







Cement and Concrete Research 34 (2004) 813-820

The pH measurement of concrete and smoothing mortar using a concrete powder suspension

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Received 10 April 2001; accepted 17 September 2003

Abstract

A simple method is presented to determine the pH value of concrete and fine smoothing mortar. The method is based on mixing powdered samples with a solvent and measuring the pH of the suspension. The parameters affecting the result are investigated. The validity of the pH results is tested by comparing the values measured from a suspension of pulverized specimen with the extracted pore solution. Measurements were done by using a pH electrode and by titration. The technique investigated furnishes results with good reproducibility and has been proven feasible. The pH value results of the proposed suspension method could be compared with the results of the extracted pore solution in two normal-strength concretes and the fine smoothing. Sufficient pore solution of the extra-air-entrained concrete and high-strength concrete could not be obtained in the reference method of extraction of the pore solution. The difference between the results of the suspension method and the extraction method was 0.07 pH units on the average with a standard deviation of 0.27 pH units when the powder-to-suspension ratio in the suspension method was 0.5.

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Keywords: Concrete; Mortar; pH; Pore solution; Powder suspension

1. Introduction

In many cases the fairly slow drying rate of concrete floors governs the obtainable building rate. In Nordic countries, a commonly used criterion for measuring the time when concrete floor can be coated with floor covers is the relative humidity (RH) value in pore structure. The floor-covering criterion used can be expressed as follows:

$$RH_{actual} < RH_{critical}$$
 (1)

in which RH_{actual} is the actual moisture condition at the surface of the subfloor, or the highest moisture conditions that will occur at the surface when the remaining moisture of construction will be redistributed. The value of RH_{actual} is compared to the value of $RH_{critical}$, which specifies the RH value of the used smoothing mortar, adhesive, floor covering, or the floor-covering material combination that it can

tolerate. RH_{critical} is a material parameter that is specified in the local standards or by the material supplier.

However, the breakdown of the glues and floor coverings is also affected by the properties of the smoothing mortar layer and the concrete floor. For instance, in high-strength concrete floors having a high cement content and a low water—binder ratio, which causes self-desiccation in the pore system, the destruction of floor coverings can take place in relatively low RH environment, compared to that of the ordinary concrete floors. This is mainly due to the higher alkali amount and the finer pore structure of the self-desiccating concrete. One way to take into consideration the ion transport properties of different concretes is to use additional criteria. According to Sjöberg [1], the problem of alkaline degradation can be expressed as

$$pH_{actual} < pH_{critical}$$
 (2)

in which pH_{actual} is the actual pH value at the surface of the subfloor as a result of the composition of the concrete or the smoothing mortar used between the floor cover and concrete. The value of pH_{actual} is compared to the value of $pH_{critical}$, which specifies the pH value the floor covering material or

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floor covering material combination used is considered to tolerate.

There remains a problem: how to measure the pH of a concrete slab surface? A commonly used method to measure the pH of concrete is to press pore water out of concrete and to analyse the extracted solution. The technique is well explained by Barneyback and Diamond [2], and a number of studies have been reported in which the method has been tested and solutions have been analysed [3-10]. Unfortunately, it is very difficult to press pore water from dried concrete slabs that are older than a few days, especially if the slabs are made from high-strength concrete. On the other hand, the samples cured in water do not express correctly the ion transport in concrete slabs and the smoothing mortar layer. In addition, the use of special high-pressure devices seems impractical as a routine test together with RH measurements in building sites, when the right covering time of concrete slab has to be determined. There is a need for a more practical and cheaper pH-measuring method of concrete.

The method presented in this article is based on mixing powdered concrete or smoothing samples with a solvent and measuring the pH of the suspension. At present the method is already in use, but the parameters affecting the results are poorly documented. In addition, a similar extraction technique has been used in determining the free chloride ion content of hydrated cement pastes [7,10,11], and the technique is widely used in determining the pH value of soil [12].

The proposed method is also suitable in measuring the pH of the pore solution in other aspects of concrete technology, for example, with regard to rebar corrosion and alkali silica reaction.

