Quantifying the impact of humidity on powder properties

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Of the many factors that influence powder behaviour, moisture, or humidity, is perhaps one of the most instantly recognised. Adding even small amounts of water to a powder can transform its properties. This is evidenced in a positive way in the process of wet granulation, which is often used to agglomerate fine, difficult to handle powders into free-flowing granules. Water can also be used beneficially to lubricate the flow of certain materials, and because it conducts charge it will effectively 'ground' a powder, reducing any electrostatic-related behaviour. Elsewhere, however, uncontrolled levels of moisture can cause significant problems.

In storage, for example, humidity levels that are ill-suited to the powder are a primary cause of caking, an issue that can adversely affect in-process and end-use performance. Poor flowability downstream of a process where water is deliberately added, such as crystallisation, wet ball milling or froth flotation, can also be detrimental to processing efficiency. Where moisture is a problem, there is usually a solution - the storage area can be maintained at lower humidity, for example, or the process stream can be dried – but these solutions are often associated with significant cost. Drying in particular is an energy intensive process that is often avoided where possible.

The challenge for formulators and process engineers is to understand the extent to which a powder will take up moisture when exposed to a humid atmosphere and, more importantly, how this moisture will affect the characteristics of the powder and its performance in any given process. Such understanding supports the development of effective strategies for process optimisation and the realistic economic assessment of measures to control moisture.

This paper explores the application of dynamic, shear and bulk property measurements to assess in detail the impact of humidity and gather relevant knowledge. It includes experimental data that illustrate the breadth of response of different powders to the presence of moisture.

Assembling an analytical toolkit

Over the last decade or so, powder processors have become increasingly aware of the need to apply a number of powder testing techniques to fully scope powder behaviour. Many different powder testing methods are available and each provides a snapshot of some aspect of powder behaviour. However, to effectively characterise powders in a way that will provide the information needed for integrated process design, optimisation and operation, it is essential to focus on methods that:

- are reliable and reproducible
- generate process-relevant data that correlate with performance
- allow sensitive assessment of the impact of variables such as moisture and degree of aeration

The improvement of more traditional powder testing techniques such as shear and bulk property testing, via the application of modern instrumentation and methodologies, has ensured that these methods retain a place in today's analytical toolkits. Shear properties are especially well known, and valued, for hopper design and more generally for characterising consolidated, cohesive powders. Bulk properties, on the other hand, such as density, permeability and compressibility, provide a general insight into powder behaviour; they generate data that may be needed directly for process design and enable the prediction of performance in certain processes.

More recently however, the development of dynamic powder testing has introduced a significant opportunity to gain a more thorough and comprehensive understanding of powder behaviour. Dynamic characterisation involves measuring the axial and rotational forces acting on a blade as it traverses through a sample along a fixed helical path. The resulting value of flow energy provides a direct measure of powder flowability. The technique is highly sensitive and has the distinct advantage of allowing powders to be characterised in a consolidated, conditioned, aerated or even fluidised state. This means that the response of a powder to the introduction or release of air can be directly measured and quantified.

When used in combination, dynamic, shear and bulk property measurements provide the information necessary to understand and rationalise powder performance in a wide range of unit operations. As such they provide a comprehensive and powerful toolkit which can be used to reliably assess the impact of humidity.

Investigating the impact of humidity

An investigation into the impact of humidity on powder properties was carried out using three different powders: limestone [BCR116, European Commission]; lactose [FlowLac100, Meggle] and microcrystalline cellulose (MCC) [PH200, FMC]. These were selected because of their industrial applicability and certain known characteristics. The limestone is used as a standard reference powder for shear testing, the MCC is known to be hygroscopic and the lactose represents an example of a widely used pharmaceutical excipient. Table 1 displays particle size data for each material.

Table 1

Material	Grade	Mean particle size (microns)
Limestone	BCR116	4
Lactose	FlowLac 100	140
Microcrystalline Cellulose	PH200	180

The first step of the experiment was to assess how much moisture each material adsorbed when allowed to equilibrate in environments of varying relative humidity. Figure 1 shows the moisture content of different samples as a function of the relative humidity at which they were stored. The results indicate that while both the limestone and lactose adsorb relatively small quantities of water, the MCC adsorbs significant amounts, an order of magnitude greater than either of the other two materials.

