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Effect of applied hydrostatic stress on the hydration of Portland cement and C3S

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Effect of Applied Hydrostatic Stress on the Hydration of Portland Cement and C_3S

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Synopsis

The hydration behavior of Portland cement and C_3S with and without stress applied were studied at water to solid (w/s) ratios of 0.35 and 0.50. A greater degree of hydration and denser microstructure were obtained with hydration under stress at an early stage of the hydration process, normally within 48 hours. This observation was confirmed by the determination and comparison of CH content, TGA results, surface area and pore-size distribution of samples hydrated with and without applied stress. Environmental SEM examination provided the evidence that micro-cracking occurred only in samples hydrated under applied stress. It is suggested that this facilitated the migration of water into the protective layers (around cement grains) that form in the early stages of hydration accelerating the reaction with the unreacted cores of the particles.

Keywords: Stress, degree of hydration

1. Introduction

The hydration and hydration kinetics of Portland cement and C_3S have been extensively studied [1-4]. Numerous techniques have been used to determine the fraction of anhydrous materials including spectroscopic and thermoanalytical methods [5]. The hydration reactions reported are however generally monitored in a stress free state, i.e. no external and continuously applied stress is maintained during the process. In practice, hydration often takes place in concrete that is in a stressed state. It is, therefore, important to have an accurate estimation of the influence of stress on reaction kinetics and the development of time-dependent engineering properties. For example, the prediction of early age creep of concrete requires an estimate of the time dependence of the degree of hydration [6].

It has recently been suggested, for cement paste and C_3S paste specimens, that the extent of reaction of the cement and C_3S as a function of time may be significantly different if hydration occurs under a uniaxially applied stress [7]. The objective of this work was to confirm (in a systematic manner) that applied stress affects the hydration kinetics (especially at early ages) of Portland cement and C_3S. The work in reference [7] was extended to a hydrostatic stress condition. Experiments were designed to permit hydration of paste specimens (kept continuously saturated) subjected to a hydrostatic stress. The extent of hydration was monitored using thermal analytical methods. These were supported by pore-size distribution and surface area determinations. ESEM examination was conducted on wet specimens to ensure that the observations were compatible with the objectives of the study.

2. Experimental

2.1 Materials

2.1.1 Cement

The cement used for this study was Canadian Standards type 10 Portland cement produced by Lafarge. It has the following composition (in percentage): SiO_2 (20.72); Al_2O_3 (5.87); Fe_2O_3 (3.07); CaO (62.66); MgO (3.46); SO_3 (2.18) and free lime (0.24). The Bogue composition is as follows: C_3S (46.5); C_2S (24.6); C_3A (10.4) and C_4AF (8.3). The fineness of the cement is 340 m^2/kg.

2.1.2 C_3S

The C_3S used was supplied by the American Cement Technical Center. It has a nitrogen surface area of 4000 cm^2/g.

2.2 Specimen Preparation

Cement or C_3S pastes were prepared by mixing the solids with de-aired distilled water at water-solids ratios of 0.35 and 0.50. A standard mixer was used to mix the pastes which were then vibrated for 1 min. The pastes were then cast in hollow cylinders, 25 mm in diameter by 250 mm long. The cylinders were slowly rotated while the paste hardened in order to avoid bleeding and to produce homogeneous products. Specimens were demoulded at selected hydration times and cut into 25 mm diameter by 50 mm long cylinders. The cut specimens were divided into two groups: one group (referred to as the loaded group) was sealed in rubber membranes which contained a small excess of saturated lime water and placed in a floating piston accumulator chamber and subjected

to an applied hydrostatic stress of 6.8 MPa at (22 ± 2)°C. The other group (referred to as the unloaded group) was stored in lime-saturated water with no applied stress at the same temperature range.

One mm thick slices were cut from both the loaded group and unloaded group of specimens at selected hydration times and dried under vacuum at 105$^{\text{o}}$C for 3 hours. The dried samples were stored in a vacuum desiccator until further use in order to minimize carbonation. All the data reported from experimental measurements (described later) are the mean values of triplicate determinations.

2.3 The Floating Piston Accumulator Apparatus

An accumulator device (illustrated in Figure 1) was used to apply a continuous hydrostatic stress to the hydrating pastes in this investigation. The procedure is briefly described as follows. The top end of the accumulator is opened and the sample is placed into the water reservoir. The accumulator is sealed and the valve (V1) is opened allowing entry of compressed nitrogen gas. The stress regulator is adjusted slowly allowing the floating piston to move upwards against the water. The flow of nitrogen gas is further adjusted until the designed stress is obtained. The volume capacity of the accumulator is 2000 ml and the stress capacity is 27 MPa.

2.4 Specimen Characterization

2.4.1 Conduction Calorimetry

Calorimetric measurements were performed to determine the heat evolution behavior during the normal hydration of pastes prepared at water/solids = 0.50 and 0.35. Samples (~10g) were hydrated in plastic containers to allow thorough mixing of the cement or C_3S with water in the calorimeter cell. The calorimeter was immersed in a thermostatically controlled water bath to ensure a constant temperature with an accuracy of $\pm 0.1^oC$ at 22^oC. The heat evolution curves were recorded for 96 hours after mixing.

2.4.2   Thermogravimetric Analysis (TGA)

A Dupont 951 Thermal Analyzer placed in an environmentally controlled chamber was used for the tests. The samples were heated from room temperature to 1050^oC at the rate of $10^oC/min$ in a N_2 atmosphere. The degree of hydration at various stages during the early hydration process was determined for loaded and unloaded samples.

In this study, the degree of hydration of cement paste (α, %) was estimated in the following two ways:

i) the determination of $Ca(OH)_2$ content based on mass of paste at 105^oC. The degree of hydration is expressed as:

$$\alpha_{CH,}\ \% = \frac{Mass\ \ of\ \ Ca(OH)_2}{Mass\ \ of\ Sample\ \ at\ \ 105^oC} \times 100,\ \% \qquad (1)$$

ii) Determination of the ratio of non-evaporable water content of the cement paste at time t to the non-evaporable water content at the complete hydration of the cement paste at $t =$

∞ [8]. For a typical Portland cement paste, the latter is generally about 25% of the mass of the cement. Therefore, the degree of hydration can be expressed as

$$\alpha_{H2O,\ \%} = \frac{W_n(t)}{0.25 * W} \tag{2}$$

where, $W_n(t)$ is, at a specific hydration time t, the bound water of the paste lost between 105^oC and 1050^oC.

W is the sample mass of cement.

