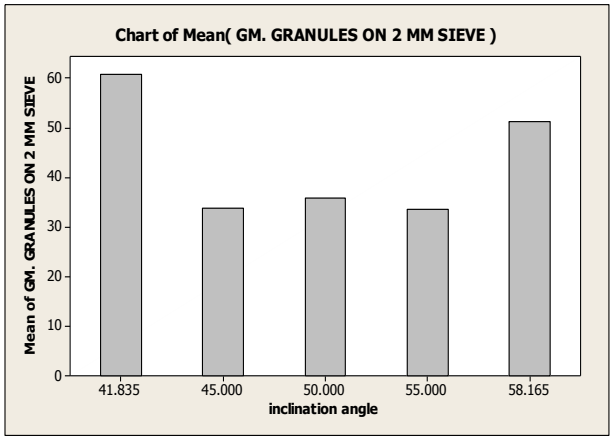


Fig. 3 The percentage 2mm granulated with respect to angle of inclination to IDG.

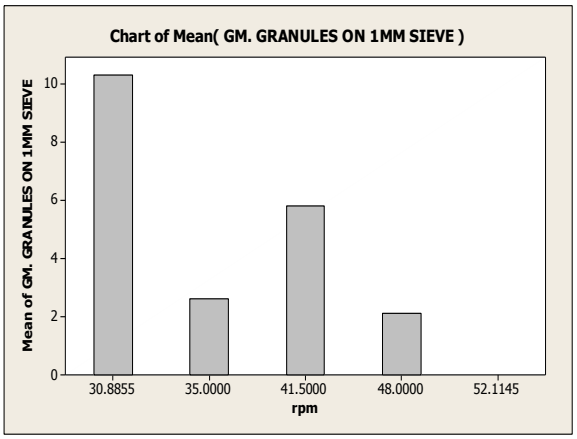


Fig. 6 Percentage of 1mm granulated with respect to speed of rotation.

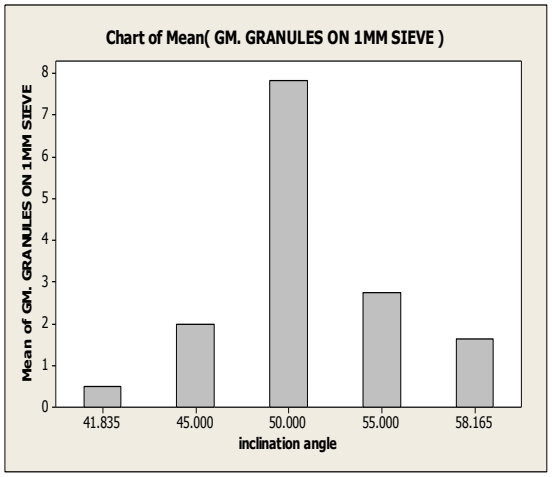


Fig. 4 The percentage 1mm granulated with respect to angle of inclination to IDG.

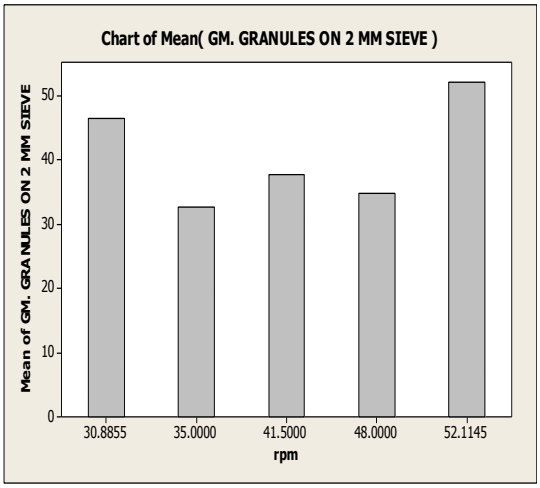


Fig.7 The relationship of rpm and percentage of 2mm granules obtained in IDG.

