CORRELATION OF CHLORIDE SOLUTION ABSORPTION-TIME IN PRE-CONDITIONED DCC CUBES

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Abstract: *Concrete structures were deterred due to chloride ingress which causes premature deterioration of steel reinforcement. Chloride ions intrusion into the reinforced concrete structures can lead to steel reinforcement corrosion in the presence of oxygen and moisture. Chlorides ingress due to water can permeable through the concrete matrix which deteriorates the embedded steel reinforcement. It's needed to quantify the chloride absorption in the concrete cubes. The present research work was made an attempted to interpret the chloride absorption to characterize the different mixtures design for in case of pre-conditioned (dry) concrete cubes which is saltly ponded with chloride solution for about 160 days at 10% NaCl solution. Seventy-two concrete cubes (100 mm^3) with different grades of concrete (25-40 N/mm^2) were prepared to evaluate the chloride absorption. It has been concluded from the results that, the chloride absorption was increased in designed mixtures type for longer time duration. Similarly, the average chloride absorption was decreased in an impregnation DCC cubes as when compared to control DCC cubes for constant higher compressive strength and varied slump value. It's also true of for in case varied compressive strength and constant slump value. Average chloride absorption was increased in impregnation DCC cubes for lesser compressive strength and constant slump value as when compared to constant higher compressive strength and varied slump value. Chloride absorption also decreased with increased compressive strength and constant slump value. It's possible to correlate mass gain (chloride absorption)-time relationship by logarithmic expression for in case of control/impregnation DCC cubes at different time intervals (31, 61, 91, 121, and 160) days.*

Keywords: Water-cement ratio, chloride ingress, impregnation, solvent/water based impregnate, dry condition

1.0 Introduction

The concrete superstructures, steel structures, and marine structures may deteriorate when they are exposing to de-icing agents. The de-icing agents were ingresses into the pores of concrete structure in modify the profile of cementitious structure. The interaction between the de-icing agents and the cementitious matrix may result in the deterioration of concrete structures [Jones, 2013]. Physical damage can occur due to exposure of concrete with a high degree of saturation like freeze-thaw cycles [Li, *et al*, 2012], scaling of concrete surfaces [Jacobsen, *et al*, 1997], enhancement of an internal stress due to salt crystallization in the pores of concrete [Scherer, 1999], and corrosion of reinforcement due to chloride-based de-icing salt is used [Wang, *et al*, 2014]. Physical attack due to de-icing salts and chemical reaction between the matrix and the de-icing salts has been investigated. De-icing salts usage can cause damage in cementitious materials [Marchand, *et al*, 1994]. This may be cause by the formation of Friedel's salt, Kuzel's salts [Collepardi, *et al,* 1994]], and calcium oxychloride, variations in the pore solution properties [Farnam, *et al*, 2014], and changes in the microstructure of hydration products [Pigeon and Regourd, 1986]. De-icing salt solution like calcium hydroxide which causes leaching in turn leads to an increase in permeability and a reduction of concrete alkalinity [Muethel, 1997]. De-icing salts have different chemical and physical interactions with cementitious materials. The usage of NaCl de-icing salt increases freeze-thaw damage and this damage has been described by an unexpected phases and creation of osmotic pressures [Farnam, *et al,* 2014]. Concrete exposed to de-icing salts indicated changes in the concrete microstructure and accompanied by a severe cracking and deterioration [Collepardi, *et al,* 1994]. The concrete infrastructures were deteriorating in different regions of the world without satisfying the stipulated service life. Therefore, there is a need to predict service life of concrete structures. In fact, chloride concentration is a major cause of deterioration of concrete structures. Because of this concrete deterioration, it may lead to concrete cover deteriorate, and reduce load carrying capacity. It may lead to pre-mature deterioration of concrete infrastructures and roads due de-icing salts. Bridge-decks were simultaneously expose to wetting-drying condition and, it has subjected to direct impact as well as repeated loading by continuous flow of traffic. Almost all the concrete structures were working under dry conditions. Researchers have made their attempt to study transport of chloride in concrete under wet conditions. In fact major diffusion models are developed to the concrete structures by considering wet condition. An experimental study is performing on the influence of water absorption in ordered to evaluate the effectiveness of durability of concrete by researchers [Zhang, and Zong, 2014].

