SORPTIVITY TESTS ON

CARBONATED CONCRETE

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CANADA MORTGAGE AND HOUSING CORPORATION POLICY DEVELOPMENT AND RESEARCH SECTOR

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Canada Mortgage and Housing Corporation, the Federal Government's housing agency, is responsible for administering the National Housing Act.

This legislation is designed to aid in the improvement of housing and living conditions in Canada. As a result, the Corporation has interests in all aspects of housing and urban growth and development.

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ABSTRACT

Rate of water absorption tests (also called sorptivity tests) were performed on both the carbonated surfaces and the uncarbonated interiors of a series of concrete core segments removed from apartment buildings in Toronto. The purpose was to try and develop a test to predict the susceptibility of in situ concretes to excessive future rates of carbonation.

The concrete samples were very small and had been stored for several years, resulting in scatter of the sorptivity data. Inspite of the limitation, there was a general trend between the depth of carbonation and the sorptivity values of the concrete surfaces.

EXECUTIVE SUMMARY

Carbonation, a reaction between atmospheric carbon dioxide and the cement paste fraction of concrete, can reduce the alkalinity of concrete. If the depth of carbonation reaches the level of reinforcement, the steel will become depassivated and in the presence of moisture, can corrode.

It would be useful to be able to predict the quality of concrete non-destructively in order to determine its susceptibility to carbonation. It is known that the rate of carbonation is diffusion related (ie.: related to the square root of time) and is influenced by concrete mix design (mainly the water to cement ratio, w/c) and the curing period provided at early ages. Both the w/c and the curing affect the porosity of concrete and hence the diffusion rate of carbon dioxide. If a low w/c concrete (eg. w/c = 0.4) is used and cured properly, carbonation after 50 years will likely only be a few mm. The rate of water absorption by a concrete surface is also a diffusion process which is related to the combined effects of w/c and curing, and is a relatively simple and inexpensive procedure. It can be done on cylindrical cores removed on a structure and also has possibilities as an in-situ, non-destructive test.

In this study, a series of 28 concrete core segments, left from a previous study of Toronto apartment buildings where depth of carbonation was measured, were tested for rate of absorption (sorptivity). Tests were done on the original surface, 2 mm below the surface, and approximately 50 mm below the surface in the uncarbonated zone. The purpose was to see a) if the sorptivity of the surface (affected by curing or lack of curing) would be higher than at depth, and b) if the concretes with high depths of carbonation were related to high sorptivity values.

The results indicate that there was a rough relationship between depth of carbonation and the sorptivity of the outer surface of the concrete.

It was concluded that the sorptivity test has potential for indicating the susceptibility of concrete surfaces to carbonation. However, the small size of the core pieces and their extended storage before test prevented obtaining a more precise relationship in this study. Therefore, this technique requires further development to be useful. Further work by the first author has resulted in a more precise test method using cores of at least 100 mm diameter. This work as well as the subsequent has helped in the development of a draft ASTM standard test method for evaluating concrete quality.

RÉSUMÉ

La carbonatation, réaction entre le dioxyde de carbone et la pâte de ciment du béton, peut réduire l'alcalinité du béton. Si le degré de carbonatation atteint l'armature, l'acier devient dépassivé et se corrode sous l'effet de l'humidité.

Il serait utile de pouvoir prévoir la qualité du béton sans le détruire afin de déterminer sa tendance à la carbonatation. Nous savons que le taux de carbonatation est lié à la diffusion (c.-à-d. lié à la racine carrée du temps) et qu'il est influencé par la compositon du béton (surtout le rapport eau/ciment) et la période de cure du début. Le rapport E/C et la cure modifient la porosité du béton et par conséquent le degré de diffusion du dioxyde de carbone. Si l'on utilise une méthode de cure convenable et que le rapport eau/ciment est faible (p. ex. E/C=0,4), la carbonatation après 50 ans ne sera que de quelques millimètres. Le taux d'absorption de la surface de béton est également un processus de diffusion lié aux effets combinés du rapport eau/ciment et de la cure et sa mesure est relativement simple et économique. Cette méthode peut être utilisée sur des pièces cylindriques enlevées d'une structure. Elle offre également la possibilité d'effectuer des essais non destructifs sur place.

L'étude comprend des essais sur le taux d'absorption (sportivité) menés sur 28 pièces centrales en béton provenant d'une étude antérieure sur le degré de carbonatation des immeubles d'appartements de Toronto. Les essais ont été effectués sur la surface originale, à 2 mm sous la surface et à environ 50 mm sous la surface dans la zone non carbonatée. Ces essais avaient pour but de vérifier si a) le taux d'absorption est plus élevé à la surface (soumise ou non à une cure) qu'en profondeur et b) si le béton dont la carbonatation est très profonde présente un taux d'absorption élevé.

