UNB
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THE INFLUENCE OF PRODUCTION PROCESSES AND MORTAR COMPOSITIONS ON THE PROPERTIES OF HISTORICAL MORTARS

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ABSTRACT

Since the Ancient Greeks and Romans lime has been the most important binder for the production of mortar in the building technology in Europe throughout many centuries. It is only by the introduction of scientific research in the field of building technology in the 18th and 19th century that gradually a new and stronger binder, known as cement, was introduced. By the beginning of this century the use of cement in the building practice had become that important, that lime as binder was only seldom used for the construction of new buildings and even so for the restoration of our cultural built heritage. At present almost all knowledge, gained by means of practice for many centuries, has been lost. For a sound restoration of our precious cultural built heritage the knowledge of the physical, chemical and mechanical behavior of this ancient binder is however essential.

As a result an extensive test program has been set up with as main objective the study of the physical, chemical and mechanical behavior of hydrated lime mortar. In a first approach the influence of the 'traditional' methods for slaking, the binder/aggregate ratio and the type of aggregates on the physical and mechanical properties of the mortar is studied. Three different production methods for the lime will be studied; commercially produced hydrated lime, wet and dry slaked quicklime. As for the binder/aggregate ratio three different ratio's will be examined for two sand types with completely different grain size distributions; one mixture with a perfect filling of the pores in the sand matrix by the lime binder, one meager and one fatter mixture of sand with lime binder.

The first results from this extensive test program will be presented and discussed.

Key words: heritage, restoration, lime mortar, slaking, granulometry, porosity

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INTRODUCTION

The influence of the 'traditional' methods for slaking of the quicklime, the binder/aggregate ratio and the granulometry of the aggregates on the physical properties of hydrated lime mortars is studied. A total of fourteen different lime mortars have therefore been prepared. In addition some information is sought on the influence of the curing conditions on the physical properties of the lime mortars. Mortar cured between two bricks is compared to mortar taken from standard beams. Also the influence of the carbon dioxide content of the surrounding atmosphere upon curing of the mortar is briefly evaluated.

For the physical properties of the hydrated lime mortars, main attention has been focused on the internal pore structure; the progress of the carbonation front inside the mortar, the total volumetric porosity and the pore size distribution. Currently the pore size distribution has only been evaluated by means of mercury intrusion and the capillary behavior. Future research will go further into depth on the internal pore structure of the mortars. Especially regarding the known problems related to the validity of the mercury intrusion test set-up.

THE HYDRATED LIME MORTARS

An air hardening or hydrated lime mortar is a mixture of lime putty, sand and water. Gypsum and other additives, often organic in nature, were in historical times regularly added to the lime mortars. A total of fourteen different hydrated lime mortars were composed in order to study the influence of the traditional slaking method of the quicklime, the granulometry of the sand and the binder/aggregate ratio on the properties of the hydrated lime mortars.

Slaking methods of the quicklime

The binder, putty lime ($Ca(OH)_2$), is obtained through the slaking of crushed and burnt limestone, also known as quicklime (CaO). The reaction is highly exothermic (1).

$$\mathrm{CaO + H_2O \rightarrow Ca(OH)_2 + 1177.2\ kJ/kg\ CaO} \tag{1}$$

Throughout history two traditional methods were developed for the slaking of the quicklime; a wet slaking method, where the quicklime is submerged in water for sometimes 20, 30 or even 50 years, and a dry slaking method, which consists of the mixing of the quicklime with wet sand (Callebaut 1999, 2000). Both traditional methods were simulated in laboratory in order to investigate the influence of the slaking method of the quicklime on the final properties of the lime mortar. A series of wet slaked (Ws) and dry slaked (Ds) mortars were prepared with the quicklime Supervical from Carmeuse. For reference a third series of lime mortars (C) was made with the commercial hydrated lime Supercalco.

Wet slaking of the quicklime was achieved upon submersion of the quicklime in tap water for a period of 1 week, during which the mixture was regularly stirred. Since the hydration of the quicklime is a highly exothermic reaction (a reaction enthalpy of 1177.2 kJ per kilogram quicklime), an amount of water, roughly 4 times the amount of quicklime, was estimated to be necessary for the slaking of the quicklime. Subsequently the wet slaked quicklime was drained for 2 hours in order to eliminate the excessive water. The final water content of the putty lime remained however too high for the realization of some of the mortar mixes. Only both meager mortar mixes could be made with the wet slaked putty lime. The determination of the physical properties of the wet slaked lime mortars is still in progress.

