

Research Article

The Influence of C_3A Content in Cement on the Chloride Transport

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The present study concerns the influence of C_3A in cement on chloride transport in reinforced concrete. Three modified cement was manufactured in the variation of the C_3A content, ranging from 6.0 and 10.5 up to 16.9%. The setting time of fresh concrete was measured immediately after mixing, together with the temperature at the time of initial set. For properties of hardened concrete in the variation in the C_3A , a development of the compressive strength and chloride permeation were measured using mortar specimens. Simultaneously, chloride binding capacity was measured by the water extraction method. To ensure the influence of pore structure on chloride transport, the pore structure was examined by the mercury intrusion porosimetry. As a result, it was found that an increase in the C_3A content resulted in an increase in chloride binding capacity. However, it seemed that increased binding of chlorides is related to the higher ingress of chlorides, despite denser pore structure. It may be attributed to the higher surface chloride, which could increase the gradient of chloride concentration from the surface, thereby leading to the higher level of chloride profiles. Substantially, the benefit of high C_3A in resisting corrosion, arising from removal of free chlorides in the pore solution, would be offset by increased chloride ingress at a given duration, when it comes to the corrosion-free service life.

1. Introduction

Chloride attack is one of the main causes of deterioration of reinforced concrete structures that is exposed on a marine environment or deicing salts. It is generally believed that steel embedded in concrete maintains a passive film (i.e., $\gamma\text{-Fe}_2\text{O}_3$) which protects the metal against corrosion due to the high alkalinity of concrete pore solution [1]. However, the passive film would be subsequently destroyed by chloride attack, once chloride ions reach steel. Likewise, to alert the risk of chloride-induced corrosion of steel in concrete, chloride threshold level can be defined as the chloride ion concentration at the depth of the steel which induce a breakdown of passive film and induce corrosion initiation. Consequently, chloride concentration at the depth of the steel is indicative of the corrosion risk and can be represented by different forms [2, 3].

Chloride binding is an interaction between hydration products and chloride ions. In particular, the hydration products of C_3A and C_4AF bind chlorides to form $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$ (Friedel's salt) and $3\text{CaO}\cdot\text{Fe}_2\text{O}_3$

$\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$, respectively [4]. It has been conventionally regarded that chloride binding is an important factor in the corrosion of reinforced steel, because it removes chloride ions from the pore solution. There are some literatures concerning the influence of C_3A in cement on chloride binding capacity, depending on the source of chlorides. For example, chlorides in cast were significantly removed or/and bound by C_3A in cement clinker [5, 6]. However, chlorides penetrated from an external source had no benefit of C_3A in being bound in the cement matrix [7].

Besides, binding of chloride ions has been intuitively deemed to impede chloride transport, due to immobilisation of chlorides. Increased chloride binding in a high level C_3A cement would lower chloride transport and thus the corrosion risk. However, the influence of C_3A content on chloride transport has been less experimented, to date. Glass and Buenfeld [8] developed a model to predict the chloride ingress with respect to C_3A content, ranging from 2.0 to 14.0%. Their calculation and modeling showed that an increase in C_3A content resulted in a decrease in the penetration of free chlorides, assuming that the pore structure

TABLE 1: Oxide composition of Portland cement determined by XRF (%).

	CaO	SiO ₂	Al ₂ O ₃	SO ₃	Fe ₂ O ₃
C ₃ A: 6.0%	70.3	17.4	4.0	4.2	4.1
C ₃ A: 10.5%	64.2	20.1	6.0	2.0	3.2
C ₃ A: 16.9%	62.8	19.3	8.3	1.9	3.0

is given (i.e., identical pore distribution). Thus, to ensure the C₃A influence on chloride transport, a well-arranged experiment set is required.

The present study concerns the influence of C₃A content on chloride transport. In fact, the C₃A content in cement is often limited by guidelines and standards due to heat generation in hydration. However, to secure the variation in the chloride binding capacity, a wide range of C₃A was selected in this study. Tests for fundamental properties, including compressive strength, setting time, and chloride profile, were conducted with cement containing different level of C₃A. Chloride binding capacity test was carried out to determine the chemical effect of C₃A. Simultaneously, the pore structure was analyzed by using mercury intrusion porosimetry to identify the role of C₃A in transport of chloride ions.

2. Experimental Works

2.1. Strength and Setting Time. Portland cement containing four different levels of C₃A in the clinker was manufactured by modifying Al₂O₃ and Fe₂O₃ in oxides, as given in Table 1. For a development of the compressive strength for hardened mortar, cylindrical mortar specimens (Ø100 × 200 mm) were cast; the mix ratio of the Portland cement, water, and sand (Grade M) was 1.00 : 0.40 : 2.52. After casting, the mortar specimens were cured in a wet chamber at 20 ± 2°C and subsequently measured at 7, 28, and 56 days.