Les résultats indiquent un certain lien entre la profondeur de la carbonatation et le taux d'absorption de la surface externe du béton.

Il a été conclu que les essais sur le taux d'absorption pouvaient possiblement indiquer le risque de carbonatation des surfaces en béton. Les dimensions restreintes des pièces centrales et leur entreposage prolongé avant les essais ont cependant empêché l'établissement d'un lien plus direct à partir de l'étude. Cette technique ne sera donc utile que si elle est élaborée davantage. D'autres travaux effectués par le premier auteur ont abouti à une méthode d'évaluation plus précise qui fait appel à des pièces d'au moins 100 mm de diamètre. Cette recherche et d'autres travaux ultérieurs ont contribué à la rédaction d'une norme provisoire ASTM sur la méthode d'essai pour l'évaluation de la qualité du béton.



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1 INTRODUCTION

Carbonation in concrete occurs when water and carbon dioxide from the air becomes dissolved in the pore water of the cement paste fraction of concrete to form carbonic acid. This acid reacts with 1) calcium hydroxide (a by-product of cement hydration) to form calcium carbonate, and 2) with the calcium silicate hydrate matrix to form calcite and silica gel. The resultant pH of concrete drops to below 9.0. This low pH causes reinforcing bars within the concrete to lose their passivity and allows for corrosion to occur.

As part of a study on evaluation of the potential carbonation problem in Canada, the University of Toronto was supplied by the CMHC (Canada Mortgage and Housing Corporation) (via Mr. J. Bickley) with a quantity of concrete cores extracted various buildings around Toronto. A previous study had been performed on these samples for CMHC by Trow Ltd.

The purpose of this study was to try to relate concrete quality via its sorptivity to its depth of carbonation. The concept was that the easily measured sorptivity can be related to the quality of the original concrete and the way the concrete was. If the concrete quality was poor, or if the curing was not done properly, sorptivity should be higher than that of the same concrete cured properly. Curing affects the quality of the surface and the cover over the reinforcement. This is also the area affected by carbonation.

1.1 Methodology

Sorptivity tests were performed on the original surface of the samples and at various interior planes. This provided rates of sorption at different depths which were subjected to different "curing", and thus, a relationship between the depth of carbonation and the sorptivity can be studied.

2 LABORATORY PROCEDURES

2.1 Introduction to the Sorptivity Test

Sorptivity is a characteristic property of a material and describes its ability to absorb water. A "poor" concrete that is relatively porous with many large pores absorbs water more quickly than does a "good" quality concrete.

The sorptivity, S, of a material as described by Hall [1], is obtained by considering one-dimensional water flow into an initially dry sample. The cumulative water absorption i, (mass per unit area of the inflow surface), increases as the square root of the elapsed time, t:

$$i = S\sqrt{t}$$

The sorptivity test involves soaking one surface of the initially dry concrete sample in distilled water and taking mass readings at certain intervals. The cumulative water absorption i, is then plotted against the square root of time. Once a linear relationship is established, the slope of this line is determined as the sorptivity S.

2.2 Sample and Test Preparation

The samples supplied were the remains of splitting tensile tests done on small diameter cores conducted by Trow Ltd for the CMHC. After splitting, the split surfaces were sprayed with phenolphthalein indicator to estimate the depth of carbonation. The dry samples were wrapped in plastic bags and labelled according to that study. The phenolphthalein indicator was still visible on the split surface, thus denoting the carbonation depth. With the exception of the building identifier, the core labels did not correspond to the numbering system in the final Trow report. However, from the depths of carbonation, it was possible to identify each core with respect to the data reported in the Trow report. This information is listed in Table 1. For the sample identification, the first number is the building identifier followed by a slash and the core number. The type of building elements, its orientation and surface condition are also noted in Table 1.

In retrospect, the cores were really too small for this test due to their small cross-sectional area with respect to the size of the coarse aggregate. However, this work helped to identify such problems and helped in the development of the draft ASTM sorptivity procedure.

Each specimen was examined to check suitability for testing. This involved the recording of the carbonation depths (3 points measured and averaged), the measurement of end areas and an examination for any cracks or damage resulting from the split cylinder tests. All samples unsuitable for testing were returned to the plastic bags.

Since each sample had one flat face end one jagged end, the jagged end had to be cut flat using a diamond saw. All pieces removed were bagged and stored.

Each sample had a different cross-sectional area exposed to wetting, therefore the sorptivity values were normalized to a unit area. The split cylinders were not of regular shape, so the area of the test surface was measured in the following way. A piece of cardboard was cut to the exact dimension of the test face and then weighed to 0.0001 g accuracy. The number was the multiplied by a constant to produce the area of the test surface.

