COMPARISON OF MAGNETICALLY ASSISTED IMPACTION COATING (MAIC) WITH TRADITIONAL MIXING TECHNIQUES FOR THE ADDITION OF A SILICA FLOW AID

Tim Freeman[1], William A. Hendrickson[2], Charles R. Bowman[2], Christopher J. Rueb[2], Robert G. Bowman[2], Katrina Brockbank[1], Jamie Clayton[1]

[1]Freeman Technology, Tewkesbury, Gloucestershire, UK
[2]AVEKA Inc., Woodbury, Minnesota, USA

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Introduction

Many powders have poor basic flow properties, they block in hoppers and die feed frames, exhibit inconsistent or pulsatile discharge rates, adhere to equipment surfaces or do not mix readily with other materials. Additionally, many powders tend to cake on storage or during transport due to changes in environmental conditions and/or consolidation. As such, flow aids are frequently added to a blend to enhance flow properties and/or inhibit caking.

In order to optimise the influence of a flow aid, it must be dispersed in a fine, uniform layer on the surface of the substrate particles. However, most flow aids, for example fumed silica or magnesium stearate, consist of very fine particles that have a tendency to form agglomerates. The mixing/coating process employed can therefore have a major influence on how efficiently the flow aid is distributed. The process must be intense enough to disperse the agglomerated flow aid without damaging the particles of the substrate or the flow aid itself. This is particularly true for many food and pharmaceutical ingredients which are often relatively friable and easily deformed by extreme mechanical forces [1].

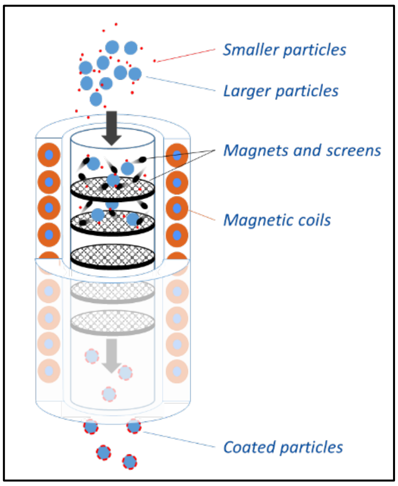

Figure 1 - Magnetically Assisted Impaction Coating (MAIC) process

Magnetically Assisted Impaction Coating (MAIC) has been demonstrated as a technique for coating host particles without causing major changes to particle shape and size [1,2]. This system relies on a pounding process in which an oscillating magnetic field is used to accelerate and spin large magnetic particles (600-800 µm, though size may vary with application), causing the magnetic particles to fluidise, through which the host and guest particles (flow aid) are passed in a continuous fashion (fig 1). The agitated magnetic particles impart energy to the host and guest particles, and coating is then achieved by means of impaction or peening of the guest particles onto the host particles as collisions occur between particles and between particles and the vessel wall [1].

Materials and Methods

Jet milled citric acid (Hawkins Chemicals, jet milled by *AVEKA*) was pre-blended with 0.05%, 0.5%, and 2.0% (w/w) fumed silica (Aerosil 200 Pharma, Evonik) using a V-blender with intensifier bar for 5 minutes. The blends were then fed through the MAIC process (US Patent 5962082 [2]). The samples were run through the MAIC (*AVEKA*, USA) in increasing mass percent of silica, so as to minimize the impact of potential contamination of subsequent samples. Prior to this, citric acid was passed through the MAIC without any treatment with fumed silica, and without being placed in the V-blender. In addition to the MAIC-treated samples, two v-blended samples not subjected to MAIC were created, with 0.5% and 2.0% (w/w) fumed silica. The untreated citric acid made for the seventh and last of the evaluated samples.

After being randomized, the samples were labeled without any indication of processing method (V-blend only or MAIC-treated) or silica loading, which facilitated a blind study of the flow characteristics. All packaged samples were then shipped from Minnesota, USA by air to Tewkesbury, UK. Each sample was then evaluated for flow on an FT4 Powder Rheometer (Freeman Technology, UK) before the identity of each sample was revealed and matched to the relevant data.

