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HYGRIC PERFORMANCE OF CONTEMPORARY AND HISTORICAL CREAMIC BRICKS

HIGROSKOPOWA ANALIZA WSPÓŁCZESNYCH ORAZ HISTORYCZNYCH CEGIEŁ CERAMICZNYCH

Abstract

This paper analyses the effect of microstructure, mineralogical composition and possible salt contamination on parameters of moisture accumulation and transport. Analysis is done for three types of contemporary and two types of historical bricks.

Keywords: *sorption isotherm*, *water vapour permeability*, *ceramic brick*, *microstructure*, *salt*

Streszczenie

W artykule przedstawiono wpływ mikrostruktury, składu mineralnego oraz możliwego występowania soli na akumulację i transport wilgoci. Analizy zostały przedstawione dla trzech typów elementów ceramicznych: materiału współcześnie stosowanego oraz dwóch stosowanych w przeszłości.

Słowa kluczowe: *sorpcja*, *przepuszczalność pary wodnej*, *cegła ceramiczna*, *mikrostruktura*, *sól*

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1. Introduction

Moisture transport and accumulation parameters are key factors in evaluation of hygro--thermal performance of building structures. Generally, moisture transport and accumulation parameters depend on actual pore structure of the building material. In cases when moisture accumulation only can be attributed to physiosorption of water vapour on the material pore system and the material does not contain very fine pores, inaccessible to nitrogen (e.g. common ceramic bricks), the hygroscopic moisture content (HMC) can be evaluated from nitrogen sorption [5, 6]. However, in case of historical bricks, which have been exposed for a long--time to interaction with chemicals from the surrounding air or water, contamination with water soluble salts is very probable. Presence of hygroscopic salts significantly increases the HMC of ceramic bricks [6, 8]. Therefore measurement of the HMC of brick samples can be used as an indication of the salt contamination [7]. In case of material contaminated with single salt relative reliable results, also on the quantity of the present salt, can be obtained from the HMC because of the linear relation between HMC and salt content. However, in case when a mix of unknown salts is present, as it is usual in reality, the relation between the HMC and the salt content is not clear [7, 8].

In this work, parameters of moisture accumulation and transport, namely water vapour sorption and water vapour permeability moisture dependence for three types of contemporary and two types of historical ceramic bricks were determined. Based on the obtained results, the effects of microstructure, mineralogical composition and possible salt contamination on the parameters of moisture accumulation and transport were analysed.

2. Materials and methods

The tested samples were three types of contemporary burnt clay bricks produced by Slovak manufacturers and two types of historical brick (nineteenth century) from western part of Slovakia.

The tested bricks were characterised by following basic parameters: bulk density, density, total porosity, open porosity and capillary moisture content. The bulk density was calculated from volume and mass of the dried out specimens (oven drying at 105°C). The density of ceramic body was determined by gas (N_2) pycnometer Pentapyc 5200e. The total porosity was calculated from density and bulk density. The open porosity was determined from water saturation test, the capillary moisture content from one dimensional time-controlled capillary water uptake experiment.

Mineral composition was determined by X-ray diffractograf Philips. The device is equipped with goniometer PW 1050 and Ni filter, and use Cu-Kα radiation in the range of angles 4–62° 2Θ. X-ray tube works at 35 kV and 20 mA. Velocity of record is 2°/min 2Θ. The data were analysed using software BedeZDS Search/Match.

Nitrogen adsorption measurements were performed with the volumetric ASAP2400 instrument, enabling isotherms in the 0.01–0.98 relative pressure range to be obtained. Prior to the measurement the samples were degassed overnight at 150°C and 2 Pa. The specific surface area of pores was determined by the BET method.

Water vapour sorption isotherms were determined by the standard gravimetric desiccator method, which consists of conditioning the samples in desiccators under constant relative humidity (RH) and temperature until the static equilibrium is achieved [2]. The samples were oven dried beforehand at 105°C.

