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Shrinkage of Water Repellent Treated Cement-Based Materials

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Abstract

Different types of cement-based mortars have been prepared and then impregnated with liquid silane. Water desorption and adsorption branches of the sorption isotherm have been measured on both untreated and water repellent mortar specimens. It was found that water repellent treatment prevents filling of capillary pores by adsorption. The pore walls of capillary pores are covered by a film of silicon resin. At relative humidity close to 100 % a small amount of water can be stored in coarse capillary pores. Nano-pores in the gel built up by the hydration products, however, can not be treated in the conventional way because of geometrical mismatch of pore size and silane molecules. Shrinkage of untreated and water repellent mortar is practically identical. This means that capillary condensed water has little, if any, influence on hygral length changes of cement-based materials. These findings are in good agreement with predictions of the Munich model. According to this model shrinkage is controlled by the colloidal interaction between water and the gel particles of the hydration products. This interaction is at the origin of a disjoining pressure which depends on the relative humidity of the surrounding air.

1 Introduction

Water repellent treatment of a porous material such as mortar or concrete changes the usual interaction between the porous system and the humidity of the environment substantially. Quite different aims can be achieved by surface treatment of porous building materials with a water repellent agent. As capillary suction is reduced considerably uptake of water and salt solutions can be minimised. Frost resistance for instance can be enhanced considerably. If the penetration depth of the water repellent agent is sufficient a chloride barrier can be built up by water repellent treatment [1].

Obviously the average moisture content of a structural element of reinforced concrete in hygral equilibrium with a given climate can be reduced by water repellent surface treatment. This has been studied by numerous authors in the past (see for instance [2, 3]). The influence of water repellent treatment on shrinkage and swelling, however, has widely been neglected so far.

There are at least two reasons why we should know more about hygral length changes after water repellent treatment. (1) If shrinkage is reduced to the same extent as moisture content, high shear stresses have to be expected in the interface between the water repellent treated and the untreated zones in mortar or concrete. These stresses may create cracks and finally may lead to spalling off of the surface near zone. This process has been used frequently to explain damage observed on natural stone monuments after treatment with water repellent agents. (2) Water repellent treatment changes the distribution of the absorbed water in a characteristic way. Reliable experimental results may allow us to better understand shrinkage mechanisms and to verify existing hypotheses on the origin of shrinkage.

In this contribution results of test series to study water sorption and shrinkage of water repellent treated and untreated mortar specimens shall be presented.

2 Experimental

2.1 Adsorption and desorption

The sorption isotherm describes well the hygral interaction between a porous material and the surrounding air with respect to variations of relative humidity. The time to reach hygral equilibrium of cement-based materials is comparatively long and it is well known that there is a marked hysteresis between the adsorption and the desorption branch. Therefore thin slices of mortar with a thickness of 5 mm and a water-cement ratio of 0.45 have been prepared. Half of the mortar specimens have been impregnated with silane. Part of the water repellent and of the untreated specimens was dried at 105 C and the remaining part has been stored in water. Then the dry mortar slices have been placed in climate boxes with constant relative humidity until they had reached hygral equilibrium. The relative humidity has been kept constant by saturated salt solutions. Mortar specimens were placed stepwise in boxes with increasing relative humidity up to 100 %. The equilibrium moisture content has been measured at each fixed relative humidity by weighing the samples. In this way the adsorption branch of the sorption isotherm has been determined. The water saturated samples were placed in boxes with stepwise lowering the relative humidity. Again the equilibrium water content has been determined by weighing and in this way the desorption branch of the sorption isotherm has been obtained [4].

For a second test series two types of mortar have been prepared. The water-cement ratio has been chosen to be 0.4 and 0.5 and river sand with a maximum aggregate size of 4 mm has been used [5]. All mortar slices have been preconditioned at RH = 60 %. Then half of the slices have been impregnated with silane. After polymerisation they were all placed in a climate box with RH = 96 % until hygral equilibrium had been reached. Finally the relative humidity has been lowered stepwise down to RH = 23 %. At each chosen relative humidity the specimens were kept until hygral equilibrium had been reached.

