STABILITY OF PORTLAND CEMENT-BASED BINDERS
REINFORCED WITH NATURAL WOLLASTONITE MICRO-FIBRES

Norman M. P. Low
Department of Civil Engineering, University of Ottawa, Ottawa, Canada

J. J. Beaudoin
Materials Laboratory, Institute for Research In Construction
National Research Council of Canada, Ottawa, Canada

ABSTRACT

The stability of Portland cement-based binders reinforced with natural wollastonite micro-fibres was investigated for hydration periods up to one year. The wollastonite micro-fibres imbedded in the hydrated cement paste were examined employing a scanning electron microscopy technique. Composite specimens were also periodically evaluated by flexural strength testing and microstructural characterization including mercury intrusion porosimetry, helium gas pycnometry, and isopropyl alcohol saturation measurement. The amount of $Ca(OH)_2$ in the hydrated matrices was also determined by differential scanning calorimetry. Wollastonite micro-fibres imbedded in hydrated cement-silica fume matrices remained stable after prolonged hydration and exhibited no surface or bulk deterioration. The flexural strength and overall pore structure of the Portland cement-based binders reinforced with wollastonite micro-fibres also remained essentially unchanged and unaffected. Flexural toughness and the post peak deflection, however, were observed to decrease with hydration time. The amount of $Ca(OH)_2$ in the hydrated matrices decreased slightly at advanced hydration times. The observed behaviour is discussed.

## Introduction

Several previous investigations have systematically demonstrated the advantages of reinforcing Portland cement-based binders with natural wollastonite micro-fibres. These included the enhancement of flexural strength, flexural toughness and ductility of cement-silica fume based composite systems [1-4]. The results were generally based on experimental data obtained after hydration for a period of about 28 days. This approach was consistent with other workers in the field studying the performance of cement-based composite mixtures reinforced with steel and carbon micro-fibres and other man-made reinforcing material [5-10]. There appears, however, to be a dearth of information with regard to long term stability of cement-based binders reinforced with natural wollastonite micro-fibres. For this reason, a systematic study was carried out to assess the one year performance of wollastonite reinforced binders. A series of specimens prepared with a base matrix consisting of 95 parts cement and 5 parts silica fume served as a reference. A second series of specimens was prepared with the same matrix reinforced with 11.5% by volume of wollastonite micro-fibres. The amount of wollastonite micro-fibres was based on a previous investigation which showed that maximum flexural strength was attained using this amount. Specimen blocks prepared from both systems were immersed in a saturated calcium hydroxide solution and hydrated for a period of one year. Periodically, a composite block from each series was removed from the solution and small beam specimens were prepared for flexural strength evaluation and microstructural characterization. The amount of $Ca(OH)_2$ in the hydrated matrices after drying was also determined by thermal analyses. The stability of the wollastonite micro-fibres imbedded in the hydrated cement matrix was examined by scanning electron microscopy. The results of the study are presented in this paper.

<u>Experimental</u>

Materials

Type 10 Portland cement* was used as the base component. The natural wollastonite micro-fibres consisting of a mixture of various acicular particle sizes were obtained from an American supplier**. The silica fume*** was similar to those used in the previous investigations. The superplasticizer solution**** is made up of about 48% solids and 52% water.

Specimen Preparation

All sample preparations were carried out in a similar manner. The silica fume was first manually blended with cement in a steel mixing bowl until a uniform mixture was obtained by visual inspection. The wollastonite micro-fibres were then gradually blended with the cement-silica fume mixture until a uniform solid mixture was obtained. Superplasticizer solution equivalent to about 0.3% by weight of cement solids was used in the preparation of the cement-silica fume composite specimens and about 1% by weight was used in the preparation of the cement-silica fume-wollastonite composite specimens. A water/cement+silica fume ratio of 0.35 was used for both composite systems. The liquid water containing the superplasticizer solution was first placed in the mechanical blender and mixed for about 1 minute. The dry mixed solids were then added to the water solution and blended for a period of about 4 minutes. The blended cement composite mixture was cast into a plastic mould which measured about 40 x 130 x 4 mm. The cement mixture was allowed to harden in a moisture controlled room at saturation for a period of 24 hours. The hardened cement specimen was then de-moulded. After the de-moulding process, the specimen blocks were kept in a closed desiccator containing the saturated calcium hydroxide solution. The specimen blocks were allowed to hydrate in a continuing manner at ambient temperature. At an early stage in the study, a sample block was removed from the calcium hydroxide solution and sliced into small beams measuring about 4 x 4 x 65 mm for evaluation every 7 days up to 49 days hydration. Subsequent to this initial period, a similar block was removed for specimen preparation and evaluation at approximate intervals of 49 days (7 weeks) hydration. The beam specimens were first used for flexural strength determination and then microstructural characterization.

