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Assessing the repeatability and reproducibility values of South African durability index tests

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An inter-laboratory test programme was conducted to estimate the repeatability and reproducibility of the durability index tests developed and used in South Africa: the oxygen permeability index (OPI) test, the water sorptivity test and the chloride conductivity test. Between the University of Cape Town and the University of the Witwatersrand, ten different concrete mixtures were cast with a range of properties. From each of these mixtures, test samples were prepared and then randomly distributed among the participating laboratories. Sufficient laboratories participated to enable nine laboratories to perform each test. The estimated repeatability was 1,4 % for the OPI test, 9,9 % for the water sorptivity test, and 9,1 % for the chloride conductivity test. The estimated reproducibility was 1,8 % for OPI, 12,8 % for sorptivity and 21,1 % for chloride conductivity. The variability in the oxygen permeability test is attributed mainly to material variability, while significant variability in the chloride conductivity test is attributed to the tolerances of the test apparatus, although there were indications that the procedures were not being followed correctly, either due to a lack of clarity in the procedures or the inexperience of the participating laboratories.

INTRODUCTION

Three tests have been developed for use in South Africa to aid in the control of concrete quality and prediction of the long-term performance of reinforced concrete subjected to aggressive environments. These three tests (the oxygen permeability test (OPT), the water sorptivity test and the chloride conductivity test) measure the transport properties of concrete at a given age, usually 28 days. They provide characterisation of the microstructure of the near-surface layer of the concrete (ie, the cover concrete), and have been shown to be sensitive to material parameters such as binder type, processing influences such as type and degree of curing, and environmental influences such as temperature and relative humidity (Gouws, Alexander & Maritz 2001; Griesel & Alexander 2001; Du Preez & Alexander 2004). These tests are therefore increasingly being adopted in the construction industry to provide a measure of the expected durability performance of concrete structures.

At this point, however, confident measures of the repeatability (single operator coefficient of variation) and reproducibility (between laboratory coefficient of variation) of the test procedures are lacking. This information is necessary for proper specification of limiting test values to obtain the desired performance. In addition, similar to the normal practice when specifying concrete strength, target and characteristic values need to be specified so as to account for variability.

An estimate of repeatability was provided by Gouws et al (2001) based on their experience with the various tests, as shown in table 1. This is limited to one operator, however, and includes no information on reproducibility. To help provide this information, an initial round robin test programme was conducted using the laboratories in South Africa that had identified themselves as being proficient in the test procedures. The results of this exercise were reported in Grieve et al (2003). This series of tests revealed problems with the test

Table 1 Estimates of repeatability (1s%) from Gouws et al (2001)

Concrete source	Oxygen permeability index	Water sorptivity	Chloride conductivity
Actual structures	3 %	13 %	14 %
Wet-cured, site mixed concrete	2 %	12 %	7 %
Wet-cured, ready mixed concrete	1 %	7 %	5 %
Laboratory	1 %	5 %	4 %

Table 2 Initial round robin series – range of results (Grieve et al 2003)

	Oxygen permeability index	Water sorptivity	Chloride conductivity
Repeatability	0,2-2,8 %	0,2-17,8 %	1,4-57,4 %
Reproducibility	0,5-3,0 %	8,0-22,6 %	10,9-56,7 %

Table 3 Concrete mixture properties

Concrete Designation	w/b	Cement type#	Stone type\$	Sand type*	28-day strength (MPa)
A	0,70	CEM I	Greywacke	Klipheuwel	33,4
B	0,50	CEM I	Greywacke	Klipheuwel	54,1
C	0,70	CEM I	Andesite	Granite	32,0
D	0,50	CEM I	Andesite	Granite	59,5
E	0,70	50:50 Corex Slag:CEM I	Greywacke	Klipheuwel	41,8
F	0,50	50:50 Corex Slag:CEM I	Greywacke	Klipheuwel	60,1
G	0,70	50:50 GGBS:CEM I	Andesite	Granite	29,8
H	0,50	50:50 GGBS:CEM I	Andesite	Granite	42,3
I	0,70	30:70 Fly Ash:CEM I	Andesite	Granite	23,5
J	0,50	30:70 Fly Ash:CEM I	Andesite	Granite	42,5

CEM I was all CEM I 42.5N
 \$ All stone was crushed
 * 'Klipheuwel' sand was a natural pit sand; 'granite' sand was a crushed granite sand

methods, resulting in the methods being reviewed and revised. A second testing programme was then undertaken (Grieve 2004). In summary, however, these exercises were not deemed a success. The repeatability and reproducibility ranges for each of the three tests from both these series are shown in table 2. The conclusions were that while the OPI test may be achieving satisfactory levels of repeatability and reproducibility, the sorptivity test and the chloride conductivity test were not.

The test procedures were again rewritten to improve their clarity and to more rigorously define and control the variation that could be tolerated in the various aspects of the test procedures. An attempt was also made to simplify some of the test procedures to make them more user-friendly wherever possible without sacrificing technical rigour. Once the laboratories were given time to familiarise themselves with the new laboratory procedures, a new round robin test was held. The detailed results of this exercise are reported in Stanish et al (2004). This paper is a summary of that report.