The dry slaking of quicklime was obtained by the stratified mixing of the quicklime with wet sand with a layer thickness of 4 cm (Callebaut 2000). An amount of water, equal to the stoichiometric amount of water necessary for a complete hydration of the quicklime, was added beforehand to the sand and mixed thoroughly. An additional 10 % of water was added in order to account for the loss of water due to evaporation. In the first hours of slaking, the temperature of the wet sand - quicklime mix can rise as high as 150 ˚C as a result of the exothermic hydration reaction. After a period of 4 days the sand and quicklime were mixed. After another 3 days the sand and putty lime mix was ready for use.

Different types of aggregate

Two different types of aggregates were used in order to study the influence of their granulometry on the final properties of the lime mortars; the very fine Zutendael sand (Z), which is a typical sand used in historic masonry mortar and still nowadays, and the coarse standardized sand (N) according to DIN EN 196-1. The grain size distribution of both sands is represented in figure 1 (DIN-4188).

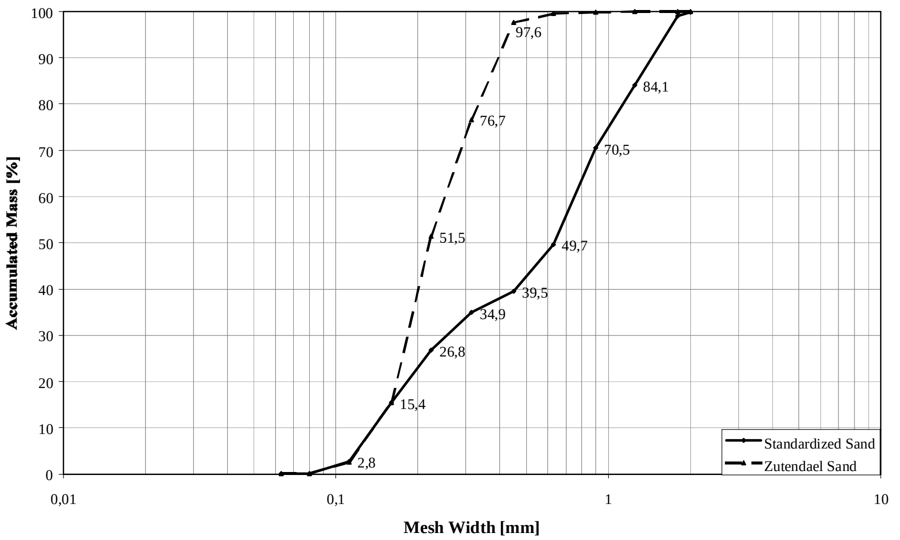


Figure 1. Grain size distribution of both sand types

Binder/aggregate ratio

Three different binder/aggregate ratios were studied; the 'ideal' binder/aggregate mix (I) together with a meager (M) and a fatter (F) lime mortar. The 'ideal' binder/aggregate mix is being defined as the composition where the hydrated lime binder fills all the pores of the sand skeleton. The amount of binder can be determined by means of the density of the putty lime and the volumetric porosity of the sand skeleton. Analysis of the ideal mortar composition gives a volumetric binder/aggregate ratio of 1:3.05 for the Zutendael sand and 1:2.97 for the standardized sand. Despite a distinct difference in grain size distribution between both sand types, there is no significant difference in binder/aggregate ratio for the respective ideal mortar compositions. The meager and fatter lime mortar compositions were consequently determined as 1:6 and 1:1 volumetric binder/aggregate ratios respectively. These ratios were chosen in order to obtain different hydrated lime mortars to such an extent that clearly different mortar properties should be expected, while in the mean time the historic context of the research was not lost.

Table 1. Basic composition of the different hydrated lime mortars in grams

Sand Type	Zutendael Sand			Standardized Sand		
B/A Ratio	1:1	1:3.05	1:6	1:1	1:2.97	1:6
Binder	372	122	62	338	114	56
Sand	1000	1000	1000	1000	1000	1000
Water	270	240	244	214	199	126
Total	1642	1362	1306	1552	1313	1182

Once the right proportions of putty lime and sand were obtained and mixed thoroughly, tap water was added to obtain a good workable hydrated lime mortar. The amount of water was determined, with the apparent workability of the mortar as criterion. The basic mortar compositions are represented in table 1. A relative comparison of the different components in the hydrated lime mortars is given in figure 2.

