Development of an Automated Foam Index Test

Tapiwa Z. Gurupira[1], Melissa P. Ochsenbein[1], John M. Stencel[1] and Ferdy Martinus[2]

[1]Tribo Flow Separations, 1525 Bull Lea Road, Suite 10, Lexington, KY 40511; [2]Department of Mechanical Engineering, University of Kentucky, Lexington, KY 40506

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INTRODUCTION

In 2003, nearly 112 million tons of cement and 11 million tons of coal combustion ash (CCA) were used for concrete construction in the US. CCA's, when used to replace cement, impart superior qualities to concrete, including increased hardness and better resistance to alkali attack. In the US, approximately 10% replacement of cement with CCA's occurs. At 30% replacement, as accepted in many construction specifications, up to $1.4 billion per year could be saved in US construction costs. Moreover, CCA's provide a way to decrease the CO_2 burden associated with cement usage because kiln operations are more energy intensive than most other manufacturing processes.

However, CCA's react differently than does cement to commonly-used and specification-required chemical admixtures like air entraining agents (AEA). Although a foam index (FI) test has been devised to measure the effect of CCA's on AEA, it is based on visual observations and non-standardized methods prone to misinterpretation and/or imprecision. Often, a person performing the FI test has other duties and responsibilities that can interfere with the attentiveness required for meaningful and repeatable measurements. The FI values obtained by visual measurements are also not transferable from one CCA usage site to another.

As suggested by the wording 'foam index', what a person tries to observe during FI measurements is surface foam instability on CCA/water/AEA mixtures after agitation. Foam instability is caused by bursting or breaking bubbles within the foam. During bubble breakage, discrete and characteristic acoustic emission (AE) emanates from foams. By quantifying the level of the AE from eight different CCA's during FI testing, Tribo Flow Separations has devised what it calls an Automated Foam Index Test (*A*FIT™) instrument. The FI results from *A*FIT™ equipment are compared to visual FI results herein.

PROCEDURES

*A*FIT™ aims to replace current, visual FI testing, a typical procedure for which is represented by the following[1]:

1. use standard, diluted AEA with distilled water (1AEA:20 H_2O);
2. place 20g of cement or pozzolan in a 4 fluid ounce bottle, add 50 ml of distilled water to bottle, cap and shake for 1 minute to thoroughly wet the cement;

3. add the diluted AEA in 0.2 ml aliquots, with vigorous shaking for 15 seconds after each aliquot. Then lay the bottle on its side and observe foam stability; and
4. the amount of dilute AEA, in ml, needed to produce a stable foam, i.e. one in which no bubbles can be seen breaking for a period of 15 seconds, is the foam index (FI).

This procedure was employed during initial FI tests with a 'standard' AEA[2,3] - $C_{12}H_{25}C_6H_4SO_4Na$ (dodecylbenzenesulfonic acid). However, to ensure compatibility with industrial practices, a review of current procedures of six CCA companies was begun. Four of these graciously provided explanations of their FI procedures; only one used shaking while the others preferred high velocity stirring for mixing of the CCA/water/AEA mixtures.

An *A*FIT™ cell was constructed to measure FI values of eight CCA's. A schematic of the glass cell and its associated hardware is given in Figure 1. Although the tests employed a contact sensor, microphone and hydrophone to provide comparisons between their AE sensitivities and repeatabilities, this paper focuses on the use of the contact and microphone sensors. The AE data were processed using PC-based software and hardware.

