Heats of Adsorption of N-Hexane by Thermal Gravimetry with Differential Scanning Calorimetry (TG-DSC): A Tool for Textural Characterization of Pillared Clays

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Abstract: Microporous materials, (materials with pore sizes with widths <2 nm) were prepared by pillaring of smectites obtained from different soil deposits. The materials were prepared by intercalation with oligomeric cations of aluminum, which were transformed to aluminum-oxide pillars by calcination. The adsorption of n-hexane in the pillared clays was studied by the determination of heats of adsorption. Heats of adsorption were measured using either a static microcalorimