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To cite this article: V Martínez-Ibáñez *et al* 2021 *IOP Conf. Ser.: Earth Environ. Sci.* **833** 012026

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# Determination of thermal conductivity variation through Modified Transient Plane Source (MTPS), and its relationship with porosity variation on thermally treated Prada limestone

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**Abstract.** In this research, the variation of thermal conductivity with temperature of a limestone and its relationship with porosity is studied. Samples from Prada formation, a lower Cretaceous limestone from the Catalan Pyrenees (Spain), obtained from the Tres Ponts road tunnel were subjected to temperatures of 105, 300, and 600 °C and then cooled at a slow rate by air-cooling to laboratory temperature. Open porosity tests were determined before and after heating to evaluate the porosity increase and the micro-cracks growth. Complementarily, thermal conductivity was measured in the rock samples before and after the application of a thermal treatment by means of C-Therm TCi device, a Modified Transient Plane Source (MTPS). This is a non-invasive, quick, and precise method, when compared with other steady-state laboratory alternatives, widely used to directly determine thermal properties of rock samples. A clear decrease in the thermal conductivity of above 10% was observed for samples heated at 600 °C, probably due to a dramatic increase in porosity. The obtained results could be of great interest for the incorporation of the effect of temperature on rock in numerical models, to evaluate the potential impacts induced by eventual fires developed inside the Tres Ponts tunnel.

## 1. Introduction

The determination of the ground thermal parameters is crucial in different fields of engineering such as geothermal energy [1], underground infrastructures [2], or sustainable construction [3]. The thermal conductivity coefficient of a material represents its ability to transfer heat by conduction. It represents the amount of heat flowing per time and surface area unit ( $J/t \cdot m^2$ ) when unit temperature gradient is applied ( $K/m$ ). Thus, the thermal conductivity  $\lambda$  is expressed as  $W/(m \cdot K)$ . Similarly to other properties, thermal conductivity is characterized by a large range of factors such as temperature, porosity, degree of saturation, pore fluid, dominant mineral phase, texture and anisotropy [1]. Such properties show dramatic variations with temperature that condition the thermal conductivity of the thermally treated rocks. Research on the effects of high temperatures on the physical, mineralogical, and mechanical properties of rocks is a topic of interest, and a number of authors have specifically focused on limestones. Thus, Yavuz et al. [4] described a marked decrease on microstructure and effective porosity at 400 °C.



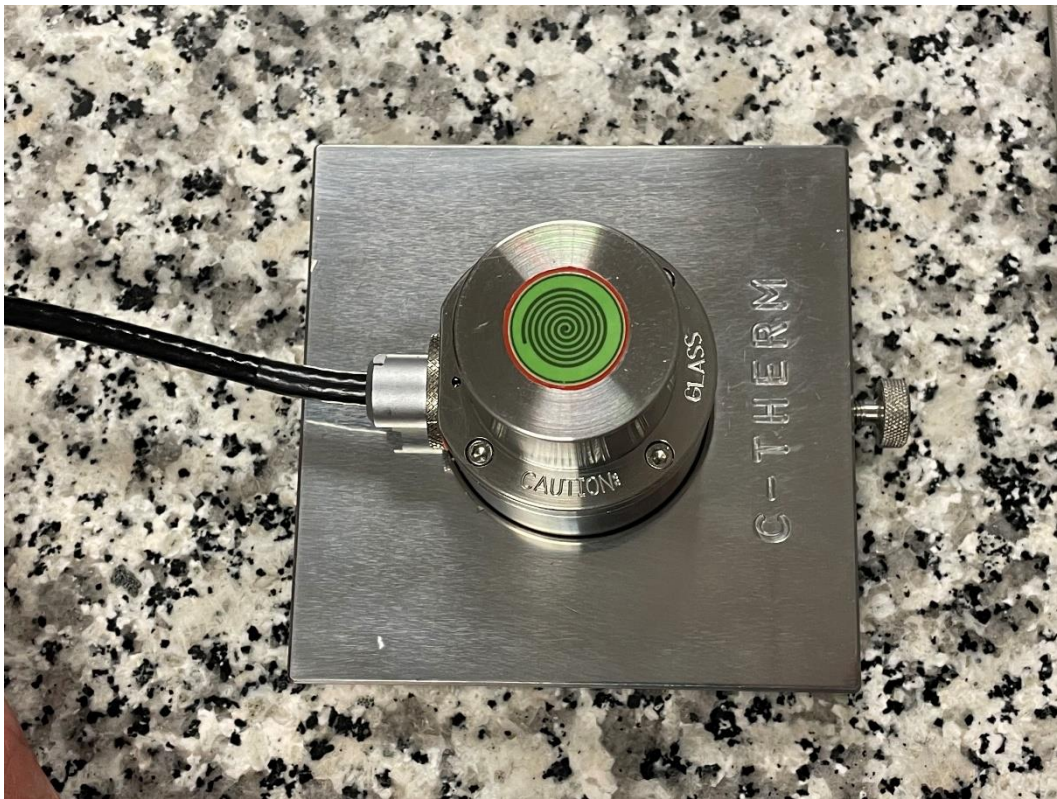
Zhang et al. [5] determined that from 200 to 500 °C porosity and pore size rapidly increased. Later, Zhang and Lv [6] described a strong relationship between the mineral content and thermal damage in limestones (China). However, few research is focused on the variation on thermal conductivity of limestones after subjected to high temperatures.

