



Contents lists available at SciVerse ScienceDirect

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saa

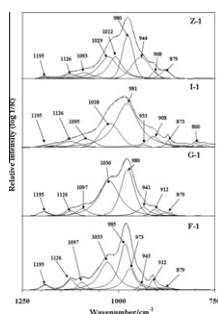
An infrared spectroscopic comparison of four Chinese palygorskites

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HIGHLIGHTS

- ▶ Infrared spectroscopy was used to characterize and evaluate the palygorskite.
- ▶ Several types of water molecules were observed in the mineral.
- ▶ A remarkable difference in the hydroxyl groups and water was observed by infrared spectroscopy.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 3 April 2012

Received in revised form 2 July 2012

Accepted 9 July 2012

Available online 17 July 2012

Keywords:

Infrared spectroscopy
Sepiolite
Palygorskite
Carbonate
Clay

ABSTRACT

Infrared spectroscopy has been used to characterize and compare four palygorskite mineral samples from China. The position of the main bands identified by infrared spectra is similar, but there are some differences in intensity, which are significant. In addition, several additional bands are observed in the spectra of palygorskite and their impurities. This variability is attributed to differences in the geological environment, such as the degree of weathering and the extent of transportation of the minerals during formation or deposition, and the impurity content in these palygorskites. The bands of water and hydroxyl groups in these spectra of palygorskite samples have been studied. The characteristic band of palygorskite is observed at 1195 cm^{-1} . Another four bands observed at 3480 , 3380 , 3266 and 3190 cm^{-1} are attributed to the water molecules in the palygorskite structure. These results suggest that the infrared spectra of palygorskites mineral from different regions are decided not only by the main physicochemical properties of palygorskite, but also by the amount and kind of impurities.

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Introduction

Palygorskite, with the ideal chemical composition $\text{MgAl-Si}_4\text{O}_{10}(\text{OH})_4\cdot\text{H}_2\text{O}$, is a crystalline hydrated magnesium silicate with a fibrous morphology. Strictly the formula is for sepiolite. Upon substitution of the Mg by for example Fe, then the term palygorskite is used. Palygorskite is known to form a continuous two dimensional tetrahedral sheet, but differs from other layered silicates in lacking continuous octahedral sheets [1]. Palygorskite and sepiolite

are fibrous clay minerals reported in soils of arid and semi-arid regions [2]. The structure of palygorskite contains ribbons of 2:1 phyllosilicates linked by periodic inversion of the apical oxygens of the continuous tetrahedral sheet every six atoms of Si (three tetrahedral chains) for sepiolite and every four atoms of Si (two tetrahedral chains) for palygorskite. The tetrahedral sheet is continuous across ribbons but the octahedral sheet is discontinuous as a result of the periodic inversion, and terminal octahedral cations must complete their coordination sphere with water molecules referred to as coordinated water [3,4].

Palygorskite is widely applied in many fields of material industry, particularly as oil refining, wastewater treatment, removal of odor, drug, and pesticide carriers, catalysts, paper and detergent

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Table 1
Palygorskite samples from China.

Palygorskite Samples	Location	Content of clay mineral	Impurities
Palygorskite (F-1)	Feidong, Anhui province of China	Palygorskite	Quartz
Palygorskite (G-1)	Guanshan, Anhui province of China	Palygorskite	Quartz
Palygorskite (I-1)	Inner Mongolia of China	Palygorskite	Quartz, Dolomite
Palygorskite (Z-1)	Zhangze, Jiangsu province of China	Palygorskite	Quartz, Dolomite

industries [5–8]. This mineral has some particularly desirable sorptive, colloidal-rheological and catalytic properties, in comparison with other clay minerals [9]. At the same time, palygorskite can form with organic compounds complexes of hydrophobic properties (so called organoclays) [10]. Technological applications are based on its physicochemical properties, principally on structure, composition, thermal behavior, surface area, among others, and especially in the structure and thermal stability. In general, the industrial raw palygorskite is a mixture of minerals, which contains different mineral impurities as well as major and minor clay minerals. The palygorskite clays from China with the major clay mineral palygorskite include minor carbonates including dolomite and calcite as non-clay minerals. Therefore, it is of great interest to undertake a comparative study of palygorskites from China.

