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COMPARISON OF NON-DESTRUCTIVE TECHNIQUES FOR ANALYSIS OF THE WATER ABSORBING BEHAVIOR OF STONE

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Abstract

Existing non-destructive techniques for analysis of the water absorbing behavior of stone material have a different nature. This leads to discrepancies in practical application and measuring area, resulting in difficulties when comparing results of different methods. The present study focuses on the comparison of different non-destructive methods in terms of practical application, influence of variable factors and accuracy in relation to the open porosity and the capillary rise measurements. The comparison was based on repetitive measurements and X-ray and neutron radiography of the water absorption by lithotypes with a varying open porosity. The methods under study are the Karsten tube (KT), the contact sponge method (CSM) and the Mirowski pipe (MIR). It can be concluded that KT and CSM have complementary fields of investigation, whereas MIR produces unreliable results due to practical discommodities. The most significant variable factor influencing the measurements is the surface of the contact area. A study of analytical models describing the capillary water absorption as reference for interpretation of results of the different methods is currently ongoing.

Keywords: capillary rise, contact-sponge method, Karsten tube, Mirowski pipe

1. Introduction

Water absorption in porous stone materials is one of the main causes of accelerated deterioration. Consequently the water absorbing behavior (WAB) is a significant factor for the stones' conservation as it determines its propensity to future deterioration. Moreover, changes in the WAB can be an indication of the degree of deterioration or the effectiveness of past treatments, e.g. a water repellent treatment (Svahn, 2006). The WAB can be defined as the behavior in which water is absorbed and retained into the stone by the characteristic pore structure and pore size distribution of each specific lithotype. Hence, analysis of the WAB is a decisive aspect when monitoring deterioration of stone and past conservation treatments, or when selecting a proper conservation treatment. Examples of the most commonly used non-destructive methods for analysis of the WAB are the Karsten tube (KT), contact-sponge method (CSM) and Mirowski pipe (MIR). In this context these methods are considered as non-destructive, because no destructive sampling is required as methods can be applied *in situ*. Even though these methods are based on the same principle (i.e. water is brought in contact with the stone surface and the amount of water absorbed in function of time is recorded), it is observed that the results of the different methods are related, but are difficult to compare since those methods are developed with different specific aims (e.g. monitoring the breakthrough through hydrophobic barriers), leading to differences in design and practical application. Moreover, at present none of these methods is standardised, leading to different interpretations of the application procedure by the operators, thwarting the comparison of results. In the last few decades, different (inter)national committees have set up numerous programmes to monitor stone deterioration and evaluate the effectiveness of protective treatments (Nwaubani & Dumbelton, 2001; Herinckx *et al.*, 2012). These programmes confirm the importance of the WAB and endorse the lack of adequate standardised non-destructive measuring techniques for the WAB. Constant attempts on developing adaptations on the already existing techniques or new non-destructive techniques (Drdácký *et al.*, 2011; Vallet, 1999) also illustrate this deficit. This paper presents a part of a larger research which focuses on the comparison of non-destructive techniques for analysis of the WAB, with the aim of developing a methodology for application of the different techniques. Firstly, this paper will focus on the comparison of the methods in terms of precision, possibilities and/or limitations due to the practical application. Secondly, it emphasizes on the relation between the results of the different methods.

2. Materials and methods

2.1. Conditions and materials tested

As non-destructive methods are developed for use in situations where destructive sampling is not an option, most investigations take place *in situ*, where variable factors such as climatic conditions, biological growth can influence the measurement. To exclude these variables, all methods have been scrutinized in laboratory (average temperature of 20-22°C and 55% RH) on stone samples in a first stage. Only distilled water was used. For the validation of the methods a selection of seven porous lime- and sandstones (Table 1) with a varying open porosity was made, based on lithotypes which are widely used for architectural and sculptural purposes in Belgium. The real density and open porosity have been determined by the Belgian Building Research Institute (BBRI, 1970; BBRI, 1997). For each lithotype the samples (7 x 7 x 2 cm^3) were cut from a bigger stone slab to obtain samples with technical properties as similar as possible. After cutting, the samples were dried in an oven at 60 ± 5°C until the constant dry mass (CDM) was reached, according to UNI/NORMAL 10859. To maintain the CDM the samples were kept in a desiccator when cooling down to room temperature. Weight measurements were made with a balance with a precision of 0.001g.

Lithotype	Symbol	Real density (kg/m³)	Open porosity (%)
Massangis Roche Claire	MC	2427 ± 5	10.7 ± 0.2
Massangis Roche Jaune	MJ	2400 ± 120	10.5 ± 0.9
Senonville	S	2388 ± 7	10.6 ± 0.2
Valanges	V	2300 ± 100	14 ± 2
Magny Doré	M	2290 ± 20	15.6 ± 0.8
Tercé	T	2060 ± 75	24 ± 5
Estaillades	E	1920 ± 20	29.3 ± 0.6

Table 1: Average values of the real density and open porosity of the selected lithotypes

2.2. Methods

2.2.1. Capillary Rise method (CR)

CR requires a sample taken from the bulk and is therefore only applicable in laboratory and considered as destructive in this context. CR measurements were performed according to UNI/NORMAL 10859, as this setup is more similar to CSM than the setup of DIN 52617. A stone sample is placed on a pile of humid filter papers, allowing the stone to absorb water by upward capillary forces. After the fixed contact time the samples are withdrawn, water on the surface which has not been absorbed is removed using a humid cloth and the samples are weighed. The WAB is calculated according to (1), where A equals the contact area between the sample and the humid paper (0.0049 m²); m_i and m_f respectively the initial and final mass of the stone sample.

$$Wa\ \left(g/m^2 .\ s\right) = \frac{\Delta m}{A\ .\ t} = \frac{\left(m_i - m_f\right)}{A\ .\ t} \quad (1)$$

$$C_{KT\Delta 5-15\min}\left(\mathrm{g/m^2\ .\ s}\right) = \frac{\left(m_{(15\min)} - m_{(5\min)}\right)}{A\ .\ t_{10\min}} \quad (2)$$

2.2.2. Karsten Tube (KT)

The Karsten tube is an open glass tube with a larger cylindrical body at the end, which is sealed to the surface of the stone by plastiline. Once attached, water is added to the tube and the amount of water absorbed over a certain period of time can be recorded by measuring the reduction of the water volume in the graded tube. The gradations go from 0 to 4 ml, divided in sub-gradations of 0.1 ml. The water column has a height of 9.8 cm, measured from the start of the gradations to the centre of the cylindrical body, exerting a pressure on the stone surface of 961.38 Pa. This pressure corresponds with rain drops hitting the wall with a static wind velocity of 140 km/h, perpendicular to the surface (Van Hees *et al.*, 1995), in order to analyse if water can penetrate through a hydrophobic barrier in case of heavy rainfall. The WAB was calculated according to (1), where A equals the surface of the cylindrical body (0.000573 m²) and Δm equals the amount of water absorbed over the contact time t. According to the RILEM II.4 recommendation (RILEM, 1980), measurements were performed after 5 and 15 min contact time, in order to calculate the WAB between the 5th and 15th minute (CKTΔ5-15min) (2).

2.2.3. Contact-sponge method (CSM)

The Contact-sponge method was developed in 2004 by Tiano and Pardini (Tiano & Pardini, 2004). The method consists of a sponge (type Calypso natural make-up from Spontex®), enclosed in a 1034 Rodac® contact plate. The contact plate is composed of two parts, namely a base and a cover. The diameter of the sponge corresponds with the inner diameter of the base, whereas the height of the sponge exceeds the vertical borders of the base. For the measurement the cover of the contact plate is removed, water is added to the sponge and the sponge enclosed in the contact plate is weighed (m_i). After weighing, the cover of the contact plate is removed and the sponge is pressed manually against the stone surface until the vertical borders of the base touch the stone surface. After the selected contact time, the contact sponge is removed and weighed inside the closed contact plate (m_f). The WAB is calculated according to (1), where A equals the surface of the sponge (0.002376 m²); m_i and m_f respectively the initial and final mass of the sponge enclosed in the contact plate. In this study the sponge was placed underneath the stone sample and the sample was pressed from above against the contact sponge, thus causing the water to be absorbed by upwards capillary forces, similarly to CR.

