Laboratory study of changes in Paleogene sandstones from Slovakia exposed to high temperatures

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Abstract: Two types of Paleogene sandstone with different initial porosity and strength were studied after their exposure to high temperatures. Cylindrical rock specimens were heated for six hours in a muffle furnace at 400, 600, 800 and 1000 °C, and subsequently, cool samples were tested to determine their uniaxial compressive strength, water absorption and ultrasonic wave velocity. In addition to visual changes in the specimens, such as a change in colour or gloss, volume and changes in mineral composition were studied as well. Total and open porosity and pore size distribution were determined using mercury intrusion porosimetry (MIP) before and after the heating. Laboratory testing showed that the resistance to temperature load of both sandstones was very similar: both indicated high resistance up to 600 °C. After being heated to higher temperatures, the rocks decreased in strength rapidly. The temperature of 600 °C was the limit or the critical temperature for preservation of the sandstones quality parameters. By MIP and by detailed mineralogical analysis, it was documented that temperatures above 600 °C start changes in the microstructure and porosity of sandstones, including the creation of micro-cracks.

Key words: sandstone, thermal load, mineralogy, colour; gloss, porosity, strength

1. INTRODUCTION

The utilization of rocks for building necessarily requires a complete evaluation of their properties and behaviour under various external stresses and thermal conditions. In connection with temperature effects, the behaviour of rocks is usually evaluated in the range of climatic temperature variations, i.e. from c. -30 °C up to +50 °C. In European laboratory testing of natural stone, the destructive effects of subzero temperatures are included in the standard EN 12371 to determine the frost resistance of stones. Above zero climatic temperature fluctuations come under consideration in standards EN 14066, EN 16140 and EN 16306. The maximum positive temperature that is used in the testing of natural stone is 105 (± 5) °C. Aside from this, the routinely used temperature for drying specimens of natural stones in a laboratory is 70 (± 5) °C or 105 (± 5) °C, which are considered to be the temperatures which do not affect the properties of the majority of hard rocks, except for rocks containing clay minerals.

Temperature plays an important role in many rock engineering practices (Huang & Xia, 2015). In recent years, examining the influence of higher temperatures on rocks is significant not only with sporadic catastrophic fires of stone historical heritage objects or fires in highway tunnels but, as has been often stated in literature, knowledge on this issue can find successful implementation in many modern and perspective geotechnical engineering projects, such as deep radioactive waste or gas storage or geothermal heat extraction (Tian et al., 2012; Ranjith et al., 2012).

The effects of high temperatures on the physical or physico-mechanical properties of natural stones have been studied by many authors (e.g. Hajpál & Török, 1998; Siegesmund et al., 2000; Gómez-Heras et al., 2004; Kompaníková et al., 2011, 2014; Ranjith et al., 2012; Ozguven & Ozcelik, 2013; Tian et al., 2014; Huang & Xia, 2015; Vazquez et al., 2016). These focused on various lithological types (marbles, limestones, sandstones, granitoids and others), various temperature ranges in which influences on the rocks are monitored, and also a wide spectrum of studied effects (colour changes, bulk density, porosity, ultrasound wave velocities, expansion of mineral constituents in rocks, surface roughness, uniaxial compressive strength, flexural strength, modulus of elasticity, permeability and others). In most cases, the properties exhibit considerable changes in temperature (Brotóns et al., 2013). Different test conditions in each study (e.g. heating rate, exposition time to heating, size and shape of specimens) make it harder to compare results and draw generally valid conclusions.

The resistance of rocks to higher temperatures differs. Surprisingly, up to a certain limit, temperature does not noticeably affect the base physical properties of rocks (Kompaníková et al., 2014). Even, as stated in the literature, in certain cases the higher temperature may increase the strength of the rock material (Hajpál & Török, 1998; Ranjith et al., 2012). After reaching a definite temperature limit, which is different for every lithological type, destruction, weakness and decay of the rock will consequently occur.

Sandstones are frequently used as a building stone in Slovakia and have been the centre of attention in many studies (Čabalová, 1977, 1988; Pivko, 2010). This paper is a result of the laboratory study of temperature load on two different sandstones that are used as building and decorative stones. The research aims to monitor visual changes of sandstones and characterise their physical (predominantly the uniaxial compressive strength, water absorption and porosity) and mineralogical changes and so consequently tries to determine their limit resistance to a thermal load.

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2. GEOLOGICAL SETTING

Studied sandstones come from quarries Králiky and Spišské Tomášovce and geologically belong to the Subtatric Group in the Inner Carpathian Paleogene unit of the Western Carpathians (Fig. 1).

Clasts in the sandstone are predominantly carbonate and quartz. Feldspar and muscovite are also present. The matrix is calcite-clayey, the cement is calcite with fragments of carbonates and quartzites. Tomášovce Member represent the shallow marine sediments of the neritic zone and is of the latest Priabonian in age (Mello et al., 2000[b]).