2.4.3   Differential Thermal Analysis (DTA)

Differential thermal measurements were obtained using a 910-DuPont thermal analyzer. The unit consists of a furnace capable of attaining a temperature of 1600^oC and utilizes platinum cups for housing both the sample and reference material. The degree of hydration for C_3S was expressed in terms of the $Ca(OH)_2$ content and estimated by equation (1). Determination of the ratio of the unreacted C_3S to the total amount of C_3S was also made. The method for estimating the amount of unreacted C_3S during hydration utilizes the endothermic transition for C_3S at about 915^oC. It is described in detail in reference [9].

DTA curves were obtained (heating rate, $20^oC/min$) by heating from 750 to 1050^oC, followed by cooling to 750^oC and reheating to 1050^oC. This was designed to minimized the interfering effects at around 915^oC which may be due to decarbonation, crystallization of β-wollastonite and the dehydration effect of calcium silicate hydrate [9]. The data from the second heating was used to calculate the amount of unhydrated C_3S.

The degree of hydration of C_3S, therefore, can be expressed as:

$$\alpha_{C3S}, \% = 100 - \frac{At}{Ac_3s} \times 100 \tag{3}$$

where, At is the endothermic peak area at 915^oC of C_3S hydrated at specific time t.

Ac_3s is the endothermic peak area at 915^oC of C_3S before hydration.

2.4.4. Environmental Scanning Electronic Microscopy (ESEM)

An ESEM permits the examination of wet specimens. This is particularly important in studies of hydration kinetics. The ElectroScan E-3 ESEM, manufactured by ElectroScan Corporation, was used to provide the microstructural information of cement pastes and C_3S samples in this research. Samples were kept in saturated lime solution until they were examined. They were measured from the container, damp dried with tissue paper, fractured and fixed in sample holders. Secondary electron images of the fracture surfaces were recorded by a digital recorder.

2.4.5. Pore-size Distribution

Mercury Intrusion Porosimetry (MIP), although it has some limitations, has been used in cement research to evaluate pore-size distribution for many years [10,11]. In the present study, the major purpose was to compare the difference between loaded and unloaded samples. Pore-size distributions were obtained using a Quantachrome Autoscan-33 Porosimeter with the maximum pressure of 224 MPa. The sample was initially dried under vacuum at 105^oC for 3 hours. About 1g of dried sample was used for each test run.

2.4.6 Nitrogen Surface Area (BET)

Vacuum dried samples were ground and outgassed at $105^{\circ}C$ for 20 mins before the surface area measurement was carried out. The measurements were made with a Quantasorb surface area analyzer using N_2 gas adsorption at $-180^{\circ}C$ using a flowing mixture of nitrogen and helium, the latter being the carrier gas. The amount of nitrogen liberated upon warming the sample is measured.

3. Results and Discussion

3.1 Heat Evolution

The rate of heat evolution and the total heat versus time curves of cement and C_3S pastes prepared at water/solids = 0.5 and 0.35 are presented in Figure 2. These serve as reference curves for the starting materials. For cement pastes at water/cement = 0.50 and 0.35, the profiles of the heat evolution curves, Figure2 (a), are similar to the typical curves of Portland cement pastes published elsewhere [1]. The initial peak is attributed to a combination of exothermic wetting and the early stage reaction, resulting in a surface coating of reaction products on the cement grains. Following the induction period, the maximum rate of heat evolution is reached at about 10 hours. The main peak corresponds to formation of the main products C-S-H and CH. A distinct shoulder peak at 20 to 30 hours, in the case of the water/cement = 0.35 paste, is associated with the conversion of AFt to AFm. SEM confirms the formation of AFt in both cases. The total heats developed after 96 hours is 95 Cal/g for water/cement = 0.50 and 80 Cal/g for water/cement = 0.35, respectively.

For C_3S pastes at water/solids = 0.50 and 0.35, Figure 2(b), the heat evolution behavior is similar to that for cement pastes except i) there is no AFt formation; ii) the maximum of the heat evolution rate is observed at about 12 hours; iii) the total values of heat developed are 58 and 66 cal/g for water/solids = 0.50 and 0.35 respectively. The heat evolution curves were used to select times for measurement of the hydration parameters. The paste specimens were demoulded and subjected to an applied stress after 8, 10, 15, 18 and 20 hours.

3.2 Hydration of C_3S

Figure 3 shows the degree of hydration of C_3S expressed as α_{CH}, % and α_{C3S}, %: the water/solids = 0.50 and the stress was applied after 18 hours. It clearly shows that stress results in an increase in the degree of hydration within the first 48 hours. Both methods show similar trends for the change of hydration degree. Examination of Figure 3(b) indicates that pressure promotes the formation of more crystalline CH. This is interpreted as resulting in more extensive hydration. The character of the curves in Figure 3(a) and 3(b) is similar although the differences between loaded and unloaded are less pronounced in Figure 3(b). The initial point is the zero reference for time of loading. An immediate increase following this would be expected if microcracking allowed water to access the grains. This effect exists but it is small in Figure 3(b). ESEM images provided evidence that microcracks formed only in the loaded samples, see Figure 4. As there was no pre-preparation involving drying before the ESEM examination and the experiment was not performed under high vacuum, the presence of microcracks is attributed to the applied stress. The difference in the curves does not continue to increase after 48h but instead a

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catching up occurs. A possible explanation may involve the variance in composition with time of the outlayer of reaction products surrounding the grain. At later times the calcium hydroxide – C-S-H ratio in the layer may change, render it less permeable and diminish the influence of microcracks.

The difference in the degree of hydration between the loaded and unloaded samples at water/solids = 0.35, Figure 5, is greater compared to the difference at water/solids = 0.50. The difference between the loaded and unloaded samples is sustained until 96 hours. This observation suggests that at a lower water-solids ratio, the applied stress is more effective in enhancing the accessibility of water to cement grains through existing hydrated products or microcracks. This leads to an increase in the rate of hydration of the unreacted cement particles and the degree of hydration.

The authors discount the possibility that microcracking is a result of depressurization as opposed to pressurization. There are several arguments for this. It has been reported that microcracking in cement paste will occur while the paste is subjected to a sustained uniaxial load [12]. This can occur at the interface between an unhydrated cement particle and the hydrated cement matrix. The interface provides a discontinuity when microcracking takes place. The microstructure of pastes loaded at very early age is weak and points of contact between particles of hydration product are particularly susceptible to displacement and microcracking. It is noteworthy that the effects of a uniaxially sustained load on the reaction kinetics in cement paste were observed to be similar to those due to hydrostatic stress described here [7]. This suggests that microcracks are generated during pressurization for both types of stress application.

