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Frost resistance of clay roofing tiles: Case study

Vilma Ducman [a], Andrijana Sever Škapin [a,*], Miroslava Radeka [b], Jonjaua Ranogajec [b]

[a] *Slovenian National Building and Civil Engineering Institute, Dimičeva 12, SI-1000 Ljubljana, Slovenia*
[b] *University of Novi Sad, Bulevar Cara Lazara 1, 21000 Novi Sad, Serbia*

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Abstract

In order to give required protection of the buildings, clay roofing tiles should be resistant to freezing conditions. In the present study, clay roofing tiles were fired at different temperatures. Afterwards, direct and indirect test methods were used to evaluate their frost resistance. The direct method in standard EN 539-2 was applied, ASTM C 1167-03 was used for indirect methods, and Maage factor was calculated from Hg-porosimetry curve. It was confirmed that the temperature of firing is the main influential parameter as it affects the porosity of a ceramic body. The comparison between indirect and direct methods of prediction and test of frost resistance has also shown good correlation.

Keywords: Frost resistance; Clay roofing tiles; Hg-porosimetry; Flexural strength

1. Introduction

Frost resistance of ceramic products is a decisive factor for their durability. Water permeates into the pores of ceramic materials and, in the case of exposure to cold weather, it can freeze. The local high stress arises in surroundings of the pores and it can cause micro-cracks and fracture of material. The reaction of ceramic products to frost depends on several parameters, such as raw material and its composition, firing process, and properties of finished products, such as pore size distribution, pore shape, and strength of its structure [1,2].

For the prediction of frost resistance many standardized methods which simulate natural conditions have been developed. Besides them, there are also indirect methods which give prediction on frost resistance on the basis of knowledge how freezing phenomena is influenced by distribution of porosity, degree of saturation, rate of freezing, etc.

The direct freezing–thawing method for clay roofing tiles, which is widely used in Europe, is EN 539-2 [3], prescribing five different methods for different climatic regions. All methods specify that samples first be soaked in water and then exposed to a number of freezing–thawing cycles. After a certain number of cycles (depends on the method selected) samples, not showing significant damages, are labelled as frost resistant.

The ASTM C 1167-03 [4] indirect method is fast and simple for execution, providing a prediction of frost resistance through the saturation factor S. This factor is the ratio between water absorption after 24 h of soaking in cold water and the water absorption after 5 h of boiling.

Classification of resistance to freezing–thawing is as follows:

- $S < 0.74$ – high probability for material to be frost resistant in severe climatic,
- conditions $0.74 < S < 0.84$ – uncertain zone of frost resistance, and
- $S > 0.84$ – low probability for material to be frost resistant.

The indirect method of wide recognition is also Maage factor [5,6] of frost resistance prediction. The factor is based on experimental results and on a statistical model with two main variables: the total volume of pores (PV) and the share of pores at certain pore diameter, which is greater than 3 μm (P3). Maage [5,6] proposes the following equation, which gives factor of frost resistance DF:

$$\mathrm{DF} = \left(\frac{3.2}{\mathrm{PV}}\right) + (2.4 \times \mathrm{P3}), \tag{1}$$

where PV represents the pore volume as the volume of intruded Hg (cm^3/g), and variable P3 represents the share of pores bigger

* Corresponding author. Tel.: +386 1 28 04 268; fax: +386 1 28 04 484.
E-mail address: andrijana.skapin@zag.si (A.S. Škapin).

