Influence of the disparities between lab and in-situ application on the penetration depth of a hydrophobic agent

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Abstract. Over the last few years, the application of a hydrophobic agent on a masonry wall has become popular due to its ability to reduce the amount of rain water absorption without changing the facade’s appearance. While the hygric properties of such hydrophobised materials are often investigated, research towards its penetration depth into materials is limited. Additionally, most existing research involves small samples made in a lab rather than masonry walls. This paper therefore focuses on two key differences between lab application and application on an actual masonry wall and their influence on the penetration depth of a hydrophobic agent. As the hydrophobic agent is applied differently on an in-situ masonry wall than on laboratory samples, the method of hydrophobisation is investigated first. It is shown that the penetration depth varies significantly for different methods of hydrophobisation as well as within single samples. Secondly, the existing research often targets separate brick or mortar samples rather than full-scale masonry walls. Therefore, several experimental methods are used to quantify the penetration depth in a masonry wall. From these experiments, it is shown that the penetration depth is not only variable throughout this wall, but within separate bricks or mortar joints as well.

1. Introduction
Moisture control in the built environment has always been an important topic of research. Not only does a proper hygrothermal design contribute to a sustainable and durable building envelope, it is also crucial for maintaining a healthy and comfortable interior climate. Typically, the primary moisture source for building facades is wind-driven rain. To limit the rain water absorption, shielding of the facade with an overhang or an external render is often applied. In case of preservation-worthy facades however, more invisible solutions are required. To that aim, the application of a hydrophobic agent has gained much popularity in recent years [1].

When a hydrophobic agent is applied on a building material, it is transported via the material’s pores, hereby rendering them hydrophobic, up until its final penetration depth. Ideally, the hydrophobised zone has a severely reduced liquid water permeability while its vapour permeability remains unaltered to not hinder the drying of water towards the outside environment. The various hydrophobic agents however interact differently with a porous material and the efficiency of the application heavily depends on the compatibility between the hydrophobic agent and the building material [2-3]. Whereas the liquid and vapour permeability of hydrophobised materials are extensively researched, the penetration depth of the hydrophobic agent remains a somewhat open question.

Several techniques can be employed to measure the penetration depth of a hydrophobic agent in building materials. Some are simple, robust methods such as the visual inspection of the (dry)
hydrophobic zone after wetting of the material’s surface [2-3], others are more elaborate like tracking the water inside the material during a water uptake test (using gamma ray attenuation [4], nuclear magnetic resonance [5] or X-ray computed tomography [6]) to locate the (dry) hydrophobic zone. Most of the existing studies however perform experiments on small samples prepared in a lab environment, which causes them to differ from in-situ masonry walls in two distinct ways. Firstly, the samples made in the lab are often hydrophobised by capillary absorption of the hydrophobic agent, whereas a masonry wall is typically hydrophobised by (repeatedly) spraying the hydrophobic agent on the facade. Secondly, most research is centered around separate bricks and mortar joints rather than a masonry wall hereby ignoring the interaction between these elements. The influence of the method of hydrophobisation on the penetration depth of a hydrophobic agent and the penetration depth of a hydrophobic agent into a masonry wall are respectively investigated in section 2. and section 3. of this paper.

2. Influence of the method of hydrophobisation on the penetration depth

The experiments in this part of the paper investigate the penetration of a hydrophobic agent into bricks in function of their method of hydrophobisation. For this purpose, EC HAMSTAD bricks [7] are hydrophobised with an emulsion containing 10 vol% hydrophobic agent (SILRES BS SMK 2100 of Wacker) and 90 vol% water. This is done by bringing the smallest surface of the samples, which measure 4 x 4 x 8 cm³ (width x length x height), into contact with the hydrophobic agent emulsion. A lab environment is mimicked by allowing these samples to capillarily absorb the hydrophobic agent emulsion for a fixed period of time, while the (repeated) spraying applied in-situ is replicated by dividing this time period in 6 intervals with a waiting period in between, where the samples are taken out of the emulsion; all investigated combinations are listed in Table 1. For every combination five samples are tested. By assuming that the emulsion is absorbed exactly like water, it is predicted that absorption times of 60 s and 9 min 30 s result in approximately 10 mm and 30 mm thick hydrophobic zones where the brick is capillarily filled with the hydrophobic agent emulsion.