Setting time was determined by the penetration resistance of fresh mortar. Standard needles (645, 312, 161, 65, 32, and 16 mm² of the area of the needles in a circular shape) were used to measure the penetration resistance at regular time intervals, since the casting of mortar has been finished immediately. After obtaining the relation of the penetration resistance with time, the initial and final set were determined; the initial and final setting time were taken as the time of penetration resistance reaching 3.4 and 27.5 MPa, respectively. Also, the temperature of fresh concrete was measured at the time of initial set by a thermocouple.

2.2. Chloride Profiling. Mortar discs (Ø100 × 50 mm) were cast and cured in a wet chamber for 56 days to secure a hydration degree enough to gain the strength, of which all the surfaces were subsequently coated by a polymer-based resin except one surface for one-dimensional chloride penetration. After immersing the specimens in a 4.0 M NaCl solution for 100 days at 20 ± 2°C, the profile of free chloride in mortars was achieved by grinding the surface with 5 mm increments. Free chloride contents in each sample were measured by the water extraction method in 50°C deionized water, followed by the

potentiometric titration against silver nitrite. By substituting the measured chloride concentration with error function solution from Fick's second law, an apparent diffusion coefficient and the surface chloride concentration were determined as follows:

$$C(x, t) = C_s \left[1 - \operatorname{erf} \frac{x}{2\sqrt{Dt}} \right], \quad (1)$$

where, $C(x, t)$ is chloride concentration at depth x after time t (%/m³), C_s is surface chloride concentration (%/m³), x is depth (mm), D is apparent diffusion coefficient (m²/s), and t is time of exposure (s).

2.3. Chloride Binding Capacity. Cement pastes were cast in a 50 mm cube mould at 0.4 of a free W/C, containing eight levels of chlorides: 0, 0.2, 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0% weight by cement. The specimens were cured for 56 days by wrapping in a polythene film to prevent a leaching-out of ions from the cement paste, which would otherwise occur in a wet curing. Then, the specimens were dried in oven at 50°C for 24 hours, ground/crushed in a mill, and then passed through a 300 μm sieve to collect dust samples. The dust samples were diluted in 50 ml of distilled water at 50°C and stirred for 5 min. Then a further 30 min of standing was applied to reach a chemical equivalent, followed by being filtered out by filter papers to obtain a solution containing chlorides then to determine the concentration of water-soluble chloride in each sample, as being regarded as free one. Once free chloride content was determined at a given total, the relation between free and bound chloride was achieved in terms of the binding isotherm : the concentration of bound chloride was defined as the concentration of chlorides subtracted free from the total one.

To ensure chemical byproducts (i.e., Friedel's salt) after the reaction of chlorides in the cement matrix, the XRD analysis was simultaneously performed. Ground cement paste containing 1.0% chloride was used representatively. The D/MAX-2500 model was employed and the diffraction pattern was performed in the range of 6–60° (2θ) at 0.1°/sec scanning rate for a 30 kV voltage.

2.4. Examination of Pore Structure. To verify the distribution of pores with the content of C₃A, the porosity of mortars containing different levels of C₃A was measured by the mercury intrusion porosimetry. To remove the pore solution remaining in mortar, the mortar sample was dried in oven at 50°C and immersed in acetone. Then, the mortar fragment obtained from the middle of the specimen was placed in a vacuum chamber to evacuate up to about 50 μm mercury (Hg). The low pressure was generated to 0.21 MPa by nitrogen gas, and then the pressure was gradually increased to 117.21 × 103 MPa at the rate of 9.1 × 103 kPa/s. The pore diameter at a given pressure was calculated by the Washburn equation as given in (2). Then, the pore volume distribution versus pore diameter was achieved in an accumulated curve.

$$d = \frac{-4\gamma \cos \theta}{P}, \quad (2)$$

where d is pore diameter (m), γ is surface tension (N), θ is contact angle (°), and P is pressure (MPa).

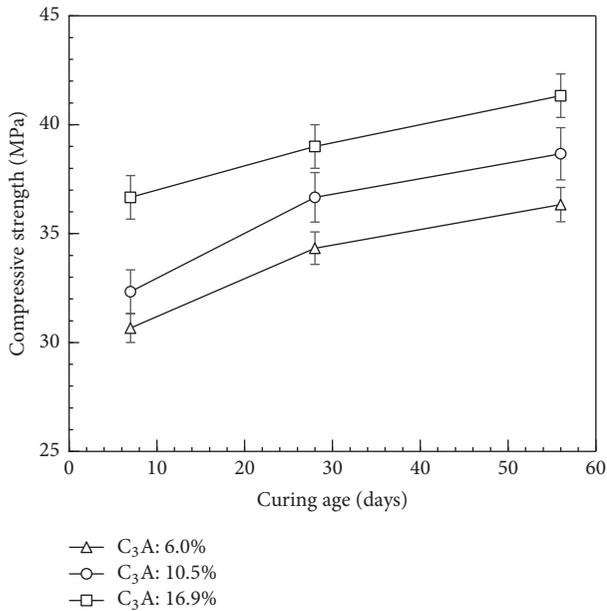


FIGURE 1: Development of compressive strength of mortar at different C_3A content in cement.

3. Results

3.1. Fundamental Properties. A development of the compressive strength for mortars containing different C_3A content in Portland cement was measured at 7, 28, and 56 days, as given in Figure 1. As expected, all the compressive strength increased with time, irrespective of C_3A content. It is evident that the rate of strength development is strongly dependent on the C_3A content in cement; an increase in the C_3A content resulted in an increase in the compressive strength at all ages. In particular, the mortar specimen containing 16.9% of C_3A in cement clinker indicated a quite high strength at an early age, of which the value accounted for about 36.7 MPa at 7 days. However, at exceeding 28 days, the compressive strength was present in the range of 39.0–41.3 MPa, as being in a small increase. In turn, the mortar specimen with the lowest C_3A content in cement imposed the lowest strength, which exceeded, however, 30.7 MPa at 7 days and then reached 36.3 MPa at 56 days. The influence of C_3A content on the strength at an early age may be attributed to the set of C_3A into the form of insoluble calcium sulfoaluminate ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$). In fact, the strength at an early age is mostly equivalent to the strength of C_3A rather than silicates (C_3S , C_2S), which would be subsequently raised by activated silicate crystal lattice in the presence of Al_2O_3 [9].

The influence of C_3A on setting and hardening of fresh concrete is very crucial. As seen in Figure 2, the penetration resistance of fresh mortar obtained immediately after casting was strongly dependent on the C_3A content in cement clinker; an increase in the C_3A in cement resulted in a rapid hardening. For example, the fresh mortar specimen containing 16.9% of C_3A was hardened at 78 min for the initial set and 202 min for the final, while mortar containing 6.0% of C_3A indicated 254 min and 434 min of the initial

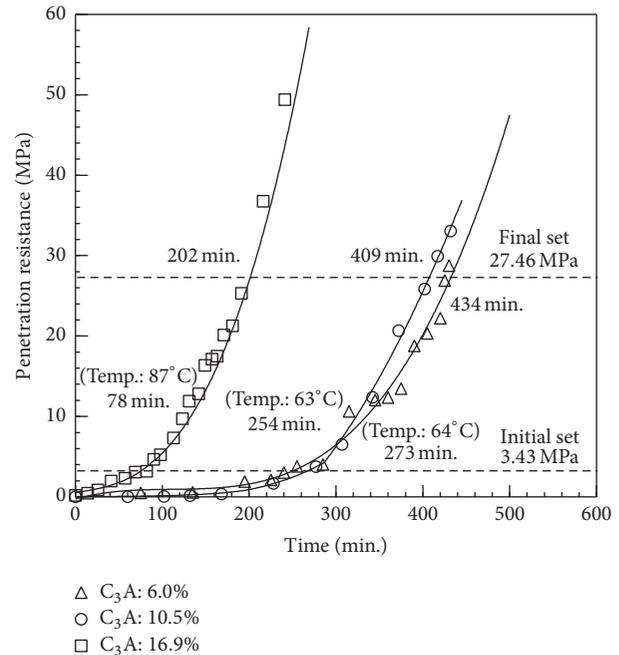


FIGURE 2: Setting time of different C_3A containing binder measured by penetration resistance of fresh concrete (initial set: 3.43 MPa; final set: 27.46 MPa).

and final sets, respectively. Apart from C_3S , significantly attributed to early setting and hardening as well, C_3A imposes the formation of AFt phases at fresh state, leading to a rapid hardening of Portland cement [10]. Substantially, the accelerated hardening of cement paste arising from the higher C_3A may accompany a rapid development of the strength. However, at a value only exceeding about 10.5% of C_3A , a rapid set was observed; at 10.5 and 6.0% of C_3A in cement clinker there was only a marginal difference in the setting time of fresh concrete. It is notable that the heat generation in hydration of fresh concrete was strongly dependent on C_3A content. At 16.9% of C_3A , the temperature at the initial set accounted for 87°C, while other level of C_3A produced a lower temperature, ranging about 63–64°C. This may arise from a rapid setting and hardening of fresh concrete, leading to densifying the cement matrix.

3.2. Chloride Transport. Figure 3 shows chloride profiles for the mortar exposed to a 4.0 M NaCl solution for 100 days, measured in water- and acid-soluble concentrations (i.e., mostly identical to the free and total concentration of chlorides) at each depth with increments of 2.0 mm. Then, the apparent diffusion coefficient of chlorides in mortar was calculated by Fick's second law. It is evident that an increase in the C_3A content resulted in an increase in the ingress of chloride at every depth, presumably implying that increased chloride binding capacity in terms of the C_3A content may accelerate the rate of chloride transport at a given exposure condition. In particular, the surface chloride concentration was strongly related to the C_3A content in cement clinker, at a lower C_3A content; for example 6.0% of the C_3A indicated 0.96 and 1.85% of the surface chloride

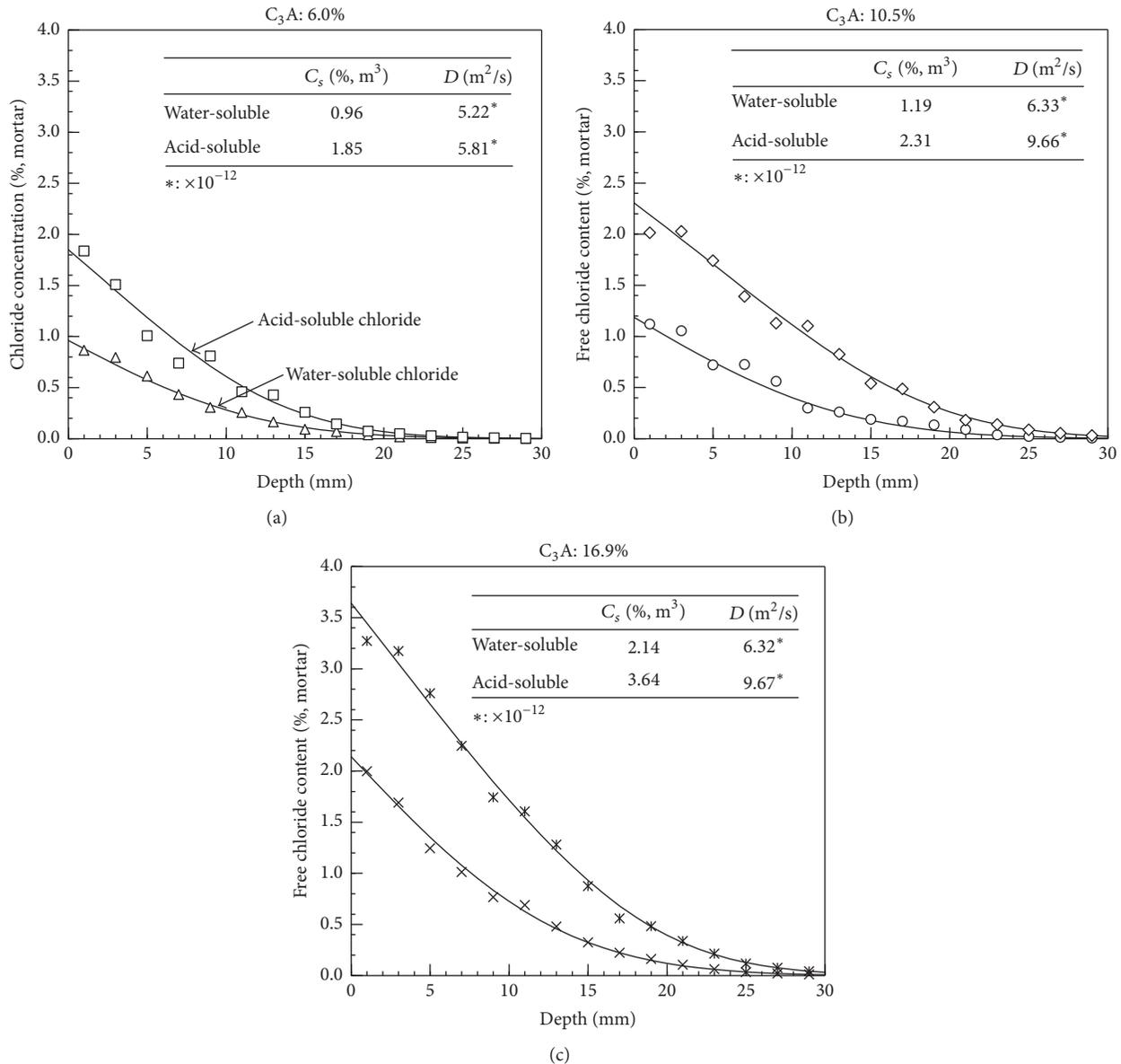


FIGURE 3: Chloride profiles for mortar with different C₃A content after 100 days of exposure to 4.0 M NaCl solution.