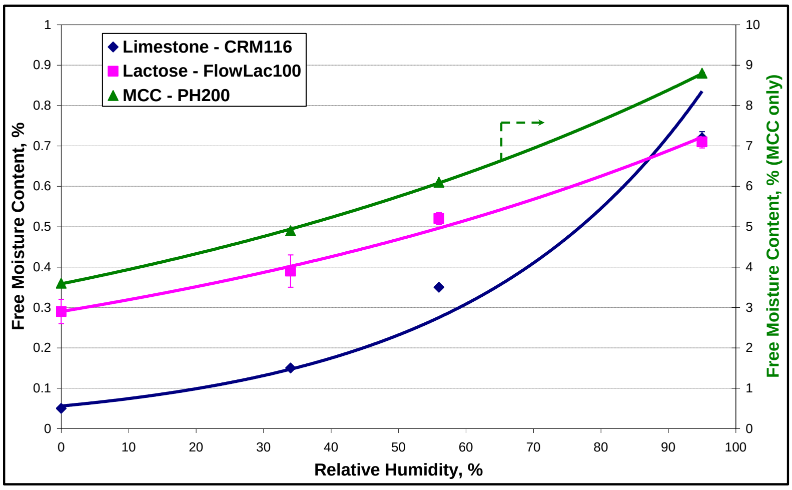

Figure 1: The effect of relative humidity on the moisture content of MCC, lactose and limestone. MCC adsorbs significantly more water than the other two materials.

These results tell part of the story of how these powders respond to the presence of moisture but the key question for processors is: How will powder properties, and most importantly, process performance change as a result of exposure to humidity and the associated adsorption of water?

To answer this question, each of the samples was subjected to dynamic, bulk and shear property testing using the FT4 Powder Rheometer, which offers all three test regimes.

Dynamic characterisation

The impact of humidity on the dynamic flow properties of the test powders is illustrated by Basic Flowability Energy (BFE), Specific Energy (SE) and Aerated Energy (AE) data.
Ref 1 gives a full description of all the test methodologies employed.

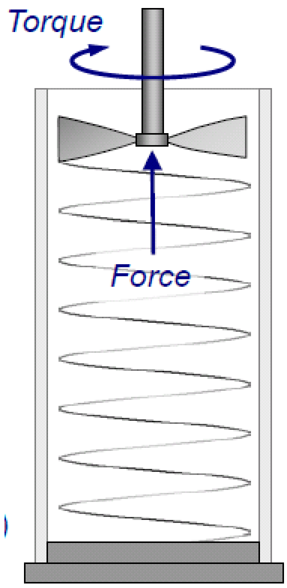

Figure 2: Schematic illustrating the principles of dynamic powder testing

Figure 3 shows BFE data for the samples. BFE is the flow energy measured as the instrument blade is rotated downwards through a conditioned sample. This rotational pattern forces the powder down against the base of the test vessel thereby imposing a compressive and moderately high stress flow regime. BFE measurements are highly differentiating and a good indicator of how the powder will flow under 'forced' conditions, such as when distributed in a feeder, or extruded.

