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FREE AND WATER SOLUBLE CHLORIDE IN CONCRETE

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ABSTRACT

Two methods were used to determine the 'free' and 'water soluble' chloride ions concentration in four differing strength grade concretes. Each grade of concrete had varying concentration of Cl^- admixed as NaCl. In all 24 different batches of concretes were made with admixed Cl^- concentration varying from 0.2 to 2%, by weight of cement. The first method was the analysis of the pore solution expressed from specimens and the second by the analysis of the decanted solution of pulverised specimens. The results suggest that the concentration of Cl^- and OH^- ions as measured in the expressed pore solution represent their true free ion concentrations. Furthermore, the Cl^- concentration of both the expressed and decanted solutions were found to depend on the (i) concentration of the admixed Cl^- ions; (ii) strength of the concrete; (iii) presence or absence of fly ash and (iv) presence or absence of a superplasticiser. Accordingly, there is no simple relationship between the Cl^- concentration as found in decanted and pore solutions. Whereas the presence of fly ash improves the chloride binding capacity of a concrete, the presence of superplasticisers tends to lower it. The paper concludes that in order to limit the damage caused by Cl^- in the concrete, an upper limit on the total Cl^- contents of the admixed chloride rather than the percentage of Cl^- by the weight of cement should be prescribed.

Introduction

Corrosion of steel reinforcement is the most significant factor of deterioration in reinforced concrete structures. Protection of steel reinforcement from corrosion is usually provided by the high alkaline environment of the concrete. If the alkalinity of the surrounding concrete is not impaired, because of carbonation for example, the steel is expected to remain passive. Nevertheless, if chloride ions exist in the vicinity of the steel, the steel will show pitting corrosion even though the pH of the environment is higher than 11.0. Chloride ions can exist as a result of aggregates contamination, use of chloride bearing water and chloride bearing admixtures within the concrete itself. However, they also can penetrate into the concrete from the outer environment if the concrete is in contact with sea water, saline water table, saline laden dust, or if the structure is in the splash zone. Whether the source of chloride is from within the concrete or from the external environment, the chloride ions will diffuse through the body of the concrete and ultimately reach the steel. Not all of the chloride ions which are within or penetrate the concrete remain free in the pore solution. Some of the ions get bound to the hydration products in a chemical reaction to form calcium chloroaluminate hydrate. Some chloride ions get adsorbed to the various hydrates of cement. Thus only a portion of the chloride ions remains free. It is this portion which is responsible for causing damage to the concrete structures.

Extensive research has been done to establish relationships between the chloride ions content and the onset of corrosion. Almost invariably, a method for the determination of chloride content had to be adopted. Methods for the determination of total chloride in a concrete sample are standardised(1). The standard method involves quite accurate chemical analysis and careful specimen preparation. Two other methods of chloride ion determination have come into use recently. These are the Free Chloride Content and the Water Soluble Chloride Content. As the name implies the water soluble chloride is that which can readily dissolve in water. A standard test for the determination of this type of chloride exists(2,3). It is however an issue which is not as clear as that of total chloride. This is so because the amount of chloride ions released into solution depends upon the method adopted which involves factors like the fineness of pulverised sample, the amount of water added, the temperature, the agitation method and the time allowed(4). In fact it has been claimed that given enough time and water, all the chlorides can be reclaimed by the solution(5). In a recent paper(6) Arya and Newman distinguished between the water soluble chloride content and the free chloride content. They defined the free chlorides as the chloride ions in the pore solution. These ions can be determined by the pore solution expression technique which has been used by many researchers.

Arya and Newman(6) reported that irrespective of whether chlorides are introduced internally or externally, the fluid expressed by the pore pressure is representative of the fluid in the specimen. Nonetheless, the expression of pore solution requires obtaining special specimens and often when these specimens are dry it becomes almost impossible to extract any pore solution(7,8). Further, this method has only limited applications for the determination of chloride ions on concretes from actual structures. Accordingly, leaching and other simpler techniques have been proposed to estimate the chloride ion concentration in pastes and mortars(4,6,9). The leaching techniques offer a more practical method of determining the free chloride. However, there are several leaching techniques and it has been reported that no single technique was sufficiently accurate over the range of chloride additions investigated(6). The leaching techniques are also known to overestimate the free chloride content when applied to concretes exposed to external chloride(6,10).