The water vapour permeability of the brick specimens was measured by the standard cup method [3]. Mass of the samples was checked before and after each cup test. At the end of all cup measurements the samples were oven dried at temperature of 105°C in order to determine actual moisture contents of the samples. All water vapour permeability measurements were performed in an air-conditioned room at temperature of 23 ± 0.5°C and RH equal to 53 ± 1%. The required RH inside the cup was established by using silica-gel, water or saturated salt solutions.

3. Results and discussion

The basic material parameters and the BET specific surface area of the tested bricks are presented in Table 1. The crystal phases identified in brick specimens by X-ray diffractometry are summarized in Table 2. As follows from the data, the tested bricks had practically identical mineral composition, with an exception in the case of illite, which was identified in bricks D, BA1 and BA2 and of montmorillonite, identified in brick BA2. The presence of illite and montmorillonite indicates an imperfect heating regime in the furnace, common for historical bricks [1]. The X-ray diffractometry results did not confirm presence of hygroscopic salts. This result does not fully exclude the possibility of salt contamination but it guarantees that in case of contamination the amount of particular salt is below of 1 wt %.

Figure 1 and 2 show the measured nitrogen and water vapour adsorption isotherms for tested bricks. In case of all three contemporary bricks very good correspondence between nitrogen and water vapour adsorption isotherms was found. The nitrogen results showed significant dissimilarity of the tested bricks microstructure. Correspondingly the HMC of brick D was practically negligible up to the RH of 94%. The determined HMC of brick S was noticeable but relatively low while the HMC of brick P was quite significant (Fig. 1). The good agreement between the water and nitrogen adsorption curves indicated that the contemporary bricks did not contain hygroscopic salts.

Table 1

Basic material parameters and BET specific surface area of tested bricks

Brick	Bulk density [kg/m^3]	Density [kg/m^3]	Total porosity [–]	Open porosity [–]	Capillary moisture content [m^3/m^3]	Specific surface area [m^2/g]
P	1370	2752	0.51	0.42	0.37	14.2
S	1460	2788	0.50	0.44	0.39	4.7
D	1780	2752	0.35	0.30	0.24	1.13
BA1	1710	2708	0.37	0.34	0.29	4.4
BA2	1700	2790	0.39	0.36	0.25	9.3

Crystal phases identified in tested bricks

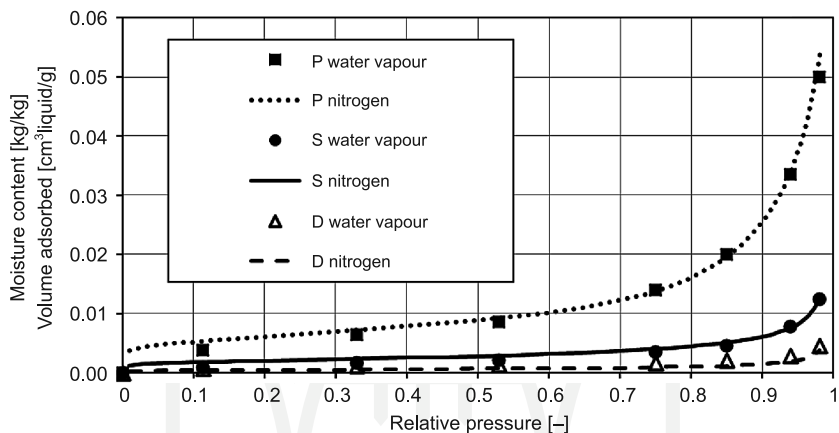| Brick	Identified phases
D	Quartz, illite, muscovite, feldspars, enstatite (Fe, Mg), hematite
P	Quartz, muscovite, feldspars, enstatite (Fe, Mg), hematite
S	Quartz, muscovite, feldspar, esseneite (Ca, Fe), enstatite, hematite
BA1	Quartz, illite, muskovite, feldspars, calcite, hematite
BA2	Quartz, Illite, montmorillonite, muscovite, calcite, feldspars, hematite

