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Effect of Process Variables on Growth Kinetics of Lactose Particles in a Wet Spray Fluidized Bed Granulator

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Abstract

The growth kinetics of Lactose particles in a top spray, small scale fluidized bed granulator is studied experimentally and the effects of binder flow rate and concentration, nozzle air pressure and the fluidizing air temperature on the growth kinetics of granules are studied. The results have been explained by the aid of sequentially main effects of each parameter in the process. The effect of each operating variable has been discussed in detail, qualitatively.

Keywords: *Fluidized Bed Granulation, Wetting, Consolidation, Breakage, Size Distribution*

1. Introduction

Fluidized bed granulation (FBG) is a process for producing granules by spraying a liquid binder onto the air-suspended solid powders that has several advantages against other powder processes such as spray drying or shear granulation [1]. The advantage of this unit operation is the coupling of the wetting, drying, particle enlarging, shaping, homogenization and separation processes, and the production in a single processing step [2]. In spite of its widespread use, economic importance and many years of research, granulation has in practice remained more of an art than a science [3]. A number of reviews were published in the past three decades to express the importance of this area of research [3-6]. There are two approaches in describing granulation mechanisms. Traditionally, the process has been described in terms of a number of different mechanisms, such as nucleation, coating and layering, coalescence, abrasion and crushing of particles (See Fig. 1a) [7]. However, in modern approach, granulation is imagined as a combination of three rate processes, namely wetting and nucleation, consolidation and growth, and attrition and breakage [8] (Fig. 1b).

In fluidized bed granulation it is important to know the end point of the process. There are a few publications in this area to find the end point of granulation process, usually with online monitoring techniques [9, 10]. However, knowing the kinetics of size enlargement can contribute to a better

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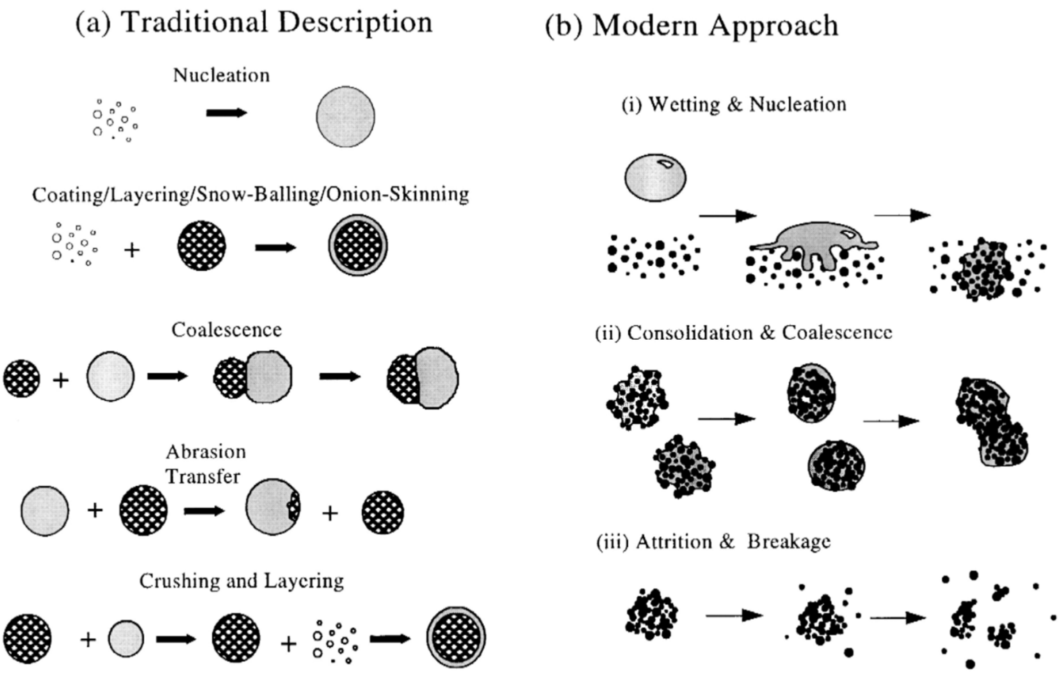

Figure 1. Traditional versus modern approaches on modeling of granulation process kinetics [3] (with permission, License number: 3110750068909).

