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Effectiveness of Crystallization Coating Materials on Chloride Ions Ingress in Concrete

Mona Elsalamawy, Ashraf Ragab Mohamed, Abdellatif Elsayed Abosen

Abstract**—**This paper aims to evaluate the effectiveness of different crystalline coating materials concerning of chloride ions penetration. The concrete ages at the coating installation and its moisture conditions were addressed; where, these two factors may play a dominant role for the effectiveness of the used materials. Rapid chloride ions penetration test (RCPT) was conducted at different ages and moisture conditions according to the relevant standard. In addition, the contaminated area and the penetration depth of the chloride ions were investigated immediately after the RCPT test using chemical identifier, 0.1 M silver nitrate $AgNO_3$ solution. Results have shown that, the very low chloride ions penetrability, for the studied crystallization materials, were investigated only with the old age concrete (G1). The significant reduction in chloride ions' penetrability was illustrated after 7 days of installing the crystalline coating layers. Using imageJ is more reliable to describe the contaminated area of chloride ions, where the distribution of aggregate and heterogeneous of cement mortar was considered in the images analysis.

Keywords**—**Chloride permeability, contaminated area, crystalline waterproofing materials, RCPT, XRD.

I. Introduction

DETERIORATION in concrete structures depend mainly on pore system, pore size and its geometry in concrete. Permeability is the most important characteristic of the concrete structural [1], where the microstructural properties of concrete, such as the size, distribution, and interconnection of pores and micro cracks has great influence on the concrete permeability [2]. Concrete barrier systems using coating are widely used to improve durability of reinforced concrete structures in order to control and/or prevent reinforcement corrosion in aggressive environment [3]. These barrier systems may be applied on new or existing concrete structure as a preventative measure or enhancing the resistivity of water or chloride ions transportation to improve their service life [4]. The surface protection systems against ingress are classified into, hydrophobic impregnation, impregnation "pore blocker" and overlay or coating [5], [6].

The aim of this paper is to study efficiency of certain surface treatment, crystallization materials, in reducing chloride permeability in concrete. The materials used for this work are cementitious and polymer-modified cementitious coating for different commercial companies classified as crystallization materials related to their brochures. A rapid test method for the chloride permeability of concrete according to standard test method of ASTM C1202-12 [7] was used for characterization of these materials against the chloride ingress.

Abd-Ellatif Elsayed/Abosen is with the Faculty of Engineering, Alexandria University, Egypt (e-mail: ae29578@gmail.com).

II. Materials

A. Portland Cement and Aggregates

Ordinary Portland cement, CEMI 42.5 N, with a specific surface area of 377 m^2/kg was used. Its chemical composition is shown in Table I. A well-graded coarse aggregate of nominal max aggregate size of 19 mm and fine aggregate with fineness modulus of 2.62 were used. The coarse to fine aggregate ratio used in this study was 1.50: 1 by weight.

TABLE I
CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF CEM I 42.5 N

Chemical Compositions	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3
Percentage (wt. %)	19.67	4.78	4.73	60.14	2.63	0.41	0.47	2.99

Calculated compound composition	
Tricalcium Silicate (C_3S)	47.9
Dicalcium Silicate (C_2S)	20.2
Tricalcium Aluminate (C_3A)	4.7
Tetracalcium Aluminoferrite (C_4AF)	14.4

B. Crystalline Waterproofing Coating Materials

Six different commercially crystallization coating materials designated as C1 to C6 were investigated. These materials are cement-based materials, while C6 is polymer modified cementitious material. The claimed mechanism of these coatings is crystallization forming cementitious materials. In order to determine the chemical compositions of these coating materials an XRD technique was conducted. Fig. 1 shows an example of XRD test results of these materials; while XRD analysis for all studied materials are given in Table II. From these results, the used materials are composed of sand, cement and sodium carbonate with different chemical compositions. It should be noted that C2 has a large amount of magnesium oxides compared with the other five materials and the XRD for C6 was conducted for the powder component.

III. Experimental Program

A. Selection of Concrete Mixtures

The less permeable quality concrete mixture, with water-cement ratio (w/c) of 0.50 and cement content of 300 kg/m^3, were selected as recommended in EN 206-1 [8], as a minimum requirements for structure exposed to chloride-induced corrosion, exposed to airborne salt but not in direct contact with sea water. Approximately 1.5 ~ 2% type F chemical admixture [9] was used to achieve the constant slump value equal to 180 ± 20 mm.