The objective of the present research was to investigate whether a relationship between water soluble chlorides as determined by a leaching method and those present in the expressed solution could be established. This paper also aims to highlight the limitations regarding such relationships and caution against hasty generalisations. It describes the results for concretes with and without fly ash and a superplasticiser with admixed NaCl of Cl^- concentration of 0.2 to 2% of the weight of cement.

Experimental Program

Concrete Mixes

The various concrete mixes used in this investigation were made using 10 mm maximum size crushed gravel, river sand, Type A normal Portland cement, similar to ASTM Type I, bituminous fly ash (ASTM Class F), reagent grade sodium chloride and a proprietary superplasticiser. Chemical composition and physical properties of the fly ash and cement are given in Table 1.

In all, four different basic types of concretes were cast. The objective was to test concretes from medium to high strength. These concretes are referred to as M, H, HSC, and HSFC concretes. The mix details of all four concretes are included in Table 2. The HSC and HSFC concretes were cast using a superplasticiser and a superplasticiser with an addition of fly ash respectively.

All the four basic types of concretes were cast with differing dosage of NaCl. Other quantities of material used to prepare the four batches of concrete are included in Table 2. In M and H concretes, the Cl^- contents were 0.2, 0.4, 0.6, 0.8, 1.0, 1.5 and 2.0% of the cement weight. However, HSC and HSFC concretes were cast with 0.8, 1.2, 1.6, and 2.0% of Cl^- by weight of cement. In all 24 batches of concretes were cast.

An individual concrete mix is designated by the basic type of concrete and by the Cl^- content by weight of the cement. For example, HSFC-1.6 stands for a high strength superplasticiser fly ash concrete with Cl^- content of 1.6% by weight of the cement.

The sand and the coarse aggregate used in the mixtures were oven-dried and contained no moisture. The concretes were mixed in a Hobart mixer. From each concrete, 6 - 4 x 100mm cylinders and 6 - 50mm cubes were cast.

Specimen Conditioning

Immediately after casting, the moulded specimens were sealed in a bigger container which was placed in the fog room. The specimens were demoulded 24 hours after casting and remained in the fog room for 27 days. At the age of 28 days, the specimens were tested for the compressive strength, evaporable and non-evaporable water, and chloride and hydroxyl ion concentration.

TABLE 1

Physical and Chemical Properties of Fly Ash and Cement

Bulk density (t / m^3)	2.10	
Unit mass (t / m^3)	1.02	
Residue on 150 μm sieve (%)	0.4	
Residue on 45 μm sieve (%)	9.5	
Specific surface (m^2 / kg)	270	
	Fly ash	**Cement**
Compound	**Compound (%)**	
Silica	57.3	20.6
Alumina	23.4	4.6
Calcium Oxide	4.0	64.7
Iron Oxide	3.8	5.0
Magnesium Oxide	0.6	1.1
Sodium and Potassium Oxide	3.8	0.53
Sulphur Trioxide	0.1	2.4
Loss of Ignition	2.2	2.0

TABLE 2
Mix Details

Compressive Strength (MPa)	Mix	Cement (c) (g)	Sand (g)	Coarse Agg. (g)	Deionised Water (w) (g)	Superplasticiser (s) (g)	Fly ash (f) (g)	$\frac{w+s}{c+f}$
37.5	M	469	1055	1923	305	-	-	0.65
60.3	H	680	969	1782	305	-	-	0.45
73.4	HSC	782	949	1760	281	9.5	-	0.37
72.6	HSFC	782	911	1760	281	11	117	0.32

6 - 50 x 50mm cubes were used for the compressive strength determination and 6 - 41 x 100mm cylinders were used for the determination of Cl^- contents in the expressed and decanted solutions.