| Total absorption time | Number of intervals | Waiting time between intervals |
|-----------------------|--------------------|-------------------------------|
| 60 s                  | 1 interval of 60 s | No waiting time               |
| 60 s                  | 6 intervals of 10 s| 3 min                         |
| 60 s                  | 6 intervals of 10 s| 10 min                        |
| 9 min 30 s            | 1 interval of 9 min 30 s | No waiting time |
| 9 min 30 s            | 6 intervals of 1 min 35 s | 3 min                   |
| 9 min 30 s            | 6 intervals of 1 min 35 s | 10 min                   |

Table 1. Different methods of hydrophobisation investigated in this study.

After the hydrophobisation process is finished, the samples are stored in the lab (ca. 23°C and 50% RH) for a minimum of two weeks to allow all chemical processes to be completed. After these two weeks, the hydrophilic side of the samples is brought into contact with water. The waterfront progresses in the sample until it is stopped by the hydrophobic zone at which point the dry zone coincides with the hydrophobic zone as shown by Figure 1. Since this allows measuring the penetration depth on the 4 outside surfaces of the samples but not in the center, the samples are then cut in half along their length after which the same measuring technique is used to quantify the ‘internal’ penetration depth on the cut surface. The penetration depth on the 4 outside surfaces as well as on the cut surface is shown on Figure 2. Because the penetration depth on the outside surfaces is more variable than inside the sample, the ‘internal’ penetration depth is used to compare different samples.
Figure 1. Progress of a water uptake test through the hydrophilic side of a brick.

Figure 2. The penetration depth (in mm) on the 4 outside surfaces as well as on the internal cut surface of a sample.

Figure 3 shows the internal penetration depth measured on the cut surface of the samples in function of the different methods of hydrophobisation listed in Table 1. In this figure WT indicates the waiting time between the application of different intervals.

The total absorption times of 60 s and 9 min 30 s were calculated theoretically to result in hydrophobic zones of 10 mm and 30 mm thick. The results in Figure 3 however diverge significantly from these theoretical penetration depths. Most probably this is a result of the (false) assumption that the hydrophobic agent emulsion is transported through the material exactly like water.

The penetration depth in Figure 3 appears to increase when the hydrophobic agent is applied in multiple intervals. For total absorption times of 60 s and 9 min 30 s, the average penetration depth increases respectively from 10.4 mm to 14-15 mm and from 16.2 mm to 19-21 mm upon applying the hydrophobic agent in multiple intervals. It should be noted however that the variation across the 5 tested samples per method of hydrophobisation is substantial. Estimating the penetration depth inside a sample for a given method of hydrophobisation is therefore difficult. Furthermore, the waiting time of either 3 min or 10 min seems to have little to no influence on the results.

Figure 3. Internal penetration depth in function of the method of hydrophobisation.

The most important conclusion of this section is that the penetration depth is difficult to determine both theoretically as well as experimentally. Not only is the hydrophobic zone irregular within single samples as shown on Figure 2, it also varies for different methods of hydrophobisation. Applying the hydrophobic agent by capillary absorption in the lab or by repeatedly spraying the facade will therefore result in different penetration depths. At the moment it is difficult to formulate the underlying reason as research concerning the transport of hydrophobic agent emulsion is lacking.
3. Penetration depth of a hydrophobic agent into a masonry wall

While the previous section focuses on the penetration depth in a single brick, here a masonry wall is investigated. For this purpose, a masonry wall, measuring 58 x 40 x 33 cm³ (width x height x thickness), is constructed using Vandersanden Robusta bricks and lime mortar with a composition of 12.5 kg Saint-Astier NHL3.5, 50 kg River sand 0/2 and 10 liters of water. It is subsequently hydrophobised by repeatedly spraying the facade with an emulsion of 10 vol% hydrophobic agent (SILRES BS SMK 2100 of Wacker) and 90 vol% water, finally amounting to 6.9 liter emulsion per m² wall. This applied amount is identical to a different in-situ test set-up described in Vereecken et al. [8]. Theoretically, it would result in a wall of which the first 30 mm of the brick is capillarily filled with the hydrophobic agent emulsion. After the hydrophobisation procedure, the wall is stored in the lab (ca. 23°C and 50% RH) for a minimum of two weeks to allow the chemical processes to be completed. Three different experiments are subsequently carried out and the penetration depth measured in each of them is compared.

