# Monitoring of Powder Homogeneity During Double-Cone Blending

Alex Wartenberg, Sierra Mirtes and Chris Schade
Hoeganaes Corporation
Cinnaminson, NJ 08077

Sarah Ackermann
C-Therm Technologies Incorporated
Fredericton, NB E3B 1B6, Canada

## ABSTRACT

A challenge central to the Powder Metallurgy (PM) process is achieving consistent and uniform products. A crucial step in reaching this goal, is ensuring powder blending sufficiently homogenizes a mix. A challenge to powder and parts producers is creating a process that both achieves homogeneity and operates efficiently. C-Therm's ESP technology offers a window into the blender by measuring the thermal properties of powder during processing. Powder chemistry and density have a significant impact on the thermal properties and measuring thermal effusivity repeatedly throughout processing can indicate once a stable, homogenous blend has been achieved.  The authors seek to establish the usability and accuracy of measuring effusivity by monitoring multiple blend conditions and comparing results to standard industry methods such as thief sampling.

## INTRODUCTION

The dimensional tolerances and final mechanical properties of parts made from the PM process depend on various manufacturing steps such as compaction, sintering and post processing such as sizing or heat treatment. Mixing is defined as the physical combination of dissimilar components to produce a homogenous mixture [1]. Since the starting material for the process is a mixture of ingredients such as graphite, lubricants (typically in the form of waxes), steel powder and metal powders such as nickel and copper, the final part properties and tolerances that can be achieved depend on how well mixed the initial powder formulation is. The mixing of powder blends or "premixes" also depends on the size, density and flowability of the various additives as well as the physical characteristics of the majority component, most commonly an iron or pre-alloy powder.

Despite the large body of work that has been documented in the literature, powder mixing is very difficult to describe using a fundamental approach. The substantial number of variables in mixing as well as the considerable number of mixer designs makes a prediction of mixing performance extremely difficult [2]. Computational techniques are commonly used in the pharmaceutical industry to predict the homogeneity of blends after mixing, however these techniques are limited to the blender design and the specific ingredients being modeled and often are representative of ideal conditions, therefore these techniques are time consuming and costly [3]. Statistical analysis in combination with design of experiments is also a common technique in

the pharmaceutical industry, but becomes impractical with the cost and large volume of material used in the powder metallurgy industry [4]. Blends of 5 to 20 tons are typically preferred so that compaction press adjustments for different powder lots are minimized.

Industrially, the current approach in PM processing to determine if a mixture is homogeneous is to compare the final properties of the blend with the predicted or targeted value required. For example, if the base metal is an iron powder and 2 w/o copper powder was added to the blend, then a powder sample taken (by approved methods- MPIF Standard 01) from the completed mixture should have a chemical composition measured at 2 w/o copper. The problem with this technique is that it does not predict how long it will take the mixture to homogenize and therefore process optimization is usually by trial and error. It also does not take account for seasonal effects such as humidity and powder temperature. Thus, the optimized mixing time may change during the year. In some cases, over-blending can occur and de-mixing of the additives will take place, making it difficult to find the minimal acceptable blend time. Other mix properties such as green strength, apparent density and powder flow rate change with blend time and are difficult to predict without doing additional experiments.

On a small scale, a generally accepted method to optimize mixing performance is to take thief samples from the blender and various times and locations [5]. These samples can be analyzed offline for the characteristic under study such as chemical composition, apparent density, green strength or flow rate. Figure 1 shows an example using this method where graphite and lubricant were added to an iron powder and then blended in a double cone blender. The blender was stopped every minute and the total carbon content was measured at the bottom of the blender for three different fill volumes (500, 1000 and 1500 lbs.). It can be seen from Figure 1b, that after approximately 10 minutes the 500-lb. mixture achieved homogeneity regarding the carbon composition (graphite/lubricant). As can be seen from Figure 1 this technique is suitable for a small volume blender in which the total height is conducive to sampling.
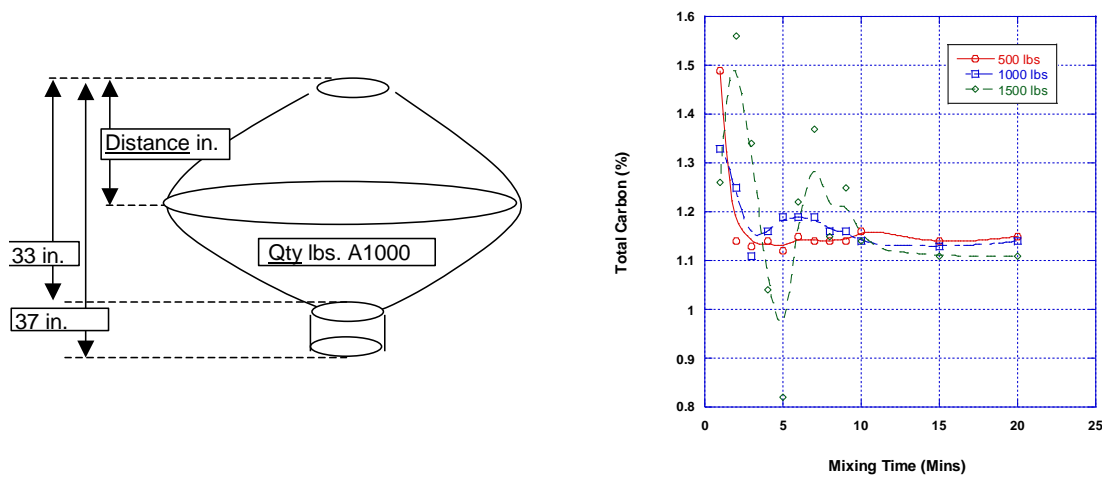


Figure 1: Schematic of double cone blender (left) and the total carbon measured by thief sampling (right) versus blend time for three different fill volumes (500,1000 and 1500 lbs.).

However, blenders in industrial use mix metal powders in quantities such as 10 to 20 tons and the height of the blender can be as high as 10 to 15 feet. This height level and the bulk density of the metal powders make any intrusive sampling very difficult.

Currently there are two methods being explored for nonintrusive- real-time methods for measuring blending parameters: Near Infrared Spectroscopy (NIR) and Thermal Effusivity. NIR is a type of vibrational spectroscopy that employs photon energy (hn) in the energy range of $2.65 \times 10^{-19}$ to $7.96 \times 10^{-20}$ J, which corresponds to the wavelength range of 750 to 2,500 nm (wavenumbers: 13,300 to 4,000 cm$^{-1}$) [6]. In NIR, the heat of mixing gives off near infrared radiation which is detected and statistically correlated with events occurring during the blending process. Although there are numerous publications citing the use of NIR for monitoring the blending process (primarily pharmaceutical) the existence of a commercial system for performing the analysis seems to be lacking. In addition, NIR works by detecting covalent bonds which do exist in some PM lubricants but do not occur in metals which usually comprise 90-95 w/o of the mixture.

Thermal effusivity is a material property that combines thermal conductivity, density and heat capacity. Therefore, it can differentiate between solids, liquids and powders in a system based on heat transfer. Each material has its own thermal effusivity as determined by Equation 1:

Equation 1:

$$Effusivity = \sqrt{k\rho c_p}$$

$Where:$
$k = thermal\ conductivity\ (W/m \cdot K)$
$\rho = density\ (kg/m^3)$
$c_p = heat\ capacity\ (J/kg \cdot K)$

The thermal effusivity of powder mixture is influenced by the physical properties of the powder composition and the properties of the inter-particle material (typically air) or additives such as lubricant, graphite or other finer metal powders such as nickel and copper. The thermal effusivity of air is very low with a value of 5 Ws$^{1/2}$m$^{-2}$K$^{-1}$ whereas the thermal effusivity of metal powders typically vary between 150 and 800 Ws$^{1/2}$m$^{-2}$K$^{-1}$. For the case of PM mixtures of iron powder, lubricant, graphite and other metal powders (i.e. copper and nickel) the effusivity will change as blending progresses eventually reaching a steady state at which time the blending can be stopped. This is the ideal case (Case 3 in Figure 2). In real situations, the ideal case may not be followed. Consider the case of powder blends with differently-sized particles. After an initial period of good blending, segregation of particles by size may occur. In such a case, this would be evident in changes in density, heat capacity or thermal conductivity of the blend in various locations, and would become apparent with increased variability between each sensor location (Case 2 in Figure 2). Following the same principle, nontypical events can also be detected by sudden changes in the effusivity or its standard deviation.
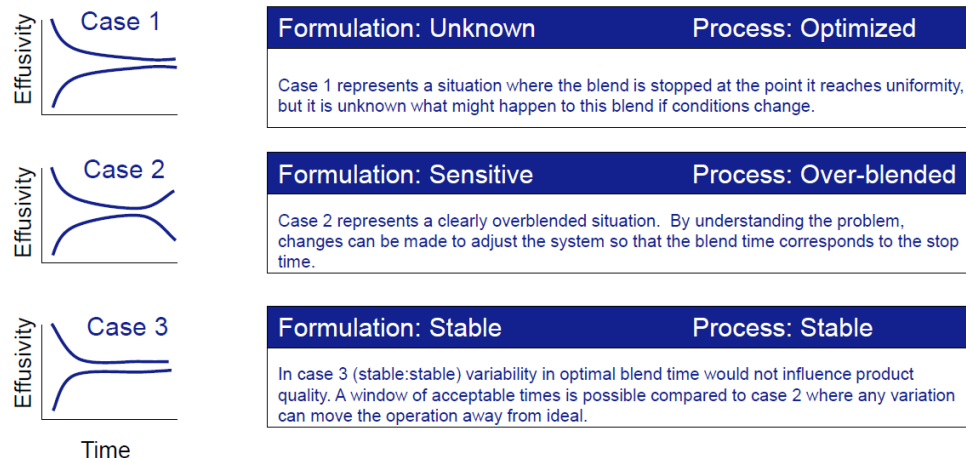
Figure 2: Three cases of effusivity measurements in powder blends.