Test Measurements

The flexural strength of each composite beam specimen was determined by a three-point loading test method using a computer controlled Material Testing System[#]. Prior to the bending test, each beam specimen was maintained in the calcium hydroxide solution after the slicing operation. During test, the excess water on the surface of the specimen was first removed using a water absorbent type paper and the specimen was then installed on the specimen support. A total of 9 specimens from each composite system was tested after each hydration period and the average result was used.

Subsequent to the bending test, two fractured beam specimens which gave similar flexural strength values were selected from each composite system for porosity measurement. Prior to the porosity evaluation process, all specimens were oven-dried at 105 °C for a period of at least 72 hours. The pore volume in each selected specimen was first determined using the helium gas pycnometry method. After the pycnometry measurement, each beam specimen was broken into two portions of approximately equal size and weight. One portion was prepared for the mercury intrusion porosimetry measurement and one portion was used for the determination of the $Ca(OH)_2$ content by thermal analyses employing a differential scanning calorimeter. The other specimen was used for isopropyl alcohol saturation measurement.

For helium gas pycnometry measurement, the true volume of the whole beam specimen was accurately measured and the measurement was repeated 3 or 4 times until a consistent value was obtained. The helium gas pycnometer[##] was operated at a gas pressure of about 0.18 MPa (20 psi).

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* Type 10 Portland cement containing about 19.8% $SiO_2$, 4.2% $Al_2O_3$, 3.2% $Fe_2O_3$, and 61.2% CaO was supplied by the St. Mary's Cement Co., Montreal, Quebec, Canada.
** The natural wollastonite micro-fibres were of the NYAD G grade obtained from NYCO Minerals, Inc., Willsboro, N. Y., U.S.A.
*** The silica fume containing about 95.2% $SiO_2$, 1.6% carbon, 0.27% $K_2O$, and 0.10% $Na_2O$ was supplied by the SKW Co., Montreal, Quebec, Canada.
**** The superplasticizer solution was the Atlas Mighty 150 RD2 grade.
# The Material Testing System is the MTS Model 810 apparatus manufactured by MTS Systems Corp., Mpls, MN, U.S.A.
## The helium gas pycnometer is the Steropycnometer manufactured by Quantachrome Corp. New York, U.S.A.

For mercury intrusion measurement, a portion of each specimen was first broken into 9 to 10 small pieces which were then inserted into the specimen container of the porosimeter. The mercury porosimeter[###] was operated at pressures up to a maximum of 414 MPa (60,000 psi).

A specimen for isopropyl alcohol saturation measurement was first weighed accurately to obtain its dry weight. The geometric volume of the specimen was determined from the width, length and height of the specimen. All specimens were vacuum-dried at ambient temperature for a period of about three hours inside a closed desiccator. Isopropyl alcohol was allowed to flow into the desiccator to saturate the specimens for a period of three hours after the vacuum drying process. The saturated weight of the specimen was obtained and the amount of isopropyl alcohol entered into the specimen was determined. The alcohol volume with respect to the geometric volume of the specimen was used to calculate the porosity.

A differential scanning calorimeter[####] was used to determine the extent of $Ca(OH)_2$ formation. The relative area of the peaks in the thermograms resulting from the thermal decomposition of the $Ca(OH)_2$ was determined using a DuPont Model 2100 Thermal Analyser system. In each measurement, a small piece of the solid was crushed into fine granules and a sample equivalent to approximately 100 mg was loaded into the specimen container and heated in a nitrogen atmosphere at a rate of 20 $^{o}$C/min.

The wollastonite micro-fibres exposed on the fractured surface of the composite specimen were examined by scanning electron microscopy[#####].

