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## AGGLOMERATE BEHAVIOUR IN FLUIDIZED BEDS

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### ABSTRACT

Fluidized bed agglomeration is used to stabilize particulate mixtures and reduce dust emissions. Agglomerates and granules that do not conform to size and shape specifications may create problems in downstream processes such as tableting, thus compromising process efficiency and product quality. The objective of the present study was to determine the critical agglomerate liquid content at which the rates of agglomerate growth and shrinkage are balanced when artificial agglomerates made from glass beads and water are introduced into a fluidized bed. This study investigated the effects of agglomerate size and fluidizing gas velocity on the critical initial liquid content. It was found that small agglomerates displayed higher critical initial moisture contents. The study also found that as the superficial gas velocity increased, the agglomerates started to break, rather than erode.

### INTRODUCTION

Particulate operations play a very large role in many industries and their performance is essential to the success of many processes. In industries such as the pharmaceutical industry, agglomerates are intentionally produced (1). In other processes such as fluid coking, however, agglomerates are not desired (2). Agglomerate properties can potentially be manipulated to ensure survivability, if they are desired, or to enhance destruction if they are undesired.

The objective of this study was to investigate how factors such as agglomerate size and superficial gas velocity affect the stability of the agglomerates and, specifically, their critical initial moisture content. Agglomerates containing a moisture level above this critical value will survive the fluidized bed conditions, whereas those with moisture contents below this critical value will begin to be destroyed in the fluidized bed. Water and glass beads, which is not a naturally agglomerating system, were used in this study (3). Some researchers have examined the effects of different parameters on agglomerate growth (4). Our study differs in that initial agglomerate parameters were controlled to examine the agglomerate stability and destruction.

### **Agglomerate Growth**

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Agglomerates are formed by the aggregation of particulate solids that are held together by short-range physical or chemical forces acting among particles, by chemical or physical modification of the particles triggered by specific process conditions, or by substances that act as binders by adhering physically or chemically to form material bridges among particles (5).

Many studies have been done to examine the effect of different parameters on the growth of agglomerates in granulating processes. Several of these studies have attempted to model the growth of agglomerates (4, 6). An important aspect of these studies is defining the different growth mechanisms of granules. Because of the arbitrary nature of earlier classifications, a more recent classification of agglomerate growth mechanisms is wetting and nucleation, consolidation and growth, and breakage and attrition (6). Attempts at modeling the agglomeration process are now considering simultaneous aggregation and breakage behaviour (7). More work is required to be able to fully understand the fundamentals of the agglomeration process and manipulate it to increase product consistency.

### **Agglomerate Destruction**

Controlling particle and agglomerate sizes is important for many industrial applications. Difficulties can arise when unintentional attrition by impact occurs, causing problems in the process due to degradation of particles and granules (8)

In a study by Salman et al. (8), wet granules were studied using impact tests. For low impact velocities, plastic deformation was observed in wet granules. At high impact velocities, wet granules were greatly reduced in size due to fragmentation. This study also found that small wet granules showed significant plastic deformation before failure. This differed from large wet granules, which exhibited localized debris formation and chipping (8). Salman et al. (9) also found that increasing granule size caused agglomerate breakage to occur at lower impact velocity in impact studies of fertilizer granules. Breakage patterns and failure modes are dictated by the agglomerate structure and the velocity of the agglomerate impact (8, 9, and 10). Researchers have found that it is very difficult to model agglomerate failure because of the complex processes that are occurring, as well as the difficulty accounting for the structure of the agglomerate material (10). Moreno-Atanasio and Ghadiri (11) used a mechanistic model that relates the number of broken contacts in an agglomerate due to impact velocity, properties of the particles that form the granule, and the interparticle adhesion energy to study breakage. More study is required to fully understand agglomerate destruction mechanisms.

## **MATERIALS AND METHODS**

### **Agglomerate Preparation**

A syringe-piston system was used for agglomerate fabrication. Known masses of solids and liquid were combined in a container and thoroughly mixed. A constant mass of wet solids was placed in the syringe-piston system and compressed to a predefined length. To test the effect of agglomerate size on agglomerate stability, three molds were used. The largest mold had a diameter of 0.0169 m and a height

of 0.0169 m, the smallest mold had a diameter of 0.0067 m and a height of 0.0070 m, and the intermediate mold had a diameter of 0.0116 m and a height of 0.0120 m. One of these molds was placed inside the syringe and the piston was fully compressed. The mold was pushed out of the end of the syringe and removed, leaving an agglomerate sitting on the bench top. For this study, the agglomerates were made with glass beads and water. The particle density of the spherical glass beads was  $2500 \text{ kg/m}^3$  and their Sauter-mean diameter was  $179 \text{ }\mu\text{m}$ . On average, agglomerates were made with a density of  $1520 \text{ kg/m}^3$ . The agglomerates that were made were very reproducible. They had the same volume each time and Table 1 shows the average mass before fluidization ( $m_o$ ) for each size of agglomerate.

