

Dependence of Capillary Properties of Contemporary Clinker Bricks on Their Microstructure

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Abstract. Contemporary clinker bricks are applied for outer layers of walls built from other materials and walls which should have high durability and aesthetic qualities. The intended effect depends not only on the mortar applied but also on clinker properties. Traditional macroscopic tests do not allow to predict clinker behaviour in contact with mortars and external environment. The basic information for this issue is open porosity of material. It defines the material ability to absorb liquids: rain water (through the face wall surface) and grout from mortar (through base surface). The main capillary flow goes on in pores with diameters from 300 to 3000nm. It is possible to define pore distribution and their size using the Mercury Intrusion Porosimetry method. The aim of these research is evaluation of clinker brick capillary properties (initial water absorption and capillary rate) and analysis of differences in microstructure of the face and base wall of a product. Detailed results allowed to show pore distribution in function of their diameters and definition of pore amount responsible for capillary flow. Based on relation between volume function differential and pore diameter, a differential distribution curve was obtained which helped to determine the dominant diameters. The results obtained let us state that face wall of bricks was characterized with the lowest material density and open porosity. In this layer (most burnt) part of pores could be closed by locally appearing liquid phase during brick burning. Thus density is lower comparing to other part of the product.

1. Introduction

Traditional macroscopic tests do not allow for precise anticipating of clinker behaviour in contact with mortars and external environment. Mechanical, thermal properties, and permeability and conductivity of materials are strictly associated with their structure and porosity. Also capillarity, drying and freezing processes of water depend on material microstructure. Material microstructure is the basic information in this area [1,2]. Generally, it is assumed that microstructure is characterized with the following values: general porosity, porosity distribution, tortuosity of pores, specific area, and permeability. Gas porosimetry is used for porosity evaluation in the range of micro- and macropores (<2nm – 60nm). In case of meso- and macropores (3nm – 360000nm) the mercury porosimetry is appropriate. The mercury porosimetry uses the following properties of Mercury:

- the liquid does not moisten material (surface tension (293K) - 428 mN/m-1) that is why in contact with porous material it does not enter material's pores,
- it shows little changeability of moistening angle (from 112° to 142°). Thus mercury porosimetry is a gradual filling of pore structure under controlled pressure. From pressure and intrusion volume the instrument generates data with Washburn equation [3]:



$$D = \frac{4\gamma \cos \theta}{P} \quad (1)$$

where:

- D – pore diameter [nm],
- γ – surface tension [mN/m],
- θ – moistening angle [°],
- P – pressure [bar].

Thus the higher is precision of pressure and mercury volume the better will be the result definition. Open porosity is affected by connected pores with different shapes and sizes which are classified according to medium radius as:

- micropores, < 2nm
- mesopores, from 2 to 50nm
- macropores, > 50nm.

The biggest adsorption abilities are possessed by macropores – they constitute the basic medium for material's sorption properties. Their size is comparative to sizes of adsorbed particles. In mesopores humidity transport goes on while sorption happens on their surface. Macropores fulfil mainly transporting function in humidity transfer to meso- and macropores. If pores surface significantly affects the hydrodynamics of fluid in their interior, such porous material has capillarity properties, and pores are capillaries [4]. It is assumed that the most intense capillary flow happens in pores in the range of 300 to 3000nm.

2. Test material and scope of tests

Input material for tests were 12 pieces of full clinker bricks (250x120x65mm) selected randomly. In order to define the capillary absorption coefficient, 6 pieces of bricks were cut out with a diamond saw. One half of a brick was a sample for water absorption test through base surface and the second one through face surface.

