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Efficiency of the saturation of concrete with liquid under vacuum conditions

Abstract

The saturation of concrete with liquid under vacuum conditions is commonly recognized as the most efficient saturation technique. Therefore, this technique is recommended to be performed in porosity measurements (to determine the water-accessible porosity) and in chloride diffusion/migration tests (to ensure that the diffusion/migration the only transport mechanism of chlorides during the test).
The efficiency of vacuum saturation was analyzed in this study by saturating concrete samples with a chloride solution (chloride is used as a tracer). After performing the vacuum saturation, the chloride penetration depth in several split samples was detected using a colorimetric indicator for chlorides, while in other samples the chloride concentration was measured at different depths. The observed chloride penetration depths indicate that the external chloride solution could not access the entire volume of the saturated samples. The measured chloride concentration profiles show that the chloride concentration gradually decreases from its maximum at the surface to lower concentrations in the deeper layers of concrete. From the results presented in this study it may be concluded that the vacuum saturation does not ensure a complete liquid-saturation of concrete, regardless of the quality (permeability) of the latter or thickness of the sample in the investigated range (of 15 up to 50 mm).

Introduction

Concrete is a porous material in which the size of pores varies greatly in the range from nanometers (gel pores) up to a few mm (entrapped air bubbles). The pore system of concrete is very complicated due to the large variety of pore sizes, their structure (tortuosity and constrictivity) and volume. Many durability issues of concrete are indirectly related to its porosity, as the aggressive substances (e.g. Cl^-, CO_2, SO_4^{2-}) are penetrating the concrete through the pores. Therefore, the pore size distribution and porosity measurement techniques are important and well documented research topics.
In order to quantify the chloride ingress speed into concrete, the chloride diffusion/migration coefficient is used, because the diffusion controls the ingress. The chloride diffusion coefficient is a parameter that reflects on the pore structure and porosity of concrete, i.e. its value is increasing when the amount of pores through which the ions can permeate increases.
In some tests performed on concrete it is necessary that all the accessible pores are saturated with liquids. In the case of the porosity (apparent density) determination, the analyzed concrete sample is firstly weighted in a water-saturated state, dried to a constant mass and weighted again (to quantify the volume of free water that can be accommodated in the pores) [1]. Therefore, a proper and efficient saturation technique is needed to determine the porosity accurately. In the case of the chloride

diffusion/migration test such as e.g. [2, 3], it is necessary to remove the air from the pores as it hinders the chloride diffusion-based transport and can additionally promote the chloride transport due to capillary suction. Despite the fact that in real conditions the capillary suction of liquids usually occurs (especially in the surface layers of concrete), for the sake of simplicity, in chloride transport models developed for the diffusion and migration test, the chloride transport is assumed to be diffusion-based and therefore the test conditions require a complete liquid-saturation of the concrete pores.

As demonstrated by Safiuddin and Heran [4], the vacuum saturation technique is more efficient than the other techniques described in the ASTM standards (i.e. immersion in water for a long period and immersion in boiling water). In this technique, the air present in the concrete samples is removed by the application of a low pressure (< 50 mbar) and replaced by liquids. Nevertheless, as shown in Spiesz and Brouwers [5], in saturated concrete samples split after performing the Rapid Chloride Migration (RCM) test, a liquid penetration front is visible, which the authors attribute to a non-complete saturation of the samples prior to the RCM test. Therefore, the objective of the present study is to demonstrate whether concrete samples can be fully saturated with liquids by performing the vacuum saturation.

Materials and concrete mixture composition

Three concrete mixtures were prepared in this study, representing different qualities (permeabilities), in order to investigate whether the vacuum saturation efficiency depends of the quality of concrete. The mixing proportions of the prepared concretes and their hardened properties are shown in Table 1.