In this research, samples from two horizontal boreholes drilled in Prada limestone, where future Tres Ponts tunnel is planned to be built, have been thermally treated at temperatures of 105, 300, and 600 °C, and then cooled at a slow rate. Thermal conductivity was directly measured using a C-Therm TCi device, a Modified Transient Plane Source (MTPS), and the variation in open porosity was also determined. Understanding the variation in the thermal conductivity of rocks is of interest for tunnel fire risk assessment. Predictive numerical models of heat propagation in the rock should consider such variation in thermal conductivity, as temperatures reaching the rock mass and affecting their mechanical and physical properties strongly depend on the evolution of thermal conductivity with temperature.

## 2. Methodology

Rock samples were taken from two horizontal boreholes in Organyà, in the Catalan south Pyrenean zone (Spain). Both were drilled in a lower Cretaceous limestone formation locally named Prada limestone, widely described in previous research [7], during the design stage of the Tres Ponts tunnel. Prada limestone samples are formed mainly by calcite (more than 90%), dolomite (less than 10%) and a small proportion (less than 1 %) of quartz, pyrites and clay [8]. Six cylindrical samples were cut with a diameter of 65 mm and 20 mm in length. Limestones were separated into three groups of two samples and subjected to temperatures of 105, 300 and 600°. Thermal treatment was limited to 600 °C as the rock lost its integrity at higher temperatures due to mass cracking, and so preventing testing. A gradient of 5 °C/min was applied to reach the target temperatures and once reached, it was maintained for one hour. Later, samples were air-cooled at a slow rate to room temperature. A thermocouple registered the temperature inside the furnace every minute using a PicoLog 6 data logger.