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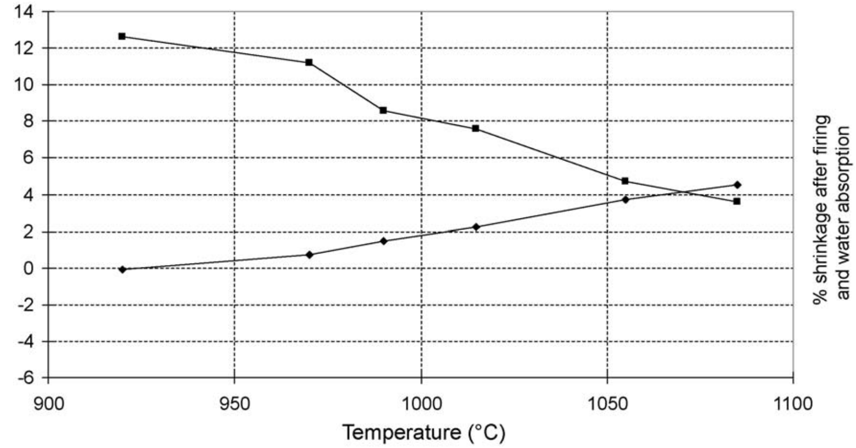

Fig. 1. Dependence of water absorption and shrinkage on firing temperature.

than 3 μm. It has been further proposed the following classification:

- DF > 70 – high probability for material to be frost resistant in severe climatic conditions,
- 55 < DF < 70 – uncertain zone of frost resistance, and
- DF < 55 – low probability for material to be frost resistant.

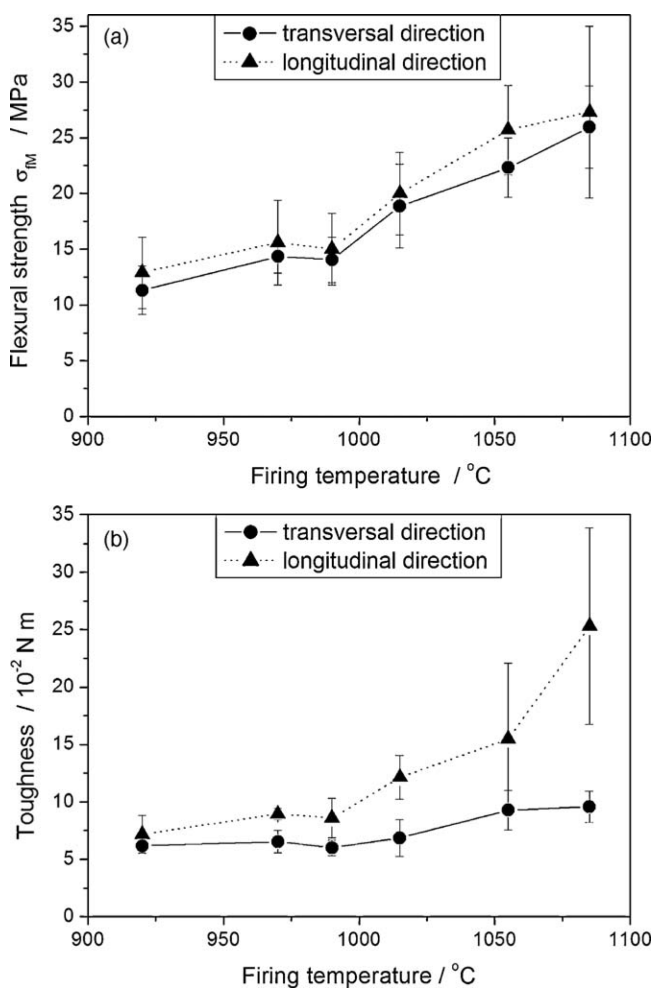

Fig. 2. Dependences of (a) flexural strength (maximum flexural stress sustained by the sample during a bending test) and (b) toughness on firing temperature (transversal direction is alongside the extrusion and longitudinal direction is perpendicular to the extrusion, respectively).

Koroth et al. [7] also developed a durability index based on the assumption that the share of pores greater than 3 μm play a crucial role in frost resistance of clay bricks, while Franke and Bentrup [8,9] proved on 40 different types of bricks (new and ancient) that the most important factor is the mean pore radius $r_{50\%}$, (assigned to 50% filling of the pore volume), where limit for resistant bricks lies at or above 1 μm.

