Influence of hydrophobic additives on protection against alkali environment

RADKA PERNICOVA Klokner Institute Czech technical university in Prague Solinova 7, 166 08 Prague CZECH REPUBLIC Radka.pernicova@klok.cvut.cz http://www.klok.cz

Abstract: - Influence of hydrophobic additives on resistance to alkali environment is described in this paper. This article is focus on surface properties of vibropressed concrete connected with hydrophobicity. The aim of the testing was in description of influence of several types of hydrophobic additives, which were applied either to the interior structure or either as a coating on the surface of the element. In some cases, it was a combination of both. The article is about specific measurement of resistance against alkalis. Higher resistance to alkali and therefore better durability properties show samples with hydrophobic coating than with inner additives. All the samples with inner impregnation exceeded its permitted limits established for the alkali resistance multi-times. Measurement involves not only resistance to alkalis but also tests of frost resistance and water absorption depending on time.

Key-Words: - vibropressed concrete, hydrophobic additives, resistance to alkali, frost resistance

1 Introduction

The principle of hydrophobicity as a function method for increases durability and service life of building materials is described in this paper. Five types of hydrophobic additives were tested, all primarily used for concrete mixture. The article is about specific measurement of resistance against alkalis [1].

Porous building materials could absorb a lot of water due to its porous structure which may cause some negative effects on material. Important negative side effect is water as the carrier medium. Transport of ions in direction away from construction could lead to leaching of the compounds and their subsequent efflorescence on the surface [2]. In the opposite direction harmful substances such as chloride ions from de-icing salt may be carried into the material and therefore slightly damage its structure [3]. Humidity changes disturb the material not only chemically but also mechanically. Significantly destructive character has the freezing of saturated material. Therefore, the main aim is to provide quality hydrophobic isolation of the material, keep it away from humidity and avoiding the possible damage. Hydrophobicity could be increased by using protective layers. The of mechanism of hydrophobic problematic impregnation is very wide. Once, the porous

building material comes into touch with water, it appears capillary force $F_{kap.}$ In case that the contact angle is wider than the capillary forces are smaller and the drop of the water will transfer into shape of sphere in order to minimize contact with surface of the specimen [4].

The hydrophobic principle is about to increase surface tension of materials together with contact angle of water due to formed hydrophobic layer on the inside surface of pore. This layer must be very thin, nearly invisible, to avoid decrease diameter of pore on practical side to ensure that pores will still remain open to permeability. The water cannot create a continuous area on the hydrophobic surface and water runs down in form of sphere [5].

The fresh concrete mixture is highly alkaline (pH is approximately 12-13.5) when the highest value is caused due to reaction of calcium hydroxide (ph is approximately 12.6). This value could be further increased depending on the amount of alkali metals in mixture (K₂O a Na₂O). Presence of water changes these alkali metals into strong hydroxide.

The resistance of hydrophobic layer against influence of alkalis is tested for several reasons:

- protection against water and regulation of humidity in order to decrease of risk corrosion,
- preserving the physical integrity and resistance of construction surface,

• Improvement of chemical properties [6].

The article is about resistance of hydrophobicity against alkalis (represent by potassium hydroxide solution), which was calculated by absorption ratio AR_{alk} obtained from several measurements of capillary absorption. The results were supplemented by measurement of water absorption and frost resistance.

2 Formulation Problem

The measurement of resistance hydrophobic impregnation against alkalis is according to the ČSN EN 13580. The test was composed of two identical measurements. Between these tests, the samples are exposured to aggressive environment for 21 days.

The measurement took place in laboratory at the temperature 20 ± 2 °C a humidity 40-50 %.

The test of frost resistance is according to the ČSN 72 2452 and the measurement of basic hydric properties, especially water absorption, is according to the ČSN EN 13057.

2.1 Materials and samples

Selected vibropressed concrete mixture with density of 2100-2200 kg/m³ has been selected for laboratory tests. These products are used primarily as architectural concrete which does not further modify. Therefore it is important to pay more attention to their surface properties especially from esthetical perspective [7].

