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An Experimental Investigation on Seeded Granulation of Detergent Powders

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Granulation is commonly used as an enlargement process of particles produce granules with desirable characteristics and functionality. Granulation process transforms fine powders into free-flowing, dust-free granules with the presence of liquid binder at certain operating conditions. The main focus of this research is on seeded granulation of detergent powders, a new phenomenon of granulation in which a layer of fine powders surround the coarse particle. This is already proven for calcium carbonate (Rahmanian et al., 2011). Here, detergent granules were produced in a 5 L high shear Cyclomix granulator using different fine/coarse powder ratio (1/3, 1, 3) and different binder ratio of 10 %, 20 % and 30 %. The granules were then characterized for their particle size distribution, strength and structure. It was found that a high percentage (70 wt. %) of granules in the desired size range between 125 - 1,000 μm were produced using the powder ratio of 1/3 and a binder content of 10 %. Low mean crushing strength (3.0 N) with a narrow distribution was obtained using this condition. Structure characterization of the detergent granules produced in the granulator shows that consistent seeded granule structures are produced under the optimum process and formulation conditions of 1/3 powder ratio with 10 % binder.

1. Introduction

Granulation is a widely-used unit operation in many industrial sectors for manufacturing a wide range of products such as food, fertilizers, metaliferous ores, nuclear fuels, ceramics, carbon black, catalyst, pharmaceuticals, agrochemicals, plastics, cement kiln feeds and detergents. Granulation converts fine powders into granular materials with controlled physical properties. A wide range of techniques have been used to form agglomerates with ~100 μm to 20 mm in size (Litster and Ennis, 2004) and different structure from loose aggregates to dense compacts (Hassanpour et al., 2008). As Simons and Fairbrother (2000) pointed out, liquid binder is used in granulation to form interparticle bonds and agitates the powder-liquid mass to promote dispersion and granule growth. The complexity of granulation process accompanied by several complicated physical phenomena occurring in the granulation vessel enables the formation of the granules. In the form of powder, the handling, storing and shipping of powders are the main problems in chemical industry. To avoid and reduce these problems, the granulation process is an excellent alternative for powder formation. The process improves flowability, reduces dust, decreases cross contamination and permits the handling of powder blends without loss of homogeneity. However, recent research shows that a new phenomenon of granulation, whereby a layer of fine powders adhere onto the coarse particle (nuclei/seed), has emerged and is called seeded granulation (Rahmanian et al., 2011). It is believed that this new phenomenon can produce granules with the consistent physical and mechanical properties and can also improve significantly the granule properties in term of flowability, bulk density, porosity, strength and so on.

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2. Experimental Design

The hypothesis of mechanism of seeded granulation (Rahmanian et al., 2011) is fully based on the concept of granulation (Litster and Ennis, 2004). Seeded granulation is a special way of the granulation where fine and coarse powders are mixed together with the presence of a liquid binder to form seeded granules at certain operating conditions. Here, the coarse powder always remains as the core of granule/nuclei and fine powder is layered onto the core of granule.

2.1 Particle Size Analysis

Size of granules were characterized by British standard sieve, which consists of a woven wire screen, with square (greater than 1 mm) or round (less than 1 mm) apertures, rigidly mounted in a shallow cylindrical metal frame (Endecotts Ltd.). Eleven sieves with apertures of 125, 180, 250, 355, 425, 500, 600, 1,000, 1,400, 2,800 and 3,350 μm were used for sieving analysis of granules. The 500 μm and 600 μm aperture sieves were mainly focused for further analysis of strength and structure properties. The desired aperture sizes were stacked from the largest aperture size on the top to the smallest aperture size at the bottom. The sieve shaking machine was operated for approximately 10 min each time with the shaking frequency of approximately 6 Hz. Sieved granules in each tray were weighed using an accurate electronic mass balance. The granule size distribution was constructed and plotted according to the sieve range. The analysis was repeated for all seeded granule samples.

