

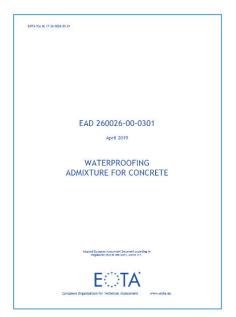


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First "Waterproofing Admixture for Concrete" Test Standard is Published

The First Hydrostatic Pressure Standard for Crystalline Concrete Admixtures

For the past several years, the Reseach & Development Group of Xypex Chemical Corporation has been working with the Deutsches Institut für Bautechnik (DIBt), to create the first hydrostatic pressure standard for crystalline concrete admixtures such as the Xypex Admix C-Series. In 2021, EAD 260026-00-0301 "Waterproofing Admixture for Concrete" was adopted by the European Organization for Technical Assessment (EOTA) and published in the EOTA Journal. Xypex was already compliant, having previously completed all the rigorous testing requirements which included at the core the harmonized test standard EN 12390-8 "Testing hardened concrete – Part 8: Depth of water penetration under pressure". Xypex Admix C-1000 NF is the first product to complete the European Technical Assessment (ETA) to this new EAD.



Existing Standards Were Not Appropriate

In EU countries, admixtures for concrete must meet the requirements of EN 934-2 "Admixtures for concrete, mortar and grout". This standard regulates concrete admixtures such as water reducing/plasticizing admixtures (Table 2), air entraining admixtures (Table 5), and set accelerators (Table 6) as well as a few specialty admixtures including the so called "water resisting admixtures" (Table 9). These "water resisting admixtures" are essentially hydrophobic absorption reducing products. Numerous other common types of admixtures are not in the standard. Notably absent are shrinkage reducing admixtures, corrosion inhibitors, and importantly for Xypex, true hydrostatic pressure permeability reducing admixtures.

In this Technical Note, we explain why the Table 9 "water resisting admixture" category was inappropriate for evaluation of hydrostatic pressure permeability reducing admixtures and why a new standard needed to be developed.



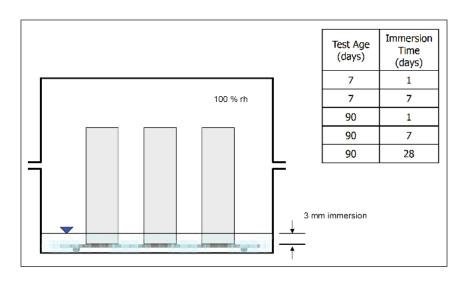
Table 9 and EN 480-5 Explained

EN 934-2 regulates admixtures for plain, reinforced and prestressed concrete. Specific performance and preEN 934-2 regulates admixtures for plain, reinforced and prestressed concrete. Specific performance and prescriptive requirements for each type of admixture are given in Tables 2 to 12. Of particular interest to Xypex is Table 9 which has the requirements for "water resisting admixtures". Table 9 specifies requirements for capillary absorption, compressive strength and air content. Tests for true hydrostatic pressure water permeability are not included.

At first glance, it appears that Xypex Admix products and integral crystalline admixtures in general should fit into Table 9 "water resisting admixtures". They do not. This is because the specified performance test is a capillary absorption test called EN 480-5 "Determination of capillary absorption". Simply put, absorption tests are not appropriate for evaluating permeability reduction and are definitely not appropriate for integral crystalline admixtures that require water to react such as Xypex Admix products.

In the EN 480-5 test, a standardized mortar mix is prepared with and without the admixture. Small prism specimens are cast and cured for 24 hrs in the molds. Specimens are then lightly air dried (65% rh) for either 7 days or 90 days.

The test is carried out as shown in the following figure. Small, 40x40x160 mm mortar prisms are set upright in 3 mm of water. The change in mass of the sample is recorded after various immersion times in order to determine the amount of water absorbed.



