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In-Process Particle Size Measurements for Diagnosis, Optimization, and Control

1.0 Introduction

In-process measurement of particle size and concentration distributions provides continuous analysis, quality control, and optimization of product yield. As process production rates continue to improve, the delay between laboratory analysis and process correction of the product stream becomes more significant and costly in many commercial applications. Elimination of sample handling and operator manipulation is now possible for most dry pneumatic and gravity flows using optical methods which are properly interfaced with the process stream. Malvern/Insitec has developed such an instrument based on ensemble laser-diffraction that has been successfully applied to a wide range of research and industrial process applications.

As part of the adaptation of instrumentation to the specific needs of different process applications, Malvern/Insitec has developed an eductor bypass interface which can be tailored to the requirements of different materials and plant configurations. To address the variety of operating concentrations that occur in practice, specific sampling interfaces have been developed that allow measurements for mass flow rates ranging from 1-100,000 kg/hr. In conjunction with these broad operating conditions, a proprietary de-convolution algorithm has also been invented that corrects multiple scattering effects at high particle concentrations which can give light transmissions down to 5%. Distribution update rates are computed and displayed in less than 1 second. This methodology is briefly described along with measurements in a cement plant. Quantitative economic benefits of real time measurement and process control are described and summarized.

2.0 Overview of EPCS Instrumentation

2.1 Principle of Operation

Figure 1 is a photograph of the optical head and real-time sampling interface of the EPCS system. Particles pass through a gas purged flow cell, which supports a fixed alignment laser transmitter and solid state detector based on the classical ensemble laser diffraction technique. As particles pass through the laser beam, light scattered in the forward direction is collected by the receiver lens and focused onto a log-scaled annular ring detector. The detector is scanned at high speed, recorded, digitized for continuous real-time analysis. This technique has been described in further detail in Ref. 1.

2.2 EPCS Process Interface for measurements

For the proprietary cement application described here, a bypass/dilution system (Figure 1) was used to extract a portion of the flow, pass it through the instrument, and then back into the primary flow line. This system incorporates a near iso-kinetic tap that is driven by a venturi eductor. A section of the primary gravity flow stream (>100t/hr) is extracted, flows through the EPCS and is re-injected into the main process line.

2.3 Measurements in a Cement Plant

The EPCS measures the specific surface area (m^2/ m^3) in conjunction with the size distribution. Figure 2 shows a 12 day comparison between the EPCS SSA (Specific Surface Area) and the traditional Blaine number measured by pressure drop. The standard error bar for a Blaine measurement has been estimated (by ASTM) and shows that the EPCS gives statistically equivalent measurements. Note the slow equilibration to steady-state operation of the process after a 1 day shutdown.

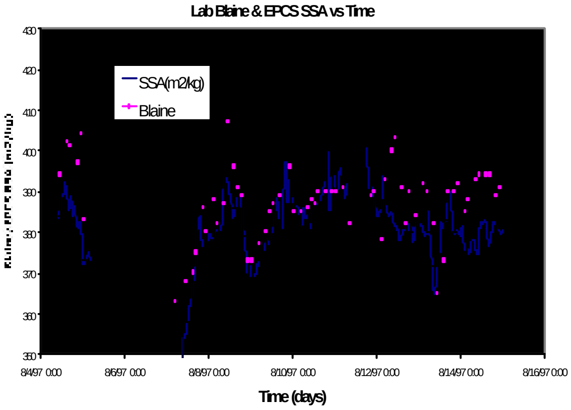
3.0 Data Analysis and Return on Investment (ROI) for a Cement Plant Application

We have recently worked closely with one of our EPCS customers to better quantify the capabilities of the EPCS and how it can improve the process control, QA/QC and production efficiency in the cement industry. The main goals of this analysis have been to:

1. Show size correlation with traditional Blaine measurements
2. Optimize process control
3. Correlate Strength with Size and Chemistry parameters
4. Quantify return on investment (ROI) for the EPCS

Figure 1. EPCS and interface in Cement Plant

Figure 2. Comparison of EPCS SSA with traditional Blaine measurement.



3.1 Size correlation

Soon after the initial EPCS installation, 30 Type I cement samples were analyzed off-line with EPCS. Initial correlation between EPCS and measured Blaine number was poor. The primary limitation was that the span of the data population was only 2 to 3 times the expected error of the measurement. To mitigate this problem three data sets have been combined using 30 samples from 2 different plants of the same company. The combined data set (Figure 3) has a span of 700 cm^2/g ($70m^2/kg$) and, as expected, the correlation coefficient R^2 values improved from a value of 0.43 to 0.79. The standard error is 7.15 m^2/kg (71 cm^2/g) or 1.8% of the measurement. This is slightly more than the ASTM estimated error of 1.5% for the air permeability test using the Blaine apparatus[2]. The error bars span +/- $7.15m^2/kg$.

