

Investigations into homogeneous and non-homogeneous caking and crusting in powders using powder rheology

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ABSTRACT Over 70% of materials in the food, chemical and pharmaceutical industries (from raw materials, additives and intermediates through to manufactured products) are supplied as relatively free-flowing powders, intended to be suitable for the manufacturing process or final application. For logistical reasons however, these materials will often have to be stored for extended periods during which time some powders have the potential to gain strength due to prolonged and undisturbed particle/particle interactions. This is generally referred to as 'caking' and can significantly limit the ability of a powder to pass through the process train without interruption as well as detrimentally impacting product quality.

This paper will present case studies which evaluate the flow properties of different powder systems that are affected by chemical, moisture and temperature based caking mechanisms. It will show how the propensity to cake can be effectively quantified with respect to the powders' flow properties and how this can assist with understanding and adapting the processing environment to retain optimal processability.

1. INTRODUCTION

Many materials in the food, chemical and pharmaceutical industries (from raw materials, additives and intermediates through to manufactured products) are supplied as relatively free-flowing powders suitable for the manufacturing process or final application. For logistical reasons these materials will often have to be stored for extended periods during which time some powders can gain strength due to particle/particle interactions. This is generally referred to as 'caking' and can significantly limit the ability of a powder to pass through the process train without interruption and can detrimentally impact product quality.

Caking occurs through one or more mechanisms – typically, mechanical, chemical and thermal – with the migration and absorption of water often the most influential. Limiting caking can be achieved by managing environmental conditions to keep materials in an optimal state; by adjusting the operational parameters (typically by limiting the time when the material is stationary) or by modifying the product formulation.

However, regardless of the mechanism, determining the precise conditions which minimize the potential for caking requires a comprehensive understanding of the changes in flow properties that occur as a consequence of such caking. Existing studies have restricted their investigations to evaluate single influences on powder caking such as storage time [1] or humidity [2], but the advent of powder rheometry [3] has allowed formulators and process engineers the capability to measure a range of powder characteristics (including dynamic, shear and bulk properties) that can quantify the progression of caking – for example, as a function of time, humidity and/or consolidation stress.

This paper will present case studies which evaluate the flow properties of a range of powder systems that are affected by chemical, moisture and temperature based caking mechanisms. It will show how the propensity to cake can be effectively quantified with respect to the powders' flow properties and how this can assist with understanding and adapting the processing environment to retain optimal processability.

2. EXPERIMENTAL

2.1 Sample Preparation – Four distinct categories of caking have been identified for evaluation in this study.

Chemical based caking – a three-component blend was stored for increasing periods of time, ranging from zero to ten days under ambient conditions; the blend was additionally subjected to an applied normal load of 9kPa to simulate storage in a silo/bin.

Temperature enhanced caking – three polymers were stored for 48 hours at 40°C with and without an additionally applied normal load of 2kPa to simulate storage in a small silo/bin.

Humidity enhanced caking – three food powders were stored for 48 hours at different constant relative humidities (RH) using a standard saturated salt solutions methodology [4].

Evaluating the extent of moisture migration (crusting) – a sulphonated methyl ester used in detergent manufacture was stored at 75% RH for four days. .

Most of the powders experience an increase in their resistance to flow through the creation of stronger inter-particulate bonds. In some instances these changes are reversible, but in many cases the powder has undergone surface chemical changes that result in permanent bonds being created; similarly the caking of a particular powder system may occur spontaneously, may require the (prolonged) interaction of atmospheric/processing induced humidity, or require an input of energy. In one instance, however, the flow behavior of the powder is enhanced and this is due to reduced electrostatic charging and/or lubrication of the powder in question.

