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The Oxygen Permeability Index Test: Its Application and Addressing the Variability Issues

GW Nganga and SM Gouws

ABSTRACT: The paper provides an overview of the Oxygen Permeability Index (OPI) test, a durability index (DI) test developed in South Africa. Firstly, a brief review is given on the application of the test and its precision. Secondly, the recent improvements in the test aimed at addressing the variability issues which include the use of ceramic discs for calibration of the apparatus and a laboratory audit are also described. From the audit exercise, the OPI test was observed to be robust. The final section of the paper presents the application of the OPI test on site and in performance-based specifications used in a large scale South African road project. In this project specifications of a limit value of OPI and cover depth were given. From the site-based test results obtained it is observed that the variability is slightly above that obtained from the inter-laboratory exercise. The OPI test is therefore a valid test to apply in the control of concrete cover quality.

INTRODUCTION

Recently there have been increasing concerns on the extensive deterioration of reinforced concrete (RC) structures due to corrosion. This occurs due to the penetration of aggressive agents, CO₂, chlorides, oxygen and moisture, that initiate and propagate the corrosion process (Bentur et al., 1997). The penetrability of the concrete cover influences the transport mechanisms of these aggressive agents by diffusion, permeation or sorptivity. To control and reduce the extent of corrosion the penetrability properties of concrete cover should be determined. The penetrability property that will be considered in this paper is air permeation.

Different test methods have been developed over the years to determine permeability properties of RC structures on site e.g. inter alia the Torrent air permeability test (Torrent, 1992), and the autoclam permeability test (Basheer et al., 1994). In addition, there are laboratory-based tests e.g. the Cembureau test (Kollek, 1989) and the South African oxygen permeability index (OPI) test developed by Ballim (1991). The OPI test is part of a suite of three penetrability tests developed in South Africa, which also include the water sorptivity and chloride conductivity (Alexander and Mackechnie, 2001).

This paper focuses on implementation of the South African -OPI test. A brief review is given on application of the test and studies undertaken to determine its precision. Review of a recent laboratory audit conducted in 2011 and observations made is also provided. The final section presents the implementation of the OPI test on site and in performance-based specifications illustrating its practicality in the control of concrete cover quality.

THE OXYGEN PERMEABILITY INDEX (OPI) TEST

The OPI test measures pressure decay of oxygen passed through a standard specimen placed (70 mm dia. \times 30 mm disc) in a falling head permeameter from which the Darcy coefficient of permeability, k, is determined (DI test manual, 2009). Details on the OPI test procedures are provided in this manual which comprises four parts, of which Part 1

(Standard procedure for preparation of test specimens) and Part 2 (Standard procedure for oxygen permeability test) are relevant to this paper. The OPI value is the negative logarithm of the average coefficient of permeability from four test determinations. The test has been applied in lab-based studies to characterize concrete mixes and evaluate effects of concrete grade, curing method and type of binder used (Mackechnie, 1996).

The OPI test is used in service life design where it has been applied in an empirical service life prediction model to determine the carbonation depth for a given OPI value (Mackechnie and Alexander, 2002; Alexander et al., 2008). This is illustrated in Figure 1 where for a given OPI value (9.70 in this case), inland exposure conditions with average relative humidity of 60% and 100% CEM I binder, the carbonation front (depth) will have penetrated to slightly less than 30 mm over a period of 100 years. The OPI value is thus used to determine the cover depth required to attain a given service life.

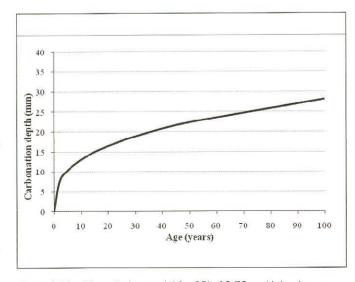


Figure 1: The DI prediction model for OPI of 9.70 and inland exposure conditions 60% humidity (Mackechnie and Alexander, 2002).

RECORDING AND COMPUTATIONS

In the laboratory, data recorded and used for computations in the OPI test include: test specimen dimensions of diameter and thickness; volume of the permeability cell (should be determined at regular intervals e.g. once in a year). On assembly of a specimen in the test apparatus pressure readings are recorded by a pressure transducer at a frequency usually of two minutes. A record of pressure change is obtained for a period of up to six hours or until pressure drops to 50kPa, whichever happens first. A minimum of eight readings is required. With the readings obtained, a plot of (In (P₂/P₂)) against time is done; P refers to initial pressure, P refers to pressure at time t. A best fit line is then made through the data points. The coefficient of correlation of this line (r2) should be equal to or greater than 0.99. On complying with the required r² value, the Darcy coefficient of permeability is computed. Formula used in this computation can be obtained from the Durability Index test manual (2010). The OPI value is based on the negative logarithm of four k-values.