EXPERIMENTAL PROCEDURES

Durability index test procedures

Conceptually, the durability index tests are relatively easy to perform. For all three tests, nominally 68 mm diameter, 25 mm long test samples are prepared at 28 days of concrete age, four samples for each test. These samples are then conditioned in a 50 °C oven for seven days prior to testing. Samples are obtained by coring from the surface zone of larger elements, either prepared in the laboratory or in situ.

The samples for the oxygen permeability test are placed in a falling head permeameter with an initial pressure of 100 kPa. The rate of pressure decay is monitored and used to calculate a D'Arcy coefficient of permeability (Ballim 1991). This is typically transformed into an oxygen permeability index value (OPI) by taking the negative log of the average permeability value (in m/s) of all four samples.

The sorptivity test is conducted by exposing one face of the dried sample to a free water surface and monitoring the mass gain with time. As long as saturation is not approached, the rate of mass gain will be linearly related to the square root of time. This is normalised by the water-accessible porosity as determined by vacuum saturation (Kelham 1988).

The chloride conductivity test is conducted by saturating the samples with a 5 M NaCl solution by vacuum saturation. The saturated sample is then placed in a test rig with a cell filled with the same salt solution on either side. Applying a voltage across the sample and measuring the current that results allows the conductivity to be calculated. The actual voltage across the sample is measured by the use of probes within the solutions contained in the test chambers, rather than taking the voltage applied by the power supply (Streicher & Alexander 1995). The water-accessible porosity of the chloride conductivity samples is also determined from the mass before and after saturation, taking into consideration the different density of the saturating fluid (5 M NaCl).

The descriptions above focus on the general concept of the test procedures, but they do not suffice to conduct the tests. More detailed test procedures were written and

these were used for this round robin test programme. While space does not allow the inclusion of these procedures in this article, a copy of the detailed procedures is available from the authors (Stanish et al 2004).

Sample production and distribution

Ten concrete mixtures were cast at two separate locations, four mixtures at the University of Cape Town (A, B, E, F) and six at the University of the Witwatersrand (C, D, G, H, I, J). Sufficient cubes were produced for all of the laboratories to receive four samples for each test that they would conduct. The oxygen permeability test samples were reused for the sorptivity test, as permitted in the test procedures. Owing to the number of cubes required, two batches of each concrete mixture were necessary. The samples for one test were all selected from the same batch, however. A summary of the concrete mixtures is shown in table 3, including the 28-day cube compressive strengths.

It was decided to reduce variability in specimen preparation by having the two laboratories that cast the mixtures prepare all the samples for their mixtures. After a minimum of 28 days' wet curing, the samples were produced, labelled in such a way that they did not reveal their composition, and then placed in the 50 °C oven for a minimum of seven days. Upon removal, the samples were randomly assigned to the various laboratories and sealed in a Ziploc bag. The four samples from one mixture were placed in the same bag, along with some silica gel to prevent moisture gain and minimise carbonation during transport. Each bag was then sealed in a second bag. The samples were tested, so far as possible, on ten days (agreed previously) spread over five weeks. Note that in the discussion below, each of the laboratories was assigned a number that is consistent within one test procedure, but varies between tests, for example laboratory 3 for the oxygen permeability discussion is not laboratory 3 for the sorptivity discussion.

Statistical analysis procedure

The procedure outlined by ASTM C 802 (1987) to evaluate the results of the round robin test programme is briefly described here. This procedure was applied to all the results from the various tests. First, the individual test determinations for each laboratory for a specific concrete were assembled in a single table and the average value and variance were calculated for each laboratory. The ratio of the highest variance to the sum of the variances was calculated, and compared to a limiting value (the upper 5 % level), which is based on the number of replicates and the number of laboratories. These limiting values were obtained from ASTM C 802. If this limit is

Table 4 Illustration of analysis procedure

ID	Average	Components of variance		Overall variance		Standard deviation		COV	
		Within	Between	Within	Between	Within	Between	Within	Between
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
	Overall average, in order	\bar{S}_{lab}	$(3) - \frac{S_{ave}}{n}$	(3)	(3)+(4)	$\sqrt{(5)}$	$\sqrt{(6)}$	$\frac{(7)}{(2)} \times 100\%$	$\frac{(8)}{(2)} \times 100\%$

\bar{S}_{lab} is the average of the variances of the individual laboratories
 S_{ave} is the variance of the averages from the individual laboratories
n is the number of replicates (4 in this case)

Table 5 OPI variance results

Mix	Ave.	Components of variance		Variance		Standard deviation		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
G	9,13	0,0199	0,0003	0,0199	0,0202	0,141	0,142	1,5	1,6
H	9,50	0,0269	0,0107	0,0269	0,0376	0,164	0,194	1,7	2,0
E	9,92	0,0078	0,0034	0,0078	0,0112	0,088	0,106	0,9	1,1
F	10,01	0,0090	0,0028	0,0090	0,0118	0,095	0,109	0,9	1,1
I	10,06	0,0246	0,0000	0,0246	0,0246	0,157	0,157	1,6	1,6
C	10,19	0,0126	0,0072	0,0126	0,0198	0,112	0,141	1,1	1,4
A	10,27	0,0148	0,0018	0,0148	0,0166	0,122	0,129	1,2	1,3
D	10,28	0,0255	0,0043	0,0255	0,0298	0,160	0,173	1,6	1,7
B	10,29	0,0306	0,2240	0,0306	0,2546	0,175	0,505	1,7	4,9
J	10,55	0,0229	0,0090	0,0229	0,0319	0,151	0,179	1,4	1,7
Average								1,4	1,8