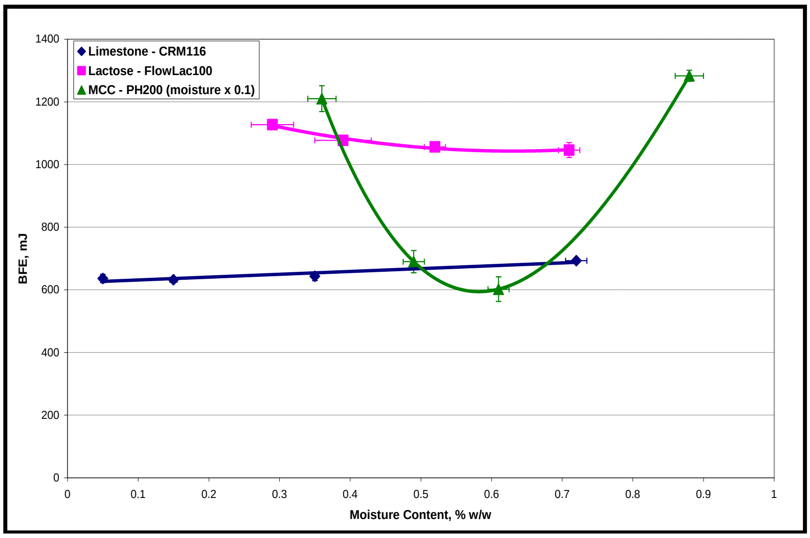

Figure 3: The effect of moisture content on Basic Flowability Energy for MCC, lactose and limestone

The results for limestone show a progressive increase in BFE with increasing moisture content, suggesting that the limestone becomes slightly more cohesive as the moisture content increases, likely due to the water acting as a binder and the associated formation of liquid bonds. The lactose in contrast exhibits a fall in BFE with increasing moisture levels, suggesting that here the moisture acts as a lubricant, reducing inter-particular forces.

The MCC shows more complex behaviour with a substantial drop in flow energy as moisture levels initially increase from the desiccated condition, and an equally dramatic increase in BFE at higher moisture levels. A minimum BFE is observed at approximately 6% moisture content.

During the study, it was also observed that the MCC sample coated the inner wall of the glass storage vessel prior to testing, suggesting a tendency towards electrostatic charging. This observation supports a rationale for the measured behaviour. If the high BFE value for the dry sample arises from electrostatic interaction between the particles then increasing moisture levels could cause a reduction in BFE by discharging the sample. The return of BFE to high values at increased levels of humidity is attributable to the material adsorbing sufficient moisture to increase inter-particulate bonding and create weak agglomerates within the sample both of which then begin to dominate flow behaviour.

It is important to note that the MCC exhibits this unusual behaviour over a range of conditions that are industrially relevant. Across the 25-50% RH range that could easily represent ambient conditions, BFE values for this material change markedly with respect to humidity, illustrating how MCC can readily exhibit variable flow characteristics when handled under conditions routinely encountered in industry.

Figure 4 shows Specific Energy data for the samples. SE is the flow energy measured as the blade traverses upward through a conditioned, unconfined sample. Because the powder is unconfined the energies measured are heavily influenced by the friction and mechanical interlocking that exists between particles and less so by factors such as compressibility. SE provides a good indication of how the powder flows in the absence of applied stress, when poured from a vessel, for example, or when flowing into an empty die.

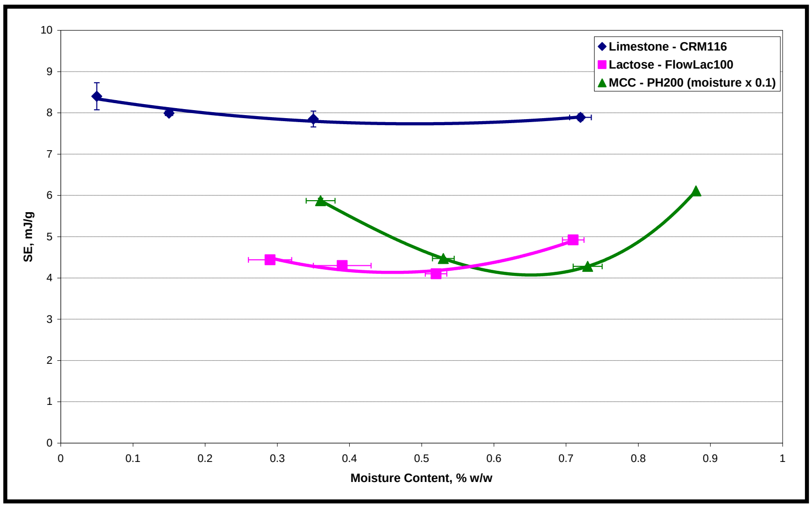

Figure 4: MCC, lactose and limestone all exhibit a minimum in SE as moisture content increases. Low levels of moisture improve flowability but higher levels are detrimental

All three powders show a similar trend for SE as they do for the BFE data, although the observed changes in SE for limestone are minor. Each material exhibits a minimum value in SE, but at different levels of moisture content. The pattern for the MCC mirrors that observed in the BFE data suggesting that the same mechanisms dominate flow behaviour under both conditions. By comparison, the results for the lactose suggest that in an unconfined state the increased cohesion induced by higher levels of moisture may dominate over the lubricating effect reported by the BFE.

