A Study of the Flow Properties of Toners in Relation to Physical and Environmental Factors

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Abstract

This is a study of the flow properties of a range of magnetic toners produced from a common base in relation to three physical variables – the milling method, the type and amount of flow additive and to a limited extent, particle size.

Two methods of milling were used for sample preparation – air jet milling and mechanical milling. The flow additives were either HMDS or PDMS silicas.

The study used the FT3 powder Rheometer (Freeman Technology) to determine the rheology of powder samples in terms of the energy required to cause a powder to flow.

The dependence upon environmental factors commonly imposed on powders such as flow rate, the level of aeration and the amount of consolidation are determined for each of the materials. The propensity to retain air is also investigated and quantified. The results clearly indicate the complex nature of toners and how their flow properties are affected by a large number of variables, of which one of the most significant is the effect of aeration and de-aeration representing the transition from fluidisation to consolidation.

Materials

All samples were produced from a magnetic mono-component type toner. One batch was produced by air jet milling and the other by mechanical milling to nominally 9µm size. (Fig1) Samples of each were then blended with different types (PDMS and HMDS) and amounts of silica (0 to 1.2%). PDMS is treated 80-120 m^2/g BET fumed silica and HMDS is treated 230-290 m^2/g BET fumed silica.

The blending with silica was done using a laboratory high shear blender at a speed of 5000 rpm for 10 minutes.

Methodology

The study used an FT4 Powder Rheometer[1] (Freeman Technology) to measure the flow properties of powders in terms of the energy required to make the powder flow. (Figure 2).

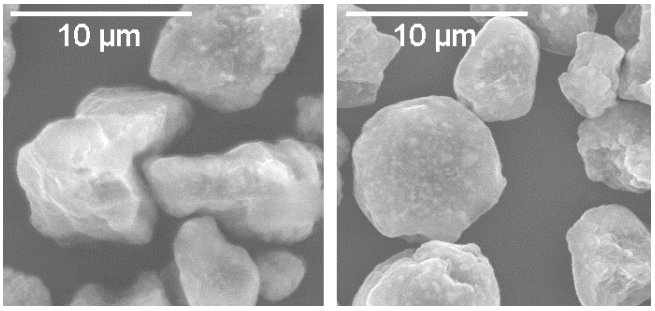

Figure 1. SEM micrographs of air jet milled toner (left) and mechanically milled toner (right).

Figure 2. FT4 Powder Rheometer.

The methodology used 130 ml powder samples that were tested in a 50 mm bore borosilicate glass cylinder or vessel. The powder was made to flow by moving a 48 mm diameter twisted blade rotationally and axially so that it moved along a helical path through the test sample. Samples were prepared for testing by a conditioning process in which the blade caused gentle displacement of the powder to establish a consistent and reproducible packing density. The test cycle that followed moved the blade along a downward helical path, (-10° at a blade tip speed of 100mm/s) but in the opposite direction, to impose compaction, thereby forcing the powder to flow around the blade.

The axial forces and rotational forces acting on the blade during the cycle through the powder were measured continuously and used to derive the work done, or energy consumed, in displacing the powder. This energy is called the Basic Flowability Energy (BFE) and is defined as the energy required to complete a standard test upon a conditioned powder. The BFE is regarded as a measure of the rheology of the powder when in a conditioned state.

A further series of tests investigated how the flow energy requirements were changed in relation to BFE by consolidation of the powders at one extreme and aeration and fluidisation at the other. All materials were tested as above.

Environmental Variables

Packing Condition

As well as the conditioned packing state (described above) used for the standard tests, samples were compacted and also aerated to determine how this affected their flow properties.

Compaction or Consolidation

Consolidation of powders, however caused, is generally detrimental to processing because the number of free particles is much reduced by physical bonding and the elimination of air so that flowability is greatly compromised.