<center>Results and Discussion</center>

The hydration of the cement-silica fume and the cement-silica fume-wollastonite composite systems in saturated calcium hydroxide solution for an extensive period was observed to produce some unique characteristics. The discussions of the results follow:

[A] Stability of Wollastonite Micro-Fibres

Natural wollastonite mineral is a rigid solid crystalline material; the surface of the individual micro-fibres is generally smooth and free from defects. The stability of the wollastonite micro-fibres imbedded in the hydrated cement-silica fume matrices was closely examined by a scanning electron microscopy technique. This was done by focusing on the surface features of a randomly selected wollastonite fibre under a magnification of about 5000X to look for surface deterioration and/or deposits of foreign substances. The individual wollastonite micro-fibre selected for examination was one of many fibres exposed on the fracture surface. Two specimens were examined, one after hydration for 28 days and the other after hydration for 350 days. In both cases, the wollastonite micro-fibres had a clean and smooth surface exhibiting no surface erosion or deposition of foreign substance resulting from chemical reaction with the hydrated cement paste, as shown in Figure 1. This result indicated that wollastonite micro-fibres are macroscopically stable in the composite matrices. Highly alkaline cement paste did not appear to have any deleterious effect on the fibres even after a prolonged hydration period.

[B] Flexural Strength

The flexural strength of the hydrated cement-silica fume composites (reference system) at 28 days was 8.5 - 9.3 MPa (1230 - 1360 psi) and the strength level remained within this range independent of hydration time. The flexural strength increased to 20.9 - 24.9 MPa (3030 - 3610 psi) when 11.5% by volume of wollastonite micro-fibres were added to the base cement -silica fume matrix. There appears to be no systematic change of flexural strength level with hydration time in the cement-silica fume-wollastonite composite system. The overall flexural strength level observed in the present study was found to be consistent with those results observed in previous investigations [2]. The variation of flexural strength with hydration time for the cement-silica fume and the cement-silica fume-wollastonite systems is shown in Figure 2. It is evident that the hydrated cement paste in both composite matrices has attained maximum strength level within 30 days of hydration and continuing hydration in calcium hydroxide solution for longer periods contributes no further strength improvement. Stability of wollastonite micro-fibres after exposure to prolonged hydration in a highly alkaline environment and relative consistency of the flexural strength in the composite system indicate that the contribution of wollastonite micro-fibres to the strengthening process is apparently unaffected by aging.

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### The mercury porosimeter is the Model 60,000 psi Porosimeter manufactured by American Instrument Co., Maryland, U.S.A.
#### The differential scanning calorimeter is the DuPont Model 951 unit manufactured by DuPont Instrument, Wilmington, DE, USA.
##### The scanning electron microscope is the Cambridge Steroscan Model S250 manufactured by Cambridge Instrument, England.
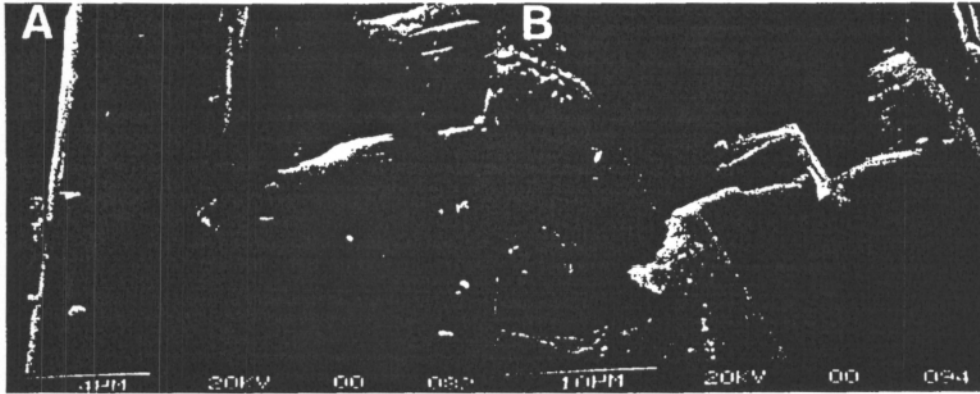
Figure 1. SEM photomicrograph of the wollastonite micro-fibres in
hydrated cement-silica fume paste
[A] After hydration for 28 days
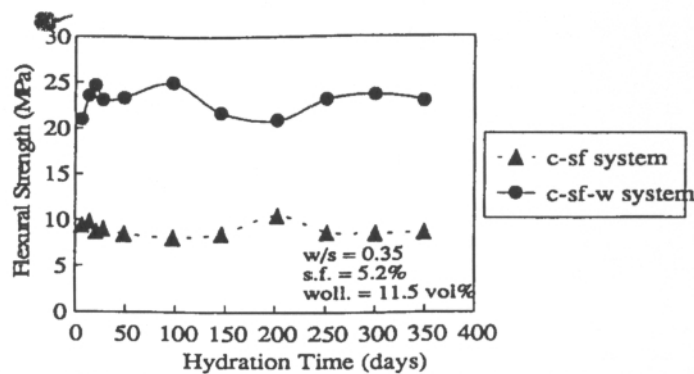[B] After hydration for 350 days



