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Moisture in screed: a non-destructive multi-sensor approach

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Abstract

Eight different screed types are tested including two different sample heights of 35 and 70 mm. The moisture of the four cement based and four sulphate based screeds are monitored during hydration and evaporation. All samples are stored in a climatic chamber at 23° C and 50 % relative humidity. Embedded sensors like temperature arrays, humidity sensor arrays, and multi-ring electrodes are embedded in the samples to yield a detailed moisture evolution with high depth resolution. Furthermore, nuclear magnetic resonance is used to quantify the water content at different depths. This multi-sensor approach allows a comprehensive monitoring of the moisture and its gradient in the different screed samples. This yields a deeper insight into the hydration, moisture convection, and diffusion processes.

1 INTRODUCTION

In order to determine the moisture content of floor screeds minor destructive testing methods like Darr drying or the Calcium Carbid (CM) method are usually applied. These require small samples, deliver only punctual information and still are proven as not very reliable. Furthermore, this testing is carried out manually and time-consuming. The correct handling and the documentation of the results are on full responsibility of the floor layers. An in-situ validation or an automated tracking of these punctual destructive tests is impossible. In case the screed has not dried enough during flooring, wooden covers might well and deform, or mold grow causes high cost for repair; followed often by legal disputes. Embedded sensors in the screed may overcome these unreliable destructive tests. The measurements are non-destructive, fast, repeatable, and data storage and data security follow up directly.

To define appropriate measurement positions for embedded sensors, the temporal and spatial moisture evolution has to be known. Therefore, sensor systems with detailed depth resolution are required to predict the capillary water convection, mass transfer through the pore system, the hydration process, and finally the evaporation as a diffusive process. Based on these information, an ideal system is independent of the screed type, the sample thickness, and does not need any further calibration.

2 SETUP

In the presented study, eight different screed types are tested with several non-destructive measurement techniques. The focus of this study is the determination of the critical moisture content determining the correct moment for laying floor covers. First, the screed samples, the manufacturing process, and the storage in the climate chamber are described. Then, the used measurement techniques, especially the embedded sensors, are discussed in detail.

2.1 Screed samples and drying

In the current study, four cement based and four calcium sulphate based screeds types are tested. The product names of the different types are listed in table 1. For each screed type, three samples are cast. Two samples are equipped with embedded sensors and tested non-destructively. For them, only the sample height, i.e. of 35 mm and 70 mm, is different. The third sample does not contain any sensors and the moisture content is determined destructively by means of the CM method and Darr drying. During dustructiv testing, only smaller parts are extracted from the sample. Thus, several destructive testes are conducted based on this sample. In total 24 screed samples exist and the inner diameter is 300 mm. The casing is polyvinyl chloride (PVC) sewage pipe with a wall thickness of 7.7 mm. A transparent polyethylene foil is used as bottom material. The foil itself is stapled directly on the PVC pipe and sealed with a polymer based sealant. A floating screed floor construction is simulated by placing the samples on top of styrodur (20 mm), styrofoam (40 mm) and washed out concrete (40 mm).

cement based	calcium sulphate based
Weber.floor 4060	Knauf FE 25 A Tempo
Weber.floor 4065	Knauf FE 50 Largo
Weber.floor 4341	Knauf FE 80 Allegro
Sakret Beton/ Estrich BE	Weber.floor 4490

Table 1: Product names of the used cement and calcium sulphate based screed samples

All screed samples are manufactured during one day. For each screed type, the water/cement ratio recommended by the producer is used. All weights are checked by a high precision balance before mixing. The resulting volume is sufficient to manufacture all three samples in a row. Thus, the material in all three samples is identical. After concreting, the samples are covered and stored over night in the production room (at approx. 22° C). On the following morning, the samples are stored in a ventilated climate chamber at a relative humidity of rH=50% and a temperature T=23° C of the ambient air. Figure 1 depicts the screed samples in the climate chamber. Thin (35 mm high), thick (70 mm high), and destructively tested samples (35 mm high) are visible. The cables coming out of the samples are for energy supply and for communication interfaces of the embedded sensors.

2.2 Measurement techniques

Figure 2 presents the setup of the embedded sensors. The temperature and relative humidity arrays consist of ten individual sensors each. The temperature sensors are MCP9700A three-pin thermistors in surface mounted device (SMD) design. They are installed directly on the board and all are covered by epoxy resin. The measurement principle is a change of the electrical resistance due to temperature variations. In the current setup, the resistance is directly converted in a voltage signal which represents the thermistor output signal. The relative humidity sensors HIH-5031 are also in SMD design. A thermosetting polymeric is sensitive to moisture variations and changes its electrical capacity [2]. The capacity is converted into a voltage output signal. The sensors are mounted on the board and then placed in the orange casing. Thereby, the casing fulfils two tasks. On the one hand, each sensor gets its own, separated housing inside the casing. Thus, any air or moisture convection through the array is disabled. On the other