2. Experimental program

2.1. Materials, mixes and specimen conditioning

Chemical analyses and other characteristics of the cements and condensed silica fume used in this work are presented in Table 1. Some data of the typical composition of the fine smoothing mortar are presented in Table 2. The mix proportions and properties of the test concretes are presented in Table 3. In Table 3, NSC denotes normal-strength concrete made from the blended cement CEM II 42.5R, NSWC is a normal-strength concrete made by white cement CEM I 52.5, HSC stands for high-strength concrete, and EAEC is extraair-entrained concrete having 8-10% air content. Both HSC and EAEC are made by blended cement CEM II 42.5R. The mineralogical composition of the aggregates was mostly granite. The superplasticizer used was Scancem SP62, which is a sulphonated naphthalene formaldehyde condensate, also known as Mighty 150. In EAEC concrete, also an airentraining agent based on a vinsol resin was used. Water used in the test concretes was Espoo city tap water. All concretes were mixed with a vertical shaft batch mixer. After mixing, concretes were cast into plastic moulds having

Table 1
Chemical analyses and other characteristics of the cements and silica fume

	White Portland cement CEM I 52.5	Blended cement CEM II 42.5R	Condensed silica fume
CaO (%)	68.6	60.6	0.35
SiO ₂ (%)	25.0	20.1	96.4
Al ₂ O ₃ (%)	1.89	4.9	0.60
Fe ₂ O ₃ (%)	0.33	2.5	1.05
SO ₃ (%)	2.05	3.3	0.40
MgO (%)	0.54	3.0	1.24
Na ₂ O equivalent (%)	0.22	1.43	1.46
Loss of ignition (%)	0.59	3.1	2.7
Insoluble residue (%)	0.07	1.2	0.48
Blaine fineness (cm ² /g)	385	460	
Compressive strength (MPa)			
1 day	18	26	
7 days	51	45	
28 days	69	54	

horizontal dimensions of 400×600 mm and a height of 170 mm. During the first 5 days after the casting, the surface of the concrete slab was covered with a plastic sheet. Thereafter the slabs were cured uncovered in plastic moulds at 45% RH and 20 °C.

The fine cover smoothing used in this investigation was Vetonit 3000. The hydraulic binder was composed of aluminate cement and Portland cement CEM I 42.5R. Water/solid ratio of the fine smoothing was 0.28. The fine smoothing was mixed with a fast rotating blade mixer and the mixing time was 2 min. The smoothing was cast into plastic moulds having horizontal dimensions of 400×600 mm and a height of 30 mm. During the first 5 days after casting, the surface of the fine smoothing slabs was covered with a plastic sheet. After that the slabs were cured uncovered in moulds at 45% RH and 20 °C.

2.2. Techniques

To avoid errors in measurements due to carbonation and moisture content gradient, the concrete samples used for the pH measurements were manually cut at the distance of 68 mm from the surface of the slab. The fine smoothing mortar samples were cut from the center of mortar slabs. The RH values of the test concretes were measured using Vaisala Oy's capacitive HMP44 temperature and humidity measuring probes. The RH values of the test concretes NSC, NSWS, EAEC and HSC at 68 mm at the test age of 7 days were 96.7%, 97.9%, 85.5% and 84.7%, respectively. At the age of 180 days, the RH values were 86.3%, 87.1%, 68.3% and 75.8%, respectively.

Large samples were crushed with a crushing press. After crushing, the samples used for the pH measurements were powdered with a grinding machine. Some 30 g of concrete or fine smoothing was powdered, the grinding time being 20 s. Thereafter, 15 g of powder and 15 g of distilled water or for solvent/solid ratio tests, an appropriate amount of

Table 2
An approximate composition of the fine smoothing

	%
Hydraulic binding agents	~ 20
Calcium carbonate	~ 70
Dispersion powder	< 5
Spread increasing agents	< 1
Foam inhibitors	< 1
Inorganic salts	< 1

distilled water, was weighed with an accuracy of 0.1 g and mixed together by using a CM200 cell mixer having a rotation speed of 35 rpm. Mixing started 1 min after the materials were weighed and it took 15 min except when the effect of mixing time on the pH was tested. After mixing some 10 ml of suspension was poured into a test tube and its pH measured. The suspensions were not filtered unless otherwise mentioned.

After crushing, the samples used for pore solution extraction were manually tightened inside the compression cylinder having a diameter of 30 mm and height of 72.5 mm. A 500-MPa pressure was applied to the piston of the high-pressure compression apparatus and the sample was completely crushed. Thereafter, typically some 5–10 ml of pore solution was extracted. From EAEC and HSC test concretes adequate amount of pore solution could not be obtained. The pore solution was delivered through a drain ring and channel and recovered in a syringe. The pore solution was poured into a test tube and pH of the solution was measured.

2.3. Determination of pH value

In evaluating the technique to determine the pH of concrete and cover smoothing by using a suspension of concrete or smoothing powder, the following parameters were considered as significant: solvent/solid ratio, fineness of particles, filtration, extraction time, stirring time, temperature of powder and solvent, and the ability of particles to

Table 3
Mix proportions and properties of the test concretes

	Mix reference (cement type)						
	NSC (CEM II 42.5R)	NSWC (CEM I 52.5)	EAEC (CEM II 42.5R)	HSC (CEM II 42.5R)			
Cement content (kg/m ³)	240	240	475	475			
Silica content (kg/m ³)	0	0	28.5	28.5			
Water (kg/m ³)	180	180	161	173			
Superplasticizer (kg/m ³)	0	0	7	7			
Air-entraining agent (kg/m ³)	0	0	1.2	0			
Aggregate amount (kg/m ³)	1950	1950	1677	1677			
Water/binder ratio	0.75	0.75	0.30	0.33			
Consistency: s VB	1.1	0.9	1.5	1.8			
Air content (%)	< 2	< 2	9	< 2			
28-Day compressive strength (MPa)	34	30	44	74			
Density (kg/m ³)	2410	2410	2220	2430			

resist pulverization. The effect of the solvent type on the pH value of concrete and cover smoothing was not tested in this research project. The solvent used in all of the pH measurements was distilled water.

The pH value of each solution and suspension was measured using the Delta OHM HD 8705 pH meter and PCFC11 combination electrode. The pH value was accepted when the change rate of the value was less than 0.01 units per minute. At least two measurements of each test were taken. The validity of the pH results was tested by comparing the values measured from the suspension of pulverized specimen of the extracted pore solution, and as measured using a pH electrode and calculated from the titration results. For validity tests, the amount of replicates was increased up to 10, in order to carry out a statistical analysis of the results. The solutions used were titrated against 0.1 N HCl; phenolphthalein, having a color change between pH 8.2 and 10, was used as an indicator. The pH meter was calibrated using two of the buffer solutions fabricated by Reagegon with pH values of 10, 11, 12 and 13 depending on the pH values to be measured. The calibration was done before and after each daily measurement, and if the difference between any of the calibrations exceeded 0.01 units the measurements were repeated. The measurement of each tested parameter was performed from the same test sample and during 1 day, if possible, to eliminate errors due to changes in calibration and testing conditions.

3. Results and discussion

3.1. Solvent/solid ratio

The pH results of concrete and fine smoothing powder are illustrated in Figs. 1 and 2, respectively. The amount of concrete and fine smoothing powder was measured as a portion of the total weight of the mixture. The results are given as averages of two replicate samples. The results in Fig. 1 show that the higher the portion of concrete powder is, the greater the pH of the suspension. This is probably

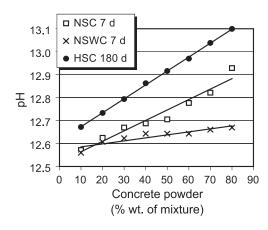


Fig. 1. Effect of concrete powder portion on the pH of concrete.

