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Low-Temperature Effects on the Microstructure of Cement Paste Exposed to Seawater

by V.S. Ramachandran, M.S. Cheung, and H.M. Hachem

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Abstract

Portland cement pastes made at a water/cement ratio of 0.25 were precured at 4°C for 3 to 15 hr and then cured in simulated seawater at -2.0 and 2°C for ages up to 180 days. Compressive strength, morphology, rate of hydration and porosity of the pastes were determined and interrelationships were established.

Résumé

On a effectué la cure de pâtes de ciment Portland préparées dans un rapport eau-ciment de 0,25 à 4 °C pendant 3 à 15 heures, puis dans de l'eau de mer simulée à -2 et 2 °C durant des périodes atteignant 180 jours. Les auteurs ont déterminé la résistance à la compression, la morphologie, le taux d'hydratation et la porosité des pâtes, et ils ont établi des relations entre ces paramètres.

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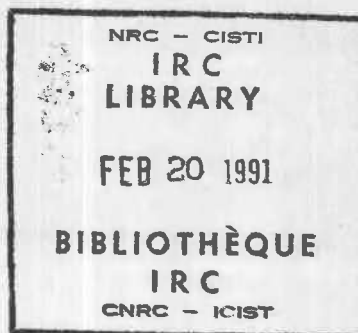
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The microstructure and compressive strength of normal portland cement pastes made with a water/cement ratio (w/c) of 0.25, precured at 4 C (39.2 F) for periods ranging from 3 to 15 hr, and then continuously cured in simulated seawater of different temperatures [−2, 0, 2 C (28.4, 32, 35.6 F)], were examined at ages up to 180 days. At low-precuring temperature, the 1 day compressive strength increased with increasing precuring duration. After 28 or 180 days of curing in seawater, however, the compressive strength showed a reversed trend. The development of physical, chemical, and microstructural characteristics of the cement paste were consistent with the strength increases observed at age 1 day. At older ages, strength was no longer a simple function of the degree of hydration, as complex interactions occurred between the cement paste and salts. It was evident from the cement paste morphology that these interactions resulted in a distinct paste composition which differs from that of specimens not cured in salt water.

Continuous curing in salt water at different temperatures [−2, 0, and 2 C (28.4, 32, and 35.6 F)] did not yield significant differences in the compressive strength development. The influence of salts on strength development seems to be more pronounced in the long term (28 days) and at higher curing temperatures. Also, results indicate that salt attack on the cement paste may continue even beyond 180 days.

During the initial curing period, low-curing temperature drastically retards the hydration process, leading to very low 1 day compressive strength. On the other hand, the initial rate of hydration and strength development appears to be accelerated by curing in salt water.

There is a linear relationship between the log of compressive strength and porosity for specimens; this relationship, however, becomes nonlinear at porosity below 20 percent when specimens are cured in salt water.

The growing interest in better understanding cold-weather concreting is evident from the large number of publications on the subject and the wide range of related research activities. This is amply reflected in the proceedings of a recent workshop on low-temperature effects on concrete.[1] Despite the difficulties associated with its use in cold climates, concrete remains a very popular construction material for many structures erected in the Arctic.

The most critical period in concrete construction is the initial curing period, when concrete is transforming from the weak plastic phase into the hardened solid state. Placing concrete in a low-temperature environment generally retards its strength gain and reduces its resistance to weathering. It will also result in finished concrete that is not as watertight as that which has not been frozen. In addition, if concrete is subjected to initial frost damage before it has attained sufficient strength, adverse effects on the properties of the hardened concrete usually result.

The curing conditions of concrete used in slip-form construction in the Arctic Sea are characterized not only by the low ambient temperature at placing but also by exposure to the cold seawater within a short period of time after placing (less than 24 hr).

Aitcin, Cheung, and Shah[2] carried out field and laboratory experiments simulating the curing conditions of concrete construction practice in the Arctic Sea. The laboratory simulation study indicated that the longer the initial curing period prior to immersion in the salt water, the higher the strength development, particularly during the first week. However, field data showed that the initial curing period had practically no influence on compressive strength development. The compressive strength of seawater-cured specimens in the field was consistently lower than that of the standard room temperature specimens. Opposite trends were observed in the laboratory when comparing the compressive strength of simulated seawater-cured specimens with that of the standard-cured specimens.

Gardner, Sau, and Cheung[3] also carried out similar experiments, and some of the results similarly led to conclusions that are not in total agreement with stan-

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dard findings. In that study, concrete with *w/c* of 0.45 and 0.35, cast and cured at 0 C (32 F) in simulated seawater, have resulted in strength gains faster than similar concrete cast and cured at 22 C (71.6 F).

The conflicting results may be explained by the difficulty in maintaining a prescribed constant curing temperature in the concrete specimen. Because of the heat of hydration resulting from the reaction between cement and water, the internal temperature of the specimen is elevated and cannot be controlled easily. The major significant chemical interaction in a concrete specimen occurs between the cement paste and the seawater. Thus, to investigate the effects of curing temperature and seawater on the structural and strength development of concrete, it was decided that cement paste specimens of relatively smaller dimensions rather than normal-sized concrete cylinders should be used, since this allows for a better control of the curing temperature.