Table 1. Average agglomerate mass and standard deviation before fluidization,  $m_o$ .

Agglomerate Diameter (m)	Average Mass Before Fluidization (g)
0.0067	$0.37 \pm 0.01$
0.0116	$1.93 \pm 0.02$
0.0169	$5.86 \pm 0.04$

### **Fluidized Bed Experiments**

A bed formed with the same glass beads as the agglomerates was fluidized in a clear column with an internal diameter of 0.10 m with compressed air through a distributor consisting of a polyethylene disk with  $70 \text{ }\mu\text{m}$  pores. The static bed height was approximately 0.15 m. A large expansion section above the bed helped return entrained particles back to the bed.

Agglomerates were weighed to determine their initial mass,  $m_o$ . All agglomerate masses were determined using an accurate scale for good precision. They were then inserted into the bed at approximately minimum fluidization conditions. A second fluidization line was then opened for fluidization at the desired superficial velocity for a predetermined time interval. The fluidization air was turned off, the expansion section of the column was removed, and the bed contents were emptied. Only one agglomerate was inserted into the bed at a time. The agglomerate was emptied onto a pile of particles outside of the bed and it could be seen if the agglomerate fractured because of fluidization or the emptying process. Very few were fractured because of the emptying process. The agglomerates that had survived fluidization were recovered and weighed to determine their mass after fluidization,  $m$ . If the agglomerates fragmented during fluidization, the mass of all of the agglomerate fragments were weighed to determine their cumulative mass,  $m_{\text{fragments}}$ . The number of agglomerate fragments was also estimated,  $N_{\text{fragments}}$ .

## **RESULTS AND DISCUSSION**

### **Fluidized Bed Experiments**

Experiments were conducted to determine a critical initial moisture content, where the mechanisms of agglomerate destruction and agglomerate growth are balanced and no net change in the agglomerate mass is observed ( $m/m_o=1$ ). This critical moisture content was determined by graphing the ratio  $m/m_o$  as a function of the initial moisture. The superficial gas velocity of  $0.17 \text{ m/s}$  ( $U/U_{mf} = 6.5$ ) was chosen because it was used in a previous study to observe the effects of different

agglomerate properties on stability (12). The maximum fluidization time was selected to be 90 seconds because the behaviour of the largest and medium sized agglomerates did not change greatly after 60 seconds as shown in Figure 1.

### Effect of Agglomerate Size

The effect of agglomerate size on the critical initial moisture content was studied using three mold sizes. The agglomerates were fluidized at  $U = 0.17$  m/s for different time intervals. The results are shown in Figure 1.

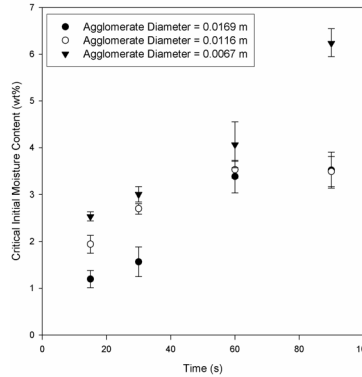


Figure 1. Effect of agglomerate size on critical initial moisture content where  $U = 0.17$  m/s. The error bars are 95% confidence intervals for each value.

Figure 1 shows that for short residence times in the fluidized bed, the critical moisture content required for the agglomerate to maintain its initial mass decreases with increasing agglomerate size. As the agglomerate residence time in the fluidized bed increases, the critical initial moisture content increases. When the residence time is 60 seconds, the increasing trend levels off and the critical initial moisture content becomes the same for both large and medium agglomerates. For fluidization times greater than 60 seconds, the medium and large agglomerates reach a dynamic equilibrium between the addition of solids and the erosion of solids, although they eventually slowly lose moisture. Therefore, the agglomerate size will then slowly decrease with time as erosion acts on the agglomerate structure. The critical initial moisture content for the smallest agglomerate size is different from the other two sizes and shows an increasing trend over the times studied.

At the superficial gas velocity of 0.17 m/s ( $U/U_{mf} = 6.5$ ), the main mechanism of agglomerate destruction was erosion. The data of Figure 1 suggest that the smallest agglomerates under these conditions required more moisture in the agglomerate structure to overcome the erosion forces. This finding disagrees with the findings of Tardos et al. (13), who found that a granule that is larger than a critical value will become unstable and fragment. Fragmentation was the behaviour observed by Tardos et al. (13), and this mechanism of reduction may be affected by agglomerate size differently than the erosion process. In the large agglomerates, it is likely that there is enough liquid within the agglomerate to provide sufficient moisture on the agglomerate surface to incorporate bed particles into the agglomerate structure and compensate for erosion forces acting on the agglomerate over a longer period of time than for the smallest agglomerates. Small agglomerates have a larger surface to volume ratio and the rate of erosion is larger while the amount of liquid available

to bind bed particles is smaller. They therefore require more liquid in their structures to overcome this effect.