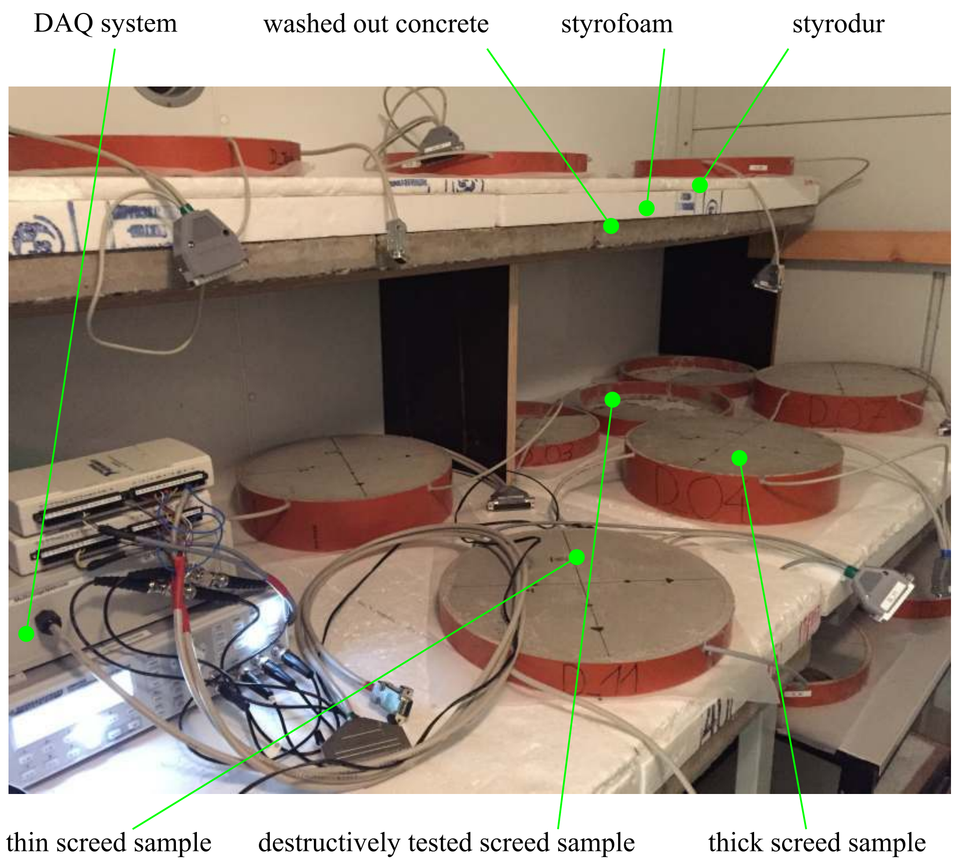

Figure 1: Screed sample storage in the ventilated climate chamber at 50% rH and 23° C

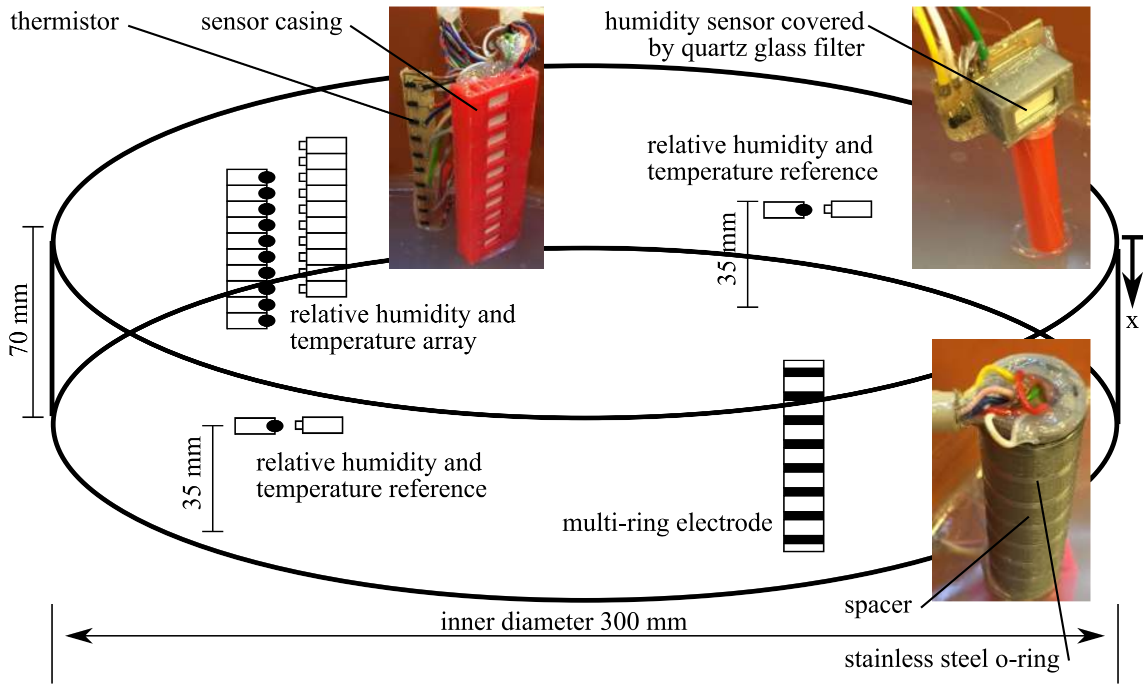

Figure 2: Sketch of the positions of the embedded sensors in the screed sample and photographs of the installed sensors before concreting; the x-coordinate is orientated from the top surface towards the bottom