2.2.4. Mirowski pipe (MIR)

The Mirowski Pipe has been developed and patented by the Polish Prof. R. Mirowski (Patent No 125504). The method consists of a graded glass tube, closed at the top and with a small cylindrical body containing a sponge on the other end. The gradations reach from 0 to 10 ml, divided in sub-gradations of 0.1 ml. For MIR, the pipe is filled with water through the open cylindrical body, after which it is closed by adding the small sponge in the cylindrical body. To attach the tube to the surface under analysis, a partially open rubber ring with two metal pins is fixed to the surface with tape above the designated measuring point. The filled pipe is then placed in the rubber ring, as such that the sponge touches the measuring point. Water is absorbed by the stone through the sponge by capillary forces. According to the manual of MIR, the amount of water absorbed can be recorded by measuring the reduction of the water volume in the graded pipe. Preliminary tests (Vandevoorde *et al.*, 2011) showed that when water is absorbed, air enters the tube, but in most cases the air bubbles stay trapped into the sponge instead of rising to the top of the tube and thus lowering the water level. As a consequence, the water uptake at a certain contact time could not be recorded correctly. Only at the moment when the tube is released from the surface, most air bubbles do start rising. To bypass this practical inconvenience, a modification was made to the protocol. The tube was weighed before measurement and after each designated period of contact with the stone, allowing calculation of the WAB according to (1), where A equals the surface of the Mirowski sponge (0.000113 m²); m_i and m_f represent respectively the initial and final mass of the Mirowski pipe. In accordance to the recommendations for KT, the WAB between the 5th and 15th minute was also calculated with (2).

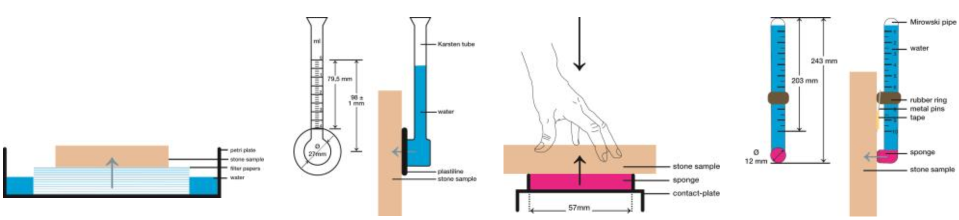


Fig 1-4: Schematic illustration of CR, KT, CSM and MIR

2.2.5. X-ray radiography and neutron radiography

X-ray and neutron radiography and tomography have become important techniques of investigation in different fields, including soil science and fluid-flow research (Cnudde et al., 2004; Dierick et al., 2005). In this study X-ray as well as neutron radiography were used to visualize the water absorption process inside stone samples of MC, MJ, V and E. X-ray radiography was performed at UGCT, Ghent, Belgium and neutron radiography at the NEUTRA beamline (NEUtron Transmission Radiography) at the Paul Scherrer Institute in Villigen, Switzerland. The samples were positioned between the source and detector and time lapse radiographs with a predefined time interval were taken. To improve the visualization of the water, the images of the absorption process were divided by an initial open beam image of the dry state. Consequently, a one-dimensional absorption process could be observed objectively through changes in grey values and quantified by measuring the height of the waterfront in function of time.

