The Effect of Firing Temperature on the Irreversible Expansion, Water Absorption and Pore Structure of a Brick Body During Freeze-Thaw Cycles

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The paper deals with the monitoring of brick body in the process of volumetric freezing and thawing. The samples were fired at temperatures of 900, 1000 and 1060 °C. Attention is focused on monitoring of the irreversible expansion, water absorption and pore structure of a brick body. We found that in all cases the endpoints take place continuously, where the amount firing temperature plays a crucial role. The greatest influence of freeze/thaw cycles on the change of the pore structure was also observed at the lowest temperature. The change of the pore system during the freeze-thaw cycles occurs in such a way, that the pore volume of small pores further decreases and conversely, the pore volume of large pores increases. The knowledge gained can be used not only in the production of new but also in predicting the remaining durability of older clay roofing tiles.

Keywords: brick body, clay roofing tile, frost resistance, irreversible expansion, water absorption, pore structure.

1. INTRODUCTION

An important property of clay roofing tiles or facing bricks is the frost resistance, which is considered as the resistance of a fired brick body in a moist condition against an alternating action of water and frost. In this case, we encounter three factors working together.

The first factor is the change in the state of matter from water to ice, it can cause a well-known expansion. The ice formation in porous material at the same time depends from the capillary diameter and extent of negative temperatures. With the decreasing diameter of capillary a negative temperature decreases and this relationship can be expressed by an exponential function [1]. The whole process of water freezing takes place in the form of rubble-ice in the larger capillaries being pressed into the smaller capillaries, the pressure increases and gradually leads to the formation of microcracks [2]. The size of the pressure in the smallest capillaries can achieve a value up to 200 MPa [3].

The second factor: the melting of ice leads to an increase of the expansion due to higher thermal linear expansion coefficient of ice as has the brick body itself. (thermal linear expansion coefficient of ice is \( \alpha \approx 50 \times 10^{-6} \, \text{K}^{-1} \) and of brick body only \( \alpha \approx 5,5 \times 10^{-6} \, \text{K}^{-1} \) [4]). The pressure from ice and melting ice applied on the pore walls causes an irreversible expansion of the brick body after each freeze-thaw cycle [5–7]. Concurrently a cumulative effect occurs until a total collapse is achieved [5, 8, 9].

The third factor: the moisture expansion of a brick body, which can induce reversible or irreversible volume changes [10–13]. The fact that the irreversible changes are of a larger dimension can pose serious problems especially for large scale products. The irreversible moisture expansion occurs mainly in brick raw material with a relatively high content of clay minerals fired at temperatures in the range about 600°C to 1050°C. It is a spontaneous process of the water reaction with the non-crystalline phase of the brick body, where the irreversible volume changes can usually exceed the value of 0.8 mm/m [13].

An intensive monitoring of the relationship between pore structure and frost resistance was carried out in the last 30 years out, where most attention was devoted to the distribution of the pores before the first freezing cycle [14–19]. Smaller attention is devoted to the distribution of pores after the end a certain number of freeze-thaw cycles, for example in the evaluation of old brick products (roofing tiles and bricks) [15, 20, 21].

It was found that the frost resistance of brick body can be better presented through the distribution of pores rather than just by its porosity, which is expressed through the pore volume or through the water absorption [22]. The actual distribution of pores is, however, influenced by several factors, such as firing temperature, mineralogical and granulometric composition of raw materials and the like [22–28].

Undoubtedly the most important impact has the firing temperature or the presence of a flux [29–33]. Insufficient firing temperature will have created a wide range of pores but it achieves only a low level of frost resistance [34]. Similarly, it is disadvantageous to have a very narrow spectrum of pores [35]. Many authors have tried to define most preferred composition of the pore structure, however neither proposal has been able to perfectly represent the...
relationship between the pore structure and frost resistance for different types of brick products [36–42].