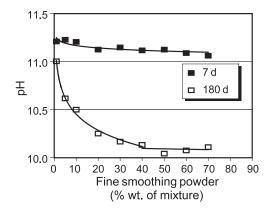


Fig. 2. Effect of fine smoothing powder portion on the pH of fine smoothing mortar.

because if there is more concrete surface in contact with distilled water, also more OH ⁻ is likely to dissolve. The correlation between the pH of concrete and the amount of concrete powder seems to be almost linear. The results also indicate that with increasing powder portion the difference between the pH values of the test concretes increases. Test concrete HSC, which had the highest cement amount and the greatest alkali portion, did undergo a remarkable increase in pH with increasing amount of concrete powder. The increase in pH of low-alkali and low-cement test concrete NSWC was moderate. The curves of NSC at the age of 7 and 180 days were alike and the results of the test concrete EAEC followed closely the curve of HSC.

With fine smoothing mortar samples low in smoothing powder the results were quite the opposite: the higher the portion of smoothing powder was, the lower the pH value of the suspension. The pH value of the 7-day-old fine smoothing mortar started to increase extensively with powder amounts lower than 40% when the increase in pH of 180-day-old smoothing mortar was fairly moderate. With samples rich in smoothing powder, the pH of both 7- and 180-day-old smoothing mortar samples remained fairly constant. The likely reason for these phenomena is the large amount of calcium carbonate in the fine smoothing layer. Calcareous soils are known to have a fairly constant

Table 4
Effect of fineness of concrete and fine smoothing mortar on the pH value

					_		
Particle size fraction (mm)	pH of concrete, 7 days		pH of concrete, 180 days			pH of fine smoothing mortar	
	NSC	NSWC	NSC	EAEC	HSC	7 Days	180 Days
>16	12.08	12.22	12.22	12.16	12.14	11.33	10.82
8 - 16	12.21	12.32	12.30	12.22	12.15	11.41	10.77
4 - 8	12.38	12.42	12.44	12.42	12.36	11.36	10.29
2 - 4	12.51	12.55	12.53	12.58	12.53	11.43	10.24
1-2	12.58	12.61	12.64	12.63	12.67	11.33	10.19
0.5 - 1	12.66	12.63	12.72	12.75	12.81	11.20	10.26
0.25 - 0.5	12.67	12.65	12.74	12.82	12.85	11.09	10.23
0.125 - 0.25	12.73	12.65	12.77	12.89	12.91	11.11	10.20
$0.075\!-\!0.125$	12.79	12.67	12.79	12.91	12.92	11.19	10.20
0.075>	12.80	12.68	12.81	12.91	12.93	11.19	10.20

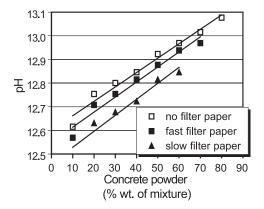


Fig. 3. Effect of filtration on the pH of EAEC, age 180 days.

pH value as long as there is enough calcium carbonate present to be dissolved [12]. If the portion of fine smoothing powder diminishes enough there is not any more sufficiently low-solubility calcium carbonate present and the pH of fine smoothing mortar starts to increase. In addition, it is known that the lower solubility of calcium carbonate gives a lower pH equilibrium value [12]. This could explain why the pH value of the 180-day-old fine smoothing mortar is lower than that of the 7-day-old fine smoothing mortar.

3.2. Fineness of particles

Table 4 shows how the fineness of concrete and fine smoothing mortar affects pH. After crushing, the samples were sieved into 10 particle size fractions presented in Table 4. The results indicate that with increasing fineness of concrete particles the pH of the suspension increases. Because of larger concrete surface in contact with solvent more OH $^-$ is leached into the water, more H $^+$ reacts with OH $^-$, and the pH of the suspension starts to increase. With coarse smoothing mortar samples the amount of calcium carbonate is not adequate to keep the pH value low, but with finer particles 1 mm or less in size, there is enough particle surface in contact with the solvent and enough calcium carbonate is leached into the water.