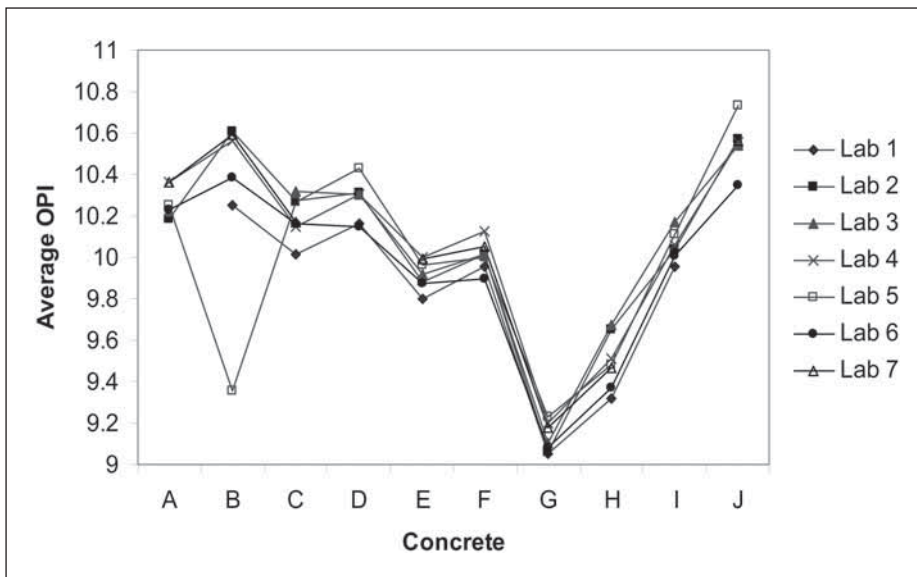


Figure 1 Average OPI values

exceeded, it can be assumed that within the set of results corresponding to the highest variance an additional, abnormal source of variance is present. For example, gross errors on the part of the laboratory could have occurred, or one of the samples could contain a flaw. To correct this, the set of data corresponding to that laboratory was examined. If one value was particularly different from the others, it was eliminated and the check redone with the remaining data of the laboratory. The number of replicates was not reduced below three, however. If there was no obvious outlier, then the data

for the entire laboratory was eliminated from the analysis of that concrete for that test. If this occurred repeatedly for a laboratory and a test, all the results from that laboratory would have been eliminated from consideration. This did not occur for any of the results in this analysis, however. To determine whether there was a laboratory with an atypically low variance, the ratio of the highest and lowest variances was determined and compared to a similar limiting value, also available in ASTM C 802. An atypically low variance would not be a problem from a testing viewpoint, but

inclusion in this evaluation may result in unrealistically high expectations. When this occurred, the data for that concrete and that test were eliminated from the analysis for that laboratory. This was done for each concrete. As a final check, the average values for each concrete were plotted for all of the laboratories to ensure that they followed a similar trend.

Once the outliers had been identified and eliminated, it was possible to calculate the overall variance, both within and between laboratories. For each concrete, four parameters were evaluated. First, the overall average was calculated as the average of the individual laboratory averages. Then what is referred to as the pooled, within-laboratory variance was determined from the average of the individual variances. Then the variance of the individual averages was calculated, and the between-laboratory component of variance was estimated from the variance of the individual averages minus the pooled within-laboratory variance divided by the number of replicates. Once this was done for each concrete, a table similar to table 4 was constructed showing the concrete ID (column 1), the average value (column 2), the within-laboratory component of variance (the pooled within-laboratory variance) (column 3) and the between-laboratory component of variance (column 4). For each lab, the within-laboratory variance was calculated as described (column 5), and the between-laboratory variance as the sum of

Table 6 Sorptivity variance results

Mix	Ave. (mm/hr)	Components of variance (mm ² /hr)		Variance (mm ² /hr)		Standard deviation (mm/ [√] hr)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
B	6,92	0,276	0,261	0,276	0,537	0,525	0,733	7,6	10,6
J	7,63	0,443	0,545	0,443	0,988	0,666	0,994	8,7	13,0
F	7,68	0,635	0,519	0,635	1,154	0,797	1,074	10,4	14,0
H	7,84	0,506	0,172	0,506	0,678	0,711	0,823	9,1	10,5
A	7,95	0,485	0,618	0,485	1,103	0,696	1,050	8,8	13,2
C	8,02	0,228	1,037	0,228	1,265	0,477	1,125	6,0	14,0
E	8,24	2,190	1,428	2,190	3,618	1,480	1,902	18,0	23,1
I	8,54	1,168	2,046	1,168	3,214	1,081	1,793	12,7	21,0
D	8,74	0,723	0,040	0,723	0,763	0,850	0,873	9,7	10,0
G	8,78	2,039	3,177	2,039	5,216	1,428	2,284	16,3	26,0
Average								10,7	15,5

