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Influence of surfactants on chloride binding in cement paste

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HIGHLIGHTS

• The influence of surfactants on chloride binding in cement paste was evaluated.

• The influences of surfactants on chemical combination and physical absorption were examined.

• Chloride binding isotherms of pastes with various surfactants were measured.

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ABSTRACT

The influence of surfactants on chloride binding in cement paste has been investigated in this paper. Three surfactants, such as TEA-Lauryl sulfate (TEALS), sodium dodecyl benzene sulfonate (SDBS) and dodecyl polyglucoside (DPG) were used to represent as cationic, anionic and nonionic surfactants, respectively. The chloride binding was determined using the equilibrium method proposed by Tang Luping. X-ray powder diffractometer (XRD) was applied to characterize the phase structures of as-obtained paste specimens. It has been found that TEA-Lauryl sulfate (TEALS) increases the chloride binding, but sodium dodecyl benzene sulfonate (SDBS) and dodecyl polyglucoside (DPG) reduce the chloride binding. All the surfactants mainly affect the physical absorption of chloride ions, and exert a minor influence on the chemical binding of chloride ions. Freundlich isotherm can better describe the relationship between the amounts of bound chloride ions in cements with various surfactants and free chloride binding similar to the former addition of surfactant.

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1. Introduction

Chloride-induced corrosion of steel is a major cause of the durability of reinforced concrete structures exposed to chloride-laden environment. Due to the porous structure of concrete, the chloride ions are liable to penetrate from the environment to the surface of steel reinforcement in concrete. The intruded chloride ions can disrupt locally the protective layer formed on the steel surface in the high alkalinity of concrete. When the chloride ions reach a critical level, the protective layer will be destroyed so that the steel corrosion is initiated [1,2].

The chloride ingress into concrete involves complex interactions between the chloride ions and cement hydrates. Some chloride ions are bound onto the cement hydrates by physical absorption or chemical reaction with AFm compounds resulting in chloride-containing AFm compounds (such as Friedel's salt, 3CaO·Al₂O₃·CaCl₂·10H₂O). The others remain free in the pore

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http://dx.doi.org/10.1016/j.conbuildmat.2016.08.075 0950-0618/© 2016 Elsevier Ltd. All rights reserved. solution of concrete. It is generally believed that only the free chloride ions are able to move to the steel surface and corrode the steel reinforcement [3]. The chloride binding in the cement hydrates can lower the free chloride ion concentration in the pore solution. Hence, it can retard the process of chloride ingress, and reduce the corrosion risk of steel reinforcement. Based on this, increasing the chloride binding capacity of concrete is often considered as an alternative method to upgrade the durability [4]. In addition, the chloride binding must be correctly determined so as to predict the service life of reinforced concrete structures [5].

The chloride binding is a complex process, which can be affected by many factors, such as chloride concentration, cement composition, supplementary, cation of chloride salt, temperature, carbonation, sulfate ions, etc. So far, many works have been carried out in order to clarify the effects of the factors. The obtained results have been comprehensively and systematically presented in recent literature review [6]. Despite this, to the best of our knowledge, the influence of surfactants on chloride binding is not still investigated until now.







As a matter of fact, surfactants as chemical additives have been extensively applied to improve the quality of cement and concrete and extend their applications. The surfactants with surface activity are served as viscosity improver, water-reducing admixture, airentraining admixture and shrinkage-reducing admixture, etc. It has been indicated that the surfactants can be absorbed on the surface of cement clinker and hydrates [7,8], inducing the changes of surface properties, such as surface charge, contact angle, etc. The physical adsorption is caused mainly by van der Waals attraction and electrostatic forces between chloride ions and cement hydrates, and hence is dependent on the surface properties [9]. Therefore, the surfactants can exert an influence on the chloride binding.

This paper aims to evaluate the influence of surfactants on the chloride binding in cement paste. Three surfactants, i.e., TEA-Lauryl sulfate, sodium dodecyl benzene sulfonate and dodecyl polyglucoside are chosen to represent as cationic, anionic and nonionic surfactants, respectively. It should be pointed out that these surfactants have been accepted as air-entraining admixtures for cement and concrete. The chloride binding was determined using the equilibrium method according to the literature [10]. Besides, X-ray powder diffractometer (XRD) was applied to characterize the phase structures of as-obtained paste specimens.

2. Experimental

2.1. Materials and sample preparation

The cement applied in our work was No. 42.5 ordinary Portland cement made in China. Its oxide composition was indicated in Table 1. TEA-Lauryl sulfate (abbreviated as TEALS, $C_{18}H_{41}NO_7S$, $M_r = 415.59$), sodium dodecyl benzene sulfonate (abbreviated as SDBS, $C_{18}H_{29}NaO_3S$, $M_r = 348.48$) and dodecyl polyglucoside (abbreviated as DPG, $C_{18}H_{36}O_6$, $M_r = 348.50$) were received from Lusen Chemical Co. Ltd in China. They were chosen to represent as cationic, anionic and nonionic surfactants, respectively and used as received. Sodium chloride was applied to supply the aggressive chloride ions. All chemical reagents in this study were analytically pure. Deionized water was applied to prepare the suspension solution to carry out the chloride adsorption and desorption tests.