Figure 2. Variation of flexural strength with hydration period in the
cement-silica fume (c-sf) and cement-silica fume-
wollastonite (c-sf-w) composite systems.

[C] Flexural Toughness and Ductility

Addition of wollastonite micro-fibres in cement and cement-silica fume matrices can result in a significant modification of the flexural toughness and ductility characteristics of composite mixtures [1,3,4]. The modifications are clearly shown in load-deflection curves obtained from the bending test. Load-deflection curves are shown in Figures 3 for the cement-silica fume system and in Figure 4 for the cement-silica fume-wollastonite composite system after various periods of hydration. It is evident that the flexural characteristics for both cement-silica fume and cement-silica fume-wollastonite composite matrices are affected by the prolonged period of hydration. The flexural toughness of both composite specimens after various period of hydration was determined by a simple calculation method from the load-deflection curve. The arithmetic average of two applied loads within a finite change of applied loads is calculated and the resulting deflection increment is measured. The product of these two values represents a finite amount of work done leading to fracture. The sum of all these finite area increments over the entire load-deflection curve thus represents the total flexural toughness. The calculation method of flexural toughness was discussed in detail in a previous investigation [3]. In the present study, the flexural toughness of the base cement-silica fume matrix after 28 days hydration was considered to be the reference. The relative toughness, defined as the flexural toughness of the composite at a specified hydration time divided by the flexural toughness of the cement-silica fume composite after 28 days hydration, was systematically determined for both composite systems. The relative toughness was observed to decrease almost linearly as the hydration time increased from 28 days to 350 days for both composite systems, as shown in Figure 5. The decrease of the relative flexural toughness for the cement-silica fume-wollastonite system was observed to be larger than that for the cement-silica fume composite system. This indicates that prolonged hydration has modified the composite matrices and decreased their ability to absorb energy leading to fracture and failure.

Prolonged hydration was also observed to affect the ductility characteristics of both composite systems as described by peak-load defection and post-peak deflection. Peak-load deflection of the cement-silica fume system was observed to decrease gradually as the hydration time increased from 28 days to about 200 days and then remain essentially unchanged with further increase of the hydration time. Data for the cement-silica fume-wollastonite composite system also displayed a similar trend. The change of peak-load deflection with hydration time for the cement-silica fume and cement-silica fume-wollastonite composite system is shown in Figure 6. The post-peak deflection for the cement-silica fume composite system decreased linearly with hydration time as shown in Figure 7. No post-peak deflection was observed for the cement-silica fume system as fracture occurred abruptly.

The change of ductility characteristics in the cement-silica fume-wollastonite composite system with hydration time may be attributed to two possible causes. The primary cause appears to be embrittlement of the fibre-matrix interface possibly altering the failure mechanism to one dominated by fibre fracture as opposed to fibre pull-out. This is a well known behaviour in cement-glass fibre composite systems; it was not expected in wollastonite fibre reinforced systems. Secondly, a change of the intrinsic material properties (e.g. modulus of elasticity and hardness) of the wollastonite micro-fibres after prolonged exposure in the highly alkaline environment within the hydrated cement matrices might also possibly have an effect on toughness. Relatively pure natural wollastonite mineral containing few impurities is known to be stable and durable in an ordinary environment [11]. However, it is uncertain about the possibility of changes under an alkaline environment within the hydrated cement paste.
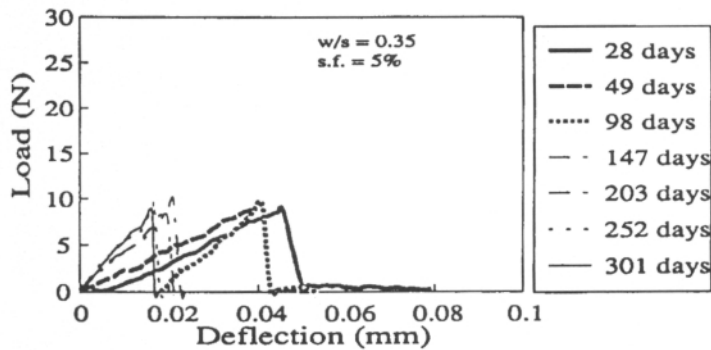


