Experimental procedure for studying the degradation and alteration of limestone slabs applied on exterior cladding

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Abstract

The studied limestone is a well known Portuguese natural stone that occurs in Valverde – Alcanede region, located in the Maciço Calcário Estremenho (centre of Portugal). This stone is used in several exterior and interior applications such as paving, cladding, masonry and decorations. Slabs made of the studied limestone were installed in ventilated facade with a “kerf” anchoring system in a building located in Valencia (Spain). After approximately five years, signs of degradation were detected on the facades through changes in color, cracks and fissures that caused instability and collapse on several slabs.

Limestone slabs comprise irregular patterns due to rock cutting across stylolites. These features represent ideal paths for fluid circulation through the slab and can be identify as vulnerability zones for exterior applications.

Experimental procedure included several laboratory analyses in order to study clays and the iron oxide contents. Microscopic petrography, XRD and SEM observations were important to identify the interaction of the clay material in stylolites and fossil contours. Results show the importance of establishing standard methods for selecting stone on cladding applications. From the results, it is possible to understand that clay minerals have a strong influence on the mechanical performance of this stone. Among other important remarks, results show the importance of the development of standard procedures that acknowledge the structure and mineral composition before setting these natural products as construction materials. Proper stone selection was found important to avoid facade degradation, and hence contribute to prevent accidents and promote user safety as well as economic impacts.

1. Introduction

The natural stone market is increasingly demanding regarding the performance of natural stone materials when applied in exterior and interior conditions. Applications, sometimes unsuccessful, can be related to a series of structural, environmental, architectural, installation and performance aspects and, not least important, with the knowledge of singularities concerning mineralogical and petrographic analysis (Baud et al 2016; Baud et al 2014;Dias et al 2020; El-Gohary 2007; Espinosa-Marzal et al 2010; Gutiérrez et al 2012; Huang et al 2020; Ivorra et al 2013; Pereira et al 2018; Pires et al 2009; Rothert et al 2007; Sebastián et al 2008; Silva et al 2009;Veniale et al 2001).

This case study refers to an experimental procedure to study the degradation of Portuguese limestone slabs with dimensions of 500x375x30 (mm) in a building facade in the city of Valencia (Spain) using a standard “Kerf” anchoring system. After 5 years of installation signs of deterioration began to be detected in the external facade of the building that led to changes in color, occurrence of cracks, swelling, fissures and loss of powdery material. These phenomena have caused instability of the facade elements with consequent implications in users’ safety and aesthetics appearance of the building (**Fig. 1**).

Figure 1 Exterior views of the building facade cladded with the studied limestone form Portugal and depicting deterioration such as cracks, fissures, colour changes and swelling. Picture credits by Juan

José Tejado Ramos.

The studied limestone is a natural stone from Portugal that for the purposed of this work was denominated as AV limestone. This stone has wide application in exterior and interior environments both in Portugal and abroad however, the main technical guidelines recommend that it should be preferably used in interior conditions (Casal Moura 2009). The reason for this concerns the fact that AV limestone may contain heterogeneous structural features such as bedding planes, stylolites, fractures, and outlines of fossils that allow the movement of water and salts on exterior applications (Casal Moura 2009; Gutiérrez et al 2012; Pires et al 2009; Sebastián et al 2008; Veniale et al 2001;). AV limestone mineralogical composition variation and the content of clay fraction and iron oxides are another potential cause for AV slabs degradation. This cause has been previously pointed in several limestone degradation studies since the aggregation/disaggregation or swelling/shrinking of the clay particles occurs when these particles interact with water causing a whole series of identifiable pathologies in building stones (Ercoli et al 2004; Gutiérrez et al 2012; Sebastián et al 2008 Veniale et al 2001). The presence of clay minerals is not usually verified when selecting a stone for cladding and this may have a negative influence on the faced since clays are minerals with expandable behavior in wet and humid environments (Ercoli et al 2004; Gutiérrez et al 2012; Sebastián et al 2008; Veniale et al 2001).

Under the light of these considerations this research conducted a laboratory analysis of AV limestone to understand the degradation phenomena and to contribute to a potential standard testing methodology on limestone clay content evaluation. Sound and altered samples of AV limestone were subjected to laboratory tests for determination of Schmidt hardness, water absorption at atmospheric pressure, stereoscopic microscopy, x-ray diffraction and scanning electron microscope. These last two assessments have been used to determine the mineralogical content and, when possible, the identification of the clay fraction.

Understanding the variability of stone characteristics increases reliability and allows lower safety factors with a direct impact on the cladding project costs. As far as the authors know, literature is scarce regarding the identification of a standard testing methodology on the impact of clay presence on the mechanical and physical properties of the selected limestone from Portugal, therefore it is clear that in this topic more research is required to gain a deeper understanding of AV limestone cladding performance. Experimental results showed that the use of additional laboratory techniques during the stone selection stage, is of critical importance particularly in stones that present microstructural and mineralogical composition variations after installed in a façade since they may substantially influence the stone performance and service life.

2. Brief Geological And Geographical Setting

AV limestone is explored in the region of Valverde and outcrops around Pé da Pedreira (Alcanede, Santarém District, Centre Portugal) (**Fig. 2** and **Fig. 3**). It belongs to the Maciço Calcário Estremenho and to the Montejunto Formation (Upper Jurassic) (Zbyszewski et al 1971). The exploited limestone beds are developed approximately EW for about 1000m long and 150m wide and are south limited by a dolerite dike (Ramos et al 2005; Zbyszewski et al 1971;).