2.2 Shrinkage and swelling

In order to study the influence of water repellent treatment on shrinkage and swelling five different types of mortar have been prepared. Cubes with an edge length of 150 mm have been cast with each type of mortar. The water-cement ratio and the cement content of the mortars are given in Table 1. River sand with a maximum aggregate size of 4 mm has been used. From

Table 1: Water-cement ratio and cement content of the five different types of mortar

Notation	**Water cement ratio**	**Cement content kg/m^3**
A	0.4	500
B1	0.5	400
B2	0.5	500
B3	0.5	600
C	0.6	500

each cube 12 cores with a diameter of 20 mm have been drilled. At the centre of both circular ends of the cylinders metallic measuring sockets have been glued. In this way weight and length changes of the cylinders while exposed to air with different relative humidity could be followed.

All cylinders were stored in water until an age of seven days. Then they were transferred to a climate box in which a relative humidity of 60 % was kept constant. Water loss and the length change have been measured as function of the drying time. After 40 days half of the cylinders were placed in liquid silane (isobutyltrietoxysilane) for 24 hours. This period was sufficient to impregnate all cylinders completely with silane. After a total drying time of 100 days all cylinders have been placed in another climate box with a relative humidity of approximately 100 %. During this last step swelling and water uptake of both water repellent and untreated mortar samples have been measured.

The hygral length change has also been measured on the water repellent and untreated specimens of the second test series (W/V = 0.4 and 0.5) which were dried stepwise from RH = 96% down to RH = 23 %.

3 Results and discussion

3.1 Adsorption and desorption

The sorption isotherms of the water repellent and untreated mortar with W/C = 0.45 are shown in Fig. 1 (see also [4, 6]). It can be clearly seen that the water repellent mortar absorbs less water than the untreated mortar. The difference is more pronounced at high relative humidity. Above RH = 55 % water uptake of cement-based materials is essentially due to capillary condensation. Quite obviously capillaries can not be filled with water by sorption after water repellent treatment. At a relative humidity close to 100 % a comparatively small amount of water can be taken up in coarse pores. The surface treatment which has been applied here most probably does not prevent water ingress in coarse pores totally. We may imagine that the silicon resin film has a limited range of efficiency in a porous system and water droplets can be formed sufficiently far away of a water repellent surface.

The desorption branch of mortar specimens with W/C = 0.4 and W/C = 0.5 after water repellent surface treatment and in the untreated state has also been determined experimentally. Results are shown in Fig. 2. It is quite obvious that in agreement with results shown in Fig. 1 water repellent mortar looses little water when drying from RH = 96 % to RH = 55 % takes place. If drying is continued, however, to even lower relative humidity, for instance down to RH = 23 %, water loss of water repellent and untreated mortar is

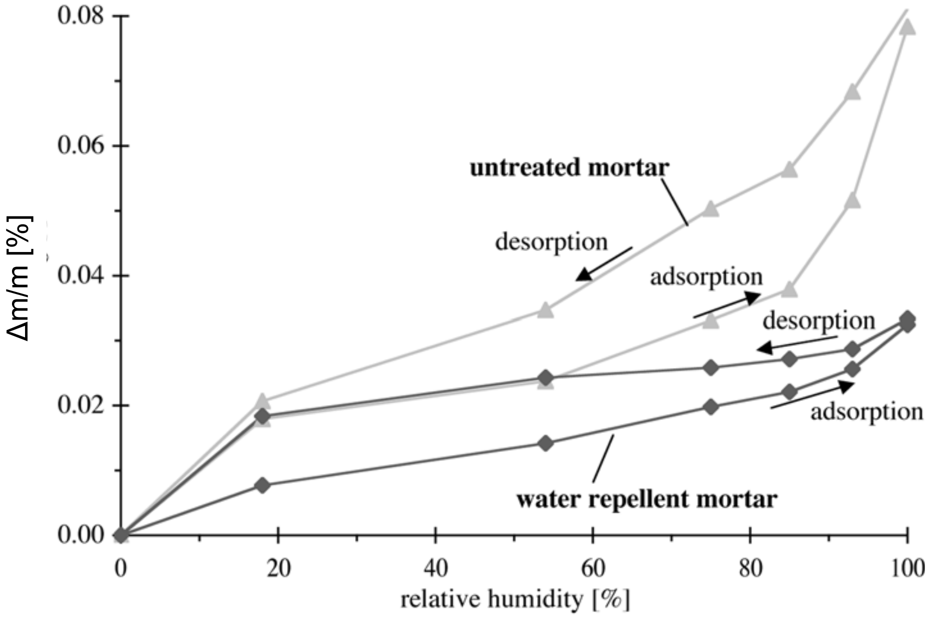

Figure 1: Sorption isotherms of water repellent and untreated cement mortar with W/C = 0.45

nearly the same. A major difference of the desorption branches of the sorption isotherm of treated and untreated mortar is observed in the region of capillary condensation. We may conclude that water repellent treatment essentially prevents capillary condensation of cement-based materials. This fact is also at the origin of the low hygral diffusion coefficient of water repellent mortar and concrete [7].