In order to meet the requirements of EN 934-2 Table 9, the admixture must have > 50% reduction in absorption compared to the control for the 7 day cure / 7 day immersion period (7/7). In addition, it must have > 40% reduction (60% of control) for the 90 day cure/28 day immersion timeframe.

There are two critical problems with this procedure that need to be considered. First, the test is a pure absorption test and no water pressure is ever applied to the samples. Second, the admixture must have an extremely high level of performance at only 7 days age. It can be clearly seen that the EN 480-5 test is a simple absorption test that does not measure true water permeability and is not appropriate for materials that are only partially developed at 7 days age. It is therefore not appropriate for the evaluation of Xypex Admix products.



Background on Absorption vs Permeability

To fully understand the importance of using the correct test method to evaluate Xypex Admix products, it is useful to review the technical meaning of absorption, permeability, porosity and other terms related to the movement of liquids and gasses through porous solids such as concrete.

Firstly, porosity is a simple measure of the volume of pores in a solid. Concrete porosity is in the range of 5 to 15%. Rock porosity is often less than 2%. Porosity is a crude but sometimes useful measure of the quality of concrete. Very porous concrete will generally be both weak and permeable to liquids and gasses.

Permeability is a material property that indicates the ease with which any liquid or gas flows through the connected pores in a solid. In our case, we are most interested in the permeability to water. For this we can define a coefficient or constant known commonly as the D'Arcy coefficient or water permeability coefficient:

$$Q = \frac{K \cdot A \cdot \Delta h}{l}$$

where: K = water permeability or D'Arcy's coefficient (m/s)

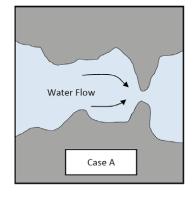
Q = flow rate through the sample (m^3/s) A = cross section area of sample (m^2)

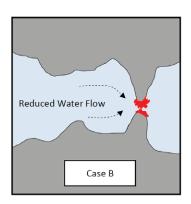
 Δh = water pressure differential across the samples (m)

1 = sample length (m)

This equation shows us that water pressure (Δh) is the driving force and a combination of the permeability (K) and path length (I) resist water flow. In other words, low permeability and adequate wall thickness are both needed to make a watertight structure. It should be emphasized that many researchers have shown that water permeability is the most fundamental parameter with respect to the durability of concrete so this property is important for both waterproofing and durability.

Porosity and permeability should not be confused. Porosity is simply a measure of the volume of pores. It is possible for materials to be porous and not permeable if the pores are not connected. This is illustrated in the following "ink bottle pore" example. In Case A, water flow is restricted somewhat by the narrow "ink bottle" opening. In Case B, the total volume of porosity is very similar to Case A. However, due to crystal growth or growth of secondary CSH (as is common with Xypex crystals and supplementary cementing materials) the neck of the ink bottle is blocked. Water flow through Case B would be stopped under the same pressure.





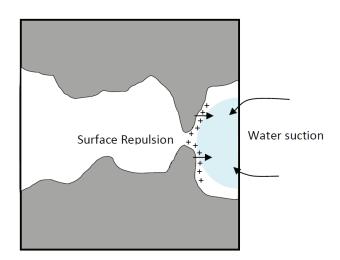


As a result, Case A and B have nearly the same porosity but Case B has much reduced permeability. Thus porosity is not a good measure of water permeability when comparing different types of materials (or mix designs with different additives).

Absorption is a somewhat different phenomenon. Dry or partially dry concrete will suck up (absorb) water and gain mass when liquid water is applied to the surface. This continues until the concrete pores are nearly full of water. When the pores are full of water the concrete is said to be saturated. This sucking of water will occur without any external water pressure. Instead, internal "capillary pressure" is the driving force.

Capillary pressure can be explained as follows. When concrete is not fully saturated, both water and air are present inside the concrete. Two important interfaces need to be considered. One is the air/water interface and the second is the water/solid interface. The air water interface results in a surface tension on the surface of the water. The water/solid interface results in a contact angle. These together result in the suction needed to pull water up into the concrete. Resistance to flow is provided by the permeability (K value) of the porous solid which is due to the size, shape and connectivity of the pores. The water will stop being absorbed into the concrete when the suction forces equal the resistance forces set up by shape and connectivity of the pathways.