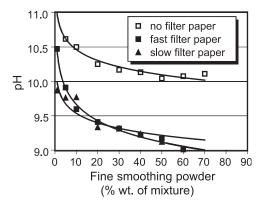


Fig. 4. Effect of filtration on the pH of fine smoothing mortar, age 180 days.

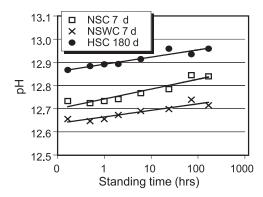


Fig. 5. Effect of extraction time on the pH of concrete.

3.3. Filtration

The effect of filtration on the pH values of concrete and fine smoothing mortar is illustrated in Figs. 3 and 4, respectively. Two filter papers manufactured by Schleicher & Schuell were used: fast-filtering no. 589/1 and slow-filtering no. 589/3. Results of all concrete tests were alike. Filtration decreased pH values slightly and this decrease remained fairly constant with different solvent/solid ratios investigated. The use of thicker, slow-filtering 589/3 caused a greater decrease of concrete pH than was detected with 589/1. As far as the effect of filtration on the pH was concerned no notable differences between the various concrete types and ages could be detected

With concrete and fine smoothing mortar powder amounts exceeding 60% and especially when the slow-filtering paper 589/3 was used, not enough pore water was always filtered in achieving a reliable pH value. With fine smoothing mortar samples that had a fairly low pH value the effect of filtration on the pH was remarkable, causing a decrease of pH even by one unit. Therefore, the effect of filtration on the pH values of concrete and fine smoothing powder solutions was detected to be dissimilar. This is suggested to be due to logarithmic pH scale. When the amount of H⁺ in the solution changes, the lower pH value of smoothing mortar solution changes more than the considerably higher pH value of concrete powder solution.

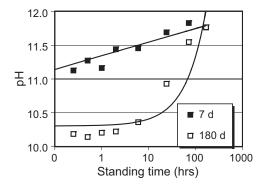


Fig. 6. Effect of extraction time on the pH of fine smoothing mortar.

Table 5
Effect of stirring time on the pH values of concrete and fine smoothing mortars

Stirring time (min)	pH of o	concrete,		pH of concrete, 180 days		rete, pH of fine smoothing morta	
	NSC	NSWC	NSC	EAEC	HSC	7 Days	180 Days
0.5	12.73	12.58	12.78	12.87	12.87	11.06	10.13
1	12.73	12.58	12.77	12.87	12.87	11.00	10.15
3	12.73	12.59	12.78	12.89	12.89	11.24	10.14
5	12.74	12.62	12.77	12.89	12.89	11.12	10.25
15	12.72	12.64	12.78	12.90	12.91	11.13	10.22
30	12.73	12.64	12.78	12.90	12.92	11.34	10.22
60	12.75	12.66	12.80	12.92	12.94	11.13	10.15
120	12.76	12.67	12.80	12.95	12.95	11.24	10.20

3.4. Extraction time

Figs. 5 and 6 show how extraction time affects the pH of concrete and fine smoothing mortar, respectively. The powdered samples were allowed to stand in distilled water from 1 min up to 1 week. As can be seen from the illustrations for all concretes and smoothing mortars tested, pH value increases continuously over the whole test period. At the test age of 1 week, the pH of 180-day-old fine smoothing mortars had reached the pH of 7-day-old fine smoothing mortar.

3.5. Stirring time

All samples were mixed by a cell mixer having moderate rotation speed of 35 rpm. The effect of stirring time on the pH values was tested by varying the duration of mixing from 30 s to 2 h. The results of the stirring time tests are presented in Table 5. It seems that an increase in stirring time increases the pH of concrete and fine smoothing mortar although the detected changes are moderate. Part of this increase can be explained by the effect of extraction time on the pH illustrated earlier in Figs. 5 and 6.