Table 1
Composition and hardened state properties of the designed concrete mixtures

Materials	**Mix 1**	**Mix 2**	**Mix 3**
	[kg/m^3]		
CEM I 42.5N	300.0	400.0	340.1
Nano-SiO_2 (dry powder)	0.0	0.0	12.8
Limestone powder	0.0	53.5	151.9
Micro-sand (sandstone)	0.0	0.0	141.4
Sand 0-4	674.9	669.6	618.0
River gravel 1-8	703.6	652.5	0.0
River gravel 4-16	500.3	433.6	0.0
Granite 2-8	0.0	0.0	735.7
Granite 8-16	0.0	0.0	274.3
Water	165.0	160.0	153.0
Superplasticizer (% wt. of cement)	0.0	0.5	1.9
w/c	0.55	0.40	0.45
w/p (< 250 μm)	0.45	0.31	0.27
Fresh density [kg/m^3]	2307	2383	2392
Air content [% vol.]	4.49	2.08	1.58
28-day compressive strength [MPa]	44.65	69.59	78.5
91-day compressive strength [MPa]	-	-	91.0

Mix 1 represents concrete with a poor quality, which consists of a low cement content and high water/cement ratio (w/c = 0.55), no superplasticizer or limestone powder. A mixture with an average quality is represented by Mix 2, in which the cement content is increased and the water/cement ratio reduced compared to Mix 1. Additionally, limestone powder is used as inert filler. Mix 3 represents concrete of a good quality (low permeability). This mixture is a self-compacting concrete with high amount of fine particles (< 250 µm) and low water/powder ratio (w/p) as shown in Table 1. To further improve the quality of this concrete, additionally a small amount (3.8% by weight of cement) of powder amorphous nano-SiO_2 is used.

Experimental plan and test methods

A set of concrete cubes (150 mm size) was cast for each mixture presented in Table 1, demolded after 24 hours and subsequently stored in water under laboratory conditions. 28 days after casting, the compressive strength test was performed on three cubes from each mixture (values given in Table 1) and from the remaining cubes cylindrical cores (diameter of 10 cm) were extracted by drilling. The compressive strength of Mix 3 was also determined at the age of 91 days on three cubes, as this concrete was cured longer in order to further improve its quality (reduce the permeability). The vacuum saturation experiments on concrete samples of Mix 1 and Mix 2 were performed at the ages of 29-36 days and on samples of Mix 3 at the age of about one year. A scheme of the sampling is shown in Fig. 1. The external layers (about 15 mm in height) of each core were cut off in order to prevent the surface effects. Samples of different thicknesses (15, 30 and 50 mm) were cut from the drilled cores. Discs of 50 mm in diameter are recommended for the chloride test [2, 3], and the thinner discs were prepared to investigate the effect of sample thickness on the vacuum saturation efficiency.

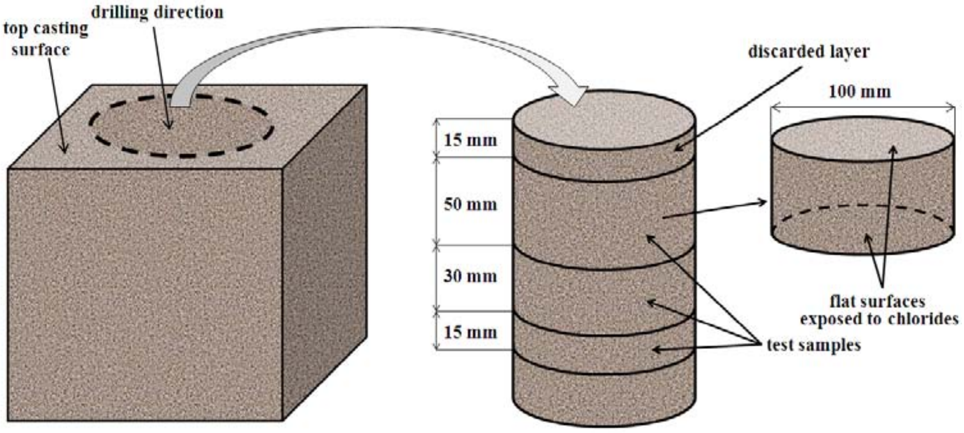

Fig. 1
Scheme of sampling for the vacuum saturation experiments

Vacuum saturation

A modified test set-up based on the vacuum saturation apparatus described in [2, 3] is used in this study, as shown in Fig. 2. A membrane vacuum pump is used in the experiments instead of an oil pump. For membrane pumps no water trap is needed (the water vapor is allowed to enter this type of pump), and therefore the test set-up is less demanding. In such a case however, more attention has to be paid by the operator in order to prevent an entrainment of liquids into the pump. An additional desiccator (5 in Fig. 2) is used to de-air the liquid prior to saturating the samples. All lids of the desiccators and hose connectors are sealed using a vacuum-grease.