Table 2 gives an overview of the codes used for the denomination of the different mortar types.

Table 2. Overview of the denomination codes for the different mortars

Sand Type	Zutendael Sand			Standardized Sand		
Binder B/A-ratio	1:1	1:3	1:6	1:1	1:3	1:6
Commercial Lime	CZ-F	CZ-I	CZ-M	CS-F	CS-I	CS-M
Dry slaked Lime	DsZ-F	DsZ-I	DsZ-M	DsS-F	DsS-I	DsS-M
Wet slaked Lime	---	---	WsZ-M	---	---	WsS-M

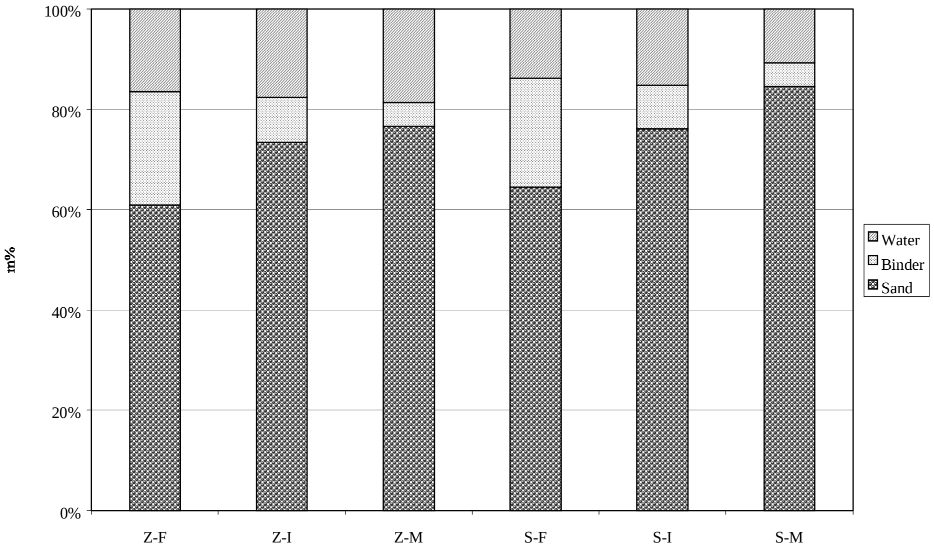


Figure 2. Relative comparison of the different hydrated lime mortars

THE LIME MORTAR SAMPLES

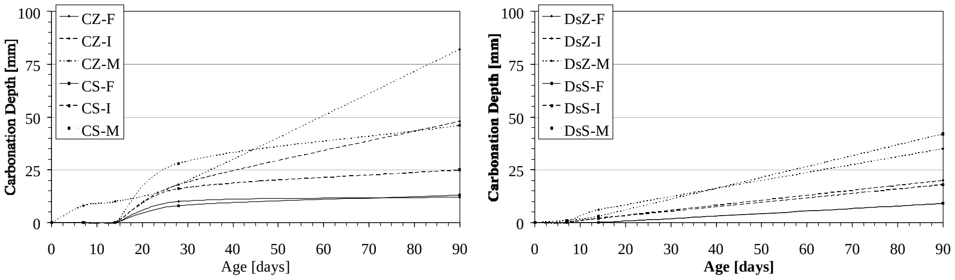
The study of the influence of the mortar composition on different physical properties was performed on mortar samples retrieved from both standard beams 160 mm by 40 mm by 40 mm and mortar joints in between two bricks. The brick used is a hand molded Spanish Red facade brick, module 50 (NBN B23-002) from Quirijnen. For the smooth retrieval of the mortar from the brick doublet, a thin filter paper is inserted between both brick-mortar interfaces upon the making of the brick doublets. Hence the water transportation between the bricks and the mortar is not hindered, while the adhesion of the mortar to the bricks is prohibited. As such the mortar properties as determined by means of the standard prescribed test set-ups can be compared with the real mortar properties within the masonry joints.

