

Assessing workability of mortar by means of rheological parameters and desorptivity

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ABSTRACT: Assessing the workability of mortars for restoration and more general purposes has been the subject of many standards and scientific publications over the last decades. A series of mortars made with different binders was brought to optimum water content for workability by an international group of masons. Two methods are proposed to assess the static yield stress and water retention of these fresh mortars, two key parameters of workability as defined by practitioners. Yield stress is measured with a vane test, originally designed for clayey soil, and found to be related to specific surface area of the binder. The increase of yield stress with resting time is quantified and the differences due to the effect of air entraining agent are demonstrated. Water retention is characterised by the desorptivity of the mortars and measured in a vacuum suction experiment. The setup is a modified version of an existing ASTM design. Both methods are complementary to assess the defined workability parameters for different mortars and eventually can be an aid to design suitable mortars for specific purposes.

1 INTRODUCTION

When selecting or designing a suitable restoration mortar for a specific situation, the first concern is for a material which will be compatible with the existing fabric in appearance, composition (type of binder and aggregate), mechanical, hygric and thermal behaviour (Van Balen et al. 2005). These properties, which refer to the hardened state, are interrelated in a complex manner to the fresh properties of the mortar, such as the rheology, water permeability and adhesion to the substrate. Workability is a general term to cover these fresh properties, and is considered by masons as highly important. A mortar should be readily moulded in the voids to be filled up, and then gain stiffness to keep its shape after application. The kneading water should be sucked out of the mortar by the substrate at a sufficient rate to provide this stiffening, but not too fast as to avoid difficulties in shaping the mortar body properly, and not too much as to avoid an insufficient water content for full hydration of the hydraulic binder.

Rheology in the most broad sense describes the deformation of matter, and more specifically of everything which lies between a perfect elastic solid and an ideal Newtonian fluid. The rheology of mortar in general is highly complex because mortar is a granu-

lar paste of questionable homogeneity with a solid fraction far above the usual in the discipline. It applies the laws of continuum mechanics, which is feasible if a sufficiently large scale is considered, and homogeneity guaranteed (Coussot 2005, Wallevik 2003). Existing methods to assess rheology of mortars are reviewed in (Banfill 2005). They can be divided into rotational methods, compressive rheology and some practice-oriented or empirical tests methods. This paper focuses on a rotational method, based on the principle of a rotating vane, which induces yielding in the circumscribing cylindrical plane. This method is particularly suited for materials which are sensitive to slip and segregation at the cylinder surface (Lidell & Boger 1996).

Existing models that describe the rheological behaviour of mortar all have a yield stress parameter, and one or more parameters quantifying the stress-strain relationship under laminar flow. It is generally recognized that the behaviour of mortar changes with time, and that thixotropy should be taken into account to properly describe these phenomena (Banfill 2005). Nevertheless, the most widely used models assume the existence of one single steady state flow curve, e.g. following the Bingham model: $\tau = \tau_0 + \mu\dot{\gamma}$, with τ the shear stress, τ_0 the shear

stress at zero shear rate, μ the plastic viscosity and $\dot{\gamma}$ the shear rate. We will show that mortar has in reality a dynamic yield stress, which is dependent on time effects, deformation history and normal force. Thus the values obtained in our vane test depend on resting time and the time scale of the imposed stress or strain. Normal force is assumed to be low in the test setup, so that the influence can be neglected. The study of the time scale of the experiment is restrained by a constant rotational velocity driving mechanism, so that possible effects on yield stress are not taken into account here. The specific apparatus used in this study was originally designed for lab measurements of clayey soil and has recently been proposed for use on binder pastes and mortars (Hendrickx et al. 2006, Bauer et al. 2007).