It is confirm from results that the most significant effect of sorptivity on long-term chloride ingress to concrete is its effect on surface chloride content. It has decided to consider an effective amount of absorption when modelling chloride ingress under cyclic wetting and drying conditions. It is also possible from research work to produce higher surface chloride contents (0·29–0·62%) that would lower the time to corrosion using the cover depths recommended in the code. Its confirmed long time ago that [Zhao, *et al*, 2008], young and uncontaminated concrete can be surface impregnated by liquid silanes in order to provide a protective barrier against ingress of chloride ions and moderate chloride content allows to apply surface impregnation of silanes successfully as a protective measure as well as to avoid further chloride ingress. It is also confirm that, higher chloride concentration and low water-cement ratio make surface impregnation more difficult. It has confirmed that deep impregnation of the concrete surfaces with water repellent agent's forms an efficient and long lasting barrier with respect to chloride ingress [Wittmann, *et al,* 2006]. In this way, service life of RC structures constructed in marine climate can be significantly extended for long time duration. It is cite by investigators [Brandt, 2009] that, the reinforcement corrosion increases volume of corrosion products, which in turn leads to cracking, and spalling of concrete. Furthermore, chloride concentration together with frost attack can cause another form of concrete deterioration such as concrete scaling. As confirmed that [Hall, 1994], the moisture retention function is necessary for the modelling of moisture flow and transport of chlorides in concrete. Chloride diffusion can only occur for a continuous water phase is present in the capillary pores of concrete to provide a path for diffusion. For in case of dry concrete, the diffusion process is lesser due to number of water filled pores decreases and that decreases the continuity of pore solution [Saetta, *et al*, 1993]. Effective diffusion coefficient is no longer a constant but a function of saturation [Garboczi, 1990] and therefore cannot be describe by simple diffusion theory under dry conditions. This is noted by researchers that [Vriesl, *et al,* 1998], hydrophobic treatment makes a concrete surface absorb lesser water and chloride. Corrosion which had already been started before the application of the hydrophobic agent was not influence by hydrophobic treatment. It has also shown that, long term absorption tests with drinking and salt water showed significantly less absorption by hydrophobic concrete. Furthermore, its highlighted by researchers [Jacob, *et al,* 1998] that, hydrophobic agents could be effective for at least 10 years when applied to a 6-month-old concrete façade provided that, the concrete substrate needs to have a minimum age of 28 days or more. Therefore there is a need to investigate about the deterioration of concrete structures due to reinforcement corrosion has now become a day to day growing problem in recent years at all over the world in cold countries region. Considerable resources were used to rehabilitate deteriorated structures around the world. In addition to that, consequently, an extensive research [McCarter, 1996] has been conduct to evaluate the effectiveness of surface treatment materials to protect concrete, hydrophobic impregnations are the least harmful to concrete appearance, mainly inhibiting capillary water absorption of the concrete.

2.0 Research Objectives

The chloride absorption has received greater attention which is a major problem for concrete durability. Pilot program is made an attempt to interpret the chloride absorption to characterize the concrete mixtures design for in pre-conditioned dry concrete cubes. Objectives of the research work are to examine the influence of dry conditioned concrete cubes on the results of chloride absorption with different mixtures proportion. Seventy-two concrete cubes with grades of concrete (25-40 N/mm^2) were prepared and evaluate the chloride absorption under specified exposure condition. For to characterize chloride absorption two sets of constraints were implemented in the present research work as slump, and w/c ratio value varied with constant compressive strength as in the first case and compressive strength, and w/c ratio value varied with constant slump as in the second case.

3.0 Experimental program

In the present research work, six different mixtures type were prepared in total as per [BRE, 1988] code standards with concrete cubes of size (100 mm^3). Three of the mixtures type were implemented with a compressive strength 40 N/mm^2, slump (0-10, 10-30, and 60-180 mm), and w/c (0.45-0.44-0.43). These mixtures were designate as M1, M2, and M3. Another Three of the mixtures type were made with a compressive strength (25 N/mm^2-30 N/mm^2- 40 N/mm^2), slump (10-30 mm), and w/c (0.5 0.45, and 0.44). These mixtures were designate as M4, M5, and M6. The overall details of the mixture proportions were represent in Table.1-2. Twelve concrete cubes were casted for each mixture and overall Seventy-two concrete cubes were casted for six types of concrete mixture. The coarse aggregate used was crush stone with maximum nominal size of 10 mm, grade of cement 42.5 N/mm^2 and, fine aggregate used was 4.75 mm sieve size down 600 microns for this research work. Water based (WB) and Solvent based (SB) impregnate materials were used in this present research work. To avoid criticizing one particular brand of impregnation materials and for confidentiality facts, the names of the products used could not be disclosed. WB is water borne acrylic co-polymer based impregnation material(less hazardous and environmental friendly). It is silicone and solvent free and achieves a penetration of less than 10mm. SB consists of a colourless silane with an active content > 80% and can achieve penetration > 10mm.

