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Experimental study of coupled transport of moisture and salt into unsaturated mortars and terracotta

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ABSTRACT

In this paper the combined evolution of liquid and chloride content is studied during experiments of sodium chloride transfer in unsaturated mortars and terracotta. The means used are gamma-ray attenuation for the liquid content and chemical analysis for the chloride content. It is experimentally very difficult to know the part played by chloride ions bound to the solid matrix. Some simplifications, allowing analysis of the results with suitable uncertainties, are proposed: for instance the quantity of bound chlorides is negligible. The decrease in the rate of reaction of chlorides with the cementitious matrix is highlighted, and is due to liquid movement. Moreover, the presence of salt does not seem to affect moisture transport for the studied materials.

1. INTRODUCTION

The aim of this work is to study the coupled transport of liquid and sodium chloride into unsaturated mortars and terracotta. It requires the determination of the space-time profiles of the liquid and chloride content during drying and wetting processes. Coupled transport in unsaturated materials are studied because transfers in saturated conditions are now rather well known [1-3]. The volumetric liquid content profiles are obtained by the method of gamma-ray attenuation and chloride profiles by chemical analysis. As it will be shown, the experimental problem is complex. The concentrations of free, bound and crystallised chloride ions must be known at each point to calculate liquid content by gamma-ray attenuation. But our experiments do not allow to determine the amount of free chloride ions. Considering this experimental difficulty and in order to reduce the number of experiments, some justified assumptions, which still lead to acceptable results, are proposed. Two kinds of materials are used in this work: mortars, which react chemically and physically with chlorides, and terracotta,

which is inert (no C_3A and C_4AF) and contains a large amount of vitreous phase.

2. THE MATERIALS

Two different kinds of mortars have been studied. M1N and M2N are normal mortars ($W/C = 0.5$; $Aggregate/C = 3$); the maximum size of sand grains is 2 mm. M1N was made with CEM I 52.5 HTS cement and mortar M2N was prepared with CEM I 52.5 cement. The first cement, used in marine environments, is low in C_3A (4.45%) and in C_4AF (6.53%) whereas the second one has more C_3A (11.3%) and the same level of C_4AF . Blocks of $4 \times 4 \times 6 \text{ cm}^3$ were realised. They were kept under water during a month after removal from the mould and dried for four months at a temperature of 35°C with a few percent relative humidity to obtain stable materials. In such conditions it has been experimentally verified, that practically no carbonation occurs [4, 5]. The principal characteristics of these materials, including terracotta, are given in references [4, 6].

3. DETERMINATION OF LIQUID CONTENT PROFILES

The four sides of each specimen were made watertight with resin to ensure a one-dimensional process. All the materials are submitted to only one wetting and drying cycle. The wetting process lasts one day for terracotta and twenty days for mortars and the drying lasts two weeks for terracotta and three months for mortars.

The different environmental conditions are:

Wetting process:

– lower side: contact with salt solutions, prepared with distilled water, at concentrations C_0 of NaCl equal to 0, 5, 20, 80, 160 and 314 gram of NaCl per litre of solution

– upper side: contact with air at 20°C and 60% of relative humidity

Drying process:

– both sides: contact with air at 35°C and a few percent of relative humidity (using silica gel).

3.1 Experimental method

The local variation of liquid content, related to the variation of the material density, induces a variation of the gamma beam attenuation. The emitter is Americium and the detector is NaI-crystal. The method is non-destructive, quantitative and gives local values point by point and does not need a heavy equipment. Moreover this experimental method has the advantage of being quick and accurate [7].

The local attenuation considering free, bound and crystallised salt is expressed by:

$$\frac{N_l}{N_0} = \exp\left(-(\mu_s \rho_0 + \mu_l \rho_l \theta_l + \mu_{salt} \rho_0 c_{salt})e\right) \quad (1)$$

N_0 and N_l are the numbers of incidental and transmitted gamma photons during Δt

μ_s is the mass attenuation coefficient of the dry solid (m^2/kg)

ρ_0 is the apparent density of dried material (kg/m^3)

μ_l is the mass attenuation coefficient of the solution (m^2/kg)

ρ_l is the density of the pore solution (kg/m^3)

θ_l is the liquid content (m^3/m^3)

μ_{salt} is the attenuation coefficient of the crystallised and bound salt (m^2/kg)

c_{salt} is the fraction of crystallised and bound sodium chloride (kg of NaCl/ kg mortar)

e is the thickness of the sample (m) ($e = 0.04$ m).