Besides porosity, for achieving frost resistance of clay roofing tiles, mechanical properties represent an important factor; if they are high enough, mechanical properties can withstand pressure applied by freezing water. The influence of the maturing temperature on porosity and consequently on mechanical behaviour of ceramic bodies was studied by Jordan et al. [10], but there is no literature data available for the correlation between mechanical properties and frost resistance for roofing tiles. The valid ASTM C62-08 [11] for bricks proposes that (a) brick resistant to severe weathering (SW) should have minimum compressive strength of 20.7 MPa (b) those resistant to moderate weathering (MW) 17.2 MPa, and (c) those resistant to negligible weathering (NW) at least 10.3 MPa.

In practice, it is recommended to use both types of methods (direct and indirect ones) and to set the correlation for certain type of raw materials. With regard to these results, producers can later decide whether to use the selected method for regular checks in the production control process or not.

The aim of our work was (a) to determine the temperature range of firing in which the selected clay based products are frost resistant, (b) to compare the use of direct and indirect methods used for the determination of frost resistance and (c) for specific type of clay products establish a correlation between both types of methods.

2. Experimental

In order to prepare specimens fired at different temperatures, dry samples of clay roofing tiles were taken from regular production and fired in laboratory (simulating the firing process of regular production) at different temperatures: 970, 990, 1015, 1055 and 1085 °C. The firing rate was 100 °C/h; the dwelling time was 2 h at a selected temperature. Samples were then tested on their frost resistance by different methods. For the verification of methods, samples were intentionally fired at the temperature 920 °C as well, at which frost resistance is not expected. Type of clay was determined by means of X-ray analysis, where it was found that raw material is of caolinit-illitic type.

Mechanical properties of clay roofing tiles were determined through testing of flexural properties (flexural strength and toughness) using the mechanical testing equipment ZWICK Z 100. Test specimens with dimensions 40 mm in length, 10 mm in width and 10 mm in thickness were cut out of fired samples (alongside and perpendicular to the extrusion). The tests were performed on 8 parallels and their mean values were reported. The speed of testing was 0.5 N/s. Span L was adjusted to 25 mm; the radius of loading edge R_1 was 2 mm, and the radius of supports R_2 was 1 mm. The flexural strength was calculated using following equation:

$$\sigma_{\mathrm{fM}} = \frac{3F_{\mathrm{M}}L}{2bh^2}, \tag{2}$$

where, σ_{fM} is the flexural strength, in MPa; F_{M} is the maximum force, in newtons; L is the span, in millimetres; b is the width of the specimen, in millimetres; h is the thickness of the specimen, in millimetres, while toughness was integrated force/deflection curve.

To get the Maage factor the Hg porosity technique was used where applied pressure was in the range of 1.6 kPa to 414 MPa and DF factor calculated by Eq. (1).

Additionally, samples fired at 970, 1015 and 1085 °C were tested for mechanical properties. Hg porosity was also tested after 50, 100 and 200 cycles of freezing–thawing. SEM microstructure was observed on samples fired at 970, 1015 and 1085 °C before and after exposure to 200 cycles of freezing–thawing using scanning electron microscopy JEOL JSM-5500LV.

Frost resistance was determined by direct method EN 539-2 (method C) [3] where 10 samples were first saturated in water under vacuum and then exposed to freezing at −15 °C in a programme-controlled freezing unit and then thawed by sprinkling with water, followed by immersion in water at +15 °C. After completing the freeze/thaw cycles, test pieces are visually examined for damages or failure of material.

Saturation factor S according to ASTM C 1167-03 [4] was determined as the ratio between water absorption after 24 h soaking in cold water and the water absorption after 5 h of boiling.