## TESTING SYSTEM AND PROCEDURES

The current study utilizes the effusivity measurement as a commercial system exists from C-Therm.  This system had been previously used for pharmaceutical applications but had not been used with a dense material like iron powder. The primary objective of this study was to see if the sensor could be used in a powder metallurgy application. Thermal effusivity of samples was measured using a C-Therm ESP wireless unit. This instrument is an interfacial device that is in direct contact with the sample and is used to measure heat flow.

The principle of operation is the measurement of heat flow which occurs from a hot body to a cold one. Some materials, such as polymers, are not good conductors of heat so they are slow to absorb the heat. Other materials, such as metals, have good thermal conductivity, so the heat flow is more rapid into the material. A schematic of the system used in this study is shown in Figure 3 and a description of the operation is as follows:

(1) A known current is applied to the sensor's spiral heating element, providing a small amount of heat.

(2) A guard ring surrounds the primary sensor coil to support a one-dimensional heat transfer into the sample. The applied current results in a rise in temperature at the interface between the sensor and the sample, which induces a change in the voltage drop of the sensor element.

(3) The rate of increase in the sensor voltage is used to determine the thermal properties of the sample. The thermal conductivity is inversely proportional to the rate of increase in the temperature at the point of contact between the sensor and the sample.  The voltage is used as a proxy for temperature and will rise more steeply when lower thermal conductivity materials (e.g. foam) are tested. Conversely, the voltage slope will be flatter for higher thermal conductivity materials (e.g. metal). Thermal conductivity and effusivity are measured directly, providing a detailed overview of the heat transfer properties of the sample material. A more detailed description of the operational theory of the system has been provided elsewhere [8-9].
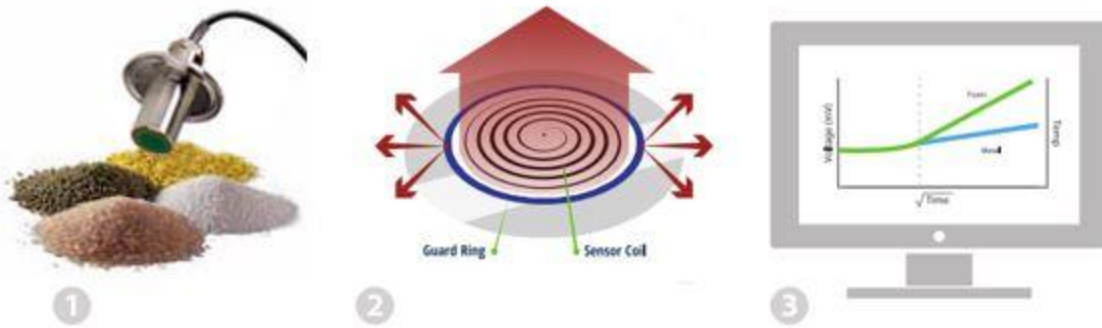
Figure 3: Schematic showing principle of operation of the C-Therm ESP™ Effusivity Sensor System.

During the rotation of the blender, the powder is not in a constant state of flow as one may believe. Instead, as the blender rotates the powder is held in place against the wall due to frictional forces until the gravitational force on the power is sufficient to overcome this, at which point it begins to slide until, due to the geometric design of the blender, the frictional forces stop the movement. There, it becomes held to the wall in the new location due to friction again. Thus, there is a region of "quasi-stationary" powder at the bottom of the blender at all time. The ESP system employs this region, where the sole mechanism of heat transfer is physical conduction through the powder medium, as opposed to regions where the powder is in motion relative to the wall of the blender, where the specific heat of the test material will also be a source of heat transfer.