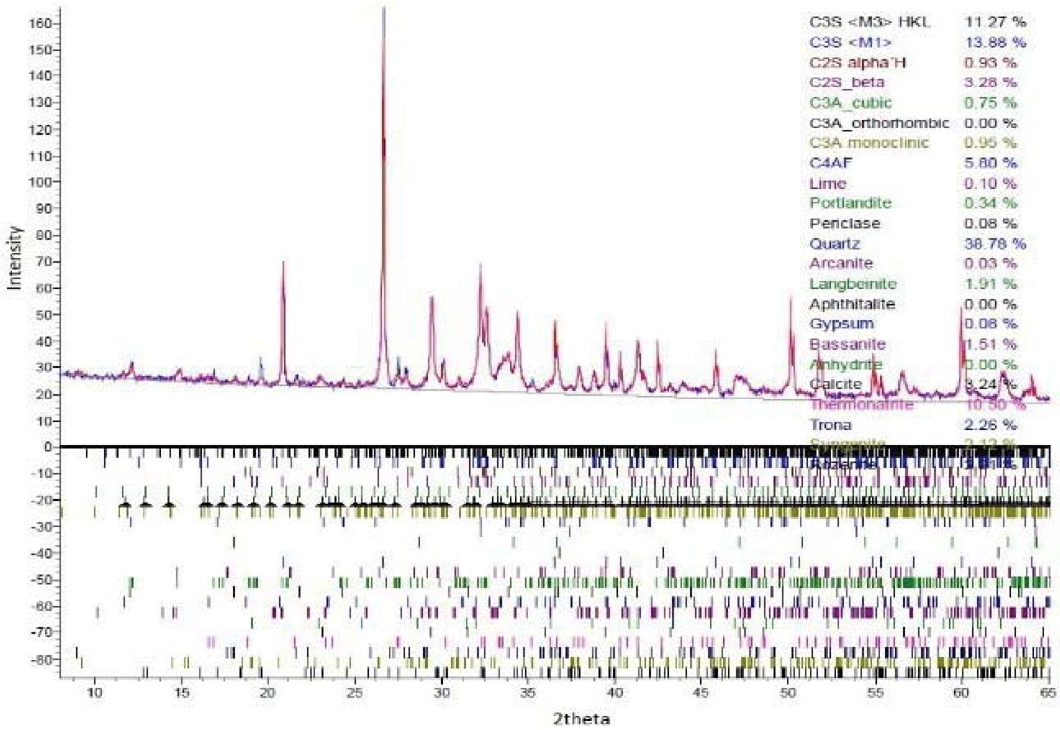

Fig. 1 XRD test results For C1

TABLE II
XRD ANALYSIS OF DIFFERENT CRYSTALLINE WATERPROOFING COATING MATERIALS

Items	C1	C2	C3	C4	C5	C6
SiO_2	44.86	31.54	37.11	41.98	36.13	50.77
Al_2O_3	2.48	6.09	2.24	3.68	3.58	1.94
Fe_2O_3	2.09	2.41	1.18	2.07	2.73	1.45
CaO	27.39	30.60	33.39	37.55	41.98	33.58
MgO	0.88	4.88	0.53	0.40	0.89	0.91
SO_3	1.96	1.20	1.08	1.66	2.51	1.25
K_2O	0.43	0.53	0.51	0.19	0.13	0.15
Na_2O	7.30	8.40	4.91	3.43	1.03	0.23
TiO_2	0.15	0.25	0.12	2.56	0.25	0.15
P_2O_5	0.04	0.05	0.05	0.07	0.10	0.04
MnO	0.11	0.06	0.03	0.04	0.04	0.02
SrO	0.04	0.07	0.03	0.05	0.07	0.33
Cr_2O_3	0.00	0.01	0.01	0.01	0.01	0.00
ZnO	0.01	0.06	0.03	0.03	0.01	0.00
Loss on Ignition	11.67	13.86	18.43	6.56	10.64	8.98

B. Test Program Overview

The age of concrete and curing conditions were investigated to illustrate the performance of crystalline coating material. To investigate these parameters, the crystallization coating materials were installed at different concrete ages, then subjected to different curing conditions.