### Effect of Superficial Gas Velocity

To determine the effect of superficial gas velocity on agglomerate stability, agglomerates were made using the large agglomerate mold. Three agglomerate moisture contents were tested: 9.1 wt%, 4.8 wt%, and 1 wt%. Agglomerates were fluidized in a bed of glass beads at different superficial gas velocities. These superficial gas velocities were 0.17 m/s ( $U/U_{mf} = 6.5$ ), 0.26 m/s ( $U/U_{mf} = 10.0$ ), 0.31 m/s ( $U/U_{mf} = 11.9$ ), 0.34 m/s ( $U/U_{mf} = 13.0$ ), and 0.40 m/s ( $U/U_{mf} = 15.4$ ).

When the superficial gas velocity was low, agglomerates were seen to erode if the moisture content was low, or gain mass if the moisture content was high. This was the dominant mechanism of agglomerate behaviour that was observed when examining the effect of agglomerate size in the previous section. When the velocity was high, agglomerates were seen to fracture into several smaller pieces. When this occurred, all of the agglomerate fragments were weighed to get their cumulative mass ( $m_{fragments}$ ). The number of fragments ( $N_{fragments}$ ) was also estimated. The relationships between  $m_{fragments}/m_o$  and superficial gas velocity and the number of agglomerate fragments and superficial gas velocity are shown in Figure 2(A) and 2(B) respectively.

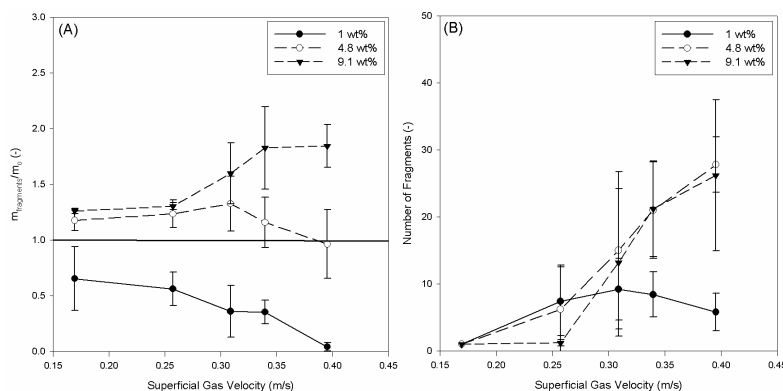


Figure 2. (A) Relationship between  $m_{fragments}/m_o$  and the superficial gas velocity. (B) Effect of superficial gas velocity on the number of agglomerate fragments. Agglomerates were made using the largest mold (diameter = 0.0169 m). Measurements were taken after 30 s of fluidization.

Figure 2 (A) shows that at the lowest superficial gas velocity, agglomerates with 1 wt% moisture content lose mass and agglomerates with higher moisture contents gain mass from the fluidized bed. Figure 2 (B) shows that at the lowest superficial gas velocity, all agglomerates remained in one piece. As the superficial gas velocity increases to approximately 0.26 m/s, Figure 2 (B) shows that the number of agglomerate fragments increases for agglomerates made with moisture contents of 1 and 4.8 wt%, while agglomerates with the highest moisture content showed no increase in the number of fragments. Agglomerates with a moisture content of 4.8 wt% have enough moisture at the surface of the fragments to continue to recruit more bed particles than are lost to erosion, shown by  $m_{fragments}/m_o$  greater than 1 in Figure 2 (A). As the velocity increases further to approximately 0.31 m/s, the

agglomerates with the highest moisture content begin to fragment as shown in Figure 2 (B). The fragmentation of these agglomerates causes the  $m_{\text{fragments}}/m_0$  ratio to increase as shown in Figure 2 (A) indicating that these agglomerates have enough moisture at the surface of the fragments to recruit more bed particles than agglomerates with lower moisture contents. Agglomerates made with the lowest moisture content continue to lose mass as erosion is dominant over particle recruitment. As the velocity continues to increase, agglomerates with 4.8 wt% and 9.1 wt% moisture contents fragment and have very similar numbers of fragments. Agglomerates with the lowest moisture content continue to exhibit fragmentation behaviour, however, the number of fragments is lower compared with the other agglomerates and also eventually starts to decrease. This behaviour may be caused by the complete destruction of the smallest fragments of these agglomerates, decreasing the number of fragments observed.

Examples of the erosion and fragmentation of large and small agglomerates are shown in Figure 3.