hand, the casing positions the sensors directly in front of a quartz glass filter. These filters are necessary in order to prevent direct contact of the humidity sensor membrane from water and screed mixture. Direct touching of screed and sensors destruct the probes. The quartz glass filters feature a mean pore size of 10 μm, hence clean water as well as moisture are able to pass the filter immediately [6]. The board and the backside of the casing are also covered with epoxy resin. Each thermistor and each humidity sensor have a distance of 6 mm from each other. There are ten/five sensors along the height of thick/thin samples. After placing the arrays in the screed sample, the lowest thermistor is 3 mm above the bottom and the lowest humidity sensor is 8 mm above the bottom. Hence, the highest temperature and humidity sensors are 57 and 62 mm above the bottom, respectively. This design ensures a clean and closed screed surface and minimises the influence of the embedded sensors themselves. Furthermore, two reference sensors, one temperature and humidity sensor each, at a height of 35 mm are installed in the sample as well to prove symmetry of the screed hydration and moisture evaporation.

The multi-ring electrode (MRE) consists of eight stainless steel o-rings which are separated by spacers. The outer diameter is 19 mm and the inner diameter 16 mm. The o-ring height is 2 mm whereas the spacer heights are 1.7 mm and 6.5 mm for the 35 mm and 70 mm samples respectively. The assumed measurement position is in the middle of two o-rings, hence the lowest one is 7.4 mm and the highest 64.9 mm above the bottom. Powered by an alternating current (AC) signal, the impedance of two o-rings placed next to each other is measured. If the moisture in the screed, which represents the surrounding dielectric material, changes, the resistance and the reactance vary as well. This gives qualitative values of the moisture and the moisture gradient across the height of the samples. In order to derive quantitative values, a calibration function for each screed material is required.

The nuclear magnetic resonance (NMR) spectroscopy is another measurement technique to quantify the moisture content with high spatial resolution across the sample height. Thereby, a strong electromagnetic field generated by the NMR spectroscope aligns atomic nuclei parallel to the generated field. Then, the transversal relaxation time is measured. This time constant represents the time duration from the parallel alignment until the thermal equilibrium is reached. It depends on the strength of the magnetic field and the magnetic properties of the isotope of the atoms.

A high precision balance with a maximum load of 72 kg and an accuracy of 0.1 g is used as reference measurement system. All screed samples are weighed at each measurement day. Thereby, the empty weight of the casing and the embedded sensors are subtracted. These gravimetric measurements give a precise and reliable reference of the averaged water content of each sample. Nevertheless, depth information are not available.

3 DATA ACQUISITION

Two USB-6210 data acquisition systems from “National Instruments” with 16-bit resolution are used to record the data from the embedded sensors. The temperature and humidity sensors are powered via a voltage regulator at 5 V DC. The generated sensor output voltage is recorded with 16 Hz for 10 s. To eliminate the influence of outliers, the median of each sensor is computed. The LCR-bridge HAMEG 8118 from “Rhode und Schwarz” is used to measure the MRE. Prior, the LCR bridge is calibrated in open and short mode to compensate the influence of the wires and the relais card. Thus, all seven MRE measurement positions are measured in a row. The relais card itself possesses a low resistance and capacity to ensure reliable measurements. The measured o-ring pairs are switched via the digital output channels of the data acquisition systems. Two AC frequencies are used for MRE, i.e. 100 Hz and 1000 Hz. 10000 Hz is also tested in preliminary studies but it generates a poor signal to noise ratio [1].

The used NMR system is the NMR-MOUSE from the company Magritek as depicted in figure 3. It is an open NMR sensor equipped with a permanent magnet and water molecules are detected by radiofrequency waves [5]. In the current setup, the amplitude of the T_2 relaxation time is determined by means of the first six spin echoes. To reduce the required measurement time, an approximation via an exponential function is not performed. Instead, the mean value of the second, third, and fourth echo is calculated and represents the used NMR amplitude. This suggested data processing already showed a sufficient robustness in preliminary studies. The proposed setup enables the data acquisition of 10 depth positions in approx. 35 minutes. Thereby, the maximum penetration depth is 25 mm and the different depth positions are reached via traversing the NMR magnet relative to the screed sample. Therefore, the screed samples are measured from the top and bottom side to get maximum information.