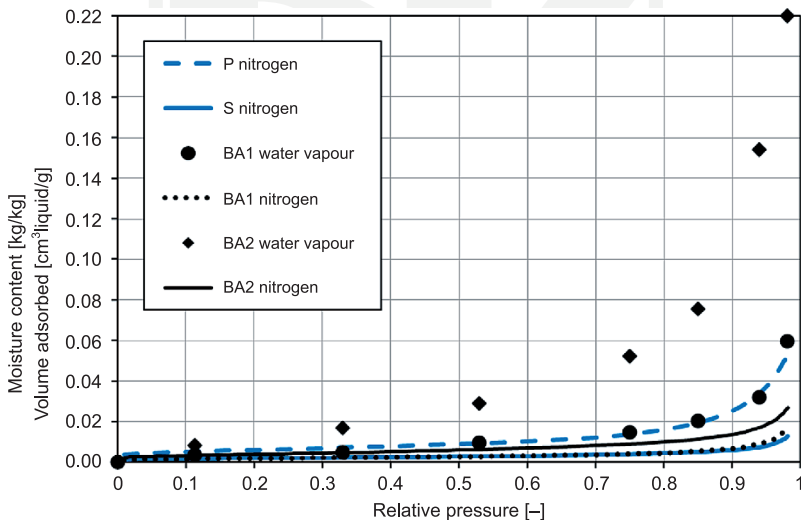Fig. 1. Water vapour and nitrogen adsorption isotherms of contemporary bricks D, P and S

Fig. 2. Water vapour and nitrogen adsorption isotherms of historical bricks BA1 and BA2 compared with nitrogen adsorption isotherms of contemporary bricks P and S

Identified concentration of salt anions and evaluated total salt concentration (historical bricks)

Brick	Cl^- [kg/kg]	SO_4^{2-} [kg/kg]	NO_3^- [kg/kg]	Total salt amount [kg/kg]
BA1	0.0014	0.0014	0.0035	0.011–0.015
BA2	0.0038	0.0002	0.01	0.023–0.03

The nitrogen adsorption curve of historical brick BA1 was practically identical with the one of brick S, the nitrogen adsorption curve of historical brick BA2 was somewhere between the ones of brick S and P. However, the obtained HMC values of the historical bricks were significantly higher than the corresponding nitrogen isotherms. This discrepancy could result from their possible salt contamination. Therefore the BA1 and BA2 samples were put to qualitative, and after receiving positive results, also quantitative chemical analysis for determination of chloride, sulphate and nitrate anions. The analysis confirmed medium chloride, low sulphate and high nitrate salt contents in BA1sample. In case of brick BA2 the analysis showed high chloride, practically negligible sulphate and very high nitrate salt content in the samples (Tab. 3). With the aim to estimate the total amount of hygroscopic salts in the samples, the presence of ammonium salts was tested but result of the test was negative. Taking into account occurrence of the most probable salts (NaCl, KCl, $CaCl_2$, $CaSO_4$, Na_2SO_4, K_2SO_4, KNO_3 and $NaNO_3$ for BA1 and NaCl, KCl, $CaCl_2$, KNO_3 and $NaNO_3$ for BA2) and the actual concentration of the salt anions (Tab. 3), total amount of the salts was evaluated. The minimum total salt amount was evaluated supposing only the salts with the lowest ratio of molar mass per one salt anion are present ($CaCl_2$, $CaSO_4$, $NaNO_3$). The maximum total salt amount was correspondingly calculated from the salts with the highest value of molar mass per one salt anion (KCl, K_2SO_4, KNO_3). The evaluated total salt amount was from 1.1 to 1.5 wt % in case of BA1 and from 2.3 to 3.0 wt % in case of BA2 (Tab. 3). However it is necessary to point out that the values presented are only approximate. Nevertheless it is possible to note that the estimated considerably higher total salt amount of BA2 is in good agreement with the significantly higher HMC of this brick. From the estimated values of the total salt amount and the results of X-ray diffractometry also follows that mixture of at least two different salts was present in bricks BA1 and BA2.