The transport of water through a volume of semi-solid mortar slurry or paste can be described using the same principles as those applied for porous solids. A complete modelling of flow from mortar to substrate using FEM could be done when the complete water retention curve and hygroscopic curve of both materials are known, as well as the initial state and boundary conditions. At present, established experimental methods do not allow the mortar to be characterised to this extent. Alternatively, a simple sharp front model to describe the dewatering of a slurry based on filter cake theory, is derived in (Hall and Hoff 2002) and experimentally validated in (Collier et al. 2007). This model presents the desorptivity R of the wet mortar at constant capillary suction of the substrate as the main parameter to characterise the mortar. R is determined experimentally for different types of mortar using a simple pressure filter used in oil industry (Carter et al. 2003). In analogy to the sorptivity of bricks, which has been accepted to be a reliable measure for its capillary absorption, desorptivity of mortar provides also more useful information than the well-known standardised tests, which measure a quantity of water absorbed by filter paper sheets (EN 413-2:2005) or filter paper plates (EN 459-2:2001). The apparatus described in ASTM C91, using vacuum suction, is adapted in the experiments described below, to be used in a procedure to measure desorptivity.

2 MATERIALS AND METHODS

2.1 Mortar composition

The description of the binders are listed in Table 1, with a standardised denomination is possible. The chemical properties measured by X-ray fluorescence

(XRF) and specific surface area (SSA) measured with the BET method are given in Table 2. Binder 2 and 3 are prefabricated admixed products; binder 7 is a lab mix of the ordinary Portland cement (binder 6), plasticizer (modified stearic acid in powder) and an air entraining agent (fatty acid / polyglycol solution). The dosage was 0.4% and 0.1% of the binder weight. The aggregate is a medium siliceous quarry sand 0/0.5 (0/1) (EN 13139:2002) from a quarry in Zutendaal, Belgium (Figure 1).

Table 1. Description of binders

N°	Description
1	calcic lime CL90S
2	calcic lime CL90S (EN 459-1:2001) (75%) with hydraulic binder (15%) and pozzolana (10%)
3	calcic lime CL90S (EN 459-1:2001) with air entraining agent
4	calcic lime CL90S (EN 459-1:2001) (66.7%) and ordinary Portland cement CEM I 42.5 R (33.3%)
5	natural hydraulic lime NHL 5 (EN 459-1:2001)
6	ordinary Portland cement CEM I 42.5 R
7	ordinary Portland cement CEM I 42.5 R with admixtures: plasticizer and air entraining agent

Table 2. Chemical properties (XRF) and specific surface area (BET) of binders

	1	2	3	4	5	6
CaO (%)	96.6	75.1	96.3	75.6	51.2	63
MgO (%)	0.94	1.88	1.05	1.49	1.68	1.74
Al ₂ O ₃ (%)	0.059	1.640	0.072	2.6	4.9	4.6
SiO ₂ (%)	0.144	6.2	0.19	9.6	16.6	16.7
Fe ₂ O ₃ (%)	0.048	1.55	0.063	2.7	2.1	3.6
CO ₂ (%)	2.0	10.6	2.0	3.0	19.8	2.9
SO ₃ (%)	0.10	1.84	0.18	2.80	2.10	4.50
SSA (m ² /g)	12.0	5.7	11.9	4.5	3.5	1.3

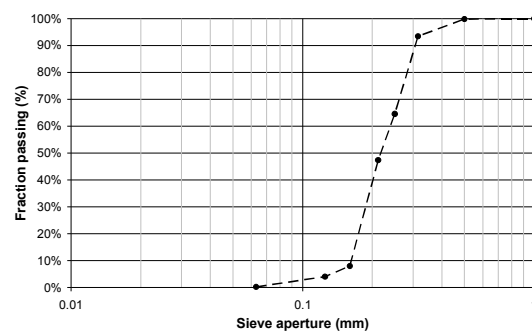


Figure 1. Grading of the aggregate (Zutendaal sand).

The compositions of the batches are given in Table 3 by their binder to aggregate weight ratio (B:A) and water to binder weight ratio (W:B). B:A values are derived from practical experience and are comparable to values given in various national standards. For the prefabricated blended binders (binder 2 and 3), they are in agreement with the producer's guidelines. W:B values have been determined for optimum workability by a panel of six masons

(Hendrickx et al. in press). Mixing is done using a Hobart planetary mixer at low speed: 1.5 min mixing, 30 s homogenising, 2 min mixing. All the components are added in the beginning: first the water, then half of the aggregate, then the binder and at last the remaining half of the aggregate.