2.2 Powder Characterization

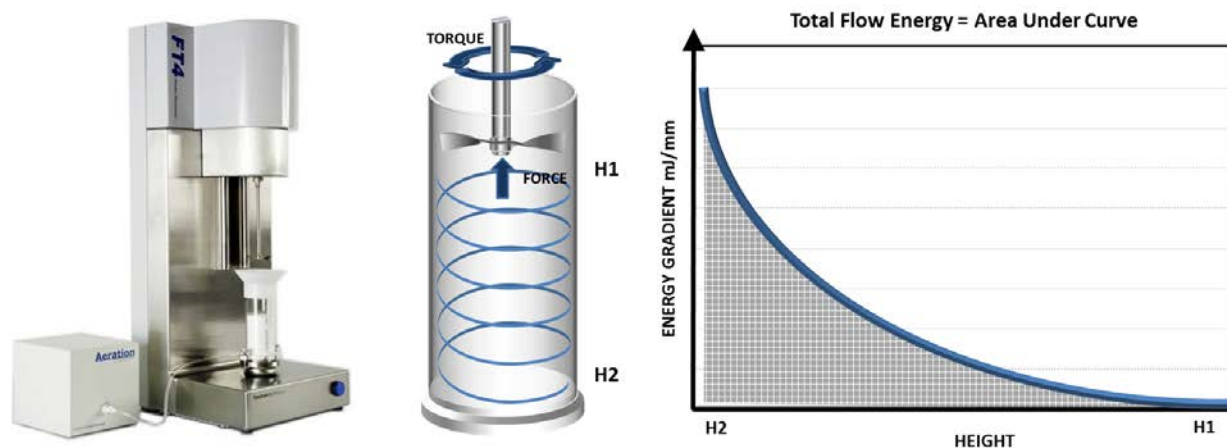


Figure 1: Measurement of flow energy using the FT4 Powder Rheometer®

An FT4 Powder Rheometer® (Freeman Technology), shown in Figure 1 (far left), was used to measure the flow energy of the samples before and after caking to quantify the resistance to flow. The flow energy results are obtained by means of a patented measurement principle that evaluates the resistance to the motion of a specially shaped twisted blade passing through a precise volume of the sample along a prescribed path. The required torque and force are recorded and converted into a flow energy [3]. The repeatability of all of the measurements was enhanced by the use of a conditioning cycle which removes packing history and operator induced variability. Conditioning was undertaken on all samples prior to being subjected to the range of modified environments which are used to induce caking in this study.

3. RESULTS AND DISCUSSION

3.1 Chemical based caking

Figure 2 shows the effect of storage time and chemical interaction for a 3-component blend, which is known to chemically interact when mixed. Two test conditions were investigated where the samples were stored under their own weight and also with an additional 9kPa consolidating stress.

In neither of the test conditions was a significant increase in the flow energy observed for the first four days due to the slow rate of reaction between the components. After this point the reaction rate increases and the mixture starts to cake and the flow energy increases. As the reaction progresses, the flow energy increases rapidly as the chemical interactions at the particles' surfaces increases and the mixture cakes. During the initial period the test group under the applied normal load is marginally more caked than the non-consolidated sample, most likely a function of reduced inter-particulate distances and as a result increased van-der Waals interactions. After four days the consolidated test group exhibits a much sharper increases in the flow energy compared with the non-consolidated sample indicating the caking reaction is enhanced by closer packing of the particles.

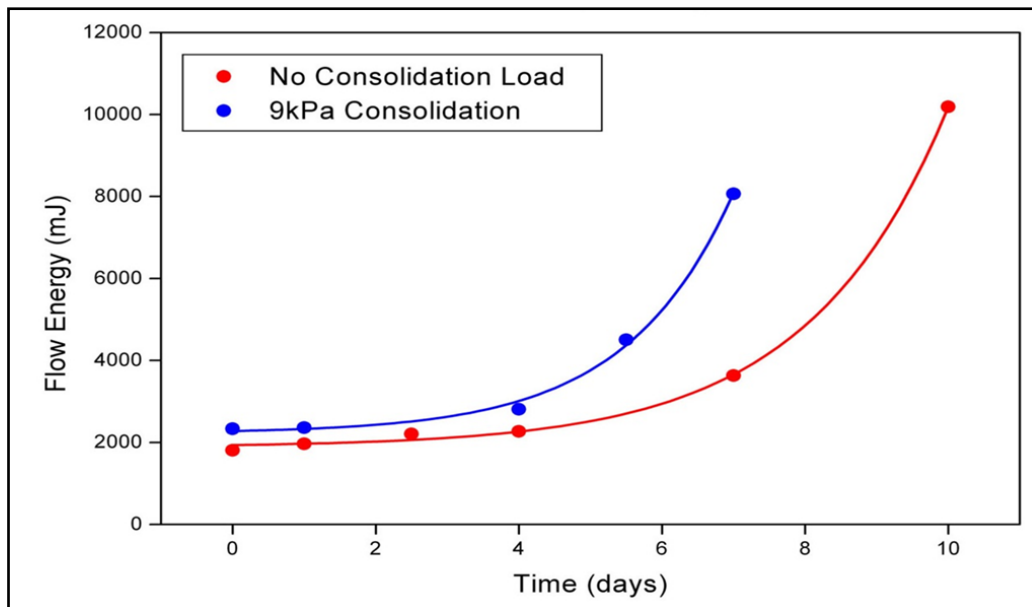


Figure 2: Investigating the impact of consolidation on caking by tracking changes in flow energy as a function of time

Using the results presented here will allow process engineers to specify maximum storage times and quantities for this particular blend of components.