3.6. Temperature of powder and solvent

The effect of powder temperature on the pH of concrete and fine smoothing mortar was also investigated. According to the experiment, when a hole is drilled in concrete, the

Table 6
Effect of powder temperature on the pH of concrete and fine smoothing mortar

Powder temperature (°C)	pH of concrete, 7 days		pH of concrete, 180 days		pH of fine smoothing mortar		
	NSC	NSWC	NSC	EAEC	HSC	7 days	180 days
20	12.73	12.67	12.76	12.88	12.90	11.14	10.17
40	12.74	12.66	12.75	12.87	12.89	10.43	10.09
60	12.71	12.64	12.75	12.86	12.89	10.49	10.15
80	12.72	12.64	12.75	12.86	12.88	10.44	10.18
100	12.73	12.64	12.75	12.85	12.84	10.30	10.22

Table 7
Effect of solvent temperature on the pH of concrete and fine smoothing mortar

Water temperature (°C)	1	concrete,	pH of c	pH of concrete, 180 days		
	NSC	NSWC	NSC	EAEC	HSC	180 days
10	12.74	12.65	12.77	12.89	12.89	10.14
20	12.74	12.65	12.77	12.89	12.88	10.19
30	12.72	12.65	12.77	12.89	12.89	10.16
40	12.72	12.68	12.77	12.89	12.89	10.19
50	12.69	12.72	12.78	12.89	12.88	10.18

temperature of the concrete powder in a drilling hole can easily rise up to 80 °C. All tested samples were powdered with a grinding machine, the grinding time being 20 s. The powder was heated and thereafter it cooled back to 20 °C. Cooled powder was then mixed with the same amount of water and after 15 min of mixing the pH value of the suspension was measured. The results are presented in Table 6. It seems that the high temperature of concrete or fine smoothing mortar powder can decrease the obtainable pH value. The detected changes in pH values were small with one exception. The pH of 7-day-old smoothing mortar decreased considerably.

Table 7 shows the effect of solvent temperature on the pH of concrete and fine smoothing mortar. Powder having the temperature of 20 °C was mixed with distilled water having different temperatures. Stirring took 15 min and the temperature of the mix at the end of stirring had already fallen down close to 20 °C. As can be seen from the results, changes in the pH values are small and no clear trend can be detected. It is possible that the increase of extraction time and the use of heat insulation around the mixing container caused greater changes on the pH values.

3.7. The ability of the particles to resist pulverization

In Fig. 7 some grinding curves of NSC and HSC are presented. Concrete properties affect the ability of the concrete to resist pulverization. The same grinding time in a grinding machine can give unequal fineness for concrete

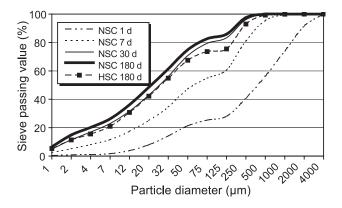


Fig. 7. Grinding curves of test concretes NSC and HSC.

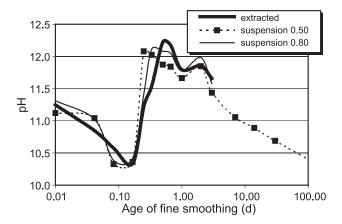


Fig. 8. pH value evaluations of fine smoothing mortars measured from the suspension and extracted pore solutions.

samples of different ages, strengths or elasticity properties. Because the fineness of concrete affects pH, the ability of concrete samples to resist pulverization also has an effect on pH. All samples had the same 20-s grinding time. Particle size distribution of the concrete powders was analysed with a Cilas Alcatel granulometer. From the grinding curves presented it can be seen that for NSC an increase in concrete age gives an increase in powder fineness. Therefore, it is likely that increasing concrete age also gives an increase of pH according to the results presented earlier in Table 4. The fineness of concrete powder increased from test age 1 to test age 7 days. HSC powder at the age of 180 days was somewhat coarser than powder of NSC at the same age.

