Preconditioning in situ concrete for permeation testing Part 1: Initial surface absorption

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Preconditioning in situ concrete for permeation testing
Part 1: Initial surface absorption

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A new method for testing concrete using the initial surface absorption test (ISAT) has been developed for site use. It is based on applying a vacuum to an ISAT cap placed on the concrete surface until drying is achieved. The progress of the drying is monitored by placing indicating silica gel desiccant in the ISAT cap and observing the colour change. The method is quick, simple and practical for in situ applications. Results of comparisons with the existing in situ method in BS 1881 show that the new method is potentially more capable of producing reliable and reproducible measurements, and therefore will allow better comparison of in situ and laboratory-obtained data.

Introduction

The measurement of the rate of ingress of water into hardened concrete is a key step in the determination of the potential durability of a structure.[1] Water is a necessary ingredient for the corrosion of embedded steel and freeze–thaw damage to concrete. Water ingress rates are also good predictors of the likelihood of the ingress of other detrimental fluids and ions into concrete.

Several in situ tests which are intended to measure the permeation characteristics of concrete in structures have been developed and investigated.[2–4] The permeation measurements from them have been shown to provide durability indices which correlate with the results from accelerated exposure testing.[5] However, the major difficulty in applying these tests in situ is that their measurements are substantially affected by the amount of water already present in the concrete, and it has been shown that any uncertainties about the original moisture content lead to poor reproducibility of the results.[4] For this reason meaningful in situ testing of concrete for permeation properties has not been possible. There are two possible approaches to overcoming this problem. The first is to measure the moisture content and compensate for it in the results, and the second is to precondition the sample by removing the moisture.

Measurement of the moisture content of in situ concrete may be achieved by measuring the electrical impedance or attenuation radiation[6] or by establishing hygral equilibrium between the in situ concrete and a specimen of which the moisture content may be established by weighing.[7] When the relative humidity is below 90%, some indication of the moisture content may be obtained by using a humidity probe in a drilled hole,[8] although difficulties may be found in calibration. After establishing the moisture content, an extensive system of correction factors taking into account the concrete grade, age, cement replacement materials, etc., would be used to calculate standard base values. The results could then be inaccurate, owing to errors arising from the test measurement itself, the measurement of moisture content or the correction factors.

This Paper reports the development of a method which works by removing the moisture, and it may be applied in situ. Results are presented from the ISAT method in which the volume of water entering an area of concrete surface under a 200 mm hydraulic head. The Authors believe that the concept may also be used with some other in situ tests.

Removal of moisture from in situ concrete

Water will remain in concrete pores with entry diameters which are sufficiently small to sustain a meniscus. The conditions for this are given by the Kelvin equation

$$r \ln (\mathrm{RH}) = \frac{2ms}{dRT}$$

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where r is the pore radius, RH is the relative humidity, m is the molecular weight of water = 0·018 kg, s is the surface tension of water = 0·073 N/m, d is the density of water = 1000 kg/m^3, R is the gas constant = 8·3 J/mol/K, and T is the temperature = 290 K.

Applying this equation indicates that at a typical external humidity of 70% no pores with radii greater than 3 nm will sustain a meniscus. The volume of pores in concrete with radii less than 3 nm is very small, so if concrete is in equilibrium with air at normal humidities in the UK it will be virtually dry. The practical difficulty for the purpose of preparing samples for permeation testing is the time taken to reach equilibrium. This will depend on the permeation properties of the concrete and the air flow at the surface.

The two possibilities for fast removal of water are raising the temperature and lowering the pressure. Raising the temperature of any part of an in situ sample, except for the immediate surface layer, would be difficult without a risk of damage from overheating. Lowering the pressure is, however, an attractive option. The maximum stress which may be applied in this way is one atmosphere, which is 0·1 N/mm^2, and this is likely to affect a typical concrete.

Vacuum drying technique

The essential requirement for sample preconditioning is that it would be simple to replicate in order to give reproducible in situ ISAT results. The time required to extract the moisture with a vacuum will depend on the pressure and the initial moisture content, as well as the permeation properties of the sample. Similarly, the depth to which the moisture should be removed will depend on the depth of penetration in the ISAT test, and this in turn will depend on the permeation properties (and the amount of moisture in the sample). Attempting to establish fixed times for vacuum treatment would therefore be exceptionally difficult. Monitoring weight loss (which is used for oven drying) is obviously not possible for in situ work.