Other factors support the view that pressurization has the dominant effect on the reaction kinetics. Water under pressure would permeate more rapidly through layers of hydration product to the reaction site (cement grain). The effect would depend on the degree of hydration and the pore structure of these layers. The overall free energy of the cement-water reaction would also change due to the pressure. Further a short depressurization period would be unlikely to have an immediate effect on the measured indicators of the extent of hydration.

3.3. Hydration of cement paste at water/cement = 0.50

3.3.1. Stress applied after hydration for 18 hours

Degree of hydration

The degree of hydration versus time for cement paste (expressed as α_{CH} % and α_{H2O}%) at water/cement = 0.50 with and without applied stress is presented in Figure 6. The stress was applied after 18 hours hydration. It indicates that there is a difference of degree of hydration between the loaded and unloaded samples. The loading increases the degree of hydration from 18 hours, when the stress was applied, up to 48 hours. Microcracking was detected in loaded specimens (see following section on ESEM).

Nitrogen surface area

The nitrogen surface area was measured by the BET method. The results for cement paste, water/cement = 0.50 are shown as Figure 7. The value of surface area of unloaded sample is higher (up to 48 hours) than that of loaded sample while values for both increase with time. It might be expected that the loaded samples would have a higher

surface area due to a higher degree of hydration. However, as a result of applied stress, an increased degree of consolidation or layering of the C-S-H (a form of aging) could decrease the accessibility of the surface for nitrogen adsorption. After 48 hours, the values of the surface areas decrease with the time. This observation is not fully understood. However, this could be due to hydration induced aging as opposed to load induced aging. Differences between loaded and unloaded specimens remain. Odler [13] explains this in general terms by ascribing it to the limited accessibility of the pore system to N_2.

Pore-size distribution

Figure 8 gives the pore-size distribution of cement pastes (water/cement = 0.50, stress applied after 18 hours) at selected hydration times, 24h, 42h, 48h and 72h. For each time, it is noted that i)the majority of the pores is in the range of 1 ~ 0.01 um; ii) the loaded sample has a lower normalized intruded volume compared with the unloaded sample over most of the pore-size range. The loaded samples may be denser than the unloaded samples. This observation is consistent with the surface area measurements (see Figure 7); iii) the difference between loaded and unloaded specimens is greater in the larger pore-size range, 100 ~ 1 um, and is gradually reduced until the maximum intrusion pressure is reached. Figure 8 shows that the difference between loaded and unloaded samples increases gradually from 24 hours to 48 hours, but is reduced at 72 hours. This observation is consistent with the changes in degree of hydration (see Figure 6).

Environmental Scanning Electron Microscopy

Similar to the observation for the C_3S hydration, microcracks were only found in the loaded samples (see Figure 9). Microcrack formation due to applied stress is a possible reason for an increase in degree of hydration in the loaded samples.

3.3.2 Stress applied at variable hydration times

Figure 10 shows the change in degree of hydration (α_{CH} %) for cement paste, water/cement = 0.50 at variable hydration times, eg. 8, 10, 15 and 20 hours. Three stages were observed: i) at 8 and 10 hours, the degree of hydration versus time curves of loaded and unloaded have the similar profiles. At this early stage, the water can easily migrate to react with cement grains. The protective layer thickens. Only a small difference in the curves is observed between loaded and unloaded samples; ii) at 15 and 18 hours, the loaded samples exhibit a larger degree of hydration than the unloaded samples. During this stage, the cement paste is getting harder and water migration through the bulk paste is slowed. However, as ESEM images show, microcracks form in the loaded samples. Water can therefore be transported through the protective layer more easily leading to the higher degree of hydration; iii) at 20 hours, hydration and layering (aging) effects are sufficiently advanced and the paste is possibly strong enough to resist microcracking. The loaded and unloaded samples have a similar degree of hydration time response, (see Figure 10).

3.4. Hydration of cement paste at water/cement = 0.35

The results obtained at w/c = 0.35 are similar to those at water/cement = 0.50 (see Figure 11). When stress was applied at 10 and 20 hours, the changes of the degree of hydration for the loaded and unloaded samples are similar. However, there is a notable difference when stress was applied at 18 hours: the loaded sample has a higher degree of hydration value up to 42 hours. The reason for this is not clear. However the following explanation is offered. The layer of hydration products that surrounds the anhydrous cement grains is porous and primarily a mixture of calcium hydroxide and C-S-H. Calcium hydroxide crystals or inclusion can promote stress concentrations and microcracking in regions throughout the layer. The interfacial regions between calcium hydroxide and C-S-H are also paths that facilitate mass transport. It appears that there may be an optimum layer composition at 15-18 hours that produces the observed effects. At later times the proportion of calcium hydroxide to C-S-H changes in the layer and may be responsible for minimizing changes in reaction kinetics.

4. Conclusions

i)   Hydration of Portland cement and C_3S under an applied hydrostatic stress of up to 6.80 MPa has a significant effect on the extent of hydration generally resulting in increasing value of the degree of hydration.

ii)   The higher degree of hydration is dependent on the time of application of the applied stress after mixing. The effect generally occurs if the application of stress takes place at an early stage of hydration for both Portland cement and C_3S hydration and normally occurs within the first 48 hours of hydration.

iii) The higher degree of hydration in the loaded samples is attributed to the formation of microcracks, as observed in the ESEM images.

iv) Applied stress during hydration leads to decreases in nitrogen surface area compared to hydration under normal conditions. Load induced aging effects (e.g. C-S-H layering and consolidation) and hydration induced aging may be responsible for the decreases in nitrogen surface area.

Acknowledgment

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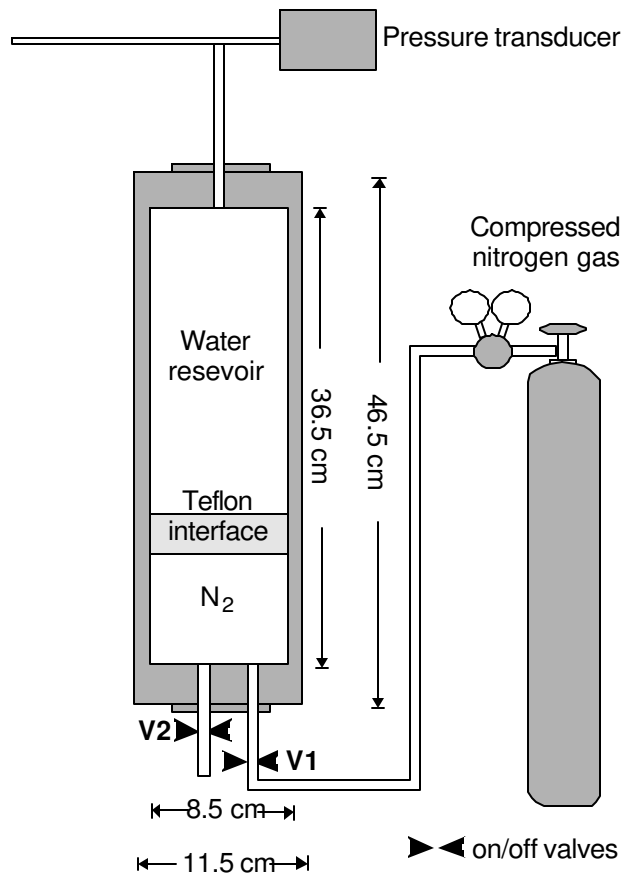
Figure Captions

Figure1   Floating piston accumulator used for application of hydrostatic stress to hydrating cement and C_3S pastes.