Cement paste samples with and without the additions of surfactants were fabricated using two water/cement ratios (w/c) of 0.35 and 0.50. They had the size of 40 mm \times 40 mm \times 160 mm. The surfactants with the contents of 0.5% and 1.0% by mass of cement were added by two ways. In one way, the surfactants were directly added to the mixing water(called former addition). In the other way, the surfactants were added to the suspension solutions of crushed cement paste particles (called later addition). Such an experimental arrangement allowed making a comparison in order to reveal the effect of surfactants on the chloride binding.

Moreover, after one day of casting in plastic molds, all the paste samples were demolded, then cured in a 95% humidity chamber at 20 ± 2 °C for three months. Subsequently, the paste samples were taken out to dry at 40 °C for three days. Then, the central region of the paste was crushed and sieved into 0.3–2.3 mm particles. The particulate specimens were dried at 40 °C in a vacuum of about 10^{-3} Pa for three days. After this, the dried specimens were transferred to an air tight container containing soda lime, and stored at 11% RH by saturated LiCl solution for ten days.

2.2. Test methods and procedure

2.2.1. Chloride adsorption

For the paste specimens with the former additions of various surfactants, 20 g particulate specimens stored at 11% RH were weighed and put into 60 mL plastic bottles under vacuum for 2 h. The bottles were then filled with approximately 40 mL of NaCl solution saturated with Ca(OH)₂. The NaCl solution had eight different concentrations of 0.005, 0.01, 0.03, 0.05, 0.07, 0.1, 0.5 and 1.0 mol/L. After this, the bottles were sealed and stored at 20 ± 0.5 °C for equilibrium. As a rule, 1–2 weeks were needed. The equilibrium of chloride adsorption was identified by monitoring the chloride concentration in the suspension until no significant decrease was observed. When the equilibrium was established, the free chloride concentration in the suspension was analyzed by means of potentiometric titration using 0.01 M AgNO₃. Besides, the amount of bound chloride ions can be determined from:

$$C_b = 35.453 * V * (C_i - C_f) / W \tag{1}$$

where C_b is the content of bound chloride ions, mg/g of the specimen; V is the volume of solution, mL; C_i is the initial chloride concentration of the solution, mol/L; C_f is the free chloride concentration at equilibrium of the suspension, mol/L; W is the mass of the dry specimen, g. Accordingly, the adsorption isotherm can be drawn from the data of C_b and C_f through a series of tests with different initial concentrations.

For the paste specimens with the later additions of various surfactants, the experimental procedure of chloride adsorption test was similar to that for the paste specimens with the former additions of various surfactants. The only difference was that the saturated $Ca(OH)_2$ solutions containing 0.5% surfactants were firstly used to fill the bottles instead of the NaCl solution. Then, the bottles were sealed and stored for one day. After this, NaCl was added at the different concentrations.

2.2.2. Chloride desorption

Desorption tests were performed for only the paste specimens with 0.50 w/c and the additions of 0.5% surfactants. After the adsorption test, the surplus solution (initially at the chloride concentrations of 0.005, 0.01, 0.05, 0.1, 0.5 and 1.0 mol/L) was filtered and the specimen with filter was moved to another bottle. Then 150 mL of deionized water saturated with $Ca(OH)_2$ (pH = 12.5) was added to the bottle. Then the bottle was stored at 20 °C for 14 days to reach new equilibrium. After this, the inside solution was analyzed in the same way described above.

2.2.3. X-ray powder diffractometer

XRD analyses of specimens (w/c = 0.50) after the desorption test with the initial chloride concentration of 1.0 mol/L were performed. The specimen was taken to grind into powder until it passed through the sieve of 0.63 mm and then vacuum-dried for three days at the temperature of 60 °C. With the D8 ADVANCEX X-ray powder diffractometer, the hydration products of dried specimens were analyzed. In the XRD test, Cu K α , 40 kV voltage, 30 mA current, scanning angle range from 5° to 50°, scanning speed of 10°/min and step length of 0.02° were used.

Oxide composition	of cement used i	in this	study (%	w/w).

Table 1

Oxide	CaO	SiO ₂	Al_2O_3	Fe ₂ O ₃	MgO	K ₂ O	Na ₂ O	SO ₃	Ignition loss
% w/w	57.27	24.99	9.32	3.11	0.86	1.03	0.15	0.98	2.16

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