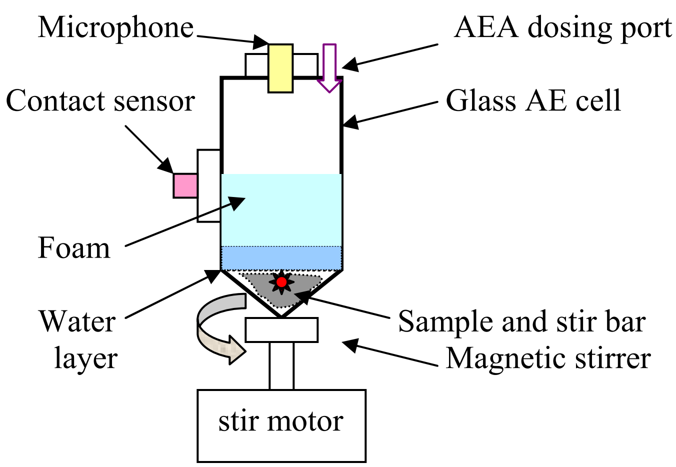

Figure 1. Components of an *A*FIT™ test cell used to measure FI values of eight CCA's.

At the beginning, careful attention was paid to the behavior of the foams and the solid-liquid mixtures during and after stirring and upon stepwise addition of AEA. They provided a repeatable procedure that was followed:

1) use standard, diluted AEA with distilled water (1AEA:20 H_2O);
2) place 40g of cement or CCA into the *A*FIT™ test cell, add 100 ml of distilled water, cap and stir for 1 minute to thoroughly mix contents;
3) after turning off stirrer, acquire background AE data to establish acoustic thresholds;
4) add the diluted AEA in 0.2 ml aliquots, stir for 15 seconds;
5) turn off stirrer, and then wait for 5-10 seconds;
6) acquire AE data for 15-30 seconds, while also observing foam stability;
7) the amount of dilute AEA needed to produce a stable foam was the FI of the sample;
8) compare visual and AE data.

Although industrial practice commonly reports FI in units of 'drops of AEA', this approach neglects differences in the drop or aliquot size and the type of instrument used to add AEA. Hence, a digital micropipette was used for dosing precisely 200 µl of diluted AEA per drop. In the following, FI values are reported as 'drops of AEA' and as 'liters AEA per metric ton' of CCA.

RESULTS AND DISCUSSION

The characteristics of AE waveforms that were assessed included: number of hits, number of counts, signal amplitude, signal rise time, and signal duration. While no correlations were found between FI values and the AE amplitude, rise time or duration, the number of hits (# Hits) and its change during AEA dosing precisely replicated visual FI observations.

This replication, displayed in Figure 2 for two CCA samples, included very high surface bubble breakage and #Hits at low AEA dosage, followed by a rapid decline in both visual and #Hits data. The overall activity then entered a low intensity plateau at intermediate AEA dosages where the number of bubbles bursting and the #Hits was small but steady. After this plateau, the visual number of bubbles bursting → 0 and the AE monitored #Hits → 0. The trends shown in Figure 2 were consistent and reproducible for all samples.

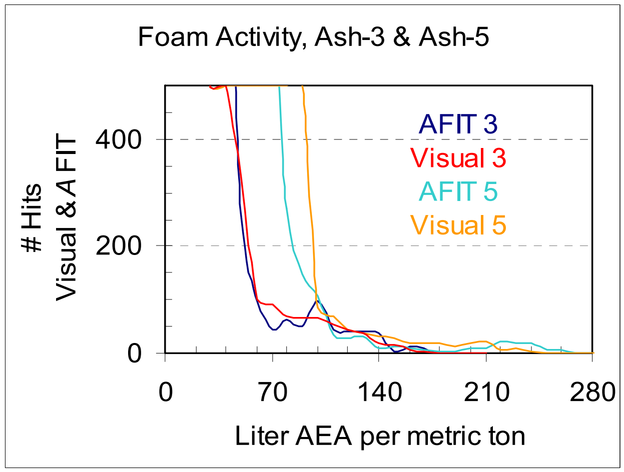

Figure 2. FI values from visual and AE #Hits.