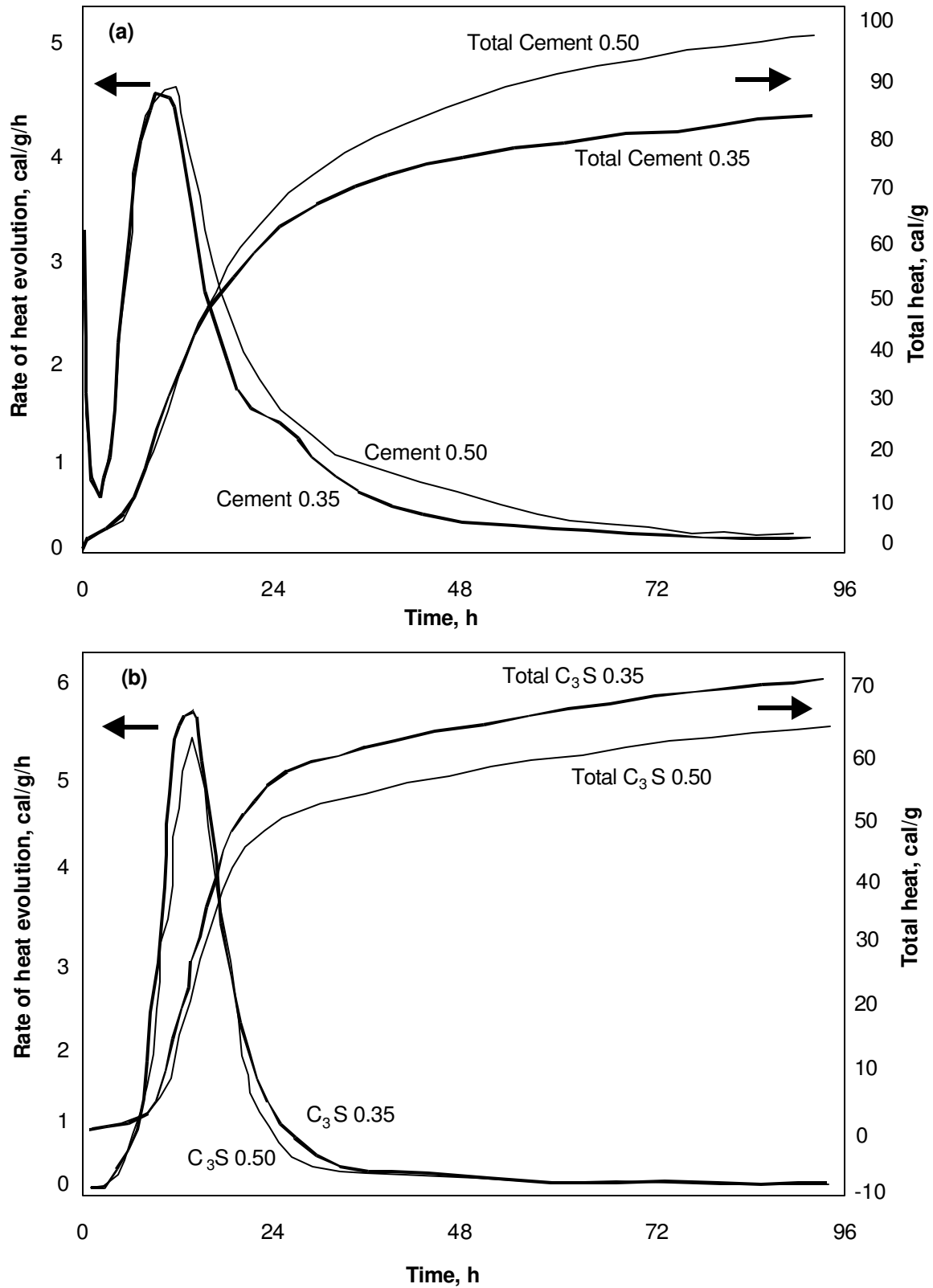Figure2.   Cement and C_3S paste: rate of heat evolution and total heat output at water/solids = 0.50 and 0.35.