3.1. Visual inspection of water droplet absorption

A visual localisation of the hydrophobic zone is performed by sawing the wall in half (perpendicular to the treated surface) and subsequently observing the rate of absorption of water droplets deposited on the cut surface. On a hydrophobic material, droplets display a contact angle of more than 90°, as shown on Figure 4 (c), after which the hydrophobic agent slows down or eliminates their absorption into the wall. The absorption rate is subsequently linked to the local degree of hydrophobicity.

![Figure 4](image.png)

Figure 4. A front view of the masonry wall (a), a side view of the wall after it has been cut in half (b) and the visual inspection of the hydrophobic zone by depositing water droplets on the surface (c).

The water droplets, shown on Figure 4 (c), show the existence of two different hydrophobic zones in the wall. While the fully hydrophobic zone does not allow water droplet absorption, in the partially hydrophobic zone the droplets are absorbed albeit significantly delayed with respect to the untreated hydrophilic material. The results from this visual inspection are summarised by the sketch in Figure 5.

![Figure 5](image.png)

Figure 5. The extent of the fully and partially hydrophobic zones determined by the visual inspection of water droplet absorption.

3.2. X-ray radiography

This part of the report is based on the visualisation of the moisture distribution inside the sample of Figure 6 (a), taken from the masonry wall, where the left side of the sample coincides with the hydrophobised surface. The composite sample, which measures 21 x 12 x 2 cm³, is subjected to a water uptake test through its hydrophilic, untreated side upon which the waterfront progresses through the elements until it is stopped by the hydrophobic zone. After 5 hours, the sample is removed from the water source and the moisture distribution inside the sample is visualized with the help of the X-ray
projection method [9]. The dry part of the sample now coincides with the hydrophobic zone and the penetration depth can be measured.

The result of this X-ray radiography image is shown in Figure 6 (b) where a high moisture content is represented by the blue colour. In the mortar layer, only a fully hydrophobic (red) zone is present in the first 1.5 cm to 2 cm. This small penetration depth can be attributed to the capillary absorption coefficient of lime mortar which is about 4 times smaller than for brick hereby making it more difficult for the hydrophobic agent emulsion to penetrate far into the lime mortar.

The penetration depth in the brick shows more variability. The top brick solely displays a fully hydrophobic zone extending up to 5 cm near the mortar joint and 5.5 cm in the middle. Contrary to this, the bottom brick exhibits both a fully hydrophobic (red) zone as well as a partially hydrophobic (yellow-red) zone of which the extent varies across the brick. While only the fully hydrophobic zone, with a penetration depth of 2-3 cm, is visible near the mortar joint, in the middle of the brick a fully hydrophobic zone with a thickness of 4 cm extends into a partially hydrophobic zone with a thickness of 1 cm. This indicates that the penetration depth not only varies for different elements in a masonry wall, but within one element as well.

Figure 6. Sample subjected to the X-ray projection method (a) and the moisture distribution in the sample derived from this experiment (b).

3.3. Variation of the capillary absorption coefficient (A_{\text{cap}})

For this experiment, the elements indicated on Figure 4 (Mortar 1, 2 and 3 and brick 1 and 2) are extracted from the wall. They are subsequently cut up along their length to result in samples with a thickness of approximately 2 cm. The first sample represents the part of the element located 0-2 cm from the (exterior) hydrophobised surface, the second sample represents the part of the element located 2-4 cm from the (exterior) hydrophobised surface and so on. Subsequently, every small sample is subjected to a capillary absorption test. To minimize water evaporation and water absorption through the side surfaces, they are coated with a grey epoxy paint as shown on Figure 7. From this experiment, the capillary absorption coefficient is determined in function of the position inside the masonry wall. Since A_{\text{cap}} is a measure of the liquid permeability in a material, the reduction with respect to A_{\text{cap}} of the hydrophilic material is correlated with its degree of hydrophobicity.

Figure 7. Test samples taken from a brick and a mortar joint.