As shown in the work of Raimondo et al. [41], a common feature of high frost resistance of a brick body on the basis of previous knowledge is a large number of pores larger than 3 mm. This requirement should satisfy the conclusions, as stated by Šveda [22, 29, 32]. The author has found with the various raw materials and at different firing temperatures that the higher value of the median pore radius, it is also higher frost resistance of brick body. The relationship between the median pore radius and frost resistance can be expressed by a mathematical function. This positive effect of the median pore radius on the frost resistance can see also in the works [30, 43].

So far, there has been a little studied area, which monitors the gradual changes in pore structure of a brick body namely during the freezing cycles. Therefore we can presume that the explanation of these changes could have a positive influence for example:

• in obtaining a high frost resistance brick product;
• in predicting the remaining durability of clay roofing tiles or facing bricks;
• in assessment the information about firing temperatures on the old roofing tiles or facing bricks.

2. EXPERIMENTAL

We have used the raw material from a plant, which produces roofing tiles. It is characterized by quaternary sediments, which are represented by layers of clay and silty loam soils of eolian origin. Its chemical and granulometric composition are shown in Table 1 and Figure 1. From the mineralogical point of view it is the montmorillonite/illite, almost without the calcium carbonates.

Table 1. Chemical composition of clay

<table>
<thead>
<tr>
<th>Chemical composition, %</th>
<th>Clay</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>71.81</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>13.37</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>5.28</td>
</tr>
<tr>
<td>CaO</td>
<td>0.46</td>
</tr>
<tr>
<td>MgO</td>
<td>0.91</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.45</td>
</tr>
<tr>
<td>K₂O</td>
<td>1.46</td>
</tr>
<tr>
<td>CaCO₃</td>
<td>0.63</td>
</tr>
<tr>
<td>MgCO₃</td>
<td>0.74</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>4.71</td>
</tr>
</tbody>
</table>

The X-ray analysis of the samples (Fig. 3) fired at temperatures from 900 °C to 1060 °C in addition to the dominant quartz contains some mineral forms from the feldspar group, micaceous species and hematite.

The test samples were prepared by cutting unfired roofing tiles lengthwise in three parts, which were brought from brickworks. They were consequently fired in an electric furnace at temperatures of 900, 1000 and 1060 °C. The firing scheme is shown in Figure 2. For the determination of an irreversible expansion we used the edge part of the beaver tail clay tile. For the determination of changes in the pore structure during freeze-thaw cycles we used only the middle part. For each firing temperature was used 5 pieces of beaver tail clay tiles.

The irreversible expansion was determined with a deformeter, where the starting distance between two dots on a face of the test specimen was 200 mm. The measurement accuracy was 0.01 %.

Frost resistance of the saturated water samples was determined though standard freeze-thaw cycles: 20 hours at air temperature of –18 ±2 °C and 4 hours in water.
temperature of 18 ±2°C, i.e. one freeze cycle was carried out in 24 hours.

The pore structure of the brick body was determined by high-pressure mercury porosimeter (Thermo Finnigan Pascal 240, firm Thermo Scientific). The test samples for this measurement during freeze/thaw cycles were gradually taken from the same batch.

4. RESULTS AND DISCUSSION

The course of irreversible moisture expansion at all three firing temperatures is shown in Figure 4. In the first ten days of measurement, a rapid increase in the expansion we observe at all three temperatures. The fastest increase was carried out at temperature of 900 °C, where the highest final values were also achieved.

After a rapid increase in expansion follows a slowdown and it goes continually until the breach of the sample. In the firing temperatures of 1000 °C and 1060 °C is this slowing down observed even during storage in an aqueous medium, but at temperature of 900 °C up during the freeze/thaw cycles.

With increasing firing temperature there occurs a significant shift in creation of the first visible crack, see Fig. 4. Similar results as with irreversible expansion we obtained well as with water absorption. The values of irreversible expansion are in accordance with authors [13].

The highest absorption after 21 days storage in water was achieved at the firing temperature of 900 °C and its steady state occurs in a rather short time, see Figure 5. With the increasing firing temperature, the values of water absorption do decrease, but their steady state shift due to a more difficult access of water into the pore structure of a brick body.