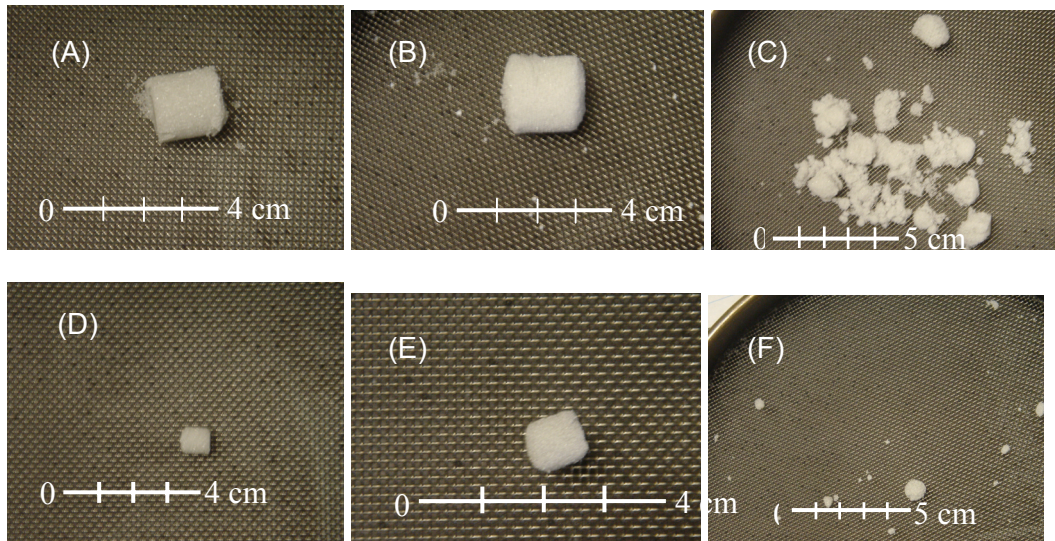


Figure 3. Examples of large (0.0169 m) and small (0.0067 m) agglomerate behaviour in fluidized beds. Moisture content was 9.1 wt%. Large agglomerate (A) before fluidization, (B) after fluidization at 0.17 m/s (erosion), and (C) after fluidization at 0.40 m/s (fragmentation). Small agglomerate (D) before fluidization, (E) after fluidization at 0.17 m/s (erosion), and (F) after fluidization at 0.40 m/s (fragmentation).

Two destruction processes are occurring simultaneously in the fluidized bed. As fragmentation is occurring, the fragments are also undergoing erosion. This contributes to decreasing the  $m_{\text{fragments}}/m_0$  ratio for the agglomerates with moisture contents of 1 wt% and 4.8 wt%. Erosion has a large impact on the decrease of the  $m_{\text{fragments}}/m_0$  ratio for the driest agglomerates at higher velocities, accounting for the low values of this ratio and the decreasing number of fragments at the highest superficial gas velocity.

Salman et al. (8) examined the impact failure modes of spherical particles, including wet agglomerates. Although impact destruction in this study is different than the mechanism of destruction in the fluidized bed, the fragmentation of the agglomerates

in the fluidized bed looked very similar to the high velocity failure of wet agglomerates in this study (see Figure 3) (8). Lower moisture contents in the fluidized bed study caused the agglomerates to be more susceptible to fragmentation because granule strength depends on local agglomerate structure (10). With less moisture available in the agglomerates, fewer liquid contact points can be maintained within their structure, causing them to be weaker than agglomerates made with more liquid.

## CONCLUSIONS

Agglomerate size was found to influence the moisture content where the processes of agglomerate destruction and growth were balanced at a particular time. Larger agglomerates had lower critical initial moisture contents than smaller agglomerates. Agglomerates with moisture contents higher than these critical values will grow in size while agglomerates with moisture contents lower than these critical values will decrease in size when the erosion mechanism of agglomerate destruction is dominant.

The effect of fluidization velocity was also observed to see how this factor affects agglomerate stability. The  $m_{fragments}/m_o$  ratio remains high as the superficial gas velocity is increased for agglomerates that have the highest moisture content. As the superficial gas velocity increases, the number of agglomerate fragments continues to increase for agglomerates made with 9.1 and 4.8 wt% moisture contents. Agglomerates made with 1 wt% moisture content showed low  $m_{fragments}/m_o$  ratios and few agglomerate fragments at the highest superficial gas velocity.

The results presented in this paper were obtained using a glass beads/water system. It is unknown to what extent these results can be applied to other systems. More work is required in this area.

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## NOTATION

$m$	Mass of agglomerate after fluidization (g)
$m_{fragments}$	Mass of fragments after fluidization (g)
$m_o$	Mass of agglomerate after agglomerate formation (g)
$N_{fragments}$	Number of fragments after fluidization
$t$	Time (s)
$U$	Superficial gas velocity (m/s)
$U_{mf}$	Minimum fluidization velocity (m/s)

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