The tests of capillary absorption were performed according to guidelines for standards [5, 6] on halves of samples (beams and clinker elements). The samples dried to solid mass were placed in a cuvette with a grid which enabled to immerse them in water at constant depth of 5 +1mm. After soaking time (1, 4, 9, 16, 25, 36, 49... min respectively) the samples were taken out, their immersed surface was dried and they were weighed. Basing on mass of water absorbed by a sample it was defined:

- Initial water absorption in $w \text{ kg}/(\text{m}^2\sqrt{\text{min}})$

$$C_{wis} = \frac{m_{so,s} - m_{dry,s}}{A_s \cdot \sqrt{t}} \quad (2)$$

- Water absorption resulted from capillarity in $\text{kg}/(\text{m}^2\sqrt{\text{min}})$

$$C_{ws} = \frac{m_{so,s} - m_{dry,s}}{A_s \cdot \sqrt{t_{so}}} \quad (3)$$

where: $m_{so,s}$ - sample mass after soaking in time t , [g], $m_{dry,s}$ - sample mass after drying, [g], t_{so} - soaking time, [min], A_s - total area of a sample immersed in water

- for a clinker brick with base-side capillarity $A_s = 0,120 \times 0,125 = 1,5 \cdot 10^{-2} \text{ m}^2$,
- for a clinker brick with face-side capillarity $A_s = 0,65 \times 0,125 = 8,125 \cdot 10^{-3} \text{ m}^2$.

The other six samples were used for microstructure tests. From each pieces there were six samples selected from the following areas:

- face layer,
- base layer.

The samples in form of a blocks (Figure 1) were cut out using a diamond saw cooled with water. The cutting residue was cleaned with pressurized water. Such prepared material was dried to solid mass.

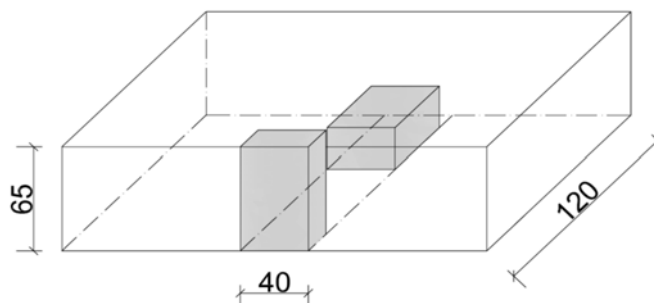


Figure 1. Clinker brick - preparation for tests

The samples created in this way were used to evaluate of volume density with macroscopic method. In the next stage part of samples were cut in order to adopt them to required volume of a penetrometer in porosimetry tests. The rest of them were use in definition of specific density.

3. Tests results and discussions

3.1. Test of capillary absorption coefficient

Results obtained for 6 samples for capillary absorption (Figure 2) indicate that single samples are characterized with significant differences in capillarity through the base area. It is particularly evident in initial phase when dynamic of humidity rising is very high. For samples with capillary rise through the face area the range of results is low as well as the dynamics of capillary rise in analogical period. In extended time of tests, the results indicate lowering of capillarity process through base area and maintaining constant velocity in case of capillarity through face area.