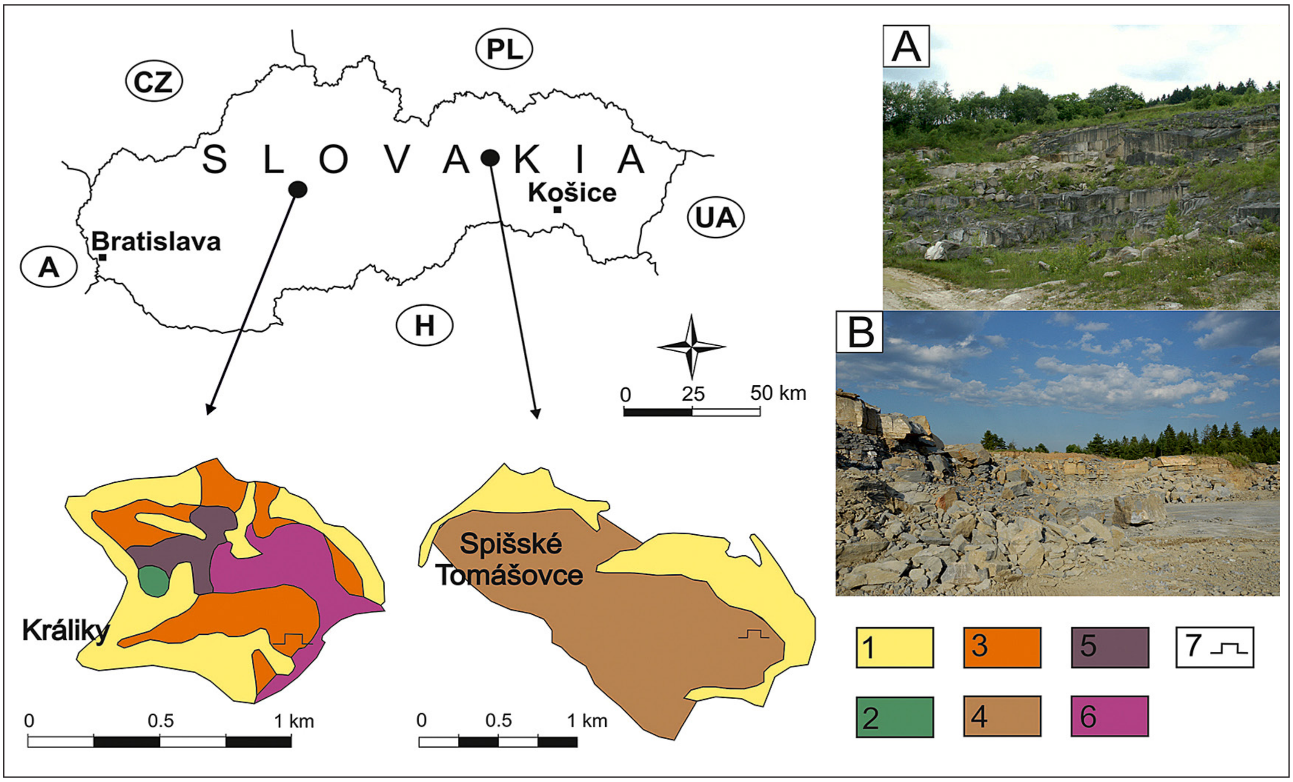

Fig. 1. Localization, geological maps and photos of the actual state of the studied quarries (from Polák et al. (2003[a]) and Mello et al. (2000[a]), modified). Explanations: 1. Pleistocene-Holocene sediments; 2. Neogene andesites; 3-4. Paleogene sandstones; 5. Triassic limestones; 6. Triassic dolomites; 7. quarry. Photos of the quarries: A – Králiky; B – Spišské Tomášovce.

On the western border of the village Králiky, the sandstones of the Borové Formation (Early Priabonian) are exposed in an abandoned quarry (Polák et al, 2003[a]). These are thickly-bedded with bed thickness between 20 and 100 cm. The bedding is relatively homogeneous (Fig. 1A). In a fresh state, the sandstone is light gray in colour; in a weathered state it is yellowish brown. Sandstone is fine-grained or medium-grained, and clasts are predominantly quartz and carbonate. Cement is mainly calcite, much less quartz or clay. By its content, especially according to its matrix and cement, the sandstone passes into a sandy limestone. According to the origin, the sandstone is considered as being a deltaic fluvial sediment (Polák et al., 2003[b]).

The second studied sandstone was excavated in the quarry near Spišské Tomášovce village, which represents a typical locality of Tomášovce Member of the Subtatric Group (Fig. 1B, Mello et al., 2000[a]). These layers contain fine-grained sandstones and siltstones with obvious beds of medium-grained carbonate sandstones or fine-grained conglomerates. The sandstone beds are from 5 to 50 cm thick; in a fresh state they are darker grey, while after weathering they are yellowish brown or rusty brown.

3. METHODS

For the evaluation of temperature influences on natural stones, two different sandstone types from territory of Slovakia were analyzed – porous sandstone Králiky (K), and stronger and denser sandstone Spišské Tomášovce (ST). Approximate 30 cylindrical (35 mm diameter, 35 mm height) samples were prepared for each stone type. Specimens were divided into groups and each group was exposed to a different temperature. One group of rocks was not heated. It was used as an etalon and also to determine basic stone characteristics according to standard laboratory methods (EN 1936; EN 13755; EN 1926; EN 14579). As a supplement to the evaluation of thermal influences, the surface faces of some specimens were polished in order to measure the gloss by glossmeter before and after the heating.

The heating of the rock cylinders was realized in a programmable muffle furnace at four different temperatures: 400, 600, 800 and 1000 °C. The specimens were exposed to the target temperature for a period of six hours and then they were allowed to cool to room temperature (ca 20 °C). Consequently, on heat-treated specimens, changes in visual appearance such as colour and gloss were described, and also changes in volume by measurement of dimensions and weight were recorded. Their mineral composition and physical properties were researched. Laboratory tests included the determination of bulk density, water absorption, ultrasound P-wave velocity and uniaxial compressive strength (UCS).

Thin sections from fresh unheated rocks and also from heated specimens were prepared for polarizing microscope analysis and electron probe microanalysis to examine changes in mineral composition and structural and textural characteristics.

The sandstones were also submitted to a detailed examination of their porosity. The parameters of the pore structure of the unheated sample and the sample heated to 600 °C were determined by the mercury intrusion porosimetry (MIP) method by using an automated porosimeter Poremaster 60GT Quantachrome.

Mineralogical and chemical changes in the sandstone were observed by polarizing microscope and analyzed via electron microprobe analyzer CAMECA SX 100 at the State Geological Institute of Dionýz Štúr.

4. RESULTS

4.1. Volume changes and loss of cohesion

Both sandstones underwent a complete heating process to 400, 600, 800 and 1000 °C. At heating above 600 °C, the rock cylinders visibly increased their volume. The effect was observed for both sandstones at temperatures 800 °C and 1000 °C (Fig. 2).

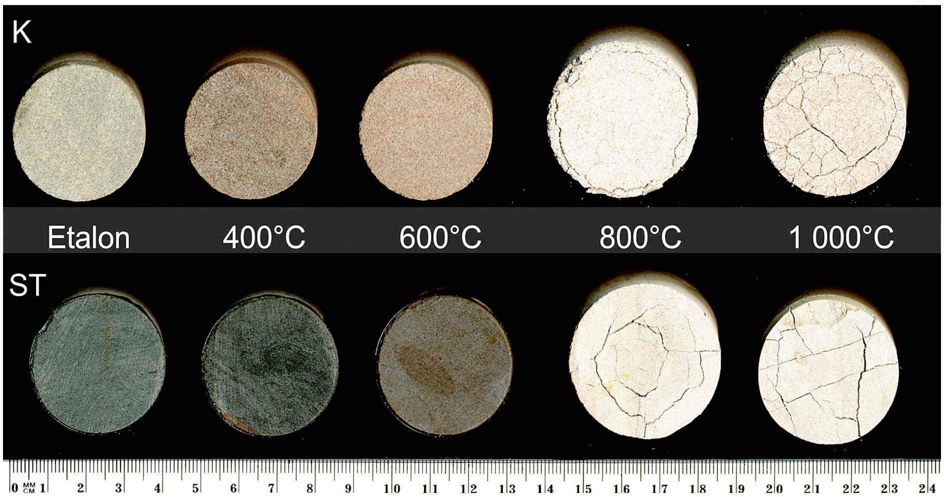

Fig. 2. Tested sandstones and temperature to which they were exposed: K – Králiky, ST – Spišské Tomášovce.

The increase in specimen volume is connected to structural loosening and weakening, which means a decrease in bulk density.