Table 3. Mortar composition in weight ratios

Binder:	1	2	3	4	5	6	7
B:A (kg/kg)	0.13	0.20	0.13	0.21	0.21	0.25	0.25
W:B (kg/kg)	2.02	0.90	1.48	1.00	1.13	0.93	0.79

2.2 Yield stress experiments

Yield stress was measured with a vane apparatus for clayey soil (ASTM D4648) (Figure 2). It measures the maximum torque necessary for a vane ($D \times H$), immersed in a sample container with mortar, when rotation at constant is initiated. Previous research has demonstrated that container dimensions of three times the dimensions of the vane, are sufficient to avoid any influence of edge effects (Lidell & Boger 1996). The mechanism is designed in a way that the inner scale indicates ϕ_1 , the amount of deflection over the spring and thus the magnitude of the torque, and the inner scale indicates ϕ_2 , the angle over which the vane has rotated inside the sample. From the moment that the mortar yields and the vane starts to move quickly, ϕ_1 remains constant and ϕ_2 starts to increase at a rate ωt .

It can be seen that $\phi_1 = \omega t - \phi_2$. The torque exerted on the vane can be expressed as: $T = B \cdot \phi_1$ with B a constant of the calibrated spring, indicating the torque per unit of rotation. Hence the shear stress in a cylindrical plane circumscribing the vane can be calculated as T/K , with the vane constant K equal to:

$$K = \frac{\pi D^2 H}{2 \cdot 10^6} \left(1 + \frac{D}{3H} \right) \quad (1)$$

The assumption that shear occurs exactly in this place is a good approximation (Lidell & Boger 1996). The homogeneity of the material close to this shear plane is discussed further. A typical test shows three stages (Figure 3). In the initial stage there is an increase of ϕ_2 due to a small repositioning of the vane and rearrangement of grains in the mortar. In the second stage, the increase of both ϕ_1 and ϕ_2 are constant. In the third stage, the spring angle reaches a maximum and the residual angle increases more, until the material yields: this last phenomenon can not be derived from the scale reading, due to the configuration apparatus. All mortars were measured

with a spring constant $B=380^\circ/\text{Nm}$, except binder 4, for which a vane with $B=260^\circ/\text{Nm}$ was used. The vane has dimensions $D=H=45\text{mm}$. Rotation speed ω was constant and equal to $9.2^\circ/\text{min}$.

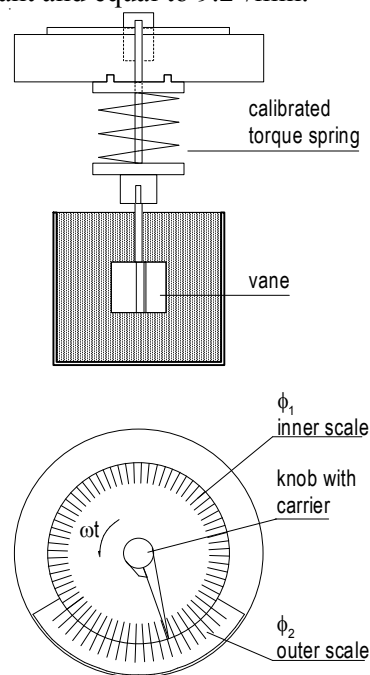


Figure 2. Working principles of the soil vane test.

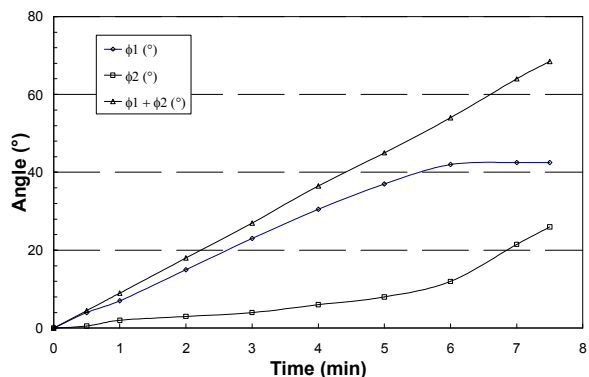


Figure 3. Typical course of an experiment at constant $\omega = 9.2^\circ/\text{min}$ (mortar with binder 7).