Therefore, in the blender, the powder is essentially stationary over the sensor for a brief period (a function of the fill level and rotational speed in RPM), allowing a window of time in which the sample makes good contact with the sensors in the lid and can be analyzed with confidence.

To evaluate the use of effusivity to optimize blend parameters on metal powders and additives used in PM, a double cone blender with the capacity of 500 lbs. was outfitted with 4 sensors as shown in Figure 4.  The sensors need to be mounted at a spot at which they will have adequate physical contact with the powder to make the measurement in real-time. It was determined that the sensors would be mounted in the lid of the blender. When the blender is rotated 180 degrees the flat surface of the lid would provide excellent contact between the sensors and the powder. The wireless controller with a NiMH battery pack was mounted to a plate that was welded to the blender. The cables from the controller to the sensor were secured with cable ties. The signals from the controller were wirelessly transmitted to a laptop computer that could process the data in real-time.
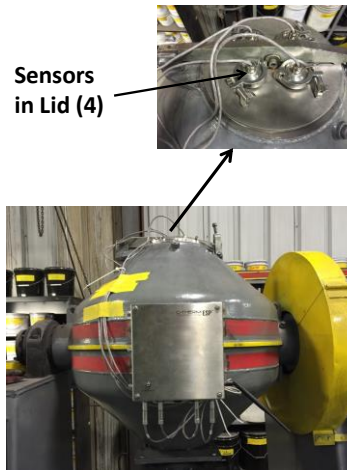
Figure 4: Photograph of the C-Therm ESP™ Effusivity Sensor System mounted to a double cone blender.

The blender used was capable of mixing 500-1500 pounds of powder and had a variable speed drive which could rotate the blender between 7 and 22 revolutions per minute (RPM). Unless otherwise noted, 500 lbs. was the standard weight of iron powder that was used and the powder had approximate apparent density of 3.00 g/cm$^3$. Experiments had been performed previously in this blender with thief sampling this mixer (@12 RPM) and it was concluded that homogeneity of a 500-lb. premix would occur in 7-10 minutes (Figure 1b). The typical procedure was to load the iron first and then other ingredients were placed on top of the iron powder before rotating the blender and taking measurements.

Two sets of experiments were completed.  The first was a set of static measurements in which the blender was stopped at time intervals and thief samples were taken from the material in the blender to try and establish a relationship with powder properties such as chemical composition, apparent density and homogeneity. These static measurements were then used as baseline information for the real-time dynamic testing (the second set of experiments) in which the blender was run continuously.

## RESULTS AND DISCUSSION

### Static Experiments

The first experiment was to determine if the diffusivity of various components used in PM had enough variation in thermal conductivity (and hence effusivity) to be detected by the sensors. A standard FC-0208 (Ancorsteel A1000B + 2.0 w/o Copper + 0.80 w/o Graphite + 0.75% Acrawax C) was mixed by adding the individual components one at a time into the iron powder. Effusivity measurements were conducted every minute for the first ten minutes, then every 5 minutes until 20 minutes of blending time had been reached. Ten minutes of blending had been previously determined to be sufficient time for complete mixing (Figure 1b). Each component of the mix was then added, one at a time, and blended for 20 minutes to see the effect on the average effusivity. Figure 5 shows the plot of effusivity versus time for this blend.
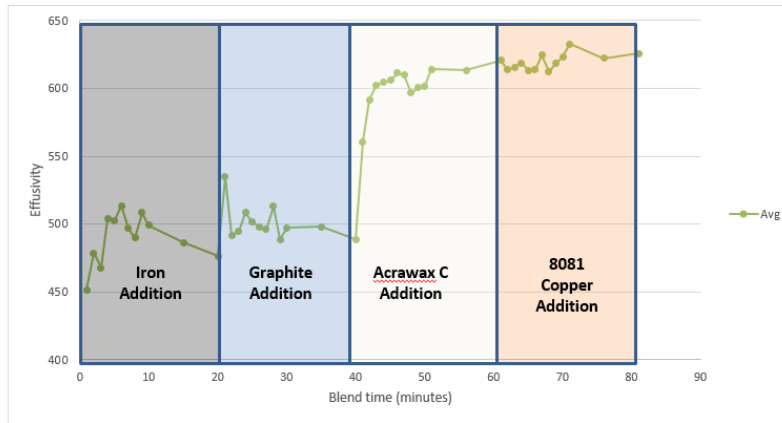
Figure 5: Effusivity measurements of individual additions made to an FC-0208 premix.