The weakness and the decay of sandstones heated to temperatures more than 600 °C was not a sudden, but a gradual process. Rocks destroyed in several stages from initial creation of cracks up to scaling, crumbling and, finally, total decomposition (Fig. 3).

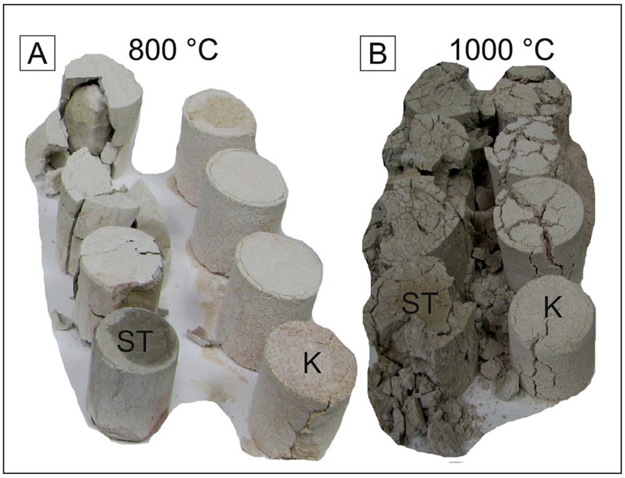

Fig. 3. The disintegration of sandstones after the heating at: A – 800 °C; B – 1000 °C. K – Králiky, ST – Spišské Tomášovce.

It was observed that immediately after the heating and cooling, the rock cylinders stayed coherent. Even it was possible to measure the ultrasonic wave velocities and uniaxial compressive strength on some specimens without special precautions. After remaining a short time (1-2 days) at room temperature and in relevant room moisture conditions, the specimens started to crumble, spall or decompose from the edges to the centre. The retaining of the specimens' shape integrity after heating conditioned the performance of follow-up tests on determining water absorption and strength.

4.2. Colour changes

The heating of the rocks to high temperatures was accompanied by changes in colour. The colour was evaluated only visually by using the Munsell geological rock-color chart (Geological Society of America, 2011). Significant changes in the colour of the sandstones were visible macroscopically (Fig. 2):

– light gray sandstone Králiky first became darker with a reddish shadow and then, at higher temperatures became lighter;

– similarly, sandstone Spišské Tomášovce, which was medium light grey originally, changed to dark grey and consequently to a lighter beige shade.

In general, it can be said that the first evident colour changes in the rocks were visible after heating to 400 °C, and less changes showed in the Spišské Tomášovce sandstone. After heating to temperatures above 600 °C and higher, the rocks became lighter and whiter. The colour alterations of the sandstones at all temperatures are summarized in Tab. 1.

Tab. 1. Colour changes of sandstones according to the Munsell Rock-Color Chart (Geological Society of America, 2011).

Sandstone	Etalon	Heating to temperature (°C)			
		400	600	800	1000
Králiky	Light gray	Light brown	Light brown	Grayish orange pink	Yellowish gray
	N7	5YR 6/4	5YR 6/4	10R 8/2	5Y 8/1
Spišské Tomášovce	Medium light gray	Medium light gray	Light brownish gray	Grayish orange pink	Pinkish gray
	N6	N6	5YR 6/1	10R 8/2	5YR 8/1

4.3. Loss of gloss

For end-use in building activities, many decorative stones are polished to demonstrate their natural colour and textural surface pattern. Polished surface faces also have better use and keeping properties, and it is simpler to clean them. For these reasons, a gloss may be listed in the specific technical properties of natural stone. Gloss effects are based on the interaction of light with the physical properties of the sample surface. The gloss is measured using a glossmeter by shining a known amount of light at a surface under a defined angle and quantifying the reflectance. Gloss intensity is expressed in gloss units GU from 0 up to 100.

For gloss measurements, one plane of the cylinder specimens from each group was polished. The gloss was measured on specimens before and after the heating at 400, 600, 800 and 1000 °C, three times on each plane to calculate the mean value. It was not possible to evaluate the gloss of Králiky sandstone for all temperatures due to structural decomposition. From the gloss mean values summarized in Tab. 2, it is evident that the gloss intensity decreases as the temperature increases.

4.4. Changes in mineral and chemical composition

Microscope analyses showed that studied sandstones have a very similar mineral composition: carbonate minerals, quartz, feldspar, muscovite and some accessory minerals (Fig. 4). Mineralogical differences between sandstones are in the percentage content of minerals only (Tab. 3). Some additional observed disparities are given in Tab. 4.

Tab. 2. Gloss units of tested stones before and after the heating (Durmeková et al., 2016, modified).

Sandstone Locality	Heating (°C)	Gloss before heating (GU)				Gloss after heating (GU)				Difference (GU)
		1.	2.	3.	Average	1.	2.	3.	Average	
Králiky	400	45.3	46.6	46.4	46	42.0	41.6	41.3	42	4
	600	46.8	46.8	47.7	47	40.2	39.6	39.0	40	8
	800	47.1	47.6	48.4	48	x	x	x	x	x
	1000	39.2	39.4	40.6	40	x	x	x	x	x
Spišské Tomášovce	400	50.3	51.1	50.1	51	44.1	42.6	42.8	43	7
	600	58.9	59.2	58.5	59	38.7	36.7	36.1	37	22
	800	57.4	56.9	54.9	56	34.3	32.7	31.5	33	24
	1000	60.5	60.6	61.2	61	28.5	26.1	24.1	26	35

x – immeasurable

Tab. 3. Mineral composition of etalon sandstone samples.

Sandstone	Composition (%)				
	Cb	Qtz	Fs	Ms	Opq
Králiky	45	40	14	1	0
Spišské Tomášovce	70	10	10	8	2

Mineral abbreviations: Cb - carbonate minerals, Qtz - quartz, Fs - feldspar, Ms - muscovite, Opq - opaque minerals

Tab. 4. Mineralogy and fabric differences of the sandstones determined microscopically.