Understanding of the spray granulation process [11-16]. Also, knowing the effect of different operating variables on the growth rate of particles could be used for controlling the mean size and size distribution of product. Usually, at the initial stages of batch spray granulation, particles grow very fast and then the size of particles remains almost constant or increases with a slight rate. The main reason for the fast growth rate at the early stages is that the numbers of particles per unit volume are considerable and, as a result, the probability of collision of wetted particle increases with number of particles per unit volume [11]. Tan *et al*. [11-16] focused on the kinetics and rate processes that occur in fluidized bed melt granulation (FBMG) process. They demonstrated the rate of different processes that occur in FBMG in a new manner and described their experimental results on kinetic behavior of granulation of glass ballotini by this new approach (Fig. 2). They studied four process variables (i.e., binder spray rate, bed temperature, atomizing air pressure, and fluidizing air velocity) on the granule growth behavior. The granule growth rate has been shown to be directly dependent on the relative amount of binder sprayed into the bed, which essentially determines the speed of the aggregation process. The final granule size distribution was also observed to become narrower with increased bed temperature.

Tan *et al*. [12] also described a sequential events scenario for aggregation and breakage of particles in fluidized bed melt granulation. Fig. 2 shows the mechanism proposed by Tan *et al*. for particle size evolution. As can be observed after an effective collision of a droplet and a particle (event 1), droplet Droplet on particle (rate I) and a film of

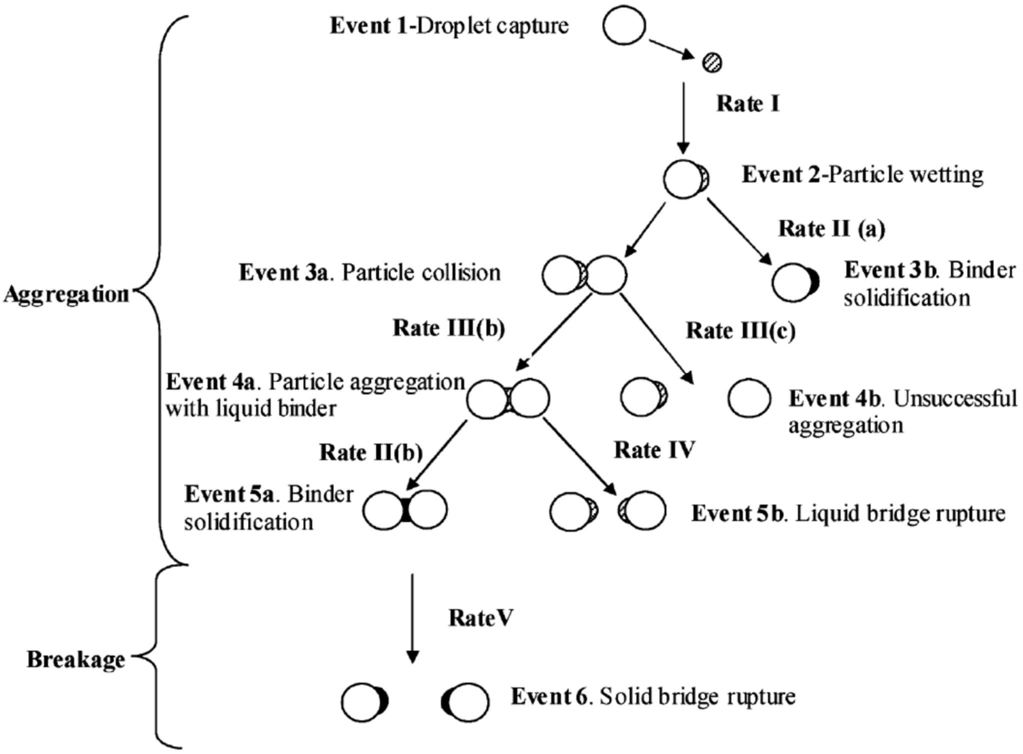

Figure 2. Sequence of events in fluidized bed melt granulation [12] (with permission, License number: 3110740697682).

binder forms on the particle (event 2). This film can be dried because of the bed temperature (rate II) and results in binder solidification (event 3-b) or can collide with other particles (event 3-a). In this state two events could take place: one of them is an effective collision (rate III-c) that results in formation of a liquid bridge between two particles (event 4-a); and the other is separation of particles because of greater kinetic energy of collision than viscous resistance of binder film (rate III-c and event 4-b). New aggregate can survive and its liquid bridge converts to a solid bridge (rate II-b and event 5-a) or disjoints to two particles by liquid bridge rupture (event 5-b and rate IV). New solidified bridge can also rupture because of collisions (rate V and event 6).

