

THERMAL ANALYSIS OF HECTORITE. PART II. DIFFERENTIAL THERMAL ANALYSIS

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ABSTRACT

The thermal behavior of the SHCa-1 hectorite source clay specimen was studied by the technique of computerized differential thermal analysis (DTA) using dynamic nitrogen, dynamic air, dynamic carbon dioxide, and static air atmospheres. The atmospheric dependence of the DTA thermal curves was established. A series of samples heated in dynamic nitrogen purge was analyzed by X-ray diffraction spectrographic techniques to establish the high temperature phases of the SHCa-1 (carbonate-contaminated) hectorite specimen. Differential thermal analysis was also performed on a processed (carbonate-cleaned) hectorite specimen using dynamic nitrogen atmosphere. Additional TG–DTG thermal curves were included in this portion of the study.

INTRODUCTION

Hectorite is a trioctahedral member of the smectite clay minerals group. As was pointed out in the previous study [1], hectorite contains little to no Al^{3+} in the silicate structure. Instead, these octahedral positions have been substituted by Mg^{2+} and Li^+ ions. The charge balancing cation located external to the silicate layer is Na^+ . Thus, the silicate structure may be considered as “magnesium-rich”. Although not given in the empirical formula of Ross and Hendricks [2], hectorite also contains fluorine atoms. A recent analysis [3] of the SHCa-1 source clays specimen found 2.75% fluorine in this raw hectorite ore.

Hectorite ore specimens in the United States are limited to those found in southeastern California (near Hector, CA) and are always found associated with large amounts of calcite and, in some cases, varying amounts of dolomite. Thus, most published thermal curves for hectorite reflect not only the thermal behavior of the smectite clay mineral but also that of the carbonate contaminant.

The thermal behavior of most smectite clay minerals has been established by Grim and co-workers [4–7] using both differential thermal analysis and X-ray diffraction techniques. Much of the DTA and TG behavior of the

dioctahedral montmorillonite members was recently repeated and reviewed by Earnest [8]. Generally speaking, in DTA one expects to observe thermal events arising from the loss of interlayer water, dehydroxylation of the silicate structure, matrix collapse of the anhydride silicate structure, and an ordering exotherm as smectite clay mineral species are heated from ambient to ca. 1020°C. However, Bradley and Grim [7] have found that the trioctahedral members of this group do not exhibit an anhydride structure after dehydroxylation as do the dioctahedral members. Thus, a simultaneous matrix collapse is believed to occur during the dehydroxylation of hectorite and a separate endothermic even is not observed in the DTA thermal curve.

The trioctahedral smectites, saponite and hectorite are considered to dehydroxylate at higher temperatures than those exhibited by the dioctahedral members. Due to the carbonate contamination of natural hectorite specimens, a problem exists in distinguishing the dehydroxylation event from the decomposition of the carbonate component. Thus, both the degree and rate of dehydroxylation of hectorite are somewhat uncertain and some previous statements regarding these may be misleading.

In the first part of this study [1], thermogravimetry in dynamic nitrogen atmosphere, acid evolved CO₂, and plasma emission spectroscopy were

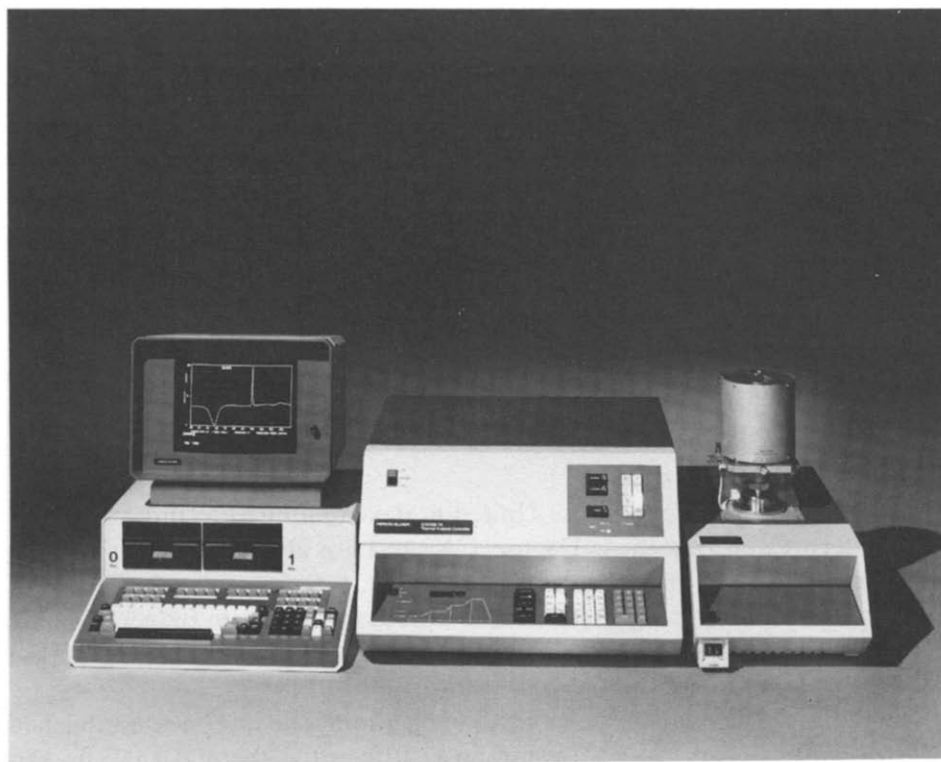


Fig. 1. Perkin-Elmer DTA-1700/TADS computerized differential thermal analysis system.