3. Results

Besides the non-destructive methods KT, CSM and MIR, CR was also included in this study, as a reference method for the analysis of the WAB. In a first stage all methods were preliminary tested by the operator to gain familiarity with the application and to examine the possibilities and limitations of the methods. In a second stage the performance of the methods is scrutinized by analysing the relation between the open porosity and WAB measured by each method (1), comparing the performance of different methods with the normalized CR (2) and studying the influence of variable factors (3). Table 1 gives an overview of the average WAB and variation coefficient measured with the different methods on seven lithotypes. To have a comparable basis, two specific contact times for the measurements were predefined, based on the provided application standards or manuals of the methods. For all methods a contact time of 90 sec was applied. For KT and MIR the WAB between the 5th and the 15th min was also measured, according to the RILEM II.4 recommendations (Van Hees *et al.*, 1995). For each lithotype the average was calculated on 16 samples for CR, CSM, KT and on 5 samples for MIR. It has to be mentioned that the WAB for KT is 10 times lower in comparison with earlier published results (Vandevoorde *et al.*, 2009), due to a miscalculation in previous results. This miscalculation has been corrected in Table 1. The WAB of KT Δ5-15 min for the stones T and E cannot be taken in account as in most cases all water from the pipe was absorbed before 15 min and the pipe was not refilled during measurement. Therefore, no valid results were obtained for these stones.

	OP	**CR 90s**		**CSM 90s**		**KT 90s**		**KT Δ5-15 min**		**MIR 90s**		**MIR Δ5-15 min**	
MC	10.7	2.3±0.2	*7.9*	2.7±0.2	*7.3*	1.6±0.5	*30.8*	0.8±0.2	*22.0*	5.0±1.3	*25.6*	1.6±1.0	*62.5*
MJ	10.5	2.8±0.6	*20.7*	3.0±0.8	*27.7*	1.8±0.7	*38.3*	0.8±0.3	*34.9*	7.4±4.7	*63.4*	1.7±1.4	*81.5*
S	10.6	2.9±0.4	*13.3*	3.3 ±0.4	*11.5*	1.5±0.5	*32.8*	1.2±0.3	*23.1*	5.5±2.0	*36.1*	2.1±0.6	*28.7*
V	13.5	2.8±0.2	*8.0*	3.7±0.3	*8.1*	1.6±0.6	*35.7*	1.1±0.3	*28.0*	8.4±6.3	*74.3*	3.5±1.5	*42.9*
M	15.6	5.4±1.5	*28.2*	5.8±1.5	*25.9*	3.8±2.6	*67.6*	1.6±0.9	*58.6*	5.8±3.4	*58.7*	3.2±2.0	*38.0*
T	23.7	13.2±1.8	*13.8*	19.4±1.7	*8.8*	13.5±2.4	*17.9*	-	-	32.3±2.1	*6.6*	16.8±6.4	*38.0*
E	29.3	31.7±5.0	*15.7*	30.6±3.2	*10.5*	29.7±9.8	*33.1*	-	-	44.3±11.9	*26.8*	27.3±8.6	*31.5*

Table 1: Average open porosity [%] and WAB (average [g/m²s] ± standard deviation and variation coefficient [%]) for seven lithotypes. For each type the average was calculated on 16 samples for CR, CSM, KT and on 5 samples for MIR.