3.8. Reliability and reproducibility of the test method

The validity of the pH results measured from the suspension of the pulverized specimen was tested by comparing these values to the values measured from the extracted pore solution. The results for fine smoothing mortars, NSWC and NSC are illustrated in Figs. 8, 9 and 10, respectively. Results are averages of two samples. In the figures, "extracted" denotes the results of the extracted pore solutions. "Suspen-

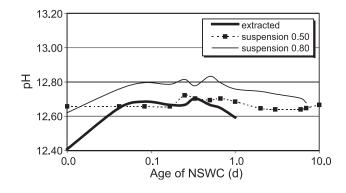


Fig. 9. pH value evaluations of NSWC measured from the suspension and extracted pore solutions.

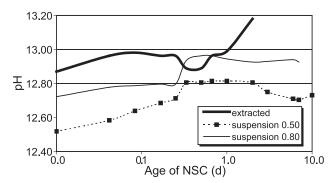


Fig. 10. pH value evaluations of NSC as measured from the suspension and extracted pore solutions.

sion" is a suspension of the pulverized specimen and the number after the name indicates the portion of concrete powder in the total weight of the mixture.

Only small differences between the curve of the extracted pore solution compared to the curves of suspensions of fine smoothing mortars can be detected in Fig. 8. During the first few days after the casting, the investigated curves were similar, although slight differences in the position of the main peaks exist. After a few days, inadequate amount of pore solution could be extracted with the compression device. In addition, the powder-to-suspension ratio of 0.80 was too dry in achieving a reliable pH value at later ages. When the ratio was 0.5, there was no difference between the results of the suspension method and the extraction method on the average with a standard deviation of 0.38 pH units. When the powder-to-suspension ratio was 0.8, the difference between the results of the suspension and extraction methods was -0.06 pH units on the average with a standard deviation of 0.22 pH units. The correlation of the results of the two methods can be considered quite good. At later ages, the suspension method with 0.5 powder-tosuspension ratio was the only method that produced measurable and consistent results.

The pH results of test concrete NSWC are illustrated in Fig. 9. When the powder-to-suspension ratio was 0.5, the difference between the results of the suspension and extraction methods was -0.04 pH units on the average with a standard deviation of 0.09 pH units. With the powder-to-suspension ratio of 0.8, the respective differences were -0.14 pH units on the average with a standard deviation of only 0.04 pH units. The results show that the difference between the methods is very small.

The pH results of test concrete NSC are shown in Fig. 10. When powder-to-suspension ratio was 0.5, the average of the differences between the results of the suspension and extraction methods was 0.25 pH units and the standard deviation was 0.12 pH units. At powder-to-suspension ratio of 0.8, the respective average difference is 0.11 pH units with a standard deviation of 0.11 pH units. These differences are similarly rather small.

At powder to suspension ratio of 0.5 the difference between the test results of the suspension and extraction methods of all three aforementioned concrete and mortar tests is 0.07 pH units on the average having a standard deviation of 0.27 pH units. At powder-to-suspension ratio of 0.8, the respective values are -0.03 pH units on the average and the standard deviation of these differences is 0.18 pH units.

The validity of the pH results obtained by using a pH electrode and the reproducibility of pH values produced by the method tested were also investigated. The values measured by a pH electrode were compared to the values calculated from titration results. Ten replicates of each test were taken. Suspension samples were filtered so that the color change of phenolphthalein could be clearly detected. The results of test concrete NSC and fine smoothing mortar samples are presented in Table 8.

No remarkable differences between the standard deviations of the pH values measured from suspension of the pulverized specimen and from the extracted pore solution can be noticed. Standard deviation of the pH values measured from the suspensions of concrete powder is small. With fine smoothing mortar samples, both the suspension and solution produced a standard deviation that is fairly large in magnitude. It is suggested that this occurred because, due to duration of the tests, not all samples could be tested exactly at the same age. As presented in Fig. 8 the pH value of fine smoothing mortars changed rapidly during the first day after the casting. The pH values of suspension samples measured using a pH electrode and those calculated from the titration results were on the same level. The large increase in the pH value of NSC at the age of 2 days measured from the extracted pore solution was detected both by using a pH electrode and from the titration results. Some differences exist between the pH values of the pore solution measured by using a pH electrode and the titration results, and it is suggested that this occurred because the pH electrode could not be calibrated at buffers exceeding pH 13.