Although the extensive use of palygorskite in industrial processes and its excellent characteristics for the preparation of organic/inorganic complexes, there is little information about the structural analysis of palygorskite, especially Chinese palygorskites and their impurities. It is noteworthy that there is certain variability in the formula and structure of palygorskite due to the influence of isomorphous substitution and various geological environments in different region. The aim of this work is to study how impurities and geological environment influence the position and intensity of the vibrations of the infrared spectra. Therefore, infrared spectroscopy study has been carried out on four Chinese palygorskites. By this means, valuable information can be obtained and infrared spectroscopy can be used to analysis palygorskite and differentiate the impurities.

Experimental methods

Materials

Four palygorskite samples, containing impurities quartz and dolomite, were selected for this study (Table 1). The samples were used directly, without prior size fraction separation, since one of the objectives was to determine the influence on the thermal behavior of mineral samples.

X-ray diffraction

X-ray diffraction patterns were collected using a PANalytical X'Pert PRO X-ray diffractometer (radius: 240.0 mm). Incident X-ray radiation was produced from a line focused PW3373/10 Cu X-ray tube, operating at 40 kV and 40 mA, with Cu K α radiation of 1.540596 Å. The incident beam passed through a 0.04 rad soller slit, a 1/2° divergence slit, a 15 mm fixed mask, and a 1° fixed anti-scatter slit.

Infrared spectroscopy

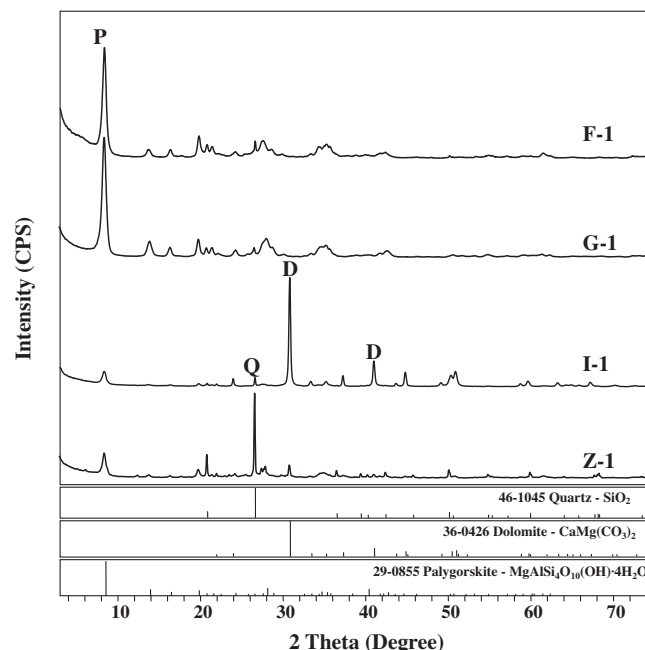
Infrared spectra were obtained using a Nicolet Nexus 870 FTIR spectrometer with a smart endurance single bounce diamond ATR cell. Spectra over the 4000–500 cm⁻¹ range were obtained by the co-addition of 64 scans with a resolution of 4 cm⁻¹ and a mirror velocity of 0.6329 cm/s. Spectra were co-added to improve the signal to noise ratio. No sample preparation was involved.

Band component analysis was undertaken using the Jandel'Peakfit'(Erkrath, Germany) software package which enabled the type of fitting function to be selected and allowed specific parameters to be fixed or varied accordingly. Band fitting was done using a Lorentz-Gauss cross-product function with the minimum number of component bands used for the fitting process. The Lorentz-Gauss ratio was maintained at values greater than 0.7 and fitting was undertaken until reproducible results were obtained with squared correlations (r^2) greater than 0.998. Band fitting of the spectra is quite reliable providing there is some band separation or changes in the spectral profile.

Results and discussion

X-ray diffraction (XRD)

The XRD patterns of the four selected palygorskite minerals with standard XRD patterns are shown in Fig. 1. These patterns show that the mineral compositions within these four palygorskite samples are various, and the notable difference between these samples is the occurrence of some impurities. Three sharp strong reflections at $2\theta = 8.32, 26.62$ and 30.86 are due to the palygorskite (P), Quartz (Q) and dolomite (D). The XRD patterns of these representative palygorskite samples from four different regions in China show that quartz is ubiquitous throughout these four samples, with slight variations in relative proportions. Samples I-1 and Z-1 contain significant amounts of dolomite. The XRD patterns of representative clay separates of these four samples reveal that these four samples have similar clay mineral compositions, consisting mainly of palygorskite. These four samples consist mainly of palygorskite. Changes in the phase compositions of the clay minerals

**Fig. 1.** XRD patterns for palygorskite samples F-1, G-1, I-1 and Z-1.

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