With dynamic testing, measurements can also be made on aerated samples to directly quantify the response of the powder to the introduction of air. Aerated Energy is the flow energy determined using the same technique previously described in the BFE methodology but with air flowing through the sample at an accurately controlled velocity. This technique provides an excellent insight into powder cohesivity and the strength of the bonds between particles. With respect to processing, AE has particular relevance for fluidisation and unit operations such as pneumatic conveying but also, more broadly, for considering the potential impact of inadvertent aeration or de-aeration of the powder during processing.

Free flowing materials will often fully aerate to a point where the measured AE stabilises at a level of a few millijoules. Conversely, the flow energy of a cohesive powder is not significantly affected by the introduction of air as the increased strength of the inter-particular bonds prevents the particles from being separated from each other in the air stream. Figure 5 shows AE data for the three samples measured at a relatively low air velocity in order to limit moisture loss.

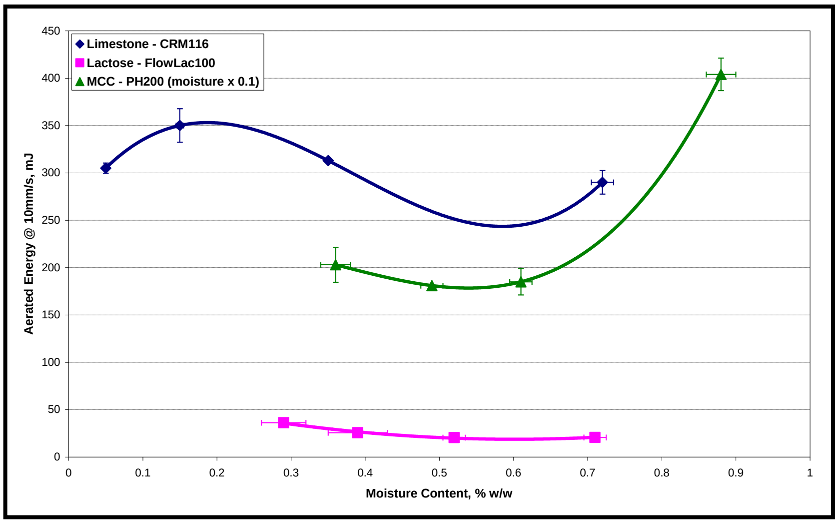

Figure 5 : The effect of moisture content on Aerated Energy for MCC, lactose and limestone samples

Limestone is a very difficult powder to aerate due to its fine particle size. The strength of the inter-particular forces presents significant resistance to air flow and results in air channelling through the powder bed. The introduction of air therefore has a limited and variable impact on flow energy for this material, with the extent and influence of the channelling effect varying erratically with moisture content

The lactose exhibits a steady decrease in AE with increasing moisture levels. This supports the observations from the BFE data and provides evidence that the presence of moisture reduces the strength of inter-particular bonds under these conditions.

The results for MCC are also consistent with the associated BFE data, but the impact at the highest humidity levels is even more apparent. This observation supports the suggestion that the flow energy increases due to the formation of larger, meta-stable agglomerates. Larger particles or agglomerates can present greater resistance to aeration because of their higher mass. In addition, they offer greater resistance to the motion of the rotating blade leading to an increased value of flow energy (see figure 6). In this test the influence of electrostatic charging at low humidity levels is much less pronounced as the particles are separated by the flowing air.