The bound and crystallised chloride ions are considered to occur with sodium to form sodium chloride at each point.

By comparison with the dry state:

$$\frac{N_{dry}}{N_l} \exp\left((\mu_l \rho_l \theta_l + \mu_{salt} \rho_0 c_{salt})e\right) \quad (2)$$

N_{dry} is the number of photons transmitted through the dried medium without salt.

In this relation the term $(\mu_l \rho_l)$ depends on the salt concentration of the pore solution C_i (in gram of NaCl/litre of solution). A separate experimental determination led to the relation [5]:

$$(\mu_l \rho_l)_{C_i} = 0.047 C_i + 19.71 \quad (3)$$

μ_{salt} is obtained by comparing attenuation for dry states after wettings, with pure water (N_{dry}), and salt solution ($N_{drysalt}$):

$$\mu_{salt} = \frac{1}{\rho_0 c_{salt} e} \ln \frac{N_{dry}}{N_{drysalt}} \quad (4)$$

c_{salt} is the total NaCl concentration determined experimentally at the end of drying (see section 5.2).

μ_{salt} has been evaluated at $0.04 m^2/kg$.

3.2 Statement of the problem

In our experiments only chloride and sodium are supposed to affect the attenuation. This means that the concentration of other possible species in the pore solution is considered as constant and does not change the results. The determination of θ_l from equation (2) requires the knowledge of c_{salt} and the salt concentration of the pore solution, C_i , which influences $\mu_l \rho_l$. Consequently the amount of free, bound and crystallised salt has to be known at each point. It is very difficult to identify experimentally the states of the various chloride by chemical analysis (see section 4.1). Only the total chloride concentration can be measured with a good accuracy, so that one piece of information is missing which is needed to determine the liquid content profiles. Some simplifying assumptions are proposed allowing the problem to be resolved for wetting and drying processes.

3.2.1 Wetting process

Mortars

The concentration of the pore solution C_i is always considered equal to the concentration of the solution used during wetting process C_0 . Then equation (2) is simplified ($c_{salt} = 0$). The liquid content is obtained by the relation:

$$\theta_l = \frac{1}{e(\mu_l \rho_l)_{C_0}} \ln \frac{N_{dry}}{N_l} \quad (5)$$

To estimate the validity of this assumption at the end of the wetting process with $C_0 = 314g/l$, two others profiles are calculated by taking into account the total salt concentration determined by chemical analysis :

- the first profile is calculated by considering all chloride ions in the pore solution. Nevertheless the concentration is limited to the saturation value ($C_i = C_{max} < 314g/l$);
- the second profile is calculated by considering that all chloride ions are bound ($C_i = 0 g/l$).

The relative differences between the concentrations obtained by these three ways are about 10% [5]. These

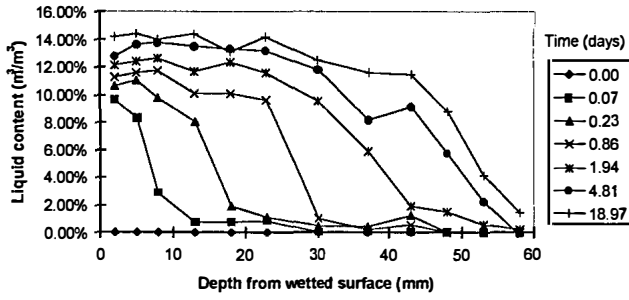


Fig. 1 – Liquid content profiles during wetting process with salt solution ($C_0 = 160 \text{ g/l}$). Mortar M1N.