The capillary absorption coefficients of the samples from the three mortar layers, indicated on Figure 4, are shown in Figure 8 where they are displayed as a percentage of the capillary absorption coefficient of hydrophilic lime mortar equal to 0.16 kg/m²s^{0.5}. Since A_{\text{cap}} can vary from sample to sample, it is
possible to exceed 100% in both Figure 8 and Figure 9. While three sets of samples were extracted from mortar 1, only one set of samples could be extracted from mortars 2 and 3 due to their brittle behaviour. As a result, no standard deviation could be determined for the latter two.

The samples of mortar 1 and 2 located 0 cm to 2 cm from the (exterior) hydrophobised surface have an almost negligible capillary absorption coefficient. This coincides with the fully hydrophobic zone seen in both the visual inspection and the X-ray projection method. No proof is found for the existence of a partially hydrophobic zone in these mortar joints since the capillary absorption coefficients of the three remaining samples (i.e. 2 cm to 8 cm from the hydrophobic surface) are comparable.

The capillary absorption coefficient in mortar 3 only seems to be lightly affected in the first 2 cm. This could mean that only a small portion of this first sample (e.g. from 0 cm to 0.5 cm from the outside surface) is affected by the hydrophobic agent. A possible explanation is that the vertical orientation of mortar 3 causes the hydrophobic agent to run off more quickly, hereby reducing its absorption of the hydrophobic agent, while the horizontal orientation of the other two mortar joints causes the hydrophobic agent to remain in contact with the mortar joints for a longer period of time. Since only one vertical mortar joint could be investigated however, this remains speculation.

![Figure 8](image.png)

**Figure 8.** The measured capillary absorption coefficient of all 2 cm thick mortar samples.

The measured capillary absorption coefficients of the bricks, expressed in percentage of the\( A_{cap} \) of the hydrophilic brick equal to 0.61 kg/m²s\(^{0.5} \), are shown in Figure 9. Brick 1 displays a fully hydrophobic zone between 0-2 cm from the (exterior) hydrophobised surface and a partially hydrophobic zone from 2-4 cm from the (exterior) hydrophobised surface. However, the individual results of the samples located 2-4 cm from the (exterior) hydrophobised surface vary substantially (the individual results are 14%, 71% and 79% of the original \( A_{cap} \) of hydrophilic brick) indicating that the hydrophobic agent has spread unevenly through brick 1. One of these samples (i.e. the sample where \( A_{cap} = 14\% \)) indicates the existence of a fully hydrophobic zone located 0 cm to 4 cm from the (exterior) hydrophobised surface. Brick 1 therefore possesses a variable fully hydrophobic zone, which is located either from 0 cm to 2 cm or from 0 cm to 4 cm from the (exterior) hydrophobised surface depending on the position in the element. Once again, this proves the variability of the penetration depth within a single element, something that was already seen on the X-ray radiography image.

Brick 2 displays a fully hydrophobic zone located 0-6 cm from the hydrophobised surface and a partially hydrophobic zone from 6-8 cm. The latter however only results in a minor reduction of \( A_{cap} \) which could indicate that the hydrophobic agent has only influenced a portion of the 2 cm thick sample (e.g. from 6-7 cm). Due to the thickness of the samples however, it is only possible to draw conclusions for increments of 2 cm.
3.4. Comparison of different experiments

Table 2 lists the size of the fully and partially hydrophobic zones determined in each experiment. The results of the visual inspection differ significantly from the other techniques which is most probably caused by its subjective nature. For example, since the $A_{\text{cap}}$ of hydrophilic lime mortar is about 4 times smaller than the one of hydrophilic brick, water droplets are absorbed slowly even in hydrophilic mortar. In the visual inspection however this slow absorption might be interpreted as a result of the hydrophobic agent which could explain the observation of a partially hydrophobic zone in the lime mortar.

In general, the other two techniques provide very similar results to each other. The bottom brick in the X-ray radiography image is hydrophobised similarly to brick 1. Both possess a fully hydrophobic zone which varies significantly across the sample and a partially hydrophobic zone with a thickness of 1 cm to 2 cm. The top brick and brick 2 display the other end of the spectrum where the fully hydrophobic zone extends further into the brick (i.e. from 0 cm to 5/6 cm from the surface) and the partially hydrophobic zone is either very faintly present or completely absent.