The vacuum was applied to the concrete surface using the cap from an ISAT apparatus, and the method chosen to determine the required application time of the vacuum was to place some silica gel indicator desiccant in the ISAT cap and to wait until the colour change was observed. Silica gel desiccant is widely available in the form of crystals which have been treated with an indicator which turns pink when moisture is present and blue when it is not. The time taken for the colour to change when in the vacuum on the concrete surface will depend on the pressure and humidity. The humidity in the vacuum depends upon the rate of moisture loss from the concrete, which in turn depends on the moisture content of the concrete surface as well as the pressure and the permeation properties. The time taken for the gel to change colour is therefore dependent on the same parameters which determine the time required for preconditioning.

Development work

Apparatus

The basic procedure using silica gel was established when laboratory work started, but it was necessary to develop the apparatus to ensure that the pressure was low enough to make it work at a practical rate. As an initial trial to determine whether the laboratory vacuum pump was performing adequately, it was tested to see if it could boil water at room temperature. The pump was unable to do this and was replaced, but even with the new pump the gel took 6–18 h to turn blue. A vacuum gauge was then fitted to the system and this revealed numerous leaks in the pipework, the ISAT heads and the seals against the samples. When these leaks had been rectified the vacuum time was reduced to 1–6 h. It is the opinion of the Authors that the use of such a gauge is essential for this technique in order to monitor and minimize leakage. In order to enable work to proceed faster a manifold was built to apply the vacuum to 6 specimens at the same time. However, this can only be used if the samples are relatively impermeable. The flow rates through permeable concrete decrease the vacuum head unacceptably.

Preparation of test samples

Two concrete mixes with mean strengths of 35 and 60 N/mm^2 were used (Table 1). The test specimens, 100 mm cubes, were cast and kept under wet hessian for 1 day before demoulding. Two curing conditions were used: water curing at 20°C; and air curing at 20°C, 55% RH, until testing at 28 days.

Time for silica gel to indicate drying

As ISAT cap containing pink silica gel was placed against a dry impermeable acrylic surface and the time required for the gel to turn blue was recorded. The pressure was then adjusted by bleeding air into the system and the pressure–time relationship shown in Fig. 1 was developed. This shows that the relationship is linear starting from about 3 mbar. It was observed that, especially at low pressures, the times were greater if the oil in the vacuum pump was substantially contaminated with water. In order to achieve rapid drying it is essential to remove this water and change the oil regularly. This problem may also be solved with a water-trap fitted to the inlet of the vaccum pump.

The times recorded using a dry impermeable surface

Table 1. Concrete mix proportions

Concrete grade: N/mm^9	Constituent materials: kg/m^3					w/c
	OPC	Water	Aggregate			
			20 mm	10 mm	Sand	
35	290	190	800	400	715	0·66
60	425	190	800	400	600	0·45