Králiky sandstone:	Spišské Tomášovce sandstone:
- bigger mineral grains, nonhomogeneity in the size of individual minerals	- mineral clasts are smaller and equal in size
- isotropic configuration of minerals	- fabric is a little anisotropic
- a great deal of quartz, predominantly in monomineral form	- less quartz, created polycrystalline aggregates
- muscovite is present only sporadically	- contents more muscovite
- more dolomite in rhombic forms, calcite is recrystalized	- less dolomite, more calcite
- less or no opaque minerals	- more ore (opaque) minerals

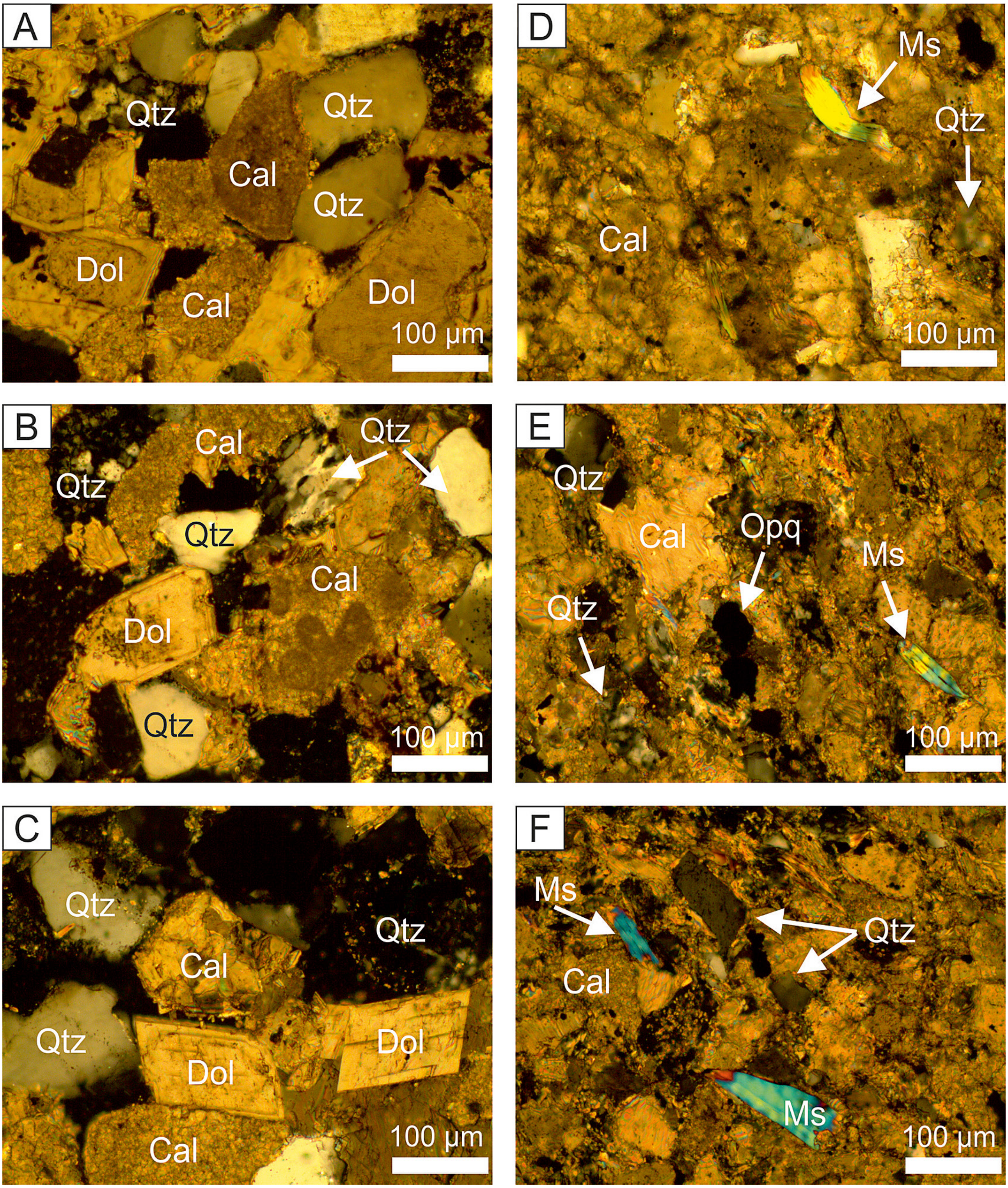

Fig. 4. Microstructure comparison of etalon sample sandstones in cross polarized light: Králiky sandstone (A, B, C); Spišské Tomášovce sandstone (D, E. F). Used abbreviations: Qtz - quartz, Dol - dolomite, Cal - calcite, Ms - muscovite, Opq - opaque minerals.

4.4.1. Králiky sandstone

The unheated sample may be identified as a fine-grained sandstone that contents quartz, dolomite crystals and calcite aggregates. Dolomite crystals are markedly limited as individual rhombic crystals. The main component of cement is calcite (which occurs in the two generations as younger micritic fine-grained and older coarse-grained calcite). Iron oxyhydroxides are also sporadically found. Obvious micro-structural and mineralogical changes occurred after heating to 800 °C, when the original colourless dolomite rhombohedra acquired a brown colouring, which was observed microscopically (Fig. 5). This effect is connected to the thermal reactions of dolomite when the calcination