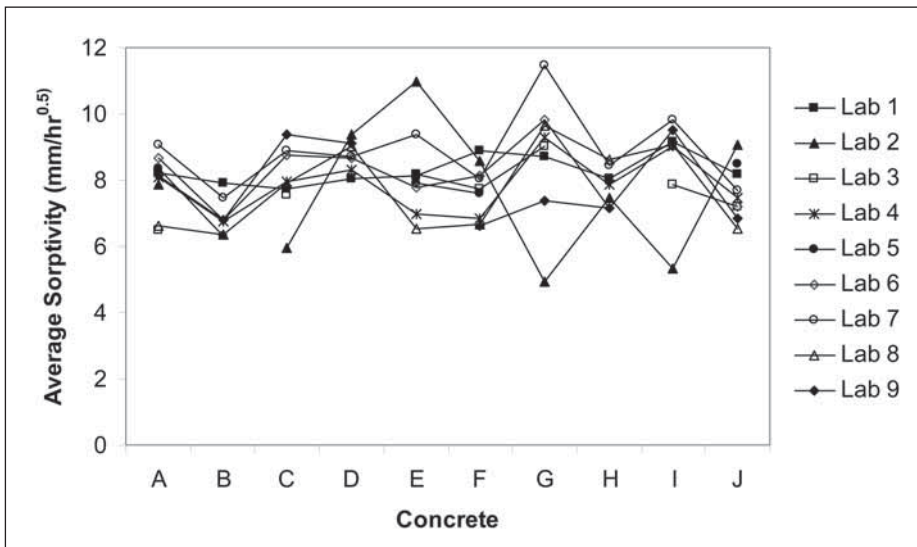


Figure 2 Average sorptivity values

the within- and between-laboratory components (column 6). The standard deviation of each type of variation was calculated as the square root of the variance (columns 7 and 8), and the coefficient of variation was calculated as the standard deviation divided by the average expressed as a percentage (columns 9 and 10). The concretes were arranged in order of increasing average value to facilitate detection of a dependence of the variance on the average value. The overall coefficient of variation was estimated as the average of the coefficients of variation for the individual concretes for that test.

RESULTS AND DISCUSSION OF INTER-LABORATORY TESTING

Oxygen permeability test

The results of seven laboratories were analysed for the oxygen permeability test. An additional two laboratories conducted the test, but their results were rejected as it was determined that they did not conduct the test according to the procedures. One of the seven laboratories was not able to test two of the ten mixtures because of internal scheduling conflicts.

The transformed oxygen permeability values (OPI values) are discussed in this paper, although the transformation desensitises the results to the underlying relationships. This process was necessary as users are more comfortable with the transformed values. In the full report, the permeability values were also analysed separately and found not to add materially to the conclusions about the test method resulting from the OPI analysis (Stanish et al 2004).

The OPI values from the individual laboratories are plotted versus concrete type in figure 1. Note that these values are the averages of the transformed individual values, rather than the usual transformation of the average values. This was done so that the plotted values agree with the statistical analysis procedure. Essentially the transformation of the individual values was considered as part of the test. With the exception of the average value of concrete B from laboratory 5, the values are in good agreement between the various laboratories.

A summary of the variance of the OPI values is presented in table 5. Following the procedures in ASTM C 802, one value for one concrete from one laboratory was

eliminated once due to high variance, and one laboratory was eliminated for one concrete due to an atypically low variance. From table 5, based on these data, a repeatability of 1,4 % and reproducibility of 1,8 % are estimated for the OPI.

A more important observation is that the coefficients of variation are very similar within laboratory and between laboratories. This is more apparent when the D'Arcy permeability values are investigated, rather than the transformed values. From this it can be surmised that most of the variability present is not due to the procedures, but to the inherent variability of the material to this property, although this has not been directly measured. This may be explained due to the sensitivity of the OPI test to compaction (Alexander et al 1999). While every effort was made to compact the cubes uniformly, it may be expected that the degree of compaction will vary more than, say, component proportions within an individual batch.

Water sorptivity test

Nine laboratories were identified that were able to perform the water sorptivity test on the samples. There were no obvious problems with performing the test correctly, so all the laboratories were included in the further evaluation. One laboratory did not complete a sufficient number of acceptable samples for four concrete mixtures, nor did a further laboratory for three concrete mixtures. The remaining results from these two laboratories were included, however. For this test, the repeatability and reproducibility were examined both for the water sorptivity value and the water-accessible porosity.