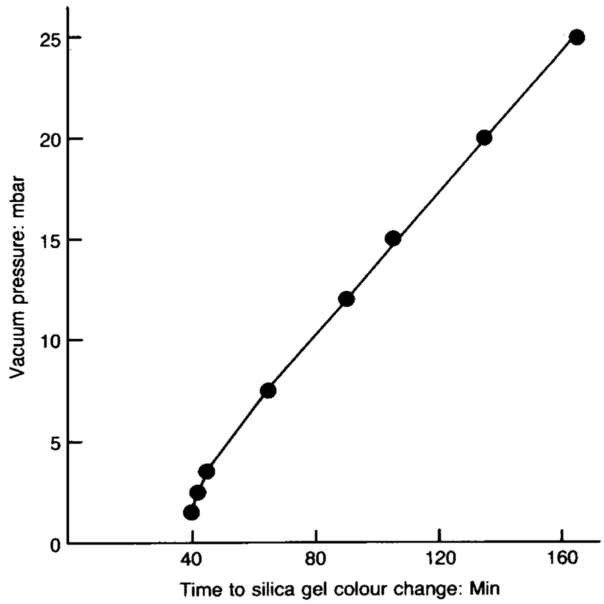


Fig. 1. Time required for silica gel to turn blue on dry impermeable surface

represent the minima for preconditioning at the different pressures; moisture loss from a concrete surface always extended them. In practice the vacuum pressure was determined by the permeability of the concrete, thus the time was less for concretes with low w/c ratios (in comparison with other mixes with the same initial moisture content). Typical times are given in Table 2. The air-cured samples are representative of site concrete, and the conditioning times of 30 and 90 min taken for 35 and 60 N/mm^2 mixes, respectively, are acceptable for site application.

Progressive change of ISAT values

ISAT tests were carried out on samples from each mix and curing condition after different periods of vacuum drying and the 10 min ISAT results were plotted in Fig. 2. It can be seen that the ISAT value was virtually stabilized prior to the colour change of the silica gel.

Comparison with BS 1881 methods

The two preconditioning methods described in BS 1881 for laboratory use are drying for two days in air and drying in an oven at 105°C to constant weight.[9] For site use the test may only be carried out after a period of at least 48 h during which no water has fallen onto the test surface. The criterion for a preconditioning method to be effective is that it should give similar ISAT results on similar samples, regardless of the initial moisture content. The experimental design was therefore based on sets of samples which had been cured in an identical manner and then brought to different moisture contents. The effectiveness of the new preconditioning system in giving similar results from each set was then compared with the BS 1881 laboratory methods using the variance ratio test, known as the F-test. The two-sided F-test was used on

Table 2. Times for gel to turn blue (samples taken directly from curing)

Concrete grade: N/mm^2	Curing: 20°C	Time: h	Pressure: mbar
35	Air	1·5	8
60	Air	1·0	5
35	Water	6·0	3
60	Water	4·0	2

the null hypothesis that the variation in results caused by the different moisture contents was the same for the different preconditioning treatments. In addition a fourth preconditioning treatment, in which a vacuum was applied for twice the time taken for the gel to turn blue, was used in order to see if further improvements could be obtained (this preconditioning is referred to as 'twice vacuum').

The following three different methods were used to produce samples with a range of different moisture conditions

(*a*) vacuum-saturate for 2 h at 10–15 mbar (typical weight gain from dry curing 2%)
(*b*) 6 h in water (typical weight gain from dry during 1%)
(*c*) dry in laboratory air for 28 days.

For each of the two mixes, two curing conditions, three different moisture contents and four preconditioning methods, two samples were tested, giving a total of 96 samples. The results are given in Table 3.

The coefficient of variation (V = the standard deviation divided by the mean) has been calculated from the ISA-10 results from two samples from each moisture condition,