The test procedure described in the next section, was similar to those of W. Dolch and J. Lovell [2] and Hall [1]. However, it was decided that the sides of the samples should be sealed to ensure water entered through the test surface only, thus producing only one dimensional flow. Epoxy paint normally for reinforcing bars was used as the sealer. It was impermeable enough to prevent water from entering the sides of the sample, and viscous enough to ensure as little penetration into the sample pores as possible.

A common way of doing a sorptivity test is to support the concrete sample on a ring at the base and have the test face in direct contact with the water. Since the samples varied greatly in size and shape, the test surface was placed on filter paper. The paper was then completely saturated with water. Samples sitting on the filter paper were about 1 mm deep in the water. This allowed all samples to be tested under the same conditions to help make the test repeatable.

To check paint penetration, a sample core was painted and then cut by diamond saw. The cross-section was then checked, and penetration was found to be less than 0.5 mm. This would not effect the cross-sectional area significantly.

Table 1 Data on Depth of Carbonation (mm), Orientation and Surface Finish of Concrete Test Cores.

Sample	Depth	Type	Ornt.	Painted
75/2	0	В	W	y-med
75/4	37	V	W	y-thin
75/15	5	В	E*	no
75/16	24	V	E	y-thin
75/18	25	V	E	y-thin
79/1	6	В	W	no
79/3	6	В	W	no
79/4	0	В	E	no
79/5	10	V	W	no
79/17	14	V	W*	y-thick
132/7	25	V	E	possibly
132/12	1	PC	S	y-thin
132/13	7	PC	S	y-thin
132/16	2	PC	S	y-thin
132/18	4	PC	S	y-thin
94/7	11	В	S	y-thin
94/10	17	V	S	y-thick
94/13	18	В	S	no
94/15	20	В	N	no
94/16	10	V	N*	y-thick
94/18	1 1	V	S*	y-thick
100/2	4	В	W	no
100/8	0	В	E	no
100/9	0	В	E	no
7/1	1	В	N*	y-vthick
7/5	0	В	N	y-vthick
7/8	17	V	N*	y-thin
7/13	1	В	W*	y-med

B: Balcony Concrete

V: Vertically Cast Concrete

PC: Precast Concrete

Y: Yes

* - Orientation is uncertain

Finally the samples were placed in a vacuum oven (approximately 500 mm Hg at 40-60°C) with a small quantity of dry air (passed through silica gel) allowed into the oven to remove all the moisture. It was desirable to have all the samples dried to the same degree. Therefore all the samples were dried for the same period, then stored in a desiccator with silica gel prior to testing. The samples remained in the oven for approximately 1 week between tests. Initial tests showed that the vacuum oven was not effective in drying the samples until the dry air was introduced into the system. It is suspected that the samples would have lost more water vapour in an oven at 105 degrees C, but it was feared this would cause microstructural damage to the samples. After completion of each test, the sample was dried until its weight closely matched that weight measured prior to the first test (to within 0.02 g).

2.3 Test Procedure

After being dried in the vacuum oven, each sample was weighed. This weight was used as a dry state reference for the second and third tests. The clock would then be started as soon as the sample test face was placed in the water on top of the filter paper. Readings were then taken each 15 seconds for the first minute, then for 30 seconds for the next two minutes, then every minute for the next 7 minutes and then each 2.5 minutes for the last 5 minutes. The clock was only running during the time in which the sample was in the water.

In order to take a weight measurement, each sample was taken from the water, wiped with a damp tissue to remove surface water, weighed and then placed back into the water. This procedure took approximately 12 seconds. In order to be consistent, the time the sample was out of the water was extended to 15 seconds for all readings. This allowed for a standard timing chart to be set up and used for all tests. As long as the surface wiping procedure was consistent, the error due to variations in the amount of surface water removed was small.

3 DATA COLLECTION

Three tests were done on each sample. They were:

- 1) Test of the exposed surface (carbonated) of the core.
- 2) Test on the inner surface of the core referred to as section A-A, (uncarbonated and generally deeper than the depth of influence due to surface curing).
- 3) Test the surface just 2 mm below the exposed surface referred to as section B-B, (carbonated, but reduces "skin" effects). The skin was sawn off after tests 1 and 2, then the sample was re-dried and re-tested.

Figure 1 illustrates the results of these three tests conducted on specimen 94/18. Notice that the slope of each line becomes roughly constant after one minute or less. By entering this data into a spread sheet, a regression analysis can determine the slope of the cumulative water mass absorption per unit area versus the square root of time for each case. This resulting slope is the sorptivity.

Regression was done individually on data from the first 1 minute of testing, the 14 minutes after the first 1 minute, and the full 15 minutes. The reason for the 1 minute test is to allow for some comparison with a new ASTM test (ASTM C1151-90), just recently introduced, where a sample is soaked in water for 1 minute and then the change in mass is noted. The 14 minute regression is done because various plots of the sorptivity revealed a constant slope after approximately 60 seconds. The 15 minute regression was done to cover the whole period of test. These slopes are listed for all the cores in Table 2.

