Titanium Dioxide has been used as a pigment and opacifier in a wide range of industries for many years, due to its bright white colour and high refractive index. However, despite such widespread use, processing Titanium Dioxide in its powdered form is often extremely challenging due to the powder’s high cohesivity. Special measures often need to be implemented when managing this material in operations such as dispensing from hoppers, feeding into unit operations and blending with other powders.

Identifying and quantifying which powder properties are conducive to efficient processing allows new formulations to be optimised without the significant cost of running samples through the process to assess suitability, making considerable savings in terms of time and raw materials, and minimising wastage due to out of specification products.

**ASSESSING BATCH-TO-BATCH VARIABILITY**

Despite meeting the existing specifications, three batches of Titanium Dioxide demonstrated significantly different behaviour when used in the same process, resulting in unacceptable variation in final product quality. A range of traditional characterisation techniques were employed, but failed to differentiate between the three batches, partially due to the high degree of variability in the test results.

Samples of the batches were analysed using an FT4 Powder Rheometer®, which demonstrated clear and repeatable differences between them that rationalised the variations in process performance, and enabled the user to reliably assess the quality of incoming batches in process-relevant terms.

**TEST RESULTS**

**Dynamic Testing: Basic Flowability Energy**

Sample B generated the highest BFE of the three materials, and Sample C the lowest. In this case, high BFE is a consequence of a more efficiently packed powder bed, meaning that the blade is required to displace more powder as it moves and with less available space for particles to move into. This results in more energy being required to mobilise the bed suggests that the powder may be problematic under dynamic, forced flow conditions, such as those experienced in a screw feeder.

**Bulk Testing: Permeability**

Sample B generated the lowest Pressure Drop of the three materials, and Sample C the highest. High Pressure Drop indicates a greater resistance to air flow through the sample, i.e. lower Permeability. The lower Pressure Drop (higher Permeability) for Sample B is typical of the uniform structure created by an efficiently packed bed, and is often associated with improved gravitational flow in low-stress environments (such as filling operations).
Shear Testing: Shear Cell

A different trend was observed in the Shear Cell results which is a consequence of the different stress and flow regimes established by this test methods. Shear Cell tests are intended to represent the high stress, static conditions experienced in operations such as gravitational hopper discharge. Sample A generated significantly higher Shear Stress values than the other two samples, indicating that it is much more resistant to incipient flow (the transition from a static to dynamic state) following storage under consolidation. Samples B and C generated similar Shear Stress values, suggesting that they would perform similarly under these conditions.

CONCLUSIONS

The FT4 has quantified clear and repeatable differences between the three samples in terms of Dynamic, Bulk and Shear properties. Sample B generated the highest Basic Flowability Energy and Permeability values, and low Shear Stress values, indicating it would perform very differently to the other samples. The results for samples A and C suggest they would exhibit more cohesive behaviour than Sample B across a range of processes: Sample C generated the lowest BFE and Permeability values, indicating the most cohesive behaviour in lower-stress processes such as blending and filling, and Sample A generated the highest Shear Stress values, indicating that this would present most resistance to flow in high-stress operations such as hopper discharge.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched and it is not uncommon for the same powder to perform well in one process but poorly in another. This means that several characterisation methodologies are required, the results from which can be correlated with process ranking to produce a design space of parameters that correspond to acceptable process behaviour. Rather than relying on single number characterisation to describe behaviour across all processes, the FT4’s multivariate approach simulates a range of unit operations, allowing for the direct investigation of a powder’s response to various process and environmental conditions.

For further information, please contact the Applications team on +44 (0) 1684 851 551 or via support@freemantech.co.uk.
In the pharmaceutical industry, the blending of a small fraction of active pharmaceutical ingredient (API) with a bulk excipient, and then subsequent processing though various unit operations, is an essential part of many manufacturing processes. The way that the blend behaves in downstream processes will be a function of the properties of the components of that blend.

When manufacturing formulations that will be delivered in capsule form, the blend requires suitable characteristics to ensure a precise and accurate fill of the dosing capsule. The ability to characterise formulations with respect to process behaviour is an essential part of Quality by Design (QbD). A method of quantifying the properties of powdered formulations that are conducive to good performance in a dosator, allows a design space to be defined, specifying material properties that will result in high quality final products. This provides significant commercial benefits in terms of higher productivity and reduced wastage.