3.2 Shrinkage and swelling

Five different mortars (see Table 1), water repellent treated and untreated, have been dried to equilibrium with RH = 60 %. Then they have been placed in a climate box with relative humidity close to 100 %. The water uptake and swelling have been measured until equilibrium had been reached [4]. Results are shown in Fig. 3 and Fig 4. As can be seen the water uptake of water repellent treated mortar is small compared to the uptake of the untreated companion specimens. This is in agreement with results shown in Fig. 1. The water uptake of the untreated mortar increases with increasing W/C and at the same W/C it increases with increasing cement content. This is a clear indication again, that in the high humidity range capillary pores of untreated cement-based materials are getting filled with water by capillary

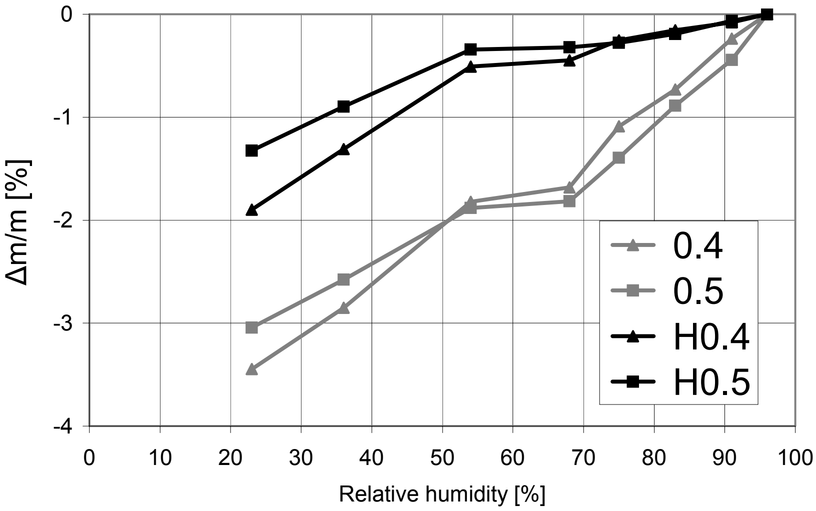

Figure 2: Desorption branch of the sorption isotherm of water repellent (H) and untreated mortar. Mortar specimens with W/C = 0.4 and 0.5 have been tested

condensation. In the case of water repellent mortar, however, the capillary space has a negligible influence on the amount of adsorbed water. Most of the water repellent treated capillaries can not be filled with water. The interfacial energy between the coated pore walls and the water prevents capillary condensation. In very large pores a comparatively small amount of water can still be stored.

Swelling of mortar as observed on the same mortar cylinders is shown in Fig. 4. At a first glance it may or ought to be surprising for many to see that swelling is nearly independent on the circumstance if capillary pores are being filled with water or not. From this observation we may conclude, however, that capillary adsorbed water and capillary pressure have very limited influence on shrinkage or swelling. The origin of hygral length changes must be explained essentially by the action of disjoining pressure in the nano-pores [8-13]. The disjoining pressure in the cement gel depends on the surrounding relative humidity. Details of this complex interaction will be discussed elsewhere. Water repellent agents can in fact not interact with the nano-pores of the hydration products as silane molecules are geometrically too big ($d_S \approx 2.4$ nm) and their interaction radius is even much bigger [14]. Therefore water repellent treatment has a small effect on shrinkage.

