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# IR Spectroscopy of ammoniated phyllosilicates and mixtures with relevance for dwarf planet (1) Ceres

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## Introduction

The surface composition of (1) Ceres has been revealed with great detail by VIR spectrometer high resolution observations [1] on board Dawn spacecraft [2]. Spectroscopic observations in the infrared range 1-5  $\mu\text{m}$  have showed an average surface composition consisting of a mixture of Mg-phyllsilicate, (Mg,Ca)-carbonate, a dark absorbing phase and  $\text{NH}_4$ -phyllsilicates [3], and bright areas locally composed by mixtures of Na-carbonates, phyllsilicates, a dark phase and ammonium compounds [4]. The reproduction in laboratory of such mineral mixtures is thus of interest in order to better constraint and interpret remote-sensing observations. In this work we focus on the preparation and IR spectroscopic measurements in laboratory of  $\text{NH}_4$ -phyllsilicates and mineral mixtures.

## 1. Sample preparation and experimental setup

A set of 8 phyllsilicates were chosen from Clay Minerals Society, grinded and dry sieved to a fine grain size ( $d < 36 \mu\text{m}$ ). Ammonium phyllsilicates were then prepared following a procedure similar to what described in Bishop et al. 2002 [5]. All powders were immersed in excess (10:1 vol/mass) solutions of ammonium hydroxide (30%  $\text{NH}_3$  in  $\text{H}_2\text{O}$ ) for several days, centrifuged and decanted: this cycle was repeated more times, and finally samples were dried. Splits of these samples were separated and treated with a leaching procedure. Other endmembers, concerning the average mixture, were prepared in the form of  $d < 36 \mu\text{m}$ -powders, specifically antigorite, (Ca,Mg)-carbonate and magnetite. A set of 8 different mixtures has been then prepared, keeping fixed antigorite, dolomite and magnetite, and varying the  $\text{NH}_4$ -clay mineral constituent.

All end-members have been spectrally characterized by means of visible/infrared spectroscopy. Spectra in the VNIR have been acquired with a FieldSpec Pro in the 0.35-2.5  $\mu\text{m}$  range, with 6 mm spatial resolution and spectral resolution  $3 \div 8 \text{ nm}$ . Fourier Transform Infrared Spectrometer (FTIR-PLAB) Vertex-80 was used in reflectance mode to acquire spectra of powders, with spectral resolution  $2 \text{ cm}^{-1}$ , spatial aperture of about 6 mm, in the spectral range 1.3-14  $\mu\text{m}$ , using an MCT detector. Each acquisition was performed by summing 256 scans in order to increase S-N ratio. Reflectance spectra were acquired from all endmembers, and separately from phyllsilicates,  $\text{NH}_4$ -phyllsilicates and “leached”  $\text{NH}_4$ -phyllsilicates, and finally from mixtures.

| Clay minerals endmembers   |        |  |
|--|--------|--|
| <b>Sepiolite</b>   | SEPSP1 |  |
| $(\text{K}_{0.01})[\text{Mg}_{5.54} \text{Al}_{0.35} \text{Mn}_{0.02} \text{Fe}^{2+}_{0.04} \text{Fe}^{3+}_{0.14}][\text{Si}_{7.90} \text{Al}_{0.1}]\text{O}_{20}(\text{OH})_4$  |        |  |
| <b>Rectorite</b>   | RAR1   |  |
| $(\text{Na,Ca})\text{Al}_4(\text{Si,Al})_8\text{O}_{20}(\text{OH})_4 \cdot 2(\text{H}_2\text{O})$  |        |  |
| <b>Nontronite-1</b>  | NAU1   |  |
| $(\text{M}^{+}_{1.05})[\text{Si}_{6.98} \text{Al}_{1.02}][\text{Al}_{0.29} \text{Fe}_{3.68} \text{Mg}_{0.04}]\text{O}_{20}(\text{OH})_4$   |        |  |
| <b>Nontronite-2</b>  | NAU2   |  |
| $(\text{M}^{+}_{0.72})[\text{Si}_{7.55} \text{Al}_{0.45}][\text{Fe}_{3.83} \text{Mg}_{0.05}]\text{O}_{20}(\text{OH})_4$  |        |  |
| <b>Illite-1</b>  | IMT2   |  |
| $\text{K}_{0.65} \text{Al}_2\text{O}[\text{Al}_{0.65} \text{Si}_{3.35} \text{O}_{10}](\text{OH})_2$  |        |  |
| <b>Illite-2</b>  | ISCZ1  |  |
| <b>Montmorillonite</b>   | SCA3   |  |
| $(\text{Mg}_{0.45} \text{Ca}_{0.15} \text{Na}_{0.26} \text{K}_{0.01})[\text{Al}_{2.55} \text{Fe}^{3+}_{0.12} \text{Mn}_{\text{r}} \text{Mg}_{1.31} \text{Ti}_{0.02}][\text{Si}_{7.81} \text{Al}_{0.19}]\text{O}_{20}(\text{OH})_4$ |        |  |
| <b>Hectorite</b>   | SHCA1  |  |
| $(\text{Mg}_{0.56} \text{Na}_{0.42} \text{K}_{0.05})[\text{Mg}_{4.60} \text{Li}_{1.39} \text{Mn}_{\text{r}} \text{Ti}_{0.01}][\text{Si}_{7.75} \text{Al}_{0.17} \text{Fe}^{3+}_{0.05}]\text{O}_{20}(\text{OH})_4$                  |        |  |

Tab.1. Clay minerals endmembers.