The region is covered by 1:50000 Portuguese Geological Map (27-C, Torres Novas) (Ramos et al 2005).

The quarries have a maximum front exploration of 10m producing large dimension blocs cut parallel to the layering (Zbyszewski et al 1971).

Figure 2 Geographical setting of AV limestone explored in the region of Valverde and outcrops in Pé da Pedreira (Alcanede, Santarém District, Centre Portugal)

Figure 3 Part of the geological map (1:50 000) showing the Montejunto Formation (Upper Jurassic) where AV limestone is explored (Zbyszewski et al 1971)

3. Petrographic Characterization

3.1 Macroscopic description

AV limestone is a fine grained calciclastic and bioclastic greyish brownish limestone with some light-coloured spots. It is a compact rock of medium to high hardness, with rounded, elongated, and elliptical components with different dimensions generally corresponding to fossil joined by a carbonated cement (Ramos et al 2005).

The most common finishing stages of external surfaces are honed and polished and, in this case, the rock color becomes grayish or grayish brown (Casal Moura 2009). The macroscopic appearance of the rock can be observed in **Fig. 4**.

Figure 4 Macroscopic aspect of a sound sample of AV limestone

AV limestone has cracks and layers sub parallel to the slabs surface with variable thickness showing darker color (see **Fig. 5**).

Figure 5 Visible cracks, fissures, and layers of on example of a sound AV limestone slab

Damaged AV slabs were collected from the building façade and in these an increase in cracking, loss of material (see **Fig. 6**), swelling - shrinking and the appearance of powdery material was clearly detected (see **Fig. 7**).

Figure 6 Fractures and loss of material on the Azul Valverde slabs in the kerf/slot region where the slab interacts with the anchoring system

Figure 7 Powdery clay material on the Azul Valverde slabs

3.2 Microscopic description

Under the stereoscopic microscope, AV limestone has a complex structure composed by rounded and elliptical elements, consisting of micritic calcite (pellets) and others made of espatic calcite as fossils and oolites structures. Displays hydrated brown oxides iron on the edge of the components and a few quartz grains (Ramos et al 2005).

4. Physical And Mechanical Properties

Reference physical and mechanical properties are presented in Table 1 adapted from Casal Moura (2007). AV average properties are within the recommended minimum characteristic value for stone cladding applications regarding flexural strength (10–12 MPa) and open porosity (< 5%) (Pinto et al 2006). Based merely upon this information an architect working with AV limestone might consider that the basic characterization in enough to perform a suitable material selection. However, as this work will emphasize, in projects located in certain external environments and in ventilated facades where the slabs are installed above the 2nd floor, it is important to assure that the stone performance will fulfill the required behavior.

Table 1
Physical and mechanical average properties of the Azul Valverde limestone

Compressive strength	**150**	**MPa**
Compressive strength after freeze-thaw test	150	MPa
Flexural strength	15.0	MPa
Apparent density	2640	kg/m^3
Water absorption at atmospheric pressure	0,5	%
Open porosity	1,2	%
Thermal dilatation coefficient	3,1	$x\ 10^{-6}$ per ºC
Abrasion resistance	2,6	mm/200m

Table 1. Physical and mechanical average properties of the Azul Valverde limestone

5. Degradation Analysis Methodology

To understand AV limestone degradation phenomena and to contribute to a potential standard testing methodology on limestone clay content evaluation, degraded samples were examined and photographed under a stereoscopic microscope (Olympus SZ 51).

Physical and mechanical properties of AV limestone sound and degraded specimens (collected from the building facade) were tested for water absorption at atmospheric pressure – testing methodology according to EN 13755:2001 and were tested to evaluate Schmidt hammer rebound hardness (Aydin

2005). As in previous works developed by Amaral et al (1999), Amaral (2005), PROCEQ (1977); Schmidt rebound test was selected because of its ability to provide quick readings of macrohardness for stone materials. Despite the natural heterogeneity of AV limestone specimens, it is expected that this method can also give consistent results, even if a considerably high number of tests is needed (as in most rock mechanics measurements). To have no prior restrictions, the Schmidt hardness of AV specimens, was obtained as the arithmetic mean of at least 40 surface readings sequentially measured on a regular grid, which gives a representative measurement AV sound and degraded slabs.

X-ray diffraction qualitative analysis was made to determine AV chemical composition; Clay fraction analysis was performed on grinded AV samples in a ring mill. Two portions of 100 grams of grinded sample were chemically attacked with concentrated hydrochloric acid Scharlau 37% to annul the carbonate fraction by effervescence reaction. Samples were then washed and decanted several times to clean the excess of acid and then dried in an oven (at 110ºC) before being weighed.

A portion of 10g dried sample at 110ºC was taken to calcination on a muffle for 3 hours at 400ºC to determine the organic matter content (Davies 1974).

Milled samples were analyzed by X-ray diffraction (XRD) in the laboratory of CENIMAT (NOVA-SST) using a Rigaku Dmáx IIIC apparatus, with an x-ray λ CuKa1 for 2θ, 40 Kv at 30.000 ma.

AV degraded slabs and clay fraction samples obtained after drying, were coated with gold and chemical analyzed in the Earth Sciences Department of NOVA-SST using a SEM (JEOL 330A) associated with a Tracor (EDS) microprobe.

6. Results And Discution

Degradation consequences on AV limestone are clear and easily identified from the fissures, cracks, swelling-shrinking and material loss, but this research aims to purpose a suitable testing method to explain the degradation causes and the problem detected.