2.3 Desorptivity experiments

The test setup from ASTM C91 was adapted to contain a larger sample volume. The sample is introduced in a perforated dish on top of a wetted filter paper. The sample height is increased with respect to the standardised version from 20 to 60 mm (Figure 4). This enables to have sufficient data points when suction is applied for identifying the initial linear regime of water loss with the square root of time (Hall and Hoff 2002):

$$i = R\sqrt{t} \quad (2)$$

with i the amount of evacuated water, expressed in mm water column, R the desorptivity and t time. The pressure difference $p_1 - p_2$ is generated by a vacuum pump, in stead of the pressure cell used in (Carter et

al. 2003). The mass of the dish with the filter paper and sample is measured at the beginning of the test, after wiping the bottom of the dish quickly with a dry cloth. The vacuum pressure is applied to the sample via the funnel by turning a valve. The seal between the funnel and the dish is made with a layer of silicone, which is applied to the funnel and moulded in contact with the dish. Sealing with petroleum or light grease is avoided because it complicates the removal and accurate weighing of the dish at different times. Measurements were done after 1, 4, 9, 25, 36, 49, 64 and 81 minutes, until the level of the vacuum could not be sustained any more due to gas breakthrough. Gas breakthrough occurs increasingly from a certain point onwards, but initially, the pressure drop over the sample can be kept constant by increasing the pump suction. The repeated procedure of closing the valve, removing and wiping the dish, weighing, replacing, and reopening the valve was done in 45 s.

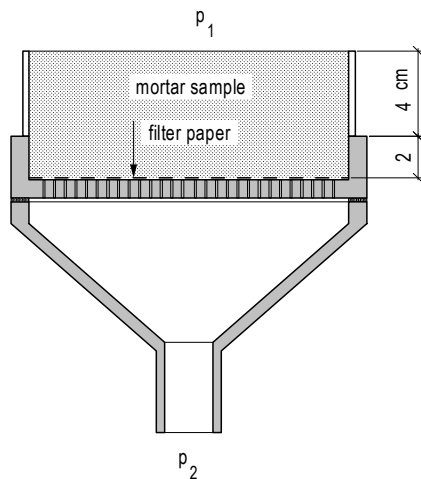


Figure 4. Geometry of the desorptivity test. The section of the dish is circular, diameter 155mm.

It is assumed that the pressure, applied through the perforations and the filter paper, is equally divided over the bottom surface of the sample. We further assume that the mortar's solid fraction does not segregate during the time of the test and that the pressure drop over the filter paper is negligible compared to the pressure drop over the sample. The maximum pressure difference that can be realised in this way is atmospheric pressure. If higher pressures are needed, use of a pressure cell is an alternative.

3 RESULTS AND DISCUSSION

3.1 Rheological experiments

The results of the vane test are represented as a function of the actual time when yielding occurs (Figure

5 and 6). By doing so, the duration of the test, which is often 10 min or more, is taken into account. The values range from 20 to 80 Pa shortly after mixing, and rise above 100 Pa after 1 hour. A distinct behaviour is observed for air entrained mortars (15 to 30 % air content, binders 3 and 7) and non air entrained mortars (3 to 7 % air content, binders 1, 4, 5 and 6). In the first group the increase of yield stress is limited during the first half hour of resting time, and becomes more important during the second half hour. The second group shows an adverse effect, with a large increase in the early stage and a smaller increase after 40 min. In both groups a large drop of yield stress is obtained by pre-shearing the mortar. This means that only a part of the stiffening is irreversible.

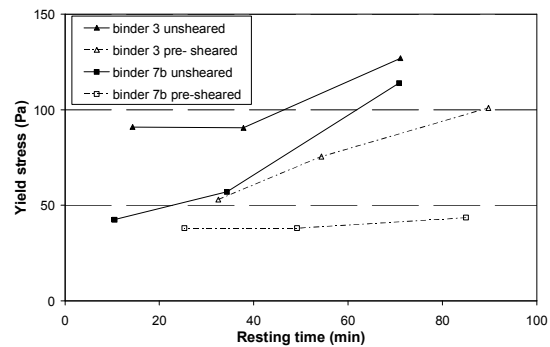


Figure 5. Yield stress unsheared and pre-sheared for air entrained mortars.