Determination of Chloride Ion Concentration

Chloride ion determination was done by potentiometry using a digital microprocessor controlled pH/mV meter with Chloride Ion Selective Electrode. The accuracy of the instrument is ± 0.1mV and has an automatic temperature compensation probe. Five standard samples containing chlorides ranging from 0.003 N to 0.5 N solutions were prepared and used to calibrate the instrument every time the measurements were taken.

Two procedures, however, were adopted to get a representative sample from the specimens to estimate the concentration of the free chloride ions. A pore expression device, manufactured according to Barneyback and Diamond$^{(11)}$ was used to extract the pore solution from the cylindrical specimens. Second, the following solution technique was used to estimate the concentration of the chloride ions in the solute.

The cylindrical specimens were crushed and sieved through a 600 µm sieve. A known quantity of the sieved powder and deionised water were mixed and stirred for five minutes and then sealed and stored for 24 hours. A ratio of powder and water of 1 - 3.5 to 4 was used for this preparation. After 24 hours, the mixture was thoroughly stirred and decanted over a filter paper. The filtrate was then used for Cl^- determination by potentiometry as described earlier. In this investigation, this solution technique was referred to as "decantation method". The Cl^- concentration was determined on two replicates of both the expressed and decanted solutions.

Representative samples of the cylinders were also used to determine both evaporable and nonevaporable water. These determinations were done in an oven and a furnace kept at 105°C and 1050°C respectively.

The Cl^- concentration as determined by potentiometry (in mM/l) has been converted to the Cl^- concentration as 'percentage of the dry cementitious materials'. This has been done in order to eliminate the effect on the results of the moisture state of the specimens. Nevertheless the concentrations expressed in mM/l are still included in Table 3. The Cl^- concentration of the various concretes is shown in Table 3.

The OH^- determination was also performed on both the expressed and decanted solutions by titration against standard hydrochloric acid of 0.01N using phenolphthalein as indicator. These are also included in Table 3.

TABLE 3
Chloride and Hydroxyl Ion Concentration Values

Mix	28 Day Strength (MPa)	Chloride Concentration				OH^- Concentration (mM/l)	
		(mM/l)		% of the cement weight			
		Expressed Solution	Decanted Solution	Expressed Solution	Decanted Solution	Expressed Solution	Decanted Solution
M-0.2	35.6	14	74	.021	0.111	96	2218
M-0.4	38.0	16	140	0.027	0.228	121	2135
M-0.6	37.0	37	236	0.058	0.373	121	2347
M-0.8	40.5	46	244	0.072	0.385	131	2219
M-1.0	51.0	56	263	0.11	0.520	163	2037
M-1.5	35.0	152	609	0.255	1.020	121	2075
M-2.0	37.0	335	762	0.615	1.398	111	1658
H-0.2	48.0	11	49	0.011	0.051	117	2337
H-0.4	62.0	31	152	0.037	0.179	190	2039
H-0.6	60.0	62	198	0.072	0.230	209	2489
H-0.8	72.0	90	238	0.099	0.262	221	2590
H-1.0	73.0	181	539	0.195	0.582	210	2572
H-1.5	46.5	321	789	0.365	0.899	215	2470
H-2.0	71.0	583	1325	0.645	1.468	198	2283
HSC-0.4	81.5	53	145	0.048	0.131	240	2566
HSC-0.8	85.5	151	409	0.126	0.343	258	3071
HSC-1.2	82.0	377	434	0.373	0.428	230	2226
HSC-1.6	75.5	710	1222	0.752	1.295	176	2374
HSC-2.0	61.0	1113	1773	1.104	1.760	163	2270
HSFC-0.4	68.5	69	145	0.074	0.156	146	2230
HSFC-0.8	60.5	257	927	0.225	0.813	176	2452
HSFC-1.2	77.5	466	1099	0.539	1.271	220	2136
HSFC-1.6	73.5	748	1175	1.107	1.740	204	1502
HSFC-2.0	84.8	1200	1906	1.283	2.038	180	2085