employed in the analysis of several hectorite specimens. These included both raw and carbonate-cleaned specimens. The total calcium carbonate level of the SHCa-1 hectorite source clay specimen was determined to be 40.0% based on the concentration of the Ca^{2+} ion in the dilute acid soluble portion of the raw clay specimen. On the other hand, the total carbonate anion (CO_3^{2-}) content in this specimen was determined to be 25.15% using acid evolved CO_2 determination of inorganic carbon. By combining the TG weight loss data and acid evolved CO_2 data, it was concluded that the major decomposition observed in the ca. 550–850°C temperature region by hectorite ores is primarily due to the decomposition of carbonate component(s). Using the results obtained for two hectorite specimens which had been industrially processed to remove most of the carbonate component, the amount of weight loss (based on sample weight at 540°C) not associated with carbonate decomposition was determined to be 2.00%.

In the following study, the SHCa-1 hectorite source clay specimen is further characterized using computerized differential thermal analysis (DTA) in a variety of atmospheres. Additional supporting TG thermal curves are also included. The results of the X-ray diffraction studies performed on samples of the SHCa-1 hectorite which were previously heated in dynamic nitrogen purge will also be reported. The DTA, TG, and DTG thermal curves obtained for a processed (carbonate-cleaned) hectorite ore are also given.

EXPERIMENTAL

All DTA thermal curves presented in this paper were obtained with the Perkin-Elmer microcomputer-based DTA-1700 differential thermal analysis system. All DTA thermal curves for the SHCa-1 hectorite source clay specimen were obtained using the DTA-1700 system with the Perkin-Elmer thermal analysis data station (TADS) and TADS printer/plotter. The computerized DTA-1700/TADS system is shown in Fig. 1. All DTA experiments for the SHCa-1 hectorite specimen were performed using the "heat flux" DSC mode of operation of the DTA-1700 system. When the instrument is used in this mode of operation, the amplified ΔT signal from two Pt/Pt-10% Rh thermocouples undergoes additional signal conditioning through the use of a programmable ROM chip. The net effect of this mode of signal conditioning is a calorimetric factoring of the ordinate signal versus temperature.

The conditioned ordinate data, along with the actual sample thermocouple temperatures, are passed through an RS-232 port to the thermal analysis data station. All ordinate scaling and data handling routines are then performed by the DTA standard software program. Both potassium chromate and gold standards were used for calibrating the temperature axis and

for establishing the ordinate calibration factor.

The DTA thermal curve for the processed (carbonate-cleaned) hectorite ore was obtained using the DTA mode of the DTA-1700 system used in conjunction with a Perkin-Elmer XY_1Y_2 recorder. When used in this mode of operation, the amplified ΔT signal from the Pt/Pt-10% Rh thermocouples undergoes additional computer firmware conditioning so that a linearized ΔT response is achieved over the entire temperature range of the instrument. When using the XY_1Y_2 recorder, the linearized sample thermocouple response is used to drive the X-axis of the recorder.

All TG thermal curves included in this portion of the study were obtained using a Perkin-Elmer TGS-2 thermogravimetric system. The TG and DTG thermal curves for the SHCa-1 hectorite specimen were obtained using the TADS as the data handling device while those for the processed hectorite ore were obtained using a Perkin-Elmer XY_1Y_2 thermal analysis recorder. The source and nature of the sample specimens were given in the earlier TG study [1]. The X-ray diffraction data were obtained using a Phillips XRD instrument external to this laboratory. The samples were randomized on a silver membrane in obtaining the XRD patterns. In all cases, when performing differential thermal analysis, the sample specimens were placed in 60 mm³ ceramic liners and a thin layer of aluminum oxide, which had previously been calcined at 1400°C, was placed over the sample to minimize radiation effects at elevated temperatures.

RESULTS AND DISCUSSION

Thermal behavior of the SHCa-1 source clay hectorite in dynamic nitrogen atmosphere

Figure 2 shows the DTA thermal curve obtained for a 56.67 mg sample of the SHCa-1 hectorite source clay specimen obtained using a heating rate of 10°C min⁻¹ and a dynamic purge of nitrogen of 50 cm³ min⁻¹. The thermal curve represents the hard copy printout from the TADS printer/plotter using the DTA-1700 in the heat flux DSC mode of operation. As is shown by the thermal curve, an endothermic peak at (T_{\min}) 121°C is observed for the interlayer water loss and a two-stage endothermic event ($T_{\min} = 752$ and 830°C) is observed in the dehydroxylation/carbonate decomposition region of temperature. Since these data were obtained in the DSC mode of operation of the instrument, the endothermic energy associated with these thermal events was assigned through the use of the DTA standard software program. Figure 3 shows the peak analysis for the dehydroxylation/carbonate decomposition as assigned by the TADS. One will observe that the extrapolated onset temperature (765.3°C) is assigned using the steepest slope of the leading edge of the DTA peak. The peak minimum (829.3°C) and the