Figure 2 shows the average sorptivity results from the various laboratories for all ten concretes. It is important to note that the results are relatively insensitive to variations in the concrete strength and composition. This is consistent with previous work, which has shown that sorptivity values are more significantly affected by curing conditions before the age of seven days. For longer water-cur-

Table 7 Porosity variance results – from sorptivity test

Mix	Ave (%)	Components of variance (%)		Variance (%)		Standard deviation (%)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
F	10,2	0,334	1,588	0,334	1,922	0,58	1,39	5,7	13,6
B	10,5	0,600	2,015	0,600	2,615	0,77	1,62	7,4	15,4
E	11,4	0,562	0,574	0,562	1,136	0,75	1,07	6,6	9,3
A	11,6	0,555	1,251	0,555	1,806	0,74	1,34	6,4	11,6
C	12,0	0,440	2,810	0,440	3,250	0,66	1,80	5,5	15,0
D	12,2	0,383	0,659	0,383	1,042	0,62	1,02	5,1	8,4
I	12,7	0,585	0,341	0,585	0,926	0,76	0,96	6,0	7,6
J	12,8	0,320	0,212	0,320	0,532	0,57	0,73	4,4	5,7
G	12,9	0,731	0,051	0,731	0,782	0,85	0,88	6,6	6,9
H	13,5	0,706	0,118	0,706	0,824	0,84	0,91	6,2	6,7
Average								6,0	10,0

Table 8 . Porosity variance results – from sorptivity test Excluding laboratories 2, 4 and 8.

Mix	Ave (%)	Components of variance (%)		Variance (%)		Standard deviation (%)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
F	10,4	0,359	0,093	0,359	0,452	0,599	0,672	5,8	6,5
B	11,0	0,429	0,061	0,429	0,490	0,655	0,700	6,0	6,4
E	11,5	0,335	0,435	0,335	0,770	0,579	0,877	5,0	7,6
A	11,8	0,531	-0,093	0,531	0,438	0,729	0,662	6,2	5,6
C	12,1	0,230	0,905	0,230	1,135	0,480	1,065	4,0	8,8
D	12,4	0,331	0,218	0,331	0,549	0,575	0,741	4,6	6,0
I	12,4	0,539	-0,110	0,539	0,429	0,734	0,655	5,9	5,3
G	12,7	0,741	-0,128	0,741	0,613	0,861	0,783	6,8	6,2
J	13,2	0,383	0,232	0,383	0,615	0,619	0,784	4,8	6,1
H	13,3	0,595	0,022	0,595	0,617	0,771	0,785	5,8	5,9
Average								5,5	6,4

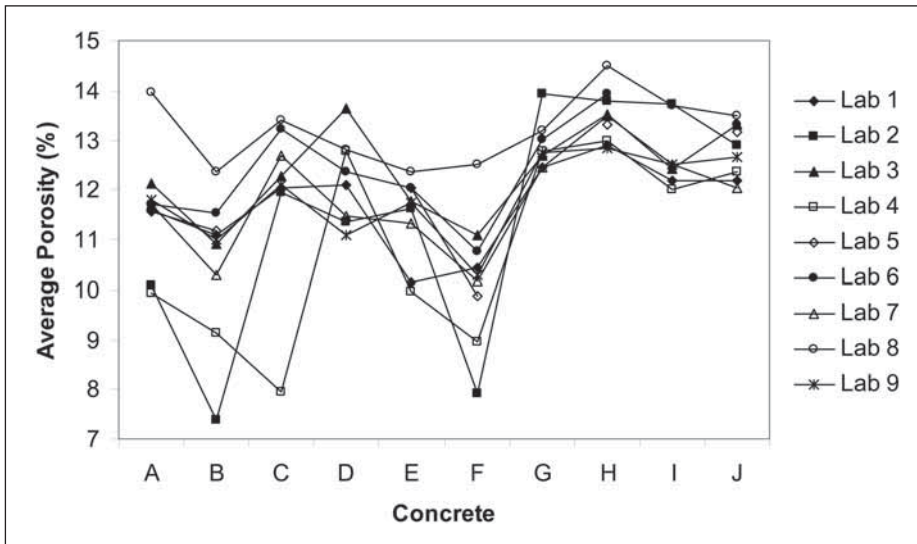


Figure 3 Average porosity values – from sorptivity test

ing periods, the results tend towards similar values. In this study, all the concretes were water-cured for 28 days and would therefore be expected to show similar sorptivity results (Ballim 1993; Alexander et al 1999). The spread of results that were obtained by the various laboratories for a given concrete mixture is of greater concern. It should be noted that there is no discernable relationship

between the laboratory and the measurement. For example, one laboratory did not consistently rate the concrete high or low compared to the other laboratories.

Table 6 presents the variances for the sorptivity test. The ASTM C 802 procedures resulted in one value of a set being eliminated twice, one set of samples being eliminated once owing to high variability and one

set eliminated once owing to atypically low variability. From this work, a coefficient of variation of 10,7 % can be estimated for the repeatability and 15,5 % for reproducibility.

