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Determination of carbonation of mortar by means of pH measurements

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PREFACE

The determination of the extent of carbonation in concrete and mortars is important in assessing the extent of curing that has occurred. Many factors affect the curing process, including the reaction of the CO_2 in the atmosphere with lime in the concrete or mortar, to form calcium carbonate. The carbonation process is accompanied by a loss of basicity. The author of this paper describes a method of assessing the extent of carbonation by measuring the pH of aqueous extracts of samples of the concrete or mortar.

The Division wishes to record its thanks to Mr. D.A. Sinclair, Translation Section, National Research Council, for translating this paper and to J.I. Davison of the Atlantic Regional Station of DBR/NRC who checked the translation.

Ottawa
May 1971

N.B. Hutcheon
Director

NATIONAL RESEARCH COUNCIL OF CANADA

TECHNICAL TRANSLATION 1464

Title: Determination of carbonation of mortar by means of pH measurements

(Zur Frage der Karbonatisierung von Mörtel mit Hilfe von pH-Messungen)

Author: G. Ruprich

Reference: Baustoffindustrie, 9: 237-239, 1966

Translator: D.A. Sinclair, Translations Section, National Science Library

DETERMINATION OF CARBONATION OF MORTAR BY MEANS OF pH MEASUREMENTS

In assessing corrosion processes in reinforcement rods, the attention of specialists is being increasingly concentrated on carbonation phenomena in concretes and mortars. The CO_2 always present in the atmosphere brings about a change in the chemical structure of the units from which these concretes and mortars are constructed. The process of carbonation in concretes and mortars, owing to CO_2 diffusion, can be clearly described in terms of the loss of basicity[1,2,3].

Experimental Setup

The mortar specimens were produced under specified conditions (Tables I to III), in such a way that an average measured value from six specimens for each case can be regarded as representative.

To speed up the reaction $Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$ the tests were conducted in an air-conditioned room at 40°C and 70% relative humidity, with the CO_2 content of air mixture kept constant (15 percent CO_2/85% air). The cubic specimens (60 × 60 × 60 mm) were compacted on a vibrating screen and stored 1, 2, 3, 7, 14 and 28 days. Accordingly, the problem was to get valid quantitative measurements. Among the methods described by several other authors, the method of electrochemical measurement of aqueous concrete extracts seemed to be the most suitable, in spite of the danger that the mortar components which are washed out by the extraction, and whose basic structure is destroyed, would show too high a pH value. The pertinent literature contains very different opinions on this point[1, 2].

If the test conditions are kept constant, this should not be very important, since in the present case the error will always be constant. In these tests, we drilled a hole 20 mm in diameter through the center of the mortar cube. Every 3 mm the filings were collected and suspended in distilled water (20% solution). Immediately after processing, the pH of the specimen was determined

with an amplified electronic pH tester of the firm of Clamann and Grahnert, Dresden.

Test Results

For the specified exposure times, the relationships shown in Figure 1 were obtained. The families of curves show the typical shapes for diffusion processes. Termination of the diffusion-reaction process after longer storage time is also evident. Under the chosen limiting conditions, it can be regarded as complete after about 20 days.

By analogy with the chloride extraction tests of Dehler, Günther and Höfer[4] it seemed very probable that the grain fractions of the filings would show different pH values. This was confirmed by the tests (Table IV). This was a convincing proof of an actual discrepancy between the true and the found pH value. However, since all the ground-up mortar material was always extracted, this behaviour should not be overemphasized. We are merely stating what was observed.

A mineralogical and x ray determination of calcite in the reaction products of the carbonated mortar specimens, conducted parallel to the pH measurements, was not successful. Treating the test specimens with dilute hydrochloric acid under the microscope revealed only the release of CO_2. There are two possible explanations of this behaviour:

1. The $CaCO_3$ arising under these conditions of formation is amorphous to x rays because pure crystal growth is prevented by a secondary reaction involving the breakdown of the primarily formed Ca bicarbonate into Ca carbonate.

2. Under the given test conditions the reaction products correspond to different modifications of the calcium carbonate (vaterite, aragonite).

The first of these two interpretations is favoured by the existence of an unstable pH behaviour after storage of the specimens in the air-conditioned room. It was often noticed that the pH values of the specimens were higher after one to two days storage

in the air following their removal from the air-conditioned room (Table V).

The lower pH limiting value in Figure 1 is 10 for an exposure time of 20 days and the upper one, for t = 0, is 12.6.

Now, if we consider the integral area components in percentage of the total area below the individual curves to be a measure of the extent of carbonation, we find the relationship of Figure 2 for the different storage times, by forming the quotient of carbonated to non-carbonated component by area. The slope of the straight line here characterizes the CO_2 permeability of the mortar employed. With the determination of this factor it is possible to describe the carbonation behaviour under the given conditions. A report will be given at a later time on the applicability of this method to mortars with grain sizes up to 5 mm in diameter.

The author wishes to thank Miss S. Lehrmann for her valuable assistance in carrying out the numerous pH measurements.