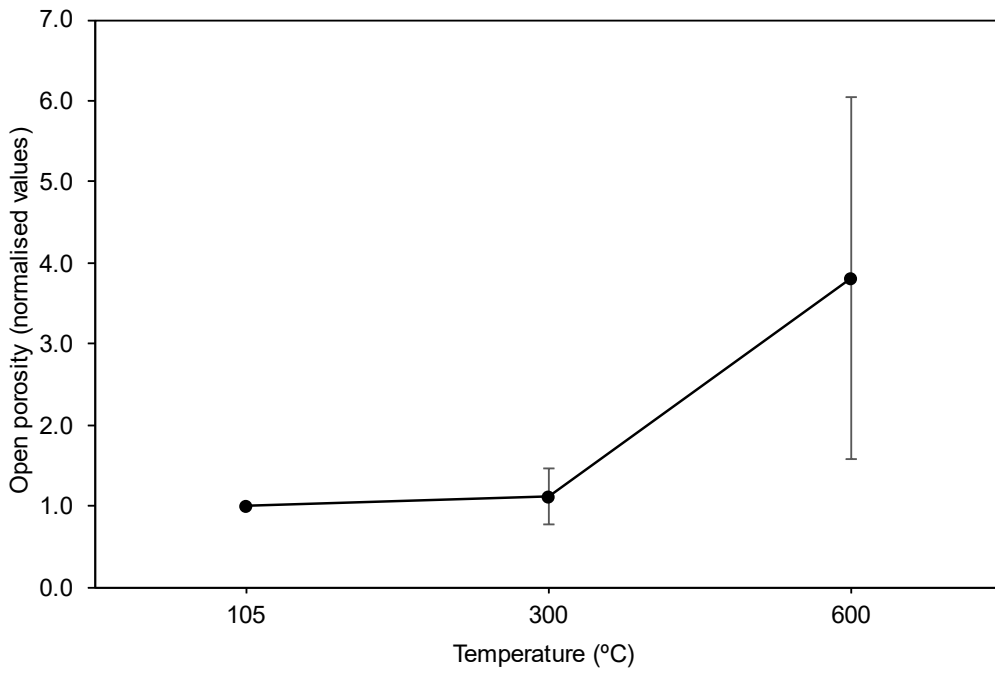
Open porosity before and after thermal treatment was determined on samples. To do so, saturation and buoyancy techniques were applied according to the suggested methods of the International Society for Rock Mechanics [9]. In this study, thermal conductivity was directly measured using a C-Therm TCi device (figure 1), a Modified Transient Plane Source (MTPS). That is a laboratory transient method that provides a non-invasive, quick and precise method when compared with other steady-state laboratory alternatives. C-Therm device accomplishes with international standard [10], and is widely used to directly determine thermal properties of geological samples [1,11,12]. Operating principle consisted on applying a known current to the sensor's spiral heating element, providing a constant one-dimensional heat source to the sample [13]. The applied current results in a rise in temperature at the interface between the sensor and the sample, which induces a change in the voltage drop of the sensor element. The rate of increase in the sensor voltage was used to determine the thermal properties of the sample, as the voltage is factory-calibrated to temperature. The thermal conductivity is inversely proportional to the rate of increase in the temperature. Samples heated to 105, 300 and 600°C were plane-cutted and faces were polished to ensure flatness. Then silica gel was applied between the sample and the sensor to reduce the contact resistance and accomplish with test requirements. Tests were repeated three times on each sample and average values and standard deviation were noted.



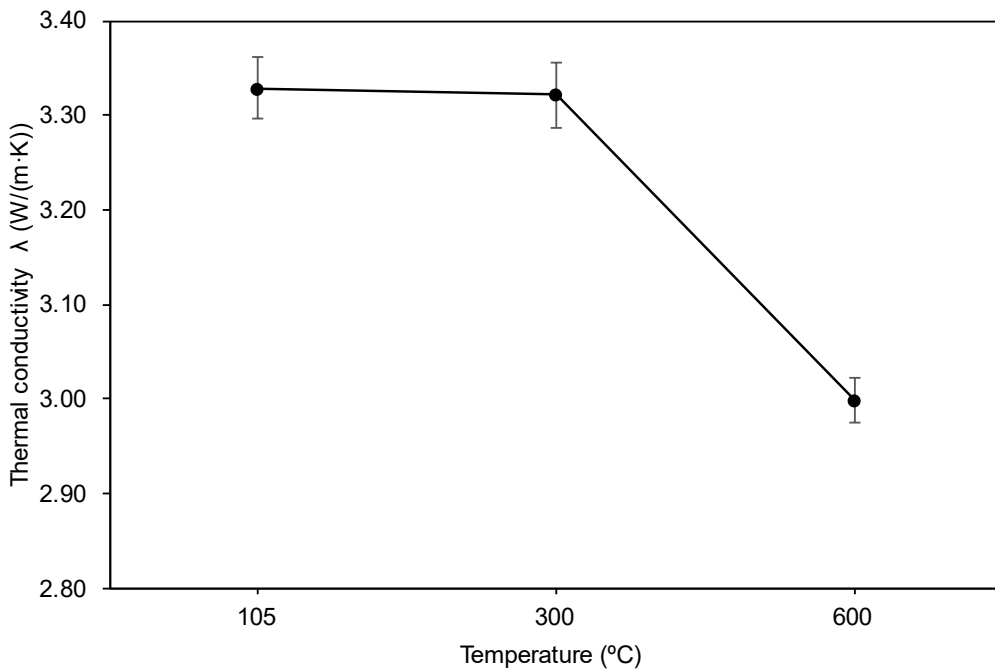
**Figure 1.** Thermal conductivity was directly measured using a C-Therm TCi device, a Modified Transient Plane Source (MTPS).

### 3. Results

Variation in open porosity with temperature was studied using mean normalised values (i.e., results after heating were divided by those of the same samples obtained at the reference temperature of 105 °C). On the one hand, the variation in open porosity was small up to 300 °C (figure 2), then dramatic variation was observed at 600 °C, where open porosity was four times greater than in samples heated at 105 °C. An appreciable rise in standard deviation was observed as the temperature increased. On the other hand, mean thermal conductivity showed little variation up to 300 °C (figure 3), then a clear decrease could be identified between 300 and 600 °C, and final value at 600 °C was 10% smaller than in samples heated at 105 °C.