2.2 Strength Analysis

The strength of the granules was measured using a Biomomentum Mach-1^{TM} Micromechanical System (Canada). Calibrating of the load cell was carried out periodically to ensure accuracy of the obtaining data. The loading rate was set at 0.05 mm/s and the plastic cylinder with a metal platform was placed and screwed underneath the load cell. The positions of the load cell, camera and plastic cylinder with metallic platform were suitably adjusted. Data acquisition was carried out at 100 Hz and the vertical z-axis and low velocity were set on manual controls throughout the experiment. Once the setup and operating conditions were set, the sample was placed on the plastic cylinder with metallic platform and underneath the load cell such that it could be viewed using a digital camera attached to the system. The load cell zero position was adjusted, until it just touched the sample’s surface. Every single granule was analyzed and 30 – 40 granules per sample were tested to obtain reliable results. The mean and standard deviation of the crushing strength were calculated.

2.3 Structure Analysis

Internal structure of the same batch of granules i.e. 500 – 600 μm were viewed by Scanning Electron Microscopy (SEM), using the FEI Quanta 400 coupled with Oxford instruments INCA X-Sight (Oxford Instruments, UK). Three steps of sample preparation, moulding, polishing and coating, were used for structure analysis. In the moulding step, volume ratio of 1 of EpoThin^{TM} 2 Epoxy hardener was mixed with 2 of EpoThin^{TM} 2 Epoxy resin in a beaker. The sample was put into a deformable plastic mould and the sample was gently shaken. Then, the resin with hardener was poured into the deformable plastic mould holding the sample. It was then left to harden for 24 h and then taken out for polishing. In the polishing process, 600 grit of carbimet paper discs was used and placed on the Buehler Metaserv motopol 12’s grinding plate. The surface of the resin with the sample was then polished on the carbimet paper discs for 5 min. The 600 grit of carbimet paper discs was then replaced with one of 400 grit and the sample polished for a further 5 min. The sample was checked regularly using a James Swift microscopy until the end of the polishing process. Afterwards, the resin’s surface with sample was placed on the Buehler AutoMet 250 grinder-polisher sample holder and polished using 9 μm diamond paste. Here, the sample holder was set to clockwise rotation whereas its grinding plate was set to anticlockwise rotation, both rotating at 40 rpm for 8 min for each sample. After this, subsequent polishing was carried out using 3, 1 and 0.25 μm paste with the same setup. Next, the well-polished resin’s surface with sample was coated with a gold film using the Emitech K550 gold coating machine prior to SEM analysis.

3. Results and Discussions

Based on the methodologies described above, the following results were obtained and discussed:

3.1 Effect of Powder Ratio (Fine: Coarse) at Constant Binder Percentage (10 %)

Powder ratio is defined as the weight of fine to coarse powder. As shown in Figure 1, when the powder ratio decreases, the trends shifts towards heterogeneous size distribution, formation of fine powder, tiny granules decreases and formation of larger granules increases.

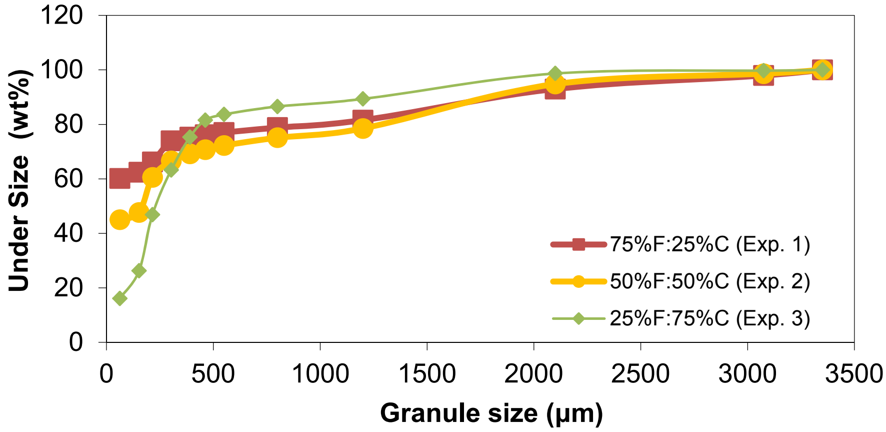

Figure 1: Granule size distribution at different powder ratio and constant 10 % of binder