Table 2. Sorptivity Data (g/mm²/S^{0.5}) of Concrete Cores at Various Horizons and for Various Time Periods

Build		1 Min.		- 	14 Min			Fuil	
Samp	Ext.	A-A	В-В	Ext.	Δ-Δ	B-B	Ext.	A-A	В-В
76/2	2.99E-06	1.82E-06	2.22E-05	1.74E-05	8.85E-08	1.82E-06	1.68E-05	7.14E-08	1.72E-05
75/4						3.10E-05			
76/16						3.11E-05			
75/16	4.01E-08	3.70E-08	4.63E-05	7.12E-05	2.8 8E-0 5	4.10E-05	5.58E-05	2.31 E-05	4.18E-05
75/18	1.75E-05	6.41E-06	3.09E-05	2.74E-05	2.00E-05	1.93E-05	2.39E-05	1.82E-05	2.12E-05
79/1	9.58E-06	4.94E-05	7.32E-06	2.52E-08	2.52E-05	4.10E-08	3.29E-06	3.03E-05	4.82E-08
79/3	7.89E-06	1.40E-05	1.33E-05	3.74E-08	1.17E-06	1.16E-06	4.19E-08	1.21E-05	1.16E-05
79/4	9.97E-08	2.02E-05	8.22E-06	3.36E-06	1.92E-05	4.88E-08	4.38E-08	2.03E-05	5.42E-08
79/6	6.64E-06	2.74E-05	5.81 E-08	1.91E-06	2.82E-05	3.49E-08	2.55E-08	3.02E-05	3.80E-06
79/17	1.48E-05	2.33E-05	2.63E-06	1.27E-05	2.53E-05	1.98E-05	1.89E-54	2.49E-05	2.09E-05
							<u> </u>	<u> </u>	
132/7	3.15E-05	2.84E-05	2.34E-05	1.75E-05	2.48E-0	1.80E-05	2.04E-05	2.55E-05	1.91E-05
132/12	1.13E-05	3.47E-05	1.20E-05	9.54E-06	1.90E-0	5.79E-08	1.09E-05	2.38E-05	6.87E-06
132/13	2.30E-05	2.58E-05	2.74E-05	1.87E-0	1.83E-08	1.93E-05	2.05E-05	2.05E-05	2.12E-05
132/16	3.00E-05	2.88E-05	1.39E-05	1.68E-0	1.84E-0	1.03E-05	2.05E-08	2.06E-05	1.07E-05
132/18	2.32E-05	8.20E-05	1.84E-0E	1.07E-0	3.14E-0	1.07E-06	1.32E-0	3.69E-05	1.16E-06
94/7	1.24E-05	4.47E-07	4.01 E-05	5.05E-08	9.01E-0	7 2.51 E-05	3.99E-08	6.78E-07	2.91 E-05
94/10	1.99E-05	1.46E-08	8.97E-0	4.30E-0	6.03E-08	2.03E-0	4.66E-08	5.69E- <u>0</u> E	2.89E-05
94/13	1.03E-05	8.01E-07	3.62E-0E	2.03E-0	2.59E-0	2.33E-0E	1.48E-0	2.15E-0E	2.54E-05
94/15	2.50E-05	1.33E-06	3.38E-0E	3.19E-0	3.99E-0	3 2.89E-0E	3.18E-0	3.34E-0€	3.08E-05
94/16	4.18E-05	8.16E-07	3.77E-06	6.19E-0	1.00E-0	3.21 E-05	5.65E-0	7.47E-06	3.47E-05
94/18	2.10E-06	5.30E-06	3.33E-0	4.87E-0	2.50E-0	3.08E-0	4.03E-0	2.25E-0	3.19E-05
			1						
100/2	2.38E-06	2.72E-08	2.08E-08	1.74E-0	5.87E-0	5 1.40E-0	1.27E-0	5 5.51 E-06	1.59E-05
100/8	6.13E-06	2.09E-06	3.30E-0	9.44E-0	4.59E-0	5 2.69E-0	1.67E-0	3.92E-0	2.72E-05
100/9	2.13E-06	8.98E-0	3.53E-06	9.95E-0	2.68E-0	8 4.11E-06	3.69E-0	5 2.44E-00	4.25E-05
				1					
7/1	8.67E-06	1.67E-0	2.58E-0	9.18E-0	1.43E-0	5 1.67E-0	8.99E-0	8 1.53E-0	1.88E-05
7/6	7.11E-06	2.10E-0	1.12E-0	4.36E-0	1.77E-0	5 6.62E-06	4.80E-0	8 1.88E-0	7.30E-06
7/8	1.78E-06	9.46E-0	1 09E-0	18.64E-0	1.03E-0	5 8.00E-0	1.00E-0	5 1.01 E-0	8.61 E-06
7/13	5.11E-06	3 7.91 E-0	319.18E-0	3 2.93E-0	8 1.25E-0	6.13E-0	3.17E-0	8 1.21E-0	6.49E-06

4 OBSERVATIONS

The sorptivity determined from each test case was plotted against the carbonation depth. Figures 2 to 10 show these plots with all the samples combined. The following was noted:

4.1 One minute regression

The external surface plot, Figure 2, showed a rough grouping of points. A line could be drawn through these points showing a linear relationship.