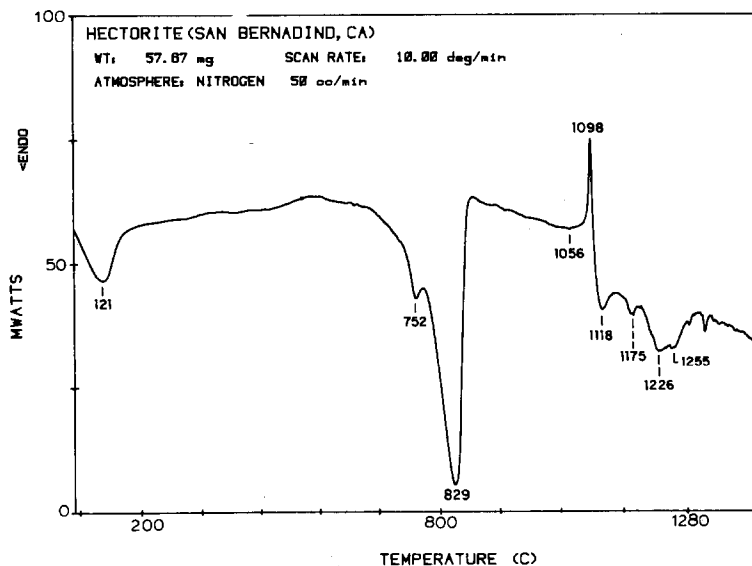


Fig. 2. DTA-1700/TADS thermal curve for the SHCa-1 hectorite specimen in dynamic nitrogen atmosphere.

endothermic decompositional energy (524 J g^{-1}) are also assigned as a part of the peak analysis. When the lower temperature endothermic peak corresponding to the loss of interlayer water is treated similarly, the endothermic energy was found to be 142 J g^{-1} .

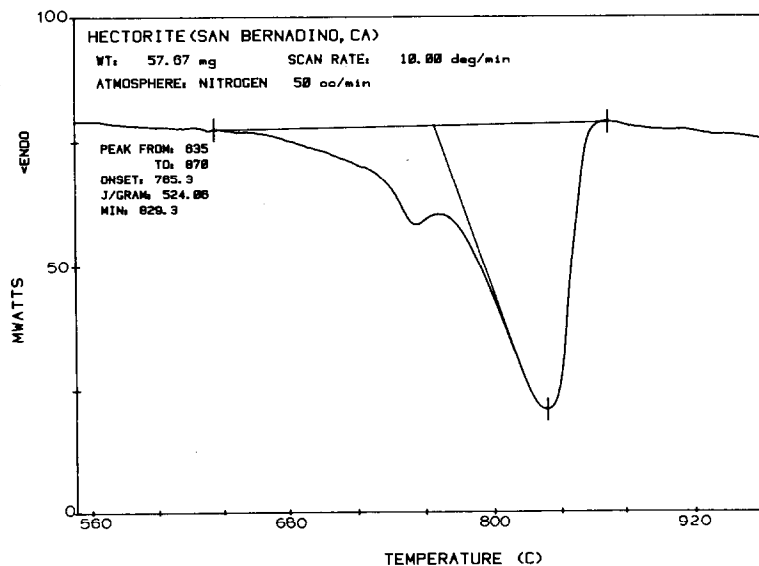


Fig. 3. TADS "peak analysis" of the carbonate/dehydroxylation decomposition region of the SHCa-1 hectorite specimen in dynamic nitrogen atmosphere.

A sharp exothermic event ($T_{\max} = 1098^{\circ}\text{C}$) followed by an immediate endothermic event and a marked endothermic shift in the baseline are also observed in the DTA thermal curve. This sharp exo-endo event was found to be very reproducible. However, as the sample size was increased to 80 mg, the exothermic portion of this event became less pronounced and a rounding effect was noted. On increasing the sample size to 100 mg, the exothermic portion of this event was completely eliminated. This suggests an atmospheric dependence due to the gaseous decomposition products arising from the preceding dehydroxylation/carbonate decomposition event. Also, this may explain why this exothermic event has not been reported by most previous workers using either much larger samples or static air environment. Prior to this work, only Smykatz-Kloss [9] reports an ordering exotherm in the DTA thermal curve of hectorite. More will be said about this matter later.

Following the exo-endo event, additional endothermic activity is observed in the DTA thermal curve. Bradley and Grim [7], MacKenzie and Caillere [10], and Schmidt and Heyster [11] have previously reported endothermic events in this temperature region. The sum of the endothermic areas observed between 1150 and 1300°C in Fig. 2 represents about 89 J g^{-1} of the SHCa-1 hectorite source clay specimen as assigned by the DTA-1700/TADS system. Bradley and Grim [7] suggest that the major endothermic activity observed at these temperatures in their study was due to the formation of clinoenstatite.

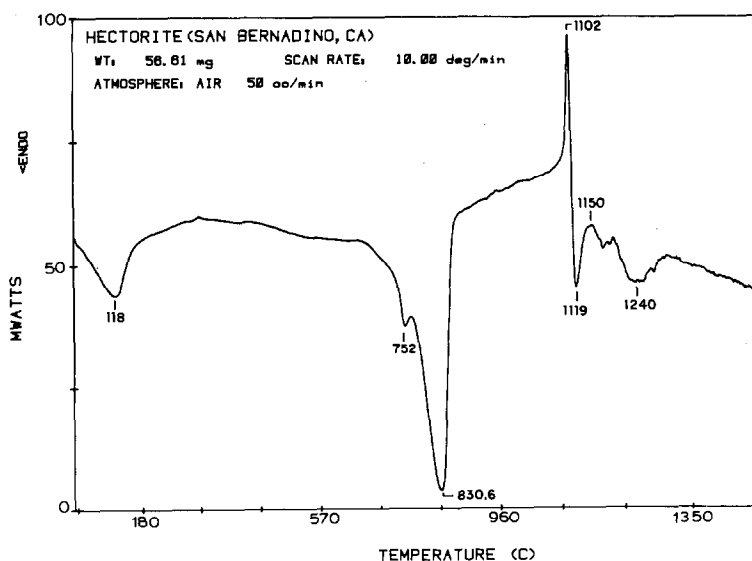


Fig. 4. DTA-1700/TADS thermal curve for the SHCa-1 hectorite specimen in dynamic air atmosphere.