In wet spray granulation process, the following parameters can lead to a better growth behavior of particles, clearly:

- good wetting of particles by binder droplets
- effective collision of wetted particles which leads to particles aggregation
- fewer granule breakage effects

The relative size of droplet to particle and the droplet thermo-physical properties such as surface tension and viscosity play important roles in wetting phenomenon. From an operational point of view, the bed temperature, the binder flow rate, the pressure of nozzle air, and the binder properties should be investigated as effective parameters on granule wetting. Ennis *et al*. [17] described a quantitative approach by defining Stokes number as criteria for effective collision of wetted particles as follow:

$$St = \frac{4\rho U_c d}{9\mu} \quad (1)$$

Where ρ is the granule density, U_c is the relative velocity of colliding particles, d is the granule mean size, and μ is the liquid viscosity. It should be noted that when Stokes number is greater than one, kinetic energy of colliding wet particles is greater than viscous dissipation energy of liquid film so coalescence does not occur and adversely when it is less than one, viscous dissipation effect is greater than kinetic energy effect and particles coalesce after colliding.

In the present work the effects of four important variables on growth kinetics of particles in a fluidized bed spray granulator have been studied. These parameters are the inlet air temperature, the binder solution flow rate, the atomizing air pressure, and the binder concentration. The experiments are carried out in a small scale batch fluidized bed granulator (Glatt Co., Model: Uniglatt) with cornstarch solution as binder and lactose particles as powder.

2. Experimental

2-1. Materials

The powder used is α-lactose anhydrate supplied by Lactochem Co., (The Netherlands). The packed density of lactose was 897.2 kg/m^3. The size distribution of the starting powder is shown in Fig. 4 as solid line. Cornstarch powder supplied by Ebnemasooye Co., (Saveh, Iran) is used for preparation of binder solution. In each experimental run, cornstarch solution in water is prepared as binder solution. For each experiment, at least 600 g of lactose and the same amount of cornstarch solution are used, corresponding to an ultimate binder to particle mass ratio (*B/P*) equal to 1.0 (*w/w*).

2-2. Method of analysis

Sizing of the primary particles and the granules is done using sieve analysis with a series of standard sieves supplied by Damavand Co., (Iran) according to the American standards. The range of sieve mesh number is from 20 to 270.

A Brookfield viscometer (model: DV-E, USA) is used to measure viscosity of binder solution at different temperatures and concentrations.

A Krüss tensiometer (model: K6, Germany) is used to measure surface tension of binder solution at different temperatures and concentrations.

2-3. Experimental setup

The experimental setup used in the present study is a small scale, top spray fluidized bed granulator (FBG) supplied by Glatt Co. (model: Uniglatt, Switzerland). The schematic of the experimental setup is shown in Fig. 3. The capacity of the granulator is about 1 kg of powder.

In each experimental run, air flow from blower (1) is heated to the required temperature in an electrical heater (2) before entering fluidized bed granulator. As can be seen in Fig. 3, the fluidized bed granulator has a cylindrical lower section with inner Diameter is equal to 15 cm in lower cylindrical section. The fluidizing air flows through a distribution plate at the bottom of the granulator. The air flow-rate could be