3.2 Temperature based caking

Figure 3 demonstrates the effect of storage time and consolidation stress for three polymer samples stored at 40°C. The change in flow energy indicates that all the samples are sensitive to caking resulting from extended storage periods and the elevated temperature. Caking due to chemical interactions/sintering, is compounded by plastic deformation of the powder bed which increases the contact area between particles and therefore the number of cohesive interactions including surface chemical interactions [5]. At elevated temperatures the molecular mobility/viscoelasticity of materials is enhanced, reducing the particles' hardness and subsequently the material undergoes greater plastic deformation. The amount of plastic deformation and consequent contact area can be further increased by the application of an applied normal load. This will also reduce inter-particulate distances and therefore increases van-der Waals interactions. This explains the substantial increases in flow energy demonstrated by Polymer B and Polymer C when subjected to an additional applied normal stress.

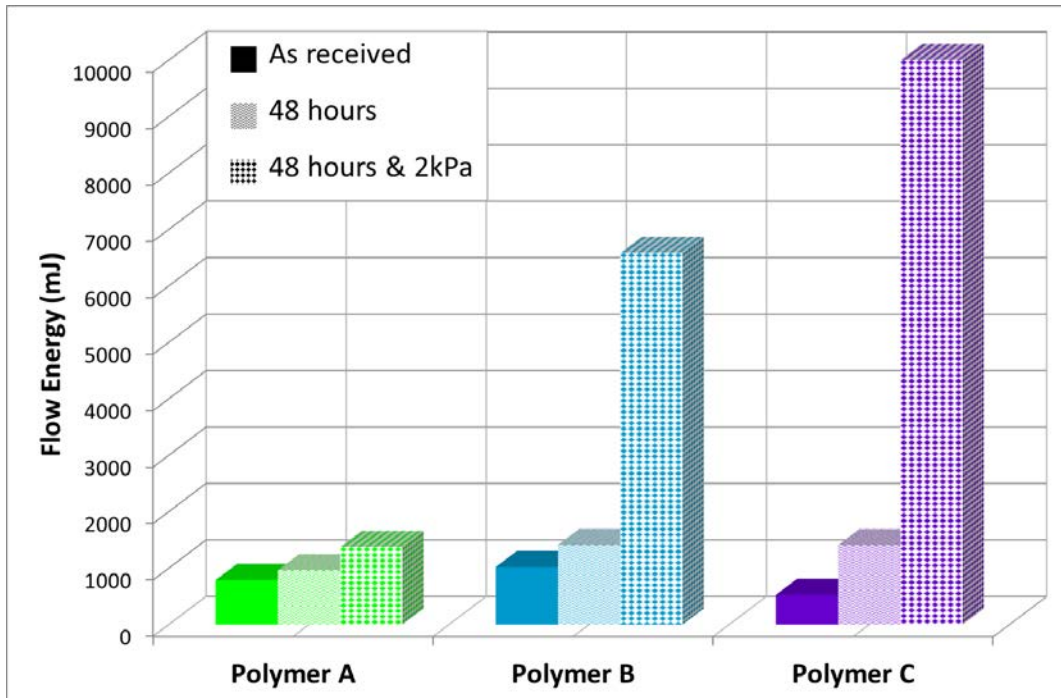


Figure 3: Flow energy as a function of time and applied normal load for three polymers stored at 40°C

In contrast Polymer A presented a smaller increase in the flow energy. This material has a higher glass transition compared with Polymer B and C and therefore exhibits a limited increase in the molecular mobility at 40°C. As such this material undergoes limited plastic deformation even at the applied normal load. Thus the level of caking is restricted and consequently this material generates only limited increases in the flow energy.

3.3 Humidity based caking

The effect of relative humidity on powders is complex due to the interaction of several factors. Moisture adsorption creates capillary bridging [6] which increases the size of any forces required to separate individual and groups of particles from each other. Over time adsorbed moisture can also result in solid bridging, facilitated chemical interactions and increased plastic deformation through increased molecular mobility primarily at particle surfaces.