Figure 3 depicting section A-A (uncarbonated), showed a non-linear relationship between sorptivity and depth of carbonation.

Section B-B (Figure 4) showed a relationship very similar to that of the external surface but with less scatter. This could be because surface skin effects were eliminated. These effects could be from paint or other compounds placed on the exposed surface in the original structures.

4.2 Fourteen minute regression

The external surface showed the same type of relationship as with the 1 minute study, but was not as widely scattered. The reduction in scatter could be due to the sample reaching a point of constant sorptivity, since it was less affected by variations in the initial "skin" capillary suction.

Section A-A showed no relationship at all. The points were not as badly scattered as with the 1 minute regression, but it still did not show any significant pattern.

Section B-B showed a relationship, which was very similar to that in the 1 minute test. From plots of the sorptivity data, it appears that in many cases, the 14 minute data (from 1 to 15 minutes of test) gives the best linear relationships.

4.3 Full 15 minute regression

The external surface plot was very scattered, but a relationship was drawn. The scatter could be due to the average of two different sorptivities (Figure 8).

Section A-A was very scattered and a relationship could not be observed (Figure 9).

Results at Section B-B showed a very good relationship, which was consistent with the other regression slopes. This is because the test done on this surface seemed to exhibit one slope consistently throughout the test.

4.4 Observations based on the method of casting

While the curing method used for each sample is unknown, further analysis can be conducted by differentiating samples by how they were cast. The samples can be subdivided into precast concrete (PC), concrete taken from the underside of balconies (B), and vertically cast concrete (V). While the orientation of each sample was also noted, the size of the data base is not large enough to draw any sufficient conclusions about the effect due to orientation. By examining sorptivities determined from the 14 minute test case, vs. carbonation depths for these three categories, the following data were tabulated (Table 3).

Table 3. Ranges of 14 Minute Sorptivity Data for Various Building Elements

	Depth from Surfaces	V	В	PC
Average Carbonation Depth (mm)		19	9	4
Ext. Sorptivity Range (10 ⁻⁵ g/mm ² /s ^{0.5})	0 mm	0.25 - 7	0.2 - 4	1 - 2
B-B Sorptivity Range (10 ⁻⁵ g/mm ² /s ^{0.5})	2 mm	1 - 6	0.1 - 6	1.7 - 3.2
A-A Sorptivity Range (10 ⁻⁵ g/mm ² /s ^{0.5})	Approx. 50 mm	0.25 - 4	0.3 - 4.6	0.5 - 2

Remarks

- 15 minute sorptivity data is tabulated.

Vertically Cast (V):

- The sorptivity of the external surface was greater than or equal to the sorptivity of the internal concrete (section A-A) in 8/10 cases.
- The external surfaces without the skin (section B-B) had sorptivities greater than those of the interior concrete in only half the cases.

- Balcony Concrete (B): The sorptivity of the external surface was less than or equal to the sorptivity of the internal concrete (section A-A) in 11/14 cases.
 - Of the 3 cases that deviated, 2 were within $0.5E-5~g/mm^2/s^{0.5}$ in sorptivity.
 - External surfaces without the skin (section B-B) had sorptivities greater than those of the interior concrete in only half the cases.

- Precast Concrete (PC): External surfaces had sorptivities greater than or equal to those sorptivities of section A-A in 4/4 cases.
 - The uncarbonated Section B-B had a greater sorptivity than the interior concrete in 3/4 cases.

5 CONCLUSIONS

A relationship can be drawn between sorptivity and carbonation depth. Figures 2 to 10 presented widely scattered plots, but this is expected because of the small sample size (coarse aggregate effects are large). The best linearity of the sorptivity data was obtained for if the first minute was ignored and slopes were calculated on the remaining 14 minutes of data. Categorizing the concrete by the type of structure cast also revealed an interesting correlation. Generally there exists a positive linear relationship between sorptivity and carbonation depth.