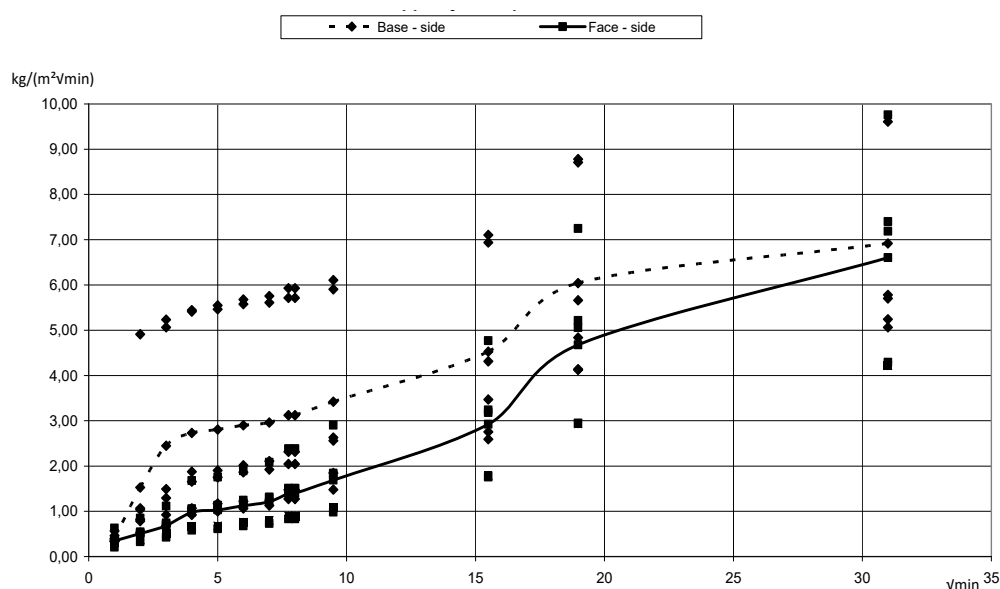


Figure 2. Capillary absorption coefficient for clinker bricks

3.2. Macroscopic tests

Macroscopic tests were performed on regular samples of material cut from clinker bricks. For definition of total porosity, the value of specific density was used. The results are listed in Table 1.

Table 1. Macroscopic tests of material samples – average values

Name	Sample cut location	
	Face layer	Base layer
Volume density [g/cm³]	2.03	2.06
Density [g/cm³]	2.72	
Total porosity [%]	25.33	24.26

3.3. Material microstructure test

As results of mercury porosimetry tests, the basic structure parameters were obtained (Table 2):

- volume density understood as relation of dry porous mass to total volume of a porous sample,
- skeleton density taking into account closed pores, defined as relation of porous mass of dry material to skeleton volume,
- open porosity – relation of pore volume contained in a given volume of porous body to its total volume,
- volume of capillary pores (300-3000nm)
- tortuosity as a geometric value defining average route length of a liquid particle moving from one point of porous medium to another, related to distance between these points along a straight line).

Table 2. Macroscopic tests of material samples – average values.

Name	Sample cut location	
	Face layer	Base layer
Volume density [g/cm³]	2.02	2.08
Skeleton density taking into account closed pores [g/cm³]	2.65	2.68
Open porosity [%]	21.58	22.58
Pore volume 300-3000nm	11.55	11.75
Tortuosity	1.57	1.51

Detailed results enabled to show pore volume distribution in function of their diameters (Figure 3). Based on relation between volume function differential and pore diameters, there was obtained the pore distribution differential curve which enabled to set dominant pore diameters. Based on integral curve of pore size distribution (Figure 4) it was defined the share of meso- and macropores in tested material samples – Table 3.

Table 3. Percentage share of pores for selected samples – average values

Sample	Pore volume [%]			Total porosity
	Micropores < 2nm	Mesopores 2-50nm	Macropores > 50nm	
Face layer	3.75	8.87	12.71	25.33
Base layer	1.68	9.30	13.28	24.26

