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Measurement of Humidity in Sandstones Treated with Water Repellent Agents

S. Behre and K. Littmann
Institut für Berufswissenschaften im Bauwesen, Leibniz Universität Hannover, Germany

Abstract

Three different sandstones, typically used in the region of Lower Saxony, were examined. The sandstones are Obernkirchener, Udelfanger sandstone and the Red Wesersandstone. Three different water repellent agents were used with formulations based on silanes and siloxanes either solvent-free or water based systems. After treatment with the hydrophobic agents, the humidity distribution when storing in different relative humidity conditions as well as the drying-out behaviour after wetting behind the water repellent zone was measured. During the experiments the relative humidity was measured in small (Ø 10 mm) drill holes. This technique allows the measurement of the humidity of the stones at different depths over a period of time.

Keywords: sandstone, water repellent treatment, humidity, measuring technique, relative humidity, sorption isotherm

1 Introduction

A question was formulated by stonemasons in Lower Saxony, as to how the humidity distribution of sandstones changes after a hydrophobic treatment. Although there is no real evidence, it is a generalized opinion that the humidity will concentrate behind a water repellent layer after treatment of the stone. Therefore, one aim of the study was to get some data of moisture distribution for the stones used in this region. Three sandstones commonly used in this region were of special interest, these are the Obernkirchener and Udelfanger sandstone as well as the Red Wesersandstone [1]. As the costs of the study had to be kept low, it was decided to use the measurement of relative humidity in a drill hole. Since the publication of this method [2], the equipment available for this type of measurement has become smaller, so that is was possible to use drill holes of only 10 mm in diameter.

2 Experimental

2.1 Natural stones

The natural stones used for this study are two stones from Lower Saxony, the Red Wesersandstone and the Obernkirchener respectively (Figure 1). The third, Udelfanger Sandstone, is from North Rhine-Westphalia. All three are relatively fine-grained but with different type of binding material, as described briefly below:

- Red Wesersandstone: Quartz grains with grain contacts, siliceous binding material with portions of mica, grey to red coloured.
- Obernkirchener sandstone: Quartz grains with grain contacts (84 %), siliceous binding material, grey to yellowish coloured.
- Udelfanger sandstone: Fine quartz grains with clay type of binding material, partly kaolinite, yellow-brown with black ferruginous dots.

Prior to the actual testing, the characteristics of the stones were determined according to the relevant standards [4] (Table 1). Whereas Obernkirchner and Wesersandstone are quite similar in bulk density and porosity, the Udelfanger has a relatively high open porosity with consequently high water uptake and lower density. As expected, the values for water vapour diffusion resistance were approximately opposite to those for porosity, as to be expected.

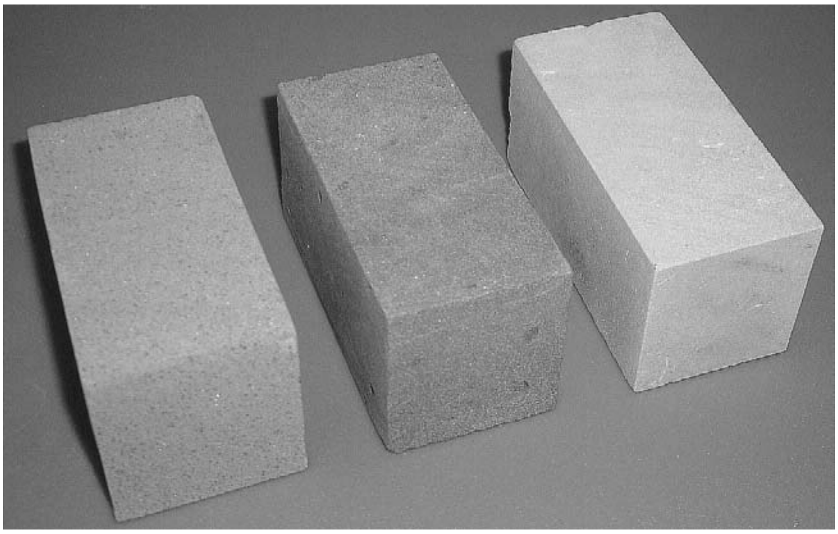

Figure 1: Udelfanger sandstone, Red Wesersandstone and Obernkirchner sandstone (from left to right)