Figure 2 shows that the powder ratio of 3 (75 %F: 25 %C) has a higher value (3.16 N) of mean crushing strength compared with powder ratio of 1 and 1/3, which have average crushing forces of 2.57 N and 3.00 N respectively. However, powder ratio of 1/3 has the lowest standard deviation of average crushing strength compared with those of powder ratios of 3 and 1 (0.95 N and 0.74 N respectively). This indicates that the higher the powder ratio, the greater the standard deviation of mean crushing strength.

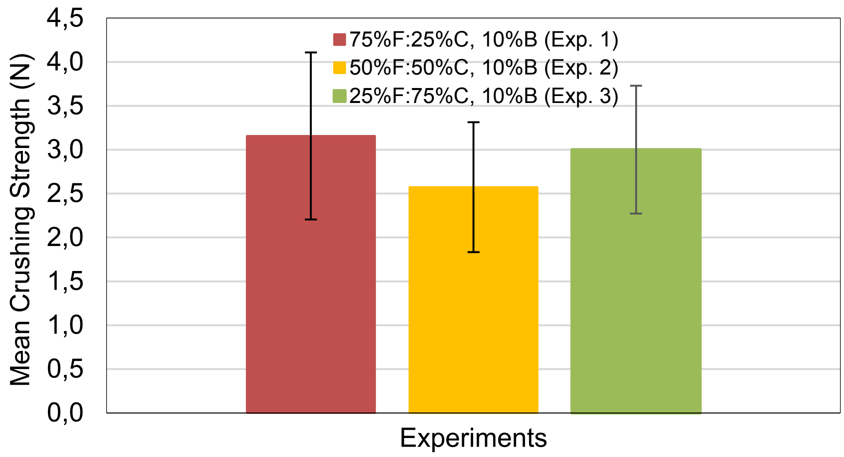

Figure 2: Mean crushing strength of 500 - 600 μm granules at different powder ratio with constant 10 % of binder

Figures 3 and 4 show the SEM images of 500 – 600 μm granules, with 10 % binder, at 30× and 200× magnification, respectively. The internal structure of granules clearly shows that the internal micro-sized granules have no seed for powder ratio of 3 and 1, but contain seed for powder ratio of 1/3. It is clear from Figure 3 that at powder ratios of 1 and 3 some granules are present with agglomeration of fine and/or coarse powder and some seeded granules are present with more than and equal to 1 coarse particle (nuclei). However, samples with powder ratio of 1/3 have followed the hypothesis of seeded granulation rule i.e. layers of fine powder onto the coarse particle (nuclei). Reducing the amount of fine powder, promotes the formation of seeded granules and this was obtained at powder ratio of 1/3 coupled with binder content of 10 %. The percentage of seeded granules at this powder ratio is about 40 % which is the highest produced compared with powder ratios of 3 and 1.

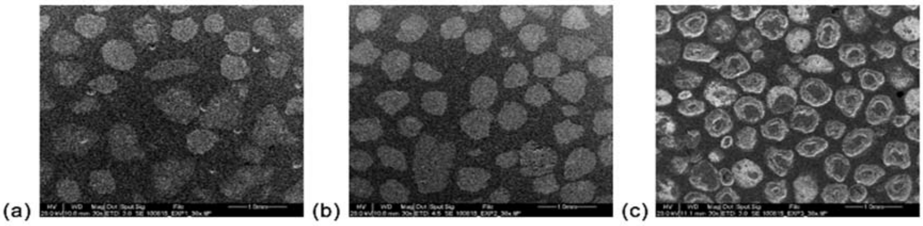

Figure 3: SEM images 30× of granules at constant 10 % of binder; (a) Powder ratio of 3 (75 % Fine: 25 % Coarse), (b) Powder ratio of 1 (50 % Fine: 50 % Coarse), and (c) Powder ratio of 1/3 (25 % Fine: 75 % Coarse)