In this study, portland cement paste specimens of 2 x 2 x 2 in. (5 x 5 x 5 cm) were precured at 4 C (39.2 F) for different durations, and then exposed to simulated seawater at −2, 0, and 2 C (28.4, 32, and 35.6 F). Additional specimens were moist-cured for 6 hr at 23 and 4 C (73.4 and 39.2 F), and then exposed to simulated seawater and freshwater, respectively. After curing for different durations in their respective environments, the physicochemical properties of the specimens were determined.

This paper presents the results of an experimental study aimed at determining the interrelationship between the microstructure and the physico-mechanical characteristics of portland cement pastes prepared and hydrated under simulated Arctic Sea conditions.

RESEARCH SIGNIFICANCE

This research was conducted to study the effects of cold temperature and seawater curing on strength, microstructure, and other properties of cement. The experimental results indicate that the physical and chemical characteristics of the cement paste, namely, compressive strength, true density, porosity, lime content, weight loss, and microstructure, are significantly influenced by the curing period, the temperature, and seawater. In addition, experiments confirmed that saltwater attacks the cement compounds even after the ini-

tial curing periods and could result in lower cement paste strength.

EXPERIMENTAL DETAILS
Materials

Normal Type 10 portland cement was used in this study. The major components were as follows: tricalcium silicate = 50.74 percent; dicalcium silicate = 21.15 percent; tricalcium aluminate = 8.64 percent; tetracalcium alumino ferrite = 8.52 percent; and calcium sulfate = 4.77 percent, totalling 93.82 percent.

Simulated seawater was prepared in the laboratory, and the concentration of constituent salts was as follows: sodium chloride = 25.0 g/l (4.0 oz-mass/gal); magnesium sulfate = 4.0 g/l (0.64 oz-mass/gal); magnesium chloride = 3.0 g/l (0.48 oz-mass/gal); calcium chloride = 1.0 g/l (0.16 oz-mass/gal); and potassium chloride = 0.7 g/l (0.12 oz-mass/gal), totalling 33.7 g/l (5.40 oz-mass/gal).

Curing baths

Five stainless steel baths 70 x 70 x 60 cm (27.6 x 27.6 x 23.6 in.) were used in the experiment. Three baths containing simulated seawater were cooled to temperatures of −2, 0, and 2 C (28.4, 32, and 35.6 F). The fourth bath was refrigerated and used for freshwater at a controlled temperature of 2 C (35.6 F), while simulated seawater at 23 C (73.4 F) was used in the fifth bath. During curing periods, the water temperature was controlled within an accuracy of ±0.5 C (0.9 F).

To prevent the formation of ice on the cooling coils of the baths, rigid plastic liners approximately 50 x 50 x 50 cm (19.7 x 19.7 x 19.7 in.) with 3 cm (1.2 in.) thick walls were fitted in the refrigerated baths. Alcohol was poured between the bath walls and liners and replenished periodically. The alcohol and water were agitated continuously by passing compressed air through a perforated copper tubing surrounding the bottom of all liners.

Procedure

The experimental program included the preparation and curing of cement paste cubes (*w/c* = 0.25) for initial periods of 3, 6, 9, 12, and 15 hr in a cold room controlled at 4 C (39.2 F), and then exposing them to the simulated seawater baths controlled at −2, 0, and 2 C (28.4, 32, and 35.6 F). Some reference specimens were cured continuously in humid rooms at 23 and 4 C (73.4 and 39.2 F). Additional specimens were cured at 23 and 4 C (73.4 and 39.2 F) only for a period of 6 hr, after which they were immersed in 23 C (73.4 F) simulated seawater and 2 C (35.6 F) freshwater, respectively.