Table: 1 (Variable: Slump & W/C value; Constant: Compressive strength)

Mix ID	Comp/mean target stg,N/mm^2	Slump (mm)	w/c	C (Kg)	W (Kg)	FA (Kg)	CA (Kg) 10 mm	Mix proportions
M1	40/47.84	0-10	0.45	3.60	1.62	5.86	18.60	1:1.63:5.16
M2	40/47.84	10-30	0.44	4.35	1.92	5.62	16.88	1:1.29:3.87
M3	40/47.84	60-180	0.43	5.43	2.34	6.42	14.30	1:1.18:2.63

Table: 2 (Variable: Compressive strength & W/C value; Constant: Slump)

Mix ID	Comp/mean target stg, N/mm^2	Slump (mm)	w/c	C (Kg)	W (Kg)	FA (Kg)	CA (Kg) 10mm	Mix proportions
M4	25/32.84	10-30	0.50	3.84	1.92	5.98	17.04	1:1.55:4.44
M5	30/37.84	10-30	0.45	4.27	1.92	6.09	16.50	1:1.42:3.86
M6	40/47.84	10-30	0.44	4.35	1.92	5.62	16.88	1:1.29:3.87

3.1 Salt ponding test

The chloride absorption tests were conduct out on concrete cubes of size (100 mm³), and tested as per code [BS: 1881-122]. Concrete specimens were cured before subjected to the salt ponding test for about160 days. Concrete specimens were oven dried to constant mass at 105±5⁰C for 72±2 hours, after that stored in airtight containers before subjected to testing. The chloride absorption test with 10% Nacl solution is carried out on pre-conditioned dry concrete cubes of size (100 mm³) which is fully submerged and noted their weights at each time interval for about 160 days. For chloride absorption test, totally 72 concrete cubes were casted, out of which 36 control concrete cubes, and 18 solvent based concrete cubes as well as 18 water based concrete cubes. The interaction between moisture diffusion and chloride penetration in concrete affects the durability of RC structures. Moisture and chloride ions are two necessary conditions for the onset of corrosion of rebar in concrete. In case of concrete is fully saturated and dominant mechanisms for both chloride diffusion and moisture diffusion is the concentration gradient of chloride. Chloride concentration gradient drives not only the chloride penetration but also the moisture movement in the concrete. The concrete is partially saturated, and the moisture concentration gradient results in the moisture penetration as well as the chloride diffusion and in this case both concentration gradients are driving forces. Thus in the present research work chloride absorption test was carried out on pre-conditioned dry concrete cubes in order to evaluate the effectiveness of two impregnation materials namely solvent/water based impregnation material respectively. In turn it's possible to interpret the effectiveness of impregnation (SB/WB) concrete cubes with control DCC concrete cubes for designed six mixtures type [Balakrishna, *et al*, 2018].

4.0 Discussion about Results

Thus in the present research work chloride absorption test was carried out on pre-conditioned concrete cubes (100 mm³) such as dry conditioned concrete cubes in order to evaluate the effectiveness of two impregnation materials namely solvent/water based impregnation material respectively. It's observed from results that (DCC control concrete cubes), the chloride absorption was slightly higher as when compared to SB and WB impregnation concrete cubes for in mixtures type (M1-M3). Chloride absorption was found to be slightly more as when compared to higher compressive strength for in case of mixtures type (M5-M6) at lower compressive strength and constant slump value. The average chloride absorption for in case of DCC concrete cubes is increased at 61th, 91th, 121th, and 160th days as when compared to 31th day in control concrete cubes, solvent based impregnation concrete cubes (SB), and water based impregnation concrete cubes (WB) in mixtures type (M1-M6) respectively. In the same way, the chloride absorption was decrease in solvent-based impregnation concrete cubes as when compared to water based impregnation concrete cubes. It's possible to correlate mass gain (chloride absorption)-time relationship by logarithmic expression for in case of control/impregnation DCC cubes at different time intervals (31, 61, 91, 121, and 160) days respectively as representing in the (Figs.1a-6c). From this relationship between chloride absorption and time, it's possible to determine chloride absorption at any specified time duration in the concrete mixes design for in the case of control/impregnation concrete cubes.