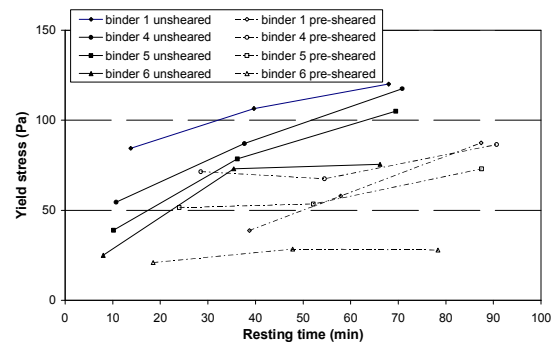


Figure 6. Yield stress unsheared and pre-sheared for non air entrained mortars.

For comparison of the different mortars, yield stress values after 30 and 60 min of resting time were calculated by linear interpolation (Table 4). It has to be stressed that these values are valid only for the (relatively low) rotation rate in this setup and that strictly spoken, this method is neither rate controlled, nor exactly stress controlled. The results are to be interpreted as a good basis for comparison: changing the test parameters will influence the order of magnitude of the absolute numbers.

Segregation of mortar by rising of water towards the surface ("bleeding") was observed during the tests, especially in mortars with high desorptivity (see below). This implies that, for those mortars, at least

part of the yield stress increase is due to increasing solid fraction, while for the other mortars, we can speak of a real increase of internal structure.

When comparing the mortars without entrained air, a correlation can be noted between the yield stress and the specific surface area (SSA) of the binder (Figure 7). The practical meaning of this correlation is that, even when W:B is optimised for each of the mortars, the differences in behaviour remain measurable. Different binders have a different optimum yield stress: binders with larger SSA are judged workable by masons even at higher measured yield stress.

Table 4. Yield stress values after 30 and 60 min of resting time.

Binder:	1	3	4	5	6	7
τ_y vane, t=30s (Pa)	98	91	78	69	64	54
τ_y vane, t=60s (Pa)	116	115	108	97	75	97

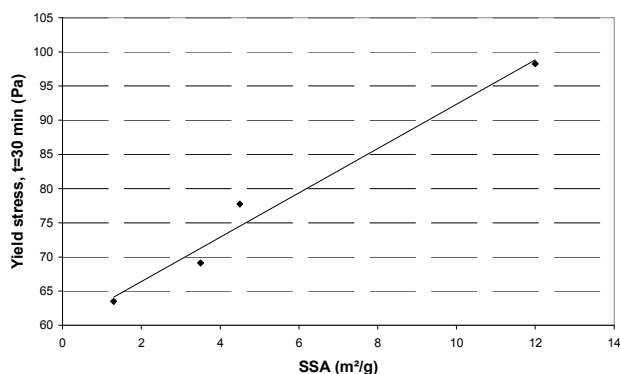


Figure 7. Yield stress measured with vane at 30 min of resting time as a function of SSA of binder powder.

3.2 Water retention experiments

The amount of water removed from the mortar at constant vacuum pressure increases proportionally to the square root of time until a certain point, from where it tends to an asymptotic value (Figure 8). This asymptotic value corresponds to the equilibrium water content of the material for the applied vacuum suction.

The observed behaviour can be fitted by least squares to a linear function for the earlier part and an exponential function for the later part. The transition point between both parts is determined by visual assessment of the curves. The slope of the linear part corresponds to the desorptivity R of the mortar, as defined in equation 2. The later data points can be fitted to an exponential equation of the form:

$$y' = A(1 - \exp(-Bx')) \quad (3)$$

where (x', y') are the coordinates relative to the transition point with the linear part. $A + x' = \mu(i_{eq})$ is an estimator for i_{eq} , the equilibrium value of i after a long time. Figure 9 gives an example for mortar with

binder 1. From $\mu(i_{eq})$, the geometry of the test and the mortar compositions, we can calculate $\mu(w_{eq})$, the estimated equilibrium water content (in kg/m^3) of the mortar after a long time at the applied vacuum pressure.

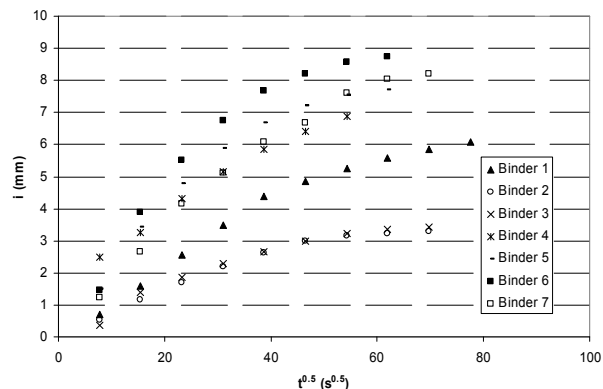


Figure 8. Quantity of desorbed water as a function of time for mortars with different binders.