The results for the water-accessible porosity determined during the vacuum-saturation procedure of the sorptivity test are also displayed. Figure 3 shows the average results from the various laboratories for the ten concretes. In general, similar trends are apparent for the results of each concrete from the various laboratories. There is a wide spread in the results between the various laboratories for one mixture, although in this case most of the spread can be attributed to laboratories 2, 4 and 8. The values for laboratory 8, which are generally high, are possibly because they experienced a problem at one point in controlling the oven temperature. This may have damaged the specimens, leading to additional micro-cracking. The porosity values determined by laboratories 2 and 4 are generally lower than those determined by the remaining laboratories. This is attributed to these laboratories not achieving the same degree of saturation as the others, probably from not achieving and maintaining the vacuum required in the procedure.

Table 9 Sorptivity variance results Excluding laboratories 2, 4 and 8

Mix	Ave (mm/hr)	Components of variance (mm ² /hr)		Variance (mm ² /hr)		Standard deviation (mm/√hr)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
B	7,06	0,221	0,333	0,221	0,554	0,470	0,744	6,7	10,5
J	7,60	0,412	0,302	0,412	0,714	0,642	0,845	8,4	11,1
H	7,76	0,339	0,237	0,339	0,576	0,582	0,759	7,5	9,8
F	7,83	0,441	0,435	0,441	0,876	0,664	0,936	8,5	12,0
A	8,16	0,617	0,618	0,617	1,235	0,785	1,111	9,6	13,6
E	8,29	2,372	-0,179	2,372	2,193	1,540	1,481	18,6	17,9
C	8,46	0,236	0,562	0,236	0,798	0,486	0,893	5,7	10,6
D	8,63	0,439	0,089	0,439	0,528	0,663	0,727	7,7	8,4
I	9,09	0,996	0,469	0,996	1,465	0,998	1,210	11,0	13,3
G	9,28	1,999	1,729	1,999	3,728	1,414	1,931	15,2	20,8
Average								9,9	12,8

Table 10 Chloride conductivity variance results

Mix	Ave (mS/cm)	Components of variance (mS ² /cm ²)		Variance (mS ² /cm ²)		Standard Deviation (mS/cm)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
F	0,20	0,0002	0,0042	0,0002	0,0044	0,0141	0,0663	7,1	33,2
E	0,42	0,0011	0,0220	0,0011	0,0231	0,0332	0,1520	7,9	36,2
H	0,59	0,0021	0,0227	0,0021	0,0248	0,0458	0,1575	7,8	26,7
B	0,94	0,0162	0,0286	0,0162	0,0448	0,1273	0,2117	13,5	22,5
G	1,16	0,0131	0,0232	0,0131	0,0363	0,1145	0,1905	9,9	16,4
A	1,21	0,0127	0,1053	0,0127	0,1180	0,1127	0,3435	9,3	28,4
J	1,33	0,0133	0,0185	0,0133	0,0318	0,1153	0,1783	8,7	13,4
D	1,62	0,0072	0,0160	0,0072	0,0232	0,0849	0,1523	5,2	9,4
I	2,24	0,0542	0,0146	0,0542	0,0688	0,2328	0,2623	10,4	11,7
C	2,56	0,0789	0,0261	0,0789	0,1050	0,2809	0,3240	11,0	12,7
Average								9,1	21,1

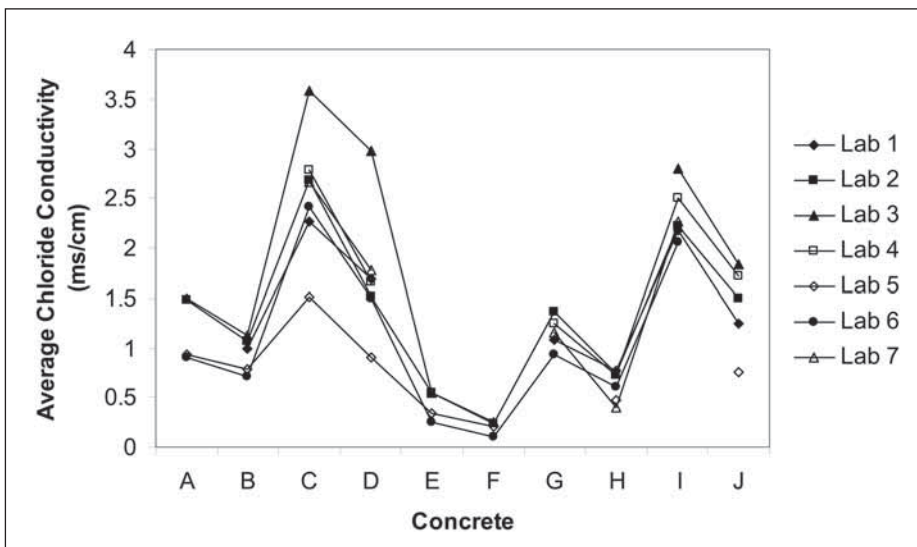


Figure 4 Average chloride conductivity values

Table 7 summarises the variance calculations for the porosity values from the sorptivity test. For the porosity measurements, none of the values needed to be eliminated. Estimates of 6,0 % for repeatability and 10,0 % for reproducibility were obtained from these data using all of the laboratories. Repeating the analysis with laboratories 2, 4 and 8 removed has little effect on the

repeatability (5,5 %), but reduces the reproducibility to 6,4 %. These calculations are summarised in table 8. The sorptivity data were also reanalysed without the data from laboratories 2, 4 and 8. The repeatability value decreased marginally (to 9,9 % from 10,7 %), but the reproducibility decreased more appreciably, to 12,8 % from 15,5 %. A summary of this analysis is included in table 9.