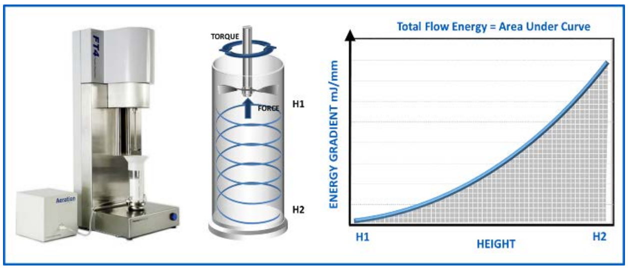

Figure 2 – Measurement of the flow energy using the FT4 Powder Rheometer

The FT4 measurements included dynamic flow, bulk and shear properties. Dynamic properties, including Basic Flowability Energy (BFE) (fig. 2), were 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 measurements was enhanced by the use of a conditioning cycle which removes packing history and operator-induced variability.

Results and Discussion

This study compared MAIC with V-blending through comparison of the flow properties of the substrate after the addition of silica. In addition, this study also investigated the effect of silica concentration on samples blended via MAIC.

MAIC vs. V-Blending

The MAIC blends at both silica concentrations (0.5% and 2.0% (w/w)) presented lower BFE, Aerated Energy (AE), Cohesion, Consolidation Index (CI) and Pressure Drop values indicating improved flow properties compared with the V-blended samples (fig. 3).

BFE measures the resistance to flow in a dynamic, constrained forced flow environment, for example feeding via a screw conveyer. Whilst all the silica blends exhibited improved flow compared to the raw material, the MAIC blends exhibited the largest decrease in flow energy, suggesting the greatest improvement in flow. This is due, in part, to the fact that MAIC creates a more uniform coating with the flow aid than can be achieved by V-blending alone [4].

The silica blends also exhibited a decrease in Specific Energy (SE) compared to the raw material suggesting reduced mechanical interlocking and friction. However, there were limited differences between the MAIC and V-blended samples at 0.50% (w/w) silica; although at the higher load of 2.0% (w/w) silica, the MAIC blend presented a greater improvement in flow compared with the V-blended sample.

The lower AE values presented by the MAIC samples not only indicate that they were more responsive to the introduction of air, but also demonstrated a clear reduction in the absolute cohesion compared with the V-blended samples. This is also indicated by the lower cohesion values obtained during the shear tests suggesting that the MAIC samples were also less resistant to flow at higher (applied) loads. This behaviour has been previously observed on a batch MAIC process, and used to improve the ability of a powder to fluidize [5].

Powders can also undergo compaction when exposed to external vibrational forces, for example during transport or due to proximity to (other) process equipment, resulting in reduced flowability. The V-blended samples both exhibited much larger increases in flow energy with notably higher Consolidation Index (CI) values following tapping, demonstrating that these samples were much more sensitive to vibrational forces and indicating reduced flow when exposed to external forces. Overall these results suggest that the MAIC blends exhibited improved flow properties at low, moderate and high stress regimes, and may be more stable during storage.