A standard Excel spreadsheet has been developed at the University of Cape Town to carry out the computations mentioned above. The record of pressure changes are transferred to the Excel spreadsheet using software that is linked to the transducer through a network connection. The Excel spreadsheet also provides internal checks of variability through computation of the $\rm r^2$ value and a variability check. The variability check is based on comparing the maximum acceptable range in results with a value obtained from considering the product of repeatability values with a given multiplier that is based on the number of test determinations (ASTM C 670-03, 2007). The repeatability values used in this check are obtained from test results in the inter-laboratory exercise reported in Stanish et al. (2004), described in the subsequent section.

PRECISION OF THE OPI TEST

In the development of a test method it is important to obtain its precision which is defined as a measure of the magnitude of variability expected between test results when undertaken in one or more competent laboratories (ASTM E 177-06b). Precision is evaluated through an inter-laboratory study (round robin test) where repeatability and reproducibility are determined (ASTM C 670-03). Repeatability is the variability of individual test results when test are carried out on same material by same operator with the same apparatus. Reproducibility is the variability of results carried out in different laboratories using materials that are as nearly identical as possible.

An early inter-laboratory exercise reported in Grieve et al. (2003) was performed due to concerns raised on differences in results on the same test between laboratories, and difficulties in achieving required test values under site conditions. This exercise was carried out in seven laboratories using two concrete mixes subjected to two different curing methods. The repeatability of the OPI test ranged from 0.43 to 2.84% while reproducibility ranged from 0.48 to 2.80% (based on OPI values). From the exercise it was recommended that the test procedures be re-written to provide more detail to reduce the possibility of different interpretation.

The test methods were revised, to make them simpler and improve clarity, by a working group that involved representatives

from both industry and researchers. The revised test methods were published in 2004 and a training workshop was held. The laboratories were then given time to familiarize themselves with the test method before another inter-laboratory exercise was conducted (Stanish et al., 2004). This involved a total of nine laboratories which conducted the three penetrability tests mentioned earlier, on ten different concrete mixes. The repeatability and reproducibility for the OPI test from the exercise are summarized in Table 1.

Table 1: Repeatability and reproducibility of the OPI test (Stanish et al., 2004)

	OPI	k-value
Repeatability (%)	1.4	32.2
Reproducibility (%)	1,8	36.6

From the inter-laboratory exercise, it is observed that variability of the OPI test is low. The repeatability of the kvalue is however much higher at 32.2%. The OPI value is a transformed logarithmic value and not the natural numerical measure of permeability, thus the lower variability. From a comparative study on permeability tests reported in RILEM TC 116-PCD (1999) it was observed that variability of permeability tests with natural numbers was in the range of 30%. Thus, the SA OPI test has variability similar to other international results. The seemingly high variability based on the natural numbers is characteristic of this type of test for penetrability parameters, where small flaws and variations in the test specimens give rise to larger variability than might be expected in, say, a bulk test for compressive strength. Thus, the variability in permeability values can be attributed to sensitivity of the test to variations in material properties, which is a desirable property for such a test.

In addition to seeking the standardization of the durability Index test methods, the working group aims at ensuring that laboratories that carry out the tests are accredited. For a laboratory to be accredited they will have to satisfy a SANAS auditor that they carry out the test exactly as stipulated in the test method (Gouws, 2010). The accreditation will ensure that data obtained from laboratories are of good quality and eliminate incidences of missing information as currently experienced e.g. missing data on samples that can only be obtained from site such as concrete mix used, curing methods.

CHECKS ON THE OPI APPARATUS

To reduce the variability in application of the OPI test, ceramic discs were developed for use as calibration standards. The ceramic discs should be carefully handled at all times; their surface should not be touched with greasy hands nor should any dirt accumulate on it. The discs were tested four times by a commercial laboratory (Contest Lab), each time in a different permeameter. The standard deviation and coefficient of variation are based on coefficient of permeability values. The variability observed was low, as shown in Table 2. These results gave confidence in the fact that the OPI test could produce results with acceptably low variability, and that variability measured in concrete test specimens therefore represented actual material variability, not test variability.