3.2 Process control

The EPCS unit has been used for feedback control for the separator at this plant since the middle of June,1999. The separator speed has been controlled using the %<30 micron parameter, which provides the best correlation with the traditional plant control parameter of [% passing 325 mesh] (wet sieve). Despite the relatively small data set available at this time, the standard deviation (RMS) on fineness has decreased by 34% from 1.5% to 1% of the indicated measurement.

Figure 4 shows a plot of process throughput vs. product fineness. We observe that product throughput is inversely proportional to the fineness, i.e. finer product requires increased milling time, which results in lower product throughput, a result consistent with previous measurements[2]. From this graph the fitted equation shows that the throughput decreases 2 tons/hr for every $100cm^2/g$ increase in surface area (approximately 2% reduction in productivity for a 2.5% surface area increase). The most economic goal is to mill sufficiently to achieve required cement strengths but not to over-grind, because this reduces total plant production and increases unit milling costs.

3.3 Strength Prediction

It is well known that early strength is dependent on both chemistry and fineness of the cement[3] .With that in mind a multi variable regression analysis was performed, correlating particle size variables and chemical analysis to 1day strengths (see Figure 5). As the general cement literature[3] indicates, it is possible to predict 1-day strength using the measured Blaine number and a measure of clinker chemistry, (namely $SiO_{2)}$. SiO_2 is the main component in two of the main cement compounds, C_3S and C_2S. Although the standard error of this correlation is not great ($R^2 = 0.59$), this is a significant improvement in correlation using particle fineness alone ($R^2 = 0.38$). Furthermore, the laboratory analysis is measuring secondary oxides SiO_2, Al_2O_3, Fe_2O_3, CaO, SO_3 and not the "real" strength producing components: C3S, C2S, C3A and C4AF. The potential composition of the cement compounds in the product (and clinker) is estimated using the Bogue formulas[3], presuming specific heating and cooling conditions in the kiln which might or might not be present at all times during production. Our model is not complete, but gives a simplified tool that describes the interaction between fineness, a chemical indicator (SiO2) and the 1-day strength. Despite its simplicity and current limitations, even this crude correlation will enable the plant to improve prediction of the early product strength because it includes a figure of merit for both the effects of chemistry and particle fineness. Changes in product chemistry can thus be compensated by product fineness, allowing optimization of the milling process.

To verify the robustness of the correlation model, it was applied to a subset of the data for which we have EPCS measurements. Figure 6 shows the result of applying the model on a data set consisting of 44 days of production from two different periods (Sep-Oct '98 and June '99). The correlation coefficient $R^2 = 0.69$, and the standard error is 107 psi, which is on the order of the uncertainty (75 psi) of the strength measurement itself. While the correlation model is based entirely on laboratory data, we find that we also obtain satisfactory results with online EPCS data. As more EPCS data becomes available the correlation model will be further confirmed and the confidence level will improve.

3.4 Economic perspective

How can this new online particle size information be used to improve production and impact the bottom line? Real time feed back control improves the consistency of the final product. Having better control over the milling process makes it possible to mill product closer to specifications thereby increasing throughput and lowering manufacturing cost. For example: our cement customer produced 14 days of type I cement. The average 1-day strength was 2280psi, 480 psi above the ASTM specification. Using the model derived earlier, (figure 5), we want to define a target Blaine # that will yield the specification 1-day strength (plus a safety margin). A conservative margin would be to use three times the standard error of the prediction model. Statistically, this will result in less than 1 % rejects. The current rate of recycle is approximately 5%, therefore this margin would reduce the recycle rate by a factor of 5. From Figure 3, the 3-sigma target for 1-day strength = 1800+3*107=2121 psi (159 psi lower than the month average). It is important to note that any improvement in the standard error of the correlation model for strength will allow even further improvement.

From the 1 day strength prediction model we can define a non-dimensional correlation relationship:

$S/S_0 = A*B/B_0 + D*C/C_0 + F$, where Eqn. (1)

S = 1 day strength (psi) and the subscript refers to a reference value of 2000 psi.
B = Blaine Number (cm^2/g) and the subscript refers to a reference value of 4000 cm^2/g.
C = Chemistry parameter (SiO2) and the subscript refers to a reference value of 21%.

A = Fineness coefficient = 1.18
D = Chemistry coefficient = -2.92
F = Constant = 2.88

In addition, we have determined a correlation (Figure 4) that gives a relation between product throughput and one-day strength:

$P/P_0 = -0.83 B/B_0 + 1.83$, where Eqn. (2)

P = Production rate in Ton/hr and the subscript refers to a reference value of 100 Tons/hr.

Using Equations 1 and 2 we can eliminate the Blaine number and show that $dP^*/dS^* = -0.7$. Thus a reduction in average strength of 159 psi or $dS^* = -8\%$ gives a 5.6% (5.4 ton/hr) increased production. This equals 1800 tons lost production for the 14 production days in June of 1999 or at a sales price of $70 /ton, a lost revenue value of $126,000.00 for the period.