Samples were consolidated prior to testing using two methods - direct incremental compaction to 0.1 bar and tapping 100 times. The standard test was then run (but without conditioning) to determine the increase of energy requirement. This increased energy figure was then divided by the BFE to determine the Compaction Index (CI).

Aeration

Samples were aerated by supplying air to the base of the testing vessel containing the sample. The standard test was repeated at different rates of airflow to determine how the flow energy varied for various levels of aeration. The BFE was then divided by the measured energy required to fully fluidise the powder to determine the Aeration Ratio (AR).

De-aeration

The readiness of the powders to release entrained air was investigated by firstly aerating the powder, then using a gentle displacement mode to de-aerate the powder, followed by a standard test to measure the flow energy requirement. This was repeated for n de-aeration cycles where n = 0 to 4.

Results and Discussion

Bulk Density Measurements

The bulk density of the conditioned air jet milled material without silica was slightly lower (0.60g/ml) than the mechanically milled base material (0.65g/ml). The HDMS silica increased the bulk density by a maximum of 18% and the PDMS silica by a maximum of 13%, roughly in proportion to silica content except for the initial HDMS silica addition that produced a disproportionately high increase.

BFE Measurements

Figure 3 shows how BFE varies with silica content for both air jet milled and mechanical milled toners.

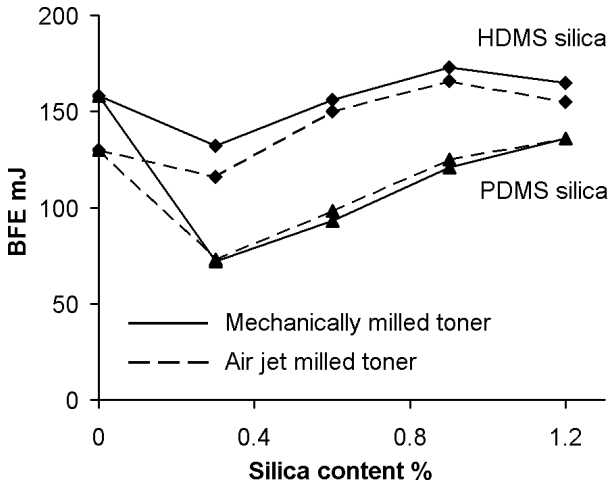

Figure 3. How BFE varies with silica type and content for air jet and mechanically milled toners

Points to note:

- BFE values can be counter-intuitive. A high value is not necessarily an indication of poorer flow properties.
- The HDMS silica produces consistently higher BFE values than the PDMS silica
- Mechanical milling produces marginally higher BFE values than air jet milling
- For all combinations the lowest BFE values occur with 0.3% silica

Compaction – by Tapping 100 Times

Samples were compacted by tapping 100 times and then tested without conditioning. Figure 4 shows how the Compaction Index (tapped) varies with silica content and method of milling.

Points to note:

- High values of CI are usually indicators of poor flowability under storage or low flow rate conditions.
- The base materials with 0% silica, however milled, have the lowest Compaction Index of about 2.2.
- The addition of PDMS silica increases the CI to 7.
- The HDMS silica produces a peak of CI at around 0.3%. This correlates with the disproportionate increase of bulk density mentioned above. Further silica addition reduces the CI value.
- Mechanically milled powders have lower CI values (tapped) than the air jet milled powders.
- Flow additives usually improve the flowability of aerated powders but have a detrimental affect on the flow of compacted powders due to the higher packing density resulting from reduced friction between particles. Closer nestling of particles and more points of contact means that higher energies are required to make the powder flow again.