All concrete mixtures were subjected to 7 days wet treatment then divided into two groups: G1 and G2. The first group "G1" was exposed to the atmosphere at room temperature for four months after that the crystalline coating materials were installed. For the second group "G2", the layers of crystalline coatings were applied immediately after initial treatment; two cases of wet treatment and air treatment were studied. All the crystalline coating layers subjected to air treatment were water sprayed for 7 days immediately after installation.

This research illustrates the effect of using the crystalline coating materials layers and its age with the different concrete age on concerning with the chloride ions transportation resistivity.

C. Specimens Preparation

For RCPT test, cylinder specimens (100 mm diameter and 200 mm height) were cast. Cured cylinders were sliced parallel to the cylinder top into 50 mm height according to ASTM C1202-12 [7]. The coating layers were installed on one clean face. It was installed using a brush as per manufactures instruction with water to coating materials ratio of 1:2 except for C6 there were two component (A, powder) and (B, liquid) mixed together by 1:4 ratio.

IV. METHODOLOGIES

A. RCPT

The procedure followed for the RCPT is the same as described in ASTM C1202 [7], where all specimens are soaked in water for 24 hours, then the 50 ± 5 mm mm-thick specimen was placed between two acrylic cells. One cell was filled with NaOH (0.3 N) aqueous solution and the other with 3.0% NaCl aqueous solution. The cells were connected to a power supply (60 Volt) for six hours. The test set up is shown in Fig. 2. The current and voltage are recorded every 30 minutes throughout the test duration: "6 hours". The total charge passed was used to specify the concrete chloride permeability according to the limits standardized by ASTM

C1202 [7]. The test was conducted after 1, 3, 7 and 28 days of installing the coating layer for G1 and after 28 days for G2. Fig. 3 shows an example of the test results, the current (mA) and time (min) relation, for concrete mixtures of (G1) after 7 days (coating layer age).

B. Contaminated Area with Chloride Ions

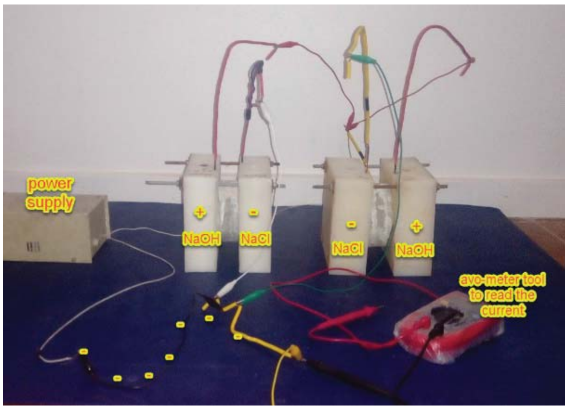

Fig. 2 RCPT setup

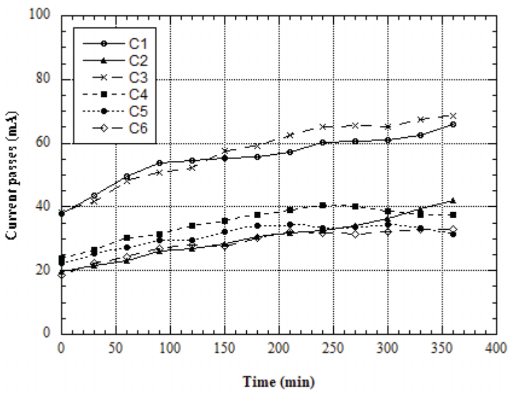

Fig. 3 Current (mA) throughout the test for G1 after 7 days

The contaminated area with chloride ions were investigated using a chemical identifier, 0.1 M silver nitrate $AgNO_3$ solution. Using silver nitrate solution was considered as the most common methods [10]. After the RCPT test was conducted the specimen was split and immediately sprayed with the chemical identifier. White color was formed by reaction between silver nitrate and chloride ions due to formation of silver chloride AgCL whereas, brown color zone means that this zone is chloride free zone due to formed Ag_2O [11]. Chloride ions' penetration depth and the contaminated area were measured using ImageJ software [12], it is open source Java-based software developed by National Institute of Health (NIH). Fig. 4 shows contaminated area and chloride ions penetration depth, where the penetration depth was calculated as the average of the 10 reading.