Five hydrophobic mixtures on organic base were chosen for experimental measurement. Additives (labeled A to D) are slightly differed in their chemical composition. All internal ingredients were used in an amount of 0.5 wt%.

Tab. 1 Basic properties and hydrophobic additives

in mixture [8]				
No.	Additive	Density		
samples	-	$[kg/m^3]$		
1	none	2100		
2	coating A	2150		
3	coating B	2070		
4	internal C	2180		
5	internal C + coating A	2190		
6	internal C + coating B	2150		
7	internal C + internal B	2150		
8	internal B	2100		
9	internal D	2190		
10	internal E	2180		

Individual samples (labeled 1 to 10) differed in using various hydrophobic additives which were applied either to the interior structure as an additive or either as a coating on the surface of the element. In some cases, it was a combination of both. Some of the basic properties, which are closely connected with resistance to alkalis, were measured in previous study [8].

Comparative measurements without any hydrophobic additives were done as well. The test has started 28 days after manufacture of specimen had been made, to give enough time for hardening samples. Absorption test started additional two days after hydrophobic coating was applied.

2.2 Experimental measurement

Basic properties of material were measured at first. Among the basic properties, the bulk density and water absorption belong. The measurement of basic parameters took place in a conditioned laboratory at the temperature of 22 ± 1 °C and 25-30 % relative humidity. Each result represents the average value from three to five measured values.

Capillary absorption was measured on the circular base of the cylinder specimen, which was exposed to the negative environmental conditions. Each sample was placed on the front surface to water so its level was (5 ± 1) mm. Whole measurement was covered to prevent the movement of air around the test body. It was determined the rise of absorbed water to the sample in specific time interval up to a steady state. In each time interval the surface of specimen was dried and sample was weighed.

Examined samples were immersed into demineralised water on supports so that the height of water above the cylinder was at least 25 ± 5 mm. Right before immersion in deionised water each sample was weighed separately (i₁). Test cylinders were removed from the water (impregnated after 24 hours, non-impregnated only one hour). All samples were reweighted right after its surface was dried (i₂).

The rate of increase in weight for each impregnated test sample (I_t) was calculated according to following relation:

$$I_t = \frac{i_2 - i_1}{\sqrt{24} \times A} \tag{1}$$

, where I_t is rate of increase in weight impregnated sample(s) [g/m²h^{0,5}], i_1 is weight of impregnated samples before immersion to the water [g], i_2 is weight of impregnated samples after removal from the water [g] and A is area of the specimen $[m^2]$.

The rate of increase in weight of each nonimpregnated test sample (I_u) was calculated according to following relation:

$$I_u = \frac{i_2 - i_1}{\sqrt{1 \times A}} \tag{2}$$

, where I_u is rate of increase in weight nonimpregnated sample $[g/m^2h^{0.5}]$, i_1 is weight of nonimpregnated samples before immersion to the water [g], i_2 is weight of non-impregnated samples after removal from the water [g] a A is area of the specimen $[m^2]$.

The absorption ratio AR was calculated as follows:

$$AR = \frac{I_{im}}{I_{um}} \cdot 100 \tag{3}$$

, where *AR* je absorption ratio [%], I_{tm} is average value of rate of increase in weight impregnated test samples [g/m²h^{0.5}] and I_{um} is average value of rate of increase in weight non-impregnated test samples [g/m²h^{0.5}].

Immediately after water immersion test was finished impregnated specimens has been inserted on the top of washers and into the beakers with solution of potassium hydroxide (5.6 g / l), specific way to ensure that the solution height level was above specimen 25 ± 5 mm. The beakers were covered with sticky foil and left for 21 days (see Fig. 1). Then, the test samples were removed and dried at room temperature ($21 \pm 2 \text{ °C}$) on the laboratory desk until its weight reached the weight right before the test immersion $i_1 \pm 2$ g. Afterwards, the second immersion test was performed, followed by the calculation rate of increase in weight each impregnated test samples after alkaline test $I_{\text{t(alk)}}$.