To determine the suitability of the procedure, it must be evaluated whether the reason for the discrepancy between the porosities determined by laboratories 2 and 4 and the remainder of the laboratories is because of a feature in the test procedure or whether it is because the test procedure was not followed correctly by these laboratories. Since the results from these laboratories were consistently low, it is believed that they were not saturating the samples completely. This would most likely be due to not maintaining the vacuum correctly during the saturation procedure. Based on this reasoning and because the remainder of the laboratories were able to achieve a reproducible result, it is unlikely that the procedures need alteration from a variability viewpoint.

Chloride conductivity test

Seven laboratories participated in the chloride conductivity test. A problem arose because of the marginally different dimensions of the core barrels used by the two laboratories that were preparing the samples. The University of the Witwatersrand used a core barrel that gave samples that were $68 \pm 0,5$ mm. This was the size of barrels that was in use when the test apparatus

Table 11 Porosity variance results – from chloride conductivity test

Mix	Ave. (%)	Components of variance (%)		Variance (%)		Standard deviation (%)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
F	9,0	0,237	1,044	0,237	1,281	0,487	1,132	5,4	12,6
B	9,7	0,556	1,731	0,556	2,287	0,746	1,512	7,7	15,6
E	10,1	0,825	1,277	0,825	2,102	0,908	1,450	9,0	14,4
A	10,5	0,494	0,173	0,494	0,667	0,703	0,817	6,7	7,8
D	11,0	0,341	0,516	0,341	0,857	0,584	0,926	5,3	8,4
C	11,8	0,582	0,474	0,582	1,056	0,763	1,028	6,5	8,7
J	11,8	0,358	0,096	0,358	0,454	0,598	0,674	5,1	5,7
I	12,2	0,269	0,254	0,269	0,523	0,519	0,723	4,3	5,9
G	12,3	0,408	0,122	0,408	0,530	0,639	0,728	5,2	5,9
H	12,4	0,599	0,700	0,599	1,299	0,774	1,140	6,2	9,2
Average								6,1	9,4

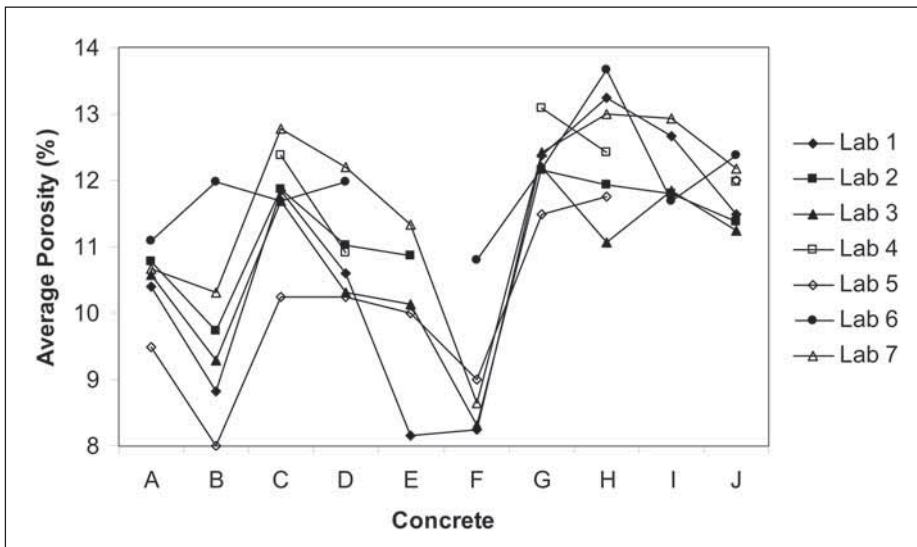


Figure 5 Average porosity values from chloride conductivity

was first designed. The University of Cape Town, however, used a core barrel that produces samples that were $70 \pm 0,5$ mm in diameter. The change arose because the core barrel supplier had changed the source for the steel tubes. It is believed that most barrels purchased in future in South Africa will be of this size unless a special order is made, with corresponding cost implications. Both these barrels, however, do produce cores that are within the tolerance as specified in the current version of the procedures (68 ± 2 mm). While this tolerance is a reasonable limitation from a production viewpoint, it is not stringent enough for the test apparatus. For laboratories using a smaller core barrel, the larger samples could not be placed into their cells. It is also believed that for some laboratories the smaller samples could not be sealed properly. The conclusion was that the tolerances on the chloride conductivity apparatus as currently designed are too narrow and the test rig needs to be redesigned. This is particularly true of the diameter of the test specimens, but applies to a lesser extent to their thickness.