Table 1: Data of Udelfanger Sandstone, Obernkirchner Sandstone and Red Wesersandstone

Type of stone	Bulk density kg/m³	Water up-take (atm. pressure) W%	Porosity V%	Fraction of open pores V%	Water vapour resistance coefficient (μ) -
Red Weser-sandstone	2200	5.18	17	80	44.5
Obernkirchner sandstone	2180	5.21	18	76	37.7
Udelfanger sandstone	2050	8.41	23	94	34.4

2.2 Hydrophobic products

Three different hydrophobic products supplied by two manufacturers were used:

- Product A: Isobutyltriethoxysilane 100 % (Evonik)

- Product B: An oligosiloxane product diluted to 7 w% in an organic solvent (Remmers)
- Product C: A silane/oligosiloxane based cream with a content of active ingredients of 40 w% (Remmers)

2.3 Preparation of stone specimens and treatment

Prismatic stone specimens of 5x5x10 cm^3 were pre-dried and their rectangular sides were sealed with an epoxy as in previous studies [3] (Figure 2). The hydrophobic products were applied on one of the square ends (5x5 cm^2) by brush: three applications wet-on-wet within one hour for products A and B while a layer of 2 mm (according 1.8 kg/m^2) for product C.

The impregnation depth was measured immediately after treatment by cracking the sample and detecting the dark part corresponding to the water repellent layer. The measurements were done again after complete reaction at 23°C / 80% RH (28 days), this time by cracking the samples and applying water drops on the cracked surface. This measurement was repeated after 77 days.

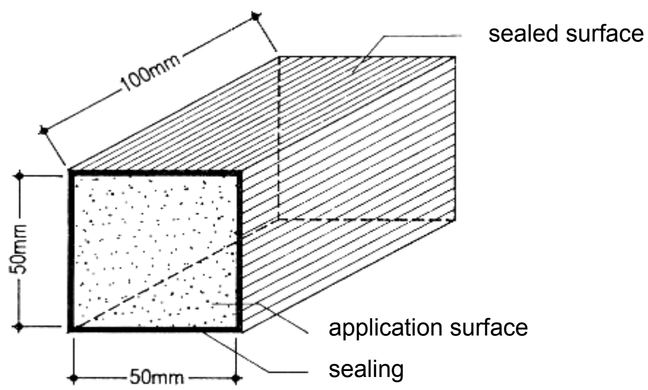

Figure 2: Preparation of the specimens

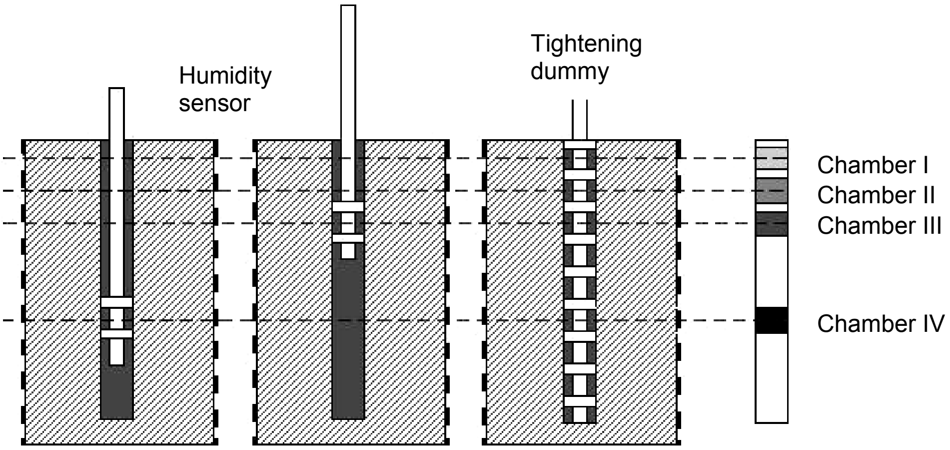

Figure 3: Positions of the humidity sensors and tightening dummy in the drilled hole of the sample