3. Results and discussion

3.1. Effect of firing temperature on shrinkage, water absorption, mechanical properties and microstructure of roofing tiles

In Fig. 1 the relation between shrinkage and water absorption in dependence upon firing temperature is presented. The shrinkage increases with higher firing temperature, and simultaneously water absorption of the ceramic body decreases. This type of curve is very often used in ceramic industry because it offers the opportunity to assess the sensitivity of the materials to firing process. If the slope of shrinkage is very steep, minimal changes in temperature cause high shrinkage, which can be a problem in the industry (differences in dimensions of final products).

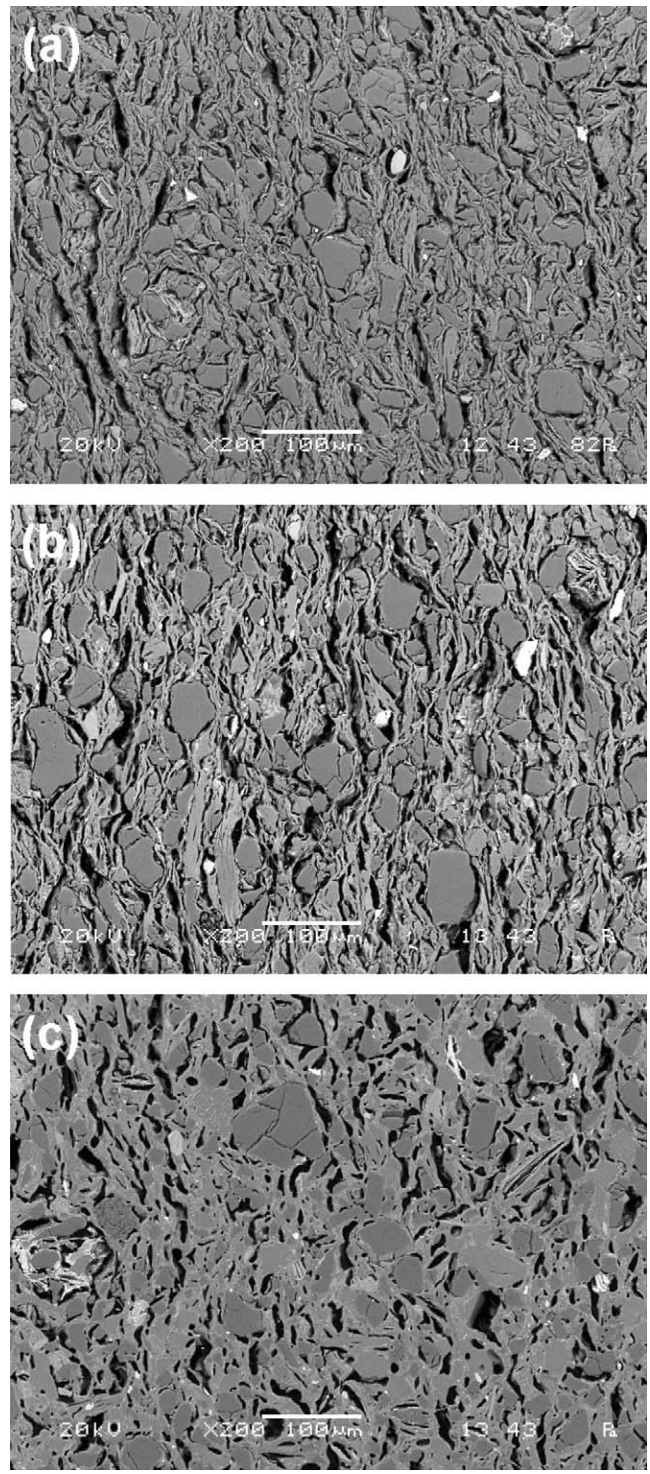

Fig. 3. SEM images of polished cross-sections of the roofing tiles fired at (a) 970 °C, (b) 1015 °C and (c) 1085 °C.

Table 1
Saturation factor S and Maage factor DF for roofing tiles fired at different temperatures.