In an attempt to further assign the high temperature phases associated with these thermal events observed at elevated temperatures, numerous samples were heated in dynamic nitrogen purge in the DTA-1700 and stopped at various points while observing the DTA thermal curve on a Perkin-Elmer XY₁Y₂ recorder. The XRD patterns showed that diopside [Ca, Mg(Si₂O₆)], forsterite (Mg₂SiO₄), and clinoenstatite (Mg₂Si₂O₆) began to form as early as 1020°C. These species all increased in amount as the temperature was raised to 1115°C. The XRD patterns showed that these three were the major crystalline forms up to 1154°C. The difference between small sample specimens showing the sharp exotherm and large specimens which did not exhibit the exotherm in the DTA thermal curve, was determined using samples which were heated to 1115°C in the DTA-1700. Those specimens exhibiting the exothermic event showed much stronger XRD patterns for all three crystalline forms than those not exhibiting the event. Hence, the difference is simply one of quantity (or crystallization rate) of the three phases.

DTA studies of the SHCa-1 hectorite specimen in both dynamic air and static air atmospheres

Figure 4 shows the DTA thermal curve for the SHCa-1 hectorite specimen obtained in dynamic air purge of 50 cm³ min⁻¹ using a heating rate of 10°C min⁻¹. One will observe this thermal curve to be very similar to that obtained in dynamic nitrogen atmosphere. As can be seen in Fig. 4, the peak minimum for the interlayer water loss is at 118°C, which is 3 degrees lower than that obtained in dynamic nitrogen purge. This small difference is due to a 1.06 mg difference in sample size. The shoulder on the major dehydroxylation/carbonate peak is at the same temperature as that obtained in dynamic nitrogen, while the strongest endothermic peak minima is 1.3°C higher than that observed in dynamic nitrogen. The major difference between the two DTA thermal curves is that the exo/endo peak near 1100°C is enhanced in dynamic air purge. By comparing peak heights of the exothermic portion, an increase of 61% is observed in dynamic air purge. Since both the exothermic and endothermic portion of the event is observed to be enhanced in dynamic air atmosphere, the total amplitude from the endothermic peak minimum to the exothermic peak maximum was determined for both DTA thermal curves. The ratio of this amplitude in dynamic air to that obtained in dynamic nitrogen was found to be 1.46.

Due to the effect of sample size, which was observed in dynamic nitrogen purge, this specimen was also studied in static air (self-generating) atmosphere. Figure 5 shows the resulting DTA thermal curve. As was expected, the endothermic peak corresponding to loss of interlayer water is shifted to a higher temperature ($T_{\min} = 131^\circ\text{C}$) due to the effect of the $P_{\text{H}_2\text{O}}$ in the self-generating atmosphere. A minor exothermic peak is observed at 305°C.

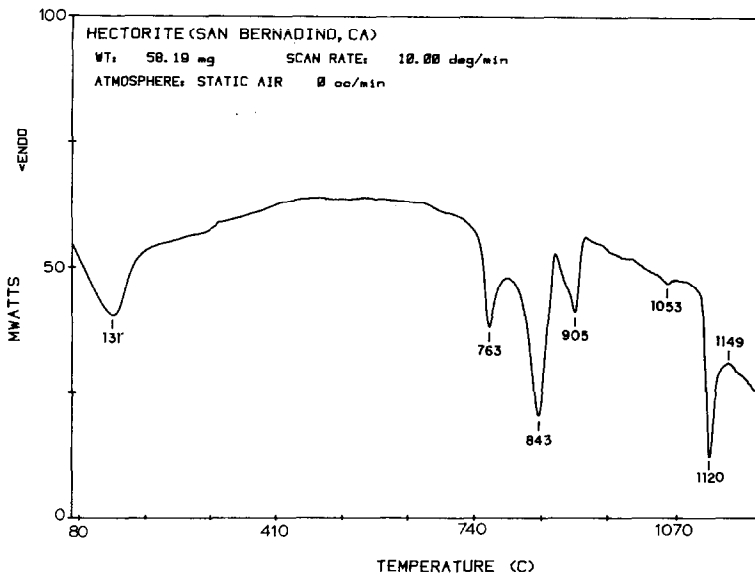


Fig. 5. DTA-1700/TADS thermal curve for the SHCa-1 hectorite specimen in static air atmosphere.

The dehydroxylation/carbonate decomposition is now observed to be split into at least three separate events. The first peak minimum (763°C) in the series is observed eleven degrees above that observed in both dynamic nitrogen and dynamic air atmospheres. This multi-step decomposition is in accord with that observed by Kerr et al. [12] with a raw hectorite ore in which it was suggested that the presence of dolomite was responsible for the multiple decomposition peaks. By observing the thermal curve in Fig. 5 and recalling that, in the previous study [1], this specimen was found to contain 40.0% total calcium carbonate, one might now suspect that there may be something more complex going on in this temperature region.

The baseline of the DTA thermal curve showed an endothermic tendency in this region and this effect was more evident after the dehydroxylation/carbonate decomposition. As was predicted from the observations made with large samples in dynamic nitrogen atmosphere, the exothermic portion of the exo/endo peak is now rounded and nearly diminished. The small peak at 1053°C in Fig. 5 is not an endothermic effect but rather represents the initiation of the exothermic activity. On comparing Figs. 2, 4 and 5, one will observe that the endothermic portion of the exo/endo peak at 1120°C is strengthened by the use of a self-generating atmosphere. This portion of the DTA thermal curve is more nearly that which has been reported by some previous workers [10,11]. It is the belief of the author that the absence of the ordering exotherm in all but one of the previously published thermal curves is due to the effect of the atmosphere generated

from the use of large samples and/or the use of static air environment in the DTA cell chamber.

Studies of the SHCa-1 hectorite specimen in dynamic CO₂ atmosphere

When differential thermal analysis of the SHCa-1 hectorite specimen is performed in dynamic CO₂ purge ($P_{\text{CO}_2} = 1 \text{ atm}$), the peaks in the dehydroxylation/carbonate decomposition region were shifted to higher temperatures. As will be seen in Fig. 6, these peaks are both sharper and more intense than those observed in the self-generating atmosphere. This shifting and intensification is normal for most carbonate decompositions in dynamic CO₂ atmosphere. Figure 7 shows the dehydroxylation/carbonate decomposition region rescaled by the TADS. One will note that, due to the shifting of the peaks, an additional weak endothermic event is now observed having a peak minimum at 743°C. Thus, four distinguishable events are observed in this temperature region of the DTA thermal curve obtained in dynamic CO₂ atmosphere.

The major portion of the carbonate fraction contained in this hectorite specimen has always been considered to be calcite. This is supported by spectroscopic studies [13] as well as XRD data obtained in this study. Also, it was found in the first part of this work [1] that the total calcium carbonate content of this specimen was 40.0%, which could possibly be as calcite and/or dolomite. Regardless of which mineral species the Ca²⁺ is found to be, the CaCO₃ decomposition should occur above 900°C when performed in

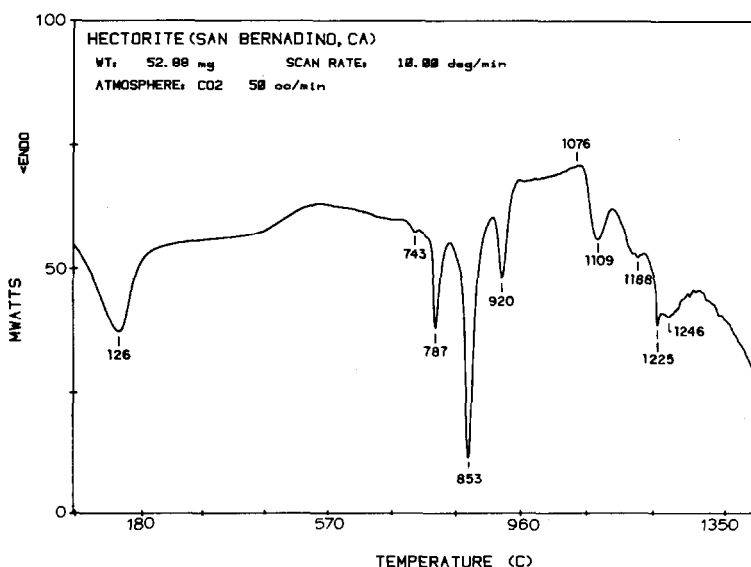


Fig. 6. DTA-1700/TADS thermal curve for the SHCa-1 hectorite specimen in dynamic CO₂ atmosphere.

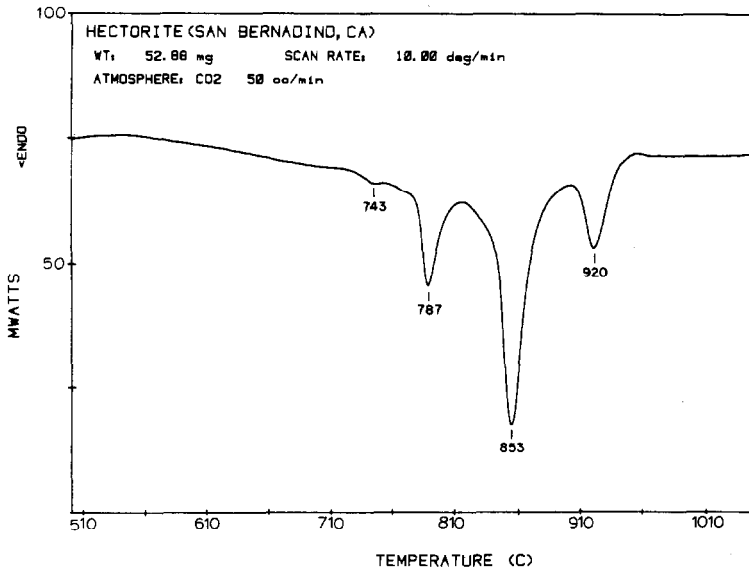


Fig. 7. TADS rescaled thermal curve for the SHCa-1 hectorite specimen for the 500–1060°C temperature region in dynamic CO₂ purge.

dynamic CO₂ atmosphere. Thus, the peak observed at 920°C in Figs. 6 and 7 should correspond to the decomposition of CaCO₃(s). However, this DTA peak is not strong enough to represent the total CaCO₃(s) content of this hectorite specimen.