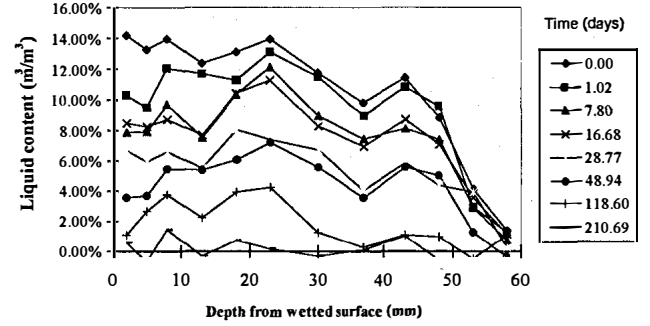


Fig. 2 – Liquid content profiles during drying process after wetting with salt solution ($C_0 = 160\text{g/l}$). Mortar M1N.

low differences are due to the fact that the variation of $\mu_1\rho_1$ can be compensated by the variation of $\mu_{\text{salt}}\rho_0c_{\text{salt}}$ (equation (2)). The mass of the sample measured by weighing is also compared to the total mass obtained by integration of the different moisture profiles at each time. The same relative differences of about 10% are found.

Fig. 1 shows liquid content profiles calculated during a wetting process with $C_0 = 160\text{g/l}$. The comparison of water content profiles obtained at the same time for different specimens of the same material shows a scattering of the results which is due to dispersion phenomena and is independent of the problem of the presence of salt. It means that the water front does not move regularly with the same profile. Consequently a strict comparison of profiles obtained at the same time is not so easy. Nevertheless the differences observed are confined to the profiles near the moisture front. The level of water content for the main part of the material is the same for different samples with relative good accuracy. This point explains why the integration of the profiles gives good results in comparison with experimental weighting.

Terracotta

As this material is inert [8], the previous assumption is correct. Then θ_1 is also calculated with equation (5).

3.2.2 Drying process

The determination of liquid content becomes more complex for drying because the crystallised salt part can be very significant especially at the end of drying.

Mortars

The total chloride profiles are evaluated at the end of wetting and drying for the different mortars and the different salt concentrations (C_0). The distribution of the chloride does not vary significantly between the beginning and the end of drying (see paragraph 5.2.1). So, the total chloride profiles are assumed to be known at each time of the drying process and the concentration of the pore solution is always considered to be equal to C_0 to calculate θ_1 . Equation (2) can be rewritten as:

$$\frac{1}{e} \ln \frac{N_{\text{dry}}}{N_1} = \theta_1 (\mu_1 \rho_1)_{C_0} + \mu_{\text{salt}} \rho_0 c_{\text{salt}} \quad (6)$$

As the concentration of the pore solution is supposed to be known ($C_1 = C_0$), c_{salt} can be expressed according to the total chloride concentration and θ_1 , by using the following relations:

$$c_{\text{salt}} = (c_{\text{tot}} - c_{\text{cf}}) \frac{M_{\text{NaCl}}}{M_{\text{Cl}}} \quad (7)$$

c_{tot} is the concentration of the total chloride (kg of chloride/kg of mortar)

c_{cf} is the concentration of the free chloride (kg of chloride/kg of mortar):

$$c_{\text{cf}} = \frac{C_0 \frac{M_{\text{Cl}}}{M_{\text{NaCl}}} \theta_1}{\rho_0} \quad (8)$$

M_{NaCl} is the molar mass of sodium chloride

M_{Cl} is the molar mass of chloride

Finally the knowledge of C_0 and c_{tot} makes it possible to calculate the liquid content with the following relation :

$$\frac{1}{e} \ln \frac{N_{\text{dry}}}{N_1} = \theta_1 \left((\mu_1 \rho_1)_{C_0} - \mu_{\text{salt}} C_0 \right) + \mu_{\text{salt}} \rho_0 c_{\text{tot}} \frac{M_{\text{NaCl}}}{M_{\text{Cl}}} \quad (9)$$

This liquid content can also be compared with those obtained by considering that the salt is totally in the pore solution or totally bound and crystallised.

The maximum differences are observed on samples soaked with a saturated solution of sodium chloride. The relative differences are about 10% at the beginning of drying and 20% at the end of drying [5]. Fig. 2 shows liquid content profiles calculated during drying process with equation (9), the same remark as for wetting (section 3.2.1) apply here also.