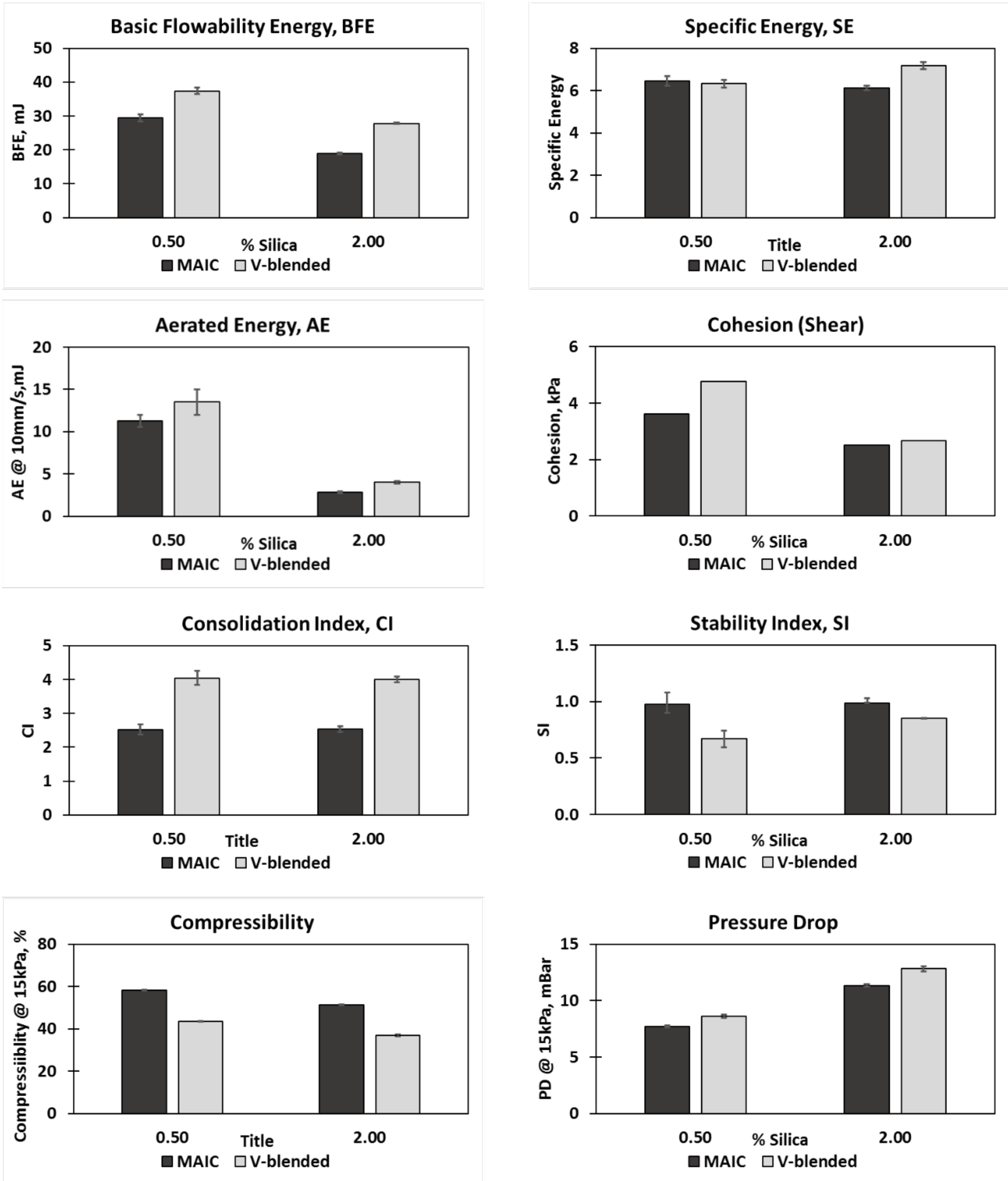

Figure 3 – FT4 Powder Rheometer Results for MAIC and V-Blended samples with 0.50% and 2.0% (w/w) fumed silica

The FT4 dynamic flow tests also provide an indication of the physical stability of the powder by measuring the change in flow energy over repeat test cycles. Whilst the MAIC blends were demonstrated to be physically stable over 7 test cycles, the V-blended samples presented a marked decrease in flow energy with Stability Index (SI) values less than 1, indicating that resistance to flow decreased as the sample was tested. This suggests that the fumed silica was not fully dispersed at the start of the test, with the mixing action of the blade as it traversed the powder bed resulting in a more uniform and wider dispersion of the fumed silica over the duration of the test. This in turn resulted in the decrease in the measured flow energy. It should be noted that these measurements were made after the samples were shipped via air from the USA to the UK. This may have generated stress conditions in the powder samples prior to testing which the MAIC process appears less affected by.

While the MAIC samples clearly showed reduced cohesion during flow, they were also demonstrated to contain more entrained air compared to the V-blended samples with lower Bulk Density (in both the Dynamic Flow and Compressibility tests) and Compressibility values. Flow aids improve particle flow behaviour by acting as a surface lubricant, reducing mechanical interlocking and friction, and/or increasing interparticular spacing, thereby removing agglomerates. Increased particle spacing may partially account for the increases in entrained air, however, the bulk of the entrained air is more likely to be due to improved separation of both the host and guest particles. Whilst this will allow for a more uniform distribution of the fumed silica on the surface of the citric acid particles as more of the host surface will be exposed, it will also cause the powder to mix with air which will subsequently be trapped in the powder bed.