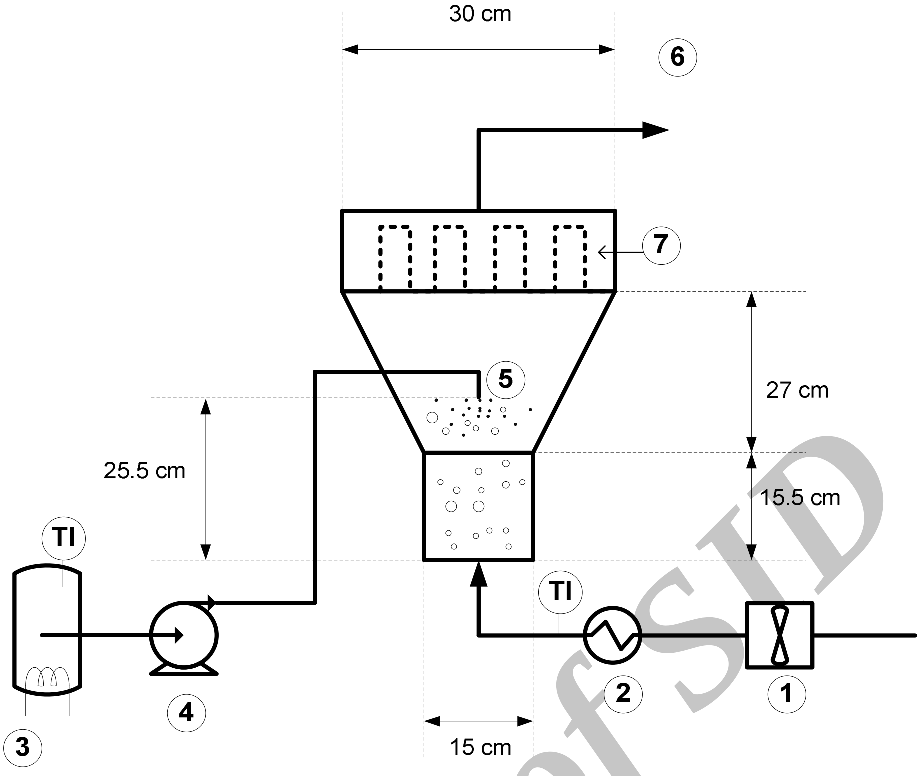

Figure 3. Experimental setup of fluidized bed granulator: (1) air blower; (2) air heater; (3) binder vessel; (4) binder pump; (5) binder nozzle; (6) air outlet connection; (7) air filters.

adjusted by opening the air flap. Monitoring and control of inlet air temperature are achieved by an on/off control system. A thermocouple is provided to monitor air temperature carefully. Bi-fluid type nozzle is used to spray binder solution. The spray nozzle is placed approximately 25.5 cm above the distribution plate. The binder solution is pumped to the spray nozzle by a peristaltic pump with variable rate. The temperature of binder solution is set to the air temperature in each experiment. The bag filters mounted on the air outlet stream are shaken in a predetermined time interval, automatically. Shaking and rest time intervals are variable in different experiments depending on the binder spraying rate. The set-up is equipped with a probe for taking granule sample over time in each experiment. Sizing of the granules is carried out using sieve analysis. It should be noted that in order to check the accuracy and repeatability of the results, some experiments are repeated two times. The results showed a standard deviation of about 1%.

3. Results and discussion

3-1. Size enlargement

Granulation is a size enlargement process in which the size of primary particles is increased by agglomeration in the presence of a liquid as binder. In fluidized bed granulators, binder solution is sprayed on fluidized bed of powders and granulation starts. Particle size distribution of granule versus time is shown in Fig. 4 for a typical experimental run. As can be observed, mean size of granules is increased over time. The same trend can be observed in all experimental runs. It should be noted that there are several criteria for considering mean size of granules. In the present study, the size that half weight of particles lie below, l_{50}, is considered as a representative of mean size of particles.

The main target of the present work is to study the effect of different process parameters on l_{50}. It should be noted that for experimental runs at different binder flow rates, it is much better to compare size of particles as a function of binder solution to particle mass ratio (*B/P*) instead of granulation time. The importance of (*B/P*) parameter is shown in Fig. 5. As can be observed, the size of granules can be compared in the whole range by introducing (*B/P*).

3-2. Effect of binder flow rate

Variation of l_{50} versus (*B/P*) at different binder flow rates is shown in Fig. 5. As can be observed, an increase in binder flow rate from 5 to 20 mL/min, results in a considerable increase in l_{50}. The effect of binder flow rate on particle growth has been studied in several researches and it is generally believed that an increase in binder flow rate has positive effect on particle growth. It should be noted that an increase in binder flow rate results in an increase in the number of droplets per unit volume of granulation bed and, as a consequence, the probability rate of particle-droplet collision increases, which has a positive effect on particle growth.