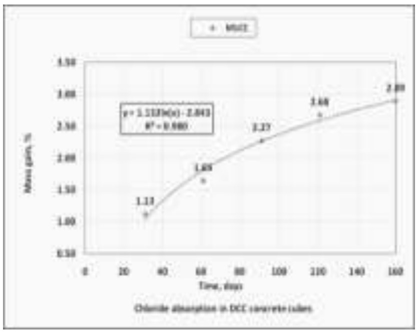


Fig.1a Mass gain-time in Mix type 1

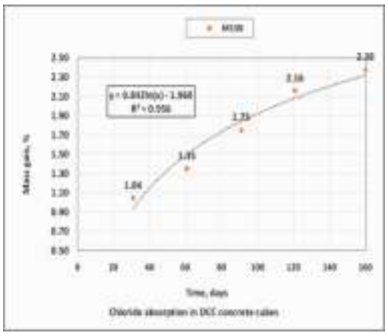


Fig.1b Mass gain-time in Mix type 1

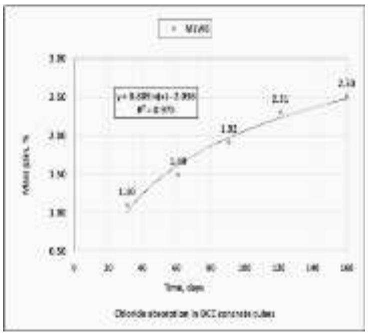


Fig.1c Mass gain-time in Mix type 1

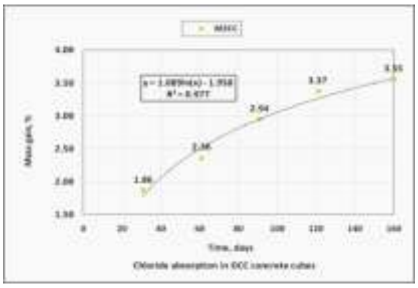


Fig.2a Mass gain-time in Mix type 2

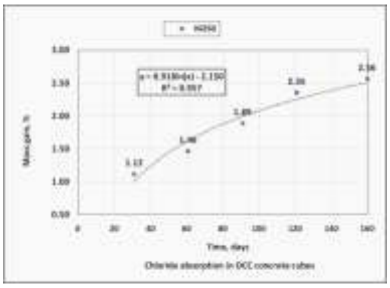


Fig.2b Mass gain-time in Mix type 2

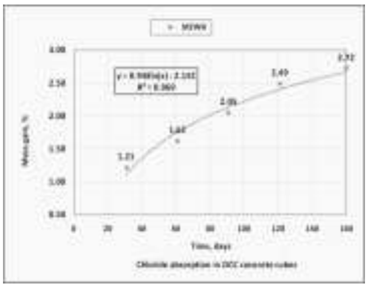


Fig.2c Mass gain-time in Mix type 2

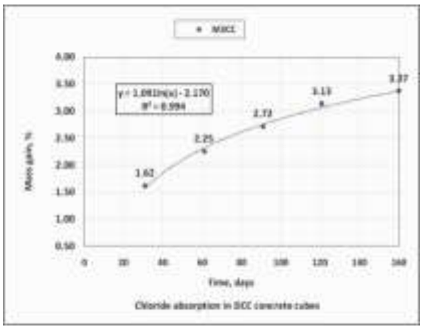


Fig.3a Mass gain-time in Mix type 3

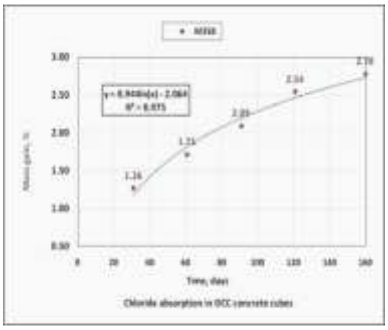


Fig.3b Mass gain-time in Mix type 3

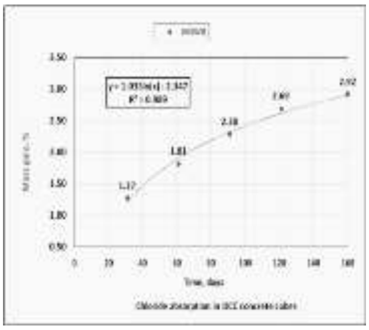


Fig.3c Mass gain-time in Mix type 3

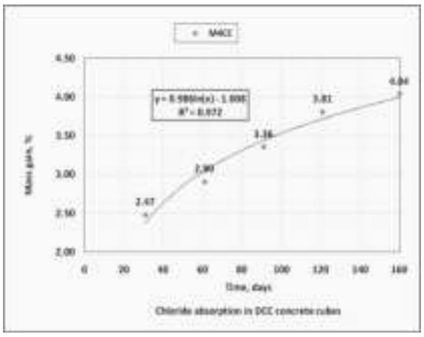


Fig.4aMass gain-time in Mix type4

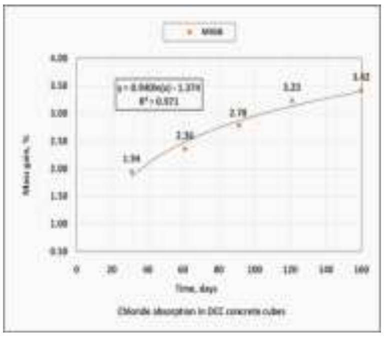


Fig.4bMass gain-time in Mix type4

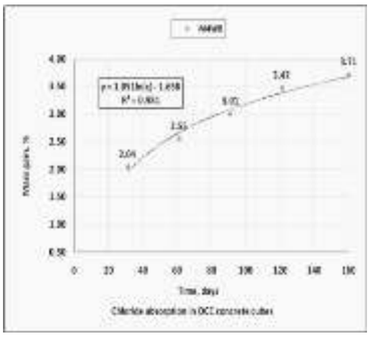


Fig.4cMass gain-time in Mix type4

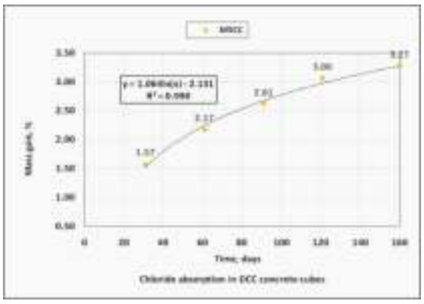


Fig.5a Mass gain-time in Mix type 5

