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Estimation of the degree of hydration of blended cement pastes by a scanning electron microscope point-counting procedure

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Abstract

A scanning electron microscope (SEM) point-counting technique was employed to study the hydration of plain portland and blended cement pastes containing fly ash or slag. For plain portland cement pastes, the results for the degree of cement hydration obtained by the SEM point-counting technique were consistent with the results from the traditional loss-on-ignition (LOI) of nonevaporable water-content measurements; agreement was within $\pm 10\%$. The standard deviation in the determination of the degree of cement hydration via point counting ranged from $\pm 1.5\%$ to $\pm 1.8\%$ (one operator, one sample). For the blended cement pastes, it is the first time that the degree of hydration of cement in blended systems has been studied directly. The standard deviation for the degree of hydration of cement in the blended cement pastes ranged from $\pm 1.4\%$ to $\pm 2.2\%$. Additionally, the degrees of reaction of the mineral admixtures (MAs) were also measured. The standard deviation for the degree of fly ash reaction was $\pm 4.6\%$ to $\pm 5.0\%$ and $\pm 3.6\%$ to $\pm 4.3\%$ for slag. All of the analyses suggest that the SEM point-counting technique can be a reliable and effective analysis tool for use in studies of the hydration of blended cement pastes.

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Keywords: Hydration; Blended cement; Fly ash; Granulated blast-furnace slag; Point counting

1. Introduction

There are many experimental techniques used to measure the degree of hydration of portland cement, such as measuring the heat of hydration, the nonevaporable water content, and the amount of calcium hydroxide produced in the hydration reactions. All of these methods are based on comparisons of the measured parameters with the predicted or measured parameters for a fully hydrated paste. When reactive mineral admixtures (MAs) are incorporated into portland cement, the system becomes more complex. It is almost impossible to determine the degree of the cement hydration by the techniques used for plain cement paste. Although some researchers [1–5] have measured the degree of reaction of MAs by a selective dissolution procedure, little information can be obtained in this manner about the cement hydration in a blended system. Thus, it would be valuable to develop a technique that can simultaneously and

directly study the degree of reaction of both MAs and the portland cement in blended cement pastes.

In recent years, scanning electron microscopy (SEM) quantitative techniques have been used in cement systems to directly estimate the degree of hydration of plain portland cement pastes [6–8]. These researchers collected backscattered electron images (BEI) of cement pastes, and then quantitatively analyzed them using a system software based on the different gray levels of the different cement phases. For example, in Ref. [6], for cement pastes with water-to-cement mass ratio (w/c) = 0.45 cured at 20 °C, the degree of hydration was about 70% at 1 day, and about 80% at 3 days. These results were much higher than those obtained from calcium hydroxide measurements and density determinations. The large disagreement may have been caused by the selection of inappropriate thresholds in the gray scale images or by limiting the image resolution so as to be able to discriminate between different gray levels. In blended cement pastes, discriminating between phases is even harder due to the similar gray levels of some of the fly-ash, the slag, and the cement clinker particles. Another SEM imaging technique, X-ray imaging, may be used to estimate the

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degree of hydration of blended cement pastes because it gives elemental composition information, which makes it easier to discriminate between phases. However, because this technique can only examine a very small field of view, it requires several hours of processing time.

In the current study, a SEM-based point-counting technique was used to directly study the degree of hydration of cement and the degree of reaction of the MA in blended cement pastes containing fly ash or slag. A similar optical-based point-counting technique is the basis for a number of ASTM standard test methods [9,10]. The technique accesses the same field of view as does X-ray imaging, but in a fraction of the time, so that enough fields of view for statistical purposes can be examined in a reasonable length of time. This is an important consideration considering that the point-counting technique requires a human operator. Loss-on-ignition (LOI) measurements of nonevaporable water content for the plain portland cement pastes were also performed to compare with the results from the SEM point-counting technique. The accuracy of the LOI measurements is not known, but they could be a precise measure because they are based on mass measurements, which can be performed with great precision.

2. Experimental procedures

2.1. Materials

Two kinds of Type I portland cements were selected for the study and were designated as T-1H and T-1D. The chemical compositions of the two cements, the Class F fly ash, and the slag are given in Table 1 in terms of oxide mass percents. The LOI of the fly ash is low compared with most Class F fly ashes because this fly ash was chosen for its low carbon content and, hence, negligible effect on air entrainment.