To gain more information about this multi-event decomposition, the specimen was also studied by thermogravimetry in dynamic CO₂ atmosphere. Figure 8 shows both the TG and DTG thermal curves which were obtained. These thermal curves ascertain that the major DTA peaks in the ca. 650–950°C temperature region are associated with weight loss events. Furthermore, since the DTG and DTA thermal curves agree in both number and relative intensity, the possibility of a thermal gradient effect is eliminated as a cause for the multi-peak nature of the decomposition. The sample weight used in obtaining the TG–DTG thermal curves is only 18.33 mg and the heating rate was only 5.0°C min⁻¹.

When the high temperature regions of the DTA thermal curves for the SHCa-1 specimen obtained in the various atmospheres are compared, it is observed that the temperature of the endothermic events are lowered in dynamic CO₂ purge. Figure 9 shows the simultaneous TADS hard copy of the 1000–1200°C ordering region obtained in both static air and dynamic CO₂ purge. The endothermic peak minima observed at 1118, 1119 and 1120°C in dynamic nitrogen, dynamic air, and static air, respectively, is lowered to 1109°C in dynamic CO₂ atmosphere. The endothermic event is also notably broadened, which corresponds to a decrease in rate. This is easily seen by both the peak amplitude and slope of the leading edge of the

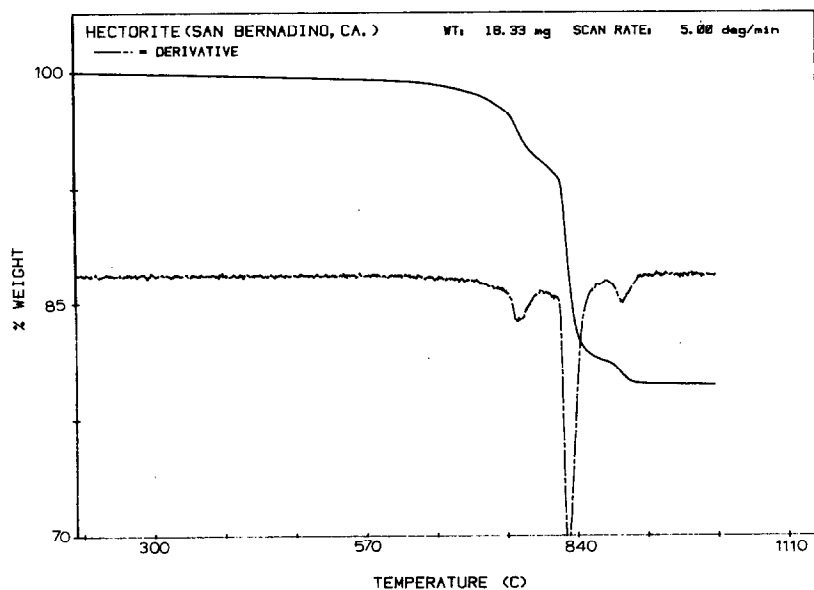


Fig. 8. TG-DTG thermal curve for a dried SHCa-1 hectorite specimen in dynamic CO_2 atmosphere.

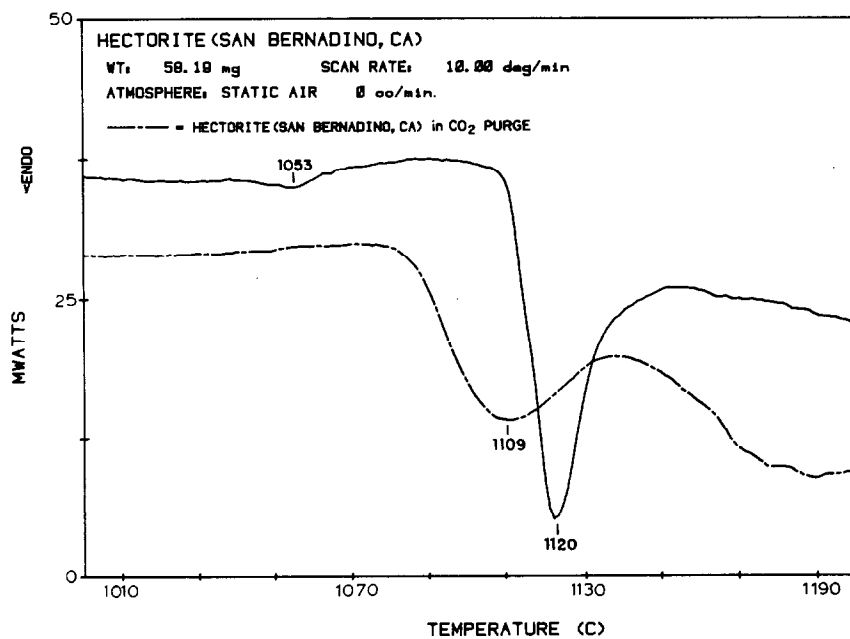


Fig. 9. Comparison of the 1000-1200°C regions of the SHCa-1 hectorite specimen in static air and dynamic CO_2 atmosphere by the TADS.

endothermic even in Fig. 9. Thus, it has been observed in the DTA studies that increasing the partial pressure of CO_2 to 1 atm not only eliminates the exothermic ordering peak but also affects the rate of endothermic ordering of this carbonate-contaminated hectorite specimen.