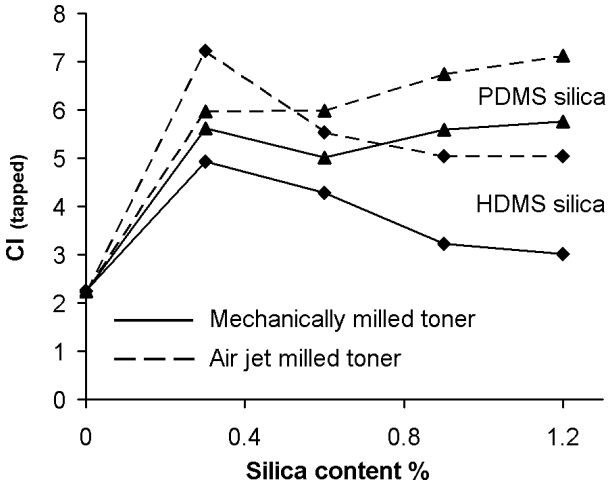

Figure 4. How Compaction Index (CI) of tapped samples, varies with silica type and content.

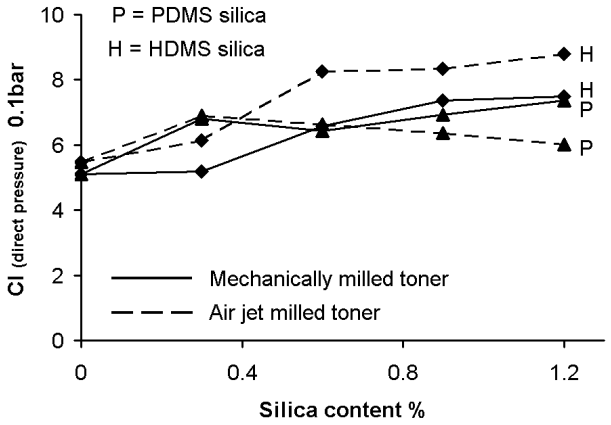

Figure 5. How Compaction Index (CI) of directly compacted samples, varies with silica type and content.

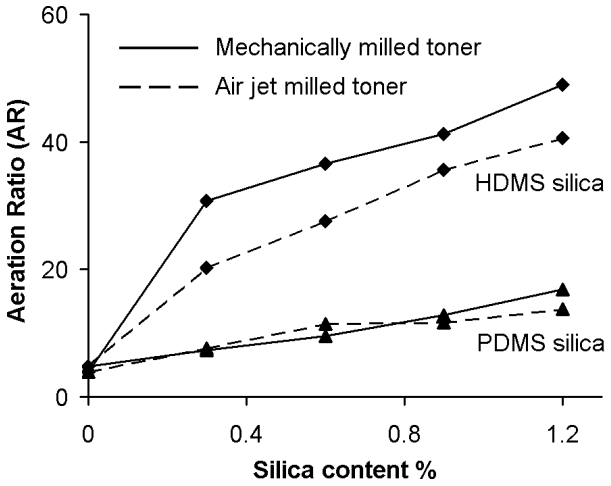

Figure 6. How Aeration Ratio (AR) varies with silica type and content.

Compaction by Direct Pressure

Figure 5 provides similar data to the above for powders that were directly compacted to 0.1 bar before testing.

Points to note:

- The untreated material has a much higher Compaction Index (CI) when compacted by direct pressure than when tapped (5 compared with 2).
- HDMS silica causes a steady increase of CI. This contrasts with the peak seen during the tapped tests. As a result the relative affect of the two silica's at high content is the reverse of the tapped compaction data.
- PDMS silica causes relatively little change.

Aeration

Figure 6 shows how the flow energy requirement is reduced by aeration, as a function of silica content. Energy is expressed in terms of the AR value defined above.

Points to note:

- The higher surface area HDMS silica's are very effective in reducing the BFE value by a factor of 50 creating a free flowing powder.
- Figure 6 shows the AR values for the HDMS silica increasing in rough proportion to the silica content.
- The PDMS silica's also have a proportionate effect but to a lesser extent.
- Mechanically milled powders have higher AR values, which possibly relates to their more spherical shape (See Figure 1).