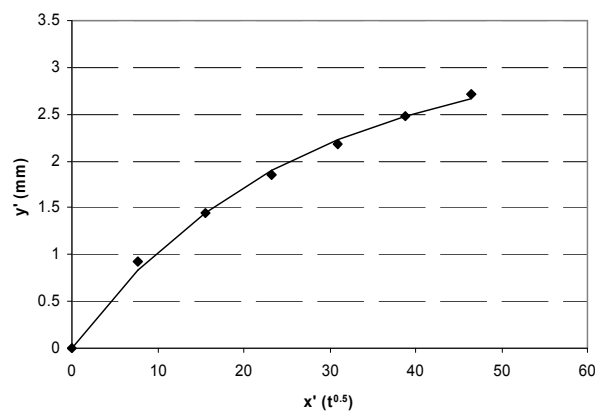


Figure 9. Fitting of data to an exponential curve; data of mortar with binder 1, vacuum pressure 200mbar.

Table 5 gives the measured values and calculated parameters for the 7 tested mortar types, as well as the values of water retention (WR) obtained for these mortars in the standardised tests with filter paper and filter paper plates.

Table 5. Numerical results of standardised water retention tests, desorptivity measurements and regression analysis.

Binder:	1	2	3	4	5	6	7
WR filter paper (%)	93	94	95	93	85	87	88
WR filter plates (%)	83	93	92	81	66	63	78
R ($\text{mm/s}^{0.5}$)	0.13	0.08	0.14	0.12	0.26	0.32	0.20
A ($\text{mm/s}^{0.5}$)	3.19	1.87	2.80	3.59	5.21	5.78	5.24
B ($10^{-3} \text{ s}^{-0.5}$)	38.7	50.0	27.4	29.8	44.2	48.3	30.0
w_{in} (kg/m^3)	353	178	217	292	326	313	236
$\mu(w_{eq})$ (kg/m^3)	237	116	144	139	175	145	72

For increasing pressures with the same mortar, we observe both a higher initial increase R and a higher final equilibrium water content w_{eq} (Figure 10 and 11). The increase of desorptivity can be empirically fitted to a power law function of the shape:

$$R = C \cdot P^n \quad (4)$$

where C and n are the empirical constants. This corresponds to the findings of other authors (Green et al. 1999, Carter et al. 2003). The exponent is related in filter cake theory to the compressibility of the filter cake, 0.50 corresponding to the condition of an incompressible cake (Green et al. 1999).

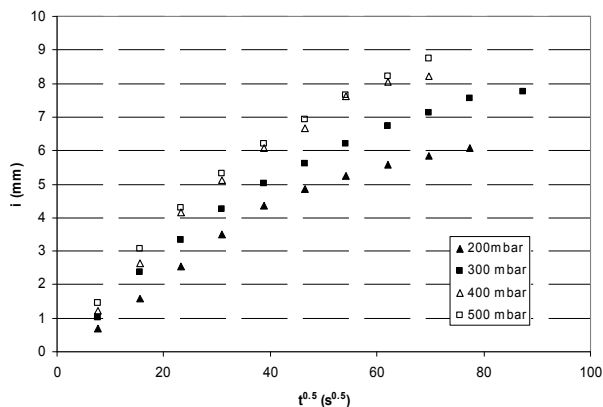


Figure 10. Quantity of desorbed water as a function of time for different vacuum pressures and mortar with binder 1.