Results and Discussions

OH^- Concentration in the Decanted and Expressed Solutions

The variation of OH^- concentration in the expressed solution of the four types of concretes, with the added Cl^- is included in Fig. 1. Initially, with the increase in the added Cl^-, OH^- concentration increased in the expressed solution of all the concretes. However, as the added Cl^- concentration in the various concretes increased a decrease in the OH^- concentration of all the concretes can be observed. In the medium (M) and high strength (H) concretes, the OH^- concentration in the expressed solutions increased up to an addition of 1 and 0.8% of Cl^-, respectively, thereafter further addition of Cl^- resulted in a decrease in the OH^- concentration of the expressed solution of the two concretes. A similar pattern in the variation of OH^- concentration of HSC and HSFC concretes can be observed, these results suggest that the variation in the OH^- concentration with the addition of Cl^- in concretes depends on both the grade of concrete and the concentration of the Cl^- in the concretes. In the M, H and HSC concretes, made without the addition of fly ash, the concentration of OH^- increased, at least initially, as the strength of the concrete increased. This, of course, is indicative of the higher concentration of $Ca(OH)_2$ in the high strength concretes.

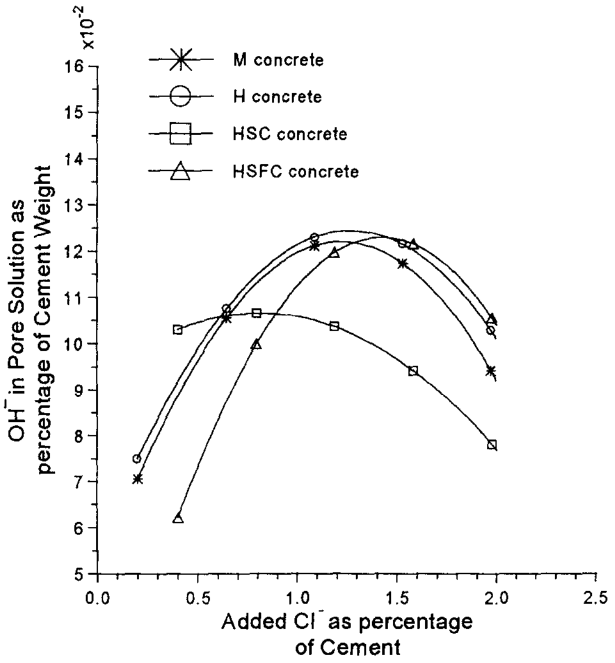

Fig. 1 - Concentration of Hydroxyl Ions in Expressed Pore Solution

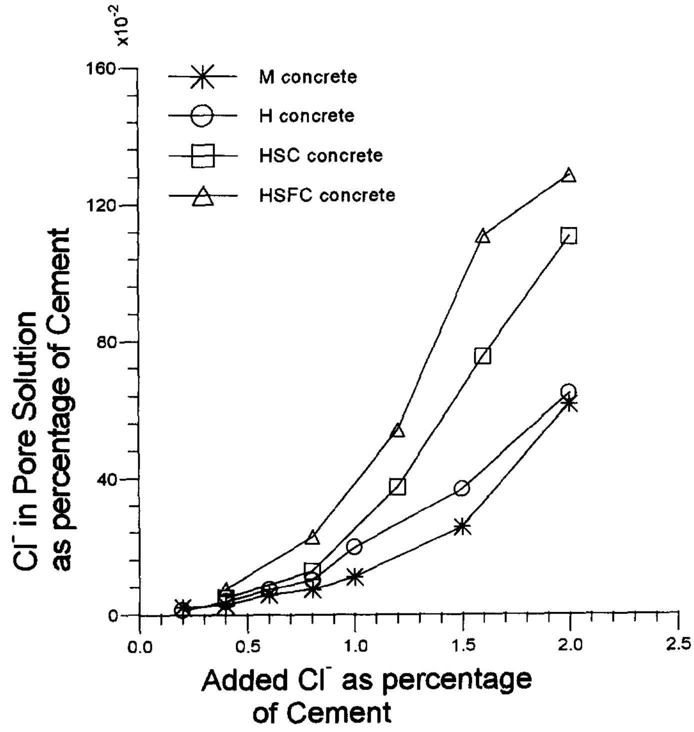

Fig. 2 - Concentration of Chloride Ions in Expressed Pore Solution

An increase of OH^- ion concentration by the addition of NaCl has been observed by Byfors et al(10), Page et al(12), Hansson et al(13) and Kawamura et al(14). For a range of added Cl^- of 0.2 to 2% of the cement weight as used in this investigation, the observation of the above investigators cannot be fully substantiated. It is also probable that the variation of OH^- of concretes is somewhat different to those of hydrated cement pastes and mortars.

The results obtained by decantation gave no definite trend in the variation of OH^- ions with the addition of differing levels of Cl^- in the four concretes tested. The results included in Table 3 also indicate that the OH^- concentration as determined from the decanted solution is higher than the OH^- ions concentration as determined from the expressed solution. On the average, the OH^- ion concentration of the decanted solution is about 13 times the OH^- ion concentration of the expressed solution. For the four grades of concretes tested, the above ratio varied between 7.4 to 27; certainly a big variation. This ratio does not seem to depend on the concentration of Cl^- of a given concrete. These results suggest that decantation methods cannot be reliably used to estimate the OH^- ion concentration of concrete. It is possible that the grinding of the concrete as performed in this method exposed unhydrated cement grains and thus unrealistically increased the OH^- content in the solution.

Cl^- Concentration in the Expressed and Decanted Solutions

The Cl^- concentration in the expressed and decanted solutions of the concretes tested are included in Table 3 and Fig. 2 and 3. As expected, with the increase in the added Cl^- contents of a given concrete, the free Cl^- in both the expressed and decanted solution increased. Further, as the strength grade of the concrete increased, the concentration of the free Cl^- within the concrete also increased. These results established that for equal concentration of the added Cl^- from NaCl, the free Cl^- concentration in the higher strength concrete is higher (see Fig. 4). Admittedly, the concentration of the free Cl^- in a concrete is an important factor responsible for the initiation and propagation of corrosion of the reinforcement. According to the results included in Table 3 (and Figs. 2-4), the higher strength concretes are likely to enhance the chances of corrosion of the reinforcement for equal concentration of the added Cl^- expressed as percent of the cement. Certainly, high strength concretes are more effective in decreasing the chloride ingress from the surrounding salt solutions. This, of course, is attributed to the lower permeability and the lower coefficient of diffusion of the high strength concretes.

It has been suggested(9) that a total chloride content of 0.4% (by weight of cement) produces a low risk of corrosion, between 0.4 and 1% produces a medium risk and greater than 1% produces a high risk. As is apparent from results in Fig. 2, beyond a total Cl^- content of 1%, the proportion of free Cl^- in the pore solution increases rapidly. It is interesting to note that while for M concrete the average proportion of free Cl^- of the total Cl^- is about 11% whereas for H concrete the corresponding value is about 20%. Likewise, for HSC and HFSC concretes, the average proportion of free Cl^- at 2% addition of Cl^- is 55 and 64% respectively. Accordingly, it is concluded from the results that the concentration of the free Cl^- in a concrete depends on the strength grade and composition of concrete and the concentration of the total added Cl^-. For a given addition of Cl^- (from NaCl), the chloride binding capacity of a concrete also diminishes, as a percent of cement, at the higher levels of chloride additions. According to Page et al(15) the cement hydrates bind a substantial portion of chloride in an insoluble form below a total chloride content of 1% and beyond this point the binding capacity is largely exhausted. In the light of the above discussion, a higher strength concrete at 1% addition of Cl^- (of the weight of cement) could be a