2.2. Paste preparation

Two plain portland cement pastes were made by mixing deionized water in a blender with either T-1H or T-1D

Table 1
Chemical compositions of cements, fly ash and slag used (mass fraction, %)

	T-1H	T-1D	Fly ash	Slag
SiO ₂				
Al ₂ O ₃				
Fe ₂ O ₃				
CaO				
MgO				
SO ₃				
K ₂ O				
Na ₂ O				
LOI				

These are taken directly from the manufacturers' analyses.

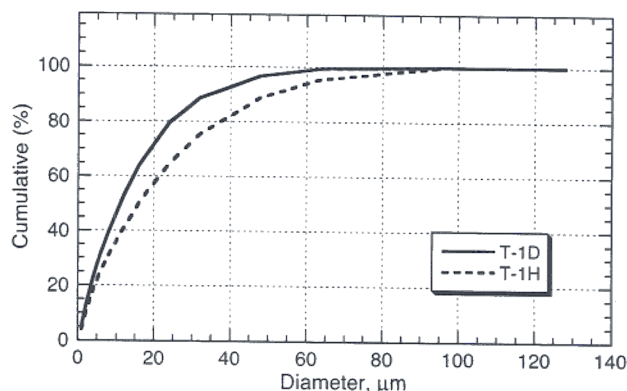


Fig. 1. Cumulative particle size distributions for the T-1D and T-1H portland cements. The minimum particle size measured was 1 μm . Air dispersion and laser scattering techniques were used.

cement at $w/c = 0.4$. The slag blended cement (designated as T-S) was made by mixing 30% (by mass fraction) slag with 70% T-1D portland cement at a 0.4 water-to-solid binder (w/s) mass ratio. Both the slag and this cement, T-1D, were supplied by Dyckerhoff. The fly-ash-blended cement (designated as T-L) was made by mixing 20% (by mass fraction) fly ash and 80% T-1H cement at a 0.4 w/s mass ratio. Both the fly ash and the T-1H cement were supplied by Holcim. Overall, four different cement pastes were studied.¹

Fig. 1 shows the measured cumulative particle size distributions (PSD) for the T-1D and T-1H cements. The minimum diameter is 1 μm . These PSD curves were measured using a laser-scattering instrument with air dispersion (dry dispersion). A standard methodology for PSD determination, worked out in the context of several ASTM round robins, was used [11]. Fig. 2 shows similar PSD curves for the slag and fly-ash materials, with the same minimum diameter of 1 μm . Wet dispersion in propanol was used for these materials. The PSD of the blended cements used in this study would, of course, be a weighted average of the relevant curves in Figs. 1 and 2. In Fig. 1, the T-1D cement was finer than the T-1H cement, and Fig. 2 shows that the slag material was finer than the fly ash material.

To eliminate entrapped air bubbles, house vacuum was employed during the mixing and casting processes, and a vibration table was used during casting. To prevent substantial bleeding, the samples were cast in sealed plastic molds and slowly rotated for 5 h on a roller. By constantly changing the orientation of the samples with respect to the direction of gravity, bleeding was prevented. After this, the pastes were kept in the molds and put in sealed plastic bags at 20 °C and 100% RH until the testing age was reached.

¹ Certain commercial equipment and/or materials are identified in this report to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment and/or materials used are necessarily the best available for the purpose.

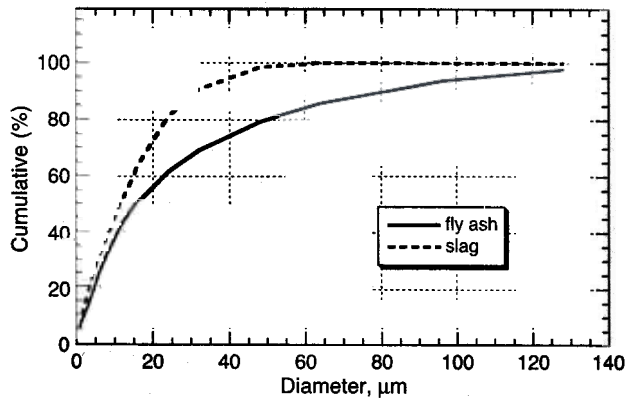


Fig. 2. Cumulative particle size distributions for the fly ash and slag used. The minimum particle size measured was 1 μm . Wet dispersion in propanol and laser scattering techniques were used.