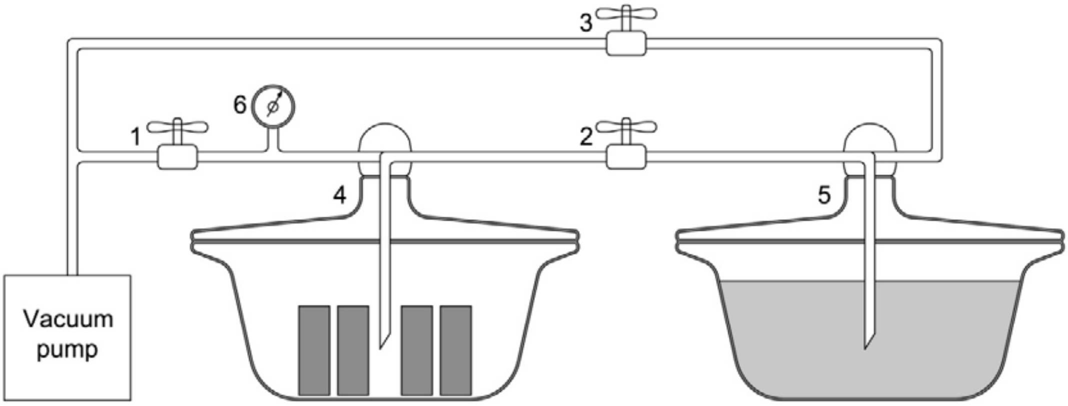

Fig. 2
Vacuum-saturation set-up: 1, 2 and 3 – valves, 4 and 5 – desiccators and 6 – vacuum gauge

Concrete samples are placed vertically in the dessicator (4 in Fig. 2) with both flat surfaces uncovered and exposed and the vacuum pump turned on for 3 h, with the valves (1) and (3) opened and valve (2) closed. The pressure indicated by the vacuum gauge (6) reaches 40 mbar (4 kPa) within about 2 minutes. After 3 h, valve (3) is closed and valve (2) opened, in order to allow the de-aired liquid (10% NaCl water solution saturated with $Ca(OH)_2$) to be slowly transported from desiccator (5) to desiccator (4). When the level of liquid in desiccator (4) is about 1 cm more than the height of the samples, valve (2) is closed and the vacuum is maintained for an additional hour. After that, the pump is turned off and the air is allowed to slowly re-enter the desiccator (4). The samples are stored in the liquid for 18 ± 2 hours.

Chloride penetration depths

The chloride penetration depths were analyzed on split concrete samples, saturated under vacuum with a chloride solution. Similarly to the vacuum saturation guidelines [2, 3], the samples were not dried prior to the vacuum treatment and therefore the chloride solution entering the samples replaced only the air present in the pores. Here the chlorides are used as tracers in order to determine how deep within the sample the external solution can penetrate (replace the air) during the vacuum saturation. The chloride penetration depths are revealed by spraying the axially split samples with $AgNO_3$ solution. AgCl, being the product of the reaction of $AgNO_3$ with chlorides, has a white colour, while AgOH (which is later transformed to Ag_2O), formed in the chloride-free regions of the sample, is brownish. Therefore, the boundary between the regions with and without chlorides becomes clearly visible and the chloride penetration depth can be measured. As shown in [5, 6], the concentration of chlorides detectable by this spraying method is about 0.1 % ($0.1\ g_{Cl^-}/100g_{concrete}$). The chloride concentration measurements are not performed on the non-dried concrete samples as the pore liquids present in these samples prior to the saturation process dilute the entering chloride solution.