4. Discussion

4.1. Comparison of results and accuracy

In relation to the open porosity, it can be noticed that each method is able to distinguish differences in open porosity. The higher the open porosity, the higher the WAB measured by each method. Although all methods follow the same trend, the absolute values differ in each method. In comparison to CR, CSM reveals a similar, though slightly higher WAB for a contact time of 90s. A possible reason for this difference is that the water on the surface of the stone is wiped off before weighing the sample with CR, which in not the case for CSM. Only the result for lithotype E for CSM does not follow this trend, which is probably due to the fact that the amount of water in the sponge was insufficient for the high water uptake by lithotype E. Therefore, it can be assumed that the result for lithotype E for the CSM90s is not valid. The absorption by KT90s is remarkably lower than CR and CSM. Firstly, this could be due to water which is already being absorbed before the actual start of the measurement. For measurements with KT water is poured into the tube, and the measurement only starts once the water level reaches the zero gradation on de graded tube. The amount of water which is already being absorbed while pouring the water in the tube is not taken in account in the measurement. Secondly, the WAB could be lowered due to the reduction of the contact area by the plastiline which seals the tube to the surface. To obtain sufficient adhesion and a watertight sealing, the plastiline is pressed between the stone and the tube, causing the plastiline to expand side wards, reducing the contact area irregularly. Secondly the plastiline leaves oily residues on the surface. In case of repeated measurements on the same area, this can lower the water absorption due to the water repellence of the measured area caused by the oily stains. For MIR90s a very high WAB is registered. This can be due to the frequent leaking. As the contact area of MIR is rather small, the quantitative water uptake is very low and only a small leak is sufficient to cause a significant deformation of the calculated WAB. The consequences of these practical encumbrances are reflected by the variation coefficients (Table 1). For CSM and CR the variation coefficient is of the same acceptable magnitude, whereas for KT90s and especially MIR the variation coefficient is almost unacceptably high. When the variation coefficient for CR and CSM are compared for each lithotype a clear relation between the intrinsic heterogeneity of the stone and the variation coefficient can be observed. The more heterogeneous the lithotype, the higher the variation coefficient. This relation cannot be observed as clearly for KT and MIR. When taking in account the high variation coefficient for KT90s and MIR90s it is suggested that neither KT nor MIR are suitable methods for the analysis of the WAB for short contact times.

The most significant difference between the methods was noticed through visualization of the water absorption by neutron radiography. The radiographs clearly depict the evolution of the waterfront inside the stone, where a significant side wards absorption is developed from the borders of the contact area on. Figures 5-8 are neutron radiographs of the water absorption by the lithotype V after a contact time of 205 sec with the different methods. The continuous line indicates the constant contact area, whereas the dotted line represents the development of the waterfront. For CR the total surface of the sample is brought in contact with water, impeding additional side wards absorption. For the other methods with a restricted contact area in comparison to the sample under investigation, pores on the borders of the contact area absorb additional water. The smaller the contact area, the more pores are surrounding the contact area relative to the contact area itself. So more additional water can be absorbed side wards in relation to the water absorbed by the contact area itself. To further analyse the influence of this sideward absorption, capillary rise tests have been performed on samples where the surface in contact with the water was taped corresponding to the respective contact areas of the different methods. Graph 1 confirms the relation between the size of the contact area and the water absorption (g/m²) in function of time: the smaller the contact area, the higher the water absorption. Moreover, for KT these results do not correspond to the results in table 1, where the WAB for KT is lower than for CR and CSM. This discrepancy could indicate the influence of the water absorbed before the start of the measurement. For CR the different standardised application procedures, namely UNI/NORMAL 10859 (CR F) and DIN 52617 (CR G) were compared, to study the influence of the contact material. It can be observed that, for V as well as for other lithotypes tested, the water absorption without filter papers is slightly lower but the differences do not exceed the standard deviation on the measurements. Consequently, it can be concluded that there is no significant influence caused by the contact material.

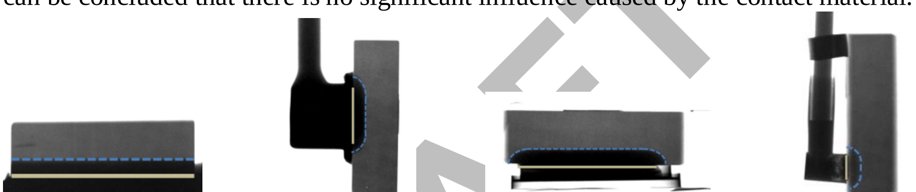
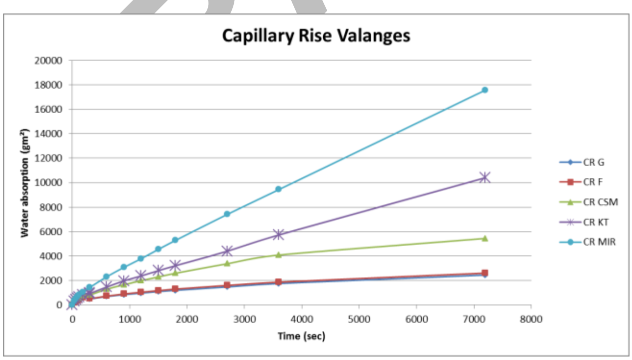