Terracotta

The chloride profiles are determined at the end of wetting and drying for various solutions (section 5.2.2). The

chloride ions move during drying in a significant way. We could calculate θ_i in the same way as for mortar but we would need the total chloride concentration profiles at each time, and this involves an extensive experimentation. The chemical analysis has been done only at the beginning and at the end of drying. An assumption has to be established to calculate the liquid content at each time: the concentration of the pore solution is always C_0 . Equation (5) is used, and the crystallised part of salt is then neglected. The profiles obtained in this way at the beginning and the end of drying can be compared with those calculated by considering that: (1) all the chloride are in the pore solution but with $C_i < 314$ g/l, and (2) all the chloride are crystallised $C_i = 0$ g/l.

The maximum relative differences are obtained for drying after wetting with a saturated solution. They are about 10% at the beginning. As the salt is strongly redistributed, large quantities of salt are present at the ends of the samples. The maximum relative differences are then obtained at these points at the end of drying and are about 40%. This is nevertheless acceptable because these relative differences only affect low liquid content.

4. CHLORIDE PROFILES

Two kinds of profiles are studied: the so-called calculated profiles and the measured profiles. The accuracy will be determined for each of them.

4.1 Experimental method

The method used is destructive. It consists in grinding and collecting powder from the sample with a profile grinder PF-1100 Mark II [9, 10]. The powder is analysed by a chemical method [11]. Total chloride is extracted by boiling powder in nitric acid for thirty minutes, while the free chloride is obtained by just shaking powder with acid solution for three minutes. The chloride ions concentrations are then measured by potentiometry using a titrated silver nitrate solution.

The technique should allow the different forms of chloride to be separated: that free in the pore solution, and that physically and chemically bound with the solid matrix. Determination of total chloride does not pose any problems. However the estimation of free chloride may include, at least partially, chloride initially physically adsorbed which moves from the solid matrix to the pore fluid during the procedure. Therefore, the amount of free chloride was overevaluated during our experiments. More generally, this difficulty

was pointed out by Tang and Nilsson in their evaluation of several methods intended to determine the different chloride profiles [12]. Considering these difficulties only total chloride profiles are measured.

4.2 The calculated chloride ion profiles

From the knowledge of θ_i , determined as explained in the previous sections, and assuming that the salt concentration of the pore solution is C_0 , it is possible to deduce chloride content profiles from liquid content profiles. These calculated profiles can be considered as total chloride profiles if all chloride would remain in the pore solution. This concentration called c_{ctot} (cal) is calculated according to the following relation:

$$c_{ctot}(cal) = \frac{C_0 \frac{M_{Cl}}{M_{NaCl}} \theta_i}{\rho_0} \quad (10)$$

The integration of these profiles represent the total mass of chloride which has penetrated into the material. The comparison between these latter profiles and those obtained by chemical analysis gives an indication of the fraction of bound and crystallised chloride ions.

5. EXPERIMENTAL RESULTS CONCERNING CHLORIDE PROFILES

5.1 During wetting

5.1.1 Mortars

Four sodium chloride solutions are used at the following concentrations: 20, 80, 160 and 314 g NaCl / litre of solution. Fig. 3 shows the measured (c_{ctot} (mea)) and calculated (c_{ctot} (cal)), chloride profiles during a wetting process with a solution of 80g/l for mortar M1N. Note that the profiles have been determined for different samples, except the calculated ones which correspond to gamma ray attenuation measurements on the same sample. Consequently we can expect a certain scattering of results.

The integration of the two profiles, at each time, should give the same amount of chloride content. From the results, we can conclude that a part of chloride is bound with the solid matrix in the first layers of the materials, 10 mm after 1 day and approximately 20 mm after 19 days. The differences observed between the two profiles after a very short time (1 h-45 mn) could be due to a strong interaction at the surface of the material which is not confirmed by the other profiles.