Effusivity is a function of the thermal conductivity of the material but can also be related to variables such as density (true and apparent), particle size, particle shape, particle size distribution, moisture, etc.  Figure 5 shows that after the iron had been blended for 20 minutes when the graphite addition was made there was an increase in the effusivity. After the twenty minutes of blending with the iron powder and graphite, there was a much larger increase in the effusivity when the Acrawax C was added. When the copper was added to these three components there was slight change in effusivity. Since the Acrawax C is much finer than the iron powder (mean particle size of 105 microns versus 6 microns) and has much lower thermal conductivity it is much easier to detect, while the graphite which is much closer to the iron powder in respect to these properties shows a smaller change. The copper powder which is similar in particle size to the iron and closer in thermal conductivity is more difficult to detect.

Since the change in effusivity was significant, as the individual components in the FC-0208 mix were easily detected, a second static experiment was performed in which the iron powder was loaded into the blender and the all the various ingredients were placed on top of the mix simultaneously. Effusivity measurements were conducted every minute for the first ten minutes, then every 5 minutes until 20 minutes of blending time had been reached. In addition to the effusivity measurements, thief samples were taken from the center of the blender.  Measurements of chemical composition and apparent density were made and compared to the effusivity values.

Figure 6 shows a plot of the average effusivity (dark blue- lower plot) versus the apparent density measurements (red line- upper plot) taken from the thief samples. On this graph, the apparent density measurements taken from thief samples at different time intervals correlates very well with the average effusivity calculated from the four sensors. Recall that effusivity is proportional to the square root of the density (Equation 1).  Both the effusivity and measured apparent density stabilize after 10 minutes of blending. This is consistent with previous experiments evaluated with thief sampling.
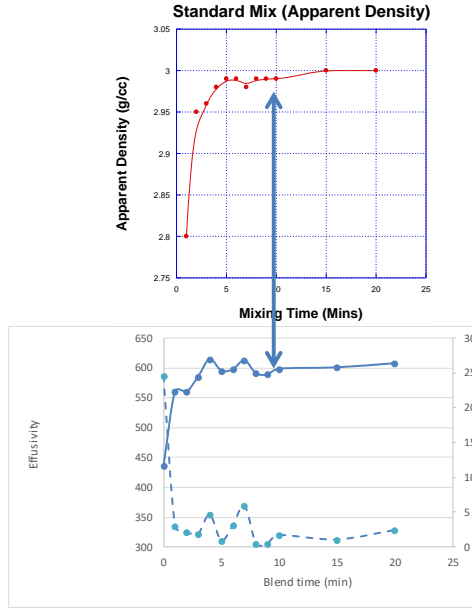
Figure 6: Effusivity measurements and apparent density measurements of a FC-0208 premix.

While the average effusivity has a relationship with the density of the mix the relative standard deviation (RSD) of effusivity can be correlated to changes in mix composition. There are two mechanisms for mass flow in blending. The first mechanism is due to convective transport, which is the transport of large volumes or blocks of powder due to the design of the blender [10]. The second mechanism is diffusive blending, which is a result of the individual particle movement with respect to these volumes or blocks of powder. Both lead to compositional variations within the mix that result in changes in the RSD.  Chemical composition was measured as a function of blend time for the same samples plotted in Figure 6.  Copper content and total carbon (which includes the carbon from both the graphite and Acrawax C) are plotted in Figure 7 and compared to the RSD.
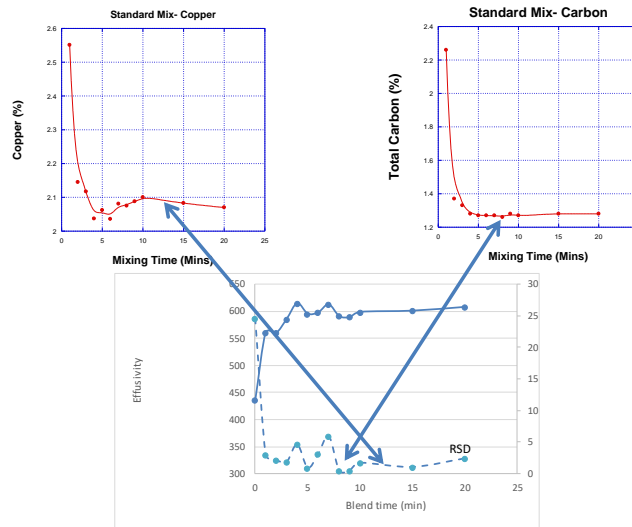


Figure 7: RSD of Effusivity versus chemical composition (copper and total carbon).

Both the copper and total carbon measurements (taken from thief samples) indicate that the blend is completely mixed after about 10 minutes.  This is supported by the measurement of the RSD in effusivity, which also starts to level out after 10 minutes. Comparable results were found when blending other additives such as, MnS, nickel and fine copper indicating that the change in RSD was a good indicator of how well mixed the additives were in the blend.