Chloride concentration profiles

In order to avoid the dilution of the chloride solution with pore liquids, another series of concrete samples was dried prior to the saturation. The drying process was performed in an oven at 105 °C until a constant mass of the samples was reached. The performed vacuum saturation procedure is similar to the one discussed above. Because the free water was removed from the samples prior to the saturation, the external chloride solution had entered the entire volume of the split samples after the vacuum saturation, as was revealed after spraying their fracture surfaces with $AgNO_3$. The concentration of chlorides was analyzed for each concrete sample in the layers which were extracted from between its surface and the middle of its thickness. During the vacuum saturation process, the chloride solution enters the concrete at a high rate and thus, the chloride binding during the application of vacuum can be neglected as the binding is a slow process. After finishing the vacuum saturation, concrete samples are stored in the solution for approximately 14 hours, during which the chlorides that already entered the samples can be bound by the hydrated cement. However, this will not influence the chloride concentration profile, as the total chloride concentration (free plus bound chlorides) is being measured. The chloride concentration profile measurement procedure is described in detail in [5, 7]. In about 2 hours after performing the vacuum saturation and the storing period in the solution, the samples are ground in layers to collect powder for further analysis. A surface layer of 0.5 mm is ground at first and then 1 mm layers are ground consecutively, until the center of the sample is reached. About 4 g of concrete powder is collected from the surface layer and about 8 g from every deeper layer. Afterwards, the powder is dried in a ventilated oven at 105 °C, until a constant mass is reached. In order to extract the chlorides from the powder, for each analyzed layer 2 g of the dried powder is poured into a beaker together with 35 ml of distilled water and 2 ml of 1 M HNO_3, shaken manually for 1 min and heated up to reach the boiling point. This is performed in order to extract the chlorides bound by the hydrated cement phases. Next, the solution is cooled down, filtered, and its volume is adjusted to 100 ml by adding distilled water. Samples of 10 ml are analyzed for the chloride concentration by using an automatic potentiometric titration unit and 0.01 M $AgNO_3$ solution as the titrant. The measured concentration is expressed as the mass of chlorides in 100 g of dry concrete.

Results and discussion

Chloride penetration depths

The chloride penetration depths, shown in Figure 3, represent those regions in concrete, in which the air from the concrete pores could be removed by the vacuum and replaced with the chloride solution. It can be clearly noticed that the chloride solution could enter the concrete of a lower quality (Mix 1, Fig. 3a) easier than the concrete of a higher quality (Mix 3, Fig. 3b). This means that for concrete with a higher permeability the vacuum saturation works more efficiently compared to denser concretes, in which the air entrapped in the finer pore structure is resisting the vacuum more strongly. Any quantitative analysis of the saturation degree cannot be performed based on the images shown in Fig. 3 as they only indicate those spots within the samples, in which the chloride concentration exceeded the value of 0.1 $g_{Cl^-}/100g_{concrete}$. In none of the analyzed samples the chloride solution was present in their entire volumes. Additionally, in the presented pictures no clear trends between the efficiency of the vacuum saturation and the thicknesses of the samples can be observed.

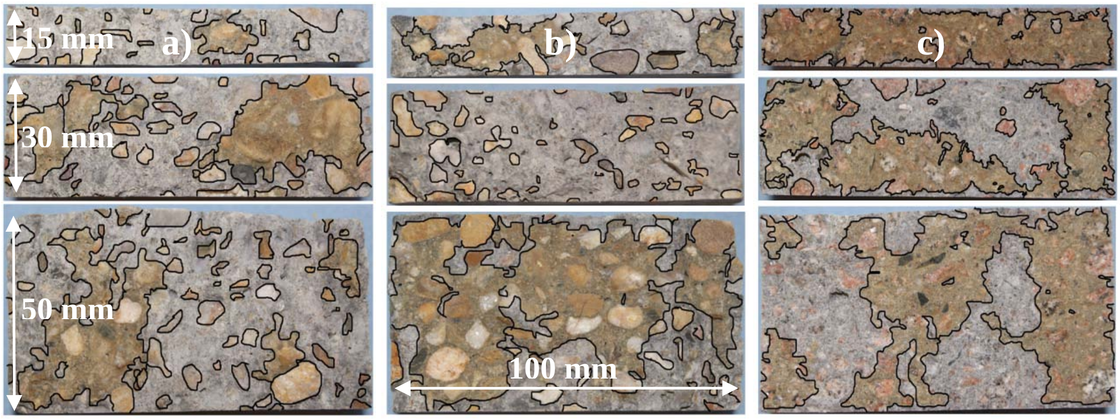

Fig. 3
Chloride penetration depths after saturating the concrete with chloride solution; a) Mix 1, b) Mix 2 and c) Mix 3

Chloride concentration profiles

As explained earlier, the free water was evaporated from some of the concrete samples prior to the vacuum saturation and therefore the dilution of the penetrating chloride solution was prevented. Assuming that the saturation of a homogenous concrete sample with a chloride solution in vacuum conditions is complete, the total chloride concentration should be at the same level in the entire sample.