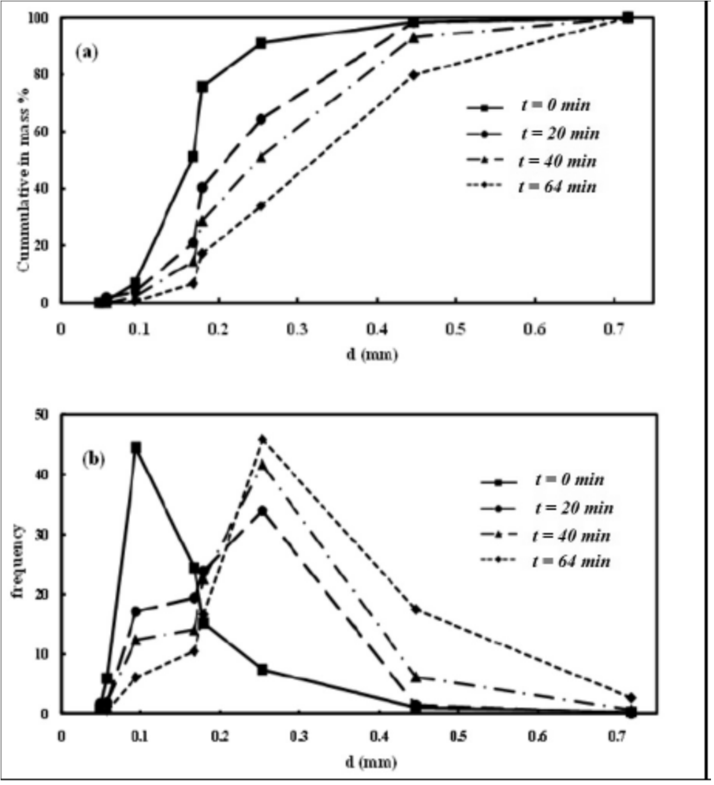

Figure 4. Granule size enlargement in a typical experimental run, (a) cumulative and (b) frequency of size distribution evolution through time (Experimental conditions: *T*= 55 °C, *P*= 2.5 bar, *Q*= 12.5 ml/min, C_{binder}= 3%).

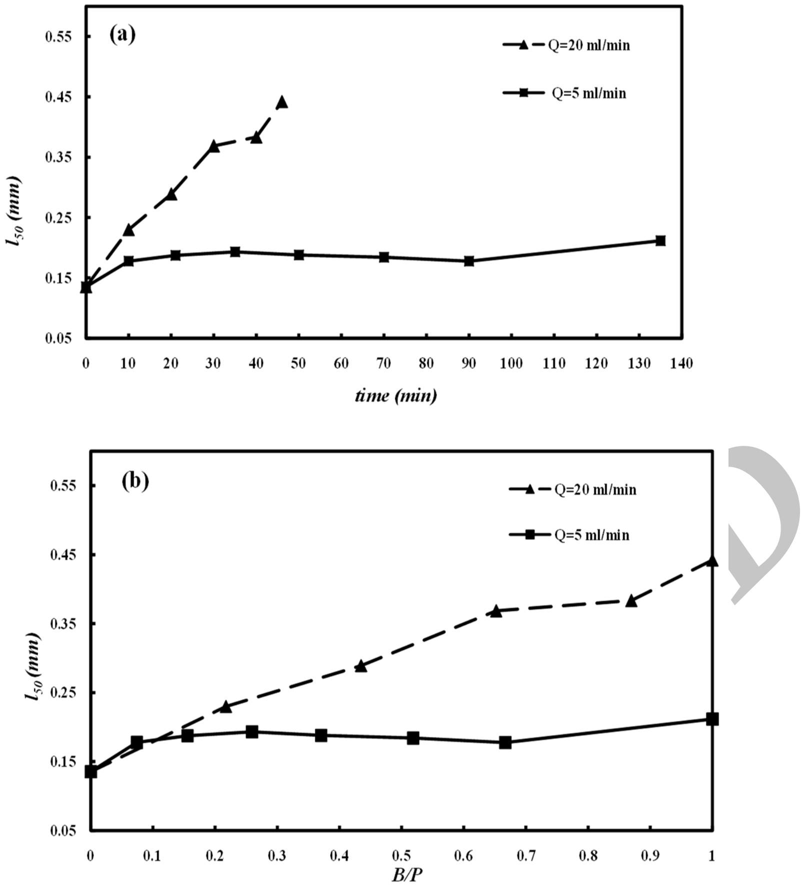

Figure 5. l_{50} versus time (a) and (*B/P*) parameter (b) in typical experimental runs (Experimental conditions: T= 40°C, P= 2.0 bar, C_{binder}= 3%).