All the mortar samples were cured in two ways; a natural curing condition in a climatic chamber and an accelerated carbonation in a CO_2-chamber. For the natural curing the mortar samples were put in a climatic chamber with an environment of 21 °C and 60 % R.H. In such a way a natural carbonation was attained for a period of 90 days for the determination of the physical properties. The carbonation speed of the mortars in these natural environmental conditions was determined by a set of standard beams. The accelerated carbonation of the lime mortar samples was obtained after a curing period of 28 days in the climatic chamber and another 7 days in an environment of 25 °C, 63 % R.H. and a CO_2-content of 20 % in the air. The curing conditions for accelerated carbonation are sufficient in order to achieve a complete carbonation of the standard beam samples (Van Balen 1991). Recent studies by Knöfel (Knöfel 1992) demonstrate however that the acceleration of the carbonation of mortars has a strong influence on its mechanical and physical properties.

COMPARISON OF THE PHYSICAL PROPERTIES

Carbonation Depth

The carbonation depth of the different mortars was determined on standard beams at ages of 7, 14, 28 and 90 days. The mortar beams were taken from their moulds after 7 days and coated with a paraffin layer on all sides, except on one of both small faces, through which surface the carbonation could continue. At the prescribed curing ages the standard beams were cut into two, lengthwise, and the carbonation depth was measured. Since the conversion of putty lime ($Ca(OH)_2$) into calcium carbonates ($CaCO_3$) changes the pH of the lime mortar from ±12,5 pH for the not-carbonated zone to 7-8 pH for the carbonated zone, the carbonation depth can be determined by means of a pH-indicator with a color transition around 9 pH. A solution of 1 % phenolphthalein in 70 % ethylalcohol was used as pH-indicator, changing color at 9.2 pH. The results of the carbonation depth in function of curing age are represented in figure 3, with the commercial lime mortar on the left and the dry slaked lime mortar on the right. The values of the carbonation depth of the dry slaked lime mortars at a curing age of 28 days have not yet been determined. The linear transition for a curing age of 14 days to a curing age of 90 days is therefore only a rough estimate of the progress of the carbonation front in the hydrated lime mortar samples.



commercial lime (C) dry slaked lime (Ds)

Figure 3. Carbonation depth

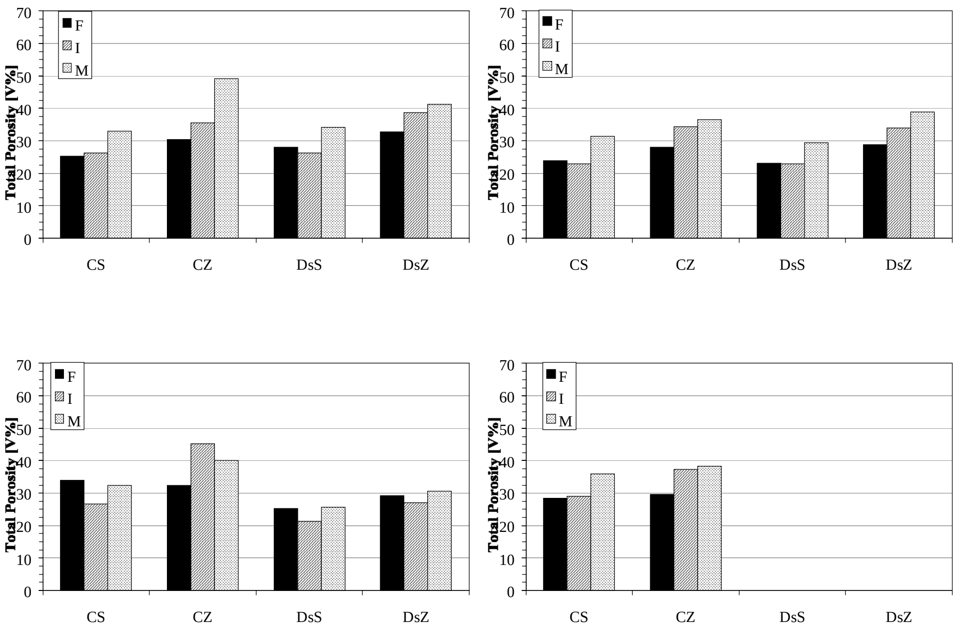
The following conclusions can be drawn from the data represented in figure 3:

- There is a clear influence of the binder/aggregate ratio on the carbonation depth at a certain curing age of the hydrated lime mortar, with the more meager lime mortar having the highest carbonation speed.
- The influence of the granulometry of the sand is less evident. Only for the commercial lime mortar a large difference in carbonation depth between both sand types is noted, with the mortars prepared with the Zutendael sand clearly having a much larger carbonation speed.