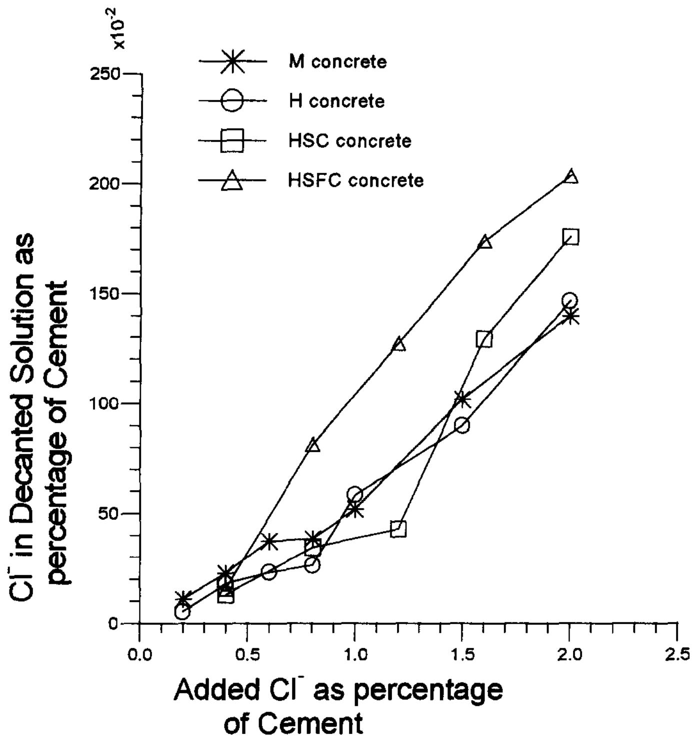

Fig. 3 - Concentration of Chloride Ions in Decanted Solution

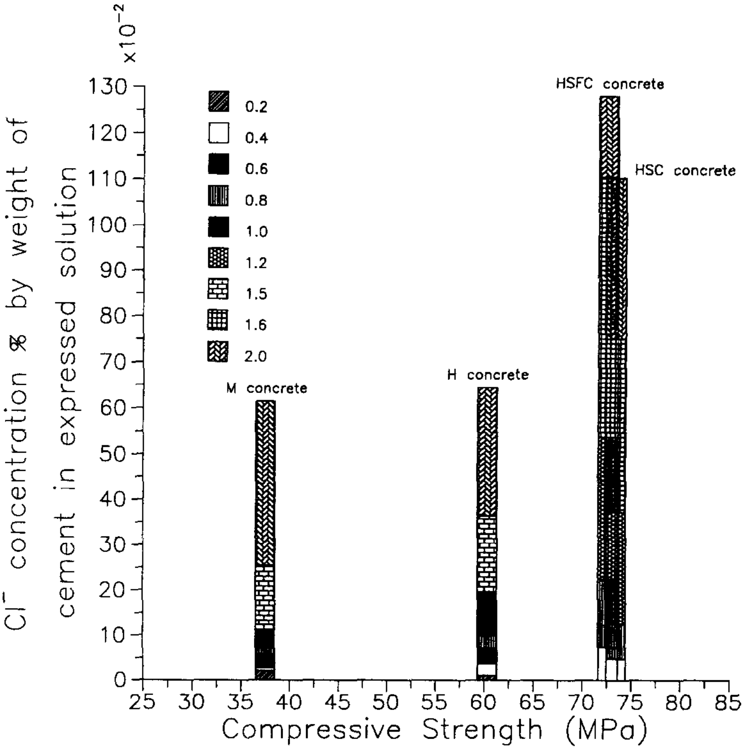

Fig. 4 - Concentration of Chloride Ions in Expressed Solution for Concretes of Different Strengths

higher risk to corrosion of steel than a lower strength concrete (with lower cement content). Accordingly, codes of practice(16) put an upper limit of chlorides in a given volume of concrete. For example, Australian Standard AS-3600 allows a maximum acid soluble chloride ion content of 0.8 kg/m^3 of concrete.

As shown in Table 3, the Cl^- ion concentration in the decanted solutions is an overestimation of the Cl^- ions in the expressed pore solution. Arya and Newman(9), while using cement paste specimens, also concluded that the water soluble chloride test will almost invariably overestimate the free chloride content of mixes containing internal and external chloride, in some cases as much as several hundred percent. For all the 24 concretes tested in this investigation, the Cl^- concentration of the decanted solutions, on the average, was about 2 times those of the expressed solution. This factor of overestimation decreased with the increase in the strength grade of the concrete and generally with the increase in added Cl^- concentration of a given concrete. The factor of overestimation varied from 8.5 to 1.6. It does not seem feasible to estimate the free chloride content of a concrete from the analysis of the decanted solutions as performed in this investigation.

On the basis of the comparison of the Cl^- contents of the concrete given in Table 3, the following two points are worth noting:

- High strength concrete which incorporated superplasticiser has invariably produced larger amount of free chloride ions than the concrete without superplasticiser. This observation was recorded in spite of the fact that the concrete with superplasticiser was generally twice as strong. Comparing medium strength and high strength concretes, it is reasonable to expect a slight rise in the residual free chlorides in the case of high strength concrete due to the increase in the cement content and thus the increase in the total chloride content which is initially added. This in itself is an indication that the amount of chlorides that can be bound to the cement hydration products does not increase proportionally with the amount of cement present, and that there is a threshold of chlorides binding capacity. However, the significant difference between the HSC and the H results while the cement content was approximately equal, and the remarkably large difference between the HSC and the M concretes, are more likely explained by the presence of the superplasticiser in HSC concrete.

- The presence of fly ash in the mixes which also were manufactured with the aid of the superplasticiser did not seem to produce any improvement in the binding of chloride ions. It is of particular interest to note here that is has been demonstrated previously that fly ash has a beneficial effect in binding chloride ions and preventing them from being free in the solution(17). It may therefore be concluded that the superplasticiser has been responsible for limiting the binding chloride ions to the cement/fly ash hydration products. The mechanism by which this effect works is not yet clear but it may be that because the superplasticiser imparts negative charges to the cement particles, these in turn repel the negatively charged chloride ions and therefore make them more freely available within the pore solution. It is believed that this observation is of particular significance if prediction of corrosion initiation due to chlorides content is the issue on hand.

Cl^-/OH^- Ratio in the Expressed and Decanted Solutions

The Cl^-/OH^- ratio for both the expressed and decanted solutions are included in Figs. 5 and 6. On closer examination, there is a great similarity between Figs. 2 and 5 and 3 and 6 respectively. That is the variation of Cl^-/OH^- ratio and the free Cl^- with the added concentration of Cl^- follows

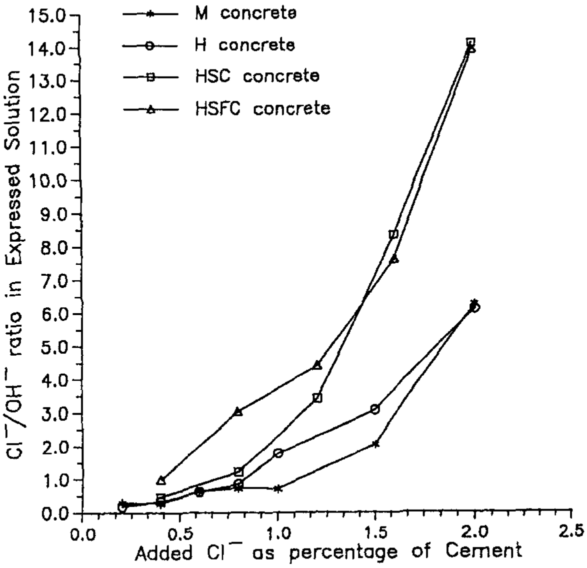

Fig. 5 - Cl^-/OH^- in Pore Solution

somewhat similar patterns. As expected, for the four grades of concretes, the ratio of Cl^-/OH^- invariably increased as the concentration of the added Cl^- increased. As shown in Fig. 5, up to 2% addition of Cl^-, the Cl^-/OH^- ratios of expressed solutions of M and H and HSC and HSFC are almost grouped together. The results tend to suggest that as the strength of the concrete increases, for a given percentage of Cl^-, the Cl^-/OH^- ratio also increases.