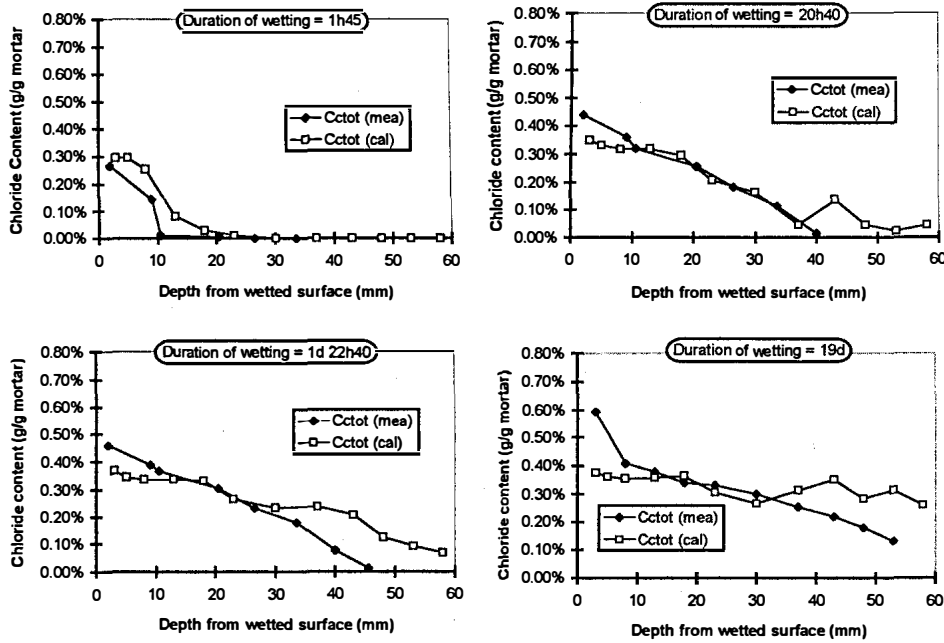


Fig.3 – Measured and calculated profiles during a wetting with the 80 g/l solution for M1N.

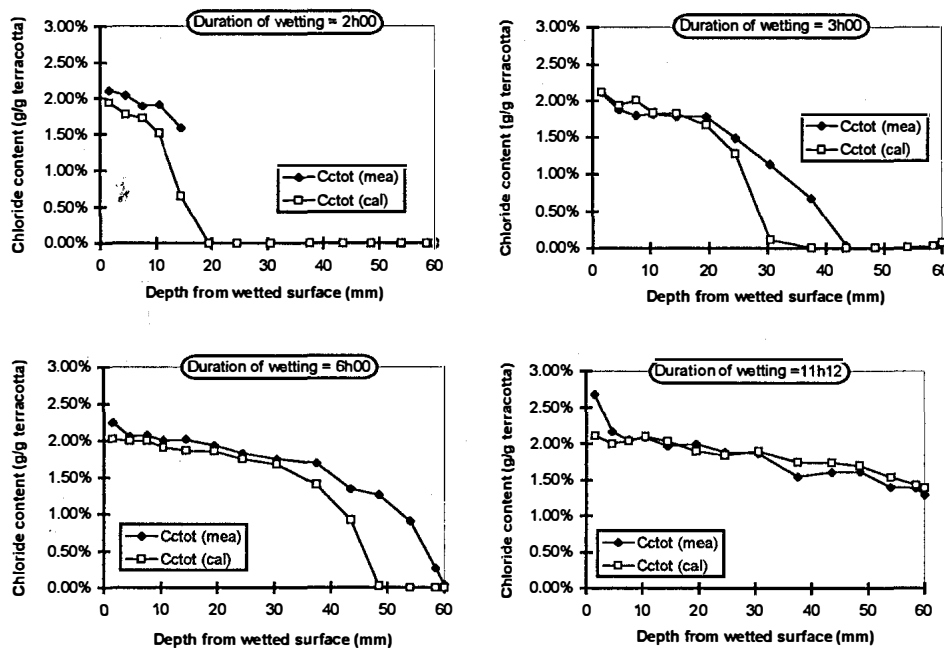


Fig. 4 – Measured and calculated profiles during a wetting with the 314 g/l solution for terracotta.

5.1.2 Terracotta

Three sodium chloride solutions are used with concentrations of 20, 80 and 314 g NaCl/litre of solution. Measured and calculated chloride profiles at different times from exposure, during wetting with saturated NaCl solutions are shown in Fig. 4. There is a quite good agreement between these profiles taking into account that the results are determined on different specimens. The differences observed between the two curves, near the wetting

front, can be attributed to the fact that the measurements in a region where the variations of water and chloride contents occur rapidly increases the scattering of experimental values.

This good agreement, which is obtained with all the solutions, confirms the lack of physical and chemical reactivity of the terracotta. The chloride ions follow the liquid water movement during the first water intake process. These results justify the assumption in section 3.2.1. The chloride content could be directly calculated from the liquid content (equation (10)) and vice versa.