at water- and acid-soluble chlorides, respectively. At 16.9% of the C₃A content, the surface chloride was dramatically increased up to 2.14 and 3.64% at water- and acid-soluble chlorides, respectively. It may be attributed to the chemical balance at chlorides in the vicinity of the interfacial region between concrete and water-based media (salt solution). As the higher C₃A content imposes the increased binding of chloride ions on the surface of concrete, more chlorides would percolate to meet the equilibrium of chloride gradient. Thus, the mobility of chlorides would increase with the chloride binding capacity so that more free chlorides could be accumulated on the surface of concrete, thereby increasing substantially both the free and bound chlorides.

The diffusivity of chloride ions in concrete was strongly affected and, in fact, increased by the C₃A content. For both water- and acid-soluble chlorides, the apparent diffusion

coefficient for 6.0% of the C₃A content was ranked at the lowest range, accounting for 5.22×10^{-12} and 5.81×10^{-12} m²/s, respectively. At exceeding 10.5% of the C₃A content, however, the apparent diffusion coefficient was not much affected, indicating about 6.32×10^{-12} and 9.66×10^{-12} m²/s for water- and acid-soluble chlorides, respectively. The influence of C₃A content on the chloride diffusivity in concrete may be ascribed to either the pore structure or chloride binding capacity. The C₃A content governs the hydration process and rate, which would subsequently modify the pore distribution, in particular, capillary pores. At the high C₃A content, hydration and hardening may be accelerated at an early age then to densify the pore structure, as long as hydration heat is restricted to the range of no generation of thermal crack. Otherwise, increased chloride binding capacity, arising from the higher C₃A content, would enhance the gradient

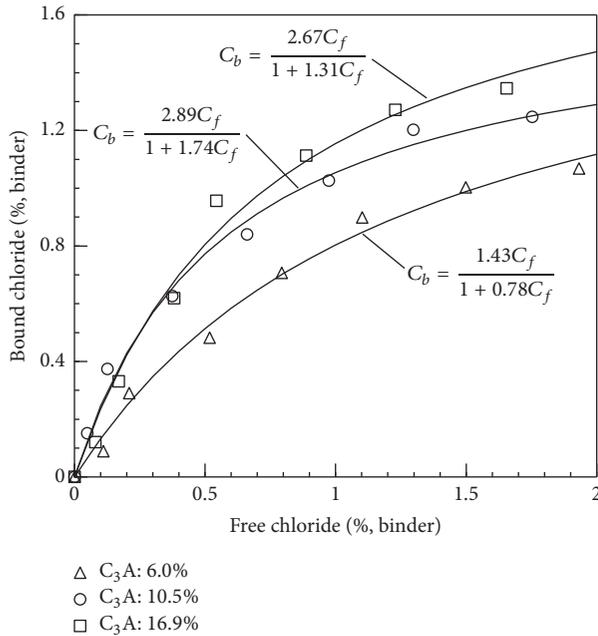


FIGURE 4: Chloride binding capacity in cement paste at different C_3A content after 56 days of curing with the variation in total chloride in cast.

of chloride concentration at depths, thereby leading to an increase in the rate of chloride transport in a diffusion-driven environment.

3.3. Chloride Binding Capacity and XRD Analysis. Chloride binding capacity, defined by the ratio of content of bound to free chloride, at different C_3A contents in cement clinker was described by the Langmuir isotherm, as given in Figure 4. The cement paste containing chlorides ranging from 0.0 to 3.0% by weight of cement was cured for 56 days and then water- and acid-soluble chloride concentrations in the paste were measured by the titration method against silver nitrate. As expected, an increase in the C_3A content results in an increase in the binding capacity at a given chloride concentration in cast. However, the chloride binding capacity was not proportionally increased to the C_3A content, because the contribution of C_3A in reacting with chlorides is limited. In fact, there are other hydration phases reacting with chloride ions to immobilize them, such as C_4AF , precipitated $Ca(OH)_2$, and C-S-H gel. Chlorides chemically reacted with hydrations (C_3A , C_4AF) are insoluble, immobile in the pore solution after forming Friedel's salt, which have been therefore regarded as an inhibitive nature with no acidification of the cement matrix. Chloride ions are simultaneously adsorbed on the surface of hydration products such as precipitated $Ca(OH)_2$, which may be usually water-soluble depending on the temperature, duration of immersion in water, and the fineness of cement paste. To avoid dissolution of adsorbed chloride in the process of titration, the temperature and immersion in water were restricted. Thus, chlorides adsorbed in the hydrations were also taken as bound ones. It is notable that the concentration of bound chloride was rapidly increased up to 1.0% of free