% Loading

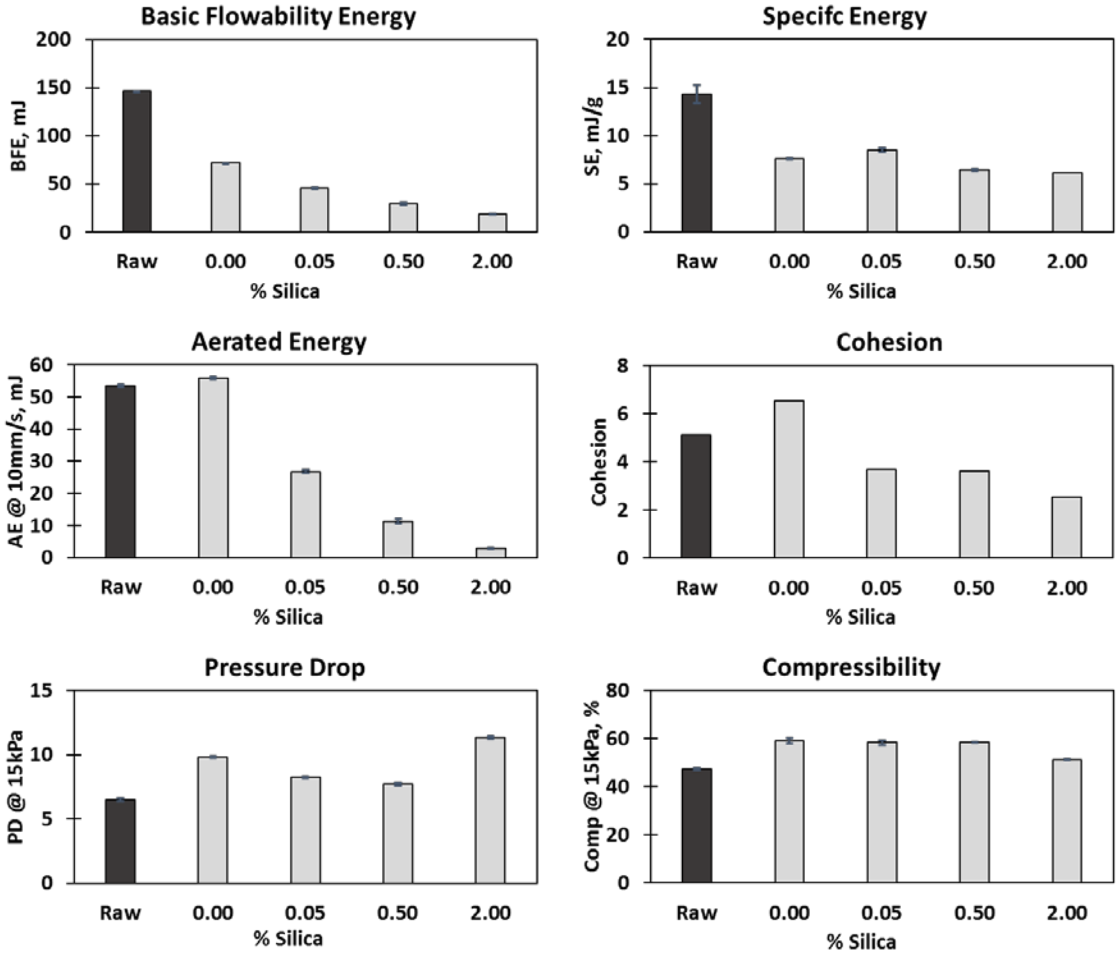

Figure 4 – FT4 Powder Rheometer Results for MAIC blended sample with increasing % silica (w/w)

As demonstrated by the results in Fig. 4, the MAIC blends exhibited a clear decrease in the BFE with increasing silica loading, with the 2.0% (w/w) presenting the lowest BFE. However, a marked decrease in flow energy was also demonstrated between the raw sample and the 0% (w/w) blend with a similar decrease demonstrated by the SE. This indicates that the MAIC process can improve reduce the resistance to flow in a low-moderate dynamic regime without the addition of silica. Indeed, there were only minor reductions in SE with addition of silica at higher loads (at 0.05% (w/w) the SE was actually slightly higher), suggesting the mechanical interlocking and friction had minimal influence following MAIC.