The measurements of the water vapour permeability of contemporary bricks were performed under different RH differences (0–53%, 11.3–53%, 33–53%, 53–75.4%, 53–84.7%, 53–94% and 53–100%) with the aim to get water vapour resistance factor values for the broadest possible range of moisture contents, including the transition zone from the higher value of water vapour resistance factor, corresponding to the water vapour diffusion without the surface diffusion, to the lower value, following from cumulated effect of the water vapour diffusion and the surface diffusion. The water vapour permeability of the historical bricks was measured under RH differences of 0–53%, 53–94% and 53–100% (Fig. 3). Analysis of the measured water vapour resistance factor/moisture content relations of contemporary bricks has shown that they can be approximated by following hyperbolic function:

$$\mu = \mu_0 - a \cdot \frac{\exp\left(\dfrac{u_m - u_{m1}}{u_{m2}}\right) - \exp\left(\dfrac{u_{m1} - u_m}{u_{m2}}\right)}{\exp\left(\dfrac{u_m - u_{m1}}{u_{m2}}\right) + \exp\left(\dfrac{u_{m1} - u_m}{u_{m2}}\right)} \qquad (1)$$

where:

u_m – moisture content [kg/kg],

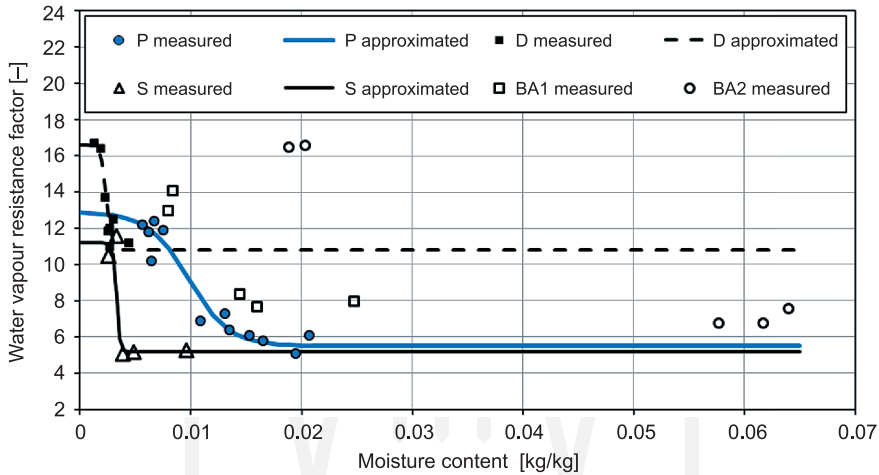μ_0, a, u_{m1}, u_{m2} – parameters.

Fig. 3. Water vapour resistance factor vs. moisture content for contemporary bricks P, S and D and historical bricks BA1 and BA2

The mean moisture content at which the transition from higher to the lower value of water vapour resistance factor occurs corresponds to parameter u_{m1}. The width of the transition zone is expressed by the parameter u_{m2} and corresponds to value of $2 \cdot u_{m2}$. The parameter μ_0 expresses the mean value of water vapour resistance factor and value of $2a$ corresponds to the difference between maximum and minimum vapour resistance factor value. The applied parameters are in Tab. 4.