When comparing the water absorption values between samples stored only in water (W) and samples exposed to freeze/thaw cycles (F/T) we can see that at the firing temperature of 900 °C their values are virtually identical up to 75 days of storage in water. Then, water absorption values for the samples F/T start to exceed the water absorption values of the samples W until their breach. The water absorption values of samples subjected to freeze/thaw cycles (F/T) are lower on a long-term basis with increasing firing temperature as compared with samples (W) and only very slowly approach these values (e.g. at the temperature of 1000 °C it is after nearly 350 days of storage in water).

Water absorption of a brick body during the freeze/thaw cycles is influenced not only by the gradual change in pore structure, but also by the change of the irreversible expansion. The effect of change in pore volume and change of the median pore radius on water absorption is virtually identical and in both cases, it is a linear dependence, see Figures 6 and 7. In case of the irreversible expansion, it is a continuous trend, where the increase of expansion couples with the increase of absorption, see Fig. 8.
Effects of freeze/thaw cycles on the change of pore structure are shown in Figures 9 and 10. The course of changes in pore volumes according the number of freeze-thaw cycles is shown in Figure 9. For all three firing temperatures we follow a continuous process. Volume changes of pores takes place the fastest on the brick body, which was fired at temperature 900°C. With the increasing firing temperature is the rate of this change slowed down significantly. With a similar trend we can also meet at monitoring of the median pore radius, see Figure 10. The difference between these two properties lies in the fact that with the increase firing temperature the initial values of the pore volume decrease and, on the contrary, the initial value of median pore radius grows. These results confirm the fact that in order to achieve a high frost resistance of brick body, two important conditions have to be met: the pore structure should have the greatest value of the median pore radius (primary condition) and the lowest value of the pore volume (secondary condition) [22, 32].

More detailed transformations of pore structure during the freeze-thaw cycles are shown in Figures 11–13. In these cases, the histograms show the percentage changes in volumes of pores on the brick body at firing temperatures of 900, 1000 and 1060°C. Based on their increasing or decreasing trend a virtual boundary has been established at the temperature of 900°C for the pore diameter of 1 µm, at temperature of 1000°C for the pore diameter of 1.5 µm and at temperature of 1060°C for the pore diameter of 3.0 µm. From this virtual boundary the volume of small pores gradually decreases with the increase in number of freeze/thaw cycles. This trend can be observed especially at the firing temperature of 900°C, see Figure 11. With an increasing firing temperature, the trend is no longer so clear.

These changes in the field of pore volume have caused an irreversible change – the increase of values of the median pore radius, see Figure 10.

5. CONCLUSIONS

Irreversible expansion and water absorption of the brick body in the process of freezing and thawing depend from the firing temperature; these phenomena significantly decrease with the rise of temperature.

The results obtained in this work show that in order to achieve a high frost resistance of brick body, two important conditions have to be met: the pore structure should have the greatest value of the median pore radius (primary condition) and the lowest value of the pore volume (secondary condition).

The change of the porous system during freeze-thaw cycles takes place so that the volume of small pores
gradually decreases and conversely, a large pore volume gradually increases. This results in a gradual increase in the value of the median pore radius. In this case, we can see that brick body has a tendency to adjust its pore structure so as to best resist the effects of freeze-thaw cycles.

Interesting results were obtained in the comparison of water absorption values of the samples stored only in water (W) and samples exposed to freeze-thaw cycles (F/T) after 21 days of common storage. Higher water absorption of samples F/T in contrast to the samples W underlines the fact that in case of the F/T samples there have been significant changes in the porous system. This should be a signal that in a short time a total destruction of the brick body will occur.

The knowledge gained can be applied when predicting the durability of older clay roofing tiles or facing bricks, which have been placed in the outdoors for long periods of time. If this product will be subjected to e.g. 50 to 100 freeze-thaw cycles and the values of the pore volume and of the median pore radius will remain practically unchanged, then we can state that the product has a suitable pore structure and can achieve high durability. Conversely, if the values of the pore volume and of the median pore radius show great changes, we can assume that the brick product will no longer perform its function in the short term.

REFERENCES


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