There are several additives in PM that have a known effect on apparent density with small addition levels. Flow enhancers and Zinc Stearate Lubricant (ZnSt) each can change the apparent density of a premix with low level of additions ( $< 0.10$ w/o).  Flow enhancers generally lower apparent density while ZnSt generally increases apparent density.  Figure 8 shows the effect of these two additions (0.05 w/o flow enhancer and 0.10 w/o ZnSt) on the effusivity when added at different times during the same blend. The time at which the apparent density drops matches the adddition time of the flow enhancer while the time at which the apparent density then increases matches the time when the ZnSt was added. Both these changes are captured by the change in effusivity.
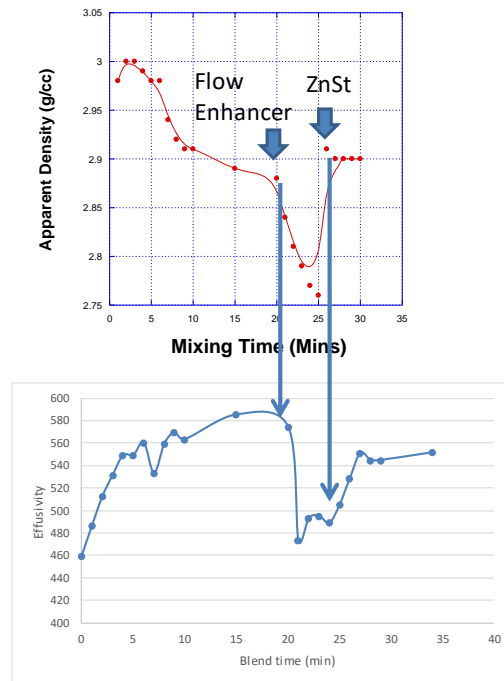


Figure 8: Effusivity (bottom) versus additions of Flow Enhancer and ZnSt (top).

The results of the static testing showed that the measurement of Effusivity and RSD corresponded to physical changes within the mix and these changes correlate with the changes in the measurement.

**Dynamic Experiments**

The static experiments allowed for a high level of confidence that phenomena occurring within the blender could be rationalized based on the effusivity measurements. However, the end use for

this system would be as a real-time measurement of the quality of the mix. The current trend in manufacturing is toward automation and data exchange to create a smart factory (Industry 4.0). Currently in the PM industry there is no real-time data collected on variables in mixing. Offline chemical analysis and physical testing is not an option in trying to improve mix consistency and precision, particularly without impacting productivity. Therefore, dynamic tests were performed to determine the suitability of this technique for in-situ measurement.

In all mixing processes, there is a competition between mixing and de-mixing or segregation. Most of the components of a PM mix vary significantly in size, apparent density, surface properties, friction coefficients, hardness and density. All these differences can lead to segregation or a de-stable mix. In general, segregation is hard to predict as it is a function of the many parameters just described. The final state of a blend is an equilibrium between complete mixing and segregation (Figure 2). Segregation can, therefore, make the prediction of the end-point of the blend (the final blend time) impossible or even prohibit a homogenous blend.

To test the theory of de-mixing the standard FC-0208 mix was loaded into the blender as previously described but it was continuously mixed for 20 minutes without any stoppage for sampling. It had been determined by previous measurements that optimum blend time for this mix was approximately 10 minutes. As can be seen from Figure 10 the average effusivity and the RSD both decrease to a minimum value and a stable blend is indicated at approximately 10 minutes. Note in this test only three of the four sensors were operational.
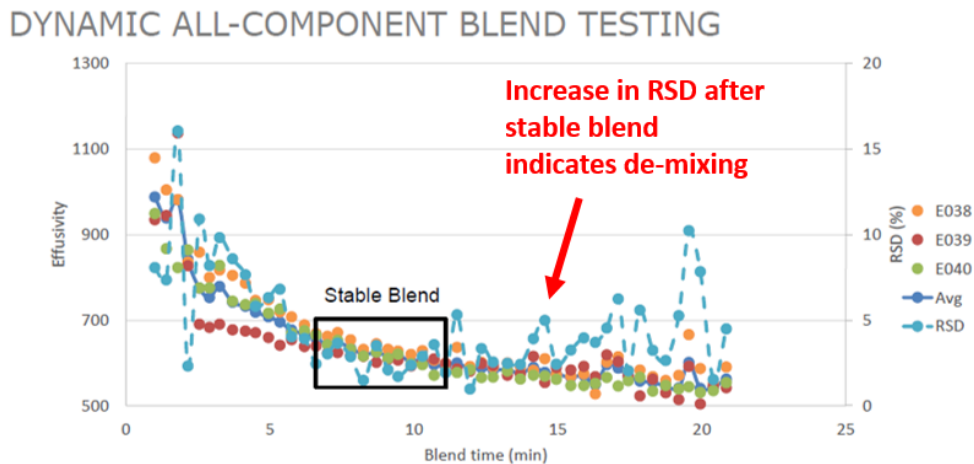


Figure 9: Extended blend time of standard FC-0208 mix.