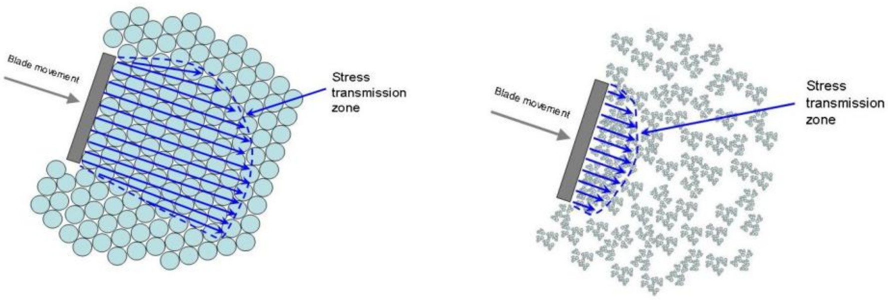

Figure 6: Efficiently packed larger particles can transmit blade movement through a large flow or stress transmission zone, to generate a high BFE value while with more cohesive powders the flow zone tends to be much smaller

Bulk property measurement

Bulk properties such as permeability, density and compressibility complement dynamic data by providing further insight into how powder behaviour is changing when it is stationary and under stress – in a hopper or a packed bed for example.

Permeability quantifies the ease with which air can pass through the powder bed. Lower permeability is indicative of greater resistance to air flow and therefore tends to be associated with more cohesive materials, which have higher inter-particular forces of attraction and reduced inter-particular pathways.

Compressibility values indicate the extent to which an applied force reduces the volume the powder occupies. Higher compressibility values are usually associated with more cohesive powders which tend to form loose agglomerates that entrain air, thereby creating a bed that can be significantly compressed. In less cohesive powders, the particles can flow more easily with respect to each other enabling them to pack more efficiently, making further compression of the bed much more difficult. Bulk density is similarly influenced by particle packing and tends to be a function of particle size and size distribution, as well as the shape and shape distribution. Close and efficient packing results in a powder with higher bulk density.

Figure 7 shows how the permeability of each of the materials varies as function of moisture content.

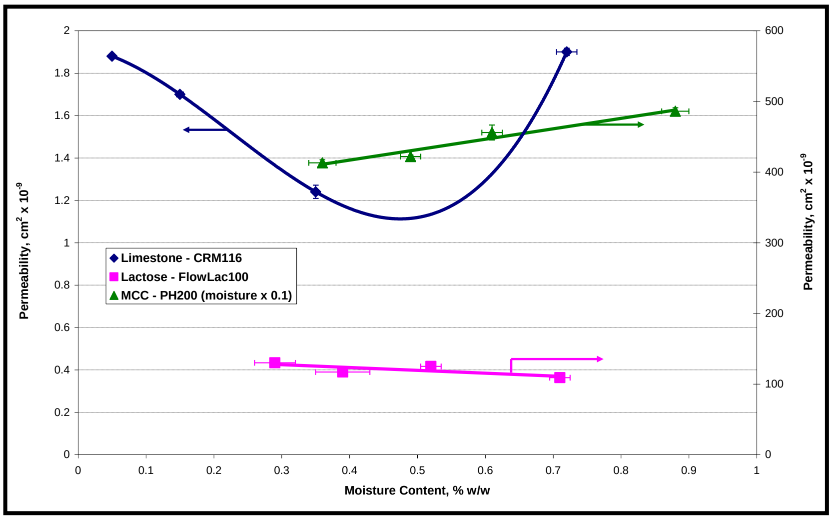

Figure 7: The effect of moisture on the permeability of MCC, lactose and limestone

The MCC sample shows a steady increase in permeability. This is consistent with the observed dynamic flowability data which suggests a decrease in cohesivity caused by a reduction in electrostatic forces followed by agglomeration. Agglomeration effectively creates 'large particles' which pack the test vessel more efficiently allowing gas to transit through the bed more uniformly. Both effects would therefore be associated with reduced resistance to air flow and a corresponding increase in permeability.

Conversely, the permeability of the lactose decreases with increasing moisture content. This is attributable to the formation of liquid bridges which, although they may lubricate flow, act as a barrier to the passage of air through the powder bed.