5. Fig. 8 and 9 show the optimum binder ratio of sodium silicate used to get total and 2mm granules is at 300% based on gamma alumina powder used.

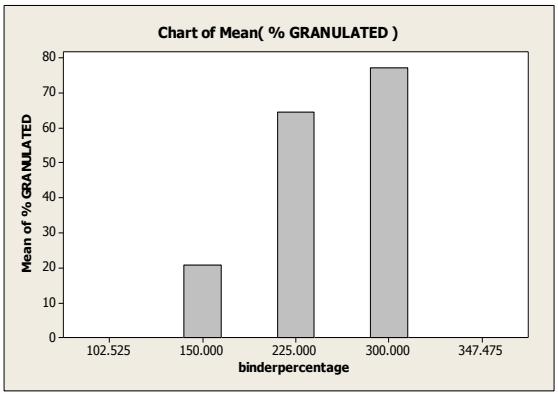


Fig. 8The relationship between the binder percentage and percentage total granulation obtained in IDG.

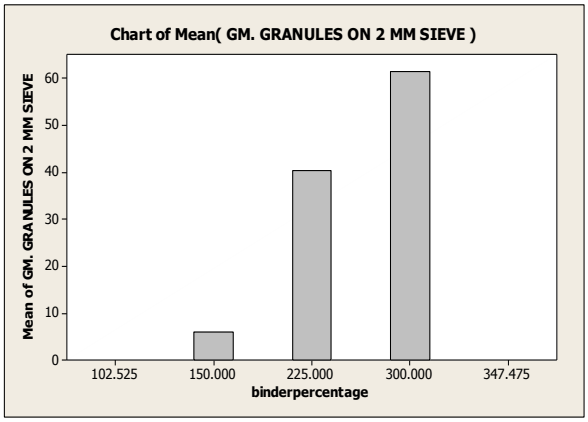


Fig. 9 the relationship between the binder percentage and percentage 2mm granules obtained in IDG.

6. Fig. 10 show the optimum binder percentage to get the 1mm granules is at 225%

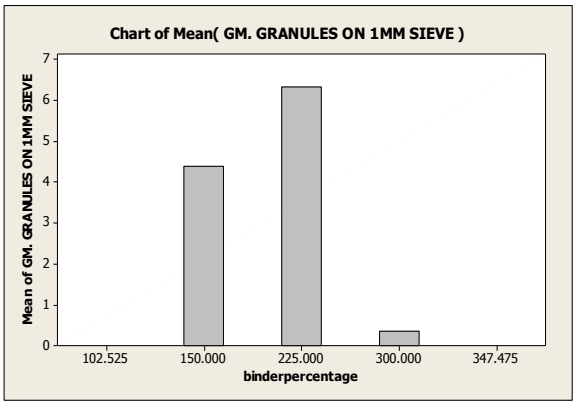


Fig.10 the relationship between the binder percentage and percentage 1mm granules obtained in IDG.

Rotary Drum Granulator

1. Fig .11 Show the optimum angle of inclination to get the total maximum granulation, it is 12.5^0.

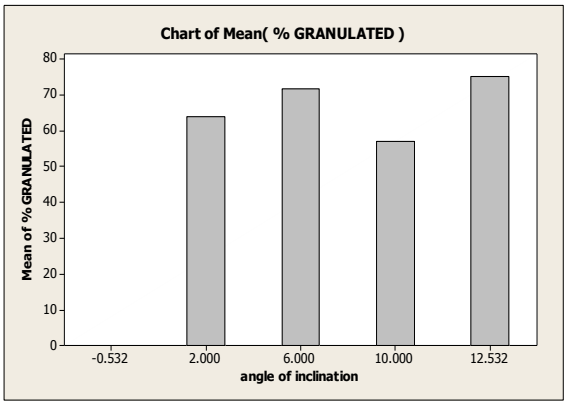


Fig.11 The relationship of angle of inclination to total granulation in the RD.

2.Fig.12 show the optimum angle of inclination to get the maximum 2mm granules in RD Granulator, it is 6^0.