Upon continued blending, at about 14 minutes the RSD increases indicating the blend in no longer homogeneous. It is not clear the actual mechanism for the de-blending, but it is known with this blender that the increased blend time leads to higher powder temperatures which can make the lubricant "sticky" and effect the flow of the powder.

The real-time indication of complete mixing is extremely important in the productivity of making mixes as well as the overall quality of the blend. Currently many blend facilities are at the maximum capacity for blending so any indication that they can predict and lower blend times

without impacting product quality will be welcome. In addition, as customers have new mix requirements and new additives are developed for mix compositions, normally a standard blend time is assumed. This approach may not achieve the best mix quality. Figure 10 shows the results of a trial with a new additive that was a very fine powder that had poor flow-ability. In previous mixes using this additive, there was obvious segregation within the mix. It was proposed that if a flow enhancer were added to this additive prior to blending the mix quality would be improved. Two mixes were then made (FC-0208) and blended continuously in the blender. One mix had no flow enhancer added to the additive while the other did. The results are shown in Figure 10. The top graph shows the RSD for the mix without the flow enhancer. In this case the blend did not become stable till about 12 minutes as indicated by an RSD < 5. In the mix where the flow enhancer was added the mix was stable in about 8 mins (RSD < 5) and the absolute value was lower than the mix without enhancer.
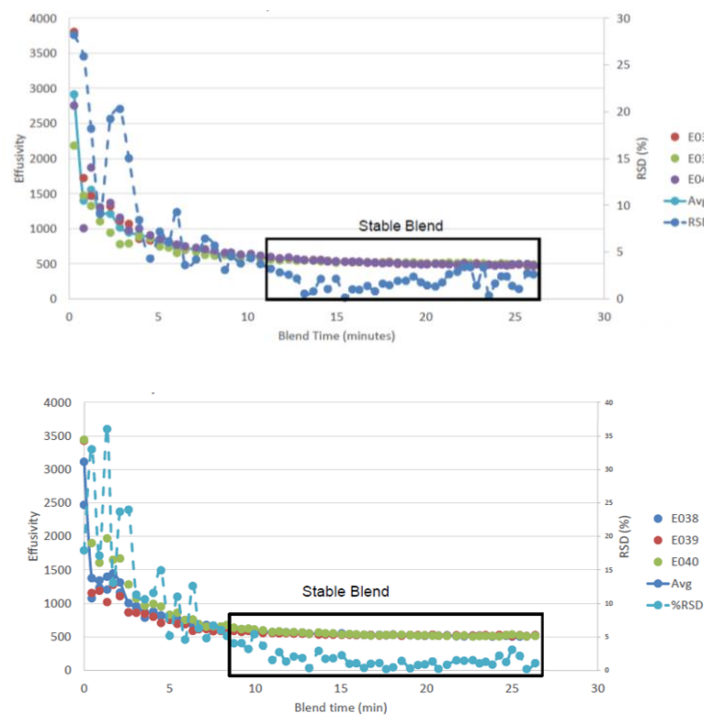


Figure 10: Additive Powder with (bottom) and without (top) flow enhancer.

Lubricants are a problem with PM premixes. In general lubricants with higher melting points are easier to mix then those with lower melting points or those that can soften from the heat of mixing. But in general, the lower melting point lubricants tend to give higher green density and better ejection characteristics. The advantage in compaction makes the use of these more difficult to blend lubricants very attractive to the user of the premixes. Currently there is no quantitative method for determining the ability to blend a specific lubricant. Many times, initial research of new lubricants is done with small blenders that do not accurately represent the amount of heat and mixing that will occur in the production blender. This can lead to problems when the lubricant is transferred to larger production sized blenders.