Sample/T_{firing} (°C)	Water absorption afer 24 h (%)	Water absorption afer 5 h in boiling water (%)	ASTM saturation factor S	Maage factor DF
920	12.6	15.4	0.82	37
970	11.2	14.6	0.76	37
990	8.6	13.2	0.65	39
1015	7.6	12.1	0.63	44
1055	4.7	9.3	0.51	94
1085	3.6	7.1	0.51	115
Sample from regular production	4.7	9.8	0.48	92

To analyze the effect of firing temperature of the test specimen on mechanical properties, flexural strength and toughness of roofing tiles at sixth different firing temperatures were determined in both principal directions: alongside and perpendicular to the extrusion. The mean value with standard deviations of flexural strength (σ_{fM}) and toughness of samples are presented in Fig. 2. It can be seen that below firing temperature 990 °C both flexural strength and toughness of samples are almost the same, while above this temperature both quantities slightly increase. In the case of flexural strength there

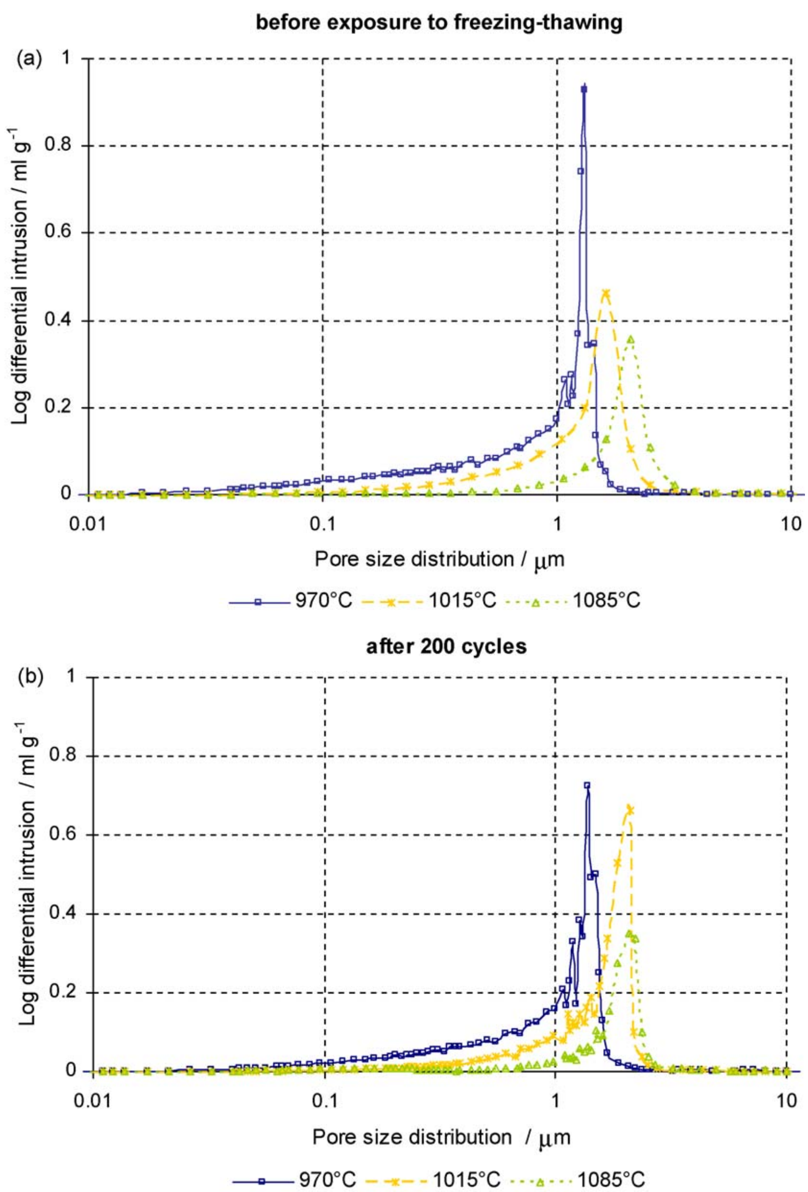

Fig. 4. Pore size distribution of samples fired at 970, 1015 and 1085 °C (a) before and (b) after exposure to the freezing–thawing procedure.

