In Situ SAXS Studies of the Temperature Structural Dependence of Sepiolite

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Introduction

Sepiolite, a clay whose structure consists of channels, has been recently used as an inorganic template to produce novel carbons useful as anodes in lithium rechargeable batteries. In order to produce the carbons, propylene or ethylene is loaded and heated up to 700°C. The inorganic matrix is then dissolved, and the carbons' properties (surface area, pore size) depend on those of the inorganic template. Upon heating the clay, it can undergo irreversible changes that ultimately affect the structure of the carbon. Studying the structural dependence of sepiolite upon heating is crucial in predicting and understanding the carbon properties and thus tailoring good candidates for rechargeable sources. *In situ* SAXS is the only technique from which this information can be derived.

Methods and Materials

Sepiolite powder was inserted into a 1-mm-diameter quartz capillary tube. At the bottom of the tube, a piece of quartz wool was placed to allow for the gas flow. The capillary tube was then inserted into a specially designed furnace that has an opening for the gas connection. The furnace temperature program was set up to ramp from room temperature to 700°C at 5°C/min, and the gas flow of nitrogen or propylene was started. SAXS data were collected every minute.

Results and Discussion

Figure 1 shows the high Q SAXS data obtained as a function of temperature upon heating a sepiolite sample under a flow of propylene gas. At 25°C, the d-spacing of the predominant peak is 12.13 Å, and there is another reflection which space corresponds to 7.56 Å. As the temperature increases, the intensity of the main peak decreases and two other reflections appeared. At 378°C, the main peak d-spacing decreases to 10.44 Å, and another peak with a d-spacing corresponding to 8.27 Å appears. Beyond 378°C, both the intensity and the position of the peaks remained the same. Propylene is then polymerized in the channels of sepiolite, as shown by Sandí et al.[1]

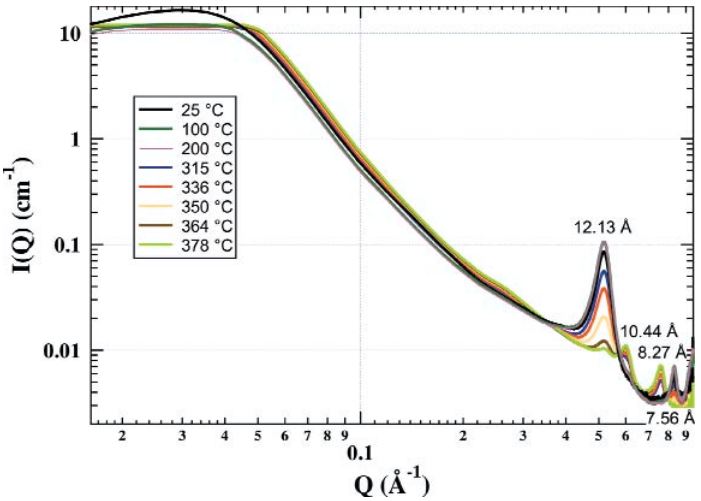

FIG. 1. In situ SAXS determination of the structural changes of sepiolite as a function of temperature.

Acknowledgments

Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Science, under Contract No. W-31-109-ENG-38. Support from BESSRC-CAT personnel is greatly appreciated.

References

[1] G. Sandí, K.A. Carrado, R.E. Winans, C.S. Johnson, and R. Csencsits, J. Electrochem. Soc. **146**, 3644-3648 (1999).