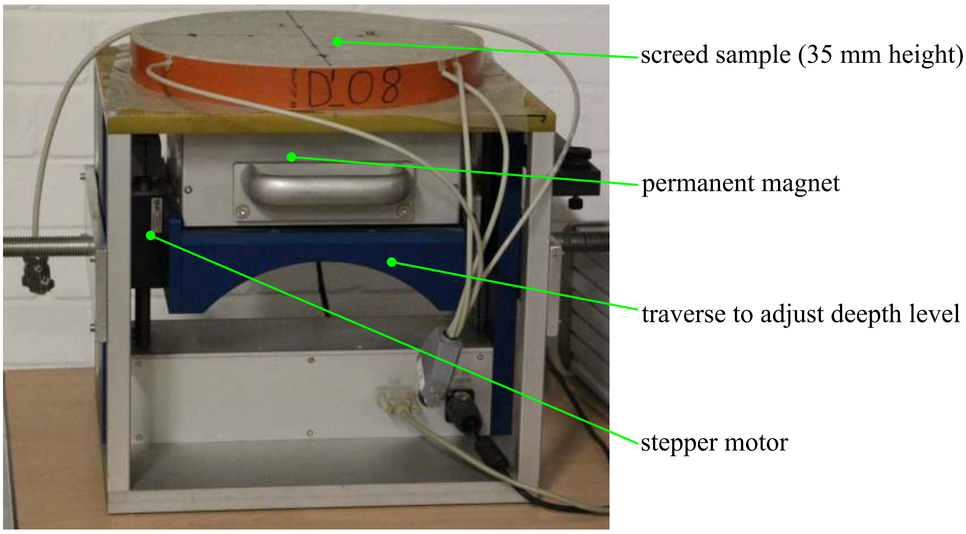

Figure 3: Photograph of the NMR-MOUSE with a thin screed sample measured from the bottom side

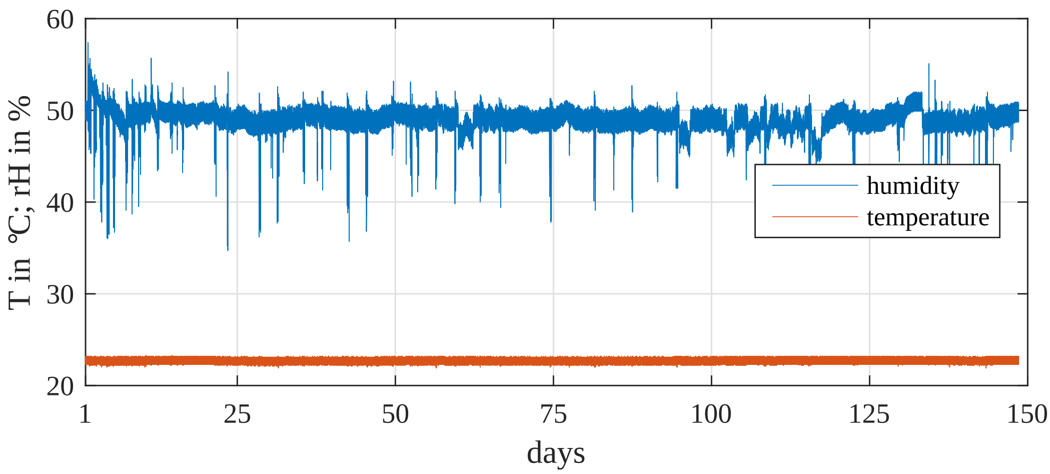

Figure 4: Temporal progress of the ambient humidity (blue line) and temperature (red line) in the climatic chamber

The samples are stored in a self-regulating climatic chamber at rH=50% and T=23° C of the ambient air. Both parameters are recorded every five minutes. Figure 4 yields the corresponding graphs. The mean temperature is 22.69° C with a standard deviation of 0.3. The humidity shows higher variations. The outliers of low values are caused by frequent door opening during measurements. Furthermore, the ambient humidity is slightly increased during the first seven days. This is likely because of the evaporation of the fresh screed samples. However, the mean humidity is 48.85% with a standard deviation of 1.5.

4 RESULTS

Although eight different screed types with two different sample heights are measured, only thin calcium sulphate based screed samples are discussed in the following. Cement based as well as thick calcium sulphate based screed samples do not reach yet the equilibrium moisture content (EMC) at all depth positions. However, the two thin screed samples, Knauf FE 50 Largo and Weber.floor 4490, and the corresponding measurement data of the embedded sensors and the NMR are compared.