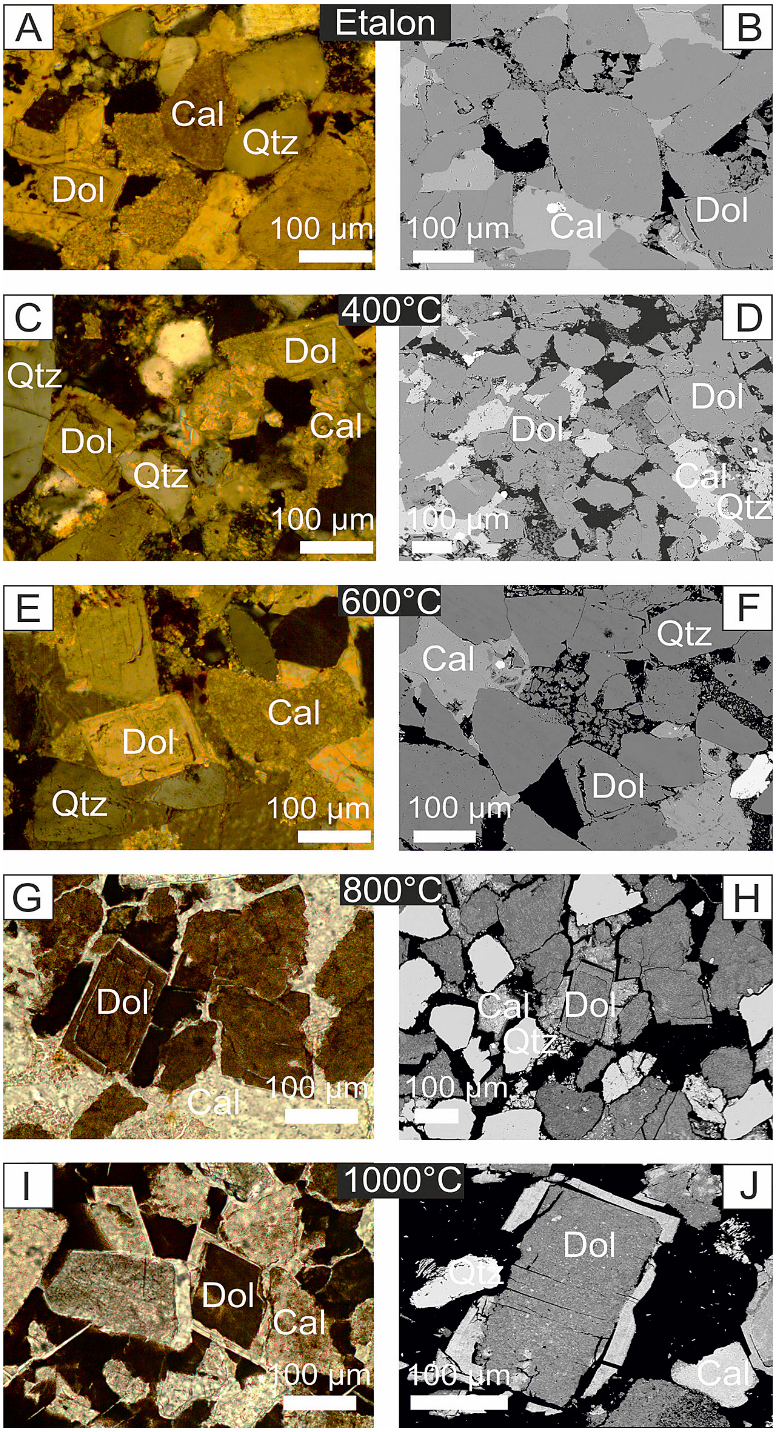

Fig. 5. Králiky sandstone microstructural changes caused by heating observed in polarizing microscope (left column) versus BSE (right column). Used abbreviations: Qtz - quartz, Dol - dolomite, Cal – calcite, BSE – back scattered electron.

process starts, and it means that the Ca constituent is being released from the original dolomite content. Electron microprobe analysis confirmed an evident decrease in CaO and increase in MgO when the samples were heated (Tab. 5).

4.4.2. Spišské Tomášovce sandstone

The etalon sample of fine-grained sandstone is characterized by a high content of carbonates (mainly calcite) and less quartz. Muscovite and opaque minerals are also present. Clasts have only a limited common contact, as they are mostly isolated in a fine grained matrix. Cement is predominantly calcite, occasionally also with quartzite. The fabric of the sandstone is gently anisotropic. Heating the samples to 400 and 600 °C did not show any evident microscopic changes (Fig. 6). Important changes came after heating to 800 °C and 1000 °C, such as: clear lightness of colour, reduction of calcite cement, creating of micro-cracks, rock weakness, and, consequently, total break-up. At higher temperatures, only quartz grains kept their original appearance but visible cracks were also in them (Figs. 6 and 7).

4.5. Changes in porosity

Changes in the total and open porosity of sandstones were analyzed in more detail by the mercury intrusion porosimetry method (MIP). The effects of heating at 600 °C on samples were compared to non-heated samples. The temperature of 600 °C was selected because it is the temperature at which the sandstones still retain their total cohesion.

MIP analysis allowed to obtain the basic parameters of the pore structure of sandstones (Tab. 6) and to determine the pore size distribution (Fig. 8), which should help to evaluate more objectively the changes in the sandstone pore structure after being heated at 600 °C.

The effect of 600 °C temperature on the Králiky sandstone with the total porosity about 16 % was not very strong or none, regarding changes of the monitored pore parameters values. The slightly lower value of the total porosity after

Tab. 5. Chemical content changes of Králiky sandstone detected by the electron-microprobe analyses.

wt. %	Dol - etalon	600 °C	800 °C	1000 °C
FeO	0.01	0.02	0	0.01
MnO	0	0.01	0.03	0.01
MgO	21.67	20.01	32.73	35.63
CaO	30.27	32.21	14.69	11.27
Total	51.95	52.25	47.45	46.92

mol. %	Dol - etalon	600 °C	800 °C	1000 °C
$FeCO_3$	0.01	0.03	0	0.01
$MnCO_3$	0	0.01	0.04	0.01
$MgCO_3$	49.89	46.34	75.58	81.45
$CaCO_3$	50.09	53.62	24.38	18.52
Total	99.99	100	100	99.99

heating are illogical and could be caused only by the natural heterogeneity of the various specimens selected for measurement. In the pore size distribution on Fig. 8 can be seen the increase of porosity in areas of bigger pores – macropores (void diameters from 1 to 100 µm). The result of this is the higher permeability of the heated sample because the pores of diameter between 0.1-10 µm are amenable for permeability.

MIP analysis indicated the different behaviour of the second tested sandstone after heating. The total porosity of the less porous Spišské Tomášovce sandstone increased with heating at 600 °C by about 36 %, predominantly by the increasing of micropores (Tab. 6). The higher ratio of micropores is documented also by the higher value of the specific surface area in comparison with the unheated sample. On the other hand, the percentage of macropores (pores bigger than 5.2 µm) in volume is lower than in the heated sample and there are also less open pores. This can be explained by changes in the microstructure following some mineral thermal reactions. A deeper explanation of obtained results requires additional repeated analyses.

4.6. Changes in other physical properties

All the above described changes in sandstones after heating were consequently revealed negatively in the decrease in physical and technical parameters. Apart from the already mentioned porosity and pore structure, changes in the water absorption, ultrasound wave velocity and uniaxial compressive strength after each target thermal load were controlled. The original basic physical and mechanical properties of the studied sandstones before heating are given in Tab. 7.

The water absorption was possible to monitor only in the case when the heated samples remained stable and coherent. Both sandstones behaved very similarly: up to 600 °C, neither had any evident change. After heating to higher temperatures, above 600 °C, it was no longer possible to monitor this property. The results of the water absorption tests are illustrated in Fig. 9A. The water absorption of sandstones increased with temperature (ST sandstone) or remained at the same level (K sandstone).