2.3. LOI measurements and degree of hydration

The LOI nonevaporable water content measurements were carried out following a procedure described elsewhere [12]. Nonevaporable water content was determined as the relative mass loss between 105 and 1000 $^{\circ}\text{C}$, corrected for the LOI of the dry cement powder itself. To convert the nonevaporable water content measurements to estimated degrees of hydration, α , it was necessary to determine the nonevaporable water content for a fully hydrated sample. Based on the compositions of the two plain portland cements listed in Table 1, the values of 0.24 g $\text{H}_2\text{O}/\text{g}$ cement and 0.234 g $\text{H}_2\text{O}/\text{g}$ cement were determined for cements T-1H and T-1D, respectively. The degree of hydration was then determined as the ratio of the measured nonevaporable water content per gram of cement to the amount at full hydration.

2.4. SEM sample preparation and point-counting procedure

Samples for SEM examination were taken out and broken into small pieces and immersed in ethanol to stop hydration. After the pore water had totally exchanged with the ethanol, the small samples were immersed in an acrylic epoxy resin for two weeks so that the resin could exchange

with the ethanol. After two weeks, the resin was cured at 70 $^{\circ}\text{C}$. The sample was then polished and carbon coated for SEM point-counting analysis.

The point-counting procedure was modified from ASTM C 1356 M. Basically, a grid was superimposed on top of the SEM image, and the phases were determined and counted at each grid point by an expert human operator. The detailed procedure was as follows.

The SEM magnification was set at $\times 1000$ to $\times 1500$, depending on the size of the particles in the paste. The purpose of using different magnifications was to avoid having adjacent grid points fall on the same particle. A 25-point grid was selected using the SEM configuration setup. A simple counting device was used to record the number of points that fell into the following two phases in the plain portland cement pastes: (a) unhydrated cement particles and (b) all others. Three phases were recorded in the blended cement pastes: (a) unhydrated cement particles, (b) unreacted fly ash or slag particles, and (c) all others. The overall number of points counted for each sample was 3000, which was chosen to be able to obtain reasonable uncertainties of the estimates. The more points counted, the smaller the uncertainties become. The phase at each grid point was identified and counted. If the phase was obscured by the area formed by the grid intersection point, then the phase on the upper right corner of the grid point was recorded as the identified phase.

Assuming that the sample had a homogeneous microstructure, the grid was moved across the specimen. There were 120 fields to be viewed in each specimen to get a total of 3000 points counted ($3000 = 120 \times 25$). The total area of the field of view was 3.60 mm^2 (higher magnification) to 8.55 mm^2 (lower magnification). After recording all the 25 points on one field, the field of view was moved systematically to examine a large sample field.

Fig. 3 shows typical fields of view that were encountered during the point-counting procedure. Fig. 3(a) shows a plain portland cement paste. At some of the grid points, the number 1 has been marked to show that this grid point has been identified as a cement clinker particle. The grid points that have not been marked have been identified as hydration

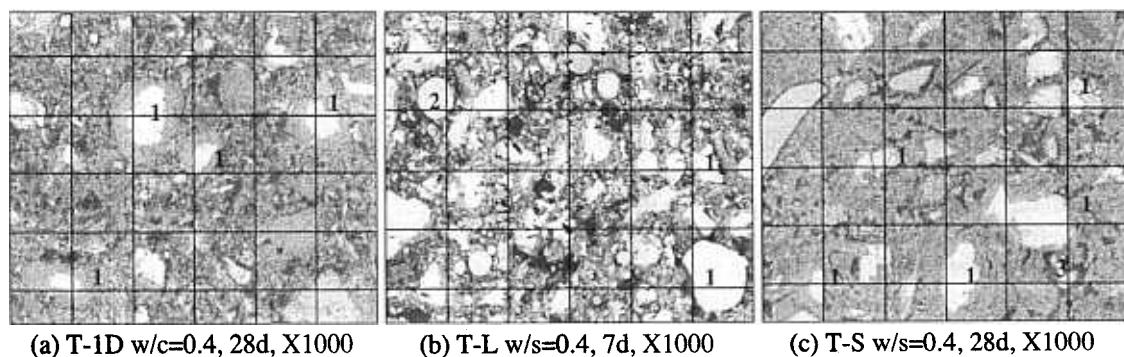


Fig. 3. Typical fields of view used for point counting on plain cement paste and blended cement pastes.