2.4 Humidity measurement

To measure the humidity, a hole of 10 mm diameter was drilled in the specimen in the center of the face that would be on the top. For the case of the treated samples, two thirds would have the hole in the treated face, and one third would have it on the untreated face. After that, the hole was filled with a tightening dummy (Figure 3) to prevent exchange of humidity. The tightening dummy is simply a screw with a series of sealing rings. The measurement itself was done with capacitive working humidity sensors from Ahlborn, Hamburg, coupled with a data recording device Almemo by the same company. The sensors have a diameter of 6 mm which allowed the use of sealers to build up a small chamber within the bore hole. By this, the humidity in different stone depths could be measured. A stable humidity was reached within approximately one minute. The relative humidity in the drill hole may be related to the humidity of the stone as described by Riecken and Pleyers [2]. As a reference, sorption isotherms of all stones were measured between 50 and 90 % RH.

The depths of the measuring chambers relative to the top surface of the stone are: 5 mm (chamber I); 20 mm (chamber II); 35 mm (chamber III); and, 65 mm (chamber IV).

Figure 4 shows some specimens with the applied measuring equipment and tightening dummies.

Figure 4: Measuring equipment and attached specimens. The tightening dummies can be seen on the lid of the chambers

2.5 Specimen exposure system

Treated and untreated specimens were submitted to a moisture flux according to the following conditions :

1. 100 % RH at the treated side of the sample and laboratory climate (around 50% RH) at the untreated backside.
2. 100 % RH at the untreated backside of the sample and laboratory climate (around 50% RH) at the treated side
3. The backside of the treated samples is in contact with liquid water to enable capillary water uptake while the RH at the treated side is kept at 50 %.

The specimens were mounted in plastic boxes filled with water (Figure 4) and in such a way, that the treated side (condition 1) or the untreated side (condition 2) was 10 mm above the water level while for condition 3, they were placed 5 mm in water.

All boxes were stored at 50 % RH and weighed daily. When a constant loss of weight was reached, indicating an equilibrium condition, the moisture measurements were carried out as described above.

3 Results

3.1 Impregnation depths

The results of the measurements of the impregnation depths are presented in Table 2. Product A, isobutyltriethoxysilane, was not effective on the Udelfanger Sandstone as no water repellent effect could be detected after 28 and 77 days. In some cases and especially for the cream material, the water repellent effect was much deeper in the stone after 77 than after 28 days. This may be explained by a slow and incomplete polymerization of the silicone compounds.

Table 2: Impregnation depths of the hydrophobic treatments on Udelfanger Sandstone (US), Obernkirchner Sandstone (OS) and Red Wesersandstone (RWS) measured after different days.

Stone	**Material**	**Impregnation depth [mm]**		
		30 min	28 d	77 d
OS	A	6.8	10.8	11.8
	B	4.3	3.3	2.7
	C	0.4	10.0	16.3
RWS	A	6.8	14.8	17.1
	B	3.8	4.0	4.3
	C	1.8	6.8	17.5
US	A	18.5	0.0	0.0
	B	12.3	8.8	8.4
	C	1.0	7.8	15.9

3.2 Humidity measurements

From the impregnation depths determined (Table 2), it can be noticed that measurement chamber I (5 mm) is generally situated in the hydrophobic layer, whereas chambers II, III and IV at respectively 20, 35 and 65 mm from the surface, are located in the untreated zone of the stones.

Humidity measurements on untreated specimens gave the expected results: a constant decrease of humidity from the side conditioned at 100 % RH to the side conditioned at 50 % RH (Figure 5).