To understand absorption test results it is important to know why low absorption is occurring. For example, the effect of a hydrophobic admixture is such that it changes the contact angle to an extent that the lining of the pores reject water. This is shown for the same pore diagram in the following figure.



In this case water suction is reduced due to a repulsive force of charged molecules of the hydrophobic additive attached to the capillary pore wall. The permeability of the pore has not changed as there has been no alteration to the pore shape characteristics. Once pressure is applied, the influence of the contact angle is negated and water will flow freely into the pores as if the nothing had been done to treat the concrete. This is why hydrophobic additives tend to have poor performance in true hydrostatic pressure permeability tests.

On the other hand, Xypex Crystalline Technology needs to absorb water to initiate crystal growth and thus reduce permeability. After these reactions occur and the permeability of the concrete is substantially reduced, the concrete will dry out. However, Xypex treated concrete will always absorb some water into the dry near surface layers as it does not reject water like a hydrophobe.



A Further Issue with EN 480-5

Historically, some hydrophobic admixture companies have specified absorption testing at 7 days (7 days curing, 7 days immersion), a measure which gives the impression of elevated absorption performance. Possibly due to the shorter testing time required, this has been adopted by EN 480-5 and gives cause for concern when evaluating permeability reduction. The reason is simple; normal concrete takes at least 28 days to achieve a reasonable degree of hydration reaction. Permeability reduction continues for many more months after this. At 7 days age, concrete is very immature and consequently will have a very high absorption. However, hydrophobic admixtures do not rely on the cement reaction to repel water and as a result are capable of absorption reduction even at a very early age. There is no engineering need for early age absorption reduction in normal situations as few structures will ever be put into service 7 days after pouring concrete. Thus not only the use of absorption testing but also the very fast testing period will not give any indication of the performance of a permeability reducing admixture under hydrostatic conditions.

How Xypex Works and How it Should be Tested

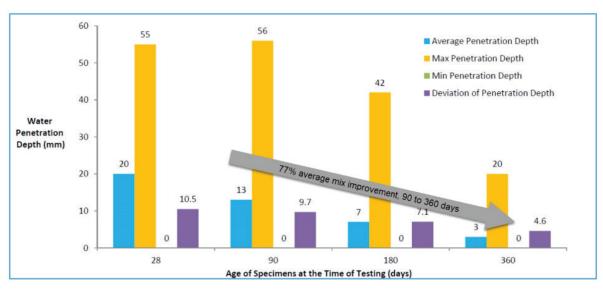
Integral crystalline technology is manufactured in the form of a dry powder compound consisting of a unique blend of proprietary chemicals. The proprietary chemicals react with the by-products of cement hydration and combine within the concrete matrix to produce a non-soluble needle-like crystalline formation that crisscross the capillary tracts and fine shrinkage cracks in the concrete, creating such a tortuous path that water cannot penetrate to any significant depth

When a cement particle hydrates, the reaction between water and the cement causes it to become a hard, solid, rock-like mass. The reaction also generates chemical by-products that lie dormant in the concrete. Crystalline technology adds another set of chemicals to the mixture. When these two groups, the by-products of cement hydration and the crystalline chemicals, are brought together, in the presence of moisture, a chemical reaction occurs which produces a new non-soluble structure in the capillaries, micro cracks and shrinkage cracks in the concrete. By means of the crystalline reaction the connectivity within the porosity of the concrete is greatly reduced and hairline shrinkage cracks are sealed against water leakage.

Due to this unique chemical reaction between water, Xypex Admix chemicals and the by-products of cement hydration, specialized curing and water permeability test procedures are needed in order to properly evaluate the admixture performance.