V. Results and Discussion

A. RCPT Result

Fig. 5 presents the calculated coulombs for the used materials under different conditions, according to ASTM C1202-12 [7], after 28 days of installing the coating layers. These results show that, the most effectiveness of the crystallization coating materials were investigated only with the old concrete (G1), where these materials satisfied the very low chloride ions penetrability [7]. On the other hand, using these materials, with the early age concrete (G2), under different curing condition, did not achieve low penetrability of chloride ions.

Fig. 6 illustrates the relative coulombs for different crystallization coating materials used in this research. The results show that, for G1, the significant reduction in coulombs for C2, C4, C5 and C6 was 73%, while about 50% for C1 and C3. For G2, the reduction was about 25% for the used materials under both curing conditions. It is very important to illustrate that, the curing procedure does not play an important role in chloride permeability with the early age concrete, while the concrete age has a pronounced effect.

Fig. 7 shows the relative charge in Coulombs related to uncoated concrete specimens with coating age for G1. It should be mentioned that, the day zero represents the age of the end of 7 days spraying water curing for the coating layers. It was clear, as shown in this figure, that the greatest rate of reduction in chloride ion penetrability (Coulombs) was investigated from three to seven days. The charges passed through the specimens decrease as the coating layers' ages increase and the significant reduction in chloride ions' penetrability was investigated after 7 days for C2, C4, C5 and C6.

B. Chloride Ions' Penetration Depth and Contaminated Area

Fig. 8 shows that the relation between the depth of chloride ions penetration and calculated surface area with total charged passed in Coulombs. These results illustrated that there is a weak correlation between the depth of chloride ions penetration and the coulombs. This weak correlation may be due to that crystalline materials contain a considerable amount of micro minerals. This observation conforms to previous researches who postulated that no correlation between depth of penetration and total charge passed in concrete with silica fume [13], [14].

The correlation between depth of penetration and calculated contaminated area, using imageJ software, with total charged passed in Coulombs from RCPT test is almost the same correlation as shown in Fig. 8 where many variables such as distribution of aggregate, heterogeneous of cement mortar and color variation of concrete were considered. Therefore, using images analysis method, imageJ software, is more reliable to describe the penetration depth and contaminated area of chloride ions. On the other hand, recent researches [15]-[17] noted that it was more effective to measure the area of surface penetration of chloride ions instead of chloride penetration depth.

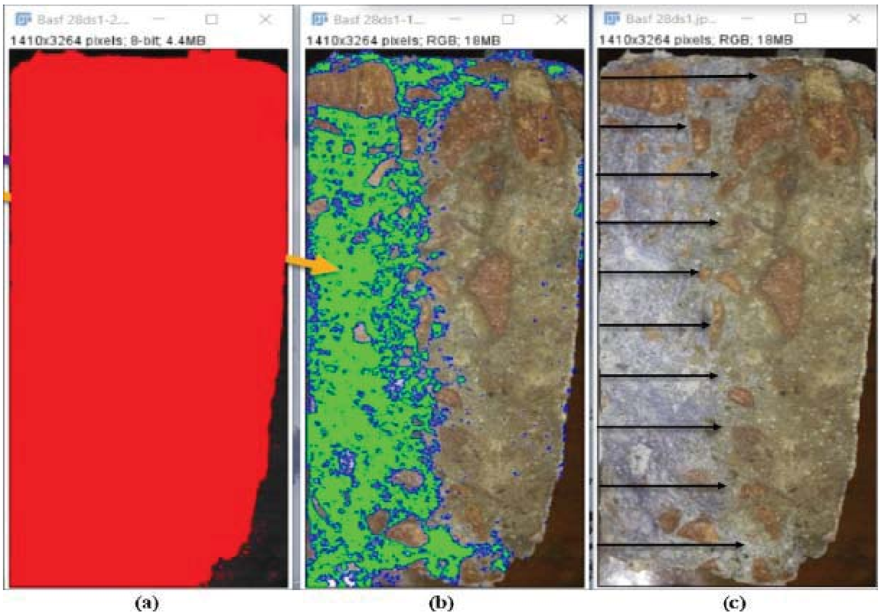

Fig. 4 (a) Total surface area of specimen, (b) the blue and green part of image represented the surface area of chloride ions penetration and (c) ten reading of the chloride ions depth on the original image