- All treated powders aerate rapidly and become fluidised at air velocities greater than 0.04cm/s (data not shown).
- It appears that the optimum silica content from a flowability viewpoint may be in excess of the maximum evaluated (1.2%).

De-aeration

Figure 7 shows the de-aeration characteristics (following aeration) of the mechanically milled base powders with varying amounts of HDMS silicas. (Other samples data not shown).

Points to note:

- Silica allows air to be retained readily.
- The smallest amount of silica (0.3%) has a disproportionately high affect.
- The optimum level of silica to enhance air retention may be greater than 1.2%.

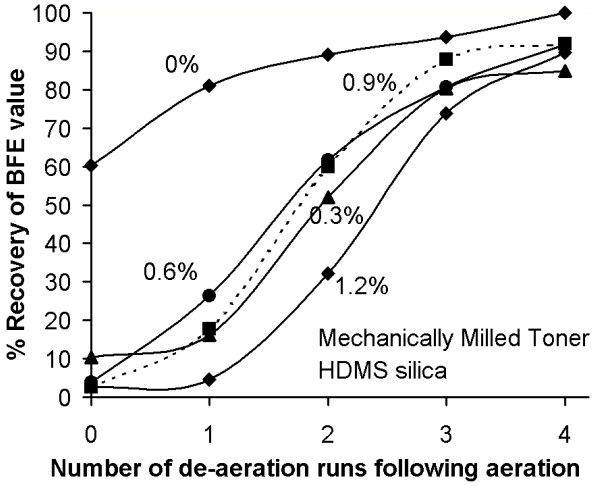

Figure 7. The de-aeration characteristics of various powders due to repeated aeration /de-aeration.

Particle Size

Figure 8 shows the BFE measurements for three air jet milled powders having 0.9% PDMS silica, milled to produce D50 particle sizes of nominally 9, 11 and 13μm.

Coarser particles tend to be less cohesive and to release air more readily so that they generally have higher BFE values, other factors being equal. This is the trend apparent here, though the amount of increase in BFE value is higher than expected.

Figure 9 shows the results of testing two sets of 9, 11 and 13μm powders after consolidation by tapping in one case and by incremental direct pressure in the other.

Points to note:

- The finer powder is less affected by tapping. This is as expected and is attributed to its higher cohesivity and ability to retain its entrained air.
- Conversely, air can be squeezed out of finer powder when directly compacted resulting in a denser powder mass and a higher energy requirement to cause flow.
- Coarser powders that more readily release their air, have higher CI (tapped) values and lower CI (direct pressure) values.

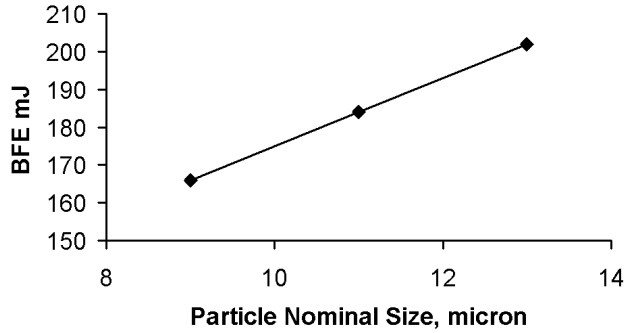

Figure 8. BFE variation with particle size

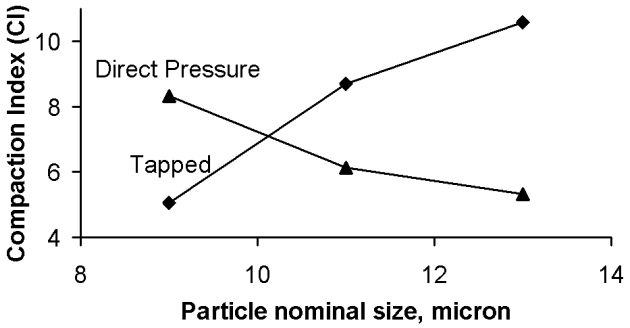

Figure 9. How Compaction Index varies with particle size and the method of consolidation used.