Cement paste cubes 2 x 2 x 2 in. (5 x 5 x 5 cm) were cast and tested according to ASTM C 109 procedures for compression testing of cement mortars. A *w/c* of 0.25 was used based on the normal consistency method recommended in ASTM C 187. Mixing of cement with water was done as outlined in ASTM C 305. The *w/c* and mixing procedure were kept constant for different
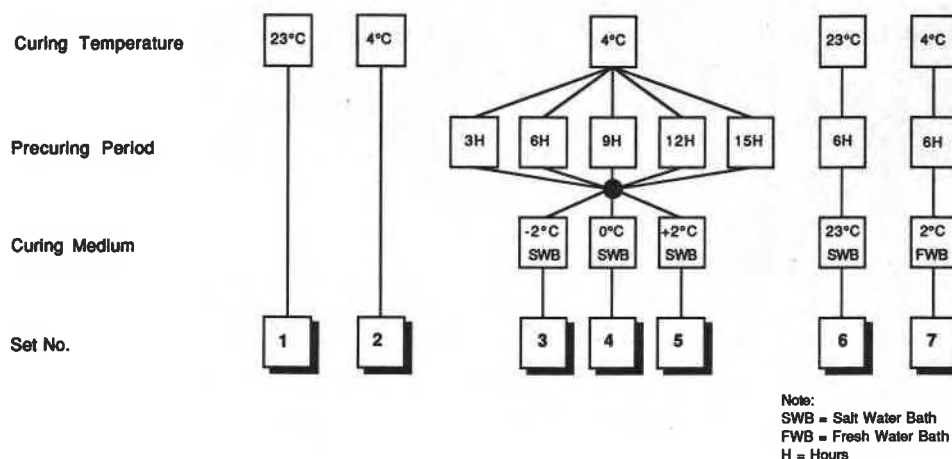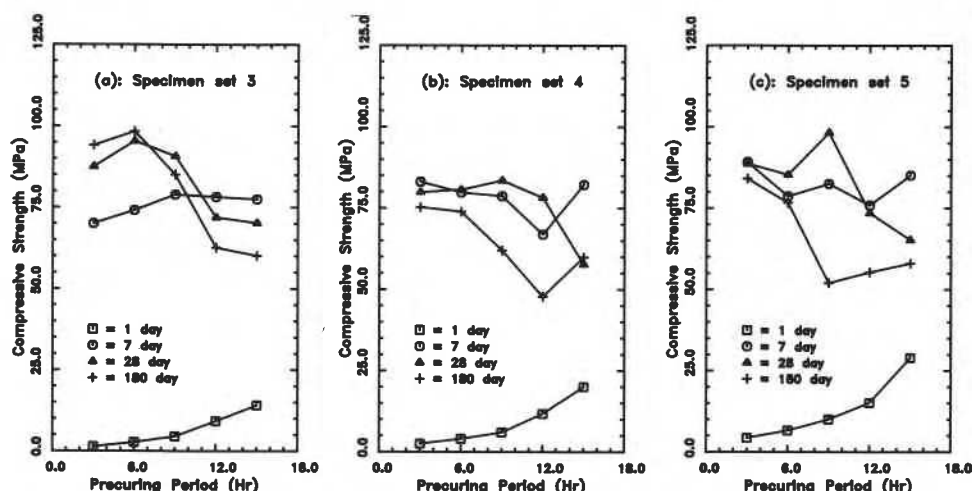
Fig. 1—Cement paste specimen description

Fig. 2—Compressive strength as a function of precuring period (1 MPa = 145.03 psi)

temperatures of mixing. For samples prepared at 4 C (39.2 F), the cement, water, molds, and mixing apparatus were cooled to 4 C (39.2 F) before mixing.

The compressive strengths reported in this paper are averaged values, each of which represents the average of three tested cement cubes. The cubes were tested within 6 min after removal from the curing bath. After breaking the cubes, pieces weighing about 20 g (0.71 oz/mass) were selected from the interior, immersed in cold alcohol, and then placed in a freezer at −18 C (−0.4 F) to stop hydration at a prescribed time. The alcohol was decanted off, and then the samples were dried under continuous vacuum for 1 day.

Thermal analysis was carried out using a 951-Thermogravimetric Analyzer (TGA) in conjunction with a 1090-Thermal Analyzer. For the TGA, 20 mg (0.71 oz-mass) samples were heated to 1000 C (1832 F) at a rate of 20 C/min (36 F/min) in a continuous flow of nitrogen. The results of the thermal analysis are expressed in terms of calcium hydroxide and weight loss, both as a percentage of ignited sample weight. Porosity, true density, and pore-size distribution were determined using the Aminco Mercury Pressure Porosimeter. Sample

sizes varied from 0.6 to 1.3 g (0.021 to 0.046 oz-mass). Cement morphology was also examined for selected samples using the Cambridge S-250 Scanning Electron Microscope.

