HARRICK SCIENTIFIC PRODUCTS

No. 21142

AN INVESTIGATION OF DRYING CLAY USING INFRARED DIFFUSE REFLECTANCE

Figure 2. The Harrick Praying Mantis™ diffuse reflectance accessory.

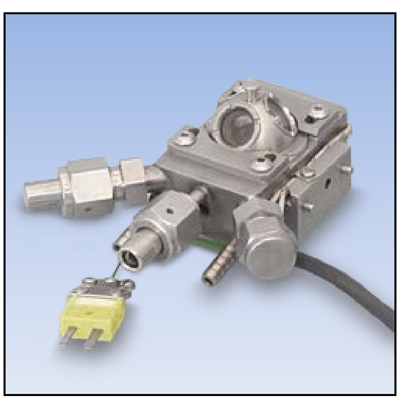

Figure 1. The Harrick High Temperature Reaction Chamber.

INTRODUCTION

Diffuse reflectance is a powerful spectroscopic technique for the analysis of powdered solids. Analysis can be carried out at elevated temperatures, allowing for reactions and physical transformations to be observed via infrared spectroscopy. A straightforward demonstration of this method is observing solids that can absorb significant amounts of water dry. In the process of drying, the water bands should decrease in intensity from the infrared spectrum.

In this application note, the drying of wet clay (Kaolin) is observed through infrared diffuse reflectance spectroscopy.

EXPERIMENTAL

The infrared measurements were carried out using the Harrick Praying Mantis™ diffuse reflectance accessory (Figure 1) with its High Temperature Reaction Chamber (Figure 2) and its Temperature Controller to heat the sample and facilitate drying. All measurements were taken with the Reaction Chamber fully open and the Temperature Controller was auto-tuned to 500°C before use.

The Praying Mantis was installed in the sample compartment of an FT-IR spectrometer. Spectra were the result of 32 averaged scans at 8 cm^{-1} resolution collected with a DTGS detector. The gain was set to 8 and the aperture was set to 100 (fully open). The spectrometer and accessory were purged. A background spectrum was collected using ground, powdered potassium bromide in the sample cup at room temperature.

A slurry of wet clay was generated by thoroughly mixing 5.8 g of Kaolin (Ward's Science) with 5 mL H_2O in a beaker. The sample cup of the High Temperature Reaction Chamber was filled with the slurry. Then sample spectra were recorded at regular intervals, first at room temperature and then from 100°C to 500°C at 100°C intervals.

RESULTS AND DISCUSSION

Figure 3 shows the changes in the spectrum of clay at various temperature, while Figure 4 shows the spectral differences in clay heated to 500°C over time. Both spectra are displayed in Kubelka-Munk units solely for convenience. The Kubelka-Munk transform is commonly used because it is analogous to the absorbance transform used for transmission spectra. It should be noted, however, that the Kubelka-

applications note

Munk transform is not strictly applicable here because the sample is too strongly absorbing.

Figure 3 focuses on the spectral range from 4000 to 2500 cm^{-1}. The reduction of the large, broad water band is clear as the temperature is increased and water is evaporated from the sample.

The loss of water on heating is also observed in the narrowing of the clay band centered around 1260 cm^{-1}, as shown in Figure 4. As the sample temperature is kept at 500°C, for longer periods of time, the band narrows and shifts. This can be attributed to a decrease in the intensity of the strong water band that occurs at 1000 cm^{-1}.

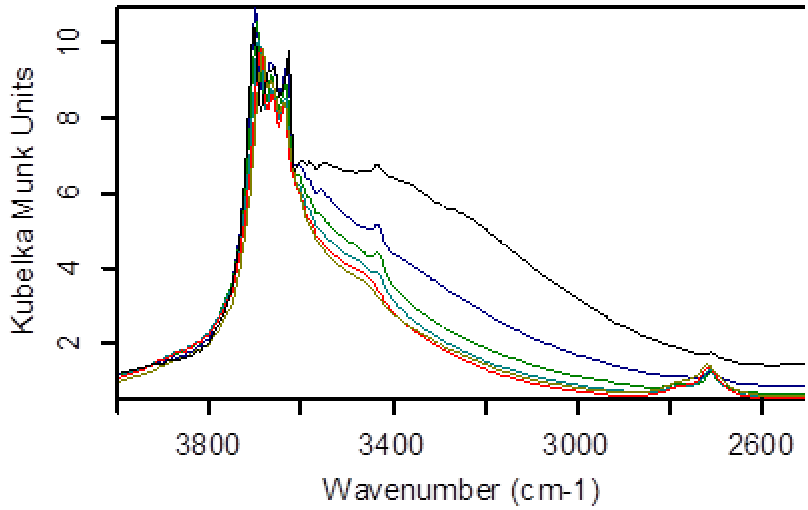

Figure 3. Spectra of clay at various temperatures: Ambient temperature, 0 min (black); 100°C, 20 min (blue); 200°C, 40 min (green); 300°C, 60 min (teal); 400°C, 80 min (red) and 500°C, 100 min (yellow).

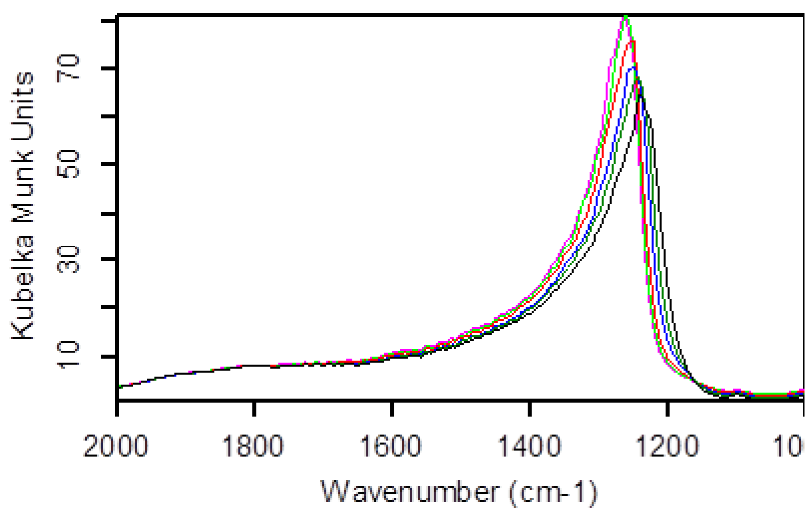

Figure 4: Spectra of clay at 500°C showing the decreased influence of an underlying water band as a function of time: 100 min (black), 110 min (green), 120 min (blue), 140 min (red), 150 min (light green) and 160 min (pink).

CONCLUSIONS

Diffuse reflectance at room temperature or high temperature is a convenient method for infrared spectroscopic study of many powdered solid samples. To demonstrate a simple application of this, the drying of clay at high temperatures over time was observed using this technique with the Harrick Praying Mantis™ and its High Temperature Reaction Chamber.

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