Table 2
Results of Hg-porosimetry for samples fired at 970, 1015 and 1085 °C and exposed to 50, 100 and 200 cycles of freezing–thawing procedure.

T_{firing} (°C)	Number of cycles of freezing–thawing	Porosity (%)	Average pore diameter (μm)	Median pore diameter (μm)
970	0	28.3	0.3373	0.9418
	50	28.4	0.3674	1.0062
	100	28.1	0.3632	0.9835
	200	28.0	0.4438	1.0755
1015	0	26.3	0.8684	1.6219
	50	25.1	0.9085	1.6841
	100	25.1	0.8153	1.7163
	200	24.8	0.9380	1.7148
1085	0	17.9	1.1467	2.1905
	50	15.8	0.7717	2.0720
	100	15.7	0.8210	2.0140
	200	15.6	0.7886	1.9723

is no significant difference between transversal and longitudinal direction, while in toughness vs firing temperature diagram samples cut in transversal direction show larger increase with temperature.

Fig. 3 shows microstructure of polished cross-sections of samples fired at three different temperatures: With increasing firing temperature grains grow and the quantity of pores decreases, which is the reason for higher flexural strength and toughness.

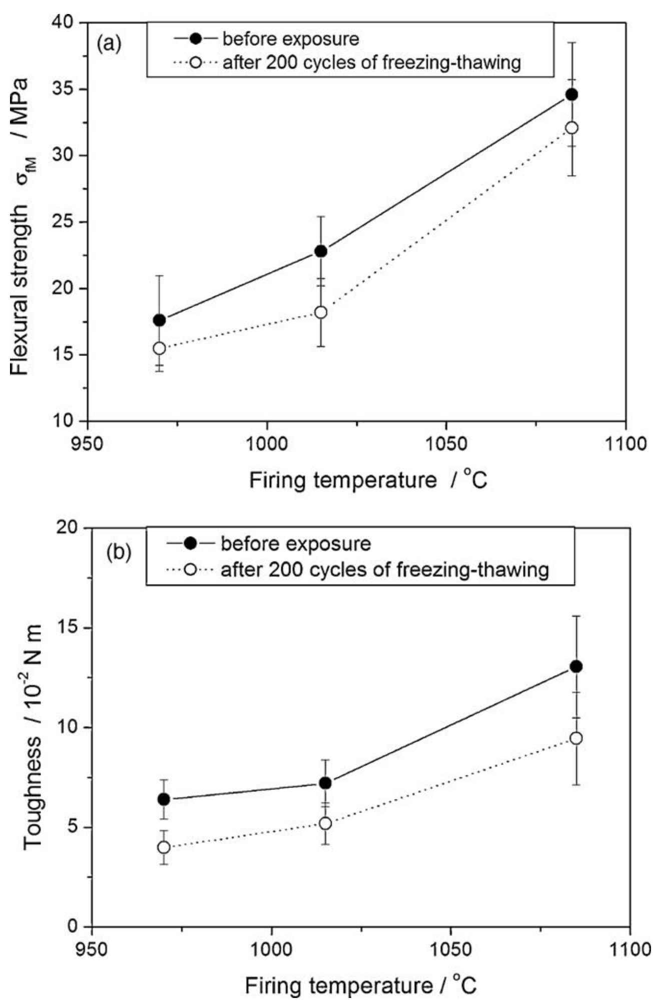

Fig. 5. Dependences of (a) flexural strength (maximum flexural stress sustained by the sample during a bending test) and (b) toughness on freezing–thawing (the standard deviations was determined from eight parallels).