Laboratory determinations of clay content in AV degraded specimens were highly informative and detected 18% of clay and almost no organic matter (0,49 %). These results show that AV is not pure limestone but a marly limestone. After drying, this clay shows high retraction cracks, as it can be observed under the stereoscopic microscopic (**Fig. 8**).

Figure 8. Clay content of sound AV limestone with retraction cracks

The marly limestones are constituted by calcite and 15 to 25% of clay minerals and typically, within the same layer the upper part is richer in clay. Marly limestones durability studies have reported that the higher the clay content, the lower the durability of the material exposed to a certain environment since most marly limestones suffer serious problems of chipping and scaling (Beretta et al 2001; Passarello et al 2002; Vecchiattini et al 2018). This compositional heterogeneity can be overcome only through a careful selection of raw materials, usual practice in historical projects for construction of major importance but less obvious in more recent times (Beretta et al 2001; Passarello et al 2002; Vecchiattini et al 2018)

XRD mineralogical analysis on AV degraded specimens (**Fig. 9**) revealed the presence of calcite, a small amount of quartz and montmorillonite (expansive clay mineral from smectite group); no iron oxides were detected by this technique. According to a study published by Delgado Rodrigues (1976), swelling/Shrinking can be defined as the volume increase experienced by any solid body when wetted. This phenomenon is typically associated with the hydration of crystalline substances and with the intra and interparticle adsorption and absorption of water by clay and a few other minerals having peculiar crystalline structures such as the smectites detected in this research with AV limestone (Delgado Rodrigues 1976).

Figure 9 XRD analysis of sound AV limestone

As stated by Delgado Rodrigues (1976) and by Delgado Rodrigues (2001), the harmful role of clays derives from their capacity to induce deformation inside the rock structure. When this deformation is restrained from increasing, the clay components build up internal stresses, up to the point of causing rupture and showing damaging results such as the ones presented in this case study (Delgado Rodrigues 1976; Delgado Rodrigues 2001).

SEM observations allowed a detailed analysis of the clayed veins in AV degraded specimens (**Fig. 10a**) and the microprobe analysis on the clay fraction (**Fig. 10b**) confirmed the presence of a Ca-montmorillonite, a very expansive clay of the smectite group.

Figure 10 SEM observation of a clay vein on Azul Valverde limestone **(a)** and microprobe analysis of clay fraction **(b)**.

As reported in previous studies by Delgado Rodrigues (1976), Delgado Rodrigues (2001) and by Sebastián et al (2008), the presence of clays inside a solid rock has always a negative potential. Clay minerals are linked to the stones mechanical performance reduction and affect other physical properties, particularly when durability is concerned (Delgado Rodrigues 1976). Many authors have pointed out the important part that clays play in natural stone performance and many studies have been dealing with limestone degradation due to this cause (Delgado Rodrigues 1976; Delgado Rodrigues et al 1990; Drew et al 1970; Félix et al 1988; Rodríguez-Navarro 1997; Struillou 1968; Thénoz et al 1968; Tourenq et al 1970;Weinert 1968; Wiis et al 2000). Clays, in this study Ca-montmorillonite, are phyllosilicates with a pronounced laminar crystalline structure. Their peculiar structure can undergo swelling-shrinking when water or other polar liquids interact with them. Expansive clays such as smectites can expand their crystal lattice as an intraparticle swelling mechanism, while others are non-expansive (kaolinite and illite) (Delgado Rodrigues 1976; Delgado Rodrigues 2001). The extent of degradation will depend on several factors, namely on the type and amount of clays, on their form of incidence inside the stone, on the interparticle and interlayer distances of clay particles, as well as on the composition and availability of percolating solutions (Delgado Rodrigues 1976; Delgado Rodrigues 2001).

Water absorption at atmospheric pressure of sound and altered specimens of AV Limestone are shown in Table 2.

Table 2
Water absorption at atmospheric pressure on sound and degraded AV specimens

Water absorption at atmospheric pressure [%]	
Sound specimens	Degraded specimens
0.5 ± 0.01	1.0 ± 0.01

Table 2. Water absorption at atmospheric pressure on sound and degraded AV specimens

Water absorption (%) comparison between reference and degraded AV limestone specimens show an increase of 50%, denoting that the degraded slabs will absorb more water because they have more fissures and cracks due to the loss of material caused by the limestone swelling-shrinkage. Similar results were reported in previous studies water absorption growth affects the limestone degradation trend and represents an important variable that may change with time, especially in the subsurface layer where the main physical, chemical, and biological attacks occur (Bellopede et al 2016; Delgado Rodrigues 1976; Delgado Rodrigues et al 1990; Delgado Rodrigues 2001; Drew et al 1970; Félix et al 1988; Rodríguez-Navarro 1997; Sebastián et al 2008; Struillou 1968; Thénoz et al 1968; Tourenq et al 1970;Weinert 1968Wiis et al 2000).

Schmidt hammer rebound hardness results are displayed in Table 3. Results corroborate the previous microstructural and physical analysis and show a decrease of 38% in the rebound hardness on the degraded slabs confirming AV loss of structural performance linked to the macroscopic features detected: i) increase of the fractures along slabs width and thickness; ii) opening of structural plans - which caused structural breakdown of planes; ii) disaggregation of fragments by clay swelling-shrinkage.

Table 3
Schmidt hammer rebound hardness for sound and degraded AV limestone.

Schmidt hammer rebound hardness	Sound	Degraded
Average	45.0 ± 5.0	28.0 ± 7.0
Variance coeficient [%]	10.0	14.0

Table 3. Schmidt hammer rebound hardness for sound and degraded AV limestone.