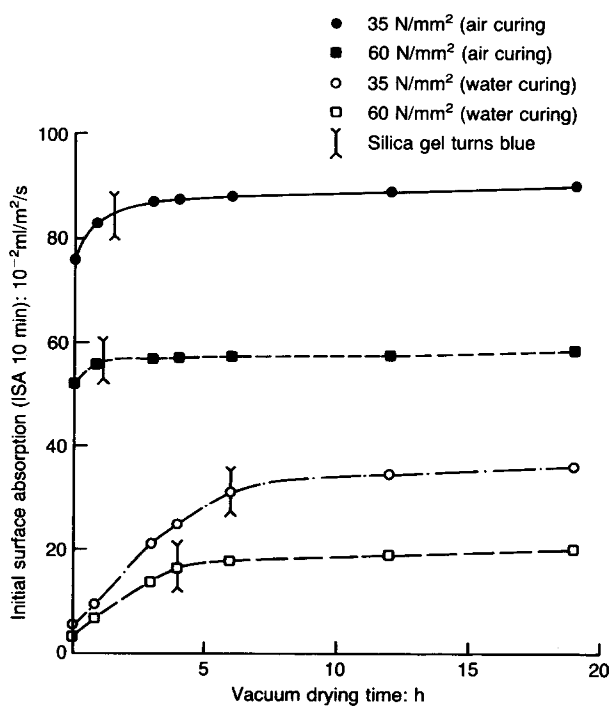


Fig. 2. ISAT values after different drying times

Table 3. Comparison between different preconditioning methods

Concrete grade: N/mm^2	Curing	Pre-conditioning	Mean ISA-10 $\times 10^{-2}$ ml/m^2/s	V of ISA-10: %	F-statistic
35	Air	Vacuum dry	80·7	8·4	1·0
		2 day air	69·9	40·4	22·9
		Oven dry	122·8	6·7	1·6
		Twice vacuum	86·3	8·4	1·0
35	Water	Vacuum dry	31·3	16·2	1·0
		2 day air	17·9	65·7	16·6
		Oven dry	53·0	6·9	5·5
		Twice vacuum	34·6	16·0	1·0
60	Air	Vacuum dry	44·9	15·2	1·0
		2 day air	24·5	54·9	13·0
		Oven dry	65·1	7·7	3·9
		Twice vacuum	46·3	15·0	1·0
60	Water	Vacuum dry	21·5	23·0	1·0
		2 day air	14·0	66·2	8·3
		Oven dry	48·7	10·9	4·5
		Twice vacuum	28·1	22·9	1·0

i.e. a total of 6 samples in each case. The critical value for the variance ratio is called the F-statistic. The F-statistic for 95% confidence limits is 7·15 (both degrees of freedom being 5). For each of the four mix/curing combinations it is therefore indicated that the coefficient of variation of the vacuum-dried samples is significantly less than for the 2 day room-dried samples, i.e. the null hypothesis is rejected. For all other cases the null hypothesis is accepted, although it may be seen that the coefficient of variation is lowest for the oven-dried samples in each set.

In Fig. 3 the standard deviations are shown as error bars. It may be seen that the standard deviations of the oven-dried sets of samples are no larger than those for the vacuum-dried samples. This results in coefficients of variation being lower for oven drying because the means are higher.

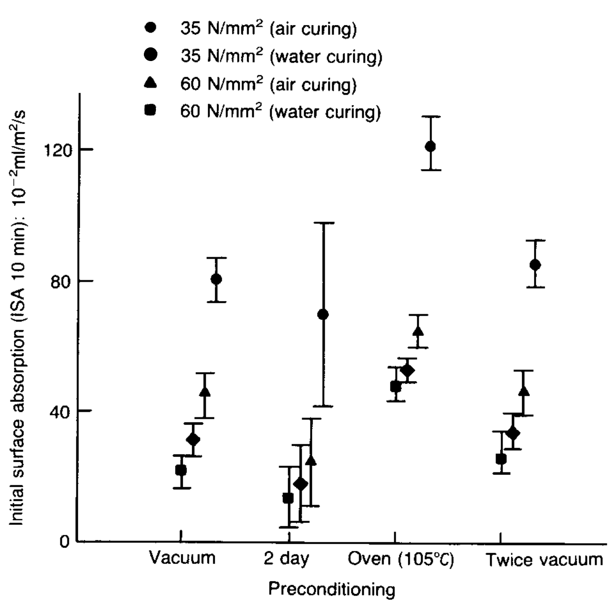


Fig. 3. Standard error bars for ISAT results

Discussion

As expected, oven drying gives highest ISAT values and lowest coefficient of variations. It can be argued that oven drying is the most efficient method, since it gives consistent results regardless of the moisture history of concrete. Unfortunately, this method cannot be used in situ. It is interesting to note from Fig. 3 that the vacuum drying results are the only ones in which the four different mix/curing combinations chosen for this study have been fully resolved, in that the error bars do not overlap. The graph indicates that the results from this preconditioning are better than the others at distinguishing between the different concrete 'qualities' and would thus be of greater practical use. Preconditioning by air drying is the only method currently used for in situ ISAT testing and the results show that changing to vacuum drying would give a significant improvement in the reproducibility of the results. It has been suggested[10] that the presence of moisture is beneficial to concrete, and absorption should therefore be measured in naturally occurring conditions. This would give lower values of absorption, indicating higher durability for structures in moist conditions. The Authors believe that drying prior to absorption testing is essential, since porous or cracked concrete would have a high moisture content. This would yield low absorption values (indicating high durability) if the concrete had not been dried before testing.