Fig 5-8: Neutron radiographs of the water absorption of V after 205 sec contact time with CR, KT, CSM and MIR.



Graph 1: Capillary water absorption by V in function of time with restricted contact areas, according to the different methods CR CSM, CR KT and CR MIR), compared to the CR according to UNI/NORMAL 10859 (CR F) and DIN 52617 (CR G).

4.2. Possibilities and limitations of the methods due to variable factors

Table 2 gives an overview of the variable factors of the methods under investigation, depending on their specific design. The consequences for the applicability of the methods is discussed below.

	Capillary Rise	**Karsten Tube**	**Contact sponge**	**Mirowski Tube**
Applicability	Lab	Lab / *In situ*	Lab / *In situ*	Lab / *In situ*

Non-destructive	No	Yes	Yes	Yes
Contact area	Size of sample	0.000573 m^2	0.002376 m^2	0.000113 m^2
Available water	Unlimited	Unlimited	5-12 ml	10 ml
Contact time	Unlimited	5 min - …	0 – 5 min	Untill end of water supply
Pressure	-	961.38 Pascal	-	'vacuum'
Precision	0.001g	0.05g	0.001g	0.05g or 0.001g
Disadvantages	Destructive	Stains of plastiline on surface	Limited water supply	Air supply through sponge
Application procedure	UNI/Normal 10859 (or DIN 52617)	RILEM II.4	Publication Tiano &Pardini, 2004	Manual according to patent No 125504

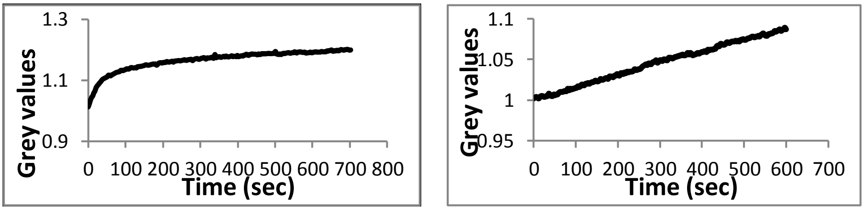
Table 2: Overview of the technical specifications, possibilities and limitations of the CR, KT, CSM and MIR.

4.2.1. Contact area

In order to obtain a representative measurement, the measured area should be in accord with the heterogeneity of the stone. For example, in case of more heterogeneous lithotypes, more measurements with a smaller contact area (e.g. MIR) are needed to obtain an average absorption value, in comparison to one measurement with e.g. CSM, which has a much larger contact area. Consequently, measurements with MIR will be more time consuming. Howbeit, the larger contact area of CSM can be a disadvantage in case of measurements on curved surfaces of sculptures or small ornaments.