5.2 After wetting and drying

5.2.1 Mortars

Fig. 5 shows the measured ($c_{ctot} (wet)$) and calculated ($c_{ctot} (cal)$) profiles at the end of the wetting process as well as the measured profiles ($c_{ctot} (dry)$) at the end of the drying process for the mortar M2N. The differences observed for the calculated profiles for the four concentrations are due to the dispersion phenomena as explained in the section 3.2.1.

However the values of $c_{ctot} (cal)$ near the wet surface can be considered as correct. We can see that these values are much lower than the measured. This difference demonstrates the importance of bound chloride in this part of the specimens.

The low difference between the measured profiles obtained with different specimens at the end of wetting and drying shows that the chloride do not move significantly with the liquid phase during drying. The method used in section 3.2.2 to calculate liquid content is then justified. Similar results are obtained for the mortar M1N.

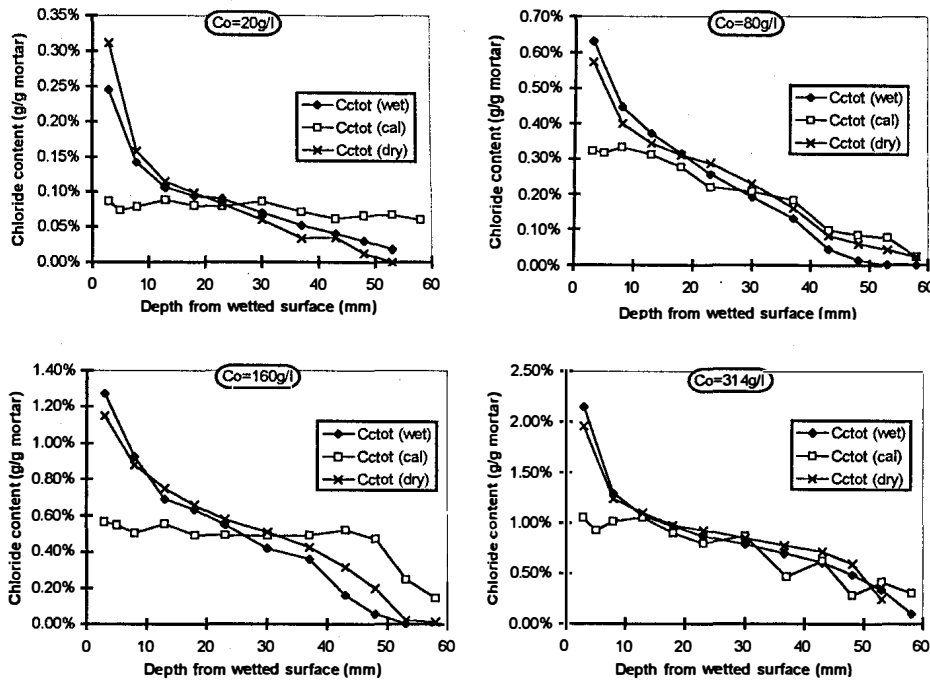


Fig. 5 – Measured and calculated profiles during wetting and drying for mortar M2N.