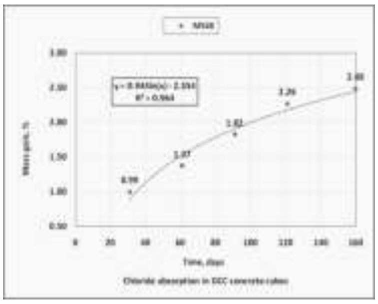


Fig.5b Mass gain-time in Mix type 5

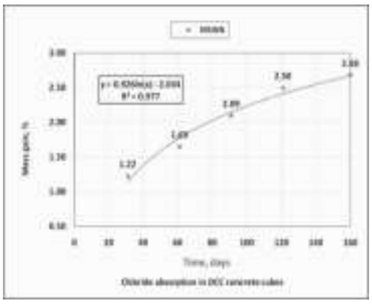
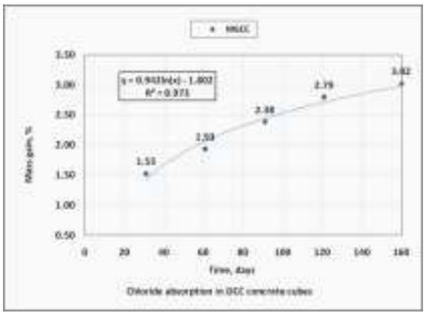
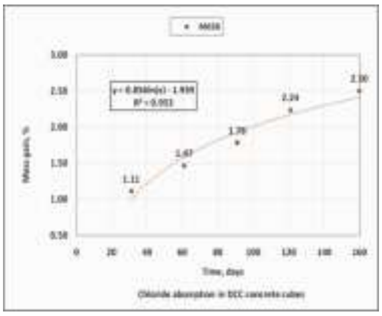


Fig.5c Mass gain-time in Mix type 5





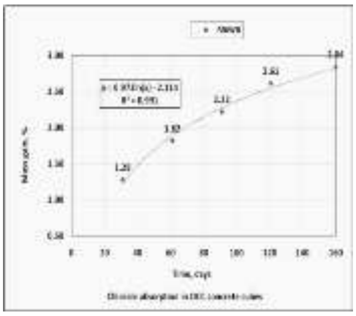


Fig.6a Mass gain-time in Mix type 6

Fig.6b Mass gain-time in Mix type 6

Fig.6c Mass gain-time in Mix type 6

It’s possible to interpret mass gain (chloride absorption)-time for in case of control/impregnation DCC cubes at different time intervals (31, 61, 91, 121, and 160) days respectively as representing in the (Figs.7a-7b). The chloride solution absorption was observed to lesser in magnitude at an initial time period (31days) as when compared to final time period (160 days). It’s also confirmed from the results that, the chloride solution absorption was predominantly increased in control concrete cubes (M1CC-M6CC) as when compared to impregnation concrete cubes (M1SB-M6SB and M1WB-M6WB) respectively.