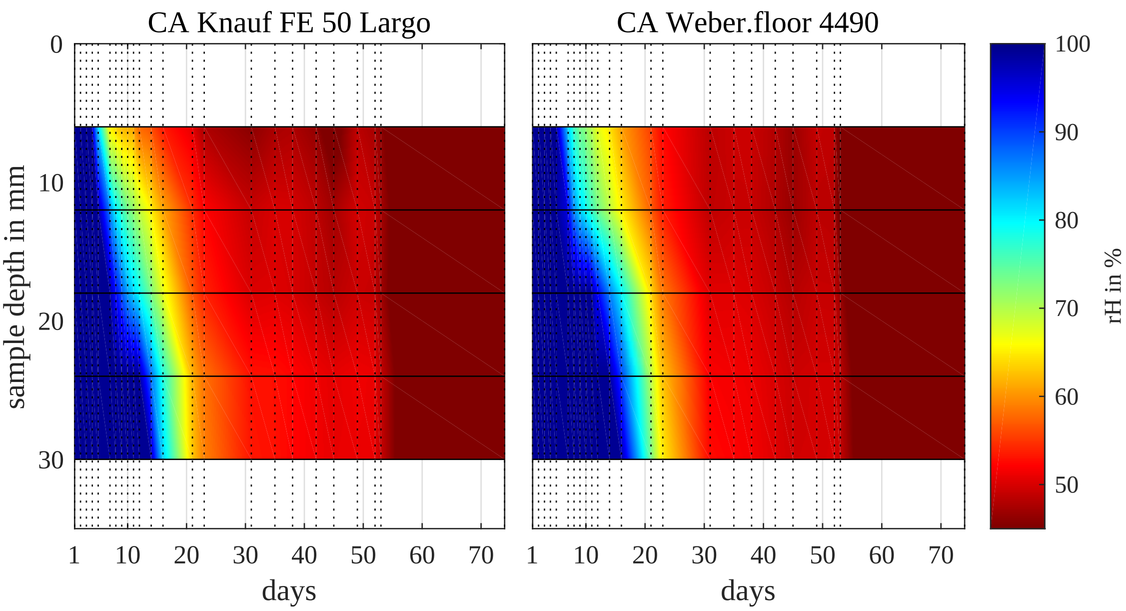

Figure 5: Measured rH in % acroos sample height and over time; the black horizontal lines mark the five measurement positions of the embedded sensors

Figure 5 shows the relative humidity measured by the sensor array during the hydration and evaporation process of the screeds. The advantages of this approach are that the humidity is measured directly and no calibration of the data is required. Hence, this method is independent of the screed types, additional substances, and their material properties. The sample height is 35 mm and the highest humidity sensor is located at 6 mm depth and the lowest one at 30 mm depth. Their distance is 6 mm resulting in five measurement positions in total. These positions are illustrated in figure 5 as black horizontal lines. The abscissa depicts the days after concreting the samples. The thin doted vertical lines mark the measurement days. During the first two weeks, measurements are taken daily and after that, two times per week. Finally, after 53 days, both samples are dried in an oven at 40° C and less then 10% rH until a constant weight is reached.

The coloured surface in these plots represents the rH in %. The corresponding colour bar is shown on the right hand side. The maximum is 100% and the shown minimum is 45%. Apart from that, the relative humidity in the samples after drying in the oven was below 10% (day 73). However, the first week after concreting, all humidity sensors show a humidity of 100%. Thus, saturation is reached and liquid water still exists in the screed pores. After one week, the humidity sensors at 6 mm depth register a reduction of rH. Hence, the corresponding relative humidity (CRH) starts to decrease towards the EMC. After two weeks, the lowest sensor at 30mm leaves saturation as well. Furthermore, both samples reach a constant humidity level and flat moisture gradients across depth after 31 days. Between day 31 and 54, no significant trends are detectable within the given measurement accuracy. In conclusion, both screed types show a similar behaviour. Both samples are completely saturated during the first week and reach a constant EMC after 31 days. Furthermore, the spatial gradients at a distinct moment of time t are comparable in magnitude as well. After 31 days, the humidity of both screed types is identical within the measurement accuracy. The final rel. humidity of 50% corresponds ideally to the ambient humidity of 50% in the climatic chamber.

The humidity sensor array shows consistent and reliable results in space and time. Thus, this new developed measurement system is sensitive to the humidity variations within the required resolution. Thereby, calibration or transformation of the measurement data according to the used material are not needed. The final CRH tends to the ambient air humidity independent of the screed type. Therefore, CRH tracking is considered to be a stable and robust technique. Although a depiction of the humidity over time is intuitive, a presentation over moisture is even more informative. Figure 6 shows the same measurement data as discussed before for the two thin samples. Now, the abscissa represents the moisture (logarithmically scaled) which is determined gravimetrically. Moisture below the solid, vertical magenta line at 0.5 M% is harmless and the screed is ready for flooring. The dashed magenta line is sometimes used for heated screed as limit for flooring. However, at 0.5 M%, only the two near-surface humidity sensors of both samples register a reduction of the humidity. The other three deeper sensors are still in saturation. Furthermore, at the more conservative limit of 0.3 M%, the two deepest sensors are still in saturation. This finding shows that the EMC is not reached at 0.5 M%. This clearly implies that free water in liquid and gaseous phase still exists in the screed sample although the screed is ready for flooring. However, the low level of free water at 0.5 M% does usually not cause damages.