Table 4

Applied parameters of hyperbolic function (1)

Brick	μ_0	a	u_{m1}	u_{m2}
P	9.2	3.7	0.0098	0.0037
S	8.2	3.0	0.0033	0.0003
D	13.7	2.9	0.0024	0.0005

During the dry cup measurements of saline samples an efflorescence occurrence is often reported on the dry sides of samples [4, 6]. The created salt crust then causes an increase

of the dry cup water vapour resistance factor value. However, the salt crust formation depends on many factors (e.g. type of salt or salts, salt concentration, way of drying) and therefore is hardly predictable. In case of the measured historical bricks a weak efflorescence was noticed in case of one sample but noticeable salt crust was not created. The presence of salt has caused slightly higher values of vapour resistance factor of BA2 samples (Fig. 3, 4).
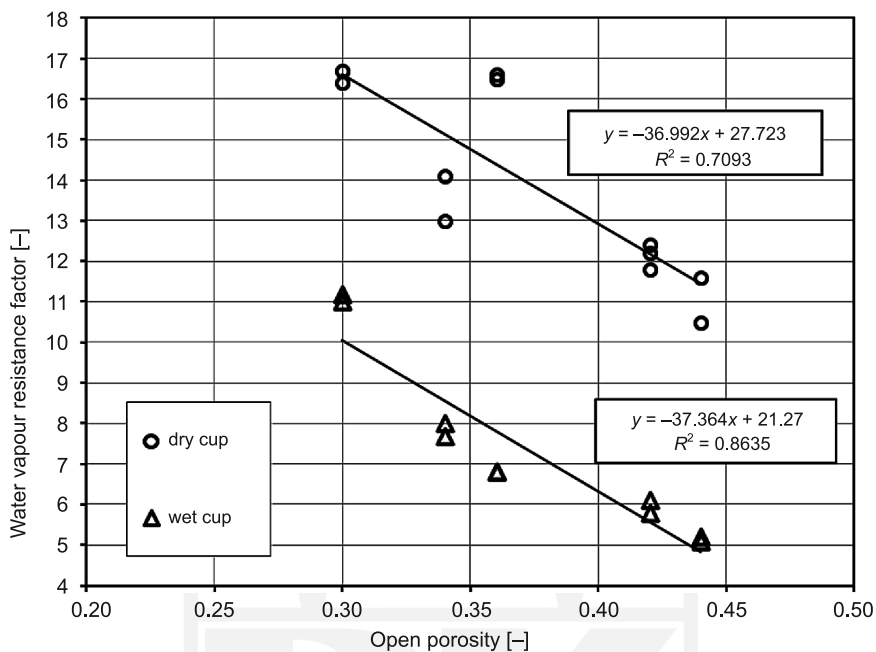

Fig. 4. Dry cup and wet cup water vapour resistance factor values vs. open porosity. Presented linear approximations involve all tested bricks

In case of contemporary bricks good correlation between the dry cup water vapour resistance factor values (measured at RH difference 0–53%) and open porosity of the bricks was obtained (Fig. 4). Compared to the contemporary salt free bricks the corresponding moisture contents of historical bricks are shifted towards the higher values (Fig. 3). This effect was significant especially for wet cup measurements of historical brick BA2 and corresponded to the high salt content as was determined. On the other hand it seems that this salt bounded moisture had minor effect on water vapour and surface diffusion. For wet cup measurements (RH difference 53–100%) relative good correlation between the water vapour resistance factor and open porosity was obtained for the whole set of tested bricks (Fig. 3, 4).

4. Conclusions

The microstructure, mineralogical composition, hygroscopic moisture content and water vapour permeability moisture dependence were determined for three types of contemporary and two types of historical ceramic bricks.

The tested bricks had very similar mineralogical composition. The microstructure of the tested contemporary bricks differed significantly. Correspondingly the potential hygroscopicity of the bricks varied from the practically negligible to the relative significant. The determined potential hygroscopicity of the historical bricks pore structure did not exceed the range determined for contemporary bricks.

Good compatibility between the nitrogen and water vapour adsorption was confirmed for salt free bricks. On the other hand it was showed that the discrepancy between nitrogen and water vapour results could indicate a contamination by hygroscopic salts.

On the whole, the water vapour permeability of the tested bricks was directly proportional to the open porosity of the bricks. However in case of historical brick BA2 the present amount of salts caused slightly lower dry cup water vapour permeability.

R e f e r e n c e s

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