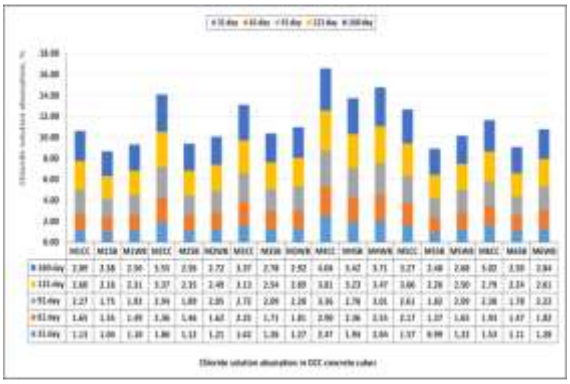


Fig.7a Chloride absorption in control/IC cubes

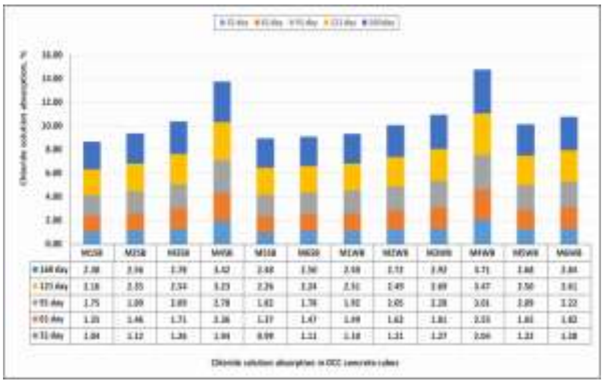


Fig.7b Chloride absorption in IC cubes (SB/WB)

The chloride solution absorption (increase) was interpreted at short time duration to longer time duration (31-61), (31-91), (31-121), and (31-160) days for in case of control/impregnation concrete cubes as representing in the Fig.7c. It's also possible to compared the chloride solution absorption (decrease) in impregnation concrete cubes ((M1SB-M6SB and M1WB-M6WB) as against control concrete cubes (M1CC-M6CC) as shown in the Fig.7d.

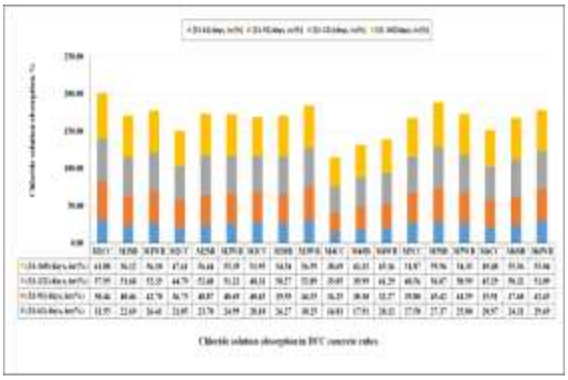


Fig.7c Chloride absorption in control/IC cubes


Fig.7d Chloride absorption in IC/control concrete cubes

The chloride solution absorption (increase) was interpreted in control concrete cubes (M1CC-M6CC) at different time intervals (31, 61, 91,121, and 160 days as when compared to impregnation concrete cubes (M1SB-M6SB and M1WB-M6WB) as representing in the Fig.7e. Chloride solution absorption (increase) was assessed in impregnation concrete cubes (M1SB-M6SB and M1WB-M6WB) by interpreting at short time duration to longer time duration (31-61), (31-91), (31-121), and (31-160) days as shown in the Fig.7f.