Description of specimens

Cement specimens were grouped into seven distinct sets, as shown in Fig. 1 and described here. Set 1: 23C-STD is the reference set. Specimens were prepared, cast, and cured at a temperature of 23 C (73.4 F) and at 100 percent relative humidity for 180 days. Specimens in Set 2: 4C-STD were prepared, cast, and moist-cured in the cold room at a temperature of 4 C (39.2 F) for 180 days. Set 3: 4C/#H/SWB/−2C represents a group of five specimens that were prepared, cast, and moist-cured in the cold room at 4 C (39.2 F) for the various curing periods (#H) of 3, 6, 9, 12, and 15 hr. Once the initial curing period was over, the specimens were submerged in the −2 C (28.4 F) cold simulated seawater bath (SWB). Set 4: 4C/#H/SWB/0C represents a group of five specimens that were prepared, cast, and moist-cured in the cold room at 4 C (39.2 F) for the various curing periods (#H) of 3, 6, 9, 12, and
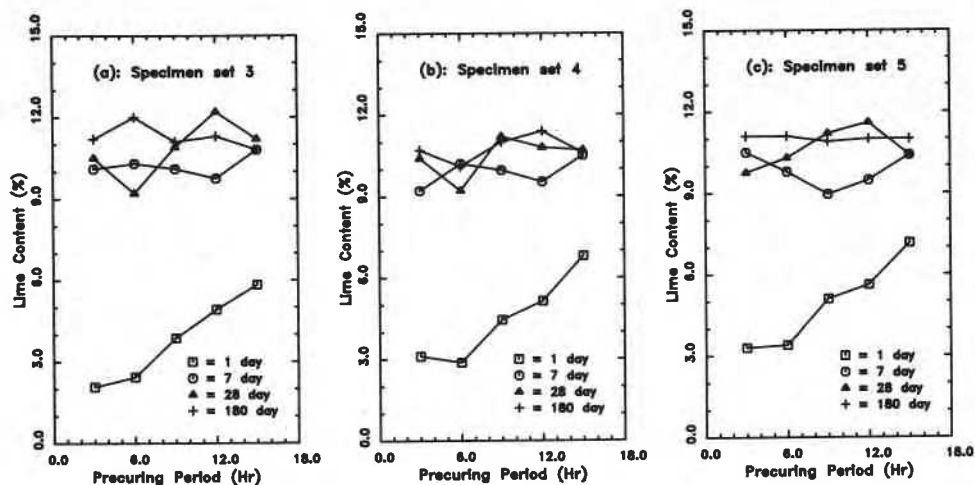
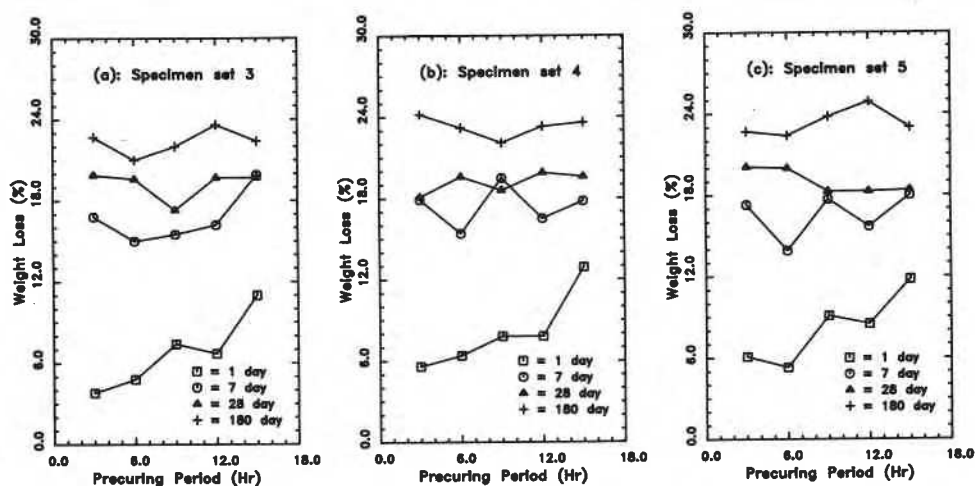Fig. 3—Variation of lime content with precuring period

Fig. 4—Influence of precuring period on weight loss

15 hr. Once the initial curing period was over, the specimens were submerged in the 0 C (32 F) simulated seawater bath. Set 5: 4C/H/SWB/2C represents a group of five specimens that were prepared, cast, and moist cured in the cold room at 4 C (39.2 F) for the various curing periods (#H) of 3, 6, 9, 12, and 15 hr. Once the initial curing period was over, the specimens were submerged in the 2 C (35.6 F) simulated seawater bath. In Set 6: 23C/6H/SWB/23C, specimens were prepared, cast, and cured in a 100 percent relative humidity humid room at 23 C (73.4 F) for a period of 6 hr and then submerged in a simulated seawater bath controlled at 23 C (73.4 F). The specimens in Set 7: 4C/6H/FWB/2C were prepared, cast, and cured in the cold room at 4 C (39.2 F) for a period 6 hr and then submerged in a freshwater bath (FWB) controlled at 2 C (35.6 F).

TEST RESULTS AND DISCUSSION
Influence of precuring period

The variation of compressive strength with precuring period for cubes of Specimen Sets 3 through 5, which

were precured at 4 C (39.2 F) and then exposed to the cold simulated seawater, are depicted in Fig. 2(a) through (c). Only data for 1, 7, 28, and 180 days are shown.

1 day—Cement strength after 1 day in the simulated seawater baths increases as the period of initial curing increases. The rate of strength increase is higher for specimens exposed to warmer simulated seawater. Despite the low-strength development in the 1 day cement cubes [Fig. 2(a)], it is expected that hydration will continue to take place even at -2 C (28.4 F), because saltwater does not freeze at -2 C (28.4 F). Even freshwater would not freeze at -2 C (28.4 F) in narrow capillaries of the cement pastes.[4] This is evident from the lime content and weight loss of the 1-day curves in Fig. 3(a) and 4(a), wherein cubes initially cured for longer periods had higher values of lime content and weight loss.