Table 2: Test results from ceramic discs used for OPI calibration (Contest Lab results)

	(Cylinder Number					
Ceramic Disc	1	2	3	4	Average	s¹	CoV ² (%)
A1	9.14	9.14	9.15	9.16	9.149	0.1305	1.84
A2	9.02	9.04	9.02	9.03	9.026	0.1663	1.77
А3	9.01	9.04	9.01	9.01	9.018	0.3186	3.32
A4	9.17	9.15	9.14	9.16	9.154	0.1858	2.65

1 s:

Standard deviation

² CoV: Coefficient of variation

REVIEW OF LABORATORY AUDIT EXERCISE

To ensure test results obtained from a laboratory are reliable, it is essential to ensure good practice which requires: clear and complete test procedures that can be correctly followed, knowledge and skill of operators in undertaking the tests, maintenance of apparatus and required test environmental conditions, proper documentation of results and calibration of test apparatus (Zimmerman, 2010; ASTM E-177).

A submission of the durability index test methods had previously been made (in 2008) to the South African Bureau of Standards (SABS) for publication. The submission was however regarded as premature, as no follow-up inter-laboratory exercise from that reported in Stanish et al. (2004) had been done to evaluate if the variability in the tests had reduced (Gouws, 2011). Despite the variability issues, the South African National Roads Agency Limited (SANRAL) implemented DI-based performance specifications in the Gauteng Freeway Improvement Project (GFIP). From site-based test results high variability was observed. To determine the reasons for the high variability the services of an accredited SANAS auditor were utilized and certain parameters that require tight specification and control were identified e.g. degree of chipping or damage allowable in discs.

To address variability issues in the test, an industry-led initiative instituted an audit exercise on the GFIP laboratories. This was done instead of a statistical validation process (determining test precision) which is an expensive undertaking. The aims of the audit were to: (a) Identify variations in test equipment and test procedure application. (b) Identify ambiguities and difficulties encountered in use of the test procedures (Raath, 2011). (c) Evaluate the experience of laboratories in application of the tests to determine best practices that can be employed in improvement of the test method (Gouws, 2011).

The audit was carried out in a total of 15 participating laboratories between February and May 2011 with observations made on Part 1 (procedure for test specimen preparation) and Part 2 (procedure for the OPI test). The audit was conducted in three main stages: (i) a seven day notification to a laboratory to condition test specimen, in this case a ceramic disc, for a period of 7 days in the oven at 50° C. (ii) Observations by the auditor on equipment used in specimen preparation and testing, and on execution of test, specifically if it was done in accordance with test procedures. (iii) Completing a checklist of questions (audit list) based on the DI test manual which required 'yes' or 'no' responses from the test operator.

Observations on preparation of test specimen

Part 1 of the test manual provides procedures for specimen preparation which are summarized below: Test specimens of diameter 70 ± 2 mm and thickness of 30 ± 2 mm are obtained from cubes or site elements at 28 to 35 days after casting. The cubes or test panels should be cored in a perpendicular direction to that of casting. A holding device that firmly holds the test sample in place is required during coring. When cores are obtained from site elements they should be properly packaged during transport to protect from adverse drying conditions and from damage due to rough handling. Specimens obtained from site should be kept at ambient conditions for a maximum period of 3 days.

The observations made in the audit with regard to Part 1 of the test procedure were:-

- a) Variations in measurement of test specimen diameter which was not always done at the widest point while the thickness was not measured on four equidistant locations on the circumference.
- b) Differences in test specimen diameter due to size of the core barrel used i.e. some laboratories used a core barrel with a diameter of 68 ± 2 mm as provided in previous test procedures while others used a 70 ± 2 mm core barrel.
- c) The holding devices used did not always provide a firm grip on the sample cored which would affect the attainment of parallel sides.
- d) Coring of test panels was not always done in a perpendicular direction to that of casting.
- e) The use of a smooth diamond saw blade to cut test specimen resulted in well-trimmed discs with less chipped edges in comparison to use of a notched blade.
- f) Discs that were damaged during coring and cutting were seldom discarded as there is no guideline with regard to extent of damage that is permissible, and which would result in discarding of a specimen.
- yariations in dimensions of test panels e.g. thickness from one end to another. The panels were observed in some cases to be of low quality due to poor handling on site.
- h) The coring to obtain test samples was generally undertaken in laboratories from test panels delivered. However, there is no procedure for transport of panels to labs i.e. when to collect panels, should the panels be delivered by site staff or collected by laboratory staff? The lack of clear procedures on transport of test panels may result in delays in coring and testing and reduce the effective application of the test in quality control.
- Lack of proper identification of test panels e.g. casting date, structural elements represented by panels, geographical locations from which they were obtained.