Experimental results seem to corroborate that the presence of expansive clay minerals and the existence of stratification features and irregular fractures (normally parallel to the surface of the rock in the facade) are the main causes for AV degradation and loss of mechanical performance.

Results suggest that the selected experimental methodology based upon macroscopic analysis, clay content evaluation, X-Ray mineral diffraction analysis and microscopic examination; display that this methodology constitutes in an appropriate way to quantify AV degradation process.

The suggested approach can contribute to the draft of a standard method for previous knowledge of the structure and mineralogical composition of carbonate rocks, allowing a proper and suitable selection of these construction materials in buildings where they can exhibit all their aesthetic and functional characteristics preventing possible economical and safety negative impacts.

AV limestone is one of the most known and chosen dark-colored limestone extracted in Portugal. Normally, it is used in facades with polished and honed finishing surfaces. But this material is frequently seen in various other applications such as external and internal paving with moderate traffic, masonry, stonework, furniture, among others. Despite its broad use, AV limestone shows limitations depending on the external environmental conditions. Regions with high-temperature variation, high humidity conditions, coastal environments with saline fog spray (such as the ones presented in this case study) are typically aggressive and contribute to permanent structural damages after a few months or years of application, for these reasons AV limestone can be less suitable for applications in external environments. The experimental method and analysis reported in this research work reinforce the need for proper systematic characterization. After five years installed on a ventilated, AV limestone started showing degradation signs detected through color changes, loss of gloss, cracks and fissures that caused instability and rupture on several slabs. These limitations have been previously pointed out by other authors mentioning that AV limestone should preferably in interior environments (Casal Moura 2009).

7. Conclusions And Final Remarks

With this study, it was possible to highlight the importance of complementary testing methodologies for natural stone when clay materials may exist to allow the identification and understanding of the potential stone degradation in certain external environments. AV limestone slabs were installed in a ventilated facade fixed with a kerf system on a building in Valencia (Spain). Sound and degraded specimens were studied in order to find the source of the problem.

AV limestone is a natural stone whose physical-mechanical properties allow it to be used as a product in a wide range of in interiors and exteriors applications. However, the application of this stone on the outside environment must be conducted under certain technical constraints.

Due to the presence of clay minerals (Ca-montmorillonite) AV limestone is sensitive to temperature variations and sensitive to contact with water or moisture, originating alterations in color, increasing fracturing, swelling, and leading to loss of material. When installed in areas near the sea under saline and humid environments the degradation process can be accelerated.

Experimental results revealed large amounts of expandable clays in the studied limestone, depicted through slabs with high fracturing potential and weakness of structural plans (stratification and stylolites) parallel to the surface of the slabs. These features lead to physical and mechanical changes in the facade. Clays have a critical play in this limestone mechanical performance and AV swelling/shrinkage will change according to the environmental conditions. Water and humidity in exterior walls can penetrate through the stone cladding slabs causing the expansion of the clay minerals and originating powdery material. The areas surrounding the main rock components (pellets, oolites, fossils) are also sensitive areas to temperature and water circulation.

Very few projects ask for clay analysis during the stone selection stage and due to this the swelling potential of most natural stones is not properly listed to establish future performance.

As a final remark the work demonstrates that the selected test set-up and chosen methodology may help to bring clear information on a stone selection procedure required to be conducted before starting the application of limestone materials such as AV. Complementary studies of the rock mineralogy, microscopic and petrographic analysis, as well an environmental assessment of the area, are important to advise the best solutions for the application of natural stones in order to prevent the material's degradation, thus contributing to avoid negative safety, economic and aesthetic impact.

Declarations

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Conflicts of interest/Competing interests

The authors have no conflicts of interest to declare. All co-authors have seen and agree with the contents of the manuscript and there is no financial interest to report. Authors certify that the submission is original work and is not under review at any other publication.

Availability of data and material

The authors confirm that the data supporting the findings of this study are available within the article [and/or] its supplementary materials.

Code availability

Not applicable.

Authors' contributions

Vera Pires: Conceptualization, Resources, Methology, Formal analysis, Writing- Reviewing and Editing.

P.M. Amaral: Conceptualization, Formal analysis, Writing- Reviewing and Editing

J.A.R. Simão: Conceptualization, Resources, Methology, Formal analysis, Writing- Reviewing and Editing.

1. Galhano: Conceptualization, Formal analysis, Writing- Reviewing and Editing.

Ethics approval

Not applicable.

Consent to participate

Informed consent was obtained from all individual participants included in the study.

Consent for publication

All co-authors have given their consent for publication.

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Figures

Figure 1

Exterior views of the building facade cladded with the studied limestone form Portugal and depicting deterioration such as cracks, fissures, colour changes and swelling. Picture credits by Juan José Tejado Ramos.