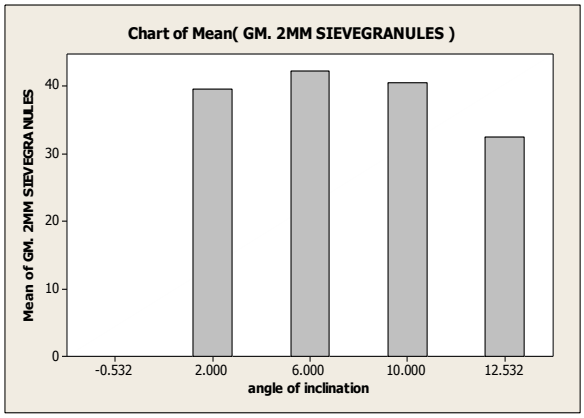
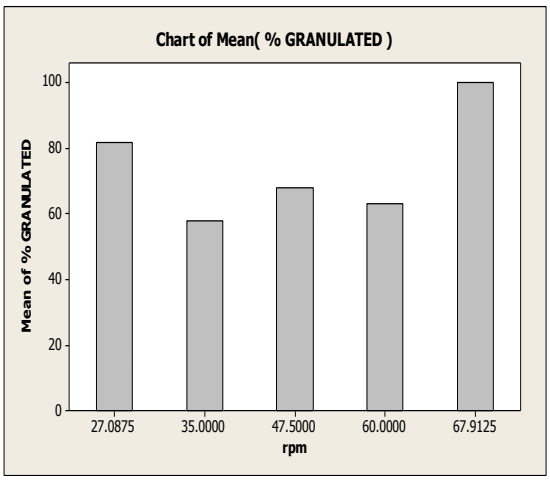


Fig. 12 The relationship of angle of inclination to get the maximum 2mm granules in RD Granulator

3. Fig. 13show the optimum speed of rotation to get the maximum total granulation and 2mm granules in the RD granulator, it is 68 rpm.



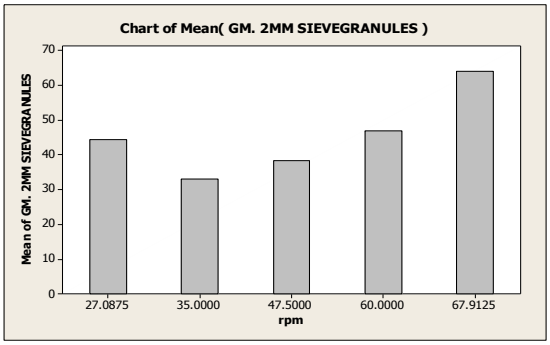


Fig.13 The relationship of speed to total granulation in the RD.

3. Fig. 14 show the optimum binder percentage to get the total rate of granulation in the RD granulator, it is 300%.

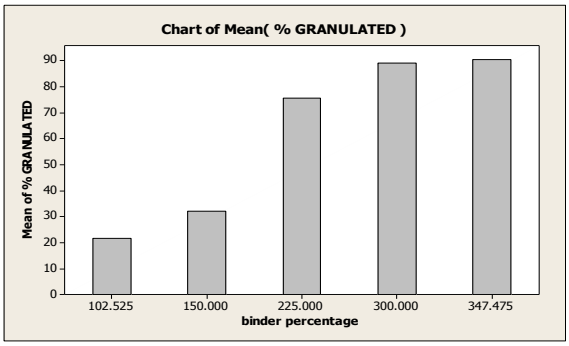


Fig. 14 The relationship of binder percentage to get the maximum total granules in RD Granulator

Caleva spheronizer

Fig. 24 shows at low speed of rotation (800 rpm) of the Caleva spheronizer, the rate of spheronization of the gamma alumina extrudate increased with increasing the time of spheronization. The spheronization percentage will be decreased after definite time because of breaking the spheres formed.