Observations on standard procedure for OPI test

The apparatuses used in carrying out the test include: oven for pre-conditioning of test specimens; permeability cell with a volume of 5L \pm 5%; compressible rubber collars; desiccators. The environmental conditions in the laboratory should



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be maintained at $23 \pm 2\,^{\circ}$ C and $\leq 60\%$ humidity. A test specimen is fitted into a rubber collar which is inserted into a rigid sleeve and placed on top of the test chamber. A cover plate is then placed on top of this assembly and centred, and the top screw tightened.

The observations made for Part 2 of the test procedure were:-

- a) The volume of the permeability cells had not been determined. Laboratories were advised to do this regularly by filling the cells with water and measuring the mass.
- b) The permeability cells were regularly checked for leaks using an impermeable (plastic) disc.
- c) The laboratory conditions, temperature and humidity, were well recorded and controlled with the exception of site-based labs.
- d) The preconditioning of samples by oven drying for 7 days \pm 4 hours was done in ovens that were observed to be standard with adequate temperature measurements. The relevance of the \pm 4 hours was questioned. One laboratory deviated from the required test procedure by measuring the mass of discs during the oven drying until a constant mass was obtained, before testing the specimen.
- e) Some laboratories left the test specimen in the oven for a longer period than that specified due to limited equipment to undertake the OPI test. The storage of test specimens in the oven until the time of testing is however preferable to exposing them to the laboratory environmental conditions.
- f) Variations were observed in test apparatus e.g. one laboratory used a plastic rigid sleeve that was shorter than the rubber collar which would result in a different degree of restraint in comparison to one made from steel.
- g) The insertion of test specimen into the rubber collar was extremely difficult, in some cases. To resolve this a few laboratories applied grease to the interface, an aspect that they were advised against doing.
- h) The hardness of the rubber collars varied which would result in different restraint under applied loads.
- i) Variations in tightening of the top screw, where some labs did it by hand while others used a spanner. This variation would result in differences in the magnitude of applied load.
- j) The record of pressure change was done at 15 minute intervals in one laboratory while another laboratory recorded pressure change at 30-second intervals. The first case would result in few readings that may be insufficient for computations, while the latter would result in too many measurements.
- k) Missing information was observed from previous records on OPI tests undertaken in laboratories e.g. binder type and amount used, water/binder ratios, curing history. This information is important for test specimen description and identification and can only be reliably obtained from site. Some laboratories made attempts to obtain this information which was however unfruitful due to poor cooperation by site staff.

Table 3: Comparison of lab audit and calibration values for OPI

Ceramic Disc ID	Lab value	Calibration value	Percentage difference
A1	9,12	9.04	0.88
D2	9.19	9.15	0.44
E1	9.01	9.01	0.00
A4	9.11	9.15	-0.44
E2	8.95	9.04	-1.00
B4	8.94	9.08	-1.54
C1	9.07	9.11	-0.44
P4	9.02	9.02	0.00
C2	9.19	9.11	0.88
C4	9.24	9.11	1.43
P2	9.17	9.21	-0.43
Mean percentage difference	-0.02		

The test results obtained for the ceramic discs used are presented in Table 3. It is observed that the differences in the laboratory audit and calibration values are low. This indicates that despite variations in apparatus and test execution, the degree of variability is low; thus valid test results are obtainable and the OPI test is robust.

APPLICATION OF THE OPI TEST: SITE-BASED STUDIES

An early study on the application of durability index tests on site is reported in Gouws et al. (2001). One of the objectives was to determine the practicality of DI tests on site to obtain valid results. The validity of results was assessed by comparing test results of site elements with those of laboratory samples, prepared with the same concrete mix. The samples used for testing were obtained from six locations using site prepared concrete. It was observed that fully wet cured samples generally yielded better DI test values than site-cured samples, as expected, but in some cases site-based DI values were better, for example for surface-finished slabs. This indicates that with proper placing, compaction and curing, test results better than those of laboratory wetcured samples can be obtained.