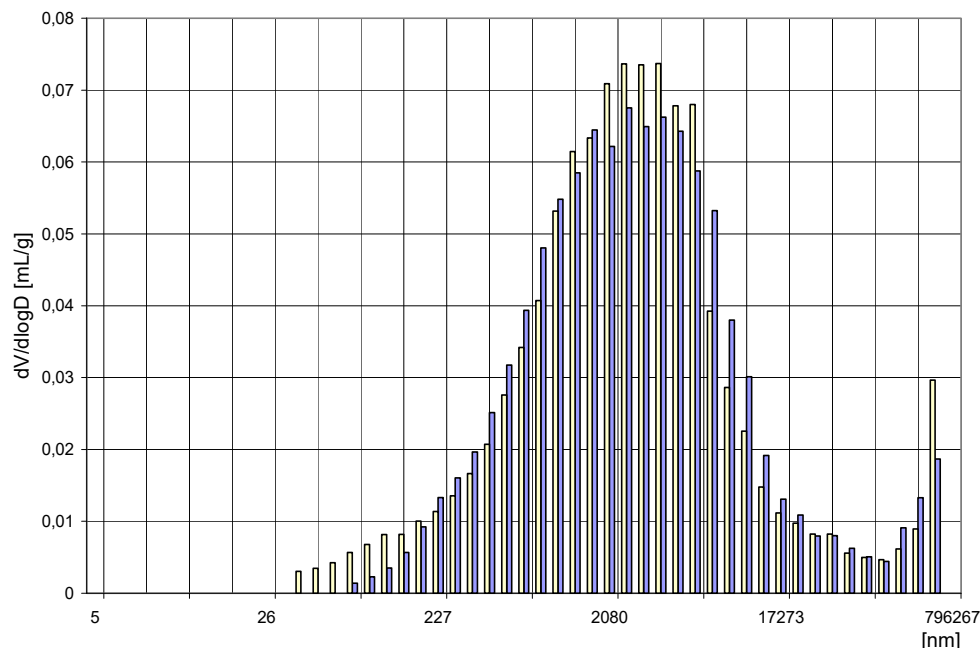


Figure 3. Differential curve of pore size distribution

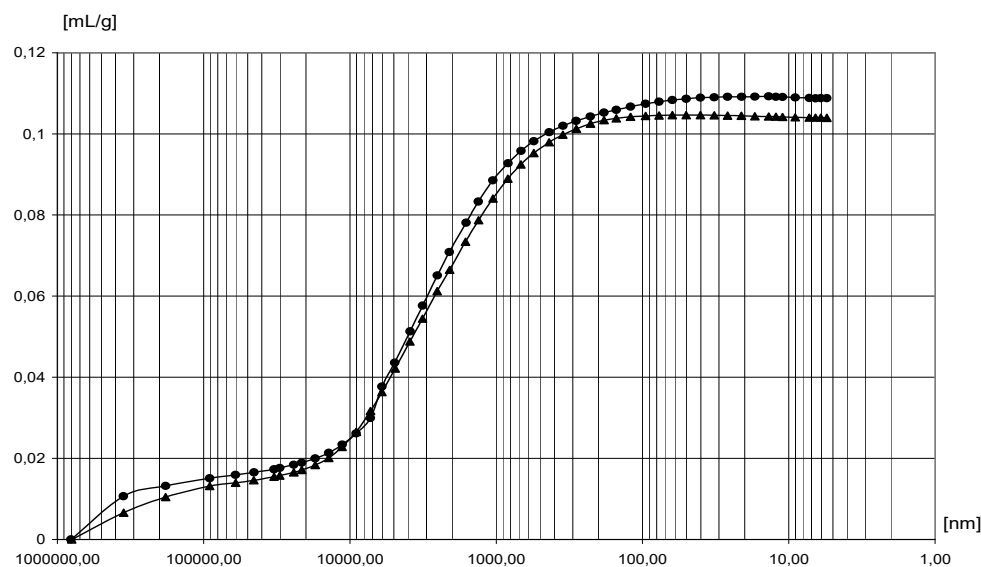


Figure 4. Integral curve of pore size distribution

4. Discussion of results and summary

Based on initial tests performed there were noticed differences in clinker structure within a single product. In the face layer there is lower volume density and consequently – higher total porosity. In the face layer the percentage share of pore volume in the range of meso- and macropores is lower than in other selected areas. However, macropores share calculated as a difference between total porosity and porosity obtained from porosimetry tests is significantly higher in relation to other layers. This is associated with the process of clinker brick burning – part of pores in this area can get closed by locally appearing liquid phase. Thus lower density value appears.

Attention must be paid to the fact that results from porosimetry tests of structure parameters concern only open pores in the range above 2nm. This means that both skeleton density, and consequently the material porosity is measured with an error resulting from macropores presence. The share of pore volume responsible for capillary flow both for face layer as well as base layer is similar and amounts to 11.55 and 11.75%. These pores are responsible for capillary absorption which is reflected in tests results showed in point 2.1 of test results. In the base layer pores with bigger diameters dominate which results in dynamic capillarity of water in the first period of tests. In case of clinker it is a desired effect because it causes a proper joint. However, in face layer appear pores with lower diameters which result in slowing of capillary penetration of water. This feature results in limiting of wall moistening caused by rain.

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