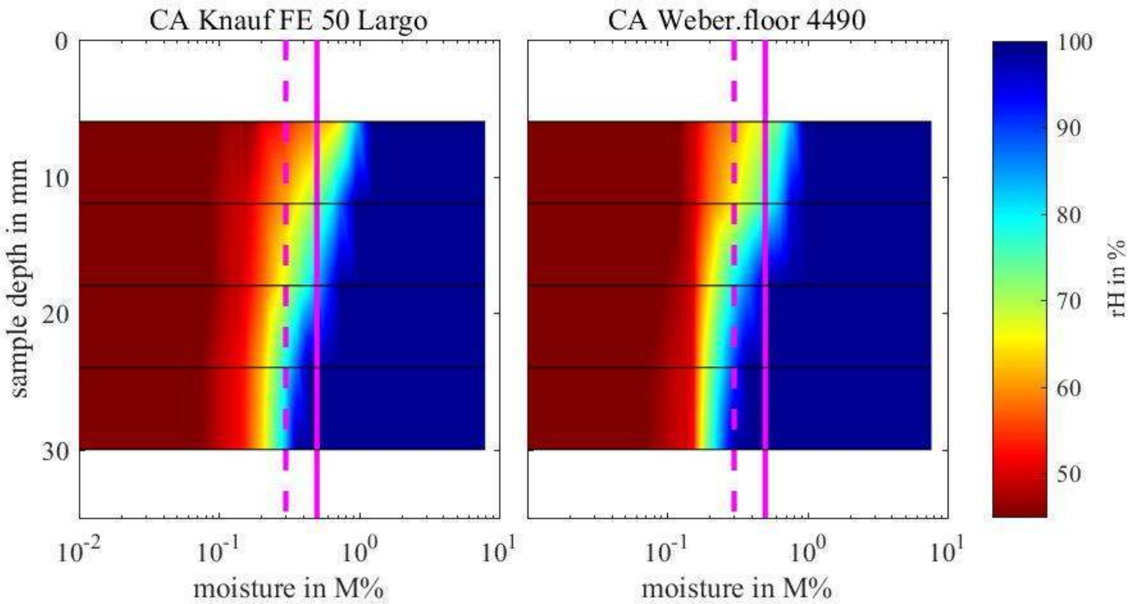

Figure 6: Evolution of the measured rH in % over moisture (logarithmic scaled); solid vertical magenta line: 0.5 M% limit for flooring; dashed vertical magenta line: 0.3 M% limit for flooring on heated screed

As illustrated, the final EMC along the entire depth is reached at approx. 0.15 M%. At this point, no free water evaporates from the sample any more. Only diffusion occurs but does not affect the moisture and humidity equilibrium. Comparing the two numbers, i.e. 0.5 M% for flooring and 0.15 M% for final EMC, it proves that the moisture for flooring is significantly higher then the moisture of EMC. This correlation is also assumed in other studies [7]. Thus, the presented results reveal critical aspects of the EMC-method. For the EMC measurement, first, a hole is drilled to get approx. 100 to 200 g screed material. After crushing and putting it in a bag or small container, the mean CRH is determined [7]. On the one hand, the drill hole depth and the sample extraction influences the mean CRH. If only screed samples from the lower and intermediate level are taken, the CRH-method may be too conservative. As shown in figure 6, the two lowest humidity sensors leave saturation not before 0.3 M%. On the other hand, which averaged CRH level is representative for the real moisture content. In Germany, a mean value of 75% rH is claimed before flooring and 65% rH for flooring on heated screed [7]. Although moisture levels significantly below 0.5 M% are already reached, the CRH-method may measure values above 75% rH, especially if a large amount of test material is taken from the lower and intermediate level.