products and voids, which are counted as “all others” in this technique. Fig. 3(b) and (c) shows fly-ash- and a slag-blended cement pastes, respectively. In both microstructures, the gray levels of some fly-ash and some slag particles were very close to those of hydration products, while the gray levels of the others were very close to the cement clinker particles. To help distinguish between these phases, the examiner can use particle morphology and, if necessary, use the X-ray signal to show the elemental abundance at a questionable point. Using these indicators, as well as the gray level, one can readily discriminate among the fly-ash and slag particles, hydration products, and cement clinker particles at each grid point. In Fig. 3(b), there is one fly-ash particle falling at grid points that is marked “2”, two cement clinker particles that are marked “1”, and the unmarked intersections are counted as “all others”. In Fig. 3(c), there are five cement clinker particles marked “1”, one slag particle marked “3”, and the unmarked grid points have been counted as “all others”.

2.5. Calculations of the degree of cement hydration, degree of reaction of mineral admixtures, and standard deviations

The volume fraction of each phase in the sample was calculated by dividing the number of points counted for that phase by the total number of points counted. When entrapped air is present, one has to correct the total number of points by subtracting the number of points that fall into the air phase. There was very little entrapped air present in these cement paste samples, certainly much less than 1% by volume. When calculating the degree of hydration of cement, one must also correct for the volume fraction of gypsum in the clinker. Detailed calculations are shown as follows.

Symbols:	
w/c	Water-to-cement ratio
$V_{(0)cem}$	Volume fraction of clinker particles in cement, %
$V_{(t)cem}$	Volume fraction of unhydrated cement at hydration time t , %
$V_{(0)ma}$	Volume fraction of unreacted MAs before hydration, %
$V_{(t)ma}$	Volume fraction of unreacted MAs at hydration time t , %
V_f^{GYP}	Volume fraction of gypsum in the clinker, %
α_{cem}	Degree of hydration of cement paste, %
α_{ma}	Degree of reaction of the MAs, %
m_{cem}	Mass of cement in 100 g binder, g
m_{ma}	Mass of MAs in 100 g binder, g
m_{H_2O}	Mass of water needed for 100 g binder by w/c ratio, g
ρ_{cem}	Specific gravity of cement, typically 3.2
ρ_{ma}	Specific gravity of mineral admixtures, 2.36 for fly ash and 2.87 for slag
ρ_{H_2O}	Specific gravity of H ₂ O, 1.0 at 20 °C

2.5.1. Plain portland cement paste

The volume fraction of cement in the paste before hydration can be calculated as:

$$V_{(0)cem} = \frac{1}{1 + \rho_{cem} \times (w/c)} \times 100\% \tag{1}$$

The volume fraction of unhydrated cement at hydration time t , $V_{(t)cem}$, is calculated by dividing the number of points counted for this phase by the total number of points counted (3000 in the current study). However, one needs to apply a gypsum correction. All the gypsum contained in the cement is substantially reacted by about 24 h after mixing [13]. Therefore, it will not be counted as a cement clinker phase during point counting. The gypsum does not contribute to the cement paste LOI measurement because it is not present, but it does contribute to the initial volume of cement. Therefore, the initial volume fraction of portland cement needs to be reduced by the amount of gypsum originally present in the portland cement when calculating the degree of hydration of the cement paste at time t :

$$\alpha_{cem}(\%) = \left\{ 1 - \frac{V_{(t)cem}}{V_{(0)cem}(1 - V_f^{GYP})} \right\} \times 100\% \tag{2}$$

2.5.2. Blended cement

In a blended cement, the volume fractions of MA and cement are calculated on the basis of 100 g of binder, and the w/s was used instead of the w/c. Thus, the volume fractions of these two solid materials in the paste before hydration can be calculated as:

$$V_{(0)cem} = \frac{\frac{m_{cem}}{\rho_{cem}}}{\frac{m_{ma}}{\rho_{ma}} + \frac{m_{cem}}{\rho_{cem}} + \frac{m_{H_2O}}{\rho_{H_2O}}} \times 100\% \tag{3}$$

$$V_{(0)ma} = \frac{\frac{m_{ma}}{\rho_{ma}}}{\frac{m_{ma}}{\rho_{ma}} + \frac{m_{cem}}{\rho_{cem}} + \frac{m_{H_2O}}{\rho_{H_2O}}} \times 100\% \tag{4}$$