- For the mortars based upon the standardized sand no significant influence of the slaking method on the progression of the carbonation front can be observed. The remarkable combination of the very fine Zutendael sand with the commercial lime shows, in comparison with the analogous mortars with the dry slaked lime binder, a much elevated carbonation speed.

Total porosity

The total porosity of the hydrated lime mortars has been determined by vacuum saturation (NBN B05-202) for both the standard beams and the mortar joints. The results are represented in figure 4. The results of the total porosity of the mortar retrieved from the standard beams are represented on the left of figure 4, while the results of the mortar from the masonry joints are represented on the right. The two graphs on the top represent the results after accelerated carbonation, the two graphs on the bottom the volumetric porosity of the mortar samples at an age of 90 days. In the case of the dry slaked lime mortars the results of the pore volume after a natural carbonation for 90 days between two bricks have not yet been determined.



Left: standard beams; Right: mortar joints
Top: accelerated carbonation; Bottom: accelerated carbonation
Figure 4. Total porosity

The following conclusions can be drawn from the data represented in Figure 4:

- The influence of the binder/aggregate ratio on the total porosity of the hydrated lime mortar is fairly evident. The higher the sand content in relation to the amount of binder, the higher the total pore volume will be. An increase of about 6 V% to 9 V% has been measured between the fat and meager hydrated lime mortars.

- The grain size distribution has also an important influence on the total porosity of the mortars, with the finer Zutendael sand giving a higher pore volume. Differences from 5 V% to 10 V% were measured in pore volumes.
- There is no distinct difference in total pore volume due to the slaking method of the quicklime.
- The mortar retrieved from the mortar joints has a somewhat lower total porosity compared to the mortar from the standard beams. Except from the exceptional case of the combination of the Zutendael sand and the commercial lime, the differences in pore volume are only to the extend of a few percentages.
- It is impossible to draw some general conclusions after the comparison of the carbonated hydrated lime mortar samples, after accelerated carbonation, with the partly carbonated mortar samples at an age of 90 days. From the data given, it is however possible to say that the total pore volume of the mortars decreases as the carbonation progresses, when comparing the fully carbonated mortar samples under natural conditions (with a carbonation depth of at least 20 mm) with the partially carbonated mortars in the same circumstances.

Pore size distribution

The pore size distribution of the different hydrated lime mortar samples from the standard beams after accelerated carbonation was studied by mercury intrusion (NBN B05-202). The results are represented in figures 5 and 6. Figure 5 represents the accumulated pore size distribution for the hydrated lime mortars based on the commercial lime, while figure 6 represents the data on the dry slaked lime.

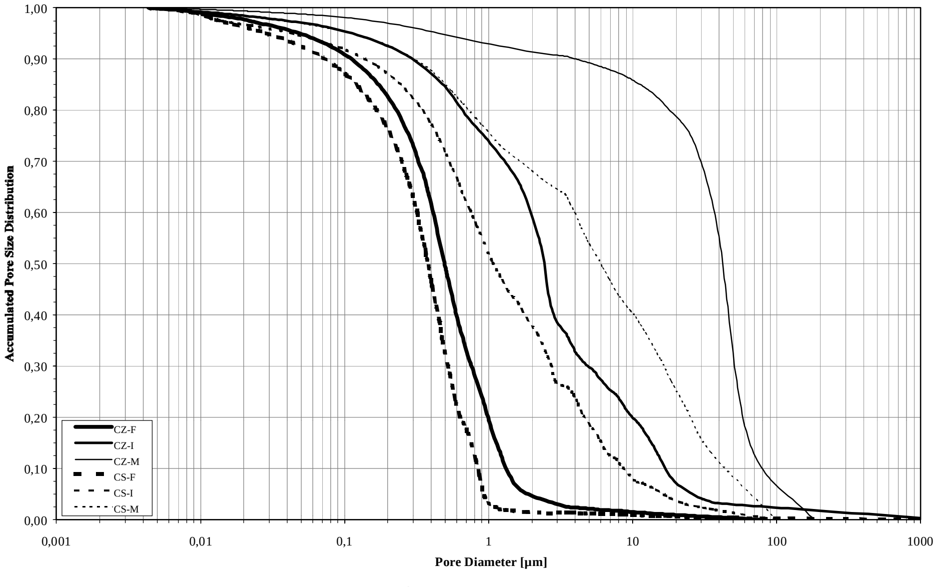


Figure 5. Pore size distribution of the mortars with commercial hydrated lime

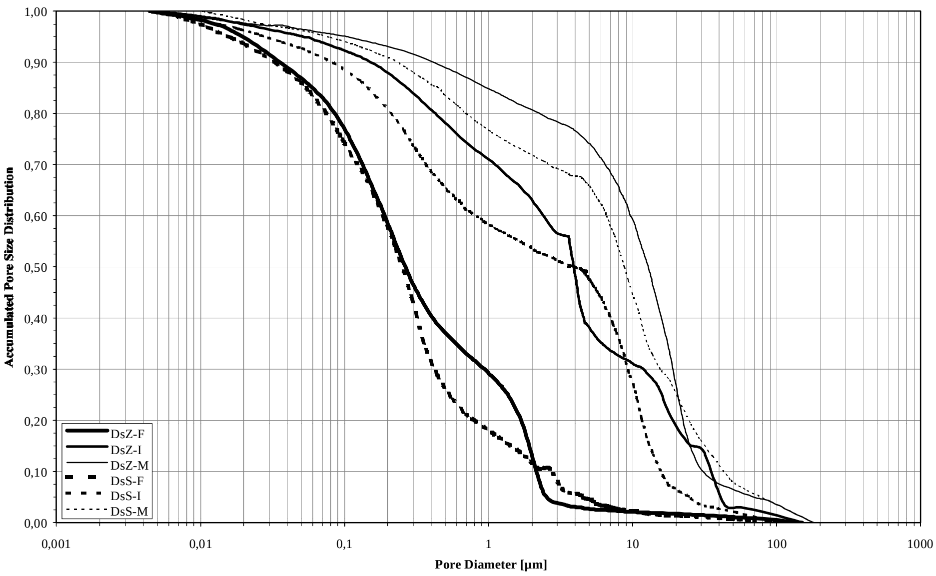


Figure 6. Pore size distribution of the mortars with dry slaked lime

The following conclusions can be drawn from the data represented in figures 5 and 6:

- The binder/aggregate ratio has a clear influence on the pore size distribution, as derived through mercury intrusion. For all the combinations of each binder type (dry slaked or commercial lime) with each sand type (Zutendael or standardized sand) higher sand contents mean coarser pore structures. For the commercial lime mortars the median pore size changes from about 0.5 µm for both fat mortar compositions to 6 µm (with standardized sand) or even 40 µm (with Zutendael sand) for the meager lime mortars. Analogous results are obtained for the dry slaked lime mortars where the median pore size changes from 0.3 µm for the fat mortar to about 15 µm for the meager hydrated lime mortars.
- The influence of the granulometry of the sand on the pore size distribution is less evident. Certainly for the dry slaked lime mortars, where there is no evidence for an important influence of the sand type on the pore size distribution. However, for the commercial lime mortar a somewhat coarser pore structure is found for the finer Zutendael sand. This influence of the sand granulometry increases clearly as the sand content increases in the composition of the lime mortars.
- A comparison of the results of the mercury intrusion tests indicates that in the case of dry slaking the band of pore sizes is wider than in the case with mortar from the commercial lime hydrate. The combination of the Zutendael sand in the meager commercial lime mortar gives a much coarser pore structure compared to the analogous dry slaked lime mortar.

The conclusions on behalf of the pore size distributions should be evaluated with the necessary precaution, regarding the possible destructive effect of large mercury pressures especially on the very weak hydrated lime mortars. The results obtained by means of mercury intrusion will in the near future be confronted with other measuring techniques for the evaluation of the pore size distribution in solid materials, like computer tomography, scanning electron microscopy, optical analysis on thin slices, etc. Earlier studies on the determination of the pore structure of very weak lime mortars, indicate that altogether a relative value can be attributed to the results retrieved by mercury intrusion (Hayen 1999).