Figure 4 provides the chloride conductivity results from the seven laboratories as

a function of concrete type. The concrete type affected the chloride conductivity results for all the laboratories in a similar manner, although there was some scatter in the results, particularly for concretes C and D. Laboratory 3 reported results for these concretes that were too high compared to the other samples, and it also gave the highest results for concretes I and J. The results from laboratory 3 are not given for concretes G and H as they were eliminated owing to their high variance, and they were again on average higher than the remaining samples. For laboratory 3, the results for concretes A, B, E and F correspond with the other results, however. This corresponds to high results for the 68-mm diameter samples and typical results for the 70-mm diameter samples. It therefore seems likely that sample size issues are the cause of the high variability and for this laboratory, concretes C, D, G, H, I and J were not included in the chloride conductivity analysis, although they were included for the porosity measurements. For laboratory 5, concretes C, D, I and J gave particularly low results. (The results for concrete I are not shown as they were eliminated owing to high variance.)

It is considered likely that some error was made during testing, although it is not obvious what this is. Incomplete saturation would result in a lower conductivity value and may be the cause. Thus the results from this laboratory have been discarded for those concretes.

Table 10 contains the variance results for the chloride conductivity test. To produce these data, nine additional sets of data had to be removed as the variance was typically high. From this evaluation, a repeatability of 9,1 % and a reproducibility of 21,1 % were estimated.

The results indicate that, if done correctly, it is possible to achieve relatively good repeatability, although the reproducibility was still high. The high number of results that qualified for elimination in order to achieve these results is of concern, however. This indicates that the required degree of control is not always being achieved. Part of this variability may be explained by the tight tolerances on the test rig, as discussed above. Improper sealing of the samples would result in high values, either for the average or for some samples in a set. Low values may be because of incomplete saturation or improper measurement. This seems to indicate that, although the test procedures may be adequate, they are not necessarily being performed correctly. This may be due to a mismatch between what is required for the procedure and the limitations of the test equipment, unclear procedures, improperly trained technicians or the influence of an unconsidered, and uncontrolled, test variable.

Figure 5 shows the porosity values from the chloride conductivity test for the ten concretes and the seven laboratories. Similar to the porosity determined from the sorptivity samples, there is a wide spread in values. Unlike the porosity data from the sorptivity samples, however, the outliers are not obvious. Laboratory 5 does appear to be low, however, consistent with incomplete saturation, which would result in low values for

Table 12 Porosity variance results – from chloride conductivity test (excluding laboratory 5)

Mix	Ave. (%)	Components of variance (%) ²		Variance (%) ²		Standard deviation (%)		Coefficient of variation (%)	
		Within	Between	Within	Between	Within	Between	Within	Between
F	9,0	0,130	1,439	0,130	1,569	0,361	1,253	4,0	13,9
B	10,0	0,534	1,350	0,534	1,884	0,731	1,373	7,3	13,7
E	10,1	0,531	1,842	0,531	2,373	0,729	1,540	7,2	15,3
A	10,7	0,526	-0,064	0,526	0,462	0,725	0,680	6,8	6,4
D	11,2	0,245	0,516	0,245	0,761	0,495	0,872	4,4	7,8
J	11,8	0,307	0,138	0,307	0,445	0,554	0,667	4,7	5,7
C	12,0	0,526	0,061	0,526	0,587	0,725	0,766	6,0	6,4
I	12,2	0,269	0,254	0,269	0,523	0,519	0,723	4,3	5,9
G	12,4	0,310	0,051	0,310	0,361	0,557	0,601	4,5	4,8
H	12,6	0,546	0,772	0,546	1,318	0,739	1,148	5,9	9,1
Average								5,5	8,9

Table 13 Test repeatability and reproducibility

	OPI	Sorptivity	Sorptivity – porosity	Chloride conductivity	CC – porosity
Repeatability	1,4 %	9,9 %	5,5 %	9,1 %	5,5 %
Reproducibility	1,8 %	12,8 %	6,4 %	21,1 %	8,9 %

chloride conductivity. The porosity data in the sorptivity test from this laboratory were also eliminated. In addition, the results for laboratory 6 may be high, particularly for concretes B and F.

Table 11 shows the variance information for the porosity data from the chloride conductivity tests, including all of the laboratories. To obtain this, two sets of data were eliminated owing to atypically low variance. A coefficient of variation for repeatability of 6,1 % and for reproducibility of 9,4 % can be estimated from these data. In addition, the data were also analysed excluding laboratory 5, shown in table 12. This reduced the variance values, but not as much as the similar process affected the variance of the sorptivity tests.

CONCLUSIONS

The repeatability and reproducibility estimated from the acceptable data for all three tests are shown in table 13. For the oxygen permeability test, the similarity of the within and between laboratory values indicates that most of the variability is likely attributable to material variability. The water sorptivity test did not present any difficulties in terms of variability, although it is suspected that some laboratories did not maintain a full vacuum during the saturation procedure. Certain laboratories had difficulty with the chloride conductivity test due to variability in the sample diameters and the tight tolerances of the testing apparatus.

In addition a large number of laboratories had to be eliminated from the analysis due to their high variances. This indicates that some laboratories had difficulty in conducting the chloride conductivity test, either because the procedures are unclear or because they do not have the experience to perform the tests correctly. The chloride conductivity test apparatus should be redesigned to increase the tolerances.

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