As cement hydrates, its volume expands and its true density decreases, as does its porosity.[5] Fig. 5(a) and 6(a) show these trends, particularly in terms of porosity, for specimens submerged in simulated seawater for 1 day. It is evident that the longer the initial curing pe-
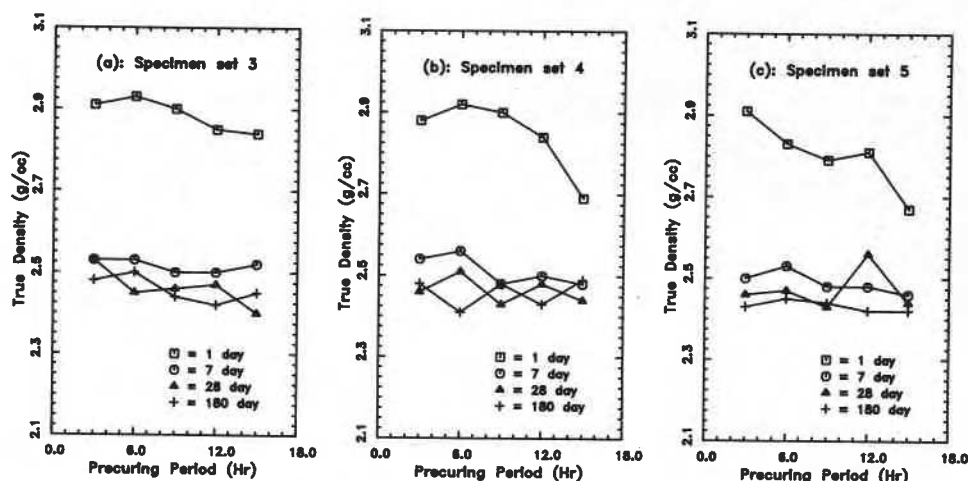
Fig. 5—*Variation of true density with precuring period (1 g/cc = 62.4 lb-mass/ft³)*
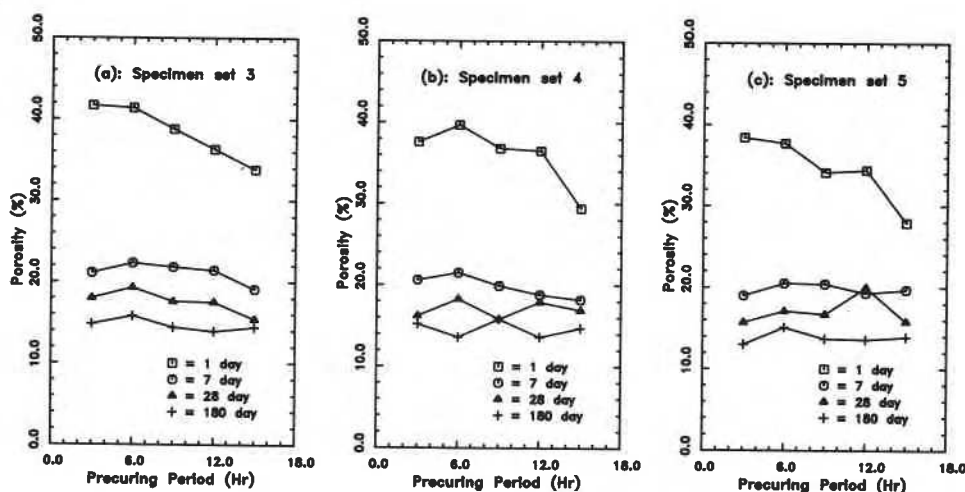

Fig. 6—*Influence of precuring period on porosity*

riod, the more the hydration and, hence, the lower the cement porosity and true density.

7 day—As expected, the 7 day compressive strengths are substantially higher than the 1-day strengths. Unlike that for the 1 day cement paste, compressive strength variation with the precuring period is no longer a simple steady climb. Fig. 2(a) through (c) show that the compressive strength is practically the same for specimens initially cured for periods varying between 3 and 15 hr. Similar trends are observed in Fig. 3(a) through (c), 4(a) through (c), 5(a) through (c), and 6(a) through (c) for the lime content, weight loss, true density, and porosity.

28 day and 180 day—The strengths at 28 and 180 days are higher [87.5 MPa and 94.1 MPa (12,691 and 13,256 psi), respectively] for specimens with an initial curing period of 3 hr than those with 15 hr of initial curing [Fig. 2(a)]. The compressive strengths for the latter precuring period are 70.2 and 60.1 MPa (10,182 and 8716 psi), respectively, for the 28 and 180-day specimens. From this, it is evident that the precuring period has a significant influence on the long-term strength of cement pastes exposed to cold seawater.

Continuous hydration takes place in specimens that

are precured in 100 percent relative humidity and subsequently immersed in seawater. The hydration is also likely accelerated by seawater in the early stages so that, by 28 days, much of the hydration is completed. Hydration tends to increase the strnegth of cement. Concurrent with hydration, however, is the attack by saltwater on the cement paste, and the complex compounds that are formed tend to decrease the strength of the paste.[6] These reactions dominate at the latter stages of curing when much of the hydration has been completed. It is, therefore, conceivable that for curing periods longer than 180 days, further attack by seawater could result in even lower cement paste strengths.