**PERFORMANCE VARIATION BETWEEN SIMILAR FORMULATIONS**

Three powdered formulations were used in the production of a DPI product. Differences in the quality of the final product were observed depending on which formulation was used. Of the three formulations, Formulation 1 represented average behaviour in terms of filling performance from the dosator, whereas Formulation 2 represented the worst behaviour, and Formulation 3 the best.

Samples from the three formulations were evaluated using an FT4 Powder Rheometer®. The tests investigated the Dynamic, Bulk and Shear properties of the samples, in order to identify trends that correlated with the dosator performance.

**TEST RESULTS**

**Dynamic Testing: Specific Energy**

Formulation 2 had the highest Specific Energy of the three materials, and Formulation 3 the lowest. High Specific Energy represents a greater degree of mechanical interlocking and friction within the bulk, typically leading to problems in operations such as filling, where gravitational flow is critical.

**Bulk Testing: Compressibility**

Formulation 2 was the most compressible of the samples, and Formulation 3 the least. High Compressibility indicates that a powder entrains a greater proportion of air within its bulk, which is a property typically associated with more cohesive powders.
Minimal differentiation was observed between the three samples, with six data sets, i.e. two repeats on each sample, generating near identical Shear Stress values to within an acceptable standard deviation. This lack of correlation with the process behaviour illustrates how Shear Cell testing, designed to investigate how a powder transitions from a static to dynamic state following consolidation, may not be relevant to the more dynamic, low-stress conditions that a powder is exposed to in the dosing process.

**CONCLUSIONS**

The FT4 has identified clear and repeatable differences between three similar formulations which correlated well with the relative performance observed in the dosator. Specific Energy and Compressibility were shown to be highly differentiating suggesting that mechanical interlocking/friction and packing structure of the powder bed have the greatest influence on process performance. Limited information was provided by the Shear Cell test, suggesting that shear properties had little or no influence on the overall performance. The Dynamic and Bulk parameters can be used to construct a design space of process-relevant measurements, which can be used as a template for future formulations and enable process behaviour to be predicted with confidence.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched and it is not uncommon for the same powder to perform well in one process but poorly in another. This means that several characterisation methodologies are required, the results from which can be correlated with process ranking to produce a design space of parameters that correspond to acceptable process behaviour. Rather than relying on single number characterisation to describe behaviour across all processes, the FT4’s multivariate approach simulates a range of unit operations, allowing for the direct investigation of a powder’s response to various process and environmental conditions.

For further information, please contact the Applications team on +44 (0) 1684 851 551 or via support@freemantech.co.uk.
Microcrystalline Cellulose (MCC) has been used as a pharmaceutical excipient for many years, due to its abundance, ease of production and resistance to degradation. As with any excipient, however, variability between batches of supplied material can in turn lead to similar variability in processing, resulting in product that is out of specification, and needs to be reworked or even scrapped. Recent Quality by Design (QbD) initiatives have made it necessary to optimise production processes to ensure the consistency and reliability of final products.

A robust method of quantifying the variations in batches of excipients that contribute to differences in downstream process behaviour enables a design space of acceptable raw material properties to be established. This approach is an essential part of QbD. However, even well-established techniques for material characterisation (such as particle size analysis) do not always provide the required differentiation, as they only evaluate of one physical property of the particles.

MULTIVARIATE ANALYSIS OF POWDER BATCHES

Three grades of MCC were used as an excipient in the production of pharmaceutical tablets by direct compression. Particle size analysis of the three grades generated almost identical D50 values (100 µm) for each grade, and it was determined that three samples were indistinguishable from each other.

All three samples were further analysed using an FT4 Powder Rheometer®, to evaluate whether differences existed that weren’t identified by the D50 value and consequently whether particle size alone is sufficient as a tool predicting in-process performance.

TEST RESULTS

Dynamic Testing: Aeration

Sample C generated a significantly higher Aeration Ratio (AR) than the other samples, demonstrating that its packing structure changes to a greater extent when air is introduced into the sample. This typically indicates a lower degree of cohesive strength between particles. Sample A and Sample B exhibited different responses, with the lower sensitivity to the introduction of air suggesting greater cohesivity compared to Sample C.