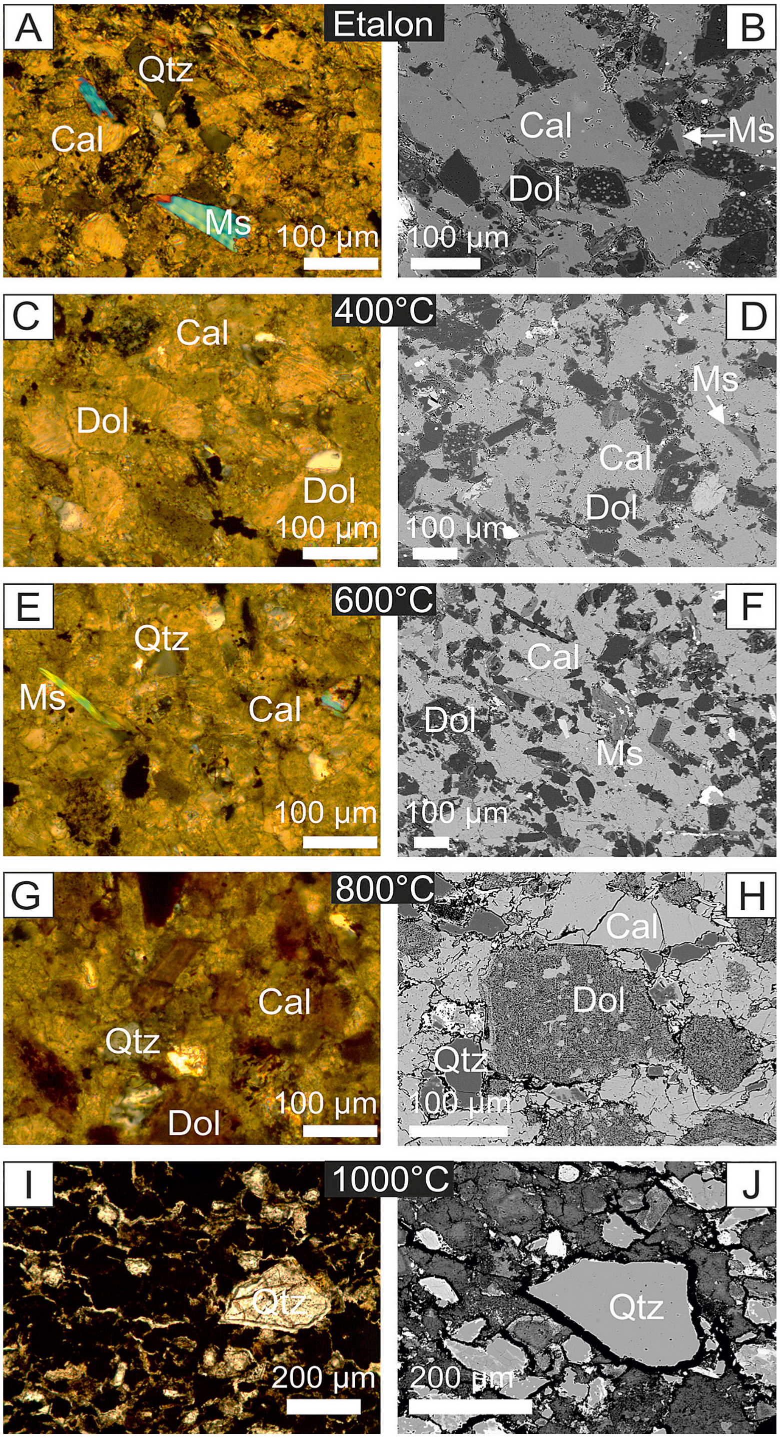

Fig. 6. Spišské Tomášovce sandstone microstructural changes after the heating visualized in polarizing microscope (left column) and BSE (right column).

The results of measurements on the determination of ultrasound wave velocity confirmed the expectation that gradually with increasing temperature the speed values would decrease due to the degradation of rock structure, the creation of cracks and the subsequent weakness of structural bonds between rock components (Fig. 3). The decreasing of ultrasound wave velocity values started continually at 400 °C (Fig. 9B). As it is visible also from Fig. 9B, some data for the Králiky sandstone are missing due to the same reason of the necessity of retaining the samples coherence. The heated Králiky sandstone disintegrated faster and it was not possible to measure this parameter after heating at 800 and 1000 °C.

One of the key targets of the presented research was the comparison of changes in the strength of rocks. The performing of the UCS tests was, as in the case of water absorption tests, also conditioned by the retaining of the structural stability and compactness of the cylindrical specimens. The results of the strength

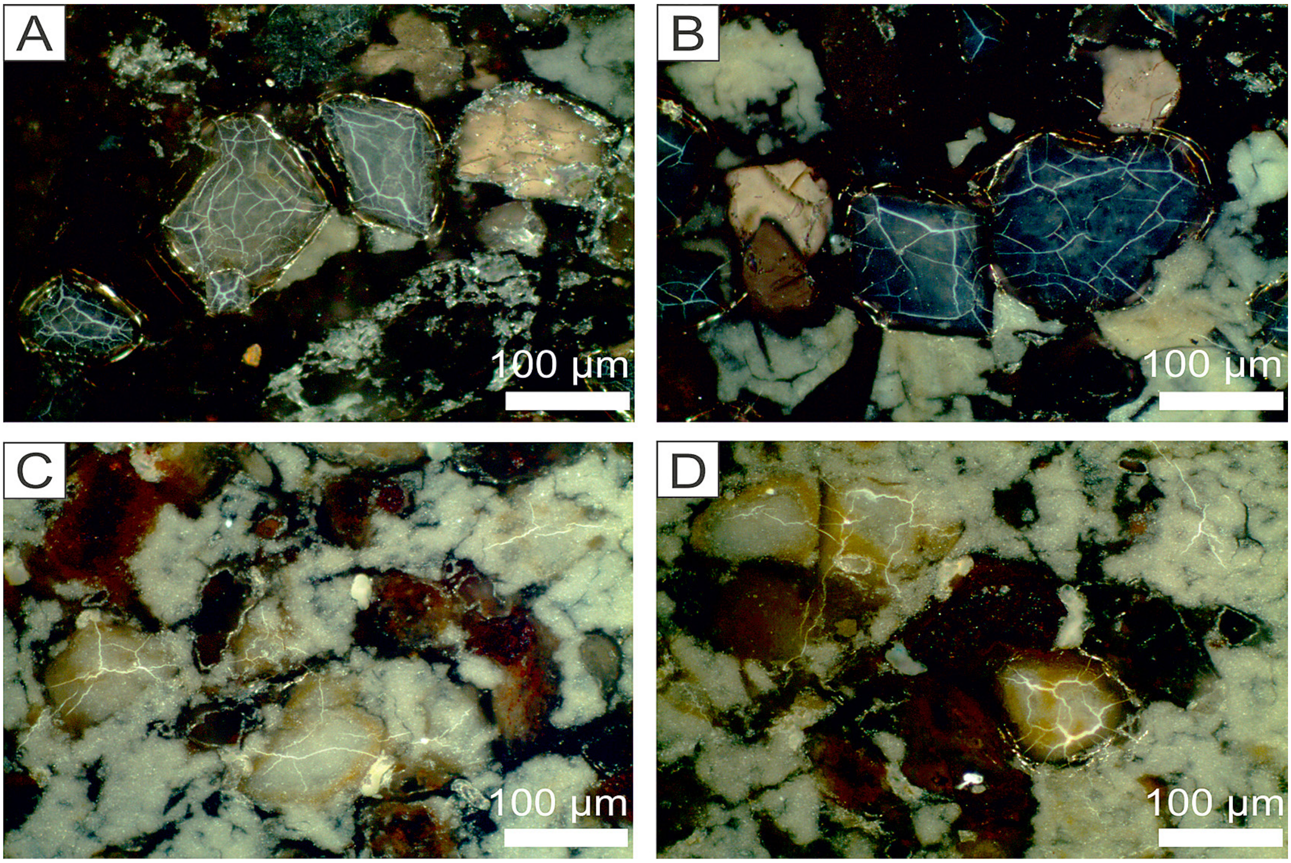

Fig. 7. Creation of micro-cracks in quartz grains of sandstones after their heating at 1000 °C: A–B) Králiky; C–D) Spišské Tomášovce.