4.2.2.Water supply and contact time

A second variable of the methods is the availability of the water, which delimitates the contact time. CSM and MIR have a limited water reservoir, while for KT water can continuously be added, depending on the amount of water absorbed. The end of the water supply and measurement can be recorded visually for KT and MIR on the graded tube. For CSM this is not the case, as the amount of water present in the sponge is not visible. The water absorption from the sponge by the stone has been visualized by X-ray radiography, where the water absorption is displayed by changes of grey values in the sponge. Graphs 2 and 3 illustrate the evolution of the grey values during measurements. It is clear that for the more porous stone E (open porosity 29.3 ± 0.6) the sponge runs out of water after approximately 36s (clear decline around 36s), while for the less porous stone MC (open porosity 10.7 ± 0.2) even after 420s water is still being absorbed from the sponge (no decline in the graph). Hence, the application methodology for CSM was adapted. An additional calculation was made for comparison of the amount of water added to the sponge before measurement with the amount of water absorbed, to control the validity of the measurement after a certain contact time. For KT the initial contact time is limited, due to the water which is already being absorbed before the actual start of the measurement while pouring the water in the tube. Hence, KT was not found reliable for measurements under 5 minutes contact time, as stated in the recommendations by Van Hees *et al.* (1995). These above mentioned limitations of the water supply mainly determine the contact time and compatibility of the methods.



Graph 2 and 3: Evolution of the grey values of the sponge for lithotype E (left) and MC (right)

4.2.3. Precision

For KT the measurement is performed by reading the lowering of the water meniscus in the graded tube with subgradations of 0.1 g. Due to the curved water meniscus, the amount of water absorbed can be estimated visually with a precision of 0.05 g. The precision of CSM depends on the balance used, where generally a precision of 0.001g is sufficient. For MIR the amount of water absorbed has to be read from the graded tube, according to its manual. Due to air bubbles staying trapped into the sponge and impairing the water level to lower, as described in the methods section, the water uptake at a certain contact time could not be recorded correctly. To bypass this practical inconvenience, the tube was weighed before measurement and after each designated period of contact with the stone. An additional advantage of this modification is that the measurement can be made with a precision of 0.001g when weighing the tube, instead of the visual estimation with a precision of 0.05g. Regarding the precision of the methods in relation to the materials under study, a high precision is mainly needed in case of a porosity lower than approximately 15% in combination with short contact times.

4.2.4. Practical encumbrances

When considering practical encumbrances when applying the methods, it are mainly KT and MIR which are under discussion. For KT the critical point is the fixation of the tube on the surface by plastiline. Firstly, the plastiline can irregularly reduce the contact area and make the surface water repellent for consecutive measurements. Secondly, it is hard to obtain a watertight sealing. Thirdly, it occurs that the plastiline does not adhere sufficiently on the surface and the tube lets go of the surface during the measurement. This occurs more frequently when the surface suffers from superficial de-cohesion or contamination. These encumbrances can cause the measurement to fail. For MIR the critical point is the sponge which closes the tube and regulates the contact with the stone. Firstly, the positioning of the sponge inside the cylindrical body, correctly and without having air entrapped in the pipe, is very hard to obtain. To position the sponge with its total surface in contact with the stone and without water leaking out is difficult. Even after extended training one out of four measurements, performed in optimal practical laboratory conditions, fails due to water leaking out at the moment of positioning the sponge against the stone or even at a random moment during the measurement. From the moment the pipe suffers the slightest movement (e.g. due to wind), leaking is occurring. Secondly, the air entrapped in the sponge during the measurements, retains a constant water supply and impairs a correct registration of the amount of water absorbed.

5. Conclusions

The WAB of stone material can be non-destructively measured with CSM, KT and MIR. As these methods have a different design to accomplish specific aims, this leads to discrepancies in practical application and measuring area. In this study the different methods are compared and the influence of variable factors on the measurements due to their specific design is scrutinized. A first conclusion was that each method has its specific field of analysis. CSM is capable of measuring the initial water uptake of less porous materials with a high precision, but is not suitable for analysis of longer measuring periods of more porous stones. KT was found incommodious for measurements of the water uptake before 5 minutes, but it has the possibility of measuring longer contact times for more porous lithotypes. MIR showed too many discommodities, leading to unreliable results. Secondly general trends of WAB were noticed, of which the influence of the side wards absorption surrounding the contact area turned out to be a significant factor to relate the results of the methods. Further study of physico-mathematical models describing the WAB is ongoing. Moreover the models will be compared to the visualisation of the WAB by X-ray and neutron radiography.

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