3-3. Effect of binder concentration

In Fig. 6, variation of l_{50} versus (B/P) at different concentration of binder solution is shown. As can be observed, an increase in cornstarch concentration in binder solution from 3 to 7%, results in a considerable growth in l_{50}. Main effects of binder concentration on particle growth are shown in Fig. 7. As can be observed, an increase in binder concentration leads to a rise in viscosity of binder solution and an increase in the contact angle between the droplets of binder solution and solid particles, simultaneously. The former results in larger droplet sizes which in turn leads to lower Stokes number and more efficient collisions while the latter leads to weaker particle wetting that in turn has negative effect of particle growth. Physical properties of binder solution are listed in Table 1. A considerable increase in the viscosity of binder solution can be observed by increasing concentration from 3 to 7%.

However, on the other hand, an increase in binder concentration leads to a decrease in solvent evaporation rate from droplet surface as well as a decrease in probability of drying of fine wetted particles and droplets, both of them have positive effects on particle enlargement.

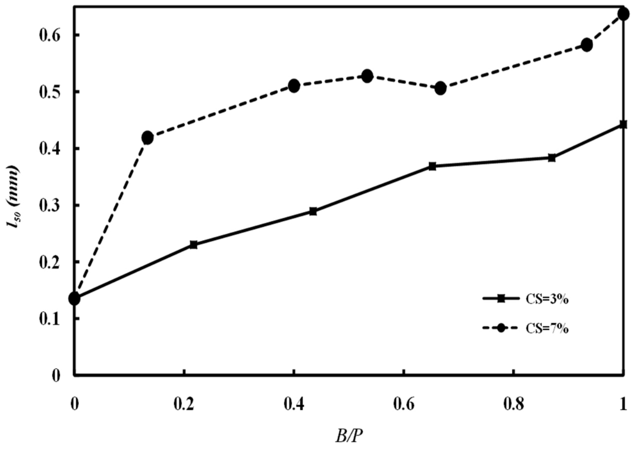

Figure 6. l_{50} versus (*B/P*) at different concentration of binder solution (Experimental conditions: *T*= 40°C, *P*= 1.5 bar, *Q*= 12.5 ml/min).

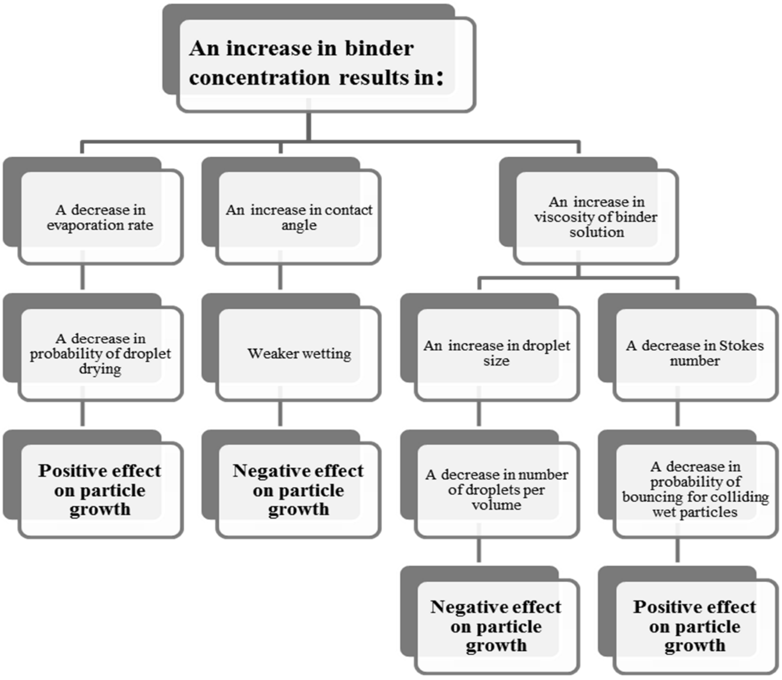

Figure 7. Main effects of binder concentration on particle growth.

Table 1
Physicochemical properties of binder solution at different temperature and mass percent of corn starch.

Corn starch mass percent in binder solution	**7%**	**3%**		
Temperature (°C)	40	40	55	70
Interfacial tension (mN/m)	48	47	42.5	42
Viscosity (mPa.S)	21.5	4.3	4.2	4.1

3-4. Effect of nozzle air pressure

In Fig. 8, l_{50} versus (*B/P*) at different nozzle air pressures are shown. As can be seen, an increase in nozzle air pressure from 1.5 to 2.0 bars, results in a growth in the size of granules but further rise in nozzle air pressure from 2.0 to 2.5 bars results in a reduction in the size of granules. The main effects of nozzle air pressure on particle growth are shown in Fig. 9. It should be noted that the nozzle air pressure affects the droplet size distribution. Ehlers *et al.* [18] found experimentally that the droplet size decreases linearly with increasing air pressure. It should be noted that an increase in nozzle air pressure leads to a decrease in droplet size of binder solution that in turn has three major effects: (1) the probability of surface evaporation is amplified which leads to drying of droplets before colliding with particles that have negative effect on particle growth. (2) The ratio of the thickness of binder on the particle to particle diameter is decreased which leads to an increase in probability of bouncing for collided particles which has a negative effect on particle growth (3). The probability of droplet-particle collision is increased, leading to better particle wetting which has a positive effect on particle growth. It should be noted that the observed trend is dependent on the relative importance of these effects in each case.

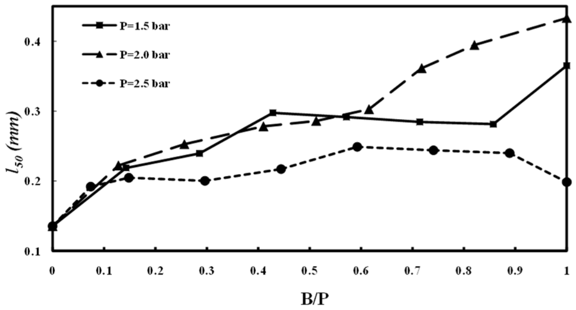

Figure 8. l_{50} versus (*B/P*) at different nozzle air pressure (Experimental conditions: *T*=70°C, *Q*= 12.5 ml/min, C_{binder}= 3%).

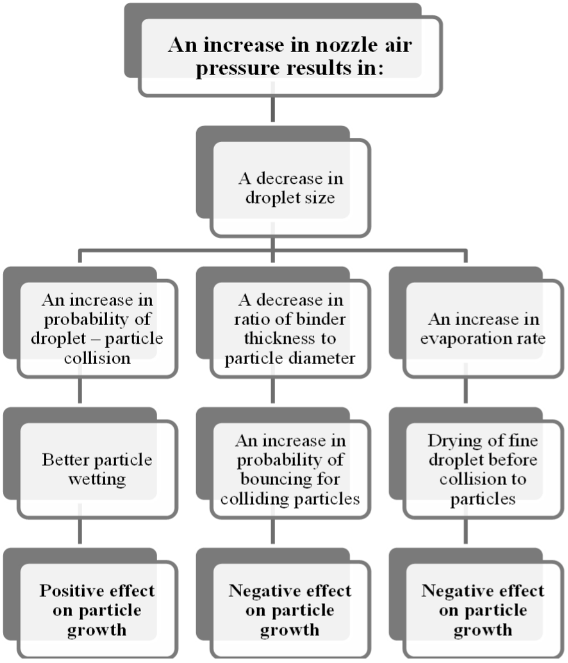

Figure 9. Main effects of nozzle air pressure on particle growth.

3-5. Effect of air temperature

In Fig. 10, l_{50} versus (*B/P*) at different air temperatures is shown. As can be observed, increasing the air temperature from 40 to 55°C results in a decrease in l_{50}, but a further rise in air temperature from 55 to 70°C results in a minor increase in l_{50}. The main effects of air temperature on particle growth are shown in Fig. 11. It should be noted that air temperature has several different effects on granulation process: (1) an increase in air temperature results in a decrease in binder viscosity that in turn leads to lower droplet size. A decrease in viscosity reduces the effect of viscous forces against velocity forces which increases the chance of particle bouncing after collision (2) On the other hand, higher temperatures result in a decrease in surface tension and as a consequence, a decrease in contact angle that finally results in better wetting (3); at higher temperatures binder evaporation rate rises and as a result, the probability of drying increases. Table 1 shows surface tension and viscosity of binder solution at different temperatures. As it was mentioned, surface tension and viscosity of binder have contrary effects on particle growth. It seems the observed trends can be explained by considering higher variation rate of surface tension from 40 to 55°C relative to 55 to 70°C.