chlorides, while the chloride binding capacity was reduced beyond this range; the concentration of bound chloride was marginally increased with free chlorides at a given total, irrespective of the C_3A content. This may be attributed to limited binding capacity of the cement matrix in Portland cement, due to a small margin in the amount of hydration products (i.e., C_3A , C_4AF , precipitated $Ca(OH)_2$, and C-S-H gel) related to the chemical reaction with chloride ions.

The XRD curve for pastes containing 1.0% chlorides at different C_3A contents is given in Figure 5. It is evident that the peak intensity and location for hydration products were mostly identical between the three Portland cement, implying that there was marginal difference in forming hydrations: alite, belite, calcite, and portlandite. However, the variation in the peak intensity at $11.2\text{--}11.4^\circ$ of 2θ was observed, where Friedel's salt is identified. It suggests that the concentration of bound chloride is dependent on the C_3A content: in fact, an increase in the C_3A content resulted in an increase in the intensity of the peak at $11.2\text{--}11.4^\circ$. Despite limited use of the XRD analysis in a qualitative measurement, the peak intensity may indicate a relative concentration or/and amount of inorganic hydration products between different samples at the same setup of the XRD. Substantially, it can be said that an increase in the C_3A content results in an increase in Friedel's salt.

3.4. Pore Structure. The pore size distribution of mortar containing different C_3A contents in cement was measured by the MIP, as shown in Figure 6. It was seen that an increase in the C_3A content resulted in a decrease in the total porosities; for example, total pore volume for 6.0, 10.5, and 16.9% of C_3A content accounted for 0.164, 0.143, and 0.119 ml/g, respectively, due to a significant reduction in the large capillary pores, of which the range is equated to $0.1\text{--}1.0\ \mu\text{m}$. The reduction of the pore volume in increased C_3A may be attributed to filler effect of the hydration products. In fact, C_3A and C_4AF generally occupy smaller margin for the pores and moreover the interfacial porous zone within C-S-H gel, while silicates cover up the larger pores, which subsequently form the C-S-H gel at hydration. Moreover, a reduction of the C-S-H gel, presumably due to an overwhelming formation of C_3A , may accompany a further reduction of the gel pores and thus the total pore volume. Apart from the matrix strength, the high C_3A in cement may benefit in raising the compressive strength of concrete by a reduction of pores and porosity in the matrix.

4. Discussion

Conventionally, the C_3A content in Portland cement clinker was modified from about 6 to 13% at the manufacturing phase according to the required performance [11, 12]. In Portland cement, C_3A content is controlled by adjusting the contents of Al_2O_3 up to 12-13% in cement. For example, the OPC type III may have 17% of C_3A to accelerate setting and hardening, while type II and type V contain the lower C_3A content to decline the hydration heat during the curing age and prevent sulfate attack, respectively. Moreover, it has an important implication on the role of Al_2O_3 in developing the early