6 RECOMMENDATIONS

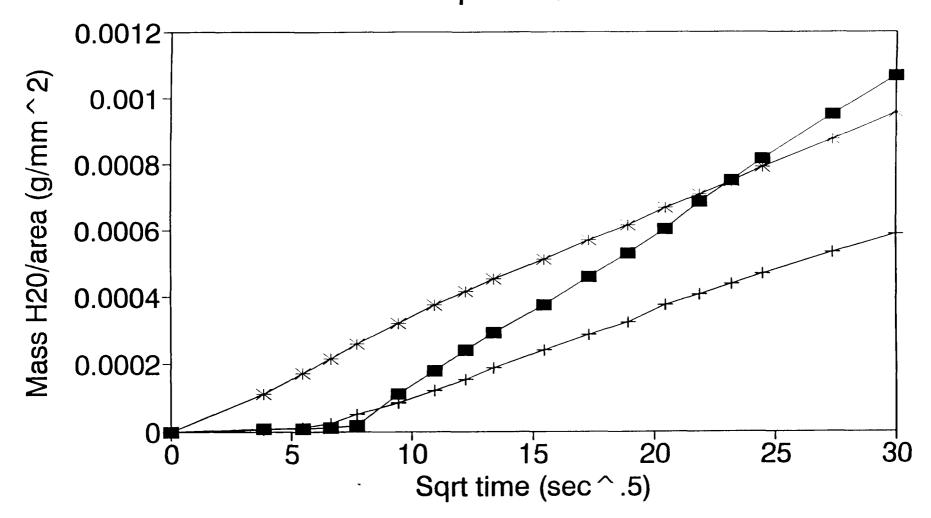
If further work is to be conducted on a sorptivity test as an indicator of concrete quality, a number of changes should be considered.

- 1. Larger diameter cores (at least 4 times larger than the maximum coarse aggregate size, and preferably at least 95 mm) should be used to reduce variability of sorptivity results.
- 2. Cores should be tested as soon as obtained. The cores in the study had been air stored for approximately 2 years and may have further carbonated during storage. This may have affected the sorptivity tests.

7 REFERENCES

- 1. C. Hall, "Water Sorptivity of Mortars and Concretes: A Review: Magazine of Concrete Research, Vol. 41, No. 147, 1989, pp. 51-61.
- 2. W. Dolch and J. Lovell, "Wetting and Drying as Indicators of Water/Cement Ratio". American Concrete Institute SP-100, 197, pp. 509-517.

Figure 1: Sorptivity Test Sample 94/18



— Ext. surface — Section A-A — Section B-B

Fig. 2 Sorption vs. Carbonation Depth External Surface (1 Minute)

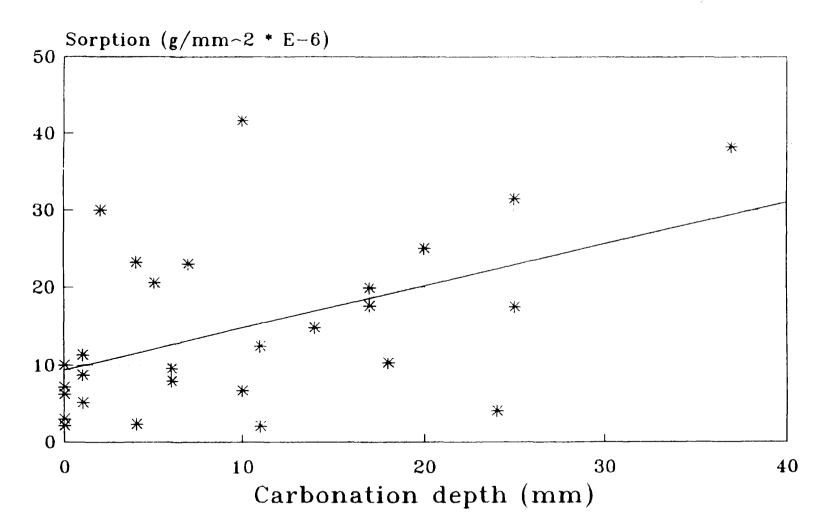


Fig. 3: Sorption vs. Carbonation Depth Section A-A (1 Minute)

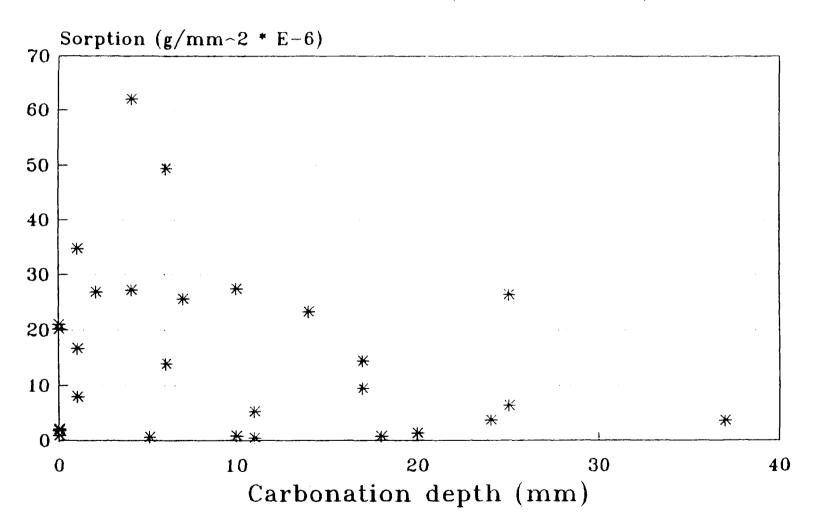


Fig. 4 Sorption vs. Carbonation Depth Section B-B (1 Minute)