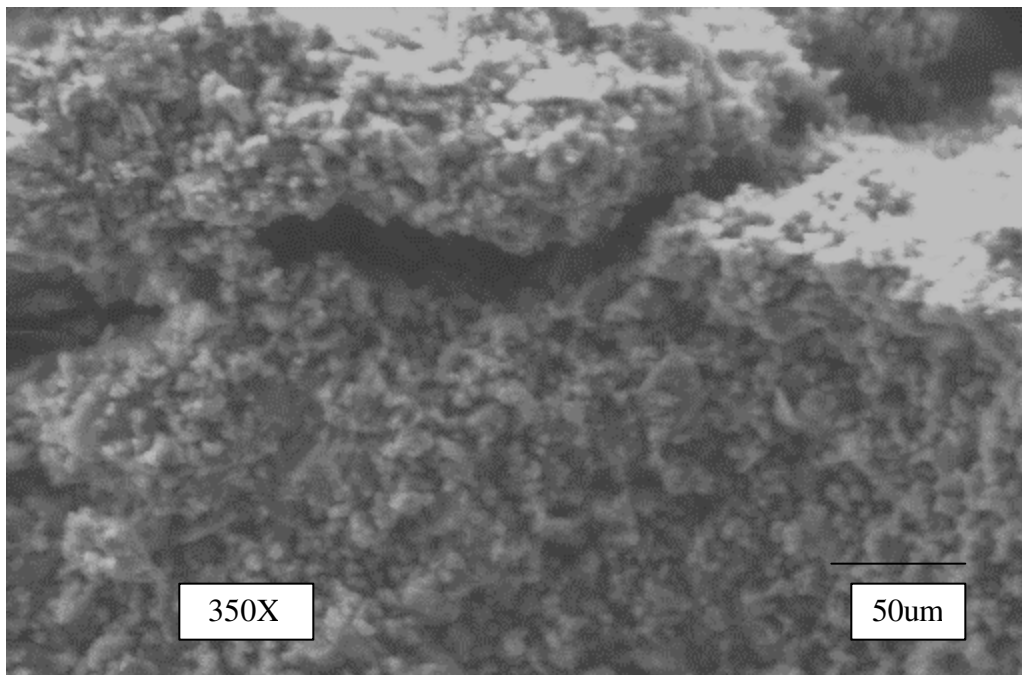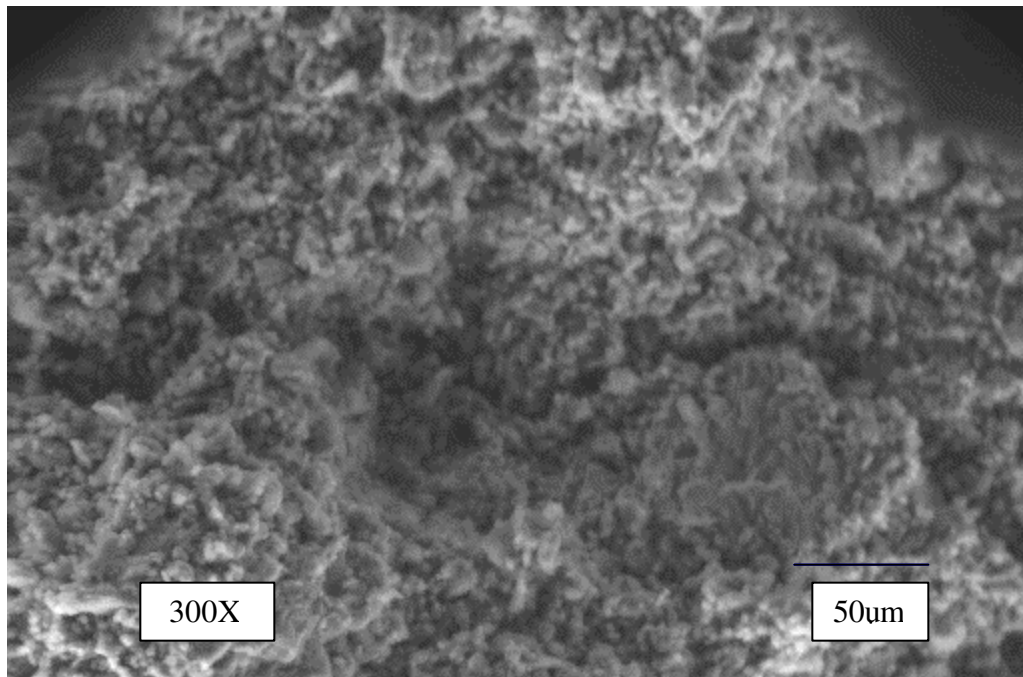Figure3   Degree of hydration of C_3S: water/solids = 0.50, stress (6.8 MPa) applied after 18 hour hydration.

Figure4   ESEM image of C_3S paste: water/solids = 0.50, stress (6.8 MPa) applied after 18 hour hydration.
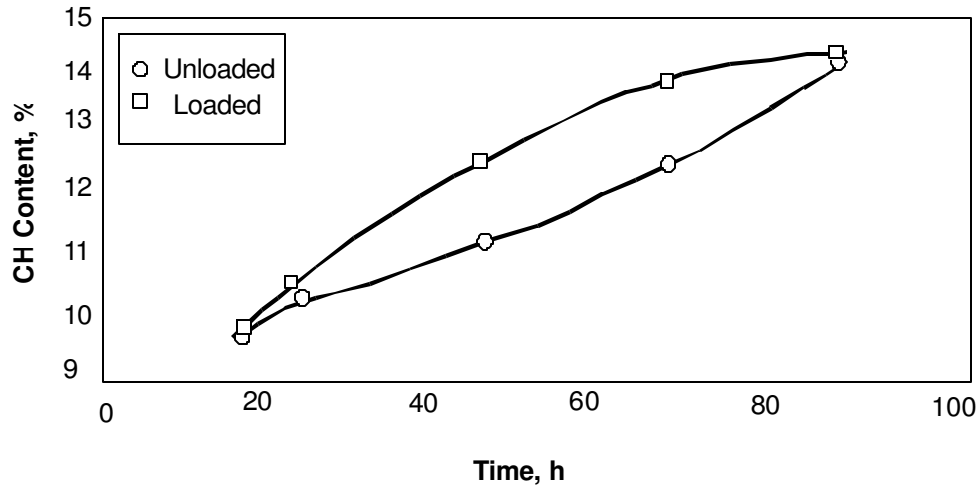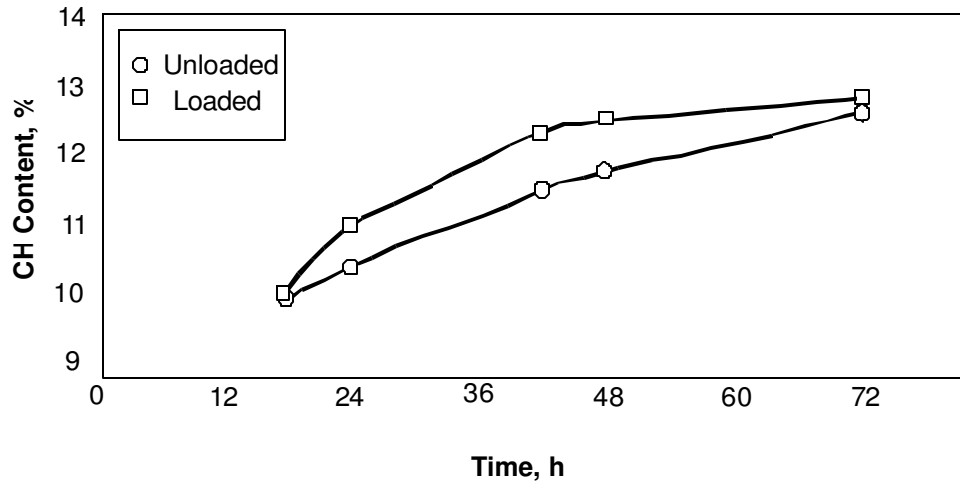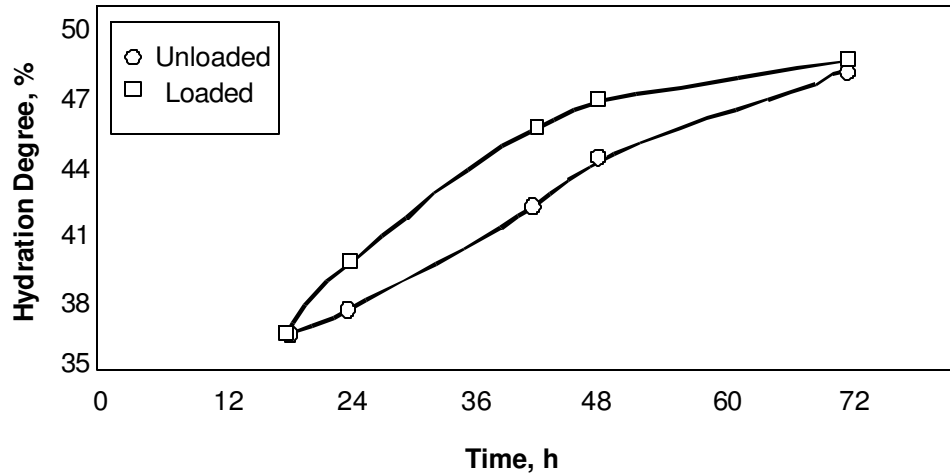
Figure5   Degree of hydration α_{CH}, % of C_3S: water/solids = 0.35, stress (6.8 MPa) applied after 18 hour hydration.