Improvements in the flow properties of the raw material following MAIC may be due to a number of factors. As previously mentioned, both the Bulk Density and Compressibility values indicate that the treated samples contain more entrained air, suggesting improved separation of the particles and wider interstitial spacing. The higher Pressure Drop values also suggest less efficient particle packing. Improved separation may result in the breakage / reduction of liquid and solid bridging, whilst wider interstitial spacing will decrease short range forces, such as van der Waals forces (effective range of approximately 100 nm) [6], and mechanical interlocking and friction. Collisions between the agitated magnetic particles and substrate may also result in the loss of surface asperities, smoothing the particle surfaces and again reducing mechanical interlocking and frictional effects, although SEM has shown limited changes in the particle physical properties. It may also be that in the absence of flow aid, the MAIC process attaches the fine particle fraction (of the citric acid) to the larger citric acid particles in the bulk, not only removing the smaller and therefore typically more cohesive particles from the bulk but also increasing interparticular spacing between the larger particles.

With respect to the effect of silica loading on the particle packing properties (as demonstrated by Compressibility and Permeability), the silica concentrations appear to have minimal effect below 2.0% (w/w) silica and even at 2.0% (w/w) there is only a slight improvement. This may be due to the reduced cohesion allowing for improved particle packing, or excess silica filling interstitial spaces. Either way, the number/size of air pockets and channels which allow for the passage of air would be reduced, explaining why the Pressure Drop increases at 2.0%(w/w) silica, whereas it presented minor decreases at lower concentrations.

Interestingly, when the effects of particle packing are negated, either by fully aerating the powder bed or through compaction (shear), the 0% (w/w) blend is marginally more cohesive compared to the raw sample with a slightly higher Aerated Energy (AE) and Cohesion values. With the addition of silica both tests present a clear improvement in flow properties with the lowest AE / Cohesion values presented at 2.0% (w/w) silica. The Aeration test presents a similar trend to the BFE with a clear reduction in the AE with increasing silica loading whereas Cohesion presents limited variation between the 0.05% (w/w) and 0.5% (w/w) blends despite a 10-fold increase in silica concentration (this is also shown by other shear parameters including Angle of Internal Friction and Flow Function). Indeed, a 10-fold increase in silica did not result in similar magnitude of change in flow properties for any of the measured parameters, as such while higher concentrations may exhibit the largest improvements, as long as the powder is fit for purpose at lower silica concentrations, further benefits may not outweigh the added costs and complexities of increasing the silica concentration. Furthermore, previous studies have shown that continuing to increase the flow aid concentration can be detrimental to flow as once the point of surface saturation is reached, the flow aid remains loose in the powder bulk adding to the total cohesion [7].

Conclusion

Although both the V-blended and MAIC samples exhibited a clear improvement in flow properties compared to the raw materials, the results were more pronounced for the MAIC samples, which were shown to be less cohesive across a range of stress regimes and modes of flow. The MAIC also resulted in a more conditioned uniform blend that is more resistant to changes in its flow properties, even after extensive shipping and handling. In terms of sample loading, whilst the 2% (w/w) silica blend was shown to be the least cohesive in a number of tests, the marked increases in silica concentrations (from 0.05% (w/w) to 2.0% (w/w)) did not yield similar magnitudes of improvement in the flow properties. As such, once the added costs and complexities of substantially increasing the silica concentration are considered, a lower silica loading may be more beneficial as these still return a marked improvement in flow behaviour. Interestingly, even without the addition of silica, the MAIC process appear to have some positive effects on the flow properties, however the reason for this requires further investigation. Overall, it can be concluded that MAIC shows an advantage over V-blending when adding flow aid.

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