Capillary water absorption

The capillary water absorption was determined on standard beams according to standard NBN B05-201. The capillary water absorption profiles of the mortars after accelerated carbonation are represented in figure 7.

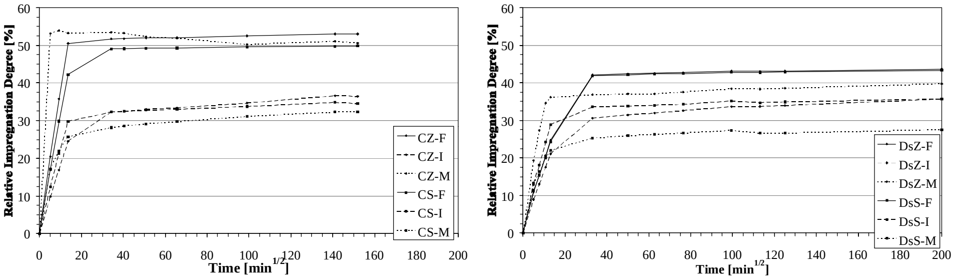


Figure 7. Capillary water absorption profiles

No general conclusions can be drawn from the data represented in figure 7, although the following considerations can be made:

- Fat hydrated lime mortars with a high binder content show an increased capillary water uptake.
- In the case of a high binder content in the hydrated lime mortar, there is a clear difference in capillary behavior between the commercial and the dry slaked lime mortars. For the other binder/aggregate mixes there is no evidence for a distinct capillary behavior due to a difference in slaking method of the quicklime.
- Anew the exceptional behavior of the meager commercial hydrated lime mortar with the Zutendael sand should be stressed. This mortar clearly shows a much higher capillary water uptake in comparison to the other meager and even ideal mortar compositions.

CONCLUSIONS

The study of the influence of the mortar composition and curing circumstances for different hydrated lime mortars on their physical properties reveals that:

- The binder/aggregate ratio has the strongest influence on the pore structure of the hydrated lime mortar. Hydrated lime mortars with low binder contents have a higher total volumetric porosity, consisting of mainly larger pores with pore diameters in the order of 6 μm to 15 μm. The coarser pore structure enables a faster penetration of the carbonation front into the mortar. The explanation of this higher carbonation speed is twofold. First the diffusion coefficient of the carbon dioxide into the coarser pore structure of the mortar is expected to be the highest for the meager mortar mixes. Second the lower lime content in a meager mortar composition results in a lower carbon dioxide uptake for a similar carbonation depth. A mortar with a high binder content seems to have a rather large capillary water suction capacity, since these mortars mainly have capillary pores, ranging from 0.1 µm to 1 µm in diameter.

- Second to the binder/aggregate ratio, the granulometry of the sand has also a strong influence on the pore structure of the hydrated lime mortars. This influence differs however from one slaking method to the other. While no important differences can be noted for the dry slaked lime mortars, the effect is very evident in the case of the commercial lime mortars. For the commercial lime mortars the finer Zutendael sand gives, remarkably, a coarser pore structure with a larger total volumetric porosity. Hence the difference in carbonation speed, where the Zutendael sand gives larger carbonation depths at comparable curing ages.
- Differences in slaking method seem to have an influence on the band width of the pore diameters and no major influence on the main physical properties of the hydrated lime mortars, except from the previous mentioned case where the influence of the granulometry of the sand was evaluated.
- The partial data on the study of the differences of the physical properties of the lime mortars in masonry compared to standard beam samples reveals an overall decrease of total volumetric porosity of only a few percentages. Further study on mortars retrieved from both between bricks and standard beams will have to reveal if important differences occur in the pore size structure of the hydrated lime mortars.
- So far no conclusions can be drawn as for the influence of the carbon dioxide content in the air during the curing of the lime mortars. Further research will be necessary.

This research is a first step in our understanding of hydrated lime as a binder in our cultural heritage. Future research on the pore structure of the different hydrated lime mortars will be performed in order to evaluate the validity of the mercury intrusion test and to have a better view of the internal structure of the mortars. For now main attention has been laid upon the study of the internal pore structure of the hydrated lime mortars. Future research will also focus on other physical and mechanical properties of the different mortar compositions. The influence of the strength and overall mechanical behavior of the hydrated lime mortars on the mechanical behavior of the masonry will in the near future be evaluated on small piers and wallets.

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