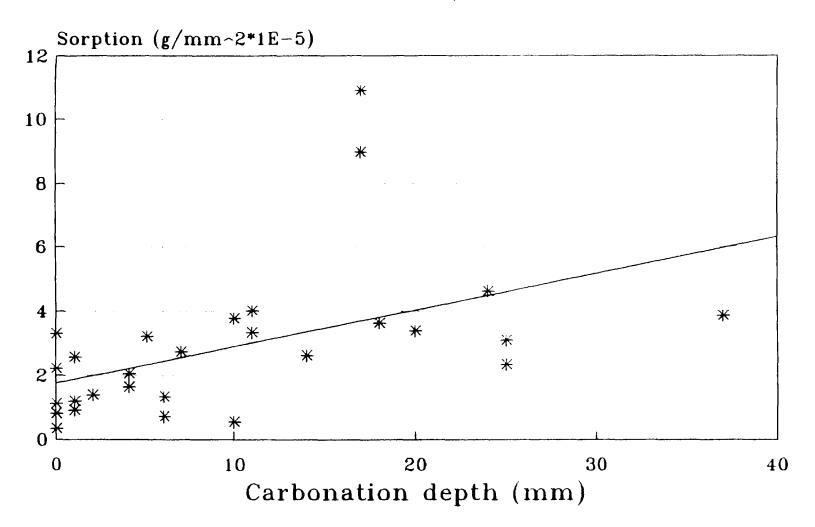


Fig. 5 Sorption vs. Carbonation Depth External Surface (14 Minute)

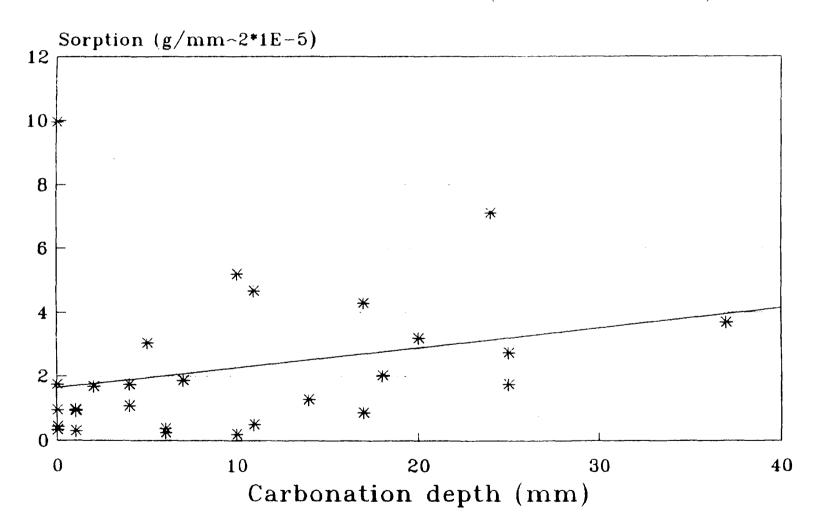


Fig. 6 Sorption vs. Carbonation Depth Section A-A (14 Minute)

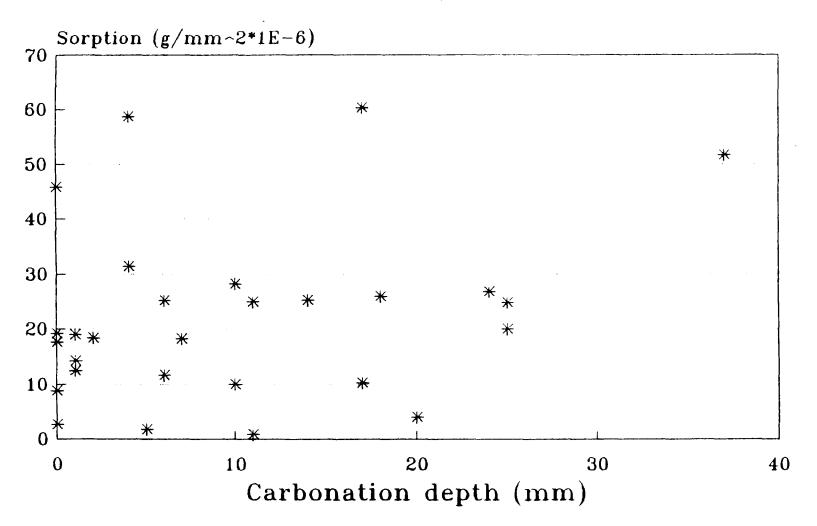


Fig. 7 Sorption vs. Carbonation Depth Section B-B (14 Minute)