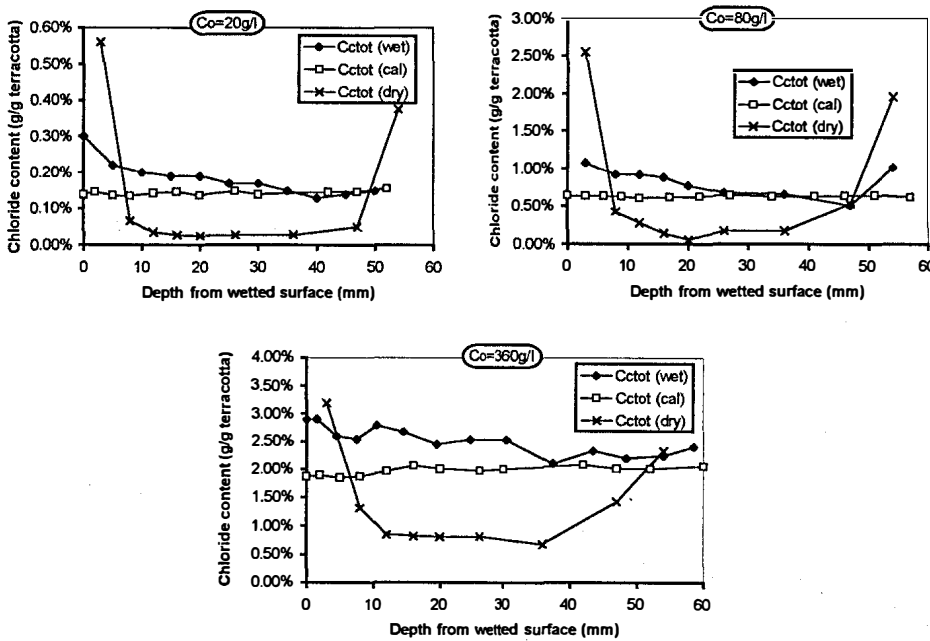


Fig. 6 – Measured and calculated profiles during wetting and drying for terracotta.

Table 1 – Comparison of the total chloride contents kg Cl/kg mortar measured and calculated from binding isotherms. Mortar M1N [4]

	20 g of NaCl/l	80 g of NaCl/l	160 g of NaCl/l	314 g of NaCl/l
$C_{ctot} (wet)$	0.0025	0.006	0.011	0.0155
C_{ctocf}	0.00087	0.0035	0.007	0.0137
C_{ctocb}	0.00163	0.00348	0.005	0.00734
$C_{ctot} = C_{cb} + C_{cf}$	0.0025	0.007	0.012	0.0210

5.2.2 Terracotta

For this material a great difference can be observed on Fig. 6 between the measured chloride profiles at the end of wetting and drying. This difference is attributed to a more important chloride redistribution than for mortars. Indeed the permeabilities to liquid and gaseous phases of the two kinds of materials as well as their binding capacity are not the same.

5.2.3 Comparison of bound chloride concentrations in saturated and unsaturated mortars

The chloride binding capacity of materials like mortars is illustrated by the interaction curves. These are determined in saturated conditions for various concentrations of the salt solution. They give the amount of chloride ions which can physico-chemically react with the solid matrix, and represent an equilibrium state. Table 1 gives values (c_{cb}) obtained for our materials by Francy [4]. Figs. 3 and 5 show the amount of bound chloride ions particularly important near the wet surface when the solution is moving. It is possible to compare the

amount of bound chloride ions for these two cases (equilibrium and transport process). Considering that the concentration of the pore solution is C_0 near the wet surface, the free chloride content, c_{cf} , can be calculated from saturated liquid content (equation (8)). The bound chloride concentrations, c_{cb} , are determined from the binding capacity. The total chloride content, c_{ctot} , is calculated by adding c_{cb} and c_{cf} . The measured total chloride content ($c_{ctot}(wet)$) is compared to c_{ctot} for mortar M1N and each concentration C_0 (Table 1).

During the wetting process the chloride needs more than two days to be bound, if we consider the external layers of the material in contact with the solution (Fig. 3), whereas binding is achieved after only few hours in saturated conditions [4]. Nevertheless the final values are the same after twenty days except for the saturated salt solution. The results are similar [5] for all mortars.

Consequently the chloride binding appears to be delayed during capillary suction. This conclusion is only valid for the external part of the material in contact with the salt solution, where the concentration of free chloride should be always equal to that of the wetting solution. The chloride ions that could be bound (according to the binding capacity in saturated materials) removed before equilibrium is reached. In fact, the flow could prevent them from reaching minor pores or from finding a free site on the pore wall.

6. MOISTURE DIFFUSIVITY

The spatial distribution of the liquid content and its variation with time are measured, as explained previously, during the processes. From them, a moisture transport property can be calculated, since during our experiments, isothermal and unidirectional conditions are used. Moreover, the influence of gravity and the gradient of total gas pressure are assumed to be negligible. The purpose of this paper is not to describe in detail the general theory of multicomponent mass transfer in porous media. The reader is invited to consult a very large bibliography on this subject [13-16]. The principle of the physical modelling of these complex phenomena, consists, starting from the expression of entropy source and in the frame of linear thermodynamics, in writing the fluxes of the species as a function of the gradient of the different potentials. A simplified formulation of the mass balance equation of the liquid phase consists in using the gradient

of the liquid concentration and using an apparent moisture diffusivity coefficient D_w [m²/s]. The mass balance equation for the solution take then the form:

$$\frac{\partial \theta_l}{\partial t} = \frac{\partial}{\partial x} \left\{ D_w(\theta_l, c_{cf}) \frac{\partial \theta_l}{\partial x} \right\} \quad (11)$$