Comparisons were made between AE data acquired using contact and microphone sensors. For a sample labeled ASH-3, the AE data in Figure 3 show somewhat different shape at AEA dosages ≤ 50 l/Mton but AE signal maxima at dosages near 55, 70, 100 and 130 l/Mton were identical for both sensors. Maxima and minima in AE intensities are associated with the dynamics of bubble stability. Furthermore, the # Hits → 0 at an AEA dosage of 195 l/Mton for both sensors, i.e. the FI value can be measured reliably using either contact and/or microphone sensors.

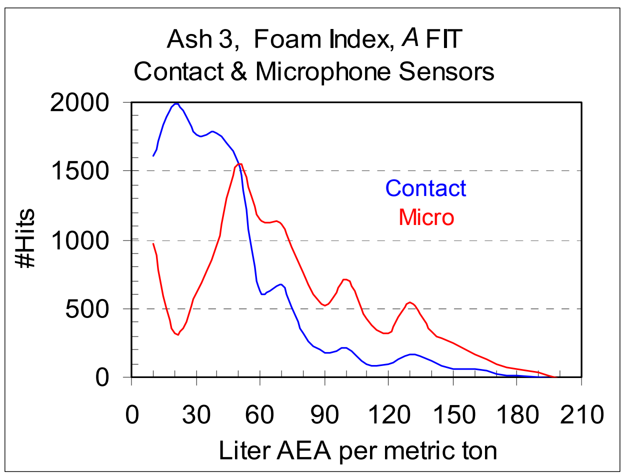

Figure 3. Characteristics of AE signals from contact and microphone sensors with increasing AEA dosage.

The FI values of eight CCA's, obtained using *A*FIT™ and visual observations, are presented in Table 1. The cumulative ±2σ deviations of FI values using *A*FIT™ were only 5% whereas the cumulative ±2σ deviations using visual measurements were two-three times greater. Typically, the deviation of visual measurements in industrial settings is significantly worse. Overall, the data presented in Table 1 show that an *A*FIT™ instrument should provide highly precise FI information to industry.

Table 1. FI values (in drops AEA) and their precision for eight CCA's using *A*FIT™ and visual observations.

Sample/ Cum. Dev	Contact Sensor		Visual
	15s, #Hits Ave ± 2σ	30s, #Hits Ave ± 2σ	15s, # Ave ± 2σ
Ash-1	10.4 ± 1.0	10.3 ± 1.2	12.0 ± 3.6
Ash-2	16.6 ± 0.8	16.7 ± 0.2	16.2 ± 3.2
Ash-3	18.4 ± 1.2	18.8 ± 1.4	19.5 ± 1.8
Ash-5	26.9 ± 0.2	29.3 ± 1.2	29.2 ± 4.1
B-2	6.6 ± 0.8	6.6 ± 1.4	6.3 ± 2.0
B-5	6.7	5.9	6.8 ± 1.2
B-R	27.8	29.9	26.0 ± 0.0
B-C	3.7	3.8	8.0 ± 0.0
Cum. Dev	**5.0%**	**6.6%**	**12.8%**

The FI values from *A*FIT™ and visual observations were also assessed relative to the LOI of the CCA's. These data showed that samples with nearly identical LOI's can have widely variant FI values. Hence, in agreement with published results (2,3), FI values rather than LOI values predict the interaction of AEA's with CCA's.

CONCLUSIONS

The foam index of commercial CCA's can be rapidly obtained by monitoring the AE emanating from CCA/water/AEA mixtures after agitation. The precision of FI values obtained from the AE data was better than from visual data. These outcomes confirm the potential application of an *A*FIT™ instrument to reliably replace current visual FI measurement methods.

The FI procedure used a specific protocol and an AEA identified as a potential 'standard'. Rather then using a 'standard' AEA, industries purchase AEA's from a multitude of manufacturers and then use their own protocols and timing during FI measurements. Because their FI values are all based on foam stability independent of the AEA and the protocol used, and because timing during AE data collection can be adjusted via computer interfaces, *A*FIT™ instruments promise the flexibility needed to meet requirements imposed by users.

ACKNOWLEDGEMENTS

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