**Figure 2.** Open porosity variation with temperature (normalised values).



**Figure 3.** Thermal conductivity variation with temperature.

#### 4. Discussion

A growing trend in open porosity from 300 to 600 °C was previously reported by other authors (e.g. Yavuz et al. [4]) according to the microstructural changes produced during heating process, specially between 300 °C and 600 °C. Changes involve an increasing number and size of fissures for that interval of temperatures. Such thermal damage in Prada limestone is linked to different mechanisms such as the anisotropic expansion of calcite, since local thermal stress concentrations occur between mineral particles that lead to microcracking [4,14]. Additionally, thermal stress concentrations occur between mineral particles of different nature due to mismatch in thermal expansion coefficients, thus increasing microcracking at the range of temperatures of 400 to 500 °C. [5,15–17]. Thermal oxidation of pyrites leads to a dramatic increase in pore-pressure on pyrite-bearing limestones, resulting on increased thermal damage at above 400 °C [8]. Moreover, the presence of quartz on dark grey samples could also cause thermal damage in limestones by the mineral phase transition between 550 and 600 °C, with a strong peak at 573 °C [18,19]. The thermal damage mechanisms explained above are coherent with open porosity variation observed in Prada limestone. The standard deviation is consistent with a microstructural heterogeneity in natural materials such as Prada limestone.

Average thermal conductivity for intact Prada limestone was about  $3.33 \pm 0.03$  W/k·m. This can be considered a normal value for low-porosity limestones [1,20]. For temperatures up to 500 °C, thermal conductivity varies inversely with temperature [21], and that was specifically observed for low-porosity limestones by Zhang and Lv [6], where a slow decrease was reported up to 400 °C, followed by a severe decrease at 600 °C. Similar results were obtained for Prada limestones, since in general for sedimentary rocks, the most controlling factor on thermal conductivity is porosity [1,22]. The reason is that as air has lower conductivity than minerals, an increase on porosity lead to a high proportion of air in the material and a decrease in thermal conductivity [23]. For all above, dramatic increase in porosity must be connected with appreciable drop in thermal conductivity at 600 °C in Prada limestone.

#### 5. Conclusions

In this paper, samples from two horizontal boreholes drilled in Prada limestone have been thermally treated at temperatures of 105, 300, and 600 °C, then cooled at a slow rate. Then the variation in open porosity and thermal conductivity was analyzed. Thermal conductivity was directly measured using a C-Therm TCi device, a Modified Transient Plane Source (MTPS). The following are the primary conclusions derived from this research:

- A dramatic increase in open porosity from 300 to 600 °C reflects thermal damage in terms of increased number and size of fissures. The final value measured at 600 °C was four times greater than that measured in samples heated at 105 °C. Such behaviour can be related to well-known mechanisms such as the anisotropic expansion of calcite, mismatch in thermal expansion coefficients of minerals, thermal oxidation of pyrites, or the mineral phase transition of quartz.
- Thermal conductivity showed a total decrease of 10% at 600°C, and that can be related to dramatic increase in porosity, that leads to a high proportion of air in the material and a decrease in thermal conductivity.
- Thermal conductivity measured trough Modified Transient Plane Source (MTPS) demonstrated a precise, non-invasive, and quick alternative for thermal conductivity measurement.

Since conduction is usually the dominant heat transfer process in rocks [3], future predictive numerical models of heat propagation during a fire in the rock should consider this variation in thermal conductivity with temperature. Real effects on underground infrastructures due to a fire event would strongly depend on the final disposition of lining or reinforced concrete protecting Prada limestone, as temperatures reaching the rock mass would depend on thermal conductivity of all materials involved.

### Acknowledgements

The authors wish to acknowledge Dr. Angel Vicente Escuder from Materials Technological Institute of Universitat Politècnica de València for his guidance in the use of C-Therm device. Also, Kreum SA, Ayesa SA, Infraestructures de la Generalitat de Catalunya, S.A.U., and the Lleida regional roads authority (Servei Territorial de Carreteres de Lleida, Generalitat de Catalunya) for providing rock samples. This work was supported by the Department of Geological and Geotechnical Engineering, Universitat Politècnica de València.

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