The variation of lime content, weight loss, true density, and porosity was reasonably constant for specimens precured for different periods (see Fig. 3 through 6). Lime content variation with different precuring periods is not very different at 28 and 180 days (Fig. 3). However, the weight loss is consistently higher for samples cured for 180 days (Fig. 4). This would indicate that the composition of the paste at 180 days is different from that at 28 days. These observations further indicate that several compounds are formed by the interaction of seawater with the cement hydrate. True
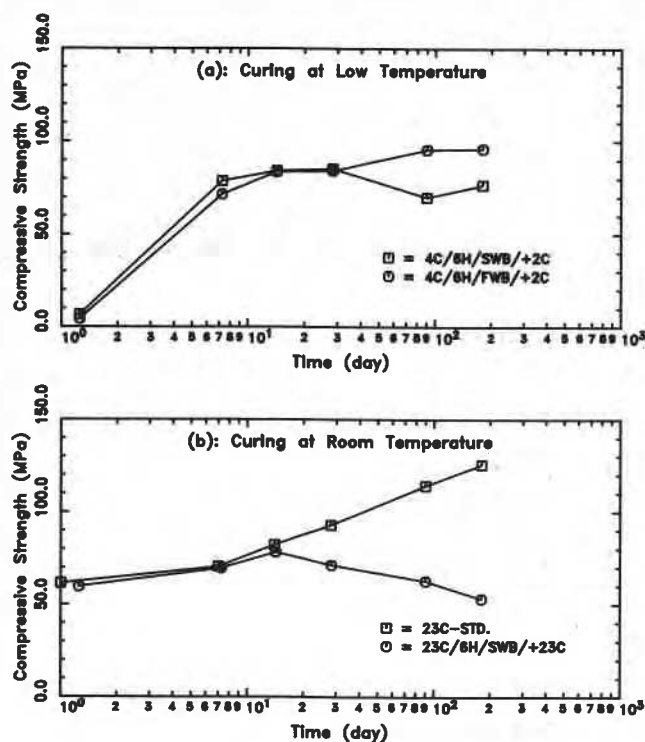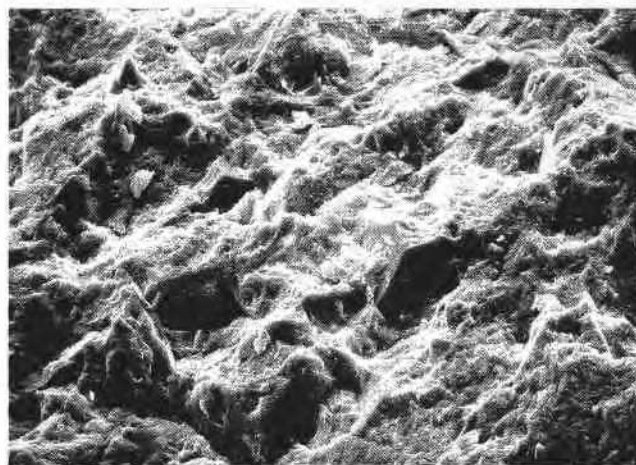
Fig. 7—Development of compressive strength with time
(1 MPa = 145.03 psi)

density is not changed at 180 days from that at 28 days
(Fig. 5). The porosity of specimens at 180 days is lower
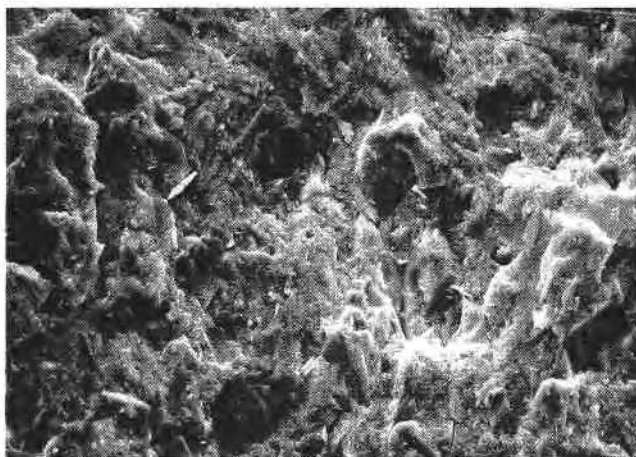than that at 28 days (Fig. 6).

Comparison of strength development in freshwater and seawater

To explain the strength development in seawater,
some investigations were carried out to compare the
strengths in cement pastes cured in freshwater for dif-
ferent curing periods. Fig. 7(a) shows compressive
strength development with time for specimens exposed
to 2 C (35.6 F) simulated seawater and freshwater. It is
evident that strength development for cement paste ex-
posed to fresh or saltwater is practically the same for
the first 28 days. However, specimens exposed to sea-
water exhibit a drop in compressive strength with time
beyond 28 days. The 180 day compressive strength for
cement paste exposed to 2 C (35.6 F) freshwater was
96.0 MPa (13,924 psi), while that for specimens ex-
posed to 2 C (35.6 F) simulated seawater was 76.7 MPa
(11,124 psi). Further investigations showed that curing
at 23 C (73.4 F) in seawater decreases strength after 14
days of exposure. Moist curing, on the other hand,
showed a continued steady increase in strength even up
to 180 days. The strength at 180 days for cement paste
at 23 C (73.4 F) is 125.6 MPa (18,217 psi), while that
for specimens exposed to seawater is 53.1 MPa (7702
psi) [see Fig. 7(b)].