The total chloride concentration profiles measured on concrete samples of different thicknesses are shown in Fig. 4. The total chloride concentration is proportional to the porosity of concrete when the chloride solution is present only in the pores. As expected, the measured chloride concentrations are larger for the more porous concrete (Mix 1 with w/c ratio of 0.55) than for the denser concretes (Mix 2 and Mix 3 with w/c ratios of 0.40 and 0.45, respectively). The extended curing period and the addition of nano-silica in Mix 3 are also reflected in the results, as the total chloride concentration measured for this concrete is slightly lower (thus the porosity is also lower) than measured for Mix 2, in which a lower w/c ratio was used.

For all the measured total chloride concentration shown in Fig. 4, a general trend can be observed that the concentrations of chlorides are gradually decreasing from the maximum value at the surface to about half of the maximum value in the middle of the samples. The increased chloride concentrations measured in the vicinity of the external surface layers of concrete can be attributed to an increased porosity of these layers, generated during the preparation of the samples (cutting using a diamond saw and drying at 105 °C). Therefore, assuming a 100% degree of saturation in the surface layers of concrete (i.e. all the pores are completely saturated), the saturation level in the middle of the samples is only 50%.

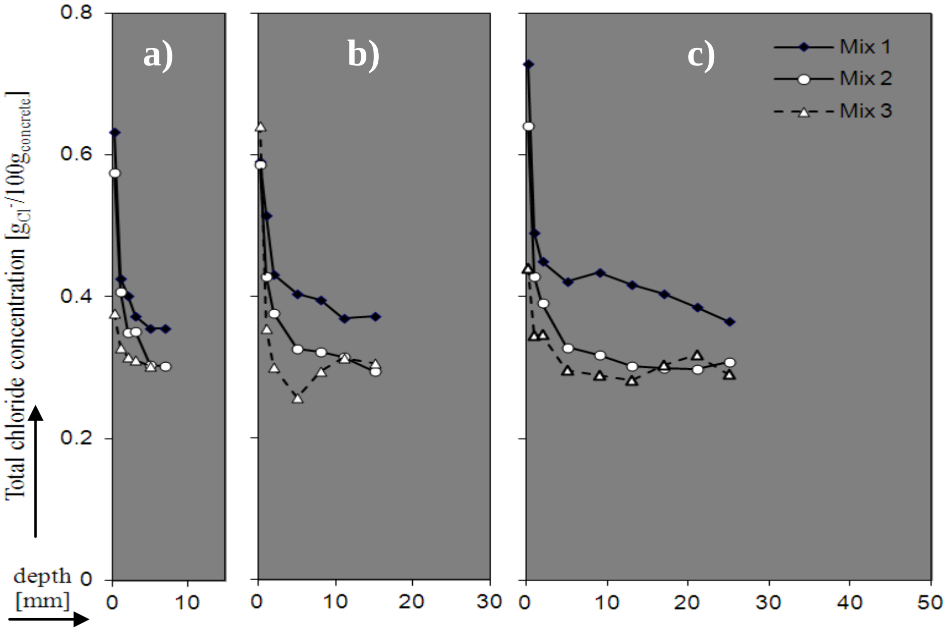

Fig. 4

Total chloride concentration profiles measured in dry concrete samples saturated under vacuum with a chloride solution; samples of a) 15 mm; b) 30 mm and c) 50 mm thickness.

When assuming a uniform porosity in different layers of the samples, the layers of concrete in Fig. 4, which have an increased chloride concentration, are also better saturated with the chloride solution. Due to a potentially larger porosity in the surface layers of concrete, the surface chloride concentration values measured in these layers could be discarded in the assessment of the saturation efficiency. In such a case, the difference in the saturation level for the remaining depths in the concrete samples is significantly reduced. However, even in this case the deeper layers of concrete show a lower saturation degree (especially in the samples of 50 mm thicknesses). Consequently, it can be stated that the vacuum saturation technique is not able to fully saturate concrete samples with liquids and the efficiency of the saturation decreases when the analyzed depth of the sample increases.

Conclusions

This study shows that the vacuum saturation of concrete with liquids does not ensure a complete saturation of the concrete pores. The experiments were performed on concrete samples with poor and good qualities and with different thicknesses. The results show that, after performing the vacuum saturation with a chloride solution, chlorides were detected only in some regions of the samples. Additionally, the measured total chloride concentration profiles show that, after performing the vacuum saturation of pre-dried concrete samples with a chloride solution, the chloride concentrations are gradually decreasing with the depth of the sample. Therefore, it can be concluded that the amount of the liquid in the deeper layers of concrete is lower than in the layers closer to the exposed surfaces, so the saturation is not complete.

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