Thermal analysis of a processed (carbonate cleaned) hectorite specimen

In the first part of this study [1], the TG thermal curve obtained in dynamic nitrogen purge was given for a hectorite specimen containing only 2.30% CO_3^{2-} , which was the cleanest of the four specimens of the study. Figure 10 shows the DTA thermal curve for this same processed hectorite ore specimen obtained in dynamic nitrogen purge using the DTA mode of the DTA-1700 system. One will note that the major endothermic peak for this specimen is that associated with the loss of interlayer water. This endothermic peak is observed to be shifted to a higher temperature than that observed in the SHCa-1 hectorite specimen which contained 25.15% CO_3^{2-} . This is in accord with the observed increase in percentage interlayer water with decreasing carbonate content which was shown in the earlier study [1]. The major DTG peak which was observed in the SHCa-1 hectorite specimen in the dehydroxylation/carbonate decomposition region is only weakly observed in this DTA thermal curve for the processed hectorite ore. This supports the conclusion of the thermogravimetric study [1] which suggests that the major decomposition event in this temperature region is almost entirely due to carbonate decomposition. This is also in agreement with the statement of Grim [4] that "in general, trioctahedral smectites show a gradual loss of hydroxyls and a broad, slight endothermic peak". One will

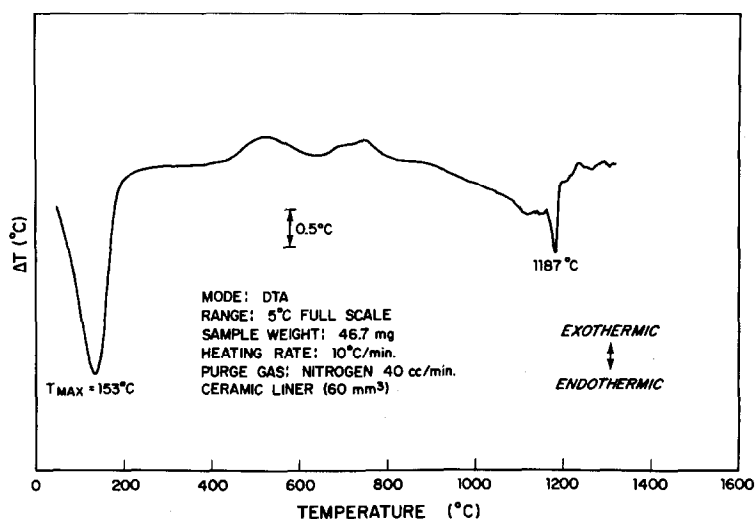


Fig. 10. DTA thermal curve for processed hectorite ore in dynamic nitrogen atmosphere.

also note the absence of the sharp exothermic event which is observed in the carbonate-contaminated specimens. This suggests that the exothermic ordering event is related to the quantity of alkaline earth oxides generated from the decomposition of the carbonate contaminant. It can be seen in Fig. 10 that the DTA thermal curve for the processed hectorite ore shows an endothermic effect beginning near 900°C and continuing to a stronger endothermic ordering region ($\approx 1100^\circ\text{C}$). This DTA thermal curve is very similar to that published by Bradley and Grim [7]. Although not as clean as the specimen of this study, it is obvious that much of the carbonate component had been removed from their specimen.

Although the TG thermal curve for this carbonate cleaned specimen was given in Part I of this study [1], an additional thermogravimetric curve is presented in Fig. 11. In this case, a sample of the processed hectorite ore specimen was held at 200°C until most of the interlayer water was lost and eliminated from the TG furnace tube. The simultaneous TG and DTG thermal curves were then obtained using dynamic nitrogen purge and an ordinate scale weight suppression of 90%. Thus, the TG thermal curves in Fig. 11 represent 10% of the dry sample weight full scale as observed on a Perkin-Elmer XY₁Y₂ recorder.

Since the TG weight loss is continuous and the thermal events do not occur in rapid fashion, the high sensitivity DTG trace is essential in

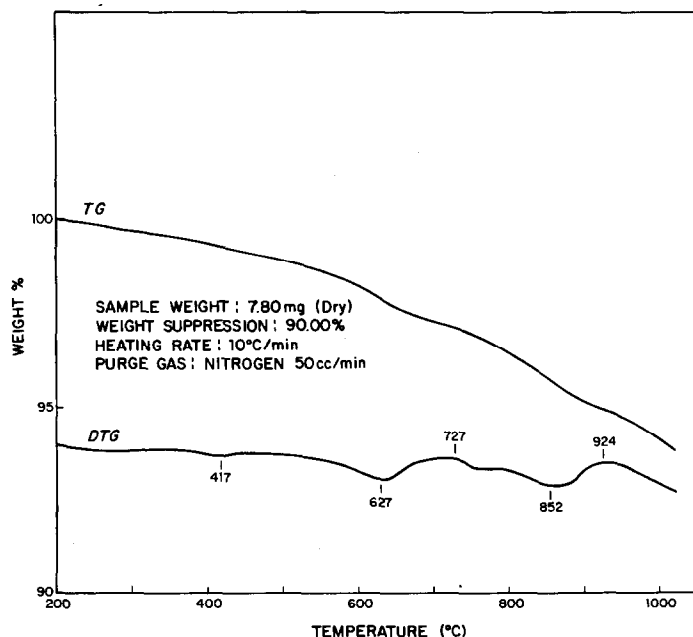


Fig. 11. TG-DTG thermal curves for processed hectorite ore in dynamic nitrogen atmosphere.