**Table 2.** The size of the fully and partially hydrophobic zones determined by the different experimental methods.

| Fully hydrophobic zone | Lime mortar |
|------------------------|-------------|
| **Visual inspection**  | Between 0 cm $\rightarrow$ 6 cm and 0 cm $\rightarrow$ 9 cm | 0 cm $\rightarrow$ 3 cm |
| **X-ray radiography**  | Top brick: 0 cm $\rightarrow$ 5/5.5 cm |
|                        | Bottom brick: 0 cm $\rightarrow$ 2/3 cm or 0 cm $\rightarrow$ 4 cm |
| **Variation of $A_{\text{cap}}$** | Brick 1: 0 cm $\rightarrow$ 2 cm or 0 cm $\rightarrow$ 4 cm |
|                        | Brick 2: 0 cm $\rightarrow$ 6 cm |
| Partly hydrophobic zone | Lime mortar |
| **Visual inspection**  | 6 cm $\rightarrow$ 9 cm |
| **X-ray radiography**  | Top brick: Not present |
|                        | Bottom brick: 4 cm $\rightarrow$ 5 cm |
| **Variation of $A_{\text{cap}}$** | Brick 1: 2 cm $\rightarrow$ 4 cm or not present |
|                        | Brick 2: 6 cm $\rightarrow$ 7/8 cm or not present |

![Figure 9. The measured capillary absorption coefficient of all 2 cm thick brick samples.](image-url)
4. Conclusion
The first part of this study has shown that the penetration depth does depend on the method of hydrophobisation and that the hydrophobic zone within a single brick can be highly irregular. However, it should be noted that only a small selection of hydrophobisation methods and only a single hydrophobic agent were tested. Research towards other popular hydrophobisation methods, like the application of the hydrophobic agent with a brush, and other hydrophobic agents is still missing.

In the second part of this study, the penetration depth of the hydrophobic agent in a masonry wall is determined using three experimental methods. While the robust visual inspection proves to be unreliable, the other two techniques provide similar results to each other. Apart from a fully hydrophobic zone where liquid water transport is almost eliminated, a partially hydrophobic zone exists where the influence of the hydrophobic agent is present albeit with a reduced intensity. Additionally, not only does the position of these two zones vary across the masonry wall, it also varies within a single brick or mortar joint making it very difficult to predict the penetration depth in a masonry wall without destructively investigating it. A more in depth study concerning the transport of the hydrophobic agent emulsion on the pore scale is therefore necessary.

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References
[1] Janssen H, Deckers D, Vereecken E, Feng C, Soulios V, Vanek A and Hansen TK 2020 Robust internal thermal insulation of historic buildings: Impact of water repellent agents on hygric properties of porous building materials
[2] Soulios V, de Place Hansen E J, Feng C and Janssen H 2020 Hygric behavior of hydrophobized brick and mortar samples Build. Environ. 176 106843
[3] Hansen TK, Bjarløv SP, Peuhkuri RH and Hansen KK 2018 Performance of hydrophobized historic solid masonry – Experimental approach Constr. Build. Mater. 188 pp 695–708
[4] Fukui K, Iba C and Hokoi S 2017 Moisture behavior inside building materials treated with silane water repellent Energy Procedia 132 pp 735–40
[5] Braun F and Orlowsky J 2020 Non-destructive detection of the efficiency of long-term weathered hydrophobic natural stones using single-sided NMR J. Cult. Herit. 41 pp 51–60
[6] van Besien T, Roels S and Carmeliet J 2004 Interlaboratory comparison of hygric properties of porous building materials Journal of Thermal Env. & Build. Sci. 27 pp 307-25
[7] Vereecken E, Deckers D, Janssen H and Roels S 2020 Field study on hydrophobised internally insulated masonry walls XV Int. Conf. on Durability Of Build. Mater. And Comp. (Barcelona) pp 1-8
[8] Roels S and Carmeliet J 2006 Analysis of moisture flow in porous materials using microfocus X-ray radiography Int. Journal of Heat and Mass Transfer 49 pp 4762-72