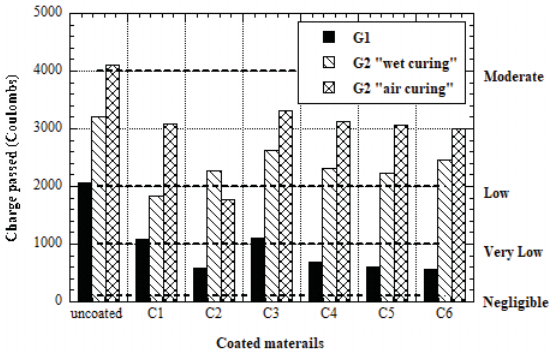

Fig. 5 Charge passed (coulombs) for different surface coated materials after 28 days for G1& G2

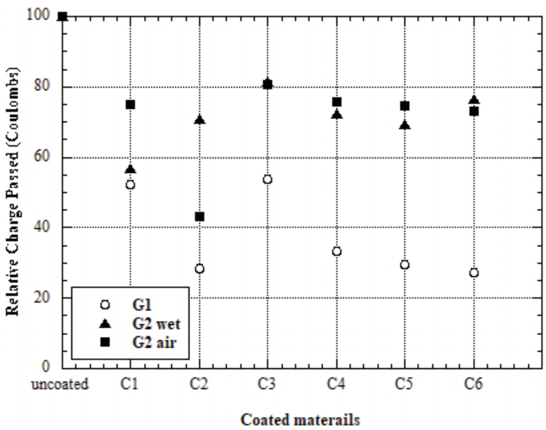

Fig. 6 Relative Charge (Coulombs) for different surface coated materials after 28 days

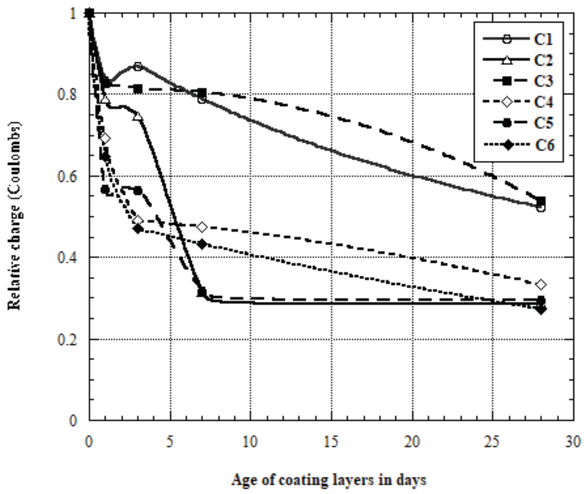

Fig. 7 Relative Charge (Coulombs) versus age of different surface coated materials "G1"

VI. Conclusion

Based on the results analysis conducted in this paper, the following conclusions can be drawn.

1- The concrete age has a pronounced effect on the effectiveness of the crystalline coating materials.
2- Using crystalline coating materials with the old age concrete satisfied the very low chloride ions penetrability.
3- Using C2, C4, C5, C6 in first group G1 achieved 73% reduction in Coulombs while this reduction was 50% for C1 and C3.
4- The curing condition, wet and air, does not play an important role in chloride penetrability with early age concrete.

5- The greatest rate of reduction in coulombs was investigated on three or seven days of the coating layers ages.
6- Using images analysis method, imageJ software, is more reliable to describe the contaminated area and penetration depth of chloride ions.
7- A weak correlation (R^2) of (0.58 and 0.50) was investigated between the coulombs and both of the chloride ions penetration and the contaminated area respectively.

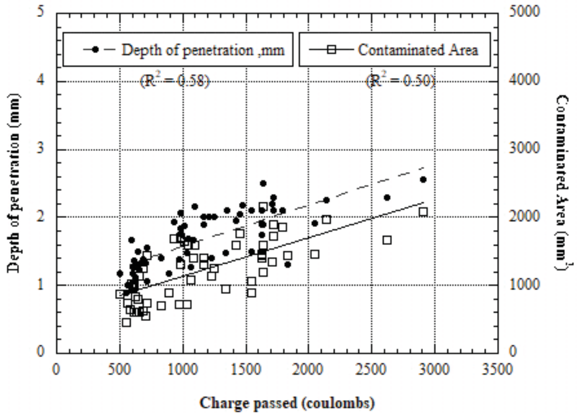

Fig. 8 Correlation between charges passed and depth of penetration and Surface area for concrete with crystalline waterproofing coating materials

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