![Aeration Ratio at 4 mm/s](image)

Bulk Testing: Permeability

Sample C generated a considerably higher Pressure Drop across the Powder Bed than the other two samples, indicating that it is the least permeable of the three. Low permeability means that any air that becomes entrained in the bulk is less able to escape. Considering the example of a tabletting process, greater air content within the dose would typically lead to capping and lamination as well as weight variation in the final product.

![Pressure Drop across the Powder Bed, mBar](image)
Shear Cell Testing

<table>
<thead>
<tr>
<th>Batch</th>
<th>Cohesion, kPa</th>
<th>UYS, kPa</th>
<th>MPS, kPa</th>
<th>FF</th>
<th>AIF, °</th>
<th>BD,  g/mL</th>
<th>SSI, kPa @ 7.08 kPa</th>
<th>SSI, kPa @ 3.06 kPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A</td>
<td>1.17</td>
<td>3.45</td>
<td>15.6</td>
<td>4.52</td>
<td>21.8</td>
<td>0.385 (±0.158%)</td>
<td>3.94 (±0.15%)</td>
<td>2.34 (±0.69%)</td>
</tr>
<tr>
<td>Sample B</td>
<td>-0.0765</td>
<td>n/a</td>
<td>14.9</td>
<td>n/a</td>
<td>34.1</td>
<td>0.390 (±0.321%)</td>
<td>4.67 (±0.371%)</td>
<td>1.98 (±1.39%)</td>
</tr>
<tr>
<td>Sample C</td>
<td>0.0621</td>
<td>0.237</td>
<td>15.5</td>
<td>65.3</td>
<td>34.8</td>
<td>0.399 (±0.381%)</td>
<td>4.92 (±1.14%)</td>
<td>2.17 (±1.88%)</td>
</tr>
</tbody>
</table>

Sample A showed considerably different behaviour to the other two samples, generating the highest Shear Stress values at low normal stress levels, but the lowest values at higher levels. This suggests that the way the powders perform in a given process would be heavily influenced by stress levels they are subjected to, and illustrates the need to characterise powders using process relevant techniques. Considering the results for Sample B, the Yield Locus, generated by a best-fit line through the data points, is so steep that it intercepts the y-axis below the origin. This leads to a negative value for Cohesion, and generates no result for Flow Function (as the minor Mohr circle cannot be constructed in order to produce a value for Unconfined Yield Strength). This illustrates the importance of recording actual measured Shear Stress values alongside parameters derived from Mohr’s circle analysis, as the mathematical model used to obtain these parameters may not always provide data for comparison.

CONCLUSIONS

The FT4 has demonstrated clear and repeatable differences between three grades of MCC, suggesting a likely difference in their process performance which is not identified by the single physical property of particle size alone. Of the three samples, Sample A displayed the highest Permeability and the lowest sensitivity to Aeration, as well as being less sensitive to changes in applied stress during Shear Cell tests. This suggests that it is the most efficiently packed of the three samples and is to exhibit more free-flowing behaviour across a range of conditions. Sample C generated the highest Shear Stress values, was most sensitive to Aeration and exhibited the lowest Permeability, suggesting a greater sensitivity to the process conditions than the other samples and illustrating the need to understand the relationship between the process environment and the material properties.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched and it is not uncommon for the same powder to perform well in one process but poorly in another. This means that several characterisation methodologies are required, the results from which can be correlated with process ranking to produce a design space of parameters that correspond to acceptable process behaviour. Rather than relying on single number characterisation to describe behaviour across all processes, the FT4’s multivariate approach simulates a range of unit operations, allowing for the direct investigation of a powder’s response to various process and environmental conditions.

If you have any questions or would like to learn more about powder characterisation, please contact our Applications team on +44 (0) 1684 851551 or email support@freemantech.co.uk.
When working with powders, in-process performance and final product quality will be influenced by properties of the raw materials delivered to the process. The ability to control these raw materials, and therefore reliably predict process performance, is essential to ensuring the properties and quality of the final product. A method of accurately measuring process-relevant properties of incoming raw materials is therefore highly beneficial to a wide range of applications in various industries. This application note considers the evaluation of bulk excipients for tableting and the impact of storage and handling conditions.

### VARIATIONS IN PROCESS PERFORMANCE AND PRODUCT QUALITY

Sorbitol was used as the bulk excipient in production of a tablet. The tablet manufacturer experienced variation in final product quality when using two batches of sorbitol that had previously been categorised as identical by the supplier. Tablets produced from one batch were consistently softer and less stable than those from the other batch. Following further investigation at the customer’s request, the excipient supplier was unable to differentiate between the batches using techniques they had available.

The only difference identified recorded about the batches was the location in which they had been stored before being supplied to the customer: the well-performing batch was stored in a silo close to the loading area (Silo 1), and the poorly-performing batch in a Silo further away (Silo 2), necessitating additional conveyance prior to processing.

Samples from both batches were analysed using an FT4 Powder Rheometer®.

### TEST RESULTS

**Dynamic Testing: Basic Flowability Energy**

The sample from Silo 2 generated a higher Basic Flowability Energy (BFE) than the sample from Silo 1. This indicates that it was more resistant to dynamic flow in a confined space (such as in a screw conveying or mixing process) requiring more energy to achieve the same outcome.

**Bulk Testing: Compressibility**

The sample from Silo 2 was more compressible than the sample from Silo 1, indicating that it entrained a greater proportion of air within its bulk, which is typically a property of more cohesive powders. Higher Compressibility can contribute to poorer behaviour in processes where a powder is subjected to an applied force, such as in mechanical feeders or tablet presses, or simply as a consequence of storage in large quantities.
Bulk Testing: Permeability

Similarly, the sample from the Silo 2 generated a higher Pressure Drop across the Powder Bed under a constant throughput of air, indicating that this sample was less permeable than the sample from the Silo 1. Low permeability can contribute to poor performance in operations where a powder is required to release entrained air, such as die filling and tableting, and can adversely impact gravitational flow in general.

Shear Cell Testing

Counter-intuitively, the sample from Silo 2 generated slightly lower Shear Stress values following consolidation at 9 kPa than the sample from Silo 1. This suggests it would more readily transition from a static, consolidated state in to a state of flow, such as when required to flow from a hopper. This illustrates how Shear Cell test results may not always be most relevant as the test conditions do not represent what the powder is subjected to in process. Even when evaluating hopper flow, shear properties alone may not fully describe performance, as Permeability is also known to be influential.

The yield loci for the two samples are non-linear and suggest that the powder from Silo 2 may generate higher Shear Stress values at lower levels of consolidation. Furthermore, due to the location and shape of the yield loci, Mohr circle’s analysis of the data could not be completed as a linear best fit line intercepts the y-axis below zero.

CONCLUSIONS

The FT4 has identified clear and repeatable differences between two very similar materials that performed differently in a process. The sample from Silo 2 demonstrates a greater resistance to forced flow (higher BFE), higher Compressibility and lower Permeability, which are all indicators of greater cohesivity. This information could be used to screen raw materials in order to identify those that are suitable for processing and those that are likely to be problematic. Furthermore, the results demonstrate that Shear Cell testing alone may not provide a reliable representation of powder behaviour in this process, due to the differing stress and flow regimes present.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched and it is not uncommon for the same powder to perform well in one process but poorly in another. This means that several characterisation methodologies are required, the results from which can be correlated with process ranking to produce a design space of parameters that correspond to acceptable process behaviour. Rather than relying on single number characterisation to describe behaviour across all processes, the FT4’s multivariate approach simulates a range of unit operations, allowing for the direct investigation of a powder’s response to various process and environmental conditions.

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In spray coating applications, a fine powder, typically a polymer, is drawn from a storage device before being fluidised and ejected through a charged nozzle onto a substrate. It is essential that the powder can be effectively and consistently fluidised, without the formation of agglomerates that may block the nozzle and affect the charging of the individual particles, leading to poor adhesion or the formation of agglomerates on the substrate. It is also essential to establish a smooth flow from the storage device, as erratic flow into the fluidisation chamber will lead to a poorly fluidised bulk.

Identifying and quantifying which powder properties correlate with the most efficient performance in a process allows new formulations to be optimised without the significant cost of running samples through the process to assess suitability, making considerable savings in terms of time and raw materials, and minimising wastage due to rejected products.

VARIATIONS IN PROCESS PERFORMANCE AND PRODUCT QUALITY

Three samples of a polymer powder were used in a spray coating application using a corona charging system. Sample A exhibited good performance in terms of flowing through the nozzle and adhering to the substrate, and Sample B showed acceptable behaviour, but Sample C was poor in both aspects; causing blockages in the nozzle and subsequently falling away from the substrate during transportation to the kiln. Particle size analysis concluded that all three powders had the same D50 and size distribution.

Samples from the three batches were analysed using an FT4 Powder Rheometer®. Clear and repeatable differences were observed between the samples in multiple tests, rationalising the variation in performance and allowing future batches to be screened prior to introduction to the process.

TEST RESULTS

Dynamic Testing

![Graph showing Dynamic Testing results] (Sample A generated the highest Basic Flowability Energy (BFE) and Specific Energy (SE) of the three samples, which together indicate greater cohesion and particle-particle interlocking. Sample C generated the lowest BFE and SE, suggesting that a degree of inter-particular cohesion is required to form a uniform coating on the substrate, and that Sample C does not meet this criterion.

Bulk Testing: Compressibility

Sample C was the most compressible of the samples, indicating a greater propensity to compact under forced flow conditions, such as when drawing the powder from the storage vessel into the fluidisation chamber. The greater propensity to compact will promote the formation of agglomerates, inhibiting both the spraying and charging operations in the nozzle.)
**Bulk Testing: Permeability**

Sample A generated the lowest Pressure Drop across the Powder Bed indicating that it is the most permeable. This suggests that it will be the most free-flowing under conveyance, and that once fluidised, is likely to flow more readily within an air stream. Sample C was least permeable, generating the highest Pressure Drop across the Powder Bed, which is likely to cause more erratic, pulsatile flow into the fluidisation chamber, and unstable flow of the fluidised mass.

![Graph showing Bulk Testing: Permeability](image)

**Shear Cell Testing**

No differentiation was observed during Shear Cell testing, with the measured Shear Stress values of the three samples identical to within an RSD of 2.5%. The lack of correlation with the process performance indicates that the highly consolidated, low-flow environment of the Shear Cell test is not indicative of behaviour in the dynamic, aerated environment of a fluidisation operation.

![Graph showing Shear Cell Testing](image)

**CONCLUSIONS**

The FT4’s multivariate approach has identified clear and repeatable differences between the three powder samples in terms of Dynamic and Bulk properties, which correlate well with in-process performance. Furthermore, the results demonstrate that Shear Cell testing alone does not provide a reliable representation of powder behaviour in this process, due to the differing stress and flow regimes present. Sample A has the highest BFE, SE and Permeability, and the lowest Compressibility, of the three samples. This suggests that a degree of cohesion is required to form a uniform coating but susceptibility to agglomeration and erratic flow is problematic to the process. Sample C, with the lowest BFE and Permeability, and the highest Compressibility, is most sensitive to compaction during conveyance to the fluidisation chamber, forming agglomerates that can block the nozzle and cause inconsistent charging.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched and it is not uncommon for the same powder to perform well in one process but poorly in another. This means that several characterisation methodologies are required to fully characterise powder behaviour in a range of operations, and rather than relying on single number characterisation to attempt this, the FT4’s multivariate approach simulates a range of unit operations, allowing for the direct investigation of a powder’s response to various process and environmental conditions.

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The size and shape of the particles within a powder are important factors in influencing behaviour, and many well-established techniques exist for measuring these properties. However, over a range of different processes, the relationship between these two parameters and flow properties is less well-understood, and due to the varying demands powders are subjected to by different processes, the effect of the two parameters may not be consistent or easily quantified.

This application note explores the influence of particle size and shape on powder flowability under a range of different conditions.

MEASURING PARTICLE SIZE AND SHAPE

Three samples of lactose used as a pharmaceutical excipient (FlowLac100, SpheroLac 100 and InhaLac 230 – Meggle) were evaluated for particle size and shape. Two of the samples (FlowLac100 and SpheroLac100) showed very similar particle size distribution and D50 but a different overall shape. Another two of the samples (SpheroLac 100 and InhaLac230) had very similar shaped particles, but differed in particle size and distribution.

The three samples were analysed using an FT4 Powder Rheometer®. The results not only differentiated between the samples, but also illustrated how changes in particle size and shape can influence flow properties.