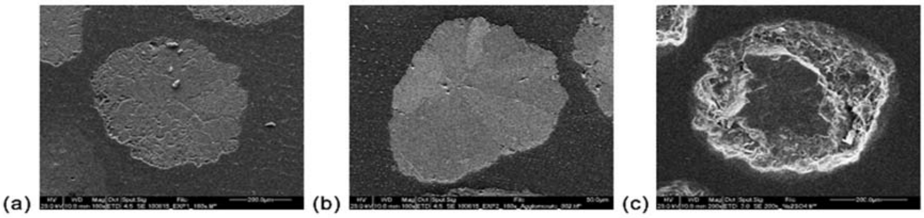

Figure 4: SEM images 200× of granules at constant 10 % of binder; (a) Powder ratio of 3 (75 % Fine: 25 % Coarse), (b) Powder ratio of 1 (50 % Fine: 50 % Coarse), and (c) Powder ratio of 1/3 (25 % Fine: 75 % Coarse)

3.2 Effect of Binder Percentage at Constant Powder Ratio 1/3 (25 %F : 75 %C)

As shown in Figure 5, when the percentage of binder increases, the trends shift towards the bigger size range, formation of fine powder, tiny granules decreases and formation of coarse granules increases.

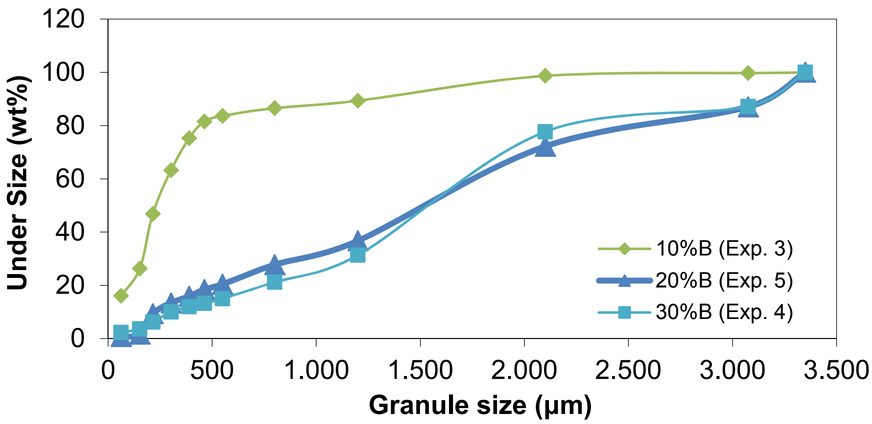

Figure 5: Granule size accumulation at different binder percentage and constant powder ratio of 1/3 (25 % Fine: 75 % Coarse)

Figure 6 shows that the 30 % binder samples have the highest mean crushing strength (4.03 N) as compared to 10 % and 20 % binder samples, which have an average crushing force values of 3.00 N and 2.13 N respectively. However, samples with 10 % binder content have the lowest standard deviation of average crushing strength as compared to those at 20 % and 30 % binder content (1.73 N and 1.28 N).