The lower strengths in seawater-cured specimens have
already been explained by the interactions between salts
in seawater and the cement hydrate. Studies involving
lime content, weight loss, true density, and porosity did
not show significant differences between moist-cured

(a) Specimen Set 1: 23C-STD

(b) Specimen Set 6: 23C/6H/SWB/23C

Fig. 8—Influence of seawater salts on cement paste
morphology at 180 days

and seawater-cured specimens. Lower strengths cannot
be directly rationalized in terms of the degree of hydra-
tion or porosity; they are better explained by the devel-
opment of microcracks as a consequence of exposure to
seawater. The two micrographs of cement paste cured
at 23 C (73.4 F) for 180 days in seawater and freshwa-
ter (Fig. 8) show that the paste cured in seawater has a
different morphology from that cured in freshwater.
The paste cured in seawater shows more discrete units
and more microcrack development.

If the cement paste specimens do not come into con-
tact with seawater, the long-term strength development
is not affected much, whether the paste is cured at 23
or 4 C (74.3 or 39.2 F). The 1-day strengths for speci-
mens cured at 4 and 23 C (39.2 and 73.4 F) are, respec-
tively, 8.70 and 61.5 MPa (1262 and 8920 psi). Beyond
about 14 days, the rate of strength development for
specimens is very similar, as indicated in Fig. 9.

Porosity-compressive strength relations

Extensive work has been carried out to relate
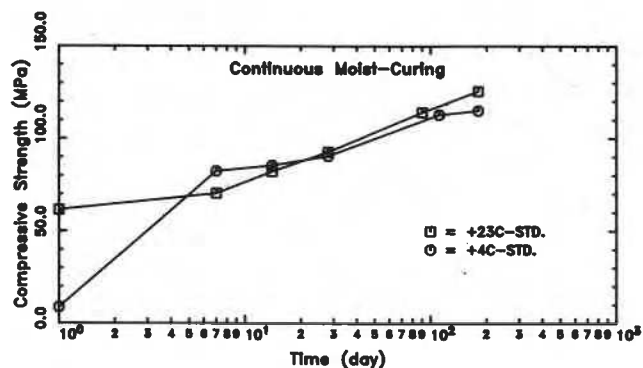compression strength to porosity. In cement paste sys-

Fig. 9—Compressive strength development for moist-cured cement pastes (1 MPa = 145.03 psi)

Fig. 11—Compressive strength relative to that of standard-cured specimens
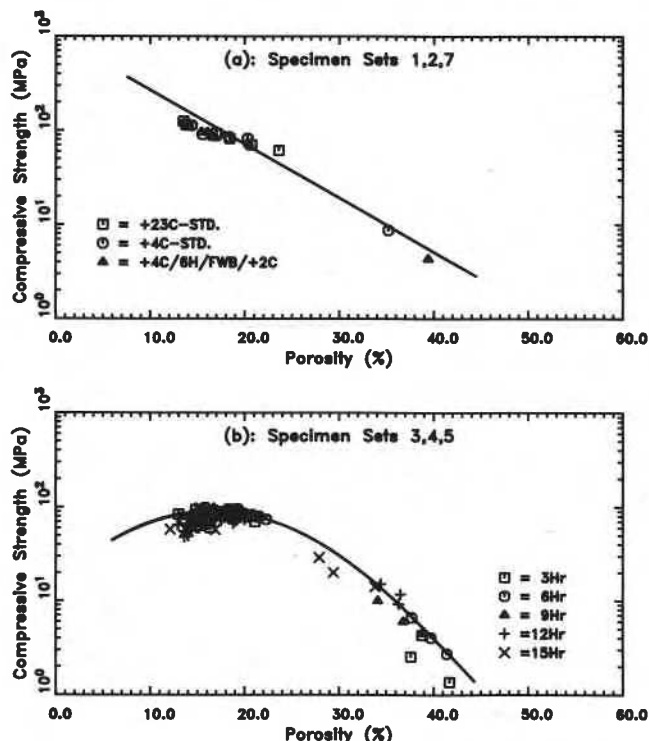

Fig. 10—Compressive strength-porosity relationships in cement pastes (1 MPa = 145.03 psi)

tems, a semilog plot of strength with porosity usually shows a linear relationship.[6]

Fig. 10(a) shows a plot of strength versus porosity for pastes moist-cured and not exposed to saltwater. A linear relationship is observed. Specimens cured in saltwater [Fig. 10(b)] show a linear strength-versus-porosity relationship greater than about 20 percent. At porosity below this value, i.e., for specimens with longer curing periods, the relationship is essentially nonlinear, indicating a cement paste composition change caused by seawater attacks.