Absorption type tests should not be used to evaluate integral crystalline admixtures. Absorption methods are not able to properly test for the effects of water pressure, chemical reactions with water, swelling, curing and time related effects, as well as more complex types of pore blocking, such as changes in connectivity, silting, and micro-crack healing. There can be a very poor correlation between absorption and permeability for integral crystalline admixtures and the use of an absorption test can often under-predict the true benefit of the admixture to waterproof structures.





Long-term improvement with time is demonstrated in this study on 90 sets of water permeability tests.

Fortunately, there are direct water permeability tests that are readily available that can be used to properly evaluate integral crystalline admixtures. Water permeability tests can be completed directly by applying water pressure to one side of a specimen and measuring steady state flow through the specimen. The saturated K value can be directly determined. The US Army Corp CRD 48 test is one such standardized method, but there are numerous similar procedures reported in the technical literature. This type of test can be difficult to complete on modern, low permeability concrete and more often water does not fully penetrate the specimen in a realistic period of time or at a realistic pressure.

To overcome this problem, a simpler but somewhat less direct method is to measure the depth of water penetration under pressure. For many years there existed a water permeability test method based on water penetration depth. Early standardization of this method goes back at least as far as 1978 (German Standard DIN 1048 Part 5 Dec 1978). In recent years, the same procedure was adopted as the European standard test method EN 12390-8 "Testing Hardened Concrete – Part 8: Depth of water penetration under pressure". In this method, concrete is cast into suitable sized molds (typically 150 mm cubes) and cured for 28 days. Water pressure of 500 Pa is applied to one face for a period of 72 hrs. Samples are split open and the depth of water penetration is measured. This procedure and other similar nationalized versions have become very commonly employed around the world. There is significant historical data on the procedure that can be used for adoption as a standardized method for concrete evaluation. The method has been used for performance evaluation and acceptance of admixture treated concrete in Europe, Australia, Brazil, Hong Kong, United States and other countries. The depth of water penetration under pressure more closely correlates to water permeability than it does to water absorption.



Future Work - Using a More Realistic Curing Regime

In recent years, Xypex has been working on modifications to these standard procedures that makes the curing regime more accurately reflect the real conditions that concrete is exposed to in-service. The use of 28 day short term curing is a convention associated with strength gain of pure Portland cement as it was manufactured decades ago but does not reflect the reality for more modern concrete or the in-service condition where concrete will continue to improve for months and even years after being cast.

This is particularly important for integral crystalline admixtures such as Xypex Admix, which continue to improve significantly after 28 days. To model this behavior, Xypex has standardized a curing procedure that allows the unique development of the chemical reactions with water. Test specimens are stored in a sealed box or cabinet with 1-2 cm of water on the bottom; they are not immersed fully in water. Conditions above the water should be 95% rh and 22 +/- °C. The samples are cured in this manner with the test side (pressurized face) in the water for 28 days. This is followed by a drying cycle in the lab air for 28 days. This simulates a period of drying expected during construction and allows certain types of chemical reactions to take place. The samples are then returned to the sealed curing chamber for a further 34 days for another cycle of wet conditions. They are then ready to test in a more completely cured state at age of 90 days since casting.

Closing Remarks

The Table 9 water absorption standard in EN 934-2 as it stands is not appropriate for the evaluation of truly water-proof concrete. Absorption tests do not accurately reflect the performance of concrete admixtures designed for resistance to hydrostatic pressure. The Deutsches Institut für Bautechnik (DIBt) and the Reseach & Development Group of Xypex Chemical Corporation have developed an alternative approach resulting in the new EAD 260026-00-0301 "Waterproofing Admixture for Concrete". Xypex then completed all the necessary testing requirements which included EN 12390-8 test. Xypex Admix C-1000 NF is now the first product to complete the European Technical Assessment (ETA) according to this new EAD.

Xypex Admix C-1000 NF is, at the time of publishing, the only admixture product approved in Europe for resisting hydrostatic pressure.