All three food samples show a propensity to cake (Figure 4), however, the magnitude of caking varies between the samples and there is no clear link between the relative humidity and increase in flow energy. Food A demonstrated limited increases in the flow energy with increased relative humidity indicating that this sample absorbed a limited amount of moisture. In contrast Food C exhibited a sharp increase in flow energy at 75% RH. This material contains a high concentration of sucrose which absorbs sufficient moisture to dissolve sugar molecules on the crystal surface. Consequently the surface of the crystal is “sticky” increasing the strength of the capillary forces and hence the strength of the cake [6].

Absorbed moisture can also inhibit/restrict consolidation as demonstrated by Food B which exhibits a decrease in the flow energy at 56%RH compared with the ambient sample. Absorbed moisture can reduce electrostatic forces and surface moisture can, in some cases, also act as a lubricant reducing the strength of the powder interactions.

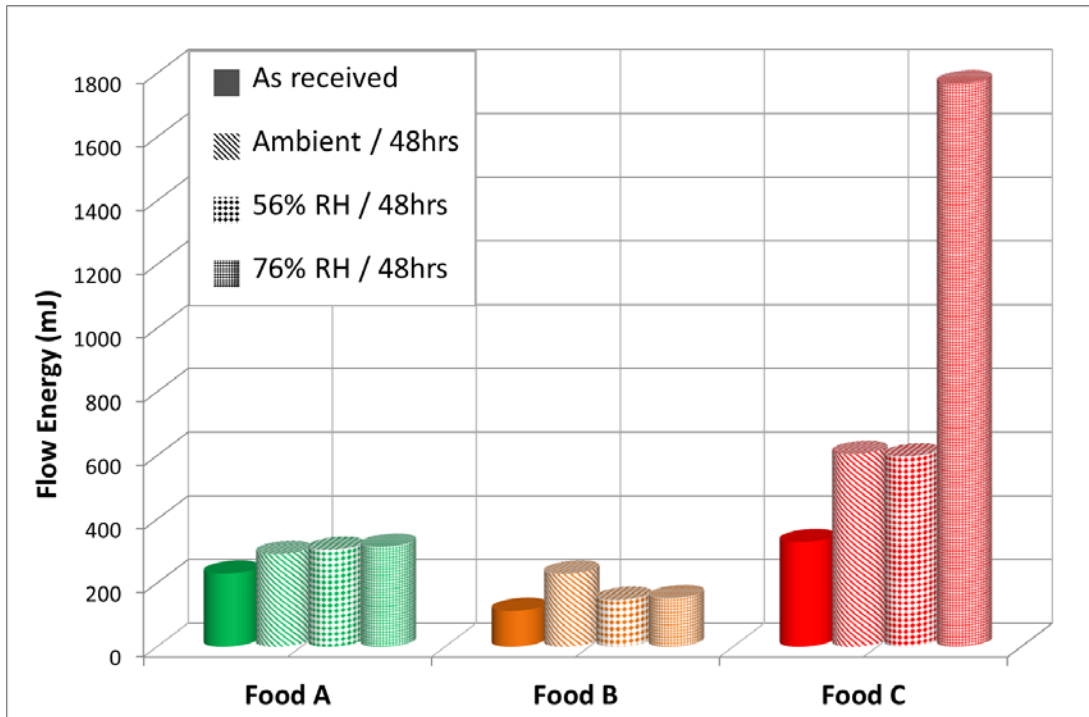


Figure 4: Flow energy as a function Relative Humidity for three food powders

3.4 Plugging/Crusting

Powder caking due to exposure to high relative humidity does not always occur uniformly throughout any given powder system. In a number of instances, caking can occur predominantly at the powder surface-air interface resulting in a strong 'crust' which is substantially more resistant to flow compared with the remainder of the powder bed beneath it. Figure 5 demonstrates this behavior in an FT4 test vessel.

