

LABORATORY SYNTHESIS AND RAMAN SPECTROSCOPIC STUDIES OF FERRIC OXIDES Yanqing Xin¹, Zongcheng Ling¹*, Hongkun Qu¹, Erbin Shi¹, Changqing Liu¹. Shandong Provincial Key Laboratory of Optical Astronomy and Solar-Terrestrial Environment, Institute of Space Sciences, Shandong University, Weihai, 264209, P. R. China (zcling@sdu.edu.cn)

Introduction: To date, Fe sulfates and Fe oxides minerals have been widely found on the surface of Mars by the Mars Exploration Rover (MER) Spirit at Gusev Crater, Opportunity at Meridiani Planum, and Curiosity at Gale Crater [1-3]. The Martian orbital remote sensing (OMEGA instrument on Mars Express and CRISM instrument on MRO) have also discovered widespread alteration phases (phyllosilicates, sulfates, etc.) on the southern highlands [4]. High-albedo regions across Martian surface show some specific absorption features from 0.4 to 1.3 μm (e.g. absorption edge at 0.53 μm , absorption center at 0.85-0.9 μm) attributed to Fe^{3+} in the minerals [5]. However, those absorption features in visible and near-infrared spectra are broad in width and easily affect by other mineral phases.

Raman spectroscopy as a promising planetary spectroscopy technique for explorations on Mars [6,7]. There are already three Raman instruments selected for future Mars missions, i.e., SuperCam and Sherloc on Mars 2020, RLS on ExoMars. Raman spectroscopy has a great advantage due to its power in fast molecular phase identification in a mixture, characterization of mineral chemistry, and the minimum-to-no sample preparation requirement. Martian analogue studies in the laboratory could help us resolve those phenomena met on Mars by rovers or orbiters. Specifically, conditions of the synthesizing process of samples (e.g., ferric oxides) can reflect past Martian environmental changes, including volcanic activities, impact craterings. In combination with Mars observations, we can infer past and present environmental conditions of the Martian surface and subsurface in order to better understand the geological evolution of Mars.

In this abstract, we adopt two starting materials, i.e., hydrated ferric sulfates ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$) and $\text{Fe}(\text{OH})_3$, for the production of hematite (Fe_2O_3) in the laboratory. We focus on the two pathways of synthesis hematite. Detailed structural and spectroscopic characterizations of the products are performed using X-ray Diffraction (XRD), and Raman methods, respectively. Our goal is to obtain the critical information (e.g., decomposition temperature, spectral data) for their potential utilization (phase identifications, geological analysis) in future Martian remote sensing and in situ investigations.

Sample synthesis:

Pathway 1: We first determined the transition temperature for the production of hematite from

hydrated ferric sulfates ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$) using differential scanning calorimetry (DSC, METTLER TOLEDO STA 449C). As shown in Fig. 1a, the transition temperature is 700.7°C. A single batch of AR grade hydrated ferric sulfates ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$) whitish powder was heated by Muffle furnace at 700.7°C for 24 hours. The product changed into reddish brown in color.

Pathway 2: AR grade powder of $\text{Fe}(\text{OH})_3$ was used to be heated by an oven at 180 °C [8] for 2 hours. Then the heated powder was taken out the oven and cooled to room temperature. The product changed into red in color.

XRD Characterization: The phase information of the products from pathway 1 and 2 were characterized by X-ray powder diffraction (XRD). A Rigaku UltimaIV diffractometer with a $\text{CuK}\alpha$ radiation ($\lambda = 1.54052 \text{ \AA}$) was employed at an acceleration voltage of 40 kV and current of 40 mA and scanned at 0.02° step from 10° to 90°. The XRD pattern of those products match with the hematite (refer to the Powder Diffraction File card no. 89-0597). And the ferric oxide from heated hydrated $\text{Fe}_2(\text{SO}_4)_3$ is no additional peaks appeared, suggested that is a pure phase of hematite.

Raman spectroscopic measurements: Raman spectroscopy acquires information on vibrational and rotated transitions depended on the symmetry of a molecule or a crystal. Renishaw inVia® Raman System (Renishaw Company) was used for the Raman measurements. Two synthesized ferric oxides Raman spectra ranging from 160 to 1400 cm^{-1} (Fig. 1c) were collected. The Raman spectra patterns of the two products are similar. The major difference is that some peaks have evident shifts in peak positions (from 213.7 to 220.4 cm^{-1} , 272.4 to 284.4 cm^{-1} and 388.4 to 400.2 cm^{-1}).

Conclusions and future work

We have performed laboratory synthesis and Raman measurements of hematite from different starting materials. For future work, we will continue the laboratory work on the pathways of hematite production. In addition to Raman measurements, we will also conduct other spectroscopic measurements (e.g., visible and near-infrared spectroscopy, mid-infrared spectroscopy), which would help for their identifications using orbital remote sensing and in-situ measurements.

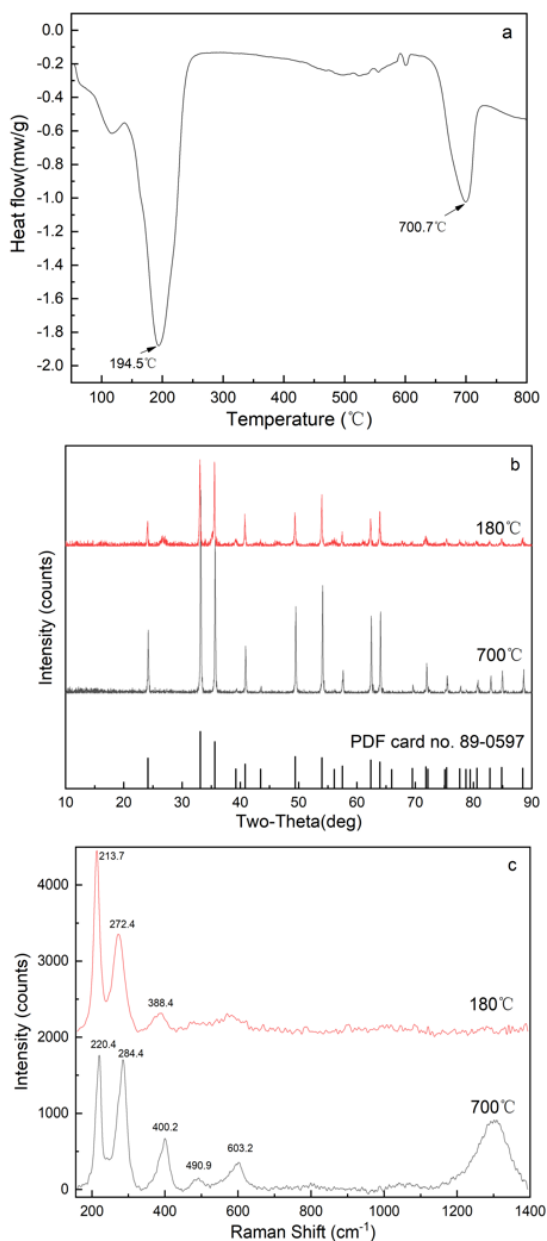


Fig. 1. a. DSC curves of the hydrated Fe-sulfates ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$). b. XRD pattern of the Fe_2O_3 powder. The black line is Fe_2O_3 powder from heated hydrated Fe-sulfates; The red line is Fe_2O_3 powder from heated $\text{Fe}(\text{OH})_3$. c. Raman spectra of the Fe_2O_3 powder. The black line is Fe_2O_3 powder from heated hydrated Fe-sulfates; The red line is Fe_2O_3 powder from heated $\text{Fe}(\text{OH})_3$.

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