THE INFLUENCE OF PHYSICAL PROPERTIES ON POWDER BEHAVIOUR

Dynamic Testing: Basic Flowability Energy

The three samples generated significantly different Basic Flowability Energy (BFE) values, but the differences in size and shape influenced the results to varying degrees. In this test regime, the difference in shape was most influential, suggesting that particle morphology is an important factor in influencing the behaviour of powders in dynamic, low stress applications such as blending, feeding and conveying.
**Bulk Testing: Compressibility**

Significant differences are also observed in the Compressibility of the three samples, but under these conditions, the influence of particle size is greater than that of particle shape, as it has a greater impact on the way particles pack. Particle size is therefore likely to be more dominant in describing the behaviour of powders during consolidating operations such as long-term storage or tablet pressing.

**Shear Cell Testing**

Less differentiation was observed between the samples during Shear Cell tests, suggesting that particle shape is potentially more influential than particle size in high stress operations where a powder is required to transition from a static to a dynamic state. Under consolidation, mechanical interlocking and friction have strong influences over flow properties, and therefore smoother, more regular shaped particles are likely to flow more easily than rough or angular particles.

**CONCLUSIONS**

Particle size and shape are important properties that will influence how a powder behaves. However, the extent of their influence also depends on the conditions to which the powder is subjected to during processing. The unique, multivariate approach of the FT4 Powder Rheometer enables operators to quantify how changes in physical properties will affect a given unit operation by subjecting the powder to the type of stress and flow regimes it will experience in process.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched, and due to the range of demands placed on a powder by different unit operations, it is not uncommon for the same powder to perform well in one process but poorly in another. It is therefore important to ensure that the test conditions for a powder match the conditions of the process as closely as possible, and to use multiple tests to evaluate a range of conditions, as the trends illustrated by a single test under a single condition may not fully characterise the behaviour in a given unit operation.

If you have any comments or questions relating to this document, please feel free to contact our Applications team on +44 (0) 1684 851551 or email support@freemantech.co.uk.
The high level of performance and low environmental impact associated with powder coating makes it an attractive option for a range of applications. Powder coating technology does not require a solvent and therefore avoids the generation of volatile organic emissions associated with their use. However, the need to fluidise a fine powder so that it behaves like a liquid, presents its own challenges and requires materials that flow readily and uniformly during the process.

Powder characterisation using the patented FT4 Powder Rheometer® provides an accurate, sensitive and reliable evaluation of powder flow properties. The FT4 aids formulation of new products, helps optimise handling and processing and can provide quality control standards that lead to improvements in productivity and quality. Factors that influence powder flowability can be independently assessed allowing information databases to be established that compare to those widely used to understand solids and fluids.

The FT4 Powder Rheometer (Figure 1) is a universal powder tester that employs a range of complementary test methodologies for the analysis of dynamic flow, bulk and shear properties.

The dynamic flow properties of a powder are determined by measuring the energy required to cause a powder to flow. This energy is calculated from measurements of the force and torque acting on a blade as it moves along a helical path, through a previously conditioned sample. A downward traverse of the blade produces a ‘bulldozing’ or compacting action within the sample. The energy can also be measured while the sample is being aerated by passing air through a porous base at the bottom of the powder column.

Powder B exhibits minimal resistance to the motion of the blade at air velocities greater than 1mm/s, indicating how this material becomes fluidised above this air velocity. Conversely, powder A, which is known to be problematic during processing, never fluidises. The ability of powder B to become fluidised at this low air velocity helps rationalise why it fluidised well in the hopper, transferred consistently to the gun and resulted in a uniform coating in final application. Powder A performed quite differently, with intermittent flow to the gun and with agglomerates present on the coated surfaces.

The correlation of the FT4 results to the processing experience allows ‘Bad’ batches to be rejected prior to committing them to processing.

CONCLUSION

Powder coating is a demanding process in terms of its requirement for consistent powder flow. The fluidisation characteristics of these powders are particularly important and the FT4 Powder Rheometer provides suitable methodologies that can accurately measure very small flow energy values in order to deliver data that are relevant to the process and therefore extremely valuable to the industry. The FT4 also allows the impact of other key variables, such as moisture, flow additive content, consolidation, attrition and recyclability to be quantified and is therefore an important tool for the optimisation of powder coating processes. This data is invaluable when developing new formulations and troubleshooting existing processes.