Figure 3. Load-deflection curves for the cement-silica fume composite mixture after hydration in calcium hydroxide solution for various periods.
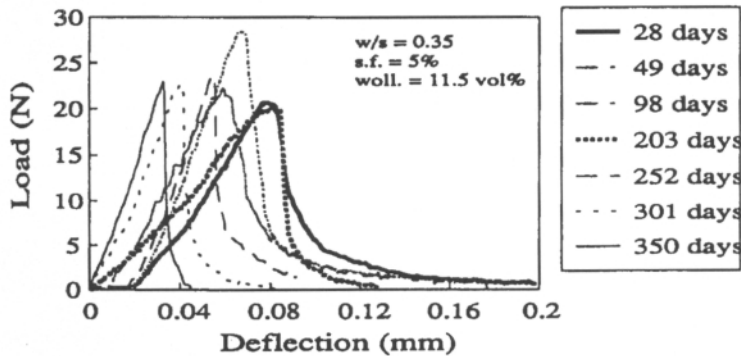


Figure 4. Load-deflection curves for the cement-silica fume-wollastonite composite mixture after hydration in calcium hydroxide solution for various periods.
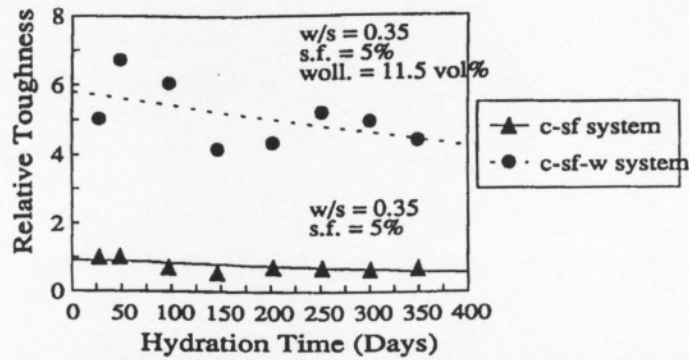
Figure 5. Variation of relative flexural toughness with hydration time in the cement-silica fume (c-sf) and cement-silica fume-wollastonite (c-sf-w) composite systems.
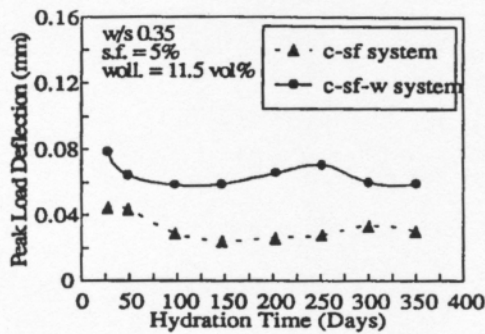


Figure 6. Variation of peak-load deflection with hydration time for the cement-silica fume (c-sf) and cement-silica fume-wollastonite (c-sf-w) composite system.
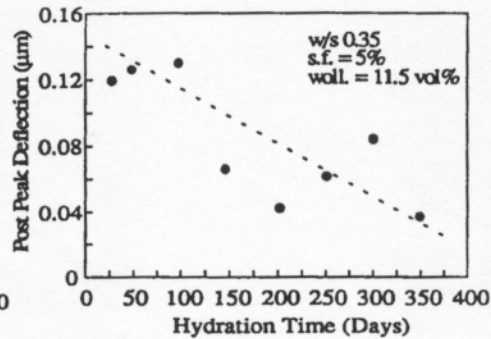
Figure 7. Variation of post-peak deflection for the cement-silica fume-wollastonite (c-sf-w) composite system.