The material is assumed to be homogeneous in order to define the single transport property: D_w . It is calculated from the liquid content profiles by using the Boltzmann method [17]. This property depends on the liquid and chloride contents. Moisture diffusivities are calculated with different concentrations C_0 to determine the influence of chloride content on them (see section 6.2).

6.1 Accuracy of moisture diffusivity determination.

If the material was macroscopically homogeneous, a constant diffusivity should be obtained, which would only depend on the liquid and chloride contents. However, scattering of moisture diffusivities occurs during transport processes even with distilled water, due to the heterogeneity of the materials, and represents dispersion effects.

Because of these problems, a mean moisture diffusivity has to be defined. It is calculated from the average of the diffusivities for a given liquid content.

6.2 Influence of chloride content on the moisture diffusivity

The mean diffusivities are illustrated for mortar M2N in Fig. 7 during the different transport processes. They do not seem to be influenced by chloride concentration of the wetting solution. Values of calculated diffusivities are between 10^{-10} and 10^{-8} m²/s during the drying process. Diffusivities are also smaller during drying than during

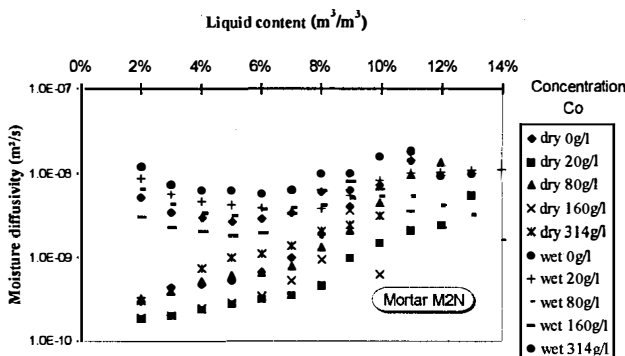


Fig. 7 – Moisture diffusivities during the wetting and drying processes for mortar M2N.

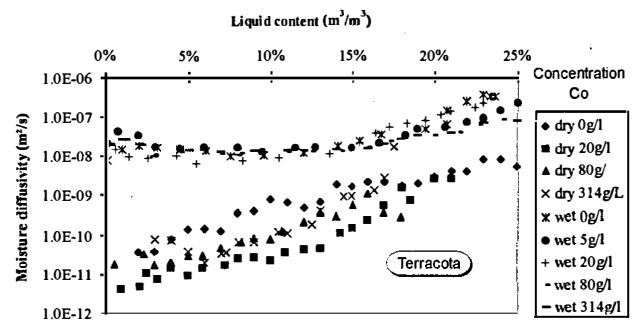


Fig. 8 – Moisture diffusivities for terracotta during the wetting and drying processes.

wetting except for high liquid contents (10^{-8} m²/s). Indeed at the beginning of drying, the liquid flow is as important as the end of wetting, the diffusivities and the solution concentration gradient being the same. The results are similar for all mortars. The moisture diffusivities for terra cotta are illustrated in Fig. 8 for each concentration C_0 and also show that the chloride content does not seem to influence the values. These results have, however, to be taken with caution considering scattering due to the heterogeneity within the material and the physical and chemical binding capacity of the materials.

7. CONCLUSIONS

The difficult question of how to perform the experimental determination of coupled transport of moisture and salt into unsaturated porous building materials are tackled in this work. The materials studied are chemically reactive or inert with respect to the chloride ions of the solution. The difficulty to determine the fractions of free, bound and crystallised ions implies some simplifying assumptions. They allow the liquid content distributions to be determined with an acceptable accuracy. Thus, the transport of moisture and chloride for the two types of building materials was analysed. The liquid flow seems to delay chloride binding during wetting process. Moreover the liquid content profiles suggest that moisture transport in materials is not affected by the presence of salt. These conclusions will be checked by numerical calculations.

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