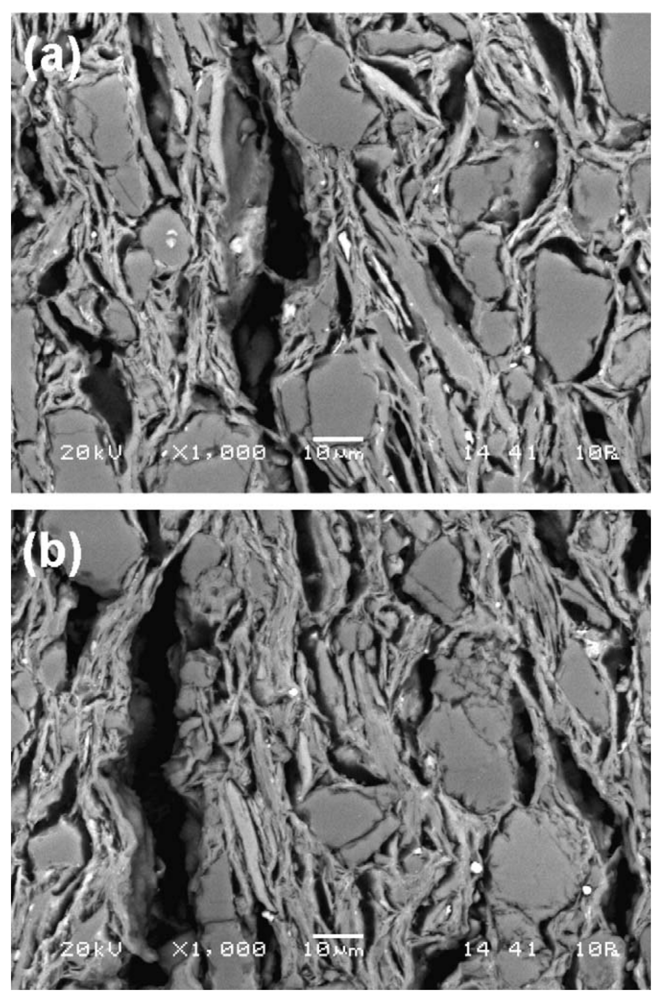

Fig. 6. SEM images of polished cross-sections of the roofing tiles fired at 970 °C (a) before and (b) after exposure to 200 cycles of freezing–thawing.

Table 3
Results of direct freezing method for roofing tiles fired at different temperatures.

Sample/T_{firing} (°C)	Water absorption under vacuum (%)	Resistance after 50 cycles	Resistance after 100 cycles	Resistance after 150 cycles	Resistance after 200 cycles	Resistance after 550 cycles
920	14.1	Yes	No	No	/	/
970	13.4	Yes	Yes	No	/	/
990	12.0	Yes	Yes	Yes	Yes[a]	Yes
1015	10.7	Yes	Yes	Yes	Yes	Yes
1055	7.9	Yes	Yes	Yes	Yes	Yes
1085	5.9	Yes	Yes	Yes	Yes	Yes

[a] Hair crack on one sample (remains the same after 550 cycles).

3.2. Assessment of frost resistance of the roofing tiles by determination of ASTM saturation factor and Maage factor

In Table 1 results of ASTM saturation factor *S* and Maage factor DF are given; according to the ASTM *S* factor prediction. Samples fired at 920 and 970 °C are not supposed to be frost resistant, while according to the Magge factor DF also samples fired up to 1015 °C have low probability to withstand severe freezing conditions. Simultaneously also samples from regular production were tested by indirect methods and both factors (*S* factor is 0.48, Maage factor is 92) predict very good frost resistance. But it was already found out by others [12] that in some cases Maage model cannot provide reasons for good or even excellent frost resistance.

3.3. Effect of freezing–thawing on pore size distribution and on mechanical properties of roofing tiles

Determination of pore size distribution and mechanical properties were carried out before and after frost resistance test according to the method C of standard EN 539-2 [4].