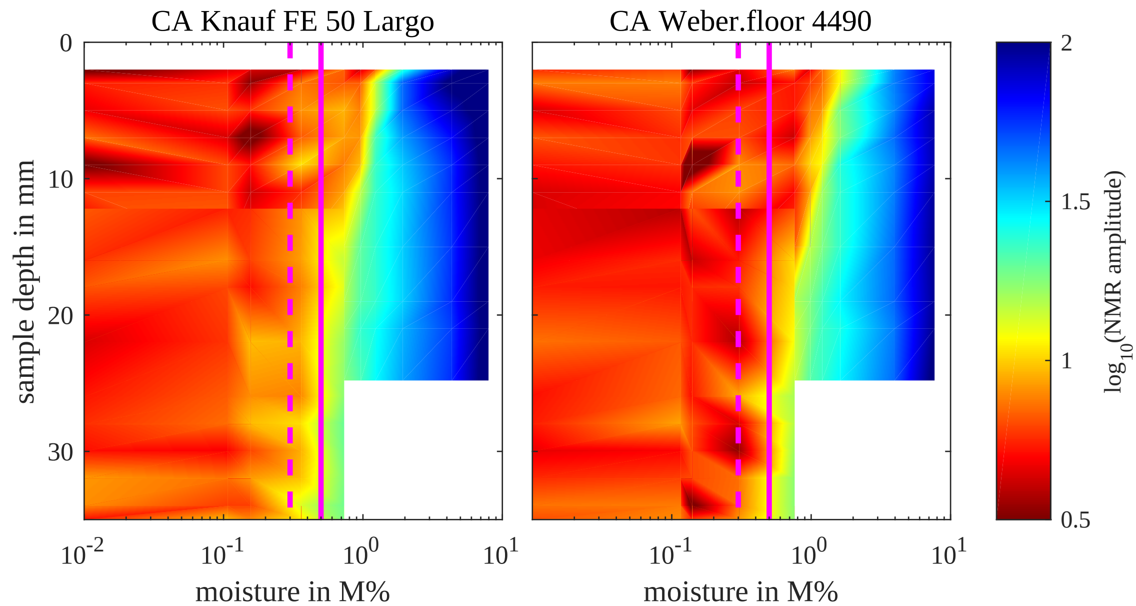

Figure 7: Logarithmic NMR amplitude over moisture (logarithmically scaled) including the flooring limits

To verify the findings of the embedded humidity sensor arrays, NMR measurements are applied as well. The humidity sensors are not able to quantify the moisture above the humidity saturation. In contrast, the NMR quantifies all water in liquid and vapour phase. In general, a linear correlation between the NMR amplitude and the moisture content is assumed. Thus, the effort of calibration is minor and gradients are measured with high spatial resolution. Figure 7 presents the corresponding results. The NMR amplitude over the sample moisture is scaled logarithmically. During the first measurement days, the samples are measured only from the top side. Thus, only moisture information to a maximum depth of 25 mm are available. Below approx. 0.7 M%, the samples are measured from both sides and the moisture along the entire depth is recorded. The NMR generates reliable and consistent results. The NMR is sensitive to moisture levels starting at around 8 M%. The NMR amplitude decreases as the moisture decreases. Below NMR amplitudes of around 6 to 10 or 0.8 to 1 on the colourbar, no significant changes are detectable any more. This corresponds to moisture of approx. 0.5 M%. Hence, the NMR is able to quantify the moisture exactly when the screed is ready for flooring. A separation between 0.3 and 0.5 M% is not possible by means of the NMR.

Although the NMR measures all free water, the measured gradients are similar to the humidity gradients. At a depth of 6 mm, the NMR records low amplitudes the first time at approx.

1 M%. This corresponds to the moment when the first humidity sensor leaves saturation and starts to tend towards the EMC. At a depth of 30 mm, a different behaviour is observed. At around 0.5 M%, the NMR is blind for any changes at all depths although the two lowest humidity sensors still show saturation. Furthermore, humidity sensors at this depth still detect changes up to 0.1 M%. Based on these findings, a linear correlation between the measured free water (by NMR) and the local humidity does not exist. Further investigations are required to disclose the relation between these two parameters in screed.