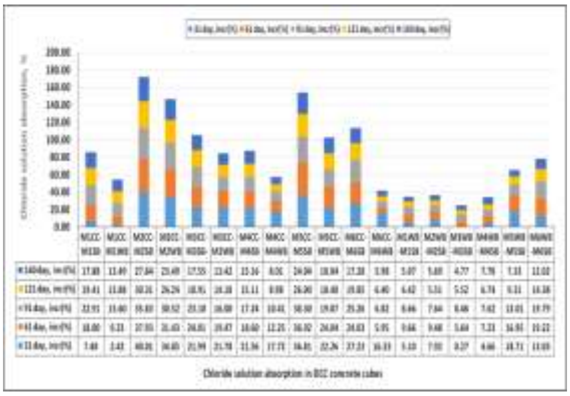


Fig.7e Chloride absorption in control/IC cubes

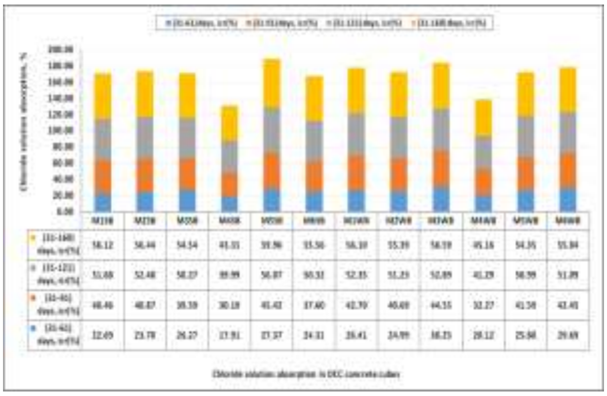


Fig.7f Chloride absorption in IC cubes (SB/WB)

The chloride solution absorption (increase) was interpreted in impregnation concrete cubes (M1SB-M6SB, and M1WB-M6WB) at different time intervals (31, 61, 91,121, and 160 days as representing in the Fig.7g-7h.

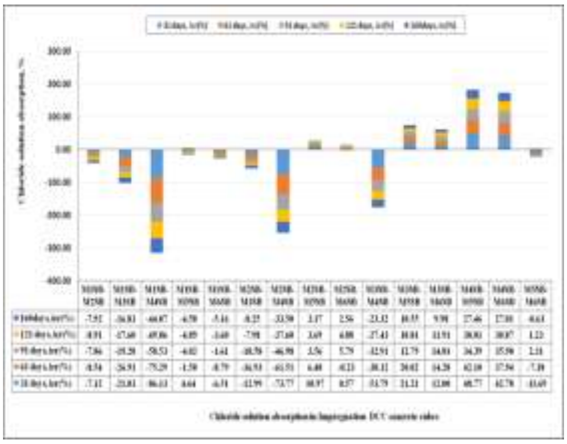


Fig.7g Chloride absorption in IC cubes (SB)

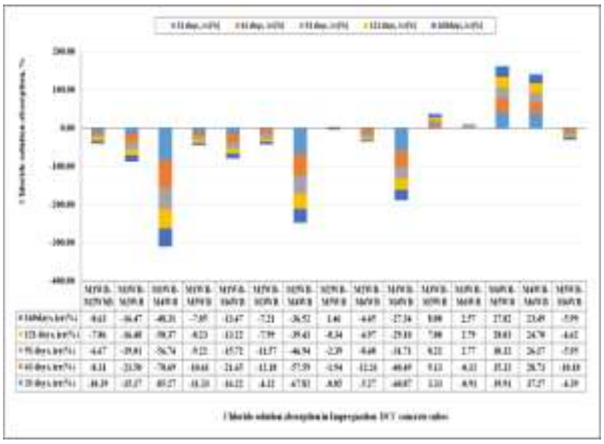


Fig.7h Chloride absorption in IC cubes (WB)

5.0 Conclusions

- Thus in the present research work chloride absorption test was carried out on 72 pre-conditioned concrete cubes such as dry conditioned concrete cubes in order to evaluate the effectiveness of two impregnation materials namely solvent based and water based impregnation material respectively. In turn to interpret the effectiveness of impregnation concrete cubes as against control cubes for six mixtures type with constant compressive strength (40 N/mm^2), and varied slump (0-10, 10-30, 60-180) mm in one case as well as varied compressive strength (25-30-40 N/mm^2) with constant slump (10-30) mm in second case.

- It's possible to correlate mass gain (chloride absorption)-time relationship by logarithmic expression for in case of control/impregnation DCC cubes at different time intervals (31, 61, 91, 121, and 160) days.

- It is observed from results that, the chloride absorption in DCC control concrete cubes was increased as when compared to the chloride absorption in DCC (SB) and DCC (WB) impregnation concrete cubes.

- From this chloride absorption-time relationship, it's possible to interpret the chloride absorption at any specified time duration in order to characterize the designed concrete mixtures type.