[D] Total Porosity

The total porosity in the hydrated cement-silica fume and cement-silica fume-wollastonite composite matrices gradually decreased as the hydration time increased from 28 days to 350 days. In the present study, all specimens selected for porosity measurements had similar flexural strength values. It is assumed that wollastonite micro-fibres are non-porous and that the porosity is concentrated only in the cement paste matrix. Two specimens were measured and the average results are presented for all porosity determinations. The porosity results are discussed in terms of the method of measurement.

(1) Mercury Intrusion Porosimetry

Measurements showed that the total pore volume in the hydrated cement-silica fume matrices prepared with a water/cement+silica fume ratio of 0.35 after various periods of hydration in calcium hydroxide solution was in the range of 20.0% - 27.5%. The total pore volume appeared to decrease slightly as hydration time increased from 28 days to 350 days as shown in Figure 8. The total pore volume in the hydrated cement-silica fume-wollastonite composite matrices was observed in the range of 21.4% - 24.5%. The overall pore volume, however, appeared to remain essentially unchanged with increase of the hydration time as shown in Figure 9. The observation of a slightly lower total pore volume in the cement-silica fume-wollastonite composite system compared to the cement-silica fume system in the present study was found to be consistent with previous investigations [1,2].

(2) Helium Gas Pycnometry

The total porosity determined by the helium gas pycnometry was observed to be significantly lower than values determined by the mercury intrusion porosimetry. Measurements showed that the total pore volume in the hydrated cement-silica fume matrices was in the range of 5.2 % - 11.5%. There appears to be a systematic and gradual decrease of the total pore volume as the hydration time increases from 28 days to 350 days. The total pore volume in the cement-silica fume-wollastonite composite matrices after a similar period of hydration was in the range of 2.2% - 7.1%.

There also appears to be a systematic decrease of total porosity with increase of hydration time. The observation of a lower total pore volume in hydrated cement composite matrices determined by the helium gas pycnometry measurement is consistent with previous investigations [1, 2]. The observation of a decrease of pore volume with hydration time is an indication that more cement reaction products (such as CH, CS-H, and ettringite) were formed in the cement-silica fume matrices. Helium gas does not appear to infiltrate into all pore space. The variation of total pore volume versus hydration time for both composite systems determined by the helium gas pycnometry method is also shown in Figures 8 and 9.
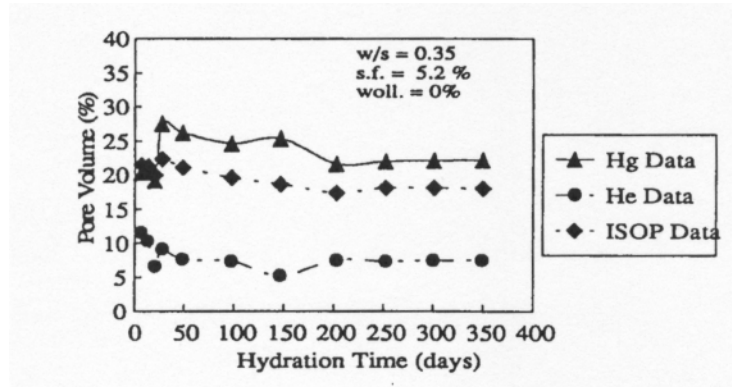


Figure 8. Variation of total porosity versus hydration time in the cement- silica fume composite systems.
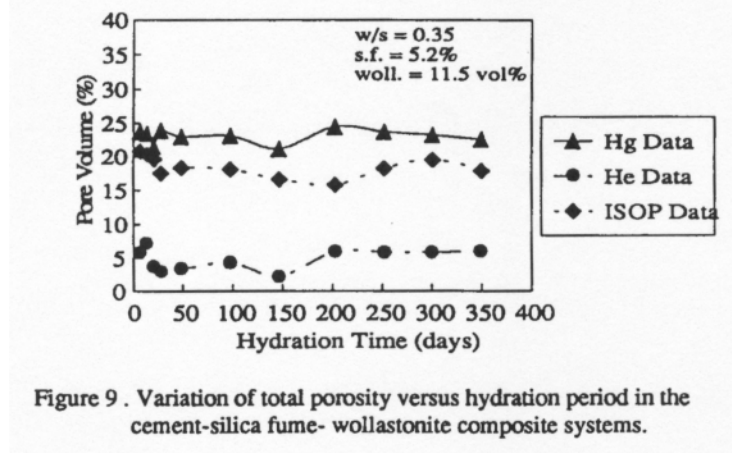


Figure 9 . Variation of total porosity versus hydration period in the cement-silica fume- wollastonite composite systems.