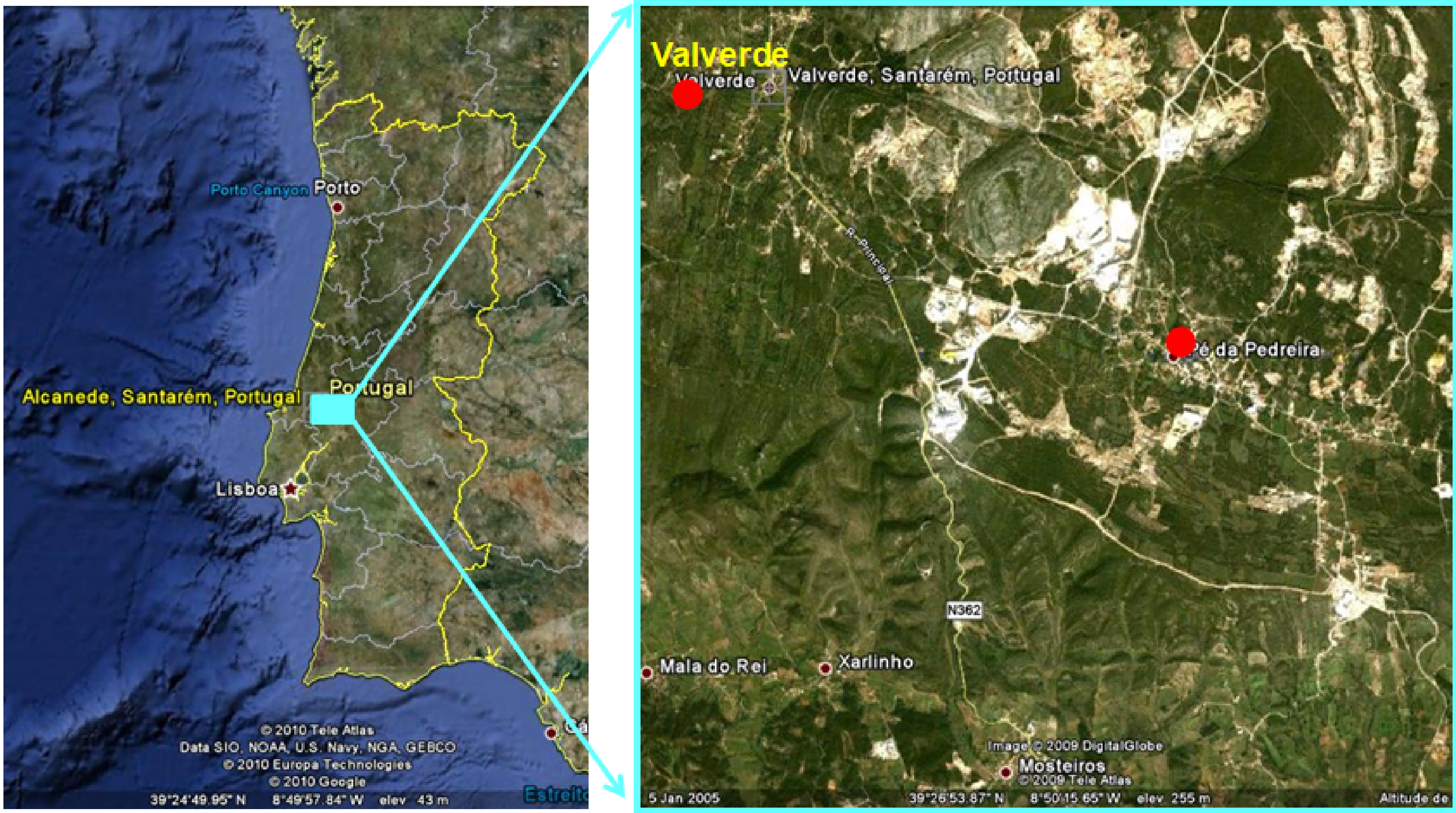

Figure 2

Geographical setting of AV limestone explored in the region of Valverde and outcrops in Pé da Pedreira (Alcanede, Santarém District, Centre Portugal)