The above example assumes that the production is at capacity and that the market can absorb the increased production. If production were below capacity, the savings would be in reduced milling costs, still substantial savings. From Peray[4], the savings, in theoretical milling is on the order of 3 % per 100cm^2/g decrease in fineness. Our current *measured* correlation is about 2% per 100 cm^2/g decrease in fineness, in reasonable agreement with the theory.

4.0 Conclusions

1. We have developed a quantitative correlation between the EPCS Blaine # and the traditional air permeability Blaine measurement.
2. An early strength (1day strength) prediction model has been established using a combination of fineness and chemical composition (Blaine # and SiO_2). The standard error of the model is on the order of the expected error of the strength measurement. This correlation model shows that **process throughput can be increased by 5%**, while at the same time meeting strength requirements.
1. The EPCS instrument package has proven to be reliable. During the first 8 months of operation no maintaince has been required. An inspection in mid-June revealed no changes in performance since installation.

5.0 References

1. Harvill, T.L., Hoog, J.H., Holve, D.J., "In-Process Particle Size Distribution Measurements and Control", Part. Part. Syst. Charact. 12 (1995) 309-313.
2. Malcolmson, A.P., Holve, D.J. "In-line particle size measurements for cement and other abrasive process environments", IEEE/PCA 40th Cement Industry Technical Conference, May 1998.
3. Lea's Chemistry of cement and concrete, John Wiley & Sons Inc
4. Kurt E. Peray, "Cement manufacturer's handbook, Chemical Publishing Co. N.Y, N.Y.

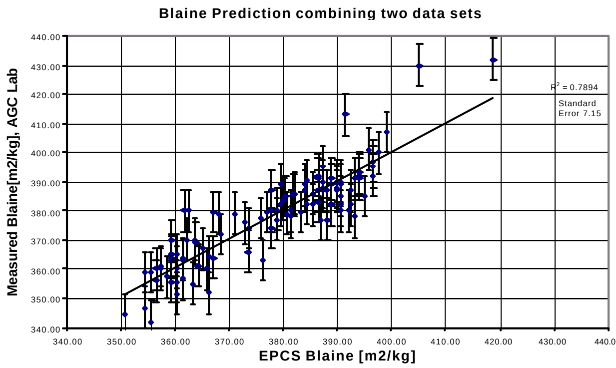


Figure 3. Comparison of Blaine numbers between EPCS and traditional Blaine measurements.

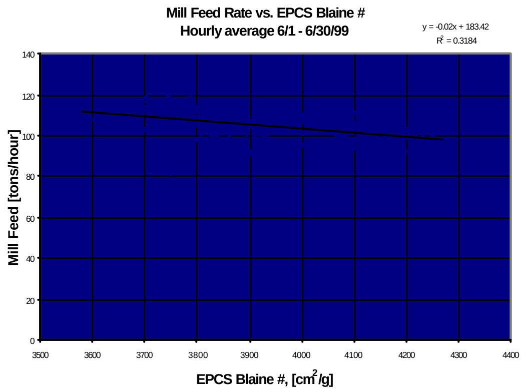


Figure 4. Process throughput vs. product fineness.

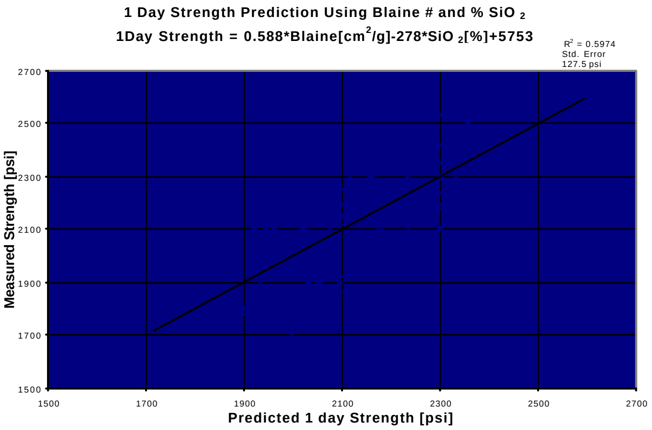


Figure 5. Correlation of measured early strength with predicted strength (Blaine/chemistry factors) using traditional Blaine measurements.

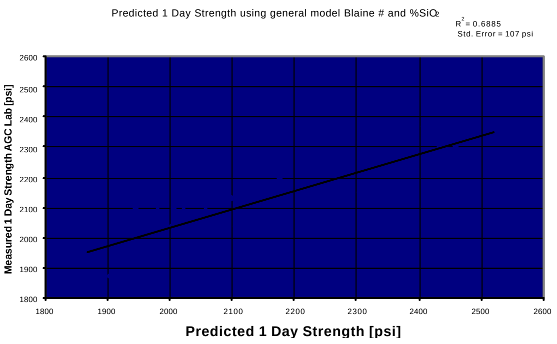


Figure 6. Correlation of measured early strength with predicted strength (Blaine/chemistry factors) using EPCS Blaine measurements.