Table 8 Reliability and reproducibility of the tested pH measuring methods

	Suspension, not filtered, by electrode	Suspension, filtered, by electrode	Extracted, not filtered, by electrode	Suspension, filtered, titrated	Extracted, not filtered, titrated
NSC, age 2 days, average S.D.	12.84 (0.03)	12.78 (0.01)	13.16 (0.05)	12.81 (0.02)	13.51 (0.05)
Fine smoothing mortar, age 1 day, average S.D.	11.86 (0.08)	11.69 (0.09)	11.77 (0.06)	Not calculated	Not calculated

4. Conclusions

A method for determining the pH value of concrete and fine smoothing mortar was investigated. The method is based on mixing powdered samples with a solvent and measuring the pH of the suspension. The parameters affecting the results were investigated. The validity of the pH results was tested comparing the values measured from suspension of pulverized specimen and extracted pore solution. Measuring was done by two methods: by using a pH electrode and by a titration method.

The averages of the differences and the standard deviations of the test results produced by the two methods were quite small. The suspension method can be used to measure the pH value of hardened concrete and its changes. To minimise errors in measurement and to observe the pH value evolution of concrete and fine smoothing mortar samples the following procedures are recommended.

Particle size of the powder should be constant and as fine as possible. The powder portion should be constant and normally between 40% and 60% of the total weight of the suspension mixture. The powder portion should be high enough that the investigated phenomena can be detected and low enough that the pH value measurements can successfully be done at all test ages. The same filtration method and extraction and stirring times should be used in all measurements performed. Temperature of the powder solution should not vary more than 10 °C.

References

- A. Sjöberg, Floor system, moisture and alkali, Healthy Buildings/ IAG'97, Global Issues and Regional Solutions, vol. 3, Healthy Buildings/IAG'97, Washington, DC, 1997, pp. 567–572.
- [2] R.S. Barneyback, S. Diamond, Expression and analysis of pore fluids from hardened cement pastes and mortars, Cem. Concr. Res. 11 (2) (1981) 279–285.
- [3] H.F.W. Taylor, A method for predicting alkali ion concentrations in cement pore solutions, Adv. Cem. Res. 1 (1) (1987) 5-17.
- [4] S. Diamond, Effects of two Danish flyashes on alkali contents of pore solutions of cement-flyash pastes, Cem. Concr. Res. 11 (3) (1981) 383-394.
- [5] K. Byfors, C.M. Hansson, J. Tritthart, Pore solution expression as a method to determine the influence of mineral additives on chloride binding, Cem. Concr. Res. 16 (5) (1986) 760-770.
- [6] M.N. Haque, O.A. Kayyali, Environmental factor and concentration of CL⁻ and OH⁻ in mortars, J. Mater. Civ. Eng. 2 (1) (1990) 24–34.
- [7] C. Arya, J.B. Newman, An assessment of four methods of determining the free chloride content of concrete, Mat. Struct. 23 (137) (1990) 319–330.
- [8] I. Odler, E.N. Stassinopoulos, Über die zusammensetzung der porenflüssigkeit hydratisierter zementpasten, TIZ-Fachber. 106 (6) (1982) 394–401.
- [9] S.E. Hussain, A.S. Al-Gahtani, A.S. Rasheeduzzafar, Chloride threshold for corrosion of reinforcement in concrete, ACI Mater. J. 94 (6) (1996) 534–538.
- [10] M.N. Haque, O.A. Kayyali, Free and water soluble chloride in concrete, Cem. Concr. Res. 25 (3) (1995) 531–542.
- [11] C. Arya, N.R. Buenfeld, J.B. Newman, Assessment of simple methods of determining the free chloride ion content of cement paste, Cem. Concr. Res. 17 (1987) 907–918.
- [12] D.L. Rowell, Soil Science: Methods and Applications, Soil Acidity and Alkalinity, vol. 3, Longman Scientific and Technical, Harlow, 1996, pp. 153–174 (Chapter 8).