The durability index tests have been further developed for use in performance-based specifications (Alexander et al., 2008). These specifications provide a limit value for OPI or chloride conductivity index (CCI), depending on environmental exposure conditions, as well as cover depth, for a given service life. As mentioned earlier, the DI-based performance specifications were implemented by the South African National Roads Agency Limited (SANRAL) in the large scale Gauteng Freeway Improvement Project (GFIP) (2008 – 2011).

A study to evaluate the effects of these specifications on the quality of construction methods is reported in Knecht (2009). The study was based on evaluating construction methods used by three precast median barrier manufactures. It was observed that the manufacturers improved on construction methods used i.e. compaction and curing to ensure they complied with requirements stipulated in the specifications. The OPI results obtained from the study complied with the limit value.



Table 4: Numerical summary of OPI values (n - sample size)

Project ID	ń		OPI (log scale)*
		Mean	s	CoV (%)
1	172	9.75	0.28	2.84
2	94	9.91	0.22	2.24
4	116	9.87	0.23	2.33
6	91	10.06	0.46	4.60
9	132	10.25	0.18	1.75

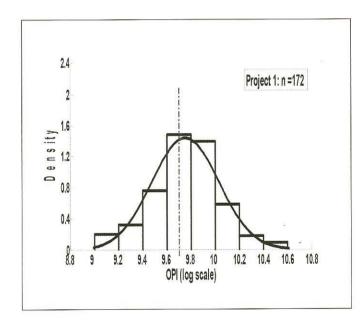
A further study to evaluate practicality of the DI-based performance specifications is reported in Nganga (2011). This was based on case studies obtained from the GFIP where data were obtained from five sub-projects and analysed. The numerical summaries of OPI values from these sub-projects are given in Table 4 while those of the k-values are presented in Table 5. From the values in Table 4, the limit OPI, on the basis of the mean value, was exceeded in the sub-projects considered. The variability (CoV) was also low when compared

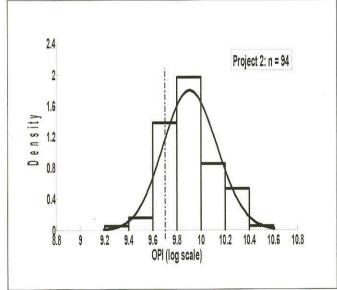
Table 5: Numerical summary of k-values

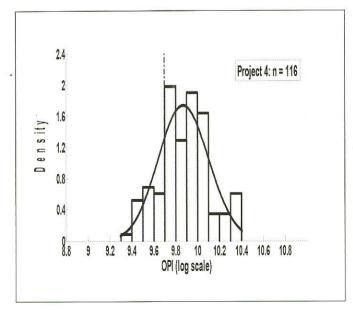
		k-values (m/s)			
Project ID	n	Mean (×10-10)	S	CoV (%)	
1	159	1.72	0.88	51	
2	92	1.37	0.63	46	
4	112	1.36	0.63	46	
6	86	1,27	1.08	85	
9	133	0.695	0.37	53	

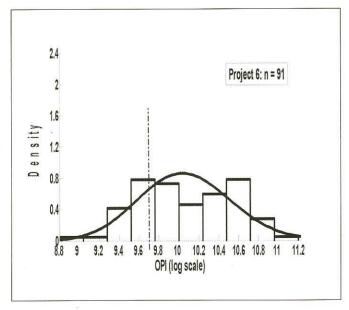
to the reproducibility values in Table 1, with the exception of that from Project 6. A graphical representation of variability in OPI values is illustrated in Figure 2.

Figure 2 shows that the spread in values is lowest for Project 9 ranging from 9.8 to 10.7, while Project 6 has the highest range in values from 8.8 to 11.2. The dotted line in the figure indicates the limit value of 9.70 provided in the specification. The area under the curve and to the left of this line indicates the proportion of values that fail to comply with the limit value.









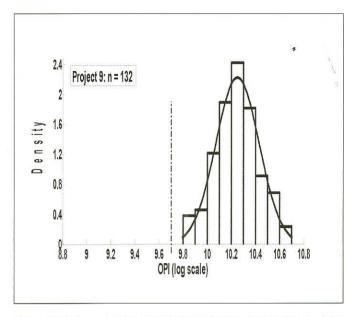


Figure 2: Histogram plots of OPI values for five projects in the GFIP (dashed line indicates limit value of 9.70)

This proportion is highest for Project 1 and lowest for Project 9 where all values comply with the limit value.