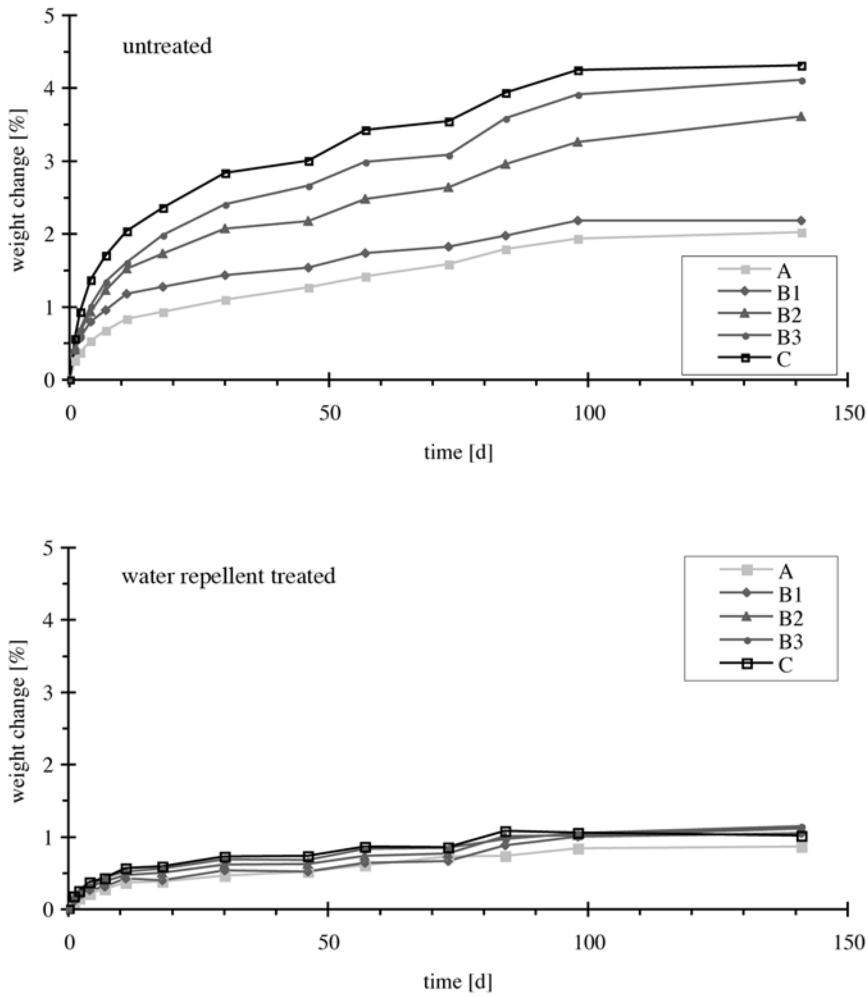

Figure 3: Water uptake of five different types of water repellent and untreated mortar samples, which were first equilibrated with a relative humidity of 60 % and then placed in a climate box with a relative humidity close to 100 %

In Fig. 2 the desorption branches of water repellent and untreated mortar specimens with W/C = 0.4 and W/C = 0.5 are represented. On identical specimens shrinkage has been measured in the same humidity range. Results are shown in Fig. 5. Shrinkage values plotted in Fig. 5 represent hygral length changes in equilibrium with the indicated relative humidity. When drying from RH = 96 % to about RH = 68 % takes place water repellent specimens show slightly less shrinkage than the untreated mortar while