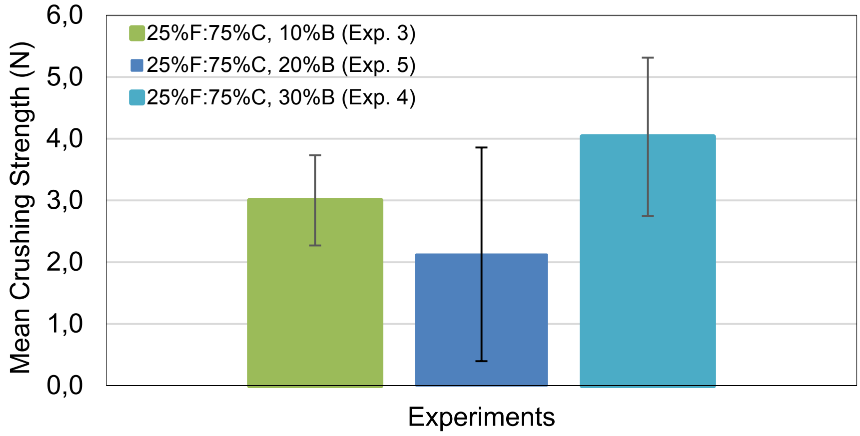

Figure 6: Mean crushing strength of 500 – 600 μm granules at different binder percentage and constant powder ratio of 1/3 (25 % Fine: 75 % Coarse)

Figures 7 and 8 show SEM images of 500 – 600μm granules at constant powder ratio of 1/3 at 30× and 200× magnifications. It can be clearly seen that the internal micro-structure of granules contain seeds. However, the percentage of seeded granules is different for each sample. All the binder percentage (10 %, 20 % and 30 %) at constant powder ratio of 1/3 therefore follow the hypothesis of seeded granulation rule i.e. layering of fine powder onto the coarse particle (nuclei). The largest numbers of seeded granules were obtained at 10 % and 30 % binder content and at constant powder ratio of 1/3. The percentage of seeded granules for the samples produced with 10 % and 30 % of binder ratio is approximately 40 % whereas for the samples with 20 % binder ratio number seeded granules are about 8 %.

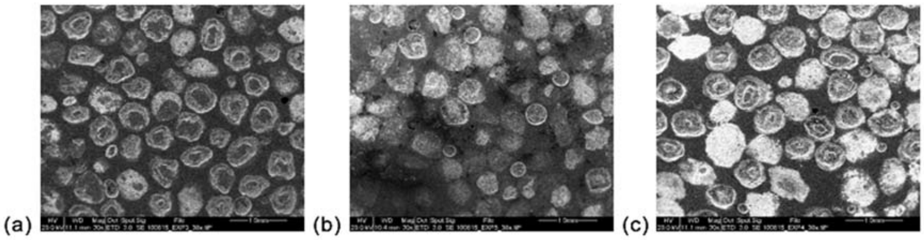

Figure 7: SEM images 30× of granules at constant powder ratio of 1/3 (25 % Fine: 75 % Coarse); (a) 10 % binder, (b) 20 % binder, and (c) 30 % binder

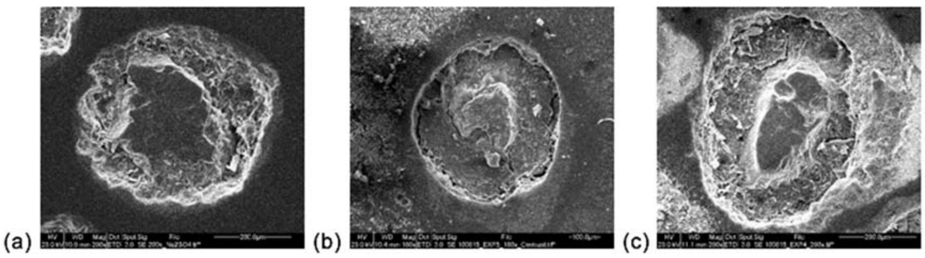

Figure 8: SEM images 200× of granules at constant powder ratio of 1/3 (25 % Fine: 75 % Coarse); (a) 10 % binder 200×, (b) 20 % binder 100×, and (c) 30 % binder 200×

4. Conclusions

Granules produced in 5 L high shear Cyclomix granulator have been characterized for their strength, size and internal structure to discover possibility of formation of seeded granules for detergent powders. It is found that 70 % of granules are produced at a desired size range between 125 – 1,000 μm using powder ratio of 1/3 (25 % Fine: 75 % Coarse) with 10 % binder. Also, low mean crushing strength of 3.0 N with a narrow distribution was obtained using powder ratio of 1/3 with 10 % binder. Finally, SEM images show that seeded granules are produced mainly by using powder ratio of 1/3 with 10 % binder.

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