Figure6   Degree of hydration of hardening cement paste: water/cement = 0.50, stress (6.8 Mpa) applied after 18 hour hydration.

Figure7   Surface area of hardening cement paste: water/cement = 0.50, stress (6.8 MPa) applied after 18 hour hydration.

Figure8   Pore-size distribution of cement paste: water/cement = 0.50, stress (6.8 MPa) applied after 18 hours hydration.

Figure9   ESEM image of cement paste: water/cement = 0.50, stress (6.8 MPa) applied after 18 hour hydration.

Figure10   Degree of hydration, α_{CH}, %, of hardening cement paste: water/cement = 0.50, stress (6.8 MPa) applied at selected times.

Figure11   Degree of hydration α_{CH}, % of hardening cement paste: water/cement = 0.35, stress (6.8 MPa) applied at selected times.

Volume capacity: 2000 ml
Pressure capacity: up to 27 MPa

Figure 1. **Floating piston accumulator used for application of hydrostatic stress to hydrating cement and C$_3$S pastes.**

Figure 2. Cement and C_3S paste: rate of heat evolution and total heat output at water/solids = 0.50 and 0.35.
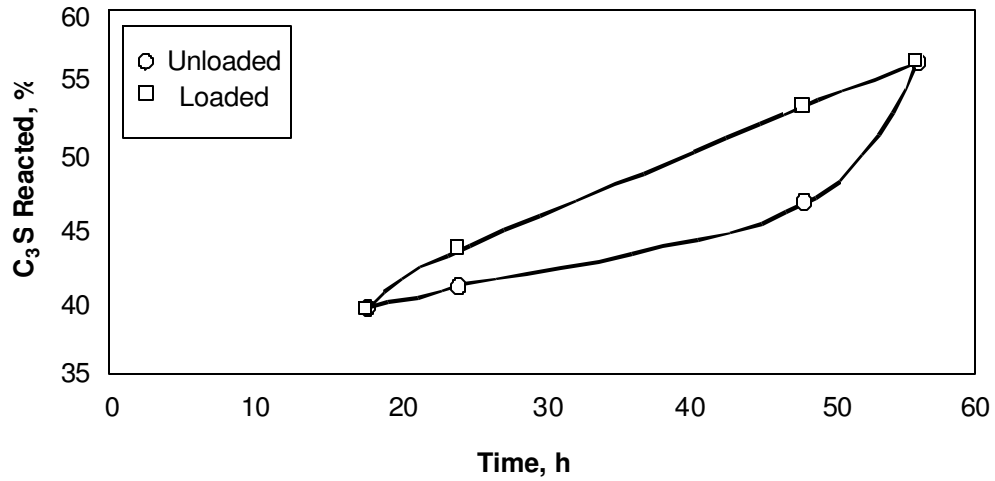
(a) degree of hydration, α_{C_3S}%
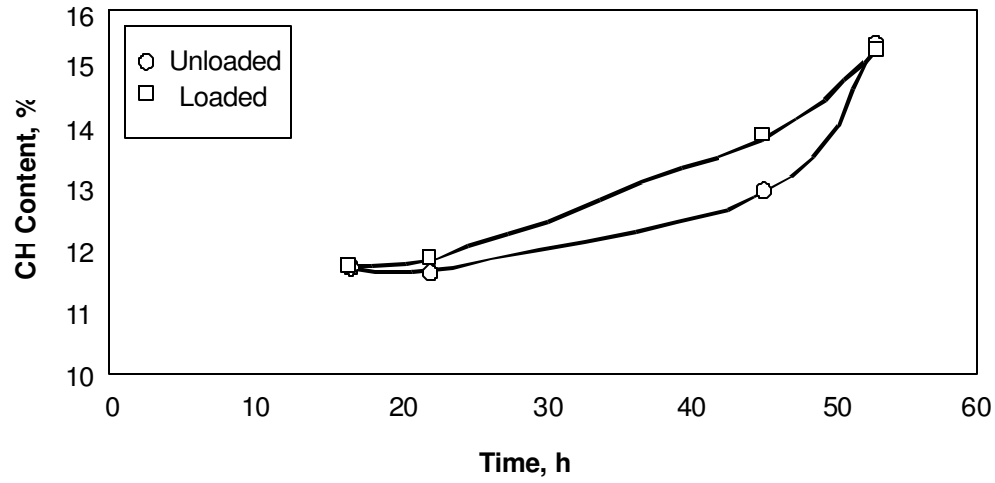
(b) degree of hydration, α_{CH}%

Figure 3. Degree of hydration of C_3S: water/solids = 0.50, stress (6.8 MPa) applied after 18 hour hydration

Figure 4. ESEM image of C_3S paste: water/solids = 0.50, stress (6.8 MPa) applied after 18 hour hydration.

Figure 5.   Degree of hydration α_{CH}, % of C_3S: water/solids = 0.35, stress (6.8 MPa) applied after 18 hour hydration.

(a) degree of hydration, α_{CH}%

(b) degree of hydration, α_{H_2O}%

Figure 6.   Degree of hydration of hardening cement paste: water/cement = 0.50, stress (6.8 Mpa) applied after 18 hour hydration.