The limit k-value, obtained from transforming the logarithm value of 9.70, is $2.0\times10\text{-}10\,\text{m/s}$. The mean values from the Projects considered all comply with this value, as indicated in Table 5. The variability in k-values is relatively high, with that from Project 6 being exceptionally high at 85%.

The higher variability in the OPI and k-values observed, when compared to that obtained for the reproducibility reported in Stanish et al. (2004) would be expected as tests

were undertaken on site-based samples where the degree of control in their production is lower than that exercised in laboratories

INTERPRETATION OF OPI VALUES IN RELATION TO COVER DEPTH REQUIREMENTS

From the OPI test values obtained from the GFIP, it is observed that variation exists among the projects with that of Project 6 being the highest at 4.60% while that of Project 9 is the lowest at 1.75%. An illustration of the practical effect of differences in OPI values on carbonation depth penetration over a period of 100 years, which has relevance in determining cover depth, is given in Figure 3.

For Project 1 with an OPI value of 9.75, the carbonation depth will have penetrated to slightly less than 40 mm, which complies with the minimum cover depth provided. For Project 9 with an OPI value of 10.25 the carbonation depth will have penetrated to a depth slightly more than 10 mm over a period of 100 years. The difference in carbonation depth for the two OPI values provided illustrates the sensitivity of the test; for a difference between 9.75 and 10.25 the penetration of carbonation depth over a period of 100 years reduces by approximately 20 mm.

Therefore, an increase in the OPI value would result in a decrease in advancement of the carbonation depth in the concrete cover. The practical consequence of this is that a lower cover depth than that provided in the specifications can be permitted where higher OPI values are obtained. However, the cover depth should not be lower than some allowable minimum value provided in the specifications, which in the case of the GFIP was 30 mm (SANRAL, 2010).

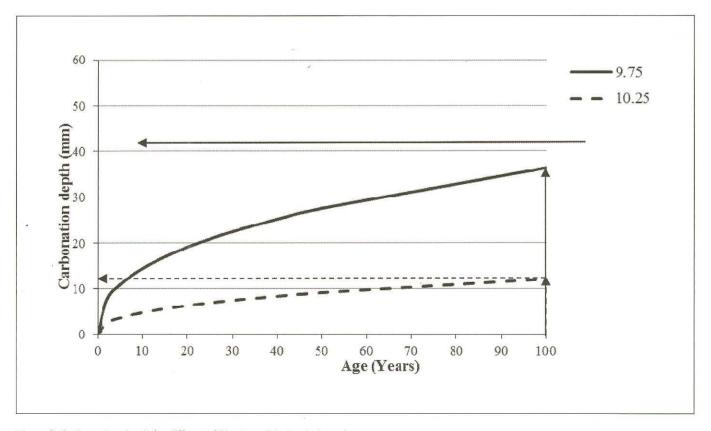


Figure 3: Carbonation depth for different OPI values (Mackechnie and Alexander, 2002).



CONCLUSIONS AND RECOMMENDATIONS

A review of the oxygen permeability index test, its application on site and measures undertaken to address variability is provided in the paper. The OPI test is applicable both in the laboratory for characterization of concrete mixes, and on site in control of quality of the concrete cover. The test precision has been determined and was found to be in an acceptable range when compared to other permeability tests in an international context. From the review of an audit report, it was observed that the test is robust and valid test results with low variability can be obtained, despite variations in apparatus and execution of the test. The test has also been applied on site in the large scale GFIP, where the variability determined from site-based data was slightly higher than that obtained for test reproducibility, which would be expected due to difference in control for site- and laboratory-based samples. The OPI test is therefore practical for application on site in the quality control of concrete cover.

Recommendations for improvements in the OPI test to address and further reduce the variability issues currently experienced include:

- a) Development of ceramic discs that are denser and from which higher OPI values, above the current values of 9.01 to 9.21, can be measured e.g. values of 9.50 10.00. The purpose of using denser ceramic discs would be to evaluate if the low variability currently observed would still be present for higher OPI values.
- b) Modification of Excel spread-sheets so as to capture site-based data which was found to be predominantly missing for the results obtained from the GFIP. A record should be made on site of geographical location, element that the test panel used represents, curing methods and binder type used. The samples from site should only be accepted for testing on supply of this site information.
- c) Further training and increased awareness of the relevance of the test method should be created among the laboratory operators and engineers on site. From the audit exercise, it was observed that poor communication existed between the two parties which would be amended if the relevance of the method in ensuring durability of structures is established.

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