(3) Isopropyl Alcohol Saturation Measurement

The total porosity determined by the isopropyl alcohol saturation measurement was observed in the range of 17.5% - 22.4% for the cement-silica fume composite system and in the range of 15.8% - 20.8% for the cement-silica fume-wollastonite composite system. It is evident that these porosity values are also higher than those values determined by the helium gas pycnometry measurement for both composite systems. The total pore volume was also observed to decrease systematically as the hydration time increased. These results appear to support the postulation that more cement reaction products have been formed during the prolonged period of hydration thus affecting the infiltration process of isopropyl alcohol into all pore space in both composite systems. In the present investigation, the total pore volume determined by the isopropyl alcohol saturation method was also observed to be slightly lower than that determined by the mercury intrusion porosimetry method for both cement-silica fume and cement-silica fume-wollastonite composite systems. This observation was found to be consistent with the results observed in a previous investigation [3]. The variation of total pore volume versus hydration period determined by the isopropyl alcohol saturation method is also shown in Figures 8 and 9.

[E] Pore Size Distribution

The distribution of pore sizes for the cement-silica fume and cement-silica fume-wollastonite composite systems was observed to be slightly different. In the cement-silica fume composite system, the amount of coarse pores in the range of 1.00 - 0.10 µm was relatively small and remained essentially unchanged with hydration time. However, the volume of smaller pores in the range of 0.10-0.01 µm increased slightly as hydration time increased. The volume of very fine pores in the range of 0.01-0.001 µm varied slightly with hydration time. The curves of pore volume versus pore diameter at several ages are shown in Figure 10 for the cement-silica fume composite system and in Figure 11 for the cement-silica fume-wollastonite composite system. It is evident that the changes with hydration time are very small.
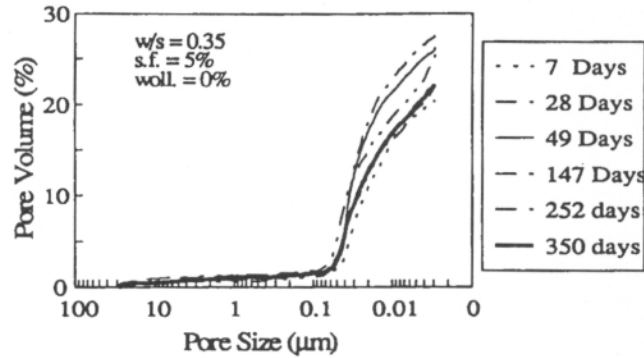


Figure 10. Curves of pore volume versus pore size for the cement-silica fume composite system
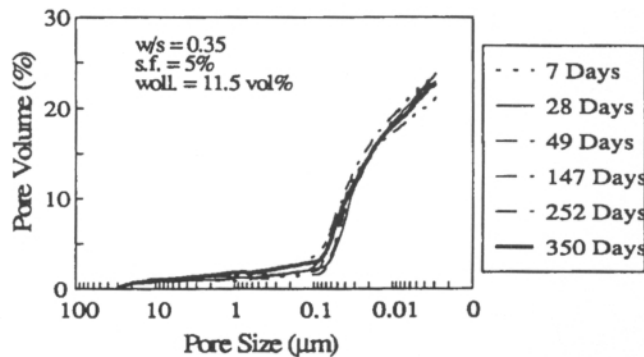


Figure 11. Curves of pore volume versus pore size for the cement-silica fume-wollastonite composite system

[G] Content of $Ca(OH)_2$

Processes associated with transition zone formation in the vicinity of inclusions such as sand particles or wollastonite micro-fibres could affect the deposition of reaction products (e.g. $Ca(OH)_2$, C-S-H, and ettringite) in this region. Pore structure in the pore size range of 0.5-0.1 µm is often affected by the quantity, size and nature of the inclusion. This can also affect the rate and extent of hydration, formation of $Ca(OH)_2$ and matrix permeability [12,13]. It is also known that silica fume reacts with $Ca(OH)_2$. The removal of $Ca(OH)_2$ from the matrix interface would therefore serve to decrease the permeability of the system. A reduction in porosity of the transition zone could also be a factor responsible for the observed decrease in ductility at later ages.