4. Conclusions

The following results were obtained in this experimental work:

The experimental results show that the binder flow rate plays an important role in growth rate. Increasing the binder flow rate leads to greater granule size at the same binder to particle mass ratio (*B/P*).

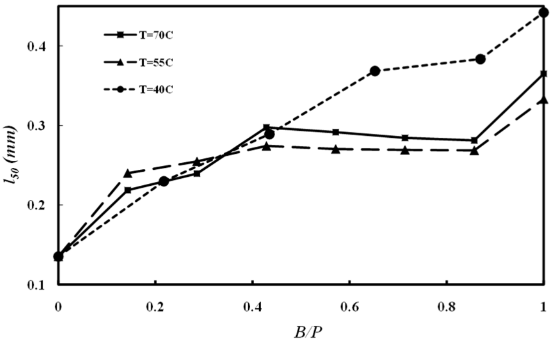

Figure 10. l_{50} versus *B/P* at different air temperatures (Experimental conditions: P= 1.5 bar, Q= 12.5 ml/min, C_{binder}= 3%).

The experimental results show that an increase in cornstarch concentration in binder solution from 3 to 7% results in a considerable increase in l_{50}. An increase in binder concentration leads to an increase in viscosity of binder solution and an increase in the contact angle between the droplets of binder solution and solid particles, simultaneously. The net result of the above mentioned effects leads to the observed trend.

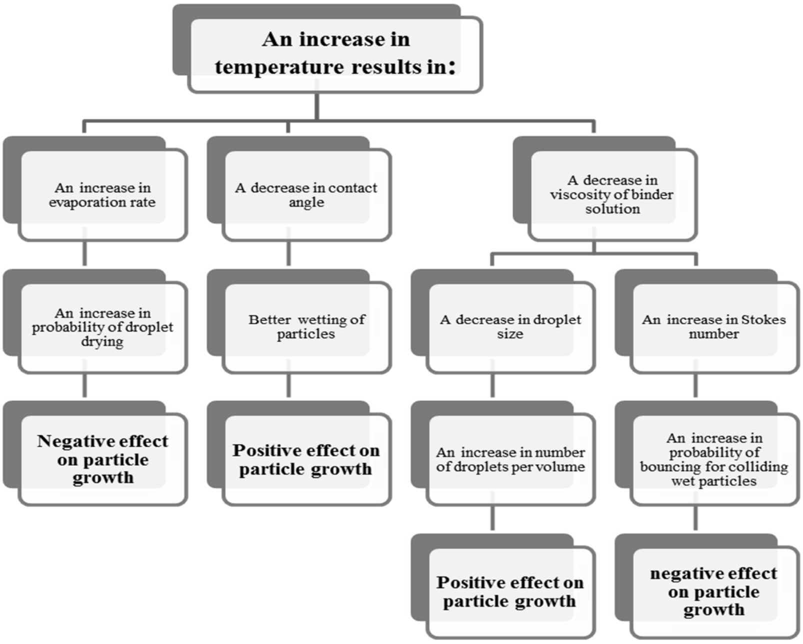

Figure 11. Main effects of air temperature on particle growth.

An increase in nozzle air pressure from 1.5 to 2.0 bars, results in an increase in l_{50} but further increase in nozzle air pressure from 2.0 to 2.5 bars, results in a decrease in l_{50}. Clearly, the droplet sizes decrease with rising nozzle air pressure. It should be noted that an increase in nozzle air pressure leads to a decrease in droplet size of binder solution that in turn has three major effects: (1) The probability of surface evaporation is increased which leads to the droplets drying before colliding with particles, that has a negative effect on particle growth. (2) The ratio of binder thickness on particle to particle diameter is decreased which leads to an increase in probability of bouncing for collided particles and this has a negative effect on particle growth. (3) The droplet-particle collision becomes more probable which leads to a better particle wetting and thus, a positive effect on particle growth. The observed trend is determined by the relative importance of these effects.

It has been observed that an increase in air temperature from 40 to 55°C, results in a decrease in l_{50} but further increase in air temperature from 55 to 70°C, results in a minor increase in l_{50}. This observation is explained by the push-pull effect of different outcomes of temperature increase in a fluidized bed granulator.

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Nomenclature

d	Particle diameter [m]
Uc	Collision velocity [m/s]
St	Stokes Number [-]
ρ	Granule density [kg/m^3]
μ	Binder viscosity [kg/m.s]

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