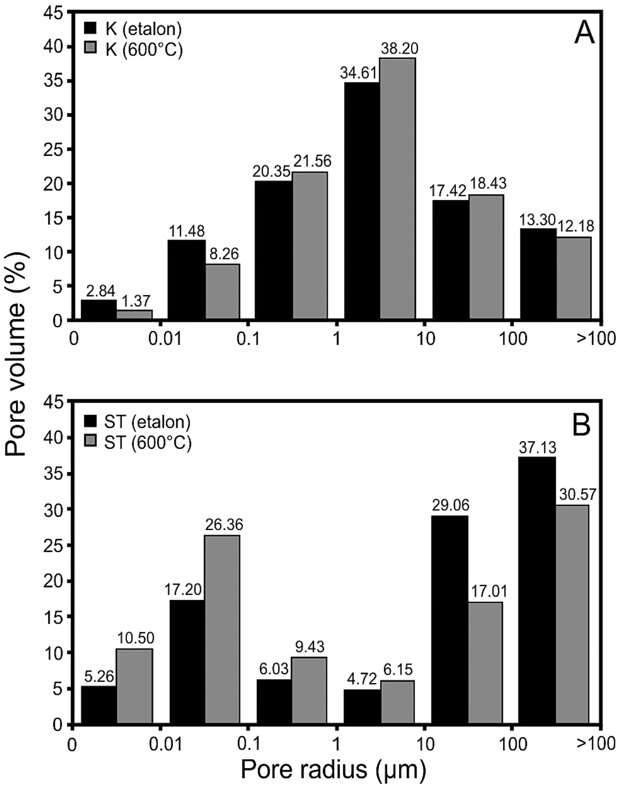

Fig. 8. Pore size distribution in sandstone samples. A – Králiky sandstone; B – Spišské Tomášovce sandstone.

Tab. 6. Main parameters of the sandstones pore structure determined by MIP.

Sandstone	Total porosity	Total volume of pores	Volume of micropores	Share of macropores (> 5.2 µm)	Share of permeable (open) pores	Specific surface area of pores
	(%)	(mm³/g)	(mm³/g)	(%)	(%)	(m²/g)
Králiky						
unheated	16.34	69.70	29.50	57.67	54.96	1.28
heated at 600 °C	14.73	63.90	25.70	59.78	59.76	0.74
Spišské Tomášovce						
unheated	4.30	16.4	4.9	70.48	70.60	0.51
heated at 600 °C	5.87	21.3	10.7	52.28	52.43	1.59

tests are given in Fig. 9C. In the graph, the mean strength values from three measurements for each temperature are given. A more significant reduction in strength was recorded on the samples after heating to temperatures above 600 °C.

Tab. 7. Physical and mechanical properties of the sand-stones – mean values (Durmeková et al., 2016, modified).

Property	Králiky	Spišské Tomášovce
Specific gravity (g/cm^3)	2.755	2.790
Bulk density (g/cm^3)	2.34	2.71
Total porosity (%)	15.1	2.9
Water absorption (%)	3.96	0.52
Ultrasound wave velocity (km/s)	3.6	5.3
Uniaxial compressive strength (MPa)	51	185

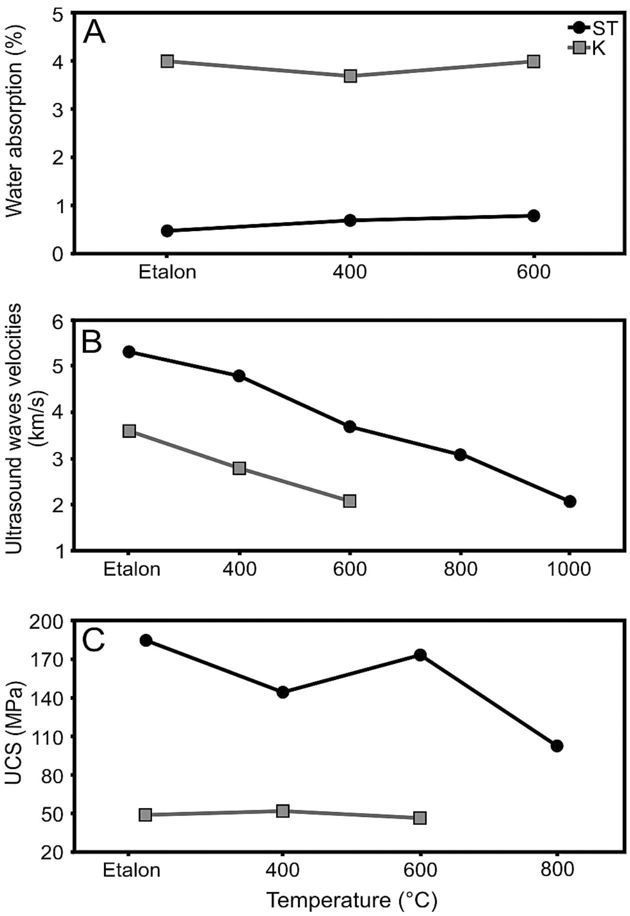

Fig. 9. Temperature versus physical properties of the sandstones: A – water absorption; B – ultrasound wave velocities; C – uniaxial compressive strength.

5. DISCUSSION

Sandstones are a rock type that are frequently researched for influences of high temperatures on property changes due to their frequent use as building and decorative stone, both in the past (historic buildings) and in the present time. The studied sandstones belong to the same geological and tectonic unit (Paleogene Subtatric Group) but they represent two different lithological types according to mineral content, structure, total porosity, and original (intact) strength.

The thermal decay induced by fire on stone materials produces strong temperature gradients between the exposed surface and sub-surface leading to linear and volumetric expansions, which create fractures, hair cracks, exfoliation, granular disintegration, powdering, flaking or gap (Gómez-Heras et al., 2009; Dionísio et al., 2012). It was also observed that the creation of inner thermal micro-cracks in fine sandstones slowly starts at temperatures above 150 °C and the first peak is reached at about 210 °C, while the second peak appears at 810 °C (Zhang et al., 2005). In our research, only minimal changes up to 600 °C were observed, and the creation of cracks in both studied sandstones was huge after heating at 800 °C and 1000 °C, and then began scaling, exfoliation and, thereafter, their total destruction (Figs. 2 and 3). As can be seen from the detailed microscopic images, the cracks were created initially between grains of minerals (Figs. 5 and 6), and, at higher temperatures above 800 °C, also across the mineral crystals (Fig. 7).

Another summarizing paper in this study area is a review of international literature published by Tian et al. (2012). Authors collected data about the effects of temperature on 12 various sandstones, especially from Chinese and German territories. Conclusions for all reviewed sandstones are the following (Tian et al., 2012): variations of bulk density are negligible below 500 °C (< 1 %); an upward trend of porosity and permeability is observed significantly above 300 °C; compressional wave velocities below 200 °C change very slightly (< 1 %); at higher temperatures a nearly linear downward trend with increasing temperature is observed. All the aforementioned is in accordance with our results obtained on two Paleogene sandstones.