The limestone is orders of magnitude less permeable than the other two samples (data is recorded against the left hand axis) due to its much smaller mean particle size and the observed response to increasing moisture content is, in absolute terms, minimal.

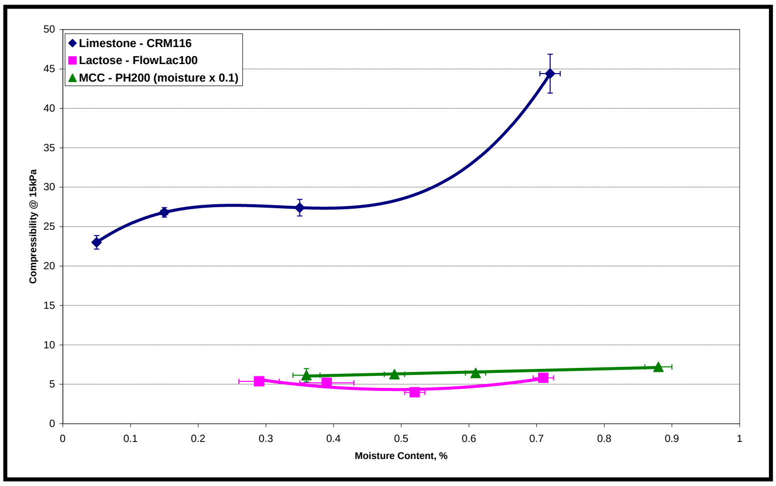

Figure 8 – The effect of moisture content on compressibility

Compressibility testing shows that, overall, all three materials become more compressible as moisture content increases (see figure 8). The lactose and MCC samples show relatively small increases in compressibility over the range of moisture contents assessed. However, with the limestone sample even minor changes in moisture content appear to have a significant effect on compressibility, a trend attributable to the increase in cohesivity observed with increasing water content, which also impacts BFE.

Conditioned bulk density (CBD) measurements are shown in Figure 9. The results for lactose and MCC indicate only small variations in CBD (approximately 2-3%) as a function of moisture content suggesting that bulk density/packing changes are not responsible for the observed trends in flowability behaviour (as quantified by dynamic test data). This suggests that changes in bulk density may not be as influential as commonly assumed in defining changes in powder behaviour, highlighting a limitation of using this parameter to quantify flow performance.

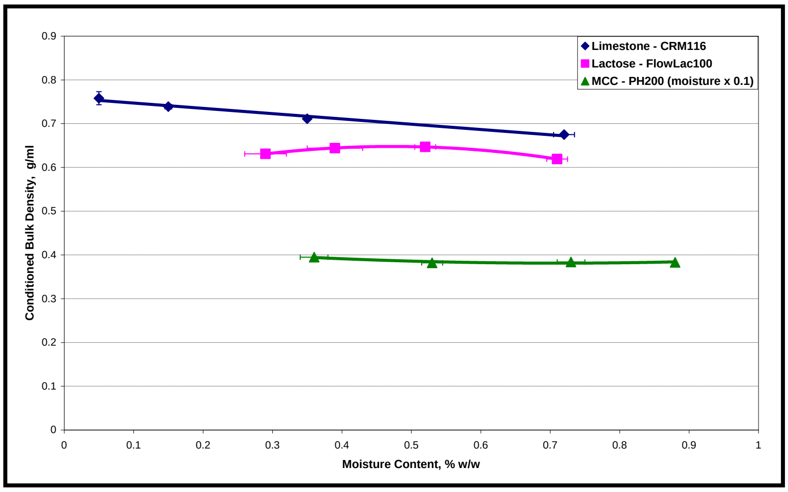

Figure 9: The effect of moisture content on conditioned bulk density (CBD) for MCC, lactose and limestone

The CBD of the limestone shows a progressive decrease with increasing moisture content, a result that suggests that more air is trapped in the bed at high moisture levels– an observation typically associated with greater cohesion. These data are therefore entirely consistent with the trend towards greater compressibility.