Figure 7. Surface area of hardening cement paste: water/cement = 0.50, stress (6.8 MPa) applied after 18 hour hydration.

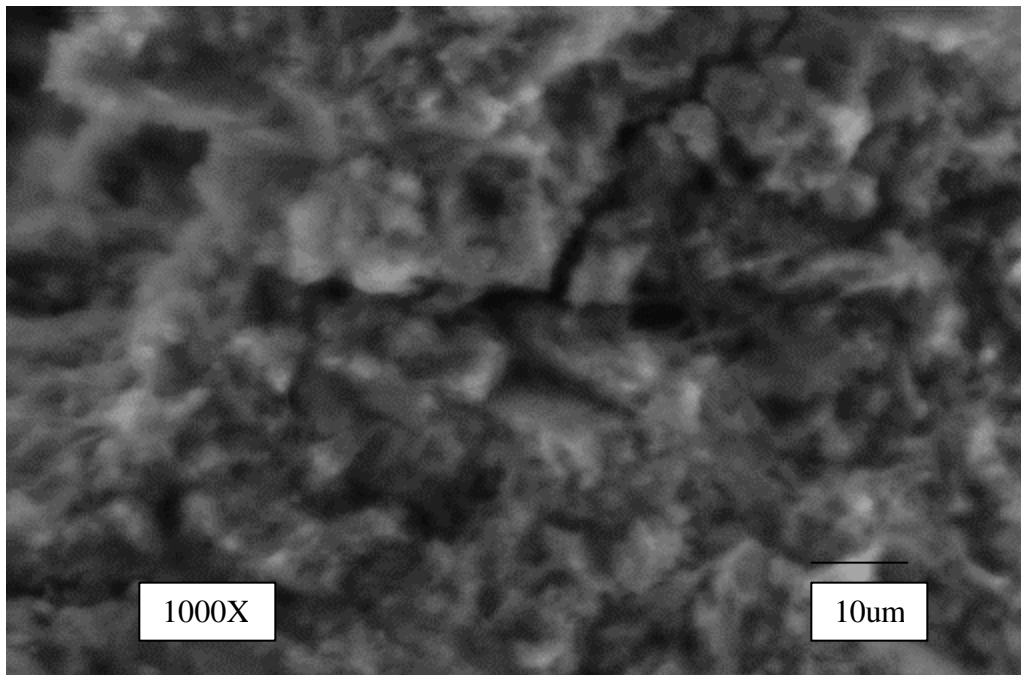**Figure 8.**   **Pore-size distribution of cement paste: water/cement = 0.50, stress (6.8 MPa) applied after 18 hours** hydration.
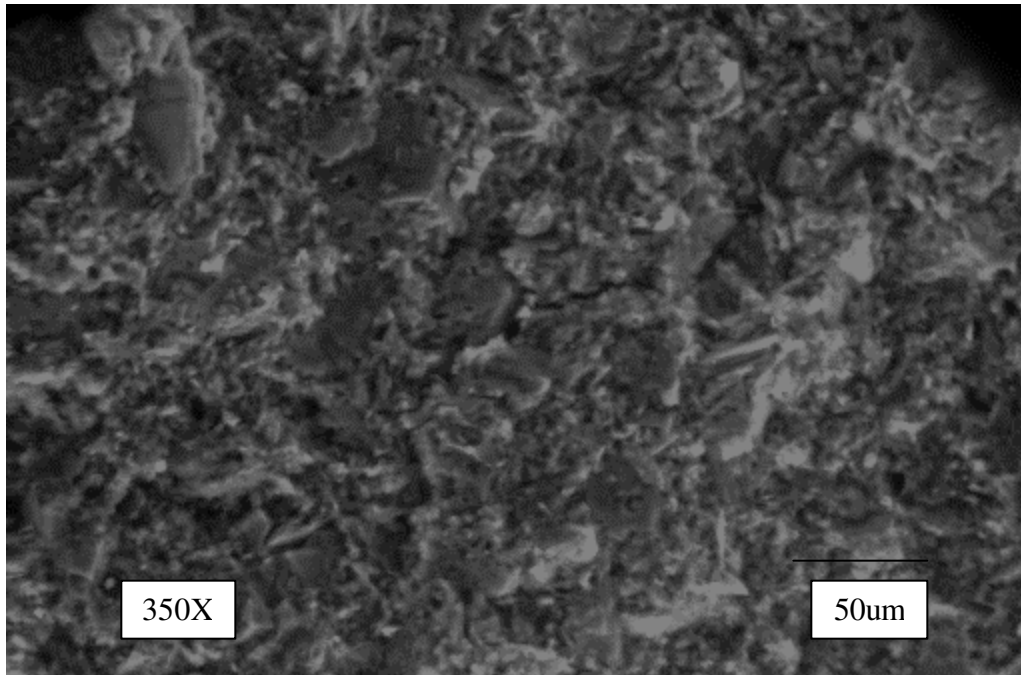
Figure 9. ESEM image of cement paste: water/cement = 0.50, stress (6.8 MPa) applied after 18 hour hydration