It is known that the behaviour of rocks under high temperatures is influenced predominantly by their mineral composition and thermal reactions related to rock-forming minerals. In the literature, it is stated that some mineral reactions to temperature, such as the desorption of clay minerals (25 – 220 °C), the decomposition of clay minerals (400 – 700 °C), the transformation of quartz forms (573 °C) and the decomposition or calcination of calcite in a temperature range of 700 – 830 °C (Somerton, 1992 in Tian et al., 2012), cause structural damage and lead to rock weakness. In the case of sandstones, the impact intensity of high temperatures naturally depends on the mineral composition of clasts and the type of cement. Both studied sandstones contain very similar mineral grains (carbonate and quartz) but in different quantities (Tab. 3). Králiky sandstone has more quartz in the form of monomineral crystals, and from carbonate clasts it has more dolomite, and less calcite. Spišské Tomášovce sandstone has many more carbonate (predominantly calcite), but less quartz, occurring predominantly in polycrystalline form. The cement in both sandstones is calcite, accessory calcite-quartzite and is sometimes slightly calcite-clayey. The fact that the sandstones are not friable, testifies about a high cementation degree in them. The mineral composition of sandstones allows us to consider the following reactions that occurred during heating:

– at 573 °C, SiO_2, α-quartz transforms into β-quartz, which may be the cause of volume changes and the creation of microcracks;

– at temperatures above 600 °C, calcite begins to decompose, predominantly if it is present in fine-grained form;

– in the case of clay components present sporadically in the cement, these are subject to dehydroxylation, which also changes the parameters of the material structure.

All these mentioned processes probably together aid the slow decrease of physical parameters of the heated samples.

Colour changes are a visible feature after the heating process. It was confirmed these changes are closely connected to the mineral content of rocks. It seems that the dispersed iron oxides and hydroxides played a role in colour changes of the sandstones, causing the colouring to red and brown after the heating. The main colouring process after heating at 600 °C and higher was certainly the calcination of calcite and therefore both sandstones became lighter and whiter (Fig. 2). A supplementary characteristic to colour was the loss of gloss, objectively measured by the glossmeter.

In our research, the aim was to determine the UCS of the studied rocks and in such a way to objectively measure the rocks weakness. As can be seen from the diagram UCS versus temperature (Fig. 9C), changes in strength of both sandstones were not so uniquely conclusive with the expectation that strength decreases with an increase in temperature. Especially, the values of the UCS of ST specimens seem to be anomalous. It could be caused by an initial structural heterogeneity of the lithological type that was impossible to determine on the samples (e.g. unapparent microcracks present) and for this reason the number of tested samples (3 pieces at each thermal load) turned out to be as insufficient. With this in mind, it can be generalized the UCS values of heated samples did not manifest any notable changes at temperature below 600 °C what is similar as the findings of authors Hajpál & Török (1998) and Török & Hajpál (2005). These researchers presented some remarkable information about the strength properties of sandstones. They noted that the UCS and the indirect tensile strength of four sandstones have no significant changes up to 450 °C. What is more interesting, in one type of sandstone with clayey cement the strength increased at 750 °C compared to the original unheated samples (Hajpál & Török, 1998). The same sandstone, after being heated to 800 °C, manifested a rapid decrease in strength. Also studies of other researchers are in support of the increasing strength with the increasing temperature up to a certain limit (Huang & Xia, 2015). They can be summarized in a statement that the UCS and elastic modulus for sandstone increases with increasing temperature for temperatures less than 500 °C and decreases with increasing temperature for temperatures greater than 500 °C (Ranjith et al., 2012). The duration of thermal load action on the specimens certainly had an important influence on the results. This circumstance was different in each study, e. g. Kompaníková et al. (2014) heated specimens for 3 hours, Hajpál & Török (1998) for 6 hours, Ranjith et al. (2012) and Huang & Xia (2015) for 2 hours. Another influencing factor, the size of specimens, was also different in various studies.

In our experimental study, the UCS testing realized on specimens that were kept for 6 hours at the target thermal load showed that a temperature of about 600 °C may be considered as a limit or a critical temperature for sandstones. The obtained UCS values confirm the decomposition or structural breakup of stones at temperatures above 600 °C. With heating, the number and size of cracks rapidly increase. Also by MIP, it was demonstrated that the applied temperature of 600 °C has relatively little but still observable influence on sandstone microstructure. The rapid decrease of sandstone strength above 600 °C could be caused by the α-quartz transforming into β-quartz and related volume changes (predominantly in K sandstone) and by decomposing of the calcite (in both sandstones, more in ST sandstone).

A further finding of the study is that the initial high strength is not a guarantee of better rock resistance against temperature load. The thermal behaviour of both studied sandstones was very similar in spite of their different initial strength and total porosity (see Tab. 7). During the heating experiment it was observed that the Spišské Tomášovce sandstone, originally significantly stronger and with lower porosity when compared with the weak and more porous Králiky sandstone, disintegrated a little slower but eventually did so, as is shown in Fig. 3.

6. CONCLUSIONS

The heating of selected two sandstones with different initial porosity and strength enables to formulate additional findings on the effects of high temperatures on rocks in a relation to previous studies.

The most important factor in the influence of high temperatures on the sandstones is certainly their mineral composition due to the thermo-chemical reactions of rock-forming minerals and also the degree of cementation in sandstones. All changes of mineral content after the heating are evidently reflected in changes of the colour, loss of gloss and in physical properties of rocks.

Colour changes and loss of gloss in the sandstones were visible after the heating at 400 °C. The strength decreasing with increasing temperature is not clearly evident in the sandstones up to 600 °C, as can be seen in both studied sandstones. In our research, critical temperatures affecting physical properties can be seen above 600 °C. After heating at this temperature it was possible to determine all key property indicators. By the mercury intrusion porosity analysis was demonstrated that the temperature of 600 °C had relatively little but observable influence on the sandstone microstructure. After the heating at higher temperatures (800 °C and 1000 °C), such characteristics as water absorption, porosity, strength properties and many others cannot be determined. A temperature of approximately 600 °C is probably the limit for many other stone types. After exposure to temperatures above 600 °C, rocks evidently lose their original strength. Above this temperature limit, the structural and strength stability of natural stones cannot be guaranteed.

The behaviour of both sandstones at the heating was very similar in spite of their different original physical characteristics. The initial high strength is not a guarantee of the better rock resistance against the temperature load. The important role certainly plays, besides the similar mineral composition, also the high degree of the cementation in both sandstones.

A very suitable indicator of the structural and strength weakness is the ultrasound wave velocities testing, and it is reliable in every respect as the non-destructive monitoring method.

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