When the exposure to alkali finished the absorption ratio AR_{alk} , was calculated by the following relation:

$$AR_{alk} = \frac{I_{im(alk)}}{I_{um}} \cdot 100 \tag{4}$$

, where AR_{alk} is absorption ratio after exposure to alkali [%], $I_{tm(alk)}$ average value of rate of increase in weight impregnated samples after immersion in to the alkali solution [g/m²h^{0,5}] and I_{um} is average value of rate of increase in weight non-impregnated samples.

The purpose of frost resistance was to specify amount of fallen away particles from main body of concrete specimen. The results were reached by measure the weight of these small parts. The test was carried out in a controlled environment at a fluctuating temperature of -20 ± 2 °C and 20 ± 2 ° C. The samples were subjected to cycling between 2 hours freezing in water (ice) and 2 hours defrosting in hot water. The test was performed in an automatic conditioning chamber HERAUS Votchi P10003 with forced air-condition. Continuously visual inspection of samples was carried out. After the required number of 100 cycles, the testing samples were conditioned. Subsequently, the measured of weight was done. The cycling was done automatically in a freezing chamber.

The simplest way, how to describe liquid water transport through a porous material is realization of a one-dimensional free imbibition experiment [9]. The water absorption coefficient was measured using a standard experimental setup [10]. Specimens sizes of 100x100x50 mm were water and vapour-proof insulated with epoxy resin on four edges, and after that dried in an oven. The face site of each sample was immersed 1-2 mm into water on top of a saturated sponge. The sample mass was measured continuously. The water absorption coefficient A [kgm⁻²s^{-1/2}] was calculated from the linear part of the dependence of the increase of tested sample's mass on the square root of time using the formula (5):

$$i = A\sqrt{t}$$
 (5)

, where $i [kg/m^2]$ is the cumulative water absorption, t is the time from the beginning of the suction experiment [11, 12].

3 Problem Solution

Results of hydrophobic impregnation resistance against alkalis were evaluated through absorption ratio according the norm ČSN EN 1504-2.



Fig.1 Settlement samples during alkali test

Ne	i 1	\mathbf{i}_2	Iu	It	AR
No.	[g]	[g]	$[g/m^2h^{0,5}]$	$[g/m^2h^{0,5}]$	[%]
1a	1461.5	1518.7	1059.5		
2	1513.5	1545.7		122.23	11.54
3	783.8	822.3		173.99	16.42
4	1487.6	1523.1		133.32	12.58
5	1530.9	1544.9		53.34	5.03
6	630.5	664.4		156.47	14.77
1b	5774.8	5843.9	656.5		
7	5714.0	5782.9		134.99	20.56
8	5704.3	5753.9		95.37	14.53
9	5706.0	5818.1		216.31	32.95
10	5708.9	5804.5		185.79	28.30

Tab. 2 Results of absorption test BEFORE alkali immersion test

Tab. 3 Results of absorption test AFTER alkali immersion test

No.	i_1	i_2	It	AR _{alk}
INO.	[g]	[g]	$[g/m^2h^{0,5}]$	[%]
1a	1481.61	1514.54		
2	1533.5	1554.35	80.33	7.47
3	788.55	810.17	151.62	9.20
4	1510.07	1524.16	52.91	4.99
5	1541.12	1555.99	56.78	5.36
6	637.29	659.26	101.62	9.59
1b	5829.9	5871.7		
7	5766.5	5823.7	112.07	17.07
8	5732.4	5812.4	153.82	23.43
9	5780.4	5849.8	133.91	20.40
10	5759.7	5857.2	189.48	28.86

Higher resistance, respectively lower absorption ratio, and the better hydrophobic properties show samples with coating rather than with inner impregnation. The final values vary according concrete mixture. Regarding that the measurement defined for test of coating is primarily hydrophobicity, it can be concluded a higher measurement deviation and uncertainty by samples with inner impregnation. Limit for fulfil hydrophobic resistance is given by the ratio AR less than 7.5%, which is fulfilled only by samples with the coating, others with the internal impregnation does not.