The degree of hydration of cement in the blended paste at time t can then be calculated as:

$$\alpha_{cem}(\%) = \left\{ 1 - \frac{V_{(t)cem}}{V_{(0)cem}(1 - V_f^{GYP})} \right\} \times 100\% \tag{5}$$

The degree of reaction of the MA in the blended paste at time t can be calculated as:

$$\alpha_{ma}(\%) = \left\{ 1 - \frac{V_{(t)ma}}{V_{(0)ma}} \right\} \times 100\% \tag{6}$$

The standard deviation in the point counting is [14]:

$$\sigma_p = \sqrt{\frac{P(100 - P)}{N}} \tag{7}$$

where σ_p is the standard deviation in the point-counting measurement of P (%) for a given constituent, P is the phase percentage of the given constituent, and N is the total number of points counted. In the current study, $N=3000$.

This standard deviation in the phase volume fraction is then converted to the standard deviation in the degree of cement hydration:

$$\sigma_{\alpha} = \left\{ \frac{\sigma_p}{V_{(0)\text{cem}}(1 - V_f^{\text{GYP}})} \right\} \times 100\% \quad (8)$$

where σ_{α} is the measured standard deviation for the degree of hydration as determined by point counting. This is done similarly for the blended cements and standard deviation in the degree of reaction in the MA.

3. Results and discussion

3.1. Degree of hydration of plain cement pastes obtained from LOI and SEM point-counting

The estimated degrees of hydration of the plain portland cement pastes made from the T-1H and T-1D cements as a function of age, determined by LOI and SEM point-counting, are listed in Table 2. The Δ column in the table stands for the difference between those two measurements (SEM point counting – LOI), divided by the LOI results and multiplied by 100%. The results are consistent with each other, as the deviations for both cement pastes are within $\pm 10\%$.

One should note that, in the Δ columns, the value of Δ is almost always negative. This indicates that the SEM point-counting results for the degree of hydration are systematically lower than the LOI measurements. This may be partly due to other materials releasing mass in the temperature range 105–1000 °C, like calcium carbonate, which releases carbon dioxide, which would tend to make the LOI results higher than the point-counting results. The calcium carbonate could come from the partial carbonation of the CH present.

Fig. 4 shows the regression analysis between the data obtained by LOI and SEM point counting for the two plain cement pastes. The values of R^2 for these regressions are .90 for the T-1H cement and .85 for the T-1D cement, with the regression lines forced to go through the origin. Note that both slopes are close to and a little larger than unity. The fact that the point-counting measurements seem to be systematically lower than the LOI measurements could be due to a multiplicative factor or to an additive factor. This kind of

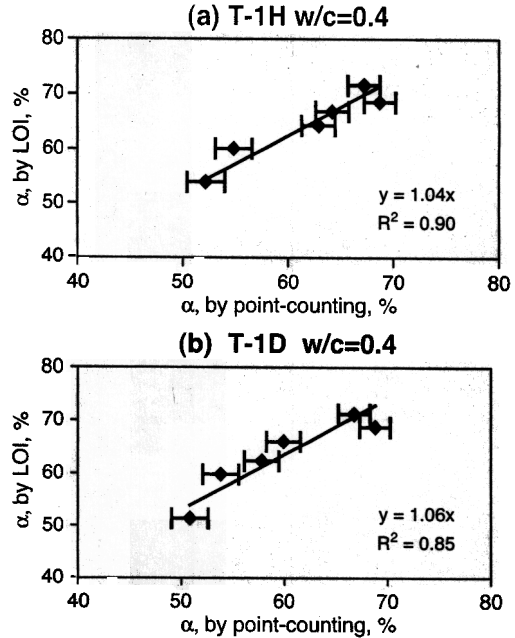


Fig. 4. Regression analysis relating SEM point counting and LOI determinations of α for the plain portland cement pastes.