Conclusions

Whilst air jet milling and mechanical milling produce different particle shape and therefore different rheology, the type and quantity of silica and the nominal particle size of the toner material has a more significant impact on flow properties.

The BFE measurement, which is a yardstick for describing the rheology of the powders, was consistently higher for the HDMS silica. In all cases the 0.3% silica samples had the lowest BFE values.

The results show large differences in the flow behaviour of compacted powders depending upon the method of compaction. Neither of the base powders is greatly affected by tapping – only doubling their energy requirement, whereas direct pressure compaction resulted in a Compaction Index (CI) of 5. This reflects their cohesive behaviour and reluctance to release entrained air.

Flow additives usually improve the flowability of aerated powders, but often have a detrimental effect on the flow of compacted powders due to closer packing resulting

from reduced friction between particles. Nestling of particles, less air and more points of contact means that higher energies are required to make the powder flow again. This was generally the case for the materials containing silica where the CI value increased to more than 8. However, the HDMS silica is an exception in relation to tapped consolidation, where the peak CI value occurs at 0.3% and reduces at higher silica content. This disproportionate affect correlates with bulk density and seems to indicate that 0.3% silica content allows close packing by reducing interparticle friction but is not highly effective in allowing the powder mass to subsequently unpack. The later is corrected by increasing amounts of silica as shown by the significant reduction in CI values – especially for the more rounded, mechanically milled toners. It is interesting that this does not occur with PDMS silica nor when the consolidation is produced by directly compressing the powder. In summary, whilst these flow additives do not improve the flow performance of consolidated powders, the higher surface area, HDMS silica is the better choice given the need to use a flow additive.

The benefits of a flow additive were clearly seen in relation to the flow performance of aerated toners. The HDMS silica reduced the flow energy requirement when fully aerated to less than 2% of the BFE value creating a very free-flowing powder. The PDMS silica having lower surface area is less effective. All samples containing silica fluidised readily requiring an air velocity of only 0.04 cm/sec to do so. As expected, the base material without silica was much less affected by this amount of air, and even when fully aerated, required much greater energy to promote flow. The results show an improvement with increasing silica content and the optimum may well be in excess of the maximum 1.2% evaluated. The de-aeration results demonstrate how the propensity of toners to retain air may be quantified. They indicate that air retention is increased by silica and that again, the optimum may be in excess of 1.2%.

Particle size has a significant impact on the rheology of most powders–including toners. Nevertheless, the difference between the 9 and 13 micron powders is greater than expected in relation to both compacted and aerated flow performance – at least in the case of the 0.9% PDMS silica.

Optimising the formulation and performance of toners requires a comprehensive understanding of their rheology and the many factors that play a part. With recent developments in instrumentation this is now possible.

References

1. Freeman Technology website www.freemantech.co.uk

Biographies

Ian Neilson obtained a B.Sc. (Hons) Degree in Physics from the University of Exeter in 1973. From 1974 -1976 he was employed by Gestetner Ltd. as a Research Physicist.

Ian joined Coates in 1976 and has been involved in the development of toners and pigmented phase change jet inks. He has formulated highly successful pressure sensitive, radiant, flash fusing magnetic and coloured toners for Electron Beam Imaging, in which Coates is a world leader. In this area he has been granted US and European patents for a fluid bed applied toner.

Currently he is employed as Research & Development Manager with responsibility for all toner development.

Reg Freeman is a mechanical engineer with extensive experience in designing testing systems for evaluating the physical properties of liquids and solids. He is CEO of Freeman Technology, the company he founded in 1988, which specialises in measuring the flow properties of powders through the provision of the patented FT4 Powder Rheometer. Reg is the recipient of a BT Entrepreneur of the Year Award and most recently a Smart Achievement Award. Email: reg@freemantech.co.uk