VNIR-SWIR spectroscopic study of samples from the sepiolite-palygorskite polysomatic series

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Palabras Clave: Sepiolita, Palygorskita, Espectroscopía VNIR-SWIR. **Key Words:** Sepiolite, Palygorskite, VNIR-SWIR spectroscopy.

INTRODUCTION

In recent years there has been an increasing interest in the use of the VNIR-SWIR reflectance spectroscopy (350–2500 nm) techniques as tools for studying samples containing different mineralogy and coming from different environments due to the increasing availability of high resolution portable equipments that can be used both in the laboratory and in the field. VNIR-SWIR spectroscopy is, precisely, used to distinguish clay minerals, since in this wavelength range the absorption features are originated by the vibration of the water molecules and of the OH^- groups bonded with different octahedral cations. However, there are several difficulties discriminating between certain clay minerals and quantifying them through VNIR-SWIR spectroscopy (Cuadros et al., 2019), because they present a chemical, structural and textural complexity, among other variables, that deeply influence the spectra of samples. In this work, a first approach at the identification of different terms of the continuous polysomatic sepiolite-palygorskite series through VNIR-SWIR spectroscopy has been performed.

MATERIALS AND METHODOLOGY

For this work, 32 samples of high purity from different locations have been studied. These samples represent different terms of the sepiolite-palygorskite continuous polysomatic series, ranging from pure sepiolite, Al-sepiolite, Mg-palygorskite and to pure palygorskite, and they have been previously identified and classified according to their crystal-chemistry obtained by using analytical electron microscopy (AEM) in transmission electron microscopy (TEM). The VNIR-SWIR spectra were acquired using an ASD FieldSpec 4 Standard-Res spectroradiometer at the laboratory. The spectroscopic data was treated using software ASD ViewSpecPro. The statistical treatment was performed using software packages SPSS and Past.

RESULTS AND DISCUSSION

The VNIR-SWIR spectra of the samples apparently helps clearly identify the samples as sepiolites and palygorskites. The differences are located in the IR region. The samples that correspond to the sepiolite extreme term, as shown in figure 1a, are characterized by a doublet of absorption bands at 1388 and 1415 nm, whose origin are the vibrations of the OH- groups, and an absorption band at 1910 nm, originated by the vibrations of water related to their structures. At 2311 nm they exhibit another absorption band due to the Mg-OH vibrations. In the case of palygorskite samples, they present the characteristic absorption bands at 1415 nm and 1910 nm and another at ~2207 nm due to the Al-OH vibrations. In intermediate terms, such as sample E9, which is an Al-sepiolite, and sample E10, a Mg-palygorskite, it is possible to observe the coexistence of the features at 2207 and 2311 nm. These features are better seen at the second derivative of the spectra.

Although apparently samples closer to the palygorskite or sepiolite extremes can be easily discriminated, if a multivariate clustering of full spectra is performed, samples identified as different minerals fall together. Using the second derivative of the spectra, which performs a normalization of the spectra and avoids the influence of the global reflectance, samples are much better classified, although there are still some samples which are mixed. This is most likely caused because they are intermediate terms of the continuous polysomatic series and show both absorption feature corresponding to Al-OH and Mg-OH vibrations. An example of this is sample E10 (Fig. 1), which was clearly identified as palygorskite in XRD but shows both features with an intermediate spectrum.

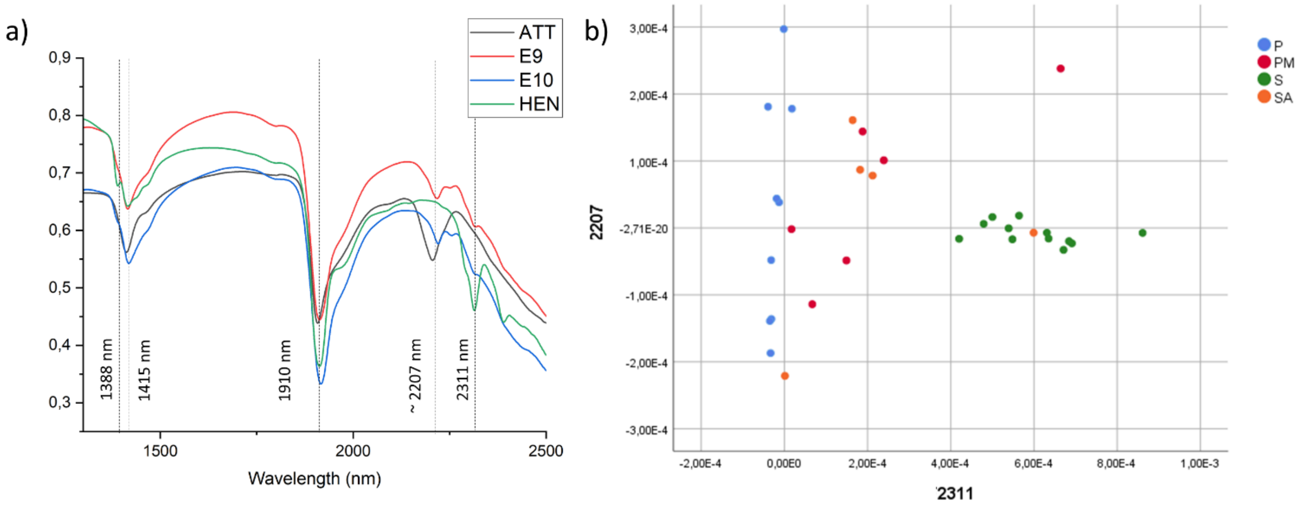

***Fig 1**. a) Spectra of samples ATT (palygorskite), E10 (Mg-palygorskite), E9 (Al-sepiolite) and HEN (sepiolite). b) Plotting of the relative intensities from the second derivative of the absorption features at 2207 nm vs 2311 nm. P: palygorskite; PM: Mg-palygorskite; S: sepiolite; SA: Al-sepiolite.*

When plotting the relative intensities obtained at the second derivative of these two characteristic features of both sepiolite and palygorskite, using the crystal chemical data and classifying the samples as pure palygorskites, sepiolites and intermediate terms, it is possible to observe a good grouping of the samples. Pure palygorskites fall along a well-defined line where the relative intensity of the feature at 2311 nm is zero or close to that value (Fig. 1b). In intermediate terms of the continuous polysomatic series, the relative intensity of this feature tends to progressively increase, until reaching their maximum for pure sepiolite terms. It must be pointed out that in the case of sepiolite both absorption features are always present.

CONCLUSIONS

The experimental data allows us to determine that there are differences between the intermediate terms of the sepiolite-palygorskite polysomatic series. Considering that the vibrations of the bonds originate in the polysome, this technique does not allow to discriminate if the spectrum corresponds to an intermediate mineral or to a mixture of two extreme terms, therefore the comparison between a spectrum of a natural sample and a set of artificial and progressive mixtures of two extreme minerals would allow to quantify the percentage of polysomes that a particular intermediate sample contains by comparison.

ACKNOWLEDGEMENTS

Financial support of the SA0107P20 (Consejería de Educación, Junta de Castilla y León, Spain) project.

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