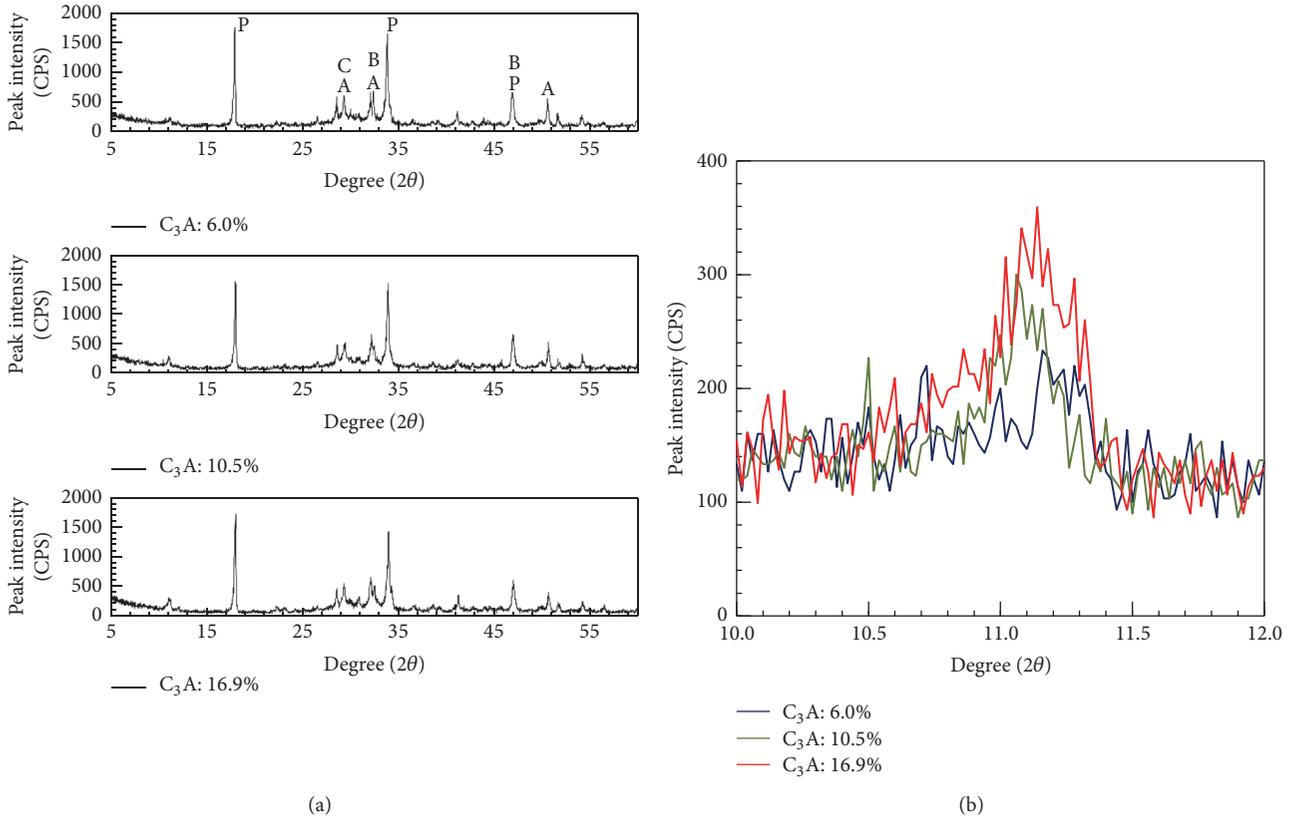


FIGURE 5: (a) XRD curves for cement paste with different C_3A content (A: alite, B: belite, C: calcite, and P: portlandite) and (b) Peak intensity for the Friedel's salt.

strength. At a high level of Al_2O_3 in cement oxides, more C_3A would be present in the clinker then to form calcium sulfoaluminate, thereby leading to a higher strength at an early age. Simultaneously, silicate activation would further enhance the strength of concrete [9]. However, the high C_3A content in cement may impose flash set or thermal cracking.

C_3A may increase the chemical reactivity with chlorides in terms of stabilization of free chlorides into Friedel's salt, of which the reactivity in turn affects chloride transport, resulting in the formation of further surface chloride. According to Glass and Buenfeld [8], the concentration of chloride in the nearer surface increased with C_3A content but decreased deeper in the concrete. An increase in the surface chloride content would arise from the binding effect, which allows the progressive build-up of higher total chloride content at increasing distances from the concrete surface. Simultaneously, increased C_3A may alternatively govern the gradient of chloride ingress at different depths, rather than pore distribution/network for ionic transport. In the present study, the higher binding capacity, induced by high C_3A content in clinker, leads to a build-up of bound chlorides that increases the surface chloride. Therefore, it can be presumed that the promotion of chloride penetration is affected by the formation of the high gradient of chloride concentration from the surface. Moreover, the hydration process of overwhelmingly increased C_3A may form denser pore structure, especially at pore size exceeding $0.1 \mu m$, leading to a reduction

of path for ionic transport. It was found that an increase in C_3A content in cement resulted in a decrease in the total pore volume, which would provide paths of chloride ions to transport. The decrease of large size pore may reduce air void in concrete, which blocks the ionic transport due to the absence of water. Furthermore, the critical path of ionic transport may be shortened by reduction of tortuosity of pore structure. Thus, the increase of C_3A gives rise to a rapid chloride transport by both progressive build-up of chloride from concrete surface and shortening of ionic transport path.