To investigate this, two lubricants were investigated using the effusivity measurement. The first is Acrawax C a bi-ethylene stearamide which is considered a robust lubricant by the PM industry. The second lubricant (Lubricant X) was one containing lower melting point constituents that aid in compaction. The results are shown in Figure 11.
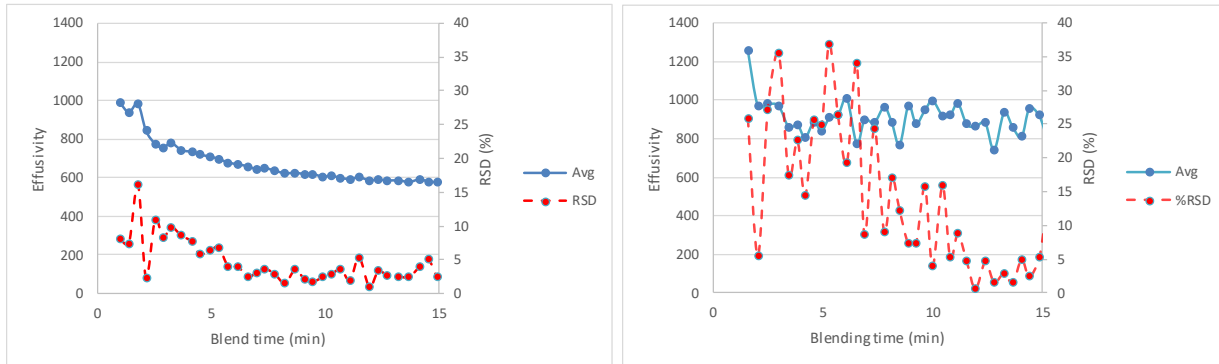


Figure 11: Comparison of Acrawax C (left) and low melting Lubricant X (right).

The effusivity and RSD using Acrawax C in an FC-0208 stabilizes within 10 minutes of blending, however when using Lubricant X, the effusivity and RSD is not as stable within this time. This may indicate a longer blend time is needed for this lubricant. The test data was only collected for a total of 15 minutes and it appears deblending was starting to occur.

Finally, it was previously discussed the considerable number of variables that can affect the premix quality. Many of these such as temperature, humidity, raw material changes, and variability of incoming materials. If the measurement of effusivity could be performed and stored according to the individual mix being made there may be a method to link the performance in the blender to the performance in the compaction press. As an example of this, during one trail documenting the performance of a new lubricant, approximately 7 minutes into a blend of FC-0208 a drop in effusivity occurred when it should have been increasing. Upon opening the blender lid, it was observed that some of the lubricant had been stuck to the walls of the blender and a "cake" of lubricant was noticed on top of the powder in the blender (Figure 12). It appears large "clumps" of lubricant had dropped into the blender lowering the effusivity. After continuing the blending, the effusivity curve trended as expected. It appears the system could detect this "upset" or "abnormal" condition during blending and this will be useful as a process monitoring and quality control of blends. Information such as this can be stored on a mix basis and used as a quality record for each lot blended. Additionally, process optimization can occur by comparing final mix properties (such as apparent density, Hall flow, dimensional change, etc.) with individual lot data, especially taking into count the time of the year (temperature and humidity effects).
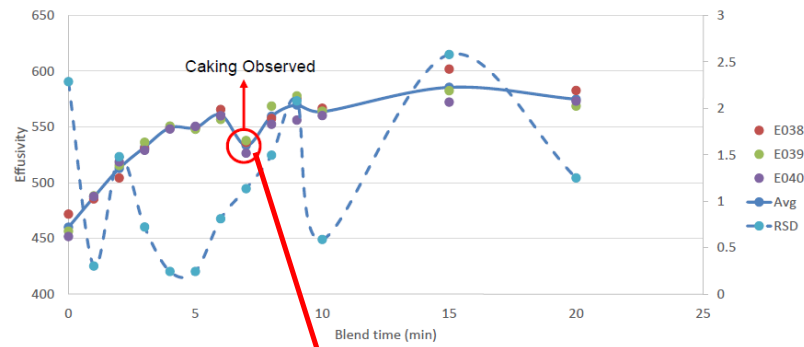
Figure 12: Upset condition measured during dynamic testing showing effusivity decrease (top) due to "cake" of lubricant.

**CONCLUSIONS**

The use of the effusivity sensors allowed for real time measurements of phenomena that occur while blending. The test results in this study showed direct correlations with phenomena that occurred in mixing of PM blends. These items included:

- Changes in apparent density.

- De-mixing or over blending.

- Optimization of mixing times.

- Improved mixing of additives through flow aids.

- Additive segregation.

- Detection of upset conditions during blending.

Additional trials examining other process variables such as powder temperature, blender speed and blender fill volume were also conducted (not reported on here) with predictable results. The sensors show excellent promise to be used as both a tool to optimize blend performance and to monitor the quality of production lots as a quality control tool. Some questions remain as to the robustness of the sensors regarding large blenders (~10-20 tons) where the force on the sensors will be large due to the static head of metal powder (which can be 3 meters high). This issue will be the subject of future work.

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