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Processing powder blends can present a number of challenges: component powders may be poorly flowing in the process, the blends may be susceptible to segregation, or the formation of agglomerates may affect homogeneity. Granulation is used in a range of industries and applications to combine multiple components of a blend into a more free-flowing, homogeneous intermediate product for downstream processing. It is frequently carried out as a wet process, but the resultant wet mass has to be dried and milled to generate a processable product. This can be time consuming and expensive, and in some cases not possible due to chemical and/or thermal degradation of the active ingredient.

Dry granulation has significant benefits in terms of both processing and cost reduction, and can be used with sensitive materials. However, there is little indication of which process parameters produce optimal granulate quality in order to achieve interruption free processing and high quality products. Most equipment suppliers and product manufacturers therefore rely on historical and ad-hoc trial information to identify suitable parameters.

This application note details the joint study undertaken by Freeman Technology Ltd. and Gerteis Maschinen+Processengineering AG, to investigate how process parameters influenced the properties of the dry granulate of a placebo formulation.

**METHODS**

A placebo formulation consisting of 70% lactose, 29.5% microcrystalline cellulose and 0.5% magnesium stearate was granulated using a Gerteis MINI-PACTOR® roller compactor, on which the Roll Gap, Compaction Force and roller speed can be varied together with the screen/sieve size. The resulting granulates were evaluated using a Freeman Technology FT4 Powder Rheometer® to quantify the dynamic, bulk and shear properties.

### THE EFFECT OF COMPACTION FORCE

Six identical batches of the feedstock were processed in the MINI-PACTOR® at different Compaction Forces:

<table>
<thead>
<tr>
<th>Compaction Force (kN/cm)</th>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.0</td>
<td>4.5</td>
<td>6.0</td>
<td>7.5</td>
<td>9.0</td>
<td>12.0</td>
</tr>
</tbody>
</table>

The roll gap was maintained at 3 mm, the roller speed at 2.5 RPM and the screen size at 1 mm. The resulting six batches of granules were subsequently evaluated using the FT4 Powder Rheometer®, to investigate the effect of Compaction Force on granule properties.

**Conditioned Bulk Density and Compressibility**

**Conditioned Bulk Density**

\[ y = 0.009x + 0.5443 \]

\[ R^2 = 0.8485 \]

**Compressibility Percentage @ 15 kPa**

\[ y = -0.6514x + 21.21 \]

\[ R^2 = 0.9103 \]
Linear correlations were observed in which the Conditioned Bulk Density (CBD) and Compressibility of the granulate varied with Compaction Force, with a higher force generating higher CBD and lower Compressibility.

A greater Compaction Force generates more uniform granules that pack more efficiently. This efficient packing leads to fewer air voids, increasing the bulk density of the material and resulting in less available space into which granules can move when subjected to an applied stress.

**Permeability**

A strong relationship was observed between Permeability and Compaction Force, with a higher force generating higher Permeability.

The granules produced at a higher Compaction Force generate a bulk that is more resistant to compaction. This means that channels between the granules can be maintained when the bulk is subjected to an external stress, allowing air to pass through more easily.

The results show a direct correlation between Compaction Force and CBD, Permeability and Compressibility. As Compaction Force increases, Compressibility decreases and Permeability and CBD of the resultant granulate increase. These properties are all indicative of more efficient packing and typically associated with more free-flowing materials.

In contrast to the Dynamic Flow and Bulk data, shear properties provided had little influence, with the Shear Cell test results providing no differentiation between the samples and no correlation observed between the Wall Friction Angle and Compaction Force. Shear Cells were primarily designed for evaluating the onset of flow for continuous, cohesive powders under high stress, so the lack of correlation to a dynamic, low stress process is not unexpected.

**THE EFFECT OF ROLL GAP**

Six identical batches of the feedstock were processed in the MINI-PACTOR® using different Roll Gaps:

<table>
<thead>
<tr>
<th>Roll Gap (mm)</th>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roll Gap</td>
<td>1.5</td>
<td>2</td>
<td>2.5</td>
<td>3</td>
<td>4</td>
<td>5</td>
</tr>
</tbody>
</table>

The Compaction Force was maintained at 4.5 kN/cm, the roller speed at 2.5 RPM and the screen size at 1 mm. The resulting six batches of granules were evaluated using an FT4 Powder Rheometer®, to investigate the effect of Roll Gap on granule properties.