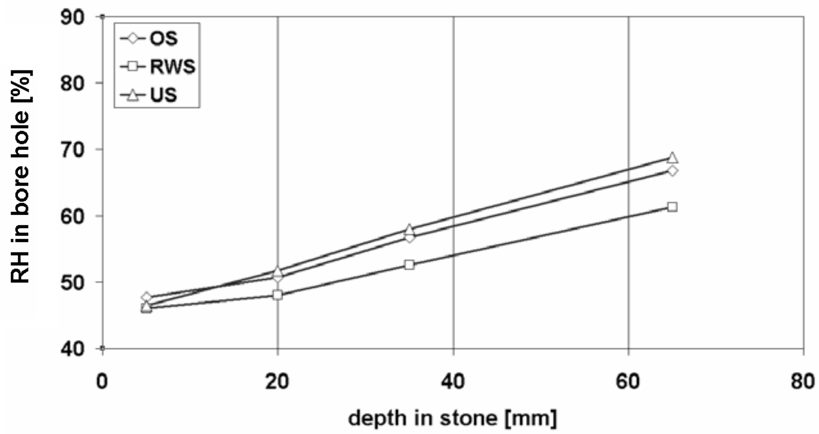

Figure 5: Humidity measurements for the untreated stones. The left side is conditioned at 50 % RH while the right side is at 100 % RH.

The humidity profiles of the untreated stones serve as reference for the evaluation of the humidity measurements for water repellent treated specimens.

When comparing the results of the untreated Red Wesersandstone with those of the treated specimens tested under the same conditions, it can be noticed that the moisture content increases about 15% at the untreated side conditioned at 100% (Figure 6) while near the treated side conditioned at 50%, the increase in the moisture content is far less.

For the other two stones smaller increases of moisture contents were noticed after water repellent treatment.

The results for the reverse climate conditions, 100 % RH at the treated side of the stone while 50 % RH at the untreated backside, are less significant. Figure 7 presents the results for the Udelfanger sandstone. It can be seen, that treatments B and C result in an overall increase of the moisture content of about 5% across the whole stone depth. Treatment A caused no change in moisture content which is in agreement with the low water repellent effect (Table 2). Similar results were obtained for the other two stones, and the moisture content increase which is fairly constant for the whole length of the sample ranges between 5 to 10 % depending mainly on the type of stone while the type of treatment had a lower influence.

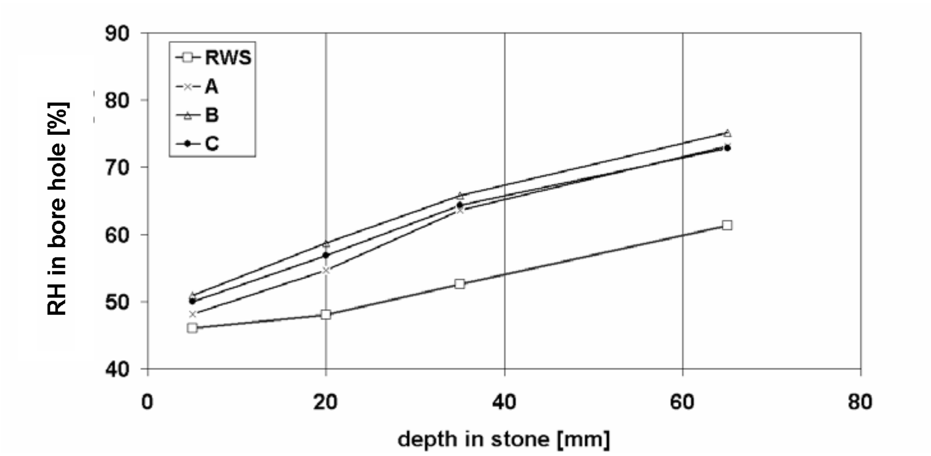

Figure 6: Percentage moisture content at different depths of the untreated Red Wesersandstone (RWS) and those treated with product A, B and C on the side exposed to 50% RH (left); the right side is at 100 % RH.