The 'twice vacuum' samples which were subjected to the vacuum for twice the time taken for the gel to turn blue were not significantly different from the 'vacuum dry' samples which were tested when the colour change occurred. This result indicates that minor increases in vacuum time will not have a significant effect on the results. This is important because the colour change is a gradual process which takes place over about 10 or 20 min, and observing an exact time for it is not possible.

The mean ISAT values were higher for the lower grade of concrete and less efficient curing, i.e. decreasing quality, as expected. Although the coefficient of variation remained significantly lower for the vacuum-dried samples relative to the two-day-dried samples, the difference decreased with increasing concrete quality. The 60 N/mm^2 water-cured samples, however, probably exceed the quality found on most existing structures. As the quality decreased towards more typical site concretes the difference became more significant and the vacuum-dried results were comparable with the oven-dried ones.

The ISAT is not the only test of its type; both the air permeability index test APT[3] and covercrete absorption test CAT[4] may be used to measure the permeation properties of concrete cover. The results of such permeation tests will also be affected by the moisture condition of concrete. Further work is in progress to develop the application of these two tests with vacuum drying for in situ testing. Initial results are proving to be very encouraging, and the Authors hope to report developments once the work has been successfully completed.

Proposed test procedure

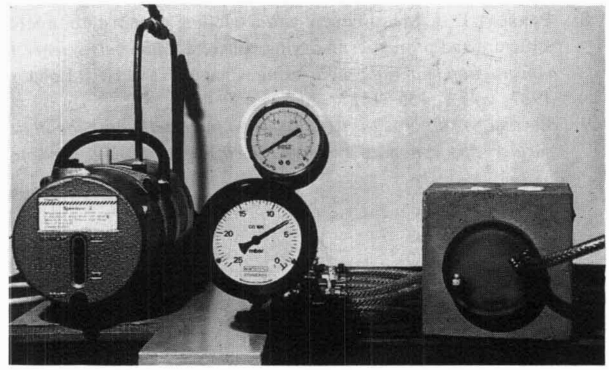
The testing procedure which was used in this project and is recommended for site is as follows

(*a*) modify the head of an ISAT apparatus by blocking one pipe connection and connecting the other to a vacuum pump (the ISAT head must be made from transparent material)
(*b*) expose some silica gel to air until it turns pink
(*c*) place the ISAT head against the area of concrete to be tested with the silica gel inside it (note that clamping is not necessary)
(*d*) start the pump and check that the pressure is 10 mbar or less
(*e*) run the vacuum pump until the majority of the gel turns blue
(*f*) carry out a normal ISAT test starting within 10 min of the release of the vacuum.

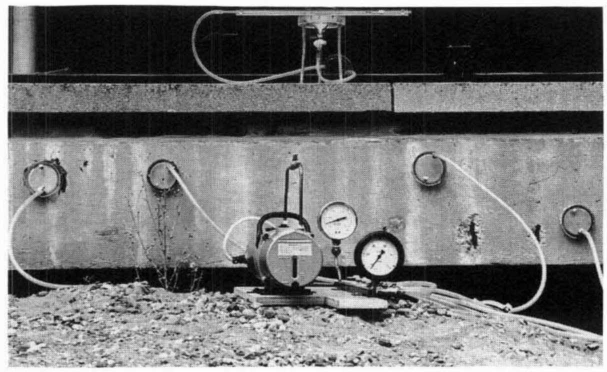
The components of the apparatus used for this work are shown in Fig. 4; they are very simple and robust and may readily be used on site. A single-stage mechanical vacuum pump (Edwards Speedivac) was connected to an ISAT cap 12 mm PVC braided tube. A 0–25 mbar vacuum gauge was used (Capsule dial type, also supplied by Edwards). The total cost of this apparatus was £600.

Conclusions

The proposed ISAT method represents a valuable improvement relative to the methods given in BS 1881.



(a)



(b)

Fig. 4. Improved in situ ISAT method: (a) vacuum apparatus; (b) application on site

The method can be used in situ. It is fast and is unlikely to affect the microstructure to a greater extent than normal atmospheric drying and gives repeatable results.

This technique will permit comparison of data from in situ and laboratory sources.

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