An investigation into the effects of binder viscosity, shear rate, mixing time, and primary particle size on the spreading of liquids in coating processes

Tyler Simmons and Richard Turton Department of Chemical Engineering West Virginia University, Morgantown 26506

Abstract

The purpose of the current study was to investigate the spreading of a liquid binder onto a particle bed. It was desired to characterize this liquid spreading in terms of common parameters including liquid viscosity, agitation intensity, mixing time, and primary particle size. A series of experiments was performed to determine the effects of these parameters on the uniformity of the coating on sugar cores (nonpareils). Silicone oils of viscosities ranging from 5 cSt to 10,000 cSt were used as the major component of the binder liquid. These silicone oils were blended with an isoparaffin to allow for the solubilizing of a blue dye in the silicone composition for visual marking purposes. Two shear rates were used for the runs. Mixing time was investigated with times ranging from 20 seconds to 200 seconds. Once the liquid spreading experiments were completed, the data were collected and analyzed. The coating uniformity was investigated by capturing images of blue-coated particles with a color camera and subsequently analyzing the color level intensity on each particle using Visilog® imaging software. The relative standard deviation (RSD) of the blue content on the particle samples was determined from the raw data, and this RSD was used as a measure of the liquid spreading onto the particle bed. A high value indicates poor mixing while a low value indicates good/uniform spreading. It was found that liquid viscosity, shear rate, mixing time, and primary particle size all have an effect on the spreading of the liquid in the bed; however, it was not possible to eliminate the occurrence of agglomeration in the system. This made it difficult to definitively characterize the spreading process alone. Nevertheless, after studying the physics of the system at a microscale level, promising results describing the agglomerate breakage step were obtained.

KEYWORDS: Binder, Coating, Agglomeration, Breakage, Uniformity.

I. INTRODUCTION

The distribution of the binder has an important effect on the granulation process and the distributed characteristics thereof. Indeed, the assumption of binder distribution and coverage is implicit to many agglomeration process models, for example, the microscale model of Ennis et al. [1]. If the wetting and spreading of the liquid is slow relative to the process time, its coverage will be poor and will lead to large variations in the inter-granule composition.

The uniformity of a batch of solids undergoing film coating using a solution of binder and solvent has been addressed previously for fluidized beds and perforated drumcoating devices by Cheng and Turton [2 and 3], Mann [4], Choi and Miessen [5], Sandadi et al. [6], and Sudsakorn and Turton [7]. The application of liquid binder solution simultaneously with a powder has also been addressed by Nakamura et al. [8]. However, the uniformity of coverage of core particles by a relatively viscous binder has received little attention in the literature. Important issues in this type of coating are the effects of binder properties (most importantly viscosity and surface tension), agitation intensity in the powder bed, mixing time, and primary particle size. In the current work, these parameters served as the focus of the investigation on the uniformity of the spreading of the binder on the surface of the core particles.

II. EXPERIMENTAL

The core particles used in this study were 18-20 mesh and 35-40 mesh nonpareils with average diameters of 920 microns and 460 microns, respectively. Silicone oils (polydimethylsiloxane) of viscosities ranging from 5 cSt to 10,000 cSt were used in this work. A blue dye tagging agent was combined with an isoparaffin and added to the silicone oils. The resulting series of binders had viscosities in the range 5 to 1700 cSt.

The experimental apparatus, shown in Figure 1, was comprised of a tilted aluminum disk 2 feet in diameter driven by a motor, the speed of which could be manipulated by the user from a control panel. A plexiglassTM enclosure was constructed and was held in place on top of the rotating disk by springs. This enclosure acted as the walls of the particle bed. The shear in the enclosure was achieved by the upward movement of particles near the surface of the disk due to the upward rotation of the disk and the cascading of particles down the bed once they reached the top wall.



Figure 1: (a) Experimental apparatus (b) Top view of particle bed enclosure

A series of experiments was performed in which 20 drops of binder were added to equal-weight (10 g) batches of core particles over a time period of 20 seconds. Four different binder viscosities, two shear rates, and three mixing times were investigated for both the 18-20 mesh and 35-40 mesh particle sizes. The mixing time is reported as the time after all the liquid had been added to the particle bed, and all experimental runs were performed at ambient temperature. Table 1 shows the experimental matrix (for each of the primary particle sizes) used for this study.

Binder Viscosity (cSt)	Shear Rate	Mixing Time (s)
5	Disk Speed = 0.58 m/s Disk Speed = 5.00 m/s	20 60 200
24	Disk Speed = 0.58 m/s Disk Speed = 5.00 m/s	20 60 200
130	Disk Speed = 0.58 m/s Disk Speed = 5.00 m/s	20 60 200
1700	Disk Speed = 0.58 m/s Disk Speed = 5.00 m/s	20 60 200

Table 1: Experimental Matrix Used in the Current Work

In order to quantify the degree of spreading of the liquid binder on the cores, it was necessary to capture an image of the blue-coated particles. Multiple samples from each coating scenario were prepared, and a Pixelink firewire color CCD camera was used to capture the image of each sample. A typical image is shown as Figure 2. The imaging analysis algorithm was performed using VisilogTM software to determine the distribution of the binder solution on the particles. First the color image was split into its component colors (red, green, blue). Then, employing the red monochrome image, various edge detection tools were used to remove any "blobs" or "noise" from the image. A boundary was then set around each particle, and this perimeter enclosed the relevant area for the color level analysis. The software then analyzed the gray level, which was an indication of the blue intensity on the particle images, within each of these particle perimeters to produce results of the binder distribution on the population of the core particles.