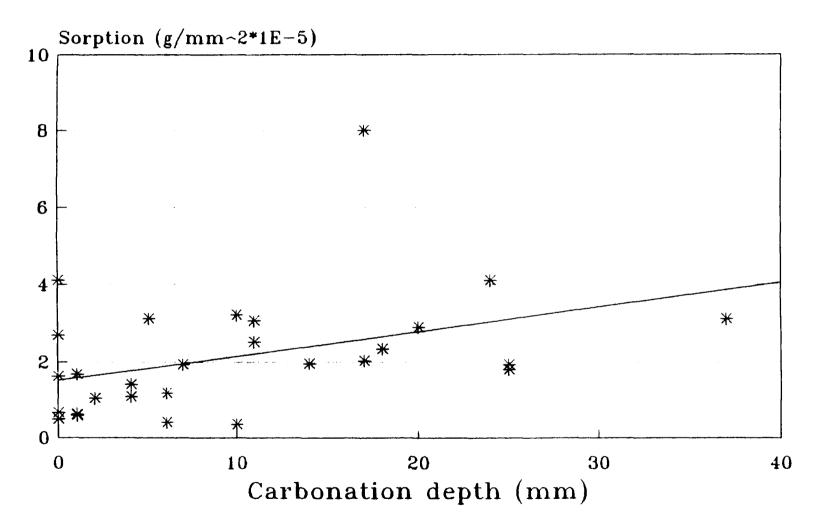


Fig. 8 Sorption vs Carbonation Depth External Surface (15 Minute)

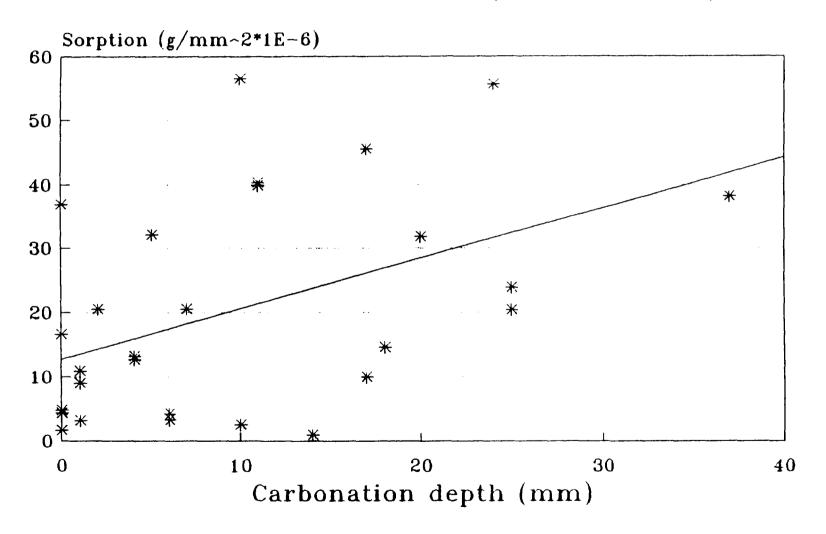


Fig. 9 Sorption vs. Carbonation Depth Section A-A (15 Minute)

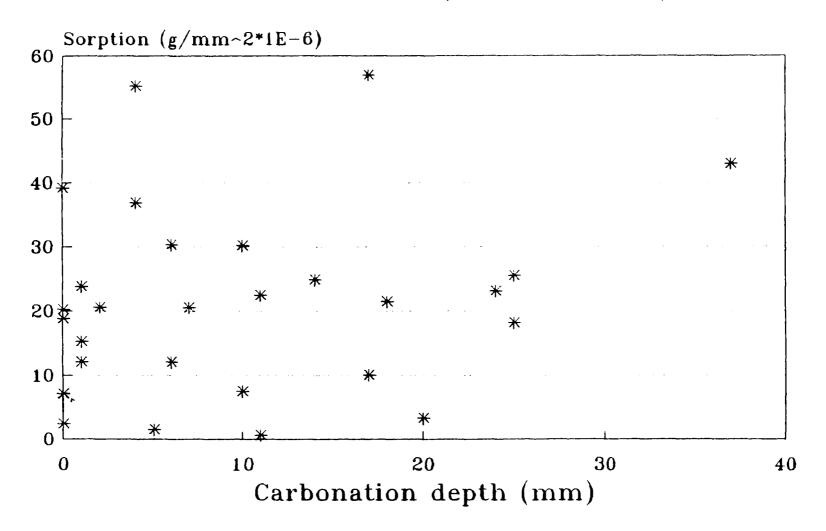


Fig. 10 Sorption vs Carbonation Depth Section B-B (15 Minute)