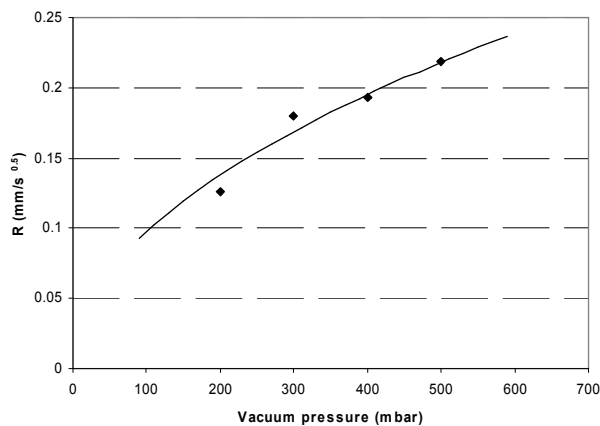


Figure 11. Desorptivity of mortar with binder 1 (air lime) as a function of applied vacuum pressure, fitted with to power law function: $R = 9.75 \cdot 10^{-3} \cdot P^{0.5}$.

The results of the standardised water retention tests using filter paper sheets (EN 413-2:2005) could not be correlated in a meaningful way to data obtained from other experiments. Observations of the importance of the transition resistance between the different sheets confirm that this test cannot yield a physically relevant value. The filter paper plate test on the other hand was found to give a good indication for the desorptivity under vacuum (Figure 12).

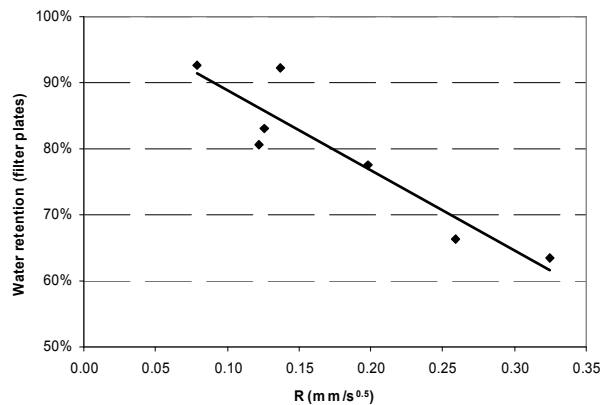


Figure 12. Water retention measured with filter paper plates as a function of calculated desorptivity.

4 CONCLUSIONS

Two important aspects of mortar workability, its rheology and water retention, have been discussed for a series of mortars with different binders. The yield stress has been measured successfully with the laboratory vane test for clayey soil. Results however remain valid only for the applied rotation rate, vane dimensions and spring characteristics. The influence of these parameters is still to be investigated. The differences in absolute value between the mortars' yield stress and the different behaviour in time for air entrained and non air entrained mortars have been demonstrated. This test has the important advantage over the flow table test or penetration tests, that it gives a meaningful physical quantity as a result, which can easily be interpreted in terms of practical use. The advantage over scientific rheometers is that the instrument has lower cost, is available in most soil mechanical labs and has a suitable torque range and dimension.

The same advantages are valid for the desorptivity experiment with the vacuum pump: it starts from a small adaptation of a standard setup which is available in a large number of building material labs. The actual process of suction by a porous brick resembles more to this setup, than to the technique with a pressure cell used by other researchers. The disadvantage is the rather laborious test procedure and the restriction of applied suction to atmospheric pressure. The result is more meaningful than the results of the traditional standardised tests with filter paper of filter paper plates, because it yields a rate of water loss in stead of one single point. Further more the data can be used to estimate the equilibrium value at the applied vacuum pressure, which allows to characterize the water retention curve of the mortar in the covered pressure range. The weakness remains that for high desorptivity mortars, a significant incli-

nation of the curve is observed already after the second point, which causes the calculated desorptivity value to be less accurate, deviating towards the lower end. The measured desorptivity value is found to be inversely proportional to the water retention value measured by filter paper plates. This means that this simple test is a suitable method to estimate desorptivity.

The observed differences between the mortars can be related to the value of the SSA and to the effect of air entraining agents in the mortar. SSA is inversely proportional to desorptivity, and addition of air entraining agent has a decreasing effect, though of less importance. There is evidence for a negative correlation between mortar desorptivity and yield stress, through their dependence on the SSA. A higher SSA gives lower desorptivity and higher yield stress for mortars without air entraining agent. Caution is needed however to interpret the results because of the different B:A values of the mortars tested. A similar trend was found for the mortars with air entraining agent. These findings illustrate the important effect of air content and SSA on both yield stress and desorptivity, two of the most important aspects in assessing the workability of mortar.

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