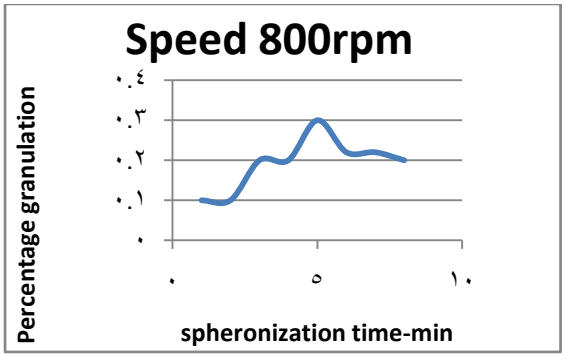


Fig. 15 spheronization of gamma alumina extrudate in Caleva spheronizer

Fig. 16 show high percentage of spheronization at speed of rotation of (1000 rpm & 1200rpm) and at low time (1 min.).

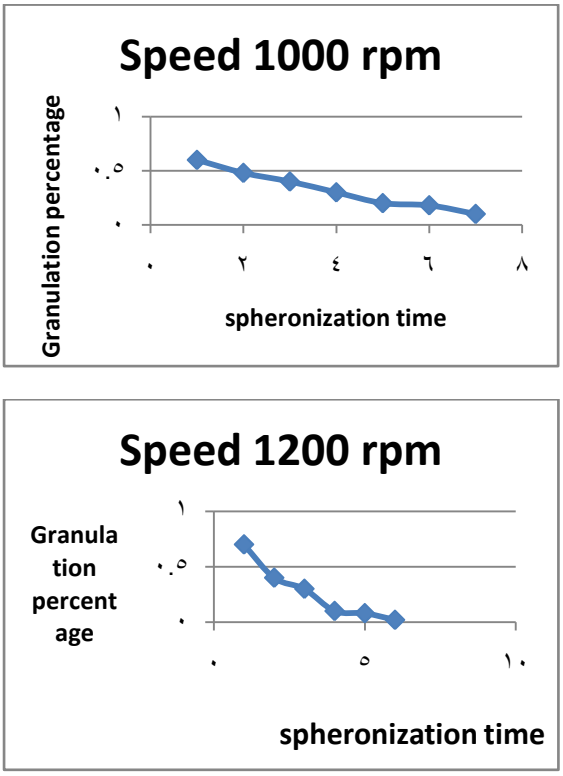


Fig. 16 spheronization of gamma alumina extrudate at 1000rpm.

Fig. 17 show low percentage of spheronization at high speed of rotation 1400 rpm and increasing the time will decrease the percentage of spheronization more.

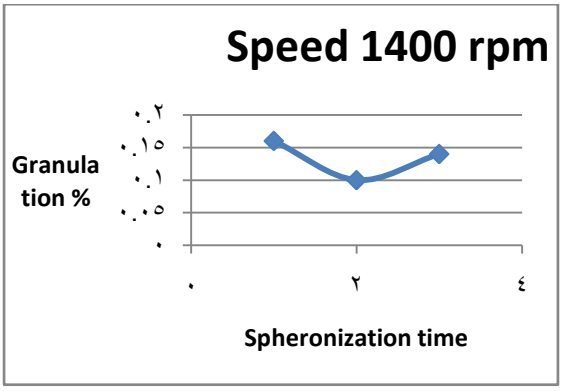


Fig. 17 Rate of spheronization at 1400 rpm speed

CONCLUSIONS

1. The optimum operating conditions in the granulation (Spheronization) of the gamma alumina catalyst support in the Inclined Disk Granulator IDG is:

   a. Inclination angle :42^0 for the total and 2mm size granules , 50^0 for the granulation of 1mm..
   b. Speed of rotation: 31 rpm for the total and 1mm granulation, 52 rpm for the 2 mm granulation.
   c. Binder percentage: 300% for the total and 2mm granulation, 225% for the 1mm granulation.