Apart from hydration process, the reactivity of C_3A with chloride ions in the concrete pore solution often forms crystallised salt in terms of Friedel's salt, which could remove, in fact, chlorides from the pore solution and thus not participate in the corrosion process. Thus, a high concentration of C_3A is regarded as an inhibitive nature against chloride-induced corrosion of steel in concrete. For example, sulfate resisting Portland cement (Type V) with a lower concentration of C_3A , usually ranging within $<5.0\%$, has increased corrosion reactivity; Page et al. [13] showed that the steel in sulfate resisting Portland cement concrete had a very high corrosion current, accounting for several hundred mA/m^2 , while ordinary Portland cement indicated the passivity. Thus, it is advised not to use sulfate resisting Portland cement in a marine environment to avoid unnecessary risk of the steel corrosion. Moreover, in the majority of the previous studies, the benefit of high C_3A cement was confirmed by their lower

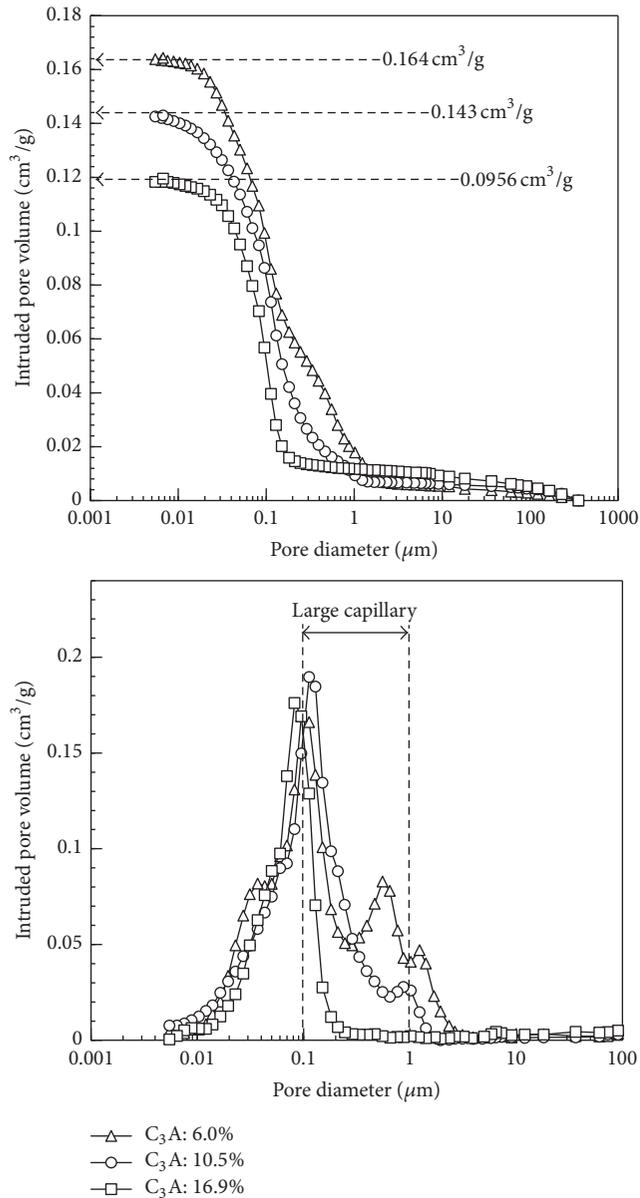


FIGURE 6: Pore distribution and pore intensity in paste to given pore diameter of each different C_3A containing binder.

corrosion rate in a beaker test and in turn a higher chloride threshold level for steel corrosion [14].

5. Conclusion

In this study, the influence of C_3A concentration in cement clinker on development of the compressive strength and chloride transport was investigated. Simultaneously, the pore structure and chloride binding capacity were experimented to explain the rate of chloride transport with C_3A content in cement. The conclusion derived from the experimental study is given as follows:

- (1) An increase in the C_3A content in cement resulted in a dramatic decrease in the setting time and an increase

in a development of the compressive strength at a given age, presumably due to accelerated hardening process. Simultaneously the temperature of fresh concrete was more or less affected by the content of C_3A . However, no thermal cracking was not observed for all specimens, irrespective of C_3A content.

- (2) The rate of chloride transport was assessed by an exposure test in terms of diffusion of chloride ions. The ingress of chloride in the higher C_3A concrete was significantly higher in the vicinity of concrete surface, due to increased chloride binding capacity, which would lead to build up of a higher chloride at a given free chloride on the surface. Due to increased chloride ingress, the diffusion coefficient was increased by C_3A , arising from the higher gradient of chloride concentration.
- (3) However, the higher C_3A in cement may be beneficial in forming denser pore structure. An increase in the C_3A resulted in a decrease in the pore volume. As the hydration of C_3A is related to a formation of expansive ettringite, the higher C_3A in cement substantially produced the denser pore structure.

Competing Interests

The authors declare that they have no competing interests.

Acknowledgments

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