6.0 References

1. Brandt, A. M. (2009). Cement based composites: materials, mechanical properties and performance,
2. Taylor and Francis.
3. Balakrishna. M. N, Fouad Mohammad, Robert Evans, and Rahman. M. M. (2018). Evaluation of chloride
4. absorption in pre-conditioned concrete cubes, MJCE, 30(2):173-192.
5. Collepardi. M, Coppola. L, and Pistolesi. C. (1994). Durability of concrete structures exposed to $CaCl_2$ based de-icing salts, in: V.M. Malhotra (Ed.), Durab. Concr. ACI SP-145, 3rd CANMET/ACI Int. Conf, pp. 107–120.
6. Farnam. Y, Bentz. D, Sakulich. A , Flynn. D, and Weiss. J. (2014). Measuring freeze and thaw damage in mortars containing De-icing salt using a low-temperature longitudinal guarded comparative calorimeter and acoustic emission, Adv. Civ. Eng. Mater. 3:316–337.
7. Garboczi, E.J. (1990). Permeability, diffusivity, and micro structural parameters: A Critical review, Cement and Concrete Research, 20:591-601.
8. Hall, C. (1994). Barrier performance of concrete: A review of fluid transport theory,
9. Materials and Structures, 27:291-306.
10. Jones. W, Farnam. Y, Imbrock. P, Sprio. J, Villani. C, and Olek. J. (2013). An overview of joint deterioration in concrete pavement: Mechanisms, solution properties, and sealers.
11. Jacobsen. S, Sœther. D, and Sellevold. E. (1997). Frost testing of high strength concrete: Frost/salt scaling at different cooling rates, Mater. Struct. 30: 33–42.
12. Jacob. T, and Hermann. K .(1998). Protection of concrete surfaces: hydrophobic impregnations. Construction Technology's. (March) 18–23 (only available in Spanish).
13. Li. W, Pour-Ghaz. M, Castro. J, and Weiss. J. (2012). Water absorption and critical degree of saturation relating to freeze-thaw damage in concrete pavement joints, J. Mater. Civ. Eng. 24:299–307.
14. Marchand. J, Sellevold. E. J, and Pigeon. M. (1994). TheDe-icer Salt Scaling Deterioration of Concrete - An Overview, in: V.M. Malhotra (Ed.), Third Int. Conf. Durab. Concr., Nice, France, pp. 1–46.
15. Muethel. R. W. (1997). Investigation of calcium hydroxide depletion as a cause of concrete pavement deterioration.
16. McCarter. W. J. (1996). Assessing the protective qualities of treated, and untreated concrete surfaces under cyclic wetting and drying. Building Environ 31(6):551–556.
17. Pigeon. M, and Regourd.M. (1986). The effects of freeze-thaw cycles on the microstructure of hydration products,
18. Durab. Build. Mater. 4:1–19.

19. Scherer. G. (1999). Crystallization in pores, Cem. Concr. Res. 29:1347–1358.
20. Saetta, A.V., Scotta, R.V. and Vitaliani, R.V. (1993). Analysis of chloride diffusion into partially saturated concrete,
21. ACI Materials journal, 90-M47.
22. Teychenné, D. C, Franklin. R. E, Erntroy. H. C. (1988). Design of normal concrete mixes, Second edition, BRE.
23. Vriesl. J. de, Polde. R. B, and Borsje. H. (1998). Durability of hydrophobic treatment of concrete, Water repellent treatment of building materials, Proceedings of Hydrophobe ll, ZLirich, Aedificatio Publishers 77-90pp.
24. Wang. Z, Zeng. Q, Wang. L, Yao. Y, and Li. K. (2014). Corrosion of rebar in concrete under cyclic freeze–thaw and Chloride salt action, Constr. Build. Mater. 53:40–47.
25. Wittmann. F. H, Zhao. T. J, Zhan. H, and Xian. Y.(2006). Establishment of an effective chloride barrier in reinforced concrete structures by means of water repellent agents, Proc. Int. Workshop on Life Cycle Management of Coastal Concrete Structures, Nagaoka University, Japan, H. Yokota and T. Shimomura, editors, 201-208pp.
26. Zhang. S. P, and Zong. L. (2014). Evaluation of Relationship between Water Absorption and Durability of concrete Materials, Advances in materials science and Engineering, Article ID 650373.
27. Zhao. T, Zhu. G , F. H. Wittmann. F. H, and Li. W. (2008). On surface impregnation of chloride contaminated cement based materials, Hydrophobe V, 5th International conference on water repellent treatment of building materials Aedificatio Publishers, 311-324p.