**Conditioned Bulk Density**

The bulk density of the granules decreased as Roll Gap increased, suggesting the larger gap generates less consistent granules with a wider size distribution.

Materials with a wide PSD typically pack less efficiently, entraining more air and reducing the density of the bulk.
Robust relationships were also observed in which the Consolidation Index (CI) and Permeability of the granulate varied with Roll Gap, with a larger Roll Gap resulting in a greater sensitivity to vibrational consolidation and lower Permeability. This again suggests that a larger Roll Gap generates granules with a wider particle size distribution that pack less uniformly and entrain more air within the bulk. When the granules are subjected to vibration, the particles readily re-align and repack into a more efficiently packed structure, expelling the air and causing a large increase in flow energy. Furthermore, the less uniform packing structure does not allow stable air channels to be established, resulting a reduction in Permeability.

As the Roll Gap increases the resulting granules are less uniform due to the less consistent consolidation regime established between the rollers. This is likely to result in greater variation in size distribution, shape and surface texture of the granules, manifested by a reduction in particle packing efficiency as demonstrated by the higher CI value, and lower Permeability and CBD values.

**VARIATION OF COMPACTION FORCE AND ROLL GAP**

Nine identical batches of the feedstock were evaluated at varying levels of Compaction Force and Roll Gap, to see if these followed similar trends compared to those observed when varying only Roll Gap or Compaction Force.

<table>
<thead>
<tr>
<th>Conditioned Bulk Density and Compressibility</th>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
<th>VII</th>
<th>VIII</th>
<th>IX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roll Gap (mm)</td>
<td>1.5</td>
<td>2</td>
<td>2.5</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>1.5</td>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td>Compaction Force (kN/cm)</td>
<td>4.5</td>
<td>4.5</td>
<td>4.5</td>
<td>4.5</td>
<td>4.5</td>
<td>9</td>
<td>9</td>
<td>9</td>
<td></td>
</tr>
</tbody>
</table>

The granules produced using a 9 kN/cm Compaction Force over a range of Roll Gap values consistently have a higher CBD and a lower Compressibility than those produced at 4.5 kN/cm, supporting the observations in the initial investigations. Comparable linear relationships were observed between Roll Gap and CBD at both 4.5 kN/cm and 9 kN/cm Compaction Force, suggesting that the relationship between Roll Gap and CBD is independent of Compaction Force. However, the relationship between Roll Gap and Compressibility does not show the same independence. At 9 kN/cm, Compressibility exhibits a sharper increase as Roll Gap increases, suggesting that Roll Gap has a greater influence on granule properties at higher Compaction Forces.
The granules produced at 9 kN/cm Compaction Force consistently have a higher Permeability than those produced at 4.5 kN/cm, indicating that the higher Compaction Force generated more uniformly packed, more permeable granules. The results confirmed relationship previously observed between Roll Gap and Permeability, however the curve is significantly more pronounced at the higher Compaction Force, reinforcing the suggestion that Roll Gap has a greater influence on granule properties at higher Compaction Forces.

CONCLUSIONS

Quality by Design dictates that the relationship between materials and processes should be well understood, in order to be able to control and optimise process performance and ensure final product quality. The results generated here demonstrate that it is possible to identify the Critical Process Parameters required to optimise a roller compaction process to produce granules with properties that directly influence performance in downstream operations and Critical Quality Attributes of the final product.

Clear and repeatable trends have been observed in the flow properties measured by the FT4, demonstrating how rheological properties of the granules are predictably influenced by the process parameters. The Permeability, Compressibility and Conditioned Bulk Density of the samples exhibited robust correlations with modes of operation of the roll compactor, with the overall suggestion that a combination of smaller Roll Gap and higher Compaction Force is more likely to result in more uniform/consistent granules which form a more efficiently packed powder bed typically associated with free-flowing powders.

This study indicates the value of powder rheology in a comprehensive, multivariate approach to powder characterisation. Flowability is not a fundamental material property, but instead reflects how multiple properties contribute to the overall ability of a powder to perform in a specific piece of equipment. Subtle variations in an individual property may lead to a noticeable difference in process performance, meaning that several characterisation methodologies are required, the results from which can be correlated with process ranking to produce a design space of parameters that correspond to acceptable process behaviour.

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