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Table I

Mortar composition

5 GT	PZ 375 Karsdorf
3 GT	distilled H_2O
8 GT	standard sand I
16 GT	standard sand II

Table II

Grain fractions of standard sand used

Standard sand I			Standard sand II		
Grain diameter from [mm]	to	Component [%]	Grain diameter from [mm]	to	Component [%]
0,250		2,185	1,600		0,370
0,200	0,250	17,700	1,000	1,600	6,220
0,160	0,200	10,100	0,800	1,000	53,892
0,125	0,160	12,000	0,630	0,800	37,958
0,100	0,125	13,100	0,500	0,630	0,890
0,080	0,100	0,100	0,250	0,500	0,300
0,063	0,080	6,660			
0,050	0,063	6,010			
0,045	0,050	15,110			
0,045		0,775			

Table III

Chemical analysis of PZ 375, from Karsdorf factory

Insoluble	0,51	So_3	2,64
Sio_2	19,46	MgO	3,10
Al_2O_3	6,30	free CaO	1,26
Fe_2O_3	2,88	Loss on ignition	1,48
CaO	63,45		

Table IV

Relationship of pH values to the various grain fractions

Grain diameter from [mm]	to	pH value	Grain diameter from [mm]	to	pH value
3,15		9,05	0,80	0,50	8,75
3,15	1,60	9,15	0,50	0,25	8,80
1,60	1,00	8,60	0,25	0,125	8,90
1,00	0,80	8,65	0,125		10,10

Table V

Change of pH value of carbonated specimens*
as a result of two days exposure in the atmosphere

Before exposure		After exposure	
Distance from surface [mm]	pH value	Distance from surface [mm]	pH value
1,5	10,21	1,5	9,07
4,5	10,25	4,5	9,00
7,5	10,17	7,5	9,30
10,5	10,38	10,5	9,45
13,5	10,52	13,5	9,57
16,5	10,84	16,5	9,62
19,5	11,02	19,5	9,67
22,5	11,17	22,5	9,77
25,5	11,37	25,5	9,50
28,5	11,48	28,5	9,55

* Specimens had been stored seven days in the air-conditioned room.

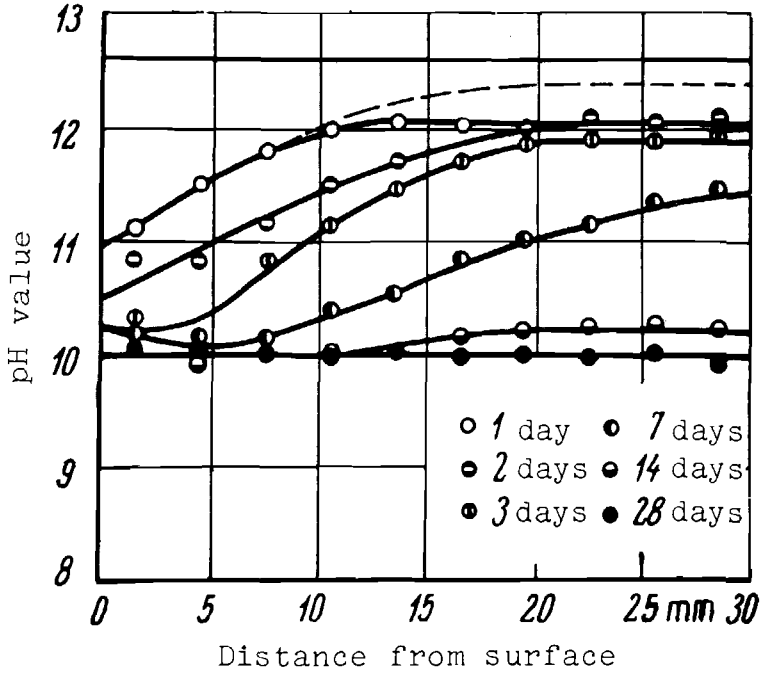

Fig. 1

pH variation in the mortar due to carbonation, as a function of time and distance from the surface

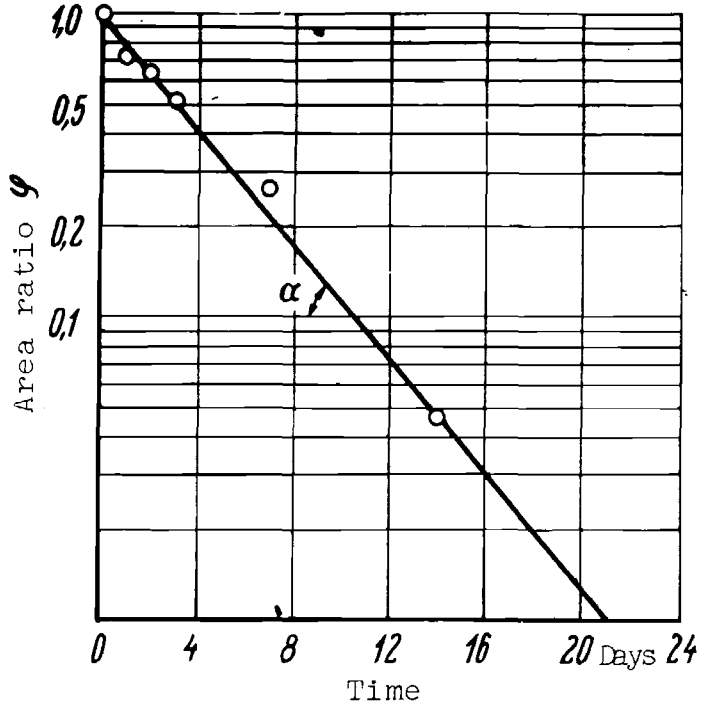

Fig. 2

Relation of area quotient ϕ to the exposure time