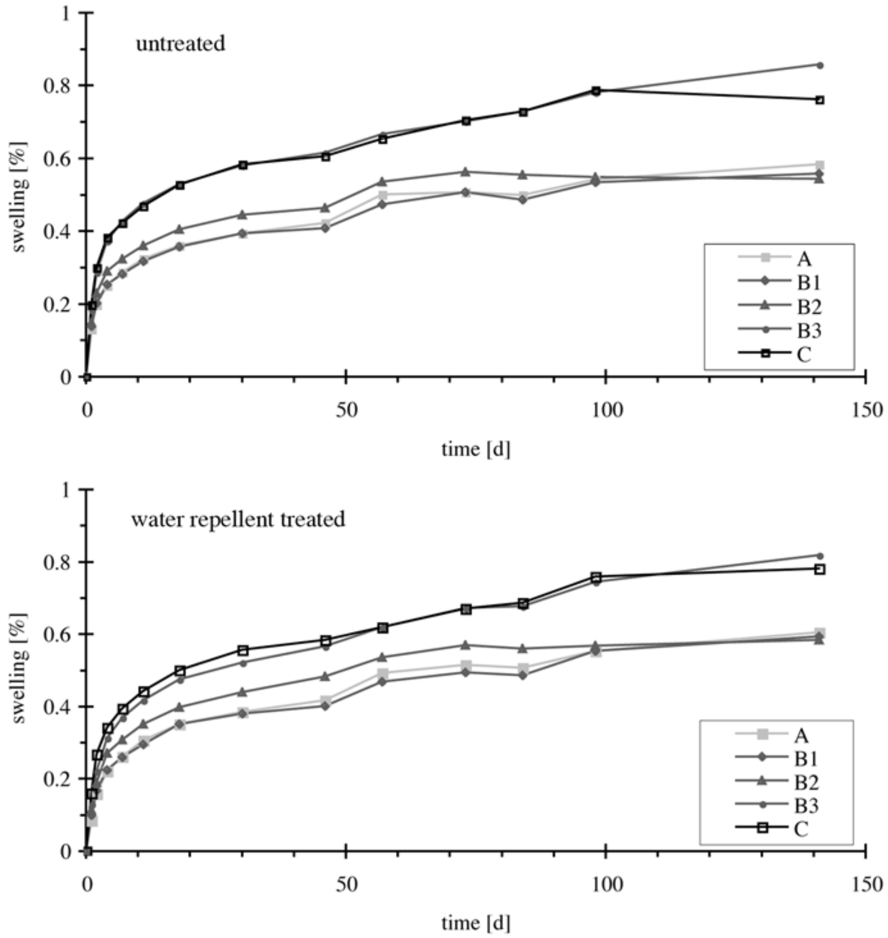

Figure 4: Swelling of water repellent treated and untreated mortar specimens when re-humidified from an equilibrium with RH = 60 % to RH 100%

further drying down to RH = 23 % leads to identical shrinkage of all specimens which have been tested. Within the accuracy of the experiments, however, shrinkage of water repellent and untreated mortar can be considered to be practically the same for both types of mortar.

But most important, it can be seen again that neither removal of water from capillary pores nor capillary condensation into the pore system have a significant influence on hygral length change of cement-based materials. Hygral length change is governed by colloidal interaction between pore water and the gel particles instead. This complex colloidal interaction can be and usually is characterised by a disjoining pressure.

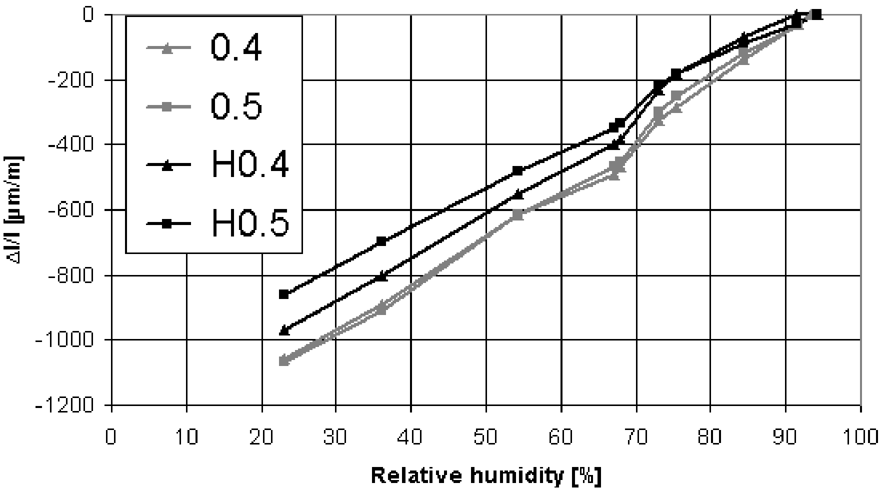

Figure 5: Shrinkage of water repellent (H) and untreated mortar with W/C = 0.4 and 0.5.

4 Conclusions

Results presented in this contribution allow the following conclusions to be drawn:

- Water repellent treatment of cement-based materials prevents capillary condensation. A comparatively small amount of water can be stored in the porous system at relative humidity close to 100 %.
- Nano-pores in the porous system of the hydration products of Portland cement are not water repellent after conventional surface treatment. Molecules of water repellent agents such as silane are too big and can not enter nano-pores because of geometrical incompatibility.
- Shrinkage of cement-based materials is hardly influenced by water repellent treatment. It is practically independent of the amount of capillary condensed water. This is considered to be a strong indication that shrinkage of concrete is controlled by the disjoining pressure in the nano-pores of the hydration products.

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