following the thermal behavior. One will note a minor weight loss event near 417°C which is associated with only 0.3% weight loss. The DTG thermal curve for the weight loss activity between ca. 520 and 919°C appears very similar to that observed for the Veegum T specimen in Part I of this study [1]. One will recall that the Veegum T specimen released 1.98% more CO₂(g) on acidification than did the processed hectorite ore. By comparing the TG and DTG thermal curves for both specimens, it is not possible to assign this difference to one single event occurring in the 520–919°C temperature range; i.e. all of the distinguishable DTG thermal events in this temperature region are lessened in the processed hectorite ore specimen. Finally, and the main reason for including these TG–DTG thermal curves, one will observe that weight loss continues at temperatures above 931°C in the TG curve. This corresponds to the endothermic trend of the DTA thermal curve (Fig. 10) for this temperature region.

CONCLUSION

The thermal behavior, as described by differential thermal analysis, of raw (carbonate-contaminated) hectorite specimens is notably different from those specimens in which most of the carbonate component has been removed. The strong endothermic events associated with the dehydroxylation/carbonate decomposition is greatly diminished by removal of most of the carbonate component. The CO₂(g) generated from the decomposition of large carbonate component(s) in raw hectorite ores can affect the appearance of the observed DTA thermal curves obtained for these specimens.

Gaseous decomposition products are not immediately expelled from a closed DTA cell chamber when using dynamic purge atmospheres. They are eliminated, however, from such dynamic purge atmospheres in an exponential fashion which is dependent upon both the purge rate and cell volume. For this reason, atmospheric effects normally observed in static air (self-generating) atmospheres, may also be observed to a lesser extent in dynamic nitrogen (or other inert gases) and dynamic air atmospheres if a large enough sample is used. The P_{CO_2} generated in the sample atmosphere has an effect on the nature and rate of high temperature ordering in raw hectorite specimens as well as upon the nature of the carbonate decomposition as observed by DTA. Therefore, the DTA thermal curve may be influenced by sample size, volume of DTA furnace tube (cell chamber), flow rate of purge gas, and heating rate. It is, therefore, conceivable that different instruments having significantly different cell volumes may produce DTA thermal curves for carbonate-contaminated hectorite specimens which differ to some extent when operating in dynamic nitrogen, dynamic air, or static air atmospheres. However, the P_{CO_2} effect is least likely to be recognized in the ordering region of raw hectorite ores when using dynamic air purge due to the rate

enhancement which is observed in this atmosphere.

The P_{CO_2} dependence of the DTA thermal curve is more pronounced in static air (self-generating) atmosphere and dynamic CO_2 ($P_{\text{CO}_2} = \text{atm}$) purge. Not only is the high temperature ordering exotherm eliminated but the dehydroxylation/carbonate decomposition region of the thermal curve exhibits inordinate features in atmospheres containing relatively high concentrations of $\text{CO}_2(\text{g})$. Based on the Ca^{2+} determination in the acid-soluble portion of the SHCa-1 hectorite specimen, as well as the published [3] CaO determination for this specimen, and the acid-evolved CO_2 determinations of this study, the carbonate component of the SHCa-1 hectorite specimen is believed to be primarily calcite. The DTA, TG, and DTG thermal curves obtained in dynamic CO_2 purge suggest that the calcite undergoes some type of endothermic reaction [liberating $\text{CO}_2(\text{g})$] prior to the normal decomposition temperature as determined by the P_{CO_2} when operating in either static air or dynamic CO_2 atmosphere. The fine particle size in which the calcite exists in the SHCa-1 hectorite specimen may contribute to this effect. It should also be mentioned that the author was unable to regenerate the CaCO_3 by cooling specimens in $\text{CO}_2(\text{g})$ which were heated to temperatures (ca. 1020°C) sufficient to decompose the carbonate component. This indicates that the $\text{CaO}(\text{s})$ is not free for recombination after heating to these temperatures.

Also, as a result of this study and the previous thermogravimetric studies [1], it is also the belief of the author that the rate of dehydroxylation of hectorite clay mineral is not sufficient to be observed as more than a weak, but broad, endothermic event in DTA thermal curves. This statement may be contrary to some who tend to think that both hectorite and saponite show rapid dehydroxylation events which would suggest strong DTA endotherms. In the case of hectorite, this study has shown that this belief is a direct result of the carbonate contamination. As of this writing, no studies have been conducted on saponite specimens in this laboratory.

Finally, on comparison of the DTA thermal curves for both the SHCa-1 hectorite specimen and processed (carbonate-cleaned) hectorite specimen, it is observed that the amount of alkaline earth oxide formed from the decomposition of the carbonate component also has an effect on the high temperature ordering behavior of hectorite specimens. Also, the removal of most of the carbonate component from hectorite specimen leads to an increase in the percentage of interlayer water. This was observed directly in the TG thermal curves given in the first part [1] of this study. This increase in percentage interlayer water intensifies the corresponding DTA peaks and shifts the peak temperatures to higher values.

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