Fig.2 Water absorption depending on time in its initial interval

Tab. 4 water absorption and frost resistance

No.	Water Absorption	Particles after 100 cycles
samples	$[kg/m^2]$	$[g/m^2]$
1	0.1057	141
2	0.1000	76
3	0.0222	86
4	0.066	61
5	0.5830	100
6	0.0389	155
7	0.0688	61
8	0.0893	67
9	0.0769	137
10	0.0678	97



Fig.3 Typical detail of edge of concrete after 100 cycles of frost resistance

4 Conclusion

Basic properties such as bulk density impregnate and non-impregnate samples remained unchanged. Differences are within the variations in measurements. There is quiet obvious difference in Water absorption depending on used hydrophobic additions. That means the samples varies in rate of hydrophobicity.

Higher resistance to alkali and therefore better durability properties show samples with hydrophobic coating than with inner additives.

Final values of resistance against alkalis of hydrophobic impregnations were calculated by absorption ratio AR_{alk} and vary by each mixture. The worst hydrophobic properties has specimen with inner additions D, sample no. 10 (28.86%) and the best hydrophobic behaviour shows specimen with inner additions A, sample no. 4 (4.99%).

To take a count the test is primarily used for measurement of coating impregnation. It is expected higher measurement deviation and uncertainty by the samples with inner hydrophobicity.

The best results were achieved at the samples with coating B together with inner impregnation A. All specimens with coating showed very good value of absorption ratio and fulfil the limit 7.5% or just slightly exceeded.

All the samples with inner impregnation exceeded its permitted limits established for the alkali resistance multi-times, i.e. result is in terms of resistance significantly negative. These samples have been washed out by large amounts of calcium ions, which later crystallized out of the sample surface. The risk of internal impregnation seems to be fact that in a real situation white crystals would not probably formed under the coating and the white maps on construction will appears caused by migration of calcium ions, which are contain in water.

5 Acknowledgement

This research has been supported by GACR Grant Agency of the Czech Republic No. GACR15-10591S.

References:

- [1] Plesser T.S.W. *Moisture in Concrete Alkaline sensitive surface treatments*. SINTEF Building and Infrastructure. 2007.
- [2] Pernicova R. Analysis of formation and testing of efflorescence on concrete elements, *Advanced Materials Research* Vol. 1025-1026. 2014. pp. 641-644.
- [3] Dobias D., Pernicova R. Diffusion of Chloride Ions in Ultra High Performance Concrete.

Advanced Materials Research Vol. 1106. 2015 pp. 21-24

- [4] Krasny I. Measurement of contact angel and determination of surface free energy of plastic materials. *Thesis*. 2010. Tomas Bata University in Zlin. Faculty of technology. Czech Republic
- [5] Selander A. *Hydrophobic Impregnation of Concrete Structures.* Doctoral Thesis, Architectural and the Built Environment Royal Institute of Technology. 2010.
- [6] Collepardi M. *The new concrete*. Grafiche Tintoretto. 2010.
- [7] Babkov V.V., Gafurova E.A., Rezvov O.A., Mokhov A.V. The problems of the occurrence of efflorescence on the surface of buildings' exterior walls made of vibropressed concrete blocks and the methods of blocking these processes. *Magazine of Civil Engineering Journal*. No.33. 2012. pp. 14-22.
- [8] Pernicová R. Influence of hydrophobic additives on susceptibility to the formation of efflorescence on concrete elements. *Recent* advances in mechanics and materials in design. 2015. pp. 283-284.
- [9] Karoglou M., Moropoulou A., Maroulis Z.B, Krokida, M.K. Water sorption isotherms of some building materials. *Drying Technology*. Vol. 23. 2005. pp. 289-303.
- [10] Lakatos A., Kalmár F. Examination of the change of the overall heat transfer coefficients of building structures in function of water content. *Building Services Engineering Research & Technology* vol. 35. 2014. pp. 508-515.
- [11] Lakatos A. Investigation of the effect of moisture in the time lag of building walls with different insulating materials. *Environmental engineering and management journal* vol. 13. 2014. pp. 2853-2858.
- [12] Stubberud L., Arwidsson G. H., Graffner C. Water-solid interactions: I. A technique for studying moisture sorption/desorption. *International Journal of Pharmaceutics*. Vol. 114. 1994. pp 55-64