regression fit indicates a multiplicative factor. If the intercepts are not forced to be 0, then, the regression equations become:

T – 1H cement paste:

$$\alpha\%_{(\text{LOI})} = 0.91 \times \alpha\%_{(\text{SEM})} + 0.08, R^2 = .91$$

T – 1D cement paste:

$$\alpha\%_{(\text{LOI})} = 0.94 \times \alpha\%_{(\text{SEM})} + 0.07, R^2 = .86$$

The R^2 factors are now somewhat closer to one, and the slopes are now a little less than one. The positive intercepts are slightly larger than zero, which means that the degree of cement hydration determined by LOI is a little larger than that determined by SEM due to an additive factor. This is reasonable because some of the very fine particles of the cement clinker may be miscounted due to the limited resolution and because of the overall uncertainty in phase identification. Fig. 4 shows the actual standard deviations in the degree of cement hydration as determined by point counting, calculated according to Eqs. (7) and (8). The standard deviation in the point-counting determination of the degree of hydration ranged from $\pm 1.5\%$ to $\pm 1.8\%$.

All of the above analyses suggest that the results from SEM point counting are close to those from the traditional LOI method and that the differences are within a reasonable range. However, the SEM point-counting results are systematically lower than the LOI measurements. The uncertainties of the LOI measurements are much lower than those for the point-counting procedures and would not show up on

Table 2
Degree of hydration of plain cement pastes by LOI and point counting

Age (days)	T-1H (w/c=0.4)			T-1D (w/c=0.4)		
	$\alpha\%$ SEM	$\alpha\%$ LOI	Δ (%)	$\alpha\%$ SEM	$\alpha\%$ LOI	Δ (%)
3	52.2	53.9	-3.1	50.8	51.4	-1.1
7	54.8	60.0	-8.7	53.8	59.8	-9.9
14	62.9	64.3	-2.2	57.8	62.4	-7.3
28	64.2	66.8	-4.0	59.9	66.1	-9.4
60	68.7	68.6	0.2	66.8	71.3	-6.3
90	67.2	71.6	-6.1	68.8	68.8	0

the scale used in Fig. 4. Thus, SEM point counting, when carefully used, can be an effective technique to determine the degree of hydration of plain cement paste. Having tested this procedure on plain portland cement pastes, attention is now turned to its much more important use on blended cement pastes, as LOI measurements are not meaningful at present for blended cements.

3.2. Degree of hydration of portland cement in blended cement systems

Fig. 5 shows the degree of hydration of the portland cement in blended cement pastes compared with the degree of hydration of plain cement pastes, as determined by point counting only. The incorporation of MAs can significantly accelerate the cement hydration in blended systems as early as 3 days, especially for the slag-blended cement. This effect is more significant between 7- and 28-day hydration, and is less significant at later ages. There are two possible reasons for this acceleration: (1) the relatively higher w/c in the blended system and (2) the pozzolanic reaction between the MA and the calcium hydroxide released from the cement hydration in the fly-ash-blended cement. The standard deviation for the degree of cement hydration in the blended cement pastes, as determined by point counting, ranged from $\pm 1.4\%$ to $\pm 2.2\%$.

One should note that, often, the addition of fly ash retards portland cement hydration. This particular fly ash had been

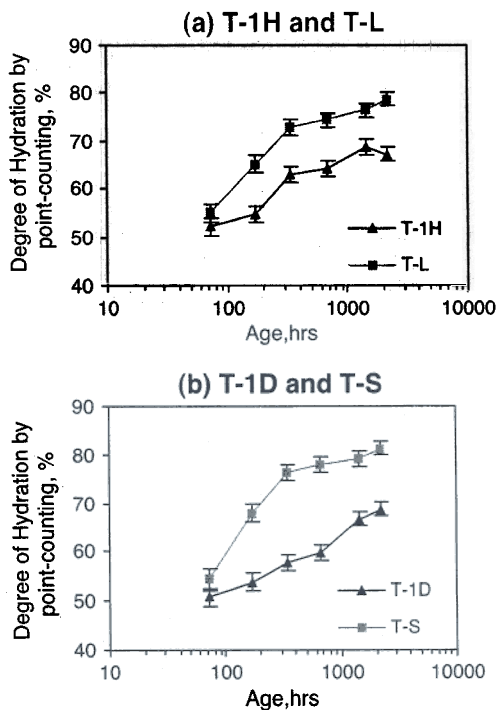


Fig. 5. Degree of hydration of cement in plain and blended cement systems: (a) T-1H portland cement, T-L blended cement (fly ash added), and (b) T-1D portland cement, T-S blended cement (slag added).