For the expressed solutions, the Cl^-/OH^- ratio for the various concretes varies between 0.19 to a maximum of 14.15 whereas for the decanted solution it varies between 0.04 to 1.91. The above ratio for the expressed solution in M, H, HSC and HSFC concretes varies between 0.28 and 6.28, 0.19 to 6.14, 0.46 to 14.15 and 0.99 to 13.95 respectively. According to Diamond[18] it is the

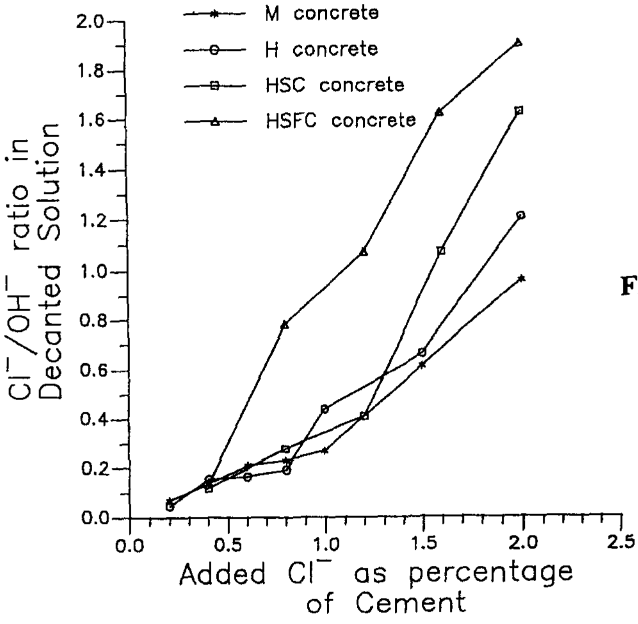

Fig. 6 - Cl^-/OH^- in Decanted Solution

conjoint concentration level of the Cl^- as well as OH^- ions in the pore solution which determines the depassivation of steel in concrete. Lambert et al(19) have suggested that for external chlorides, the Cl^-/OH^- ratio below which no significant corrosion occurs is approximately 3, but with internal chlorides, corrosion will occur at lower, but unspecified Cl^-/OH^- ion ratios. In M and H concretes, the above ratio beyond a total Cl^- content of 1% is more than one and as the Cl^- contents increase beyond 1.5%, there is a rapid increase in this ratio. In HSC and HSFC concretes, again there is a large increase in the Cl^-/OH^- ratio beyond a Cl^- addition of 0.8%. Both the HSC and HSFC concretes were dosed with a superplasticiser as well. In addition, the total cement or cementitious contents of the HSC and HSFC concretes are high. These results suggest that it is the total admixed chloride content of a concrete, rather than the percentage of the admixed chloride by the weight of cement which is critical. Since the HSC and HSFC concretes contain higher amount of the total admixed chloride (although the % of Cl^- by the weight of a given cement content may be the same), the free Cl^- content in the pore (or decanted) solution is also high.

In the decanted solutions, the ratio of Cl^-/OH^- ions is considerably lower than that in the expressed solutions. Also this difference in the above ratio increases as the chloride addition in the concrete increases. As mentioned earlier, the determination of OH^- in the decanted solution cannot be used with confidence. Therefore no importance should be attached to the value of Cl^-/OH^- obtained from the decantation method.

Conclusions

1. The OH^- concentration as determined on the decanted solution gives an erroneous and gross overestimation of the OH^- ions concentration.
2. The OH^- concentration in the pore solution seems to depend on both the grade of concrete and the concentration of the added Cl^- (as NaCl) in the concrete. Accordingly, estimation of OH^- concentration in a given concrete with given parameters does not seem feasible.
3. The Cl^- concentration both in the expressed and decanted solutions increased with the increasing contents of the admixed chloride and the increasing strength of the concrete. It seems that the higher strength concretes are likely to enhance the chances of corrosion of the reinforcement for equal concentration of the admixed chloride expressed as percent of the cement. So it is the total admixed chloride content of a concrete rather than the percentage of the admixed chloride, by the weight of cement, which is critical.
4. The Cl^- ion concentration in the decanted solutions gave an overestimation of the Cl^- in the pore solution. As the strength of the concrete increased and the admixed Cl^- increased, the factor of overestimation decreased. Accordingly, it may not be straight forward to estimate the free Cl^- of the pore solution by measuring the Cl^- of the decanted solution.
5. The presence of a superplasticiser in concretes, with and without fly ash lowered the Cl^- binding capacity of the concretes. A possible mechanism has been suggested which explains how the presence of a superplasticiser can lower the chloride binding capacity of a concrete.

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