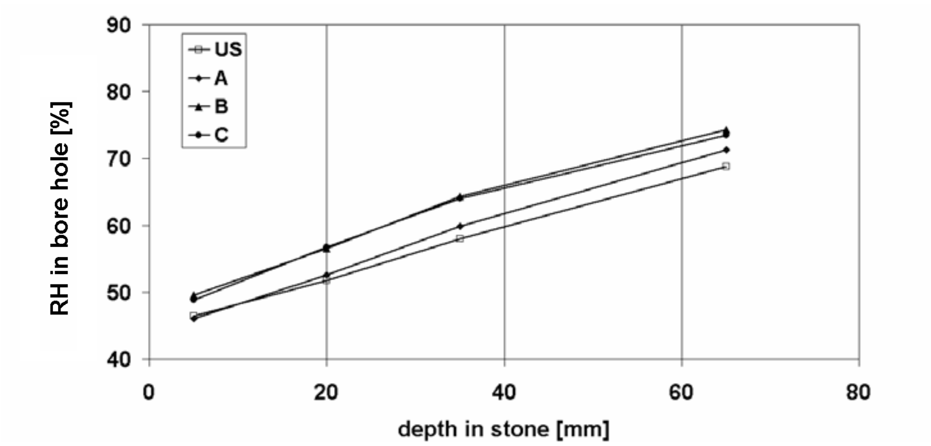

Figure 7: Percentage moisture content at different depths of the untreated Udelfanger sandstone (US) and those treated with product A, B and C on the side exposed to 100% RH (right) ; the left side is conditioned at 50 % RH.

It is important to note that the plots of the measured RH as a function of the distance from the treated surface appear to be practically linear between 30 and 70 % RH.

Finally the results from the capillary water uptake revealed the experimental limitations of the measuring equipment. Over the whole stone depth, relative humidity values above 90 % were measured. In this range of humidity, capacitive sensors of this size show a tendency to drift and do not produce accurate data. Hence, it may only be concluded that the small sensors are not suitable for recording high humidity since this effect was not observed in earlier experiments using equipment fitted for drill holes with a diameter of 16 mm.

4 Conclusions

The equipment for measuring the relative humidity in small bore holes was found to be suitable to get humidity profiles in the depth of the stone. However, the technical equipment is limited to a certain RH measuring range. In case the moisture content is near saturation, no accurate results are obtained using the small sensors.

The study has shown that the studied stones treated with a water repellent and exposed to a high humidity gradient will actually lead to a rise of humidity in the stone as the stonemasons suspected. However, the measured humidity does not lead to water saturation of the pores. The increase in humidity is more dependent on the nature of the stone than on the type of hydrophobic treatment.

References

[1] Grimm, W.: Die Naturwerksteine der Bundesrepublik Deutschland im geologischen Rahmen. In: Bildatlas wichtiger Denkmalgesteine der Bundesrepublik Deutschland. Hrsg. Michael Petzet. Arbeitsheft 50. Bayerisches Landesamt für Denkmalpflege. München. 1990

[2] Riecken, B,; Pleyers, G.: Zerstörungsarme Messung der tiefenabhängigen Feuchte poröser Baustoffe. In: Moderne Verfahren und Geräte zur Materialfeuchtemessung: 9. Feuchtetag 1997 in Weimar; Vorträge und Posterbeiträge, 17./18. September 1997

[3] Littmann, K. ; Riecken, B. ; Sasse, H.R.: Steinschutzstoffe nach dem Aachener Konzept. Stuttgart: IRB, 1998. - In: Denkmalpflege und Naturwissenschaft Natursteinkonservierung Band II – (1998), (Snethlage, R. (Ed.)), S. 207-226

[4] DIN EN 1936-06 Prüfverfahren für Naturstein – Bestimmung der Reindichte, der Rohdichte, der offenen Porosität und der Gesamtporosität DIN EN ISO 12571-00 Wärme- und feuchtetechnisches Verhalten von Baustoffen und Bauprodukten - Bestimmung der hygroskopischen Sorptionseigenschaften