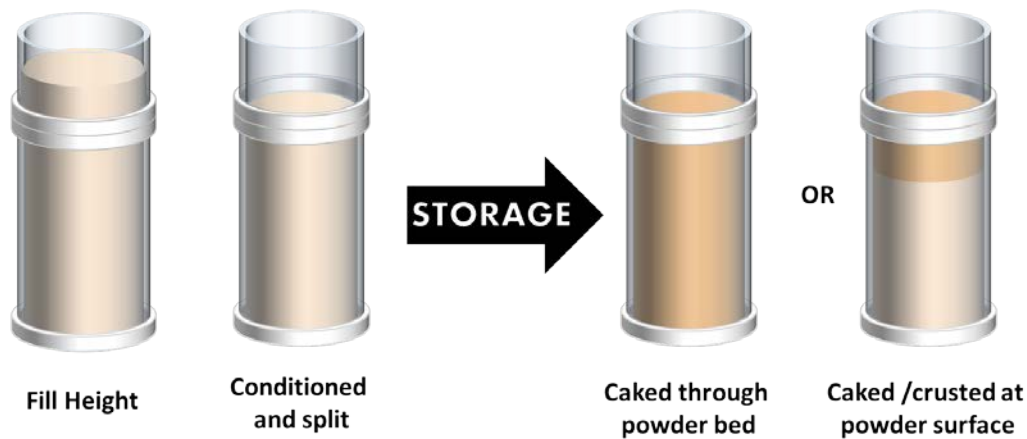


Figure 5: Schematic of the experimental set up used to evaluate moisture migration and crusting behavior and caking mechanisms, once the test vessel is filled, the same is conditioned to obtain a uniform packing structure. The vessel is then split to ensure a uniform bed height/test volume.

Figure 6 displays the force and torque measurements for the repeat tests of the 'as received' sample (blue + green traces) and crusted (red + pink traces) sample.

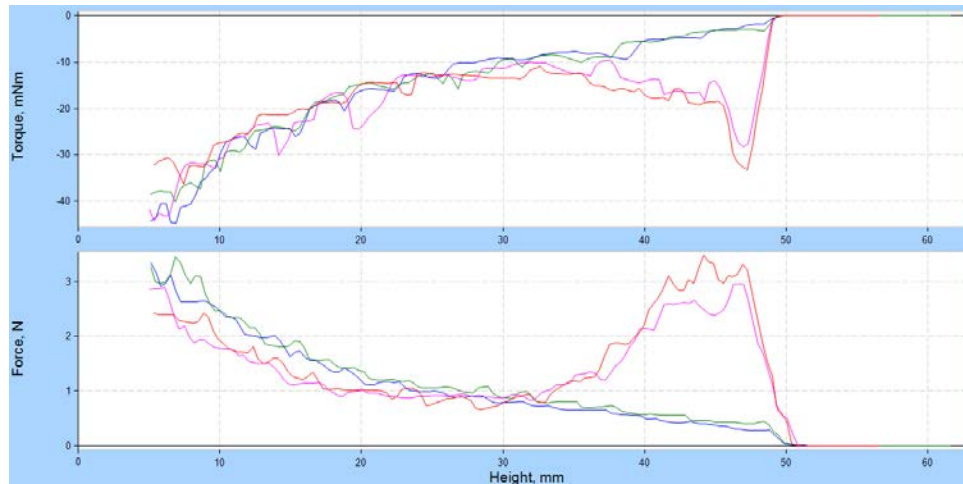


Figure 6: Force and Torque measurements for the ‘as received’ sample (blue and green traces) and crusted sample (pink and red traces) which demonstrates the presence of a stronger layer of powder between 30-50mm height.

Where there is moisture induced crusting, the data exhibits significant peaks in both torque and force between the top and bottom of the crust at 50mm and 35mm respectively and is due to increased resistance of this layer to the movement of the blade. Once the blade has passed through the plug, then both the force and torque measurements show similar levels to the as received sample and from this result it should therefore be possible to extend the testing to precisely evaluate the range and rate of moisture migration and thus quantify the extent of the crusting with respect to RH and time.

4. CONCLUSIONS

Over time, the physico-chemical nature of some powders subjected to humidity and/or; temperature and/or; stress, can result in the development of a caked structure. This can occur through a number of mechanisms which are clearly not limited to any one of these external factors and can have a significant impact on the flow properties and hence processing behavior and final product quality of a powder. In most instances these external factors result in reduced flowability, but this is not always the case, as evidenced by the behavior of Food B when subjected to increased humidity environments.

These studies show that, whatever the mechanism, the propensity to cake can be effectively quantified with respect to the powders’ flow properties and how this, in turn, can assist with understanding and ultimately adapting the processing environment to limit caking and retain optimal processability.

5. REFERENCES

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