Variation of the content of $Ca(OH)_2$ with time was observed to be different for the hydrated cement-silica fume system and the hydrated cement-silica fume-wollastonite system. The content of $Ca(OH)_2$ of the cement-silica fume composite system increased from about 3.1% to 7.6% (based on ignited weight) as hydration time increased from 28 days to 150 days. The content then gradually decreased to about 5.4% as the hydration time further increased to 350 days. It is suggested that the decrease may indicate a change in the nature of the C-S-H. The silica fume content is relatively low and it should be completely reacted with CH prior to 150 days. In the cement-silica fume-wollastonite composite system, the unadjusted content of $Ca(OH)_2$ was in range of 3.5% - 4.8% after a similar period of hydration. These results were obtained from a hydrated composite matrix made up with 88.5% by volume of cement-silica fume and 11.5% by

volume of wollastonite micro-fibres. As a result, a correction for the mass difference in the composite system is required. The corrected content of $Ca(OH)_2$ is in the range of 4.1% - 5.5%. In the cement-silica fume-wollastonite composite system, the maximum content of $Ca(OH)_2$ was observed after a hydration time of 28 days. As the hydration time increased to 350 days, the content of $Ca(OH)_2$ gradually decreased. The variation of the content of $Ca(OH)_2$ with hydration time for the two systems is shown in Figure 12. It is likely that most of the $Ca(OH)_2$ in the hydrated cement matrices has been consumed by reaction with the silica fume. The decrease of the content of $Ca(OH)_2$ with increase of hydration time was observed to be consistent with a previous study of hydration reactions in Portland cement-silica fume blends [14]. It appears that the presence of wollastonite micro-fibres may affect the type of C-S-H deposited and it may also accelerate its formation.
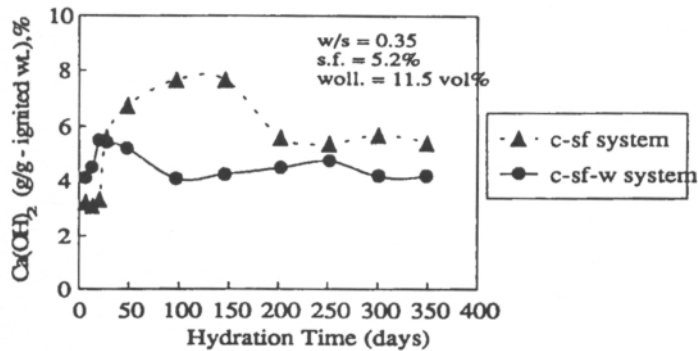


Figure 12. Variation of $Ca(OH)_2$ content versus hydration time in the cement-silica (c-sf) composite system and the cement-silica fume-wollastonite(c-sf-w) composite system.

Conclusions

The experimental results obtained in this investigation appear to support the following conclusions.

1. Natural wollastonite micro-fibres are relatively stable in cement composite systems and apparently unaffected by exposure to prolonged hydration in calcium hydroxide solution.

2. The flexural strength of the cement-silica fume matrices reinforced with 11.5% by volume of wollastonite micro-fibres is unchanged and unaffected by prolonged hydration in calcium hydroxide solution up to one year.

3. The flexural toughness and post-peak deflection of the cement-silica fume and cement-silica fume-wollastonite composites decreases linearly with increase of hydration time.

4. The total porosity in the hydrated cement-silica fume and cement-silica fume-wollastonite composite systems determined by mercury intrusion, helium gas pycnometry and isopropyl alcohol saturation methods decreases slightly with increase of hydration time.

5. Pore volume in the 1.0 - 0.001 µm region of the cement-silica fume and cement-silica fume-wollastonite composite systems appears to be unchanged with increase of hydration time.

6. Wollastonite micro-fibres may affect the stoichiometry and the rate of formation of C-S-H.

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