Note: The 300% binder percentage is really equal to 44% of pure sodium silcate , because The binder was diluted with 2 parts of distilled and its real concentration is 44% water as mentioned in the experimental work. Similar works [8]mentioned that it is in excess of 55% volume (usually 59-73% volume) for closely sized powders and between 40% and 55% volume for materials with wide size distributions .In practice ,however , the water content may range from roughly 90-110% of the critical liquid saturation.

2. The optimum operating conditions to the granulation of gamma alumina in the Rotary granulators(RD) is:

   a. Inclination angle: 6^0 for the granulation of gamma alumina of size 2mm, 12.5^0 for total granulation of gamma alumina catalyst support.

   b. Speed of rotation: 68 rpm for total and 2mm granulation.

c.Binder percentage:300% for total granulation .Please refer to the Note in paragraph 1 above.

3. The optimum operating conditions in the caleva spheronizer to get spheres of gamma alumina catalyst support is:

a. Speed of rotation: 1200 rpm .Maximum spheronization rate (70%) obtained at 1200 rpm speed , while at 800 rpm , only 30% maximum spheronization obtained and only 60% maximum spheronization obtained for 1000rpm.

b.Time of spheronization: The minimum time of spheronization with maximum spheronization rate (1min.) was obtained at 1200 rpm and 1000 rpm ,while 5 min. time of spheronization obtained at 800rpm speed with maximum spheronization rate.

REFERENCES

[1]Irshad,U.,Sharif,M.N,Khan,R.U and Rizivi,Z.H. ,"Granulation of Urea in a Pan Granulator", Journal of Qualty and Technology Management.2010.

[2]www.alibaba.com,Disc pellitizer,Disc granulator,Pan Granulator.

[3]Lister,Jim , Ennis , Bryan, "The Science and Engineering of Granulation Processes",Kluwer Academic Publisher,Netherland,2004.

[4]D.F. Wu,J.C. Zhou and Y.D.Li,AIChE J.,2007,53,2618-2629.

[5]Antos,George J., Aitani, Abdullah M., Parea,Jose M., " Catalytic Naphtha Reforming Science and Technology", Marcel Dekker Inc.,USA,1995.

[6]Van Santen,R.A, VanLeeuwen,P.W.N.M,Moulijin,J. A. and Averill,B.A,"Catalysis:an integrated approach",Elsevier,1993.

[7]Le Page,J. F.,"Applied Heterogenous Catalysis", institute francais du petrole publications,France ,1987.

[8]Ramachandran, R., Jonathan M.H., Constantjin,F.W.Thomas ,G. Charles ,D. Immanuel,Francis,J.,Lister,James D. and Franisek Stepanek," Experimental studies on distrubitions of granular size, binder content and porosity in batch drum granulation:Influences on process modeling requirements and process sensitivities",Powder Technology, 188(2008) 89-101.

[9]Wang,Fu.Y.,Cameron,Ian.T.,"R eview and future directions in the modeling and control of continuous drum granulation",Powder Technology 124(2002)238-253.

[10]PSTT (Pharmaceutical Science & Technology Today) Vol. 2, No. 4 April 1999

[11]Chatlapalli,R.,Rohera,Bhagwa n D., "Physical characterization of HPMC and HEC and investigation of their use as pellitization aids" International Journal of Pharmaceutics 161 (1998) 179–193

[12]Caleva Spheronizer MBS , installation & operation manual.

[13]Kapur,P.C.,"Balling and granulation" in Advances in Chemical Engineering

Vol.10,Academic press Inc.,New York,1978.

[14]Montgometry,"Design and Analysis of Experiments", John Wiley & Sons, 1997.

[15]Jawad,Alaa Dhari,Richeh ,Ibrahim, Saleh,Riad Ahmed,"Effect of the forming conditions in the production of Gamma Alumina Catalyst Support on the crushing strength property",IJCPE,11,2,June 2010 ,43-48.

[16]Mukhlyonov,I.P.,Dobkina, E.I,Deryuzhkina,V.I,Soroko,V.E.," Catalyst Technology" ,MIR Publishers,Moscow, 1976.