Compressive strength development

In Fig. 11, the strength development in cement pastes (a) moist-cured continuously at 4 C (39.2 F); (b) precured for 6 hr at 4 C (39.2 F) and then continuously exposed to freshwater; (c) precured for 6 hr at 4 C (39.2 F) and then saltwater-cured at 2 C (35.6 F); and (d)
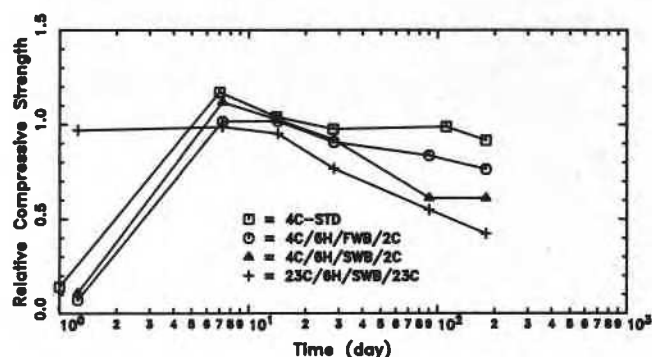
precured at 23 C (73.4 F) for 6 hr and then cured in saltwater at 23 C (73.4 F), is compared with that in specimens continuously at 23 C (73.4 F).

The deleterious effects of low-temperature curing in saltwater on strength development are most significant during the early period of hydration (1-day-old cement paste). During this period, the low-curing temperature retards the hydration process, leading to a very low 1 day compressive strength. The relative compressive strength can be as low as 0.1 at 1 day. With time and particularly during the first week of hydration, the relative compressive strength increases sharply to a value between 1.0 and 1.2 at Day 7. After 7 days, the relative compressive strength begins to decrease gradually with time. At 180 days, relative compressive strength can be as low as 0.4. It is evident that the rate of decrease in the compressive strength is dependent on the curing conditions. The rate of strength decrease is lowest for the specimen continuously moist-cured at 4 C (39.2 F). Specimens precured at 23 C (73.4 F) for 6 hr and then immersed in simulated seawater show the highest rate of strength decrease.

Because detrimental effects of saltwater on cement physicochemical properties of the cement paste are mainly chemical in nature, and since chemical reactions are usually accelerated at higher temperatures, detrimental effects in specimens exposed to 23 C (73.4 F) simulated seawater bath are, therefore, the most pronounced. For specimens continuously cured at low temperature [4 C (39.2 F)] but not exposed to saltwater, the relative strength stabilizes around 1.0 after 28 days. For specimens exposed to simulated seawater, the relative strength is seen to decrease with time. This indicates that compressive strength reduction with time is caused by the saltwater attack. Chemical attacks on the cement paste seem to continue at the same rate even after 90 days.

CONCLUSIONS

The physical and chemical characteristics of cement paste, namely, compressive strength, true density, porosity, lime content, weight loss, and microstructure, are influenced by the precuring period, the temperature at which the specimens are precured, and the curing environment.

Specimens precured in moisture at 4 C (39.2 F) and subsequently exposed to simulated seawater at −2, 0, and 2 C (28.4, 32, and 35.6 F) for different durations show that the 1-day strength increases with the precuring period. Lime content and weight loss showed similar trends, while porosity and true density followed decreasing trends with the precuring period. At longer curing periods of 28 and 180 days, the strength decreases as the precuring period increases, whereas porosity, true density, lime content, and weight loss did not show significant variation. In other words, strength is no longer a simple function of degree of hydration because of the complex interactions between the paste and salts. Long-term strengths developed by the cement paste, cured in simulated seawater at −2, 0, and 2 C (28.4, 32, and 35.6 F) are similar.

Specimens continuously moist-cured at 4 C (39.2 F) and those cured in simulated seawater at 2 or 23 C (35.6 or 73.4 F) developed strengths at almost the same rate up to 14 days, after which those exposed to saltwater showed gradual loss of strength. This is caused by the attack of salts on the cement paste, which is more pronounced at the higher curing temperature. It is evident from the cement paste morphology that the interactions between salts and cement components result in a distinct cement composition characterized by discrete units and microcrack developments. These characteristics lead to long-term strength degradation in the cement paste.

At later stages of curing, specimens that were moist-cured at ambient temperature developed compressive strengths that are higher than those in specimens cured at 4 C (39.2 F) and those in specimens exposed to saltwater. During the early curing stages (less than 7 days), specimens cured in seawater showed higher strengths than those cured in freshwater, because seawater may have accelerated the rate of hydration. In the early periods of seawater curing, specimens cured at low temperature develop strengths at a slower rate and approach the strength of standard-cured specimens [23 C (73.4 F)] at around 7 days, but subsequent to this the strengths decrease considerably, relative to the standard-cured specimen. Moreover, specimens cured in moisture at any comparable temperature show higher strength than those cured in seawater.

There is a linear relationship between log compressive strength and porosity for moist-cured specimens. Specimens that were exposed to seawater continuously also show a linear relationship, but only for porosity greater than about 20 percent; below this porosity value, the relationship is essentially nonlinear. This indicates a change in the physical and chemical structure of the paste.

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