## 2. IR Spectral measurements: results

In fig.1 an example of spectra acquired on nontronite endmember (NAU-1) is shown, in which the non-

treated sample (blue line) is compared with the  $\text{NH}_4$ -treated sample (green). The full spectral coverage has been subdivided in five ranges for clarity, specifically 0.35-2.5  $\mu\text{m}$  (FieldSpec) and then 1.3-2.5  $\mu\text{m}$ , 2.5-4.1  $\mu\text{m}$ , 4-6 and 6-14  $\mu\text{m}$  for FTIR data. The ammoniated sample is characterized by the occurrence of several new features in four of the five displayed ranges, although a global change in the overall spectrum is visible in the full range; namely a substantial decrease in reflectance level is observed, together with a reduction of spectral contrast. New absorption bands putatively attributable to  $\text{NH}_4^+$  ions appear near 2 and 2.1  $\mu\text{m}$ , near 3.1  $\mu\text{m}$  and at 7  $\mu\text{m}$ .

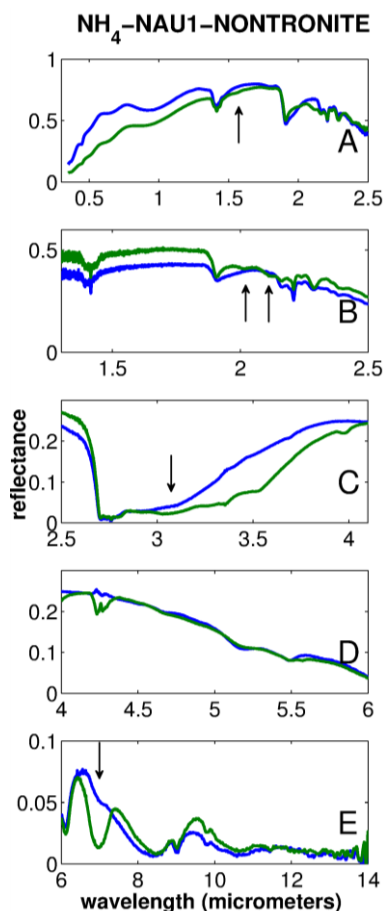


Fig.1. Nontronite NAU-1. Untreated sample (blue line) vs  $\text{NH}_4$ -treated sample (green line). A: 0.3-2.5  $\mu\text{m}$ . B: 1.3-2.5  $\mu\text{m}$ . C: 2.5-4.1  $\mu\text{m}$ . D: 4-6  $\mu\text{m}$ . E: 6-14  $\mu\text{m}$ . The  $\text{NH}_4^+$  3.1- $\mu\text{m}$  band here appears as a bump overlapped with the water band at 3  $\mu\text{m}$ , which is difficult to remove unless heating the sample in vacuum.

In fig.2 the VIR average spectrum of Ceres (purple line) [3] is compared with mixture spectra measured with FTIR. Here the mixture is composed by serpentine-antigorite, dolomite, magnetite and  $\text{NH}_4$ -

NAU1 (nontronite). Laboratory spectra show a good matching with VIR spectrum; the agreement is better for what concerns the  $\text{OH}^-$  2.7  $\mu\text{m}$ -band of serpentine and the 4  $\mu\text{m}$ -band of carbonate. The feature near 3.1  $\mu\text{m}$  due to  $\text{NH}_4^+$  in laboratory spectra is evident, although additional adsorbed water is present in the sample and influences the band.

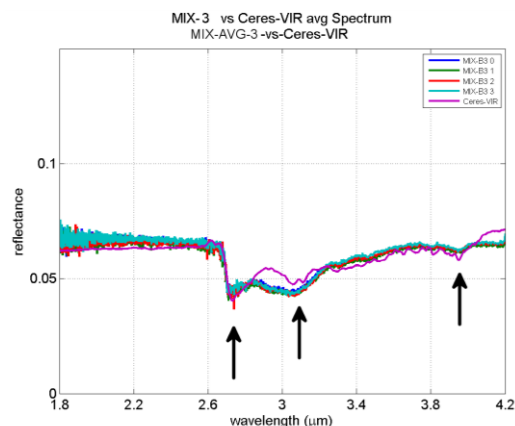


Fig.2. Dawn-VIR average spectrum of Ceres (purple line) vs Mixture AVG-3 laboratory spectra. Arrows indicate, from left to right, absorptions in the mixture due to antigorite (2.72  $\mu\text{m}$ ),  $\text{NH}_4^+$ + $\text{H}_2\text{O}$  (3-3.1  $\mu\text{m}$ ) and dolomite (4  $\mu\text{m}$ ).

### 3. Summary and Conclusions

Treatment of phyllosilicates with ammonia shows that different minerals behave in different ways:  $\text{NH}_4^+$  ions are easily accepted by several crystal structures (nontronite, montmorillonite), while other structures accept these ions with difficulty. Laboratory spectra of the mixture show a good agreement with VIR spectrum. Further work is ongoing to remove adsorbed water from phyllosilicates, in order to facilitate  $\text{NH}_4^+$  inclusion in mineral structures, and to separate  $\text{NH}_4^+$  and  $\text{OH}^-$  absorption features in the 3- $\mu\text{m}$  spectral region.

### Acknowledgements

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