Samples fired at selected temperatures 970, 1015 and 1085 °C were tested for changes in porosity after process of freezing–thawing. As it can be seen from Fig. 4a and b, which represents samples before exposure and after 200 cycles of freezing–thawing, respectively, there are changes in the structure of pores. From Table 2 and Fig. 4a and b it is clearly seen that with higher firing temperatures there is a decrease in porosity and an increase in average as well as median pore diameter, which is in an agreement with theory of sintering. It can also be observed that the dominant pore size is above 1 μm, which is important to ensure frost resistance [13].

After exposure to freezing–thawing the changes in the porosity are not very evident (Fig. 4b), but there is a slight change in the shape of the curves. Before freezing curves of pore size distribution are wider, while after exposure they are narrower and biased. In the case of samples fired at 970 °C there is a slight increase in average pore diameter after freezing–thawing processes which could be attributed to the breakage of cell between pores due to the lower mechanical properties of ceramic body fired at lower temperature (see Fig. 2a).

The results of determination of flexural properties of samples fired at three temperatures 970, 1015 and 1085 °C before and after exposure to freezing–thawing are shown in Fig. 5. It can be seen that the value of flexural strength and toughness of samples after exposure is reduced (flexural strength is reduced up to 20% with respect to not exposed samples and toughness up to 38%, respectively).

Fig. 6 shows the microstructure of samples before and after freezing and there are no significant differences in porosity, the quantity of larger pores seems to be constant, but it can be seen that sample after exposure to freezing–thawing contains more hair cracks within grains than before exposure.

Even though compressive strength does not correlate directly to flexural strength, there is an indication that compressive strength increases [14] with flexural strength. Therefore samples fired at higher temperature having higher mechanical properties are also more frost resistant as suggested in ASTM C62-08 [11].

Based on the theory of Franke and Bentrup [8,9] samples having median pore diameter greater than 1 μm samples fired at 1015 and 1085 °C, should be frost resistant. To the contrast, those fired at 970 °C should be susceptible to weathering. The prediction was confirmed by direct test methods (Table 3).

From Table 3 it can be seen that the roofing tiles are resistant to freezing when fired at or above 990 °C. ASTM prediction factor *S* (see Table 1) shows very good correlation with testing by direct method as indicated in Table 3. Also samples fired at lower temperature were resistant after 50 cycles of freezing–thawing what are the requirements of the C method of EN 539-2 [3] standard, but after following 50 and 100 cycles samples have shown frost resistance damage (Fig. 7).

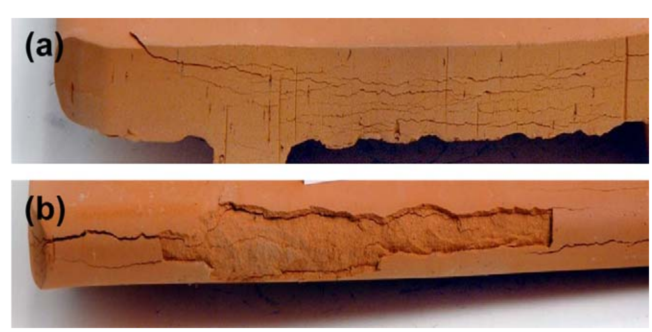

Fig. 7. Damage on samples fired at (a) 970 °C and (b) 920 °C after 150 cycles of freezing–thawing.

4. Conclusions

Frost resistance was determined by different direct and indirect methods and good correlation between direct freezing test and ASTM saturation factor for investigated caolinit-illitic type of clay was confirmed. In real conditions, after 200 cycles only samples fired at or above 990 °C are frost resistant, which is supported by the results for the *S* factor. The Maage method also provides a good correlation with real frost test for samples fired below 990 °C. It suggests that samples will be resistant in severe climatic conditions only when fired at or above 1055 °C which is also the set temperature in regular production. The methodology presented in the paper could be used effectively in the production process when changing the process parameters, such as mixture composition or firing regime.

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