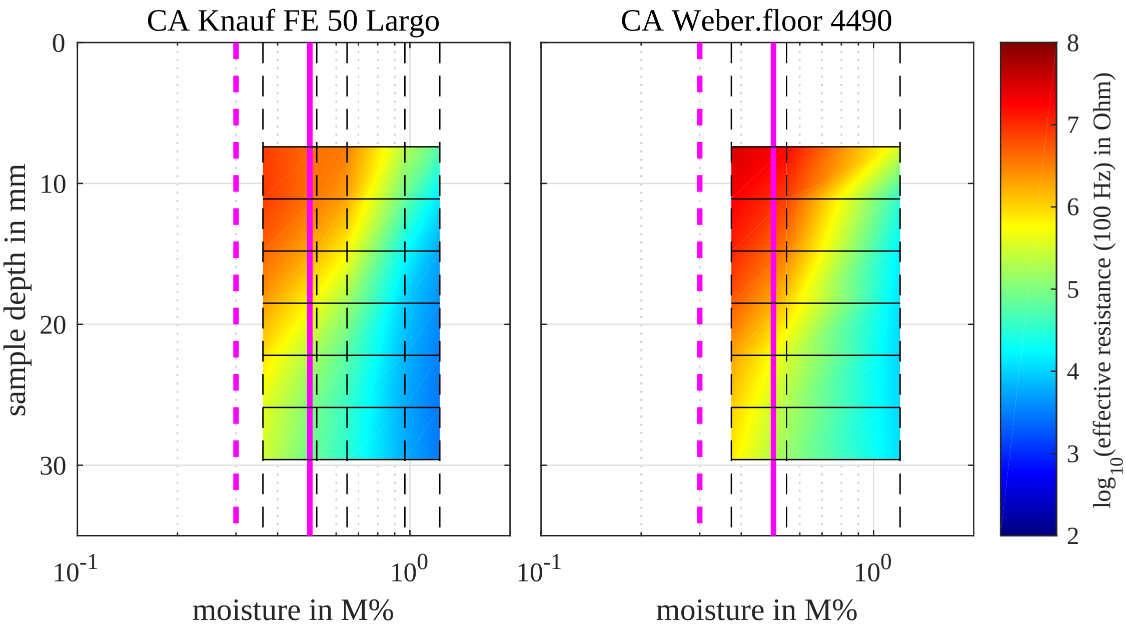

Figure 8: Logarithmic electric effective resistance of the MRE at 100 Hz AC versus moisture content (logarithmically scaled) including the flooring limits

The last discussed measurement technique is the embedded multi-ring electrode. Figure 8 illustrates the measured effective resistance at 100 Hz AC. The resistances are scaled logarithmically. At high moisture level of more then 1 M%, the values are fairly low, i.e. between 100 and 10,000 Ohm. With decreasing moisture, the resistance increases exponentially. Below a moisture content of 0.5 M%, the measured resistances reach values with a magnitude of 10^7 Ohm. This exceeds the measurement range of the used LCR bridge and measurements at lower moisture levels are impossible. Thereby, the high resistance consists of two parts. On the one hand, the electrolytic resistance of the screed itself increases due to less capillary water. This effect may be also measured at the top surface with a Wenner probe for instance [3]. On the other hand, at lower moisture levels, the contact resistance of the embedded electrodes increases significantly as well. Thereby, the contact resistance raises the total effective resistance by several magnitudes [4]. A separation of these two effects is possible via the Archie equation but this approach requires additional parameters of material properties. Furthermore, to transform the measured electric resistance into moisture, a calibration is needed. Thus, only qualitative results are discussed in the following.

The MRE's are very sensitive to moisture changes. Repeated measurements with a time delay of one minute already recognises increased resistances at all positions. Therefore, an almost arbitrary high temporal resolution is possible. The spatial evolution of the qualitative moisture content is similar to the NMR and the humidity sensors. The measured near-surface resistance increases faster compared to higher depths. Above 1 M%, the resistances between 15 and 30 mm have the same magnitude (although a clear trend is detectable). In the range of

0.5 M%, the near-surface resistance reaches already high values. Moisture levels of 0.3 M% cannot be measured any more due to the measurement range of the used LCR bridge. This makes this approach questionable for application in practice. An user cannot judge weather the MRE is not working due to damage or due to a too high resistance.

Beside the resistance, the capacity is measured as well. If the moisture decreases, the capacity decreases as well. At around 1 M%, the capacities are in the range of nanofarad. At lower moisture levels, the capacity magnitude trends to picofarad. However, an optimisation of the MRE geometry may shift the resistance to lower values and the capacity to higher values.

5 CONCLUSION

Eight different screed types with two different sample heights of 35 and 70 mm are tested. The samples are successfully equipped with embedded sensors, i. e. temperature and humidity sensor arrays and MRE. This yields a detailed depth profile of the moisture content. Furthermore, additional NMR measurements generate further moisture depth profiles with high spatial resolution.

The presented measurement systems show different sensitivities. The humidity sensors saturate in the beginning but are able to detect changes down to a moisture content of 0.1 M%. The NMR in the proposed setup quantifies the moisture from high levels down to approx. 0.5 M%. Further changes at lower moisture values vanish due to measurement noise. A longer measurement time may overcome this issue. However, the MRE yields a high temporal resolution. Tiny variations during a time span of 1 minute are detectable. The exponential increase of the resistance generates values of larger than 10 MΩ at a moisture of around 0.5 M%. Furthermore, a calibration is required to transform the MRE values into moisture.

The represented multi-sensor approach yields several major benefits. The humidity sensors show constant values below 0.1 M%. Thus, the resting time until a full equilibrium moisture content is reached is too conservative to use it as an indicator for flooring. The tested calcium sulphate based screed samples are ready for flooring much earlier. Furthermore, the officially recommended values in Germany for the averaged corresponding relative humidity are 75% or 65% for heated screed. As shown, these averaged values are sensitive to depth of the extracted test material. At moisture levels below 0.5 M%, the lower humidity sensors still show saturation.

Consideration of the moisture gradient discloses further consequences. Although the equilibrium of the moisture is achieved within the first 10 mm of the sample, below 25 up to 35 mm, high moisture values are recorded. In this lower region, high NMR amplitudes are measured and the humidity sensors still show saturation which shows that the depth position of the used material of the destructive CRH method has a strong influence of the final result. Hence, a more detailed description of the test material extraction is required to ensure reliable results.

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