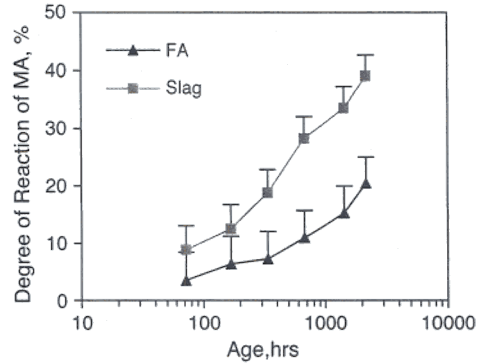


Fig. 6. Degree of reaction of MA in blended cement systems.

used in the manufacture of a fly-ash-blended cement by Holcim, so that a fly ash that retarded cement hydration would not have been so used. Hence, it is not surprising that no apparent retardation with the fly ash present was seen experimentally.

3.3. Degree of reaction of MAs in blended cement systems

The degrees of reaction of MA in the blended systems are shown in Fig. 6, along with the standard deviations. These degrees of reaction come from a direct point-counting measurement. At all ages, slag reacts much faster than fly ash due to its inherent hydraulic and higher pozzolanic reactivity. The standard deviation for fly ash reacted is from $\pm 4.6\%$ to $\pm 5.0\%$, while for slag reacted, it is from $\pm 3.6\%$ to $\pm 4.3\%$. The higher standard deviation for fly ash than for slag is due to the lower replacement of fly ash (20%) in the blended cement pastes than for slag (30%). In Eq. (8), similar point-counting standard deviations will be divided by smaller numbers, thus giving larger standard deviations for the extents of reaction. These results are comparable with the results from selective dissolution methods as performed by other researchers [2,3]. For example, with a 20% fly ash replacement, a 10% fly-ash extent of reaction at 28 days was reported by Hardtl [2]. Our work here shows an 11% reaction extent, with a standard deviation of $\pm 4.8\%$. With a 30% slag replacement, Lumley et al. [3] found a 35% slag degree of reaction at 28 days, while in our work, a $28 \pm 3.9\%$ reaction extent was measured. Standard deviations for the literature measurements were not given.

4. Conclusion

For blended cements containing fly ash or slag, LOI measurements cannot, at present, be used to determine the degree of hydration. Therefore, a SEM point-counting procedure was adopted to determine the amount of unreacted cement and mineral admixture left at a given time, which gives the degree of reaction if the original amount is known.

This procedure was first checked by comparing SEM point-counting with LOI measurements on two plain portland cements. It was found that the degree of hydration of plain portland cement pastes obtained from SEM point-counting techniques were consistent with the results from the traditional LOI method, with differences between the two methods no more than $\pm 10\%$. The SEM point-counting results were systematically lower than the LOI measurements. The standard deviation in the point-counting determination of the degree of hydration of plain portland cement ranged from $\pm 1.5\%$ to $\pm 1.8\%$. This is for a single operator, on a single small sample cut from a larger sample. Once this technique was established, it was used to measure the degrees of hydration of cement in blended cement systems for the first time. The standard deviations for the degree of hydration of cement in the blended cement pastes were within $\pm 1.4\%$ and $\pm 2.2\%$. The same SEM point-counting technique was also used to measure the degree of reaction of the MAs in the blended cements. The results were comparable with previous results obtained using a selective dissolution method. The standard deviations for the degrees of fly-ash reaction ranged from $\pm 4.6\%$ to $\pm 5.0\%$ and, for the slag-blended cements, from $\pm 3.6\%$ to $\pm 4.3\%$.

Results of the present study indicate that the SEM point-counting technique can be a reliable and effective analysis tool to measure the degree of hydration of the portland cement and MAs in blended cement pastes. These measurements will allow models like the Virtual Cement and Concrete Testing Laboratory [15] to be validated on these materials. With the growing use of blended cements utilizing waste stream materials, this combined measurement and prediction capability will allow better and more controlled use of these materials.

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