Shear analysis

Shear cell testing determines the stress required to shear one critically pre-consolidated powder plane relative to another. The results provide an indication of how easily a powder will move from a static, consolidated condition into a dynamic flow regime, for example, when the outlet on a hopper is opened, and form the basis of hopper design strategies [ref 2]. Key shear parameters include the angle of internal friction and shear stress (which is reported at a defined applied normal stress). Higher values of either of these figures are associated with more cohesive materials.

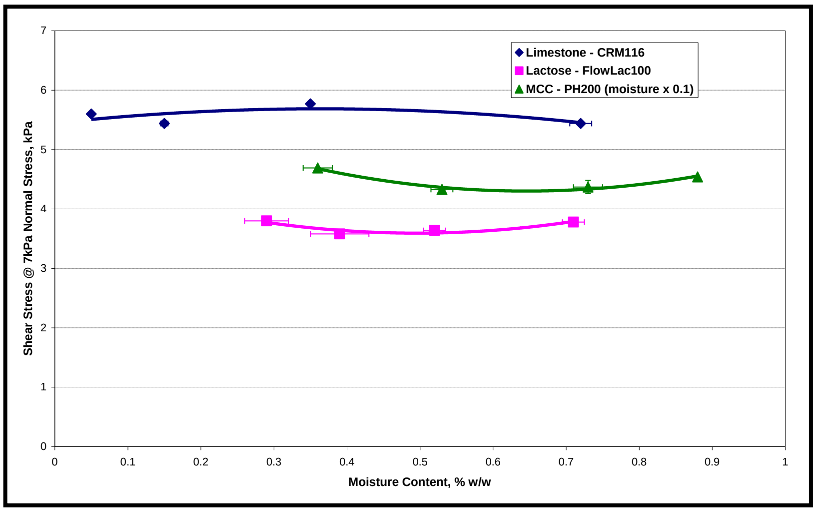

Figure 10: The effect of moisture content on shear stress for MCC, lactose and limestone.

The shear data for the lactose and MCC show similar trends to those observed in the SE data but perhaps the most noticeable feature of these results is that differences between the samples are very small; any trends are not pronounced. This is true of the limestone also and further demonstrates the greater value of alternative powder testing techniques, such as dynamic testing, for evaluating the effect of humidity.

To conclude

To accurately manage the impact of humidity on process performance, it is important to understand the extent to which moisture is adsorbed by a powder and, more importantly, how the adsorbed moisture impacts on powder properties. In this experimental study the hygroscopic MCC adsorbed more water, by an order of magnitude, than either the limestone and lactose samples tested at any given level of relative humidity. However, powder properties relevant to process behaviour changed significantly for all three powders, in ways that could neither be predicted from first principles nor inferred from any single measurement.

The data presented here strongly suggest that the assumption that all adsorbed moisture is detrimental to powder behaviour is misleading. For example, the adsorption of water improved the flowability of the MCC under certain conditions, possibly because it dissipated accumulated electrostatic charge. Moisture also had a positive impact on the BFE of lactose, an observation attributed to the ability of water to, in effect, lubricate particles. What is clear from the results, though, is that small quantities of adsorbed water can have a significant effect on powder behaviour, even with hydrophobic powders, and that for hydrophilic materials flow properties can be severely affected by storage under the relative humidity values typically observed in many industrial environments. Furthermore, the effect of adsorbed moisture on any given powder characteristic can be far from linear.

These findings reinforce the premise that to properly optimise powder processing it is essential to comprehensively characterise powders using a variety of techniques and under conditions that reflect the environmental conditions to which they may be subjected. Measuring only a single property, or assessing a powder under standard or uncontrolled conditions, will not provide the insight required. Although the response of a powder to moisture is complex and cannot be easily predicted, it can be quickly and reliably assessed through the application of suitable analytical techniques in order to provide the necessary information to effectively and economically optimise process behaviour.

References:

[1] Freeman R. "Measuring the flow properties of consolidated, conditioned and aerated powders — A comparative study using a powder rheometer and a rotational shear cell", Powder Technology 174 (2007) 25–33.
[2] Storage and flow of solids, Bulletin 123 of the Utah Engineering Experiment Station, November 1964 (Revised 1980), A.W.Jenike, University of Utah.

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