Figure 2: Color image of blue-coated particles, high shear setting and 60 s mixing time

III. RESULTS AND DISCUSSION

Coating Uniformity

Five samples (of approximately *x* particles each) were analyzed for the spread of binder solution for each experimental scenario, and the raw data were combined. This raw data comprised the relative color level, which was based on two standard values. One of the standard values represented a relative color value of 0 (no blue dye). This value was found by capturing images of uncoated white particles and determining the value of the whitest particle. The other represented a relative color value of 1 (large amount of blue dye), which was found in a similar fashion. Therefore, the relative blue level on each of the particles in the sample images was normalized. The mean (\bar{x}), standard deviation (σ_x), and relative standard deviation RSD (σ_x/\bar{x}) for these color level values were determined for all scenarios. Figure 3 illustrates the effects of binder viscosity, shear rate, and mixing time on RSD for the 18-20 mesh particle size. Low binder viscosities, high shear rates, and longer mixing times lead to a smaller RSD, and therefore, better spreading of the liquid binder.

Results were also obtained for the experiments using the 35-40 mesh primary particles; however, there appeared to be no significant trend of RSD with viscosity, shear rate, and mixing time. It is believed that because of the flow pattern observed in the powder bed for these experiments with the smaller particles, the data obtained is confounded. It was observed that, after the total amount of binder was added to these particles, the flow of the particles had a "slugging" motion. This "slugging" motion comprised of large groups of particles moving as a single entity down the length of the particle bed. In this situation, the flow field was quite different from that found with the larger particles. No longer did individual particles move separately in the bed but rather clumps of particles moved in a non-uniform motion. Not surprisingly, it was found that all RSD values for the smaller particle size were significantly larger than the corresponding values using the 18-20 mesh particles. This particle size effect may be overcome using higher shear rates in the apparatus, but such conditions could not be produced in the current equipment.

The mechanism by which the liquid binder spreads on the powder bed was also examined. Because the binder was added in a drop-wise fashion, agglomerates were initially formed in all experiments. Subsequently, due to the movement and impact of the dry bed particles, these agglomerates broke up and the liquid binder redistributed itself over the bed. The mechanism by which these initial agglomerates broke up was also investigated. The work of Simons and Fairbrother [9] and Willett and Seville [10] was used to develop a model to describe this behavior. The formulation of the model and results will be discussed in the presentation, if time allows.





Figure 3: Effect of mixing time and shear rate for 18-20 mesh primary particle size at binder viscosity of (a) 5 cSt, (b) 24 cSt, (c) 130 cSt, and (d) 1700 cSt

IV. CONCLUSIONS

This work demonstrates a methodology to directly measure binder dispersion on particles in a mixer-granulator or coating process. From the coating uniformity results obtained from this work, it is hypothesized that parameters including binder viscosity, shear rate, mixing time, and primary particle size can be incorporated to model the spreading process. However, due to the presence of agglomeration in the system, it was not possible to definitively characterize the spreading mechanism alone. On the other hand, it is believed that there exists a strong coupling between binder dispersion and agglomeration, and the agglomeration and breakage mechanisms were investigated.

V. REFERENCES

- 1. Ennis, B.J., Tardos, G.I., and Pfeffer, R., A Microlevel-Based Characterization of Granulation Phenomena, Powder Technology, 65, 257-272 (1991)
- Cheng, X.X., and R. Turton, The Prediction of Variability Occurring in Fluidized Bed Coating Equipment Part 1: The Measurement of Particle Circulation Rates in a Bottom Spray Fluidized Bed Coater, Pharmaceutical Development and Technology, 5(3), 311-322 (2000)
- Cheng, X.X., and R. Turton, The Prediction of Variability Occurring in Fluidized Bed Coating Equipment Part 2: The Role of Non-Uniform Particle Coverage as Particles Pass Through the Spray Zone, Pharmaceutical Development and Technology, 5(3), 323-332 (2000)
- 4. U. Mann, E.J. Crosby, Can. J. Chem. Eng., 53, (1975) 579-581
- 5. M.S. Choi, A. Miessen, Chem. Eng. Sci., 52, (1997) 1073-1086
- Sandadi, S., Pandey, P., and R. Turton, In-situ, Near Real-time Acquisition of Particle Motion in Rotating Pan Coating Equipment Using Imaging Techniques, Chem. Eng. Sci., 59, 5807-5817 (2004)
- 7. Sudsakorn, K., and R. Turton, Nonuniformity of Coating on a Size Distribution of Particles in a Fluidized Bed Coater, Powder Technology, 110, 37-43 (2000)
- 8. H. Nakamura, E. Abe, N. Yamada, Powder Technology, 99, 140-146 (1998)
- S.J.R. Simons, R.J. Fairbrother, Direct Observations of Liquid Binder-Particle Interactions: The Role of Wetting Behavior in Agglomerate Growth, Powder Technology, 110, 44-58 (2000)
- C.D. Willett, J.P.K. Seville, An Examination of the Effects of Contact Angle on the Properties of Exact and Toroidal Liquid Bridges, World Congress on Particle Technology 3, Paper 79 (1998) IChemE