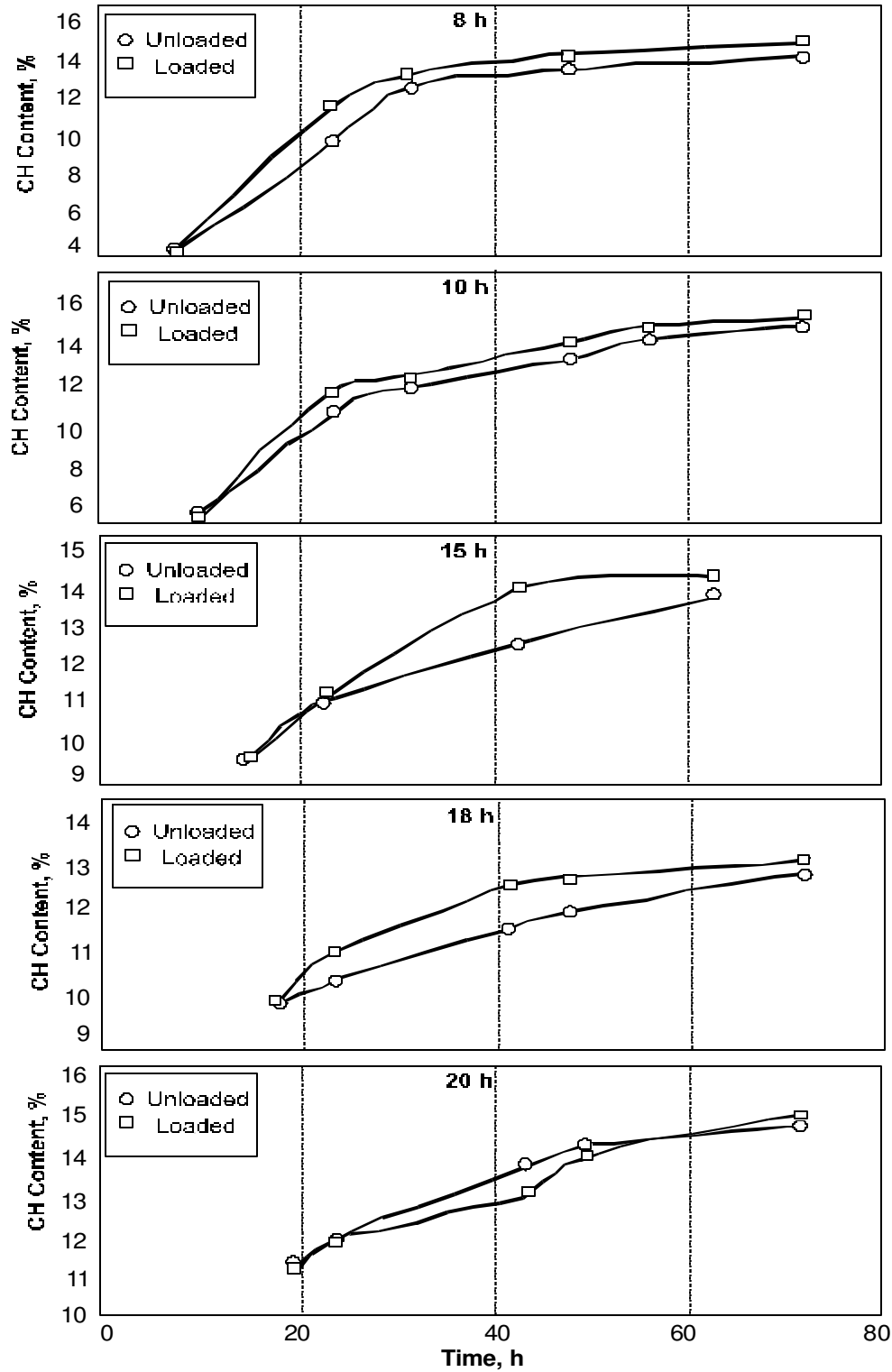
Figure 10.   Degree of hydration, α_{CH}, %, of hardening cement paste: water/cement = 0.50, stress (6.8 MPa) applied at selected times.
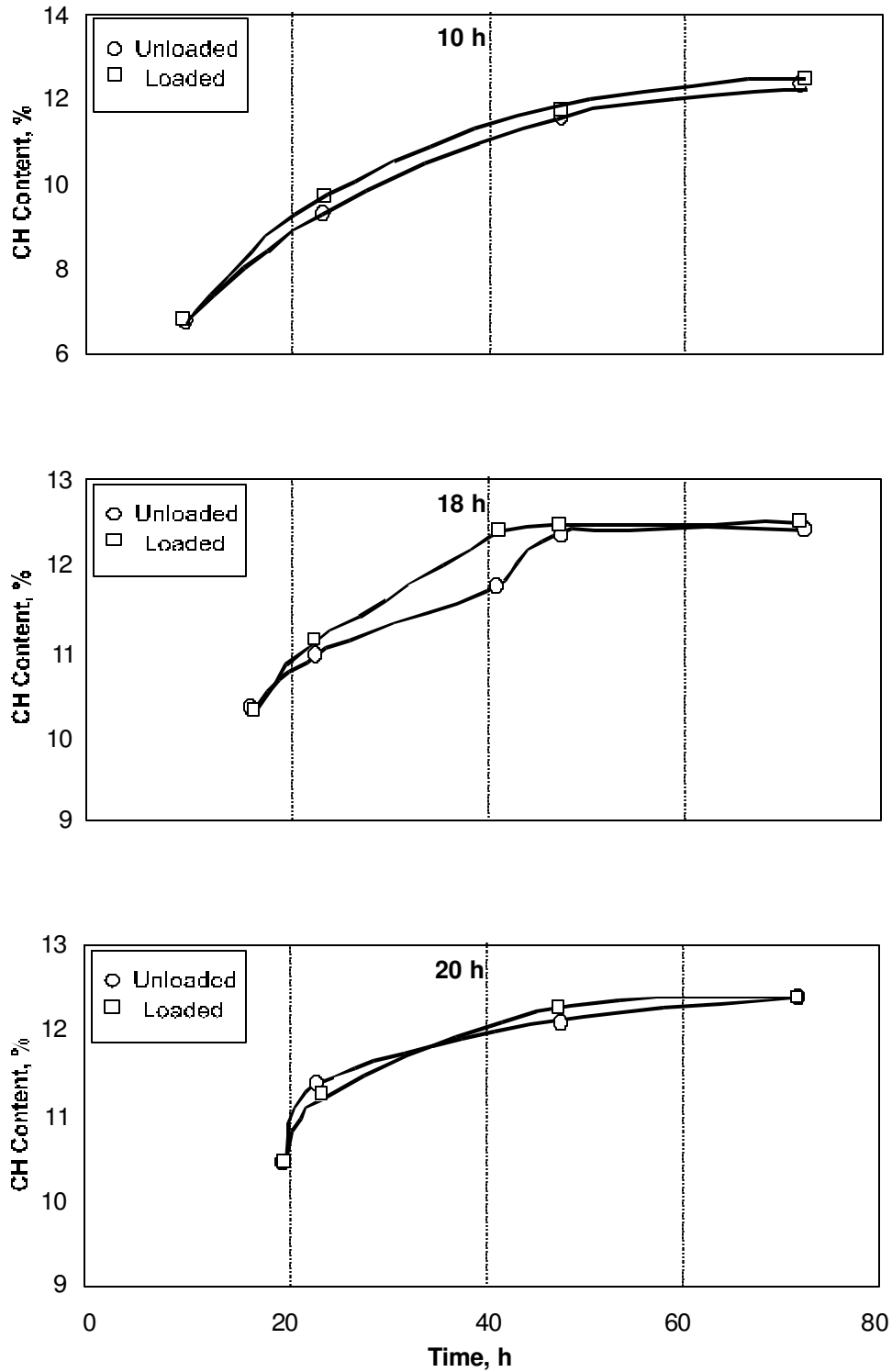
Figure 11. Degree of hydration α_{CH}, % of hardening cement paste: water/cement = 0.35, stress (6.8 MPa) applied at selected times.