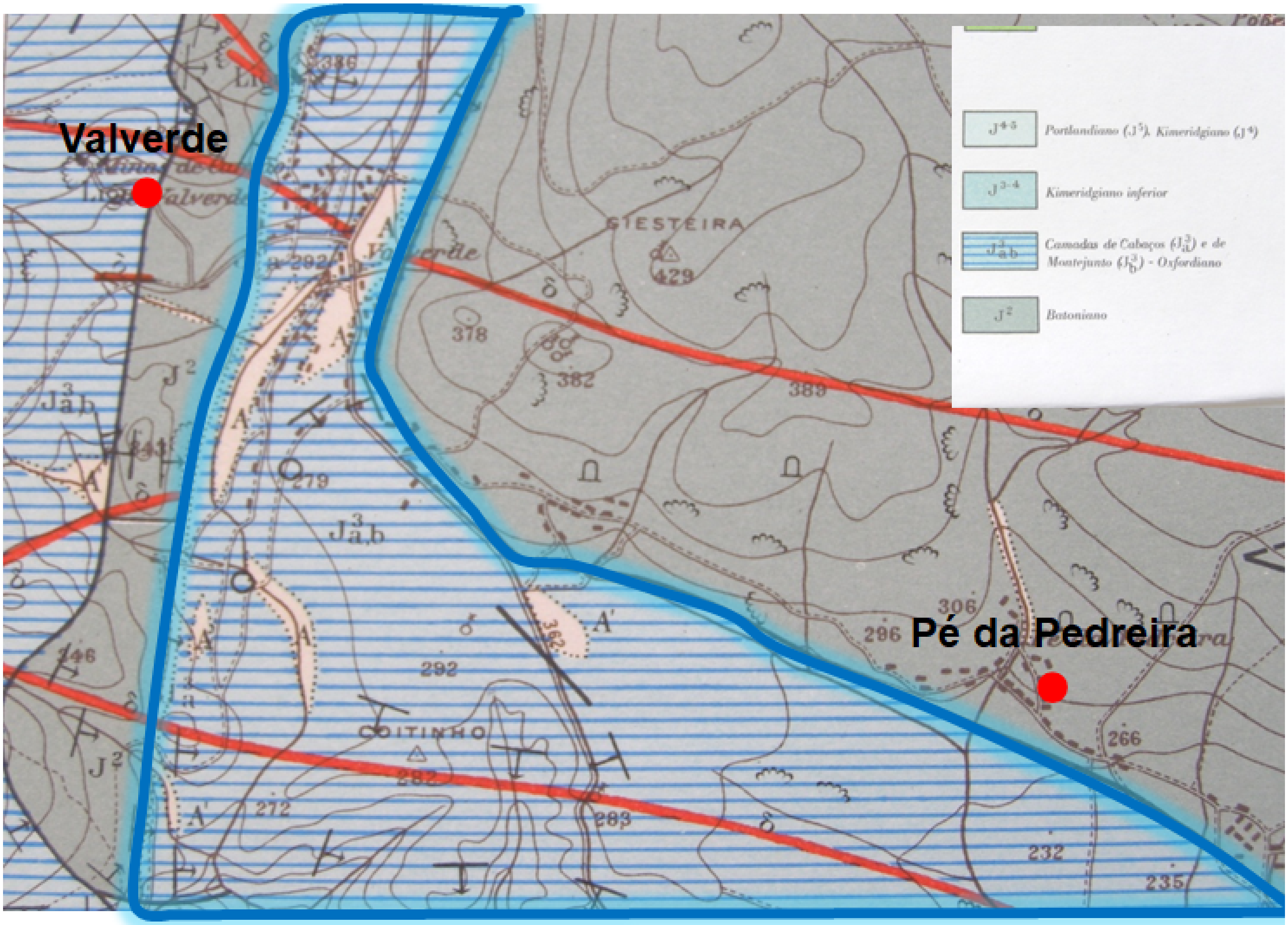

Figure 3

Part of the geological map (1:50 000) showing the Montejunto Formation (Upper Jurassic) where AV limestone is explored (Zbyszewski et al 1971)

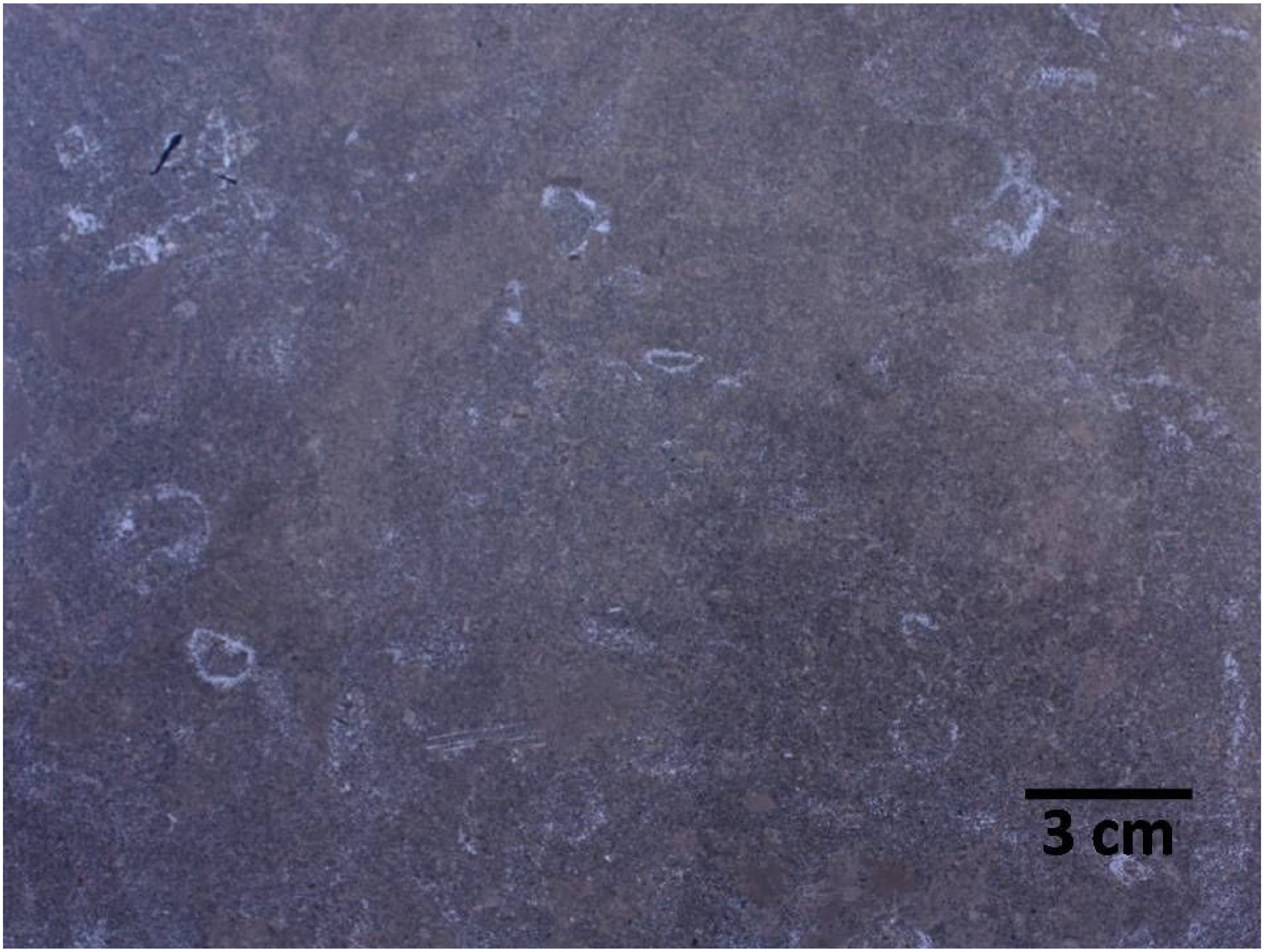

Figure 4

Macroscopic aspect of a sound sample of AV limestone

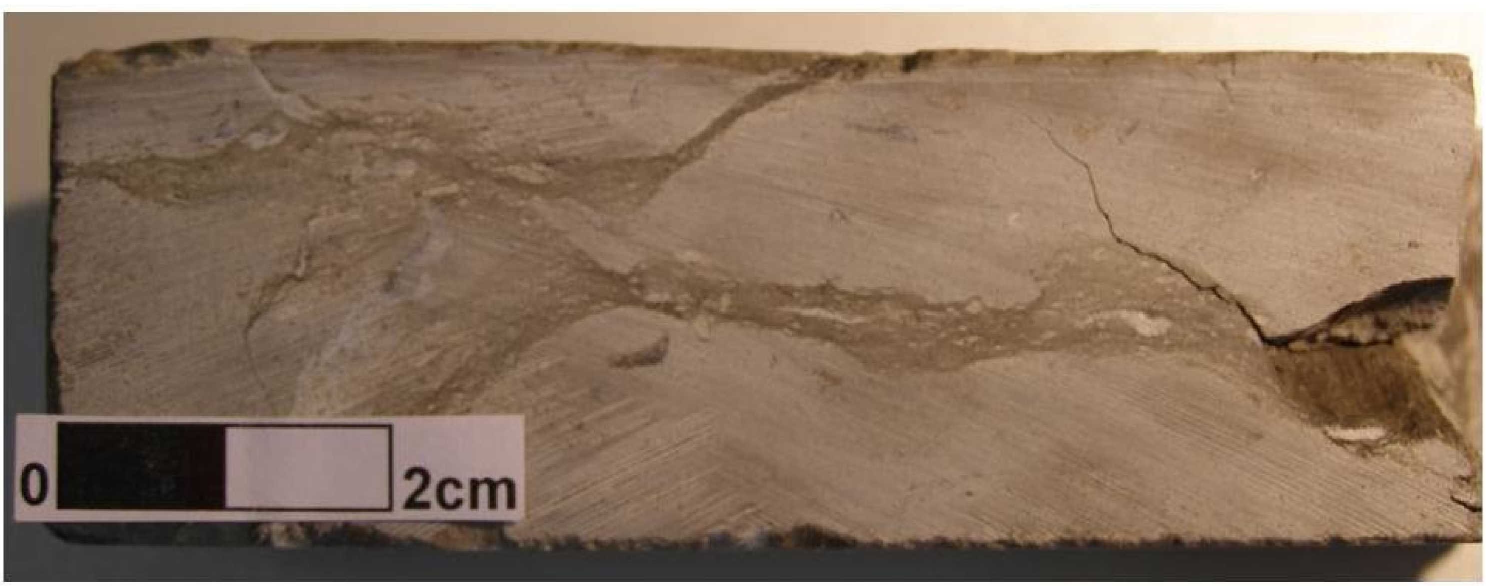

Figure 5

Visible cracks, fissures, and layers of on example of a sound AV limestone slab

Figure 6

Fractures and loss of material on the Azul Valverde slabs in the kerf/slot region where the slab interacts with the anchoring system

Figure 7

Powdery clay material on the Azul Valverde slabs

Figure 8

Clay content of sound AV limestone with retraction cracks

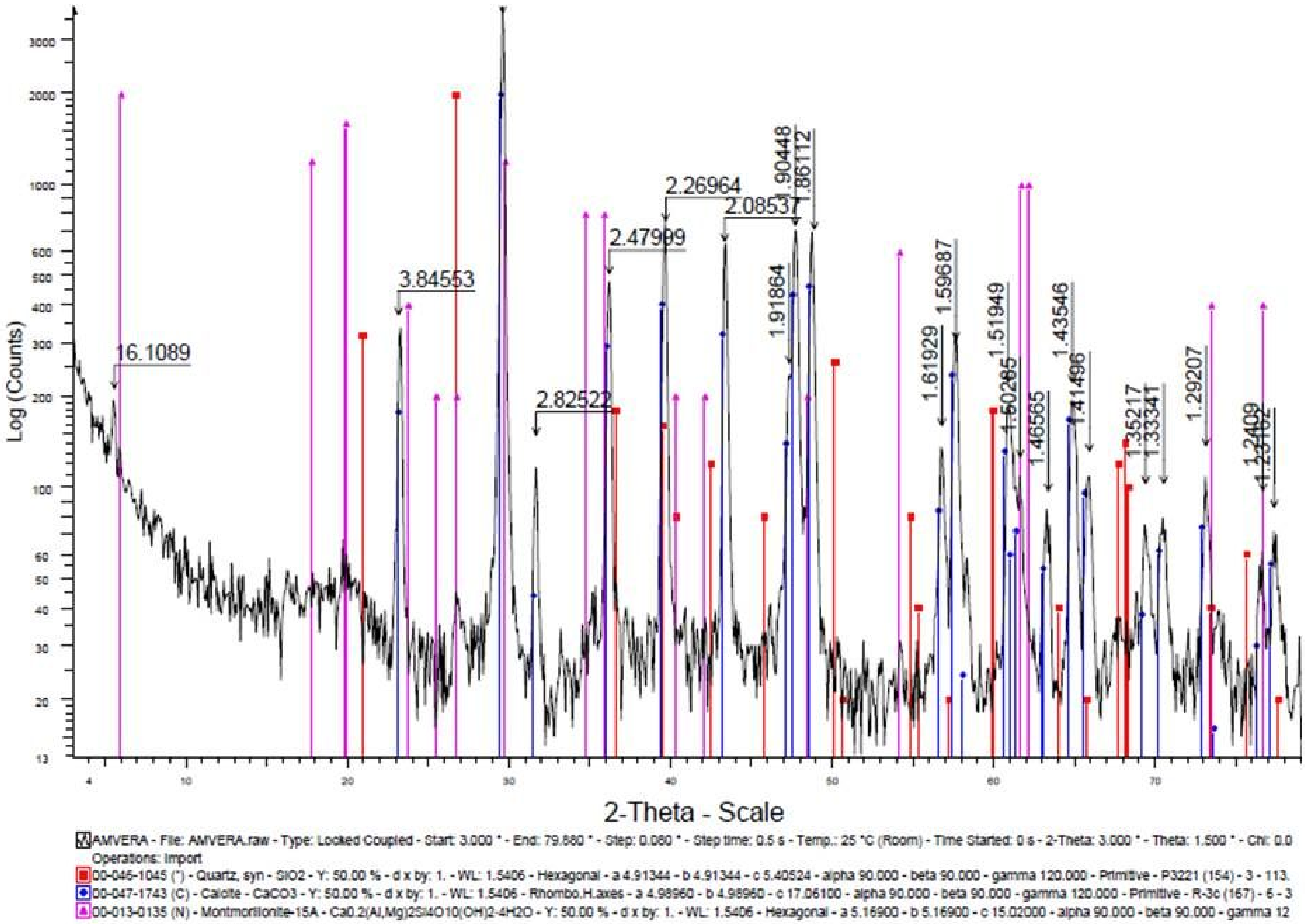

Figure 9

XRD analysis of sound AV limestone

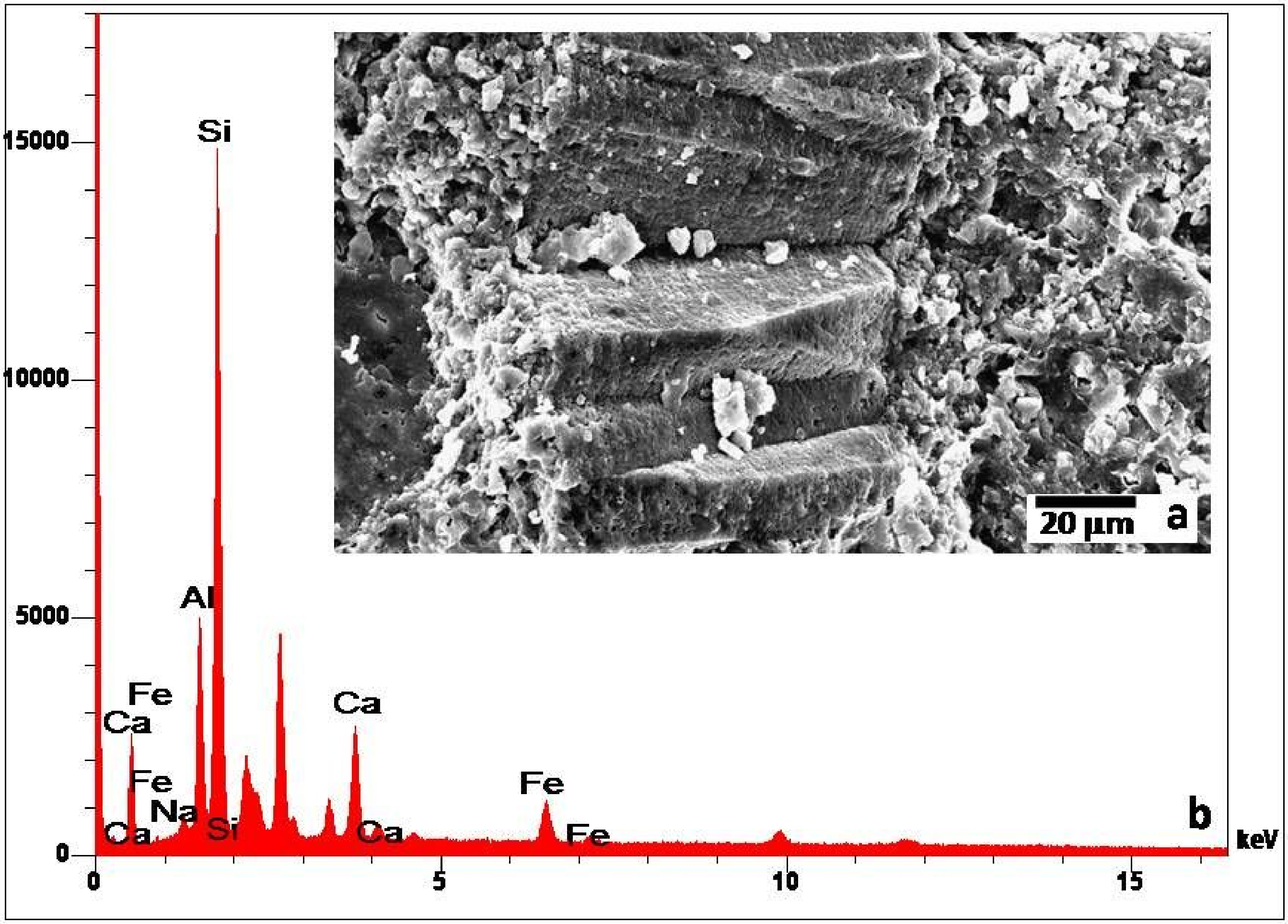

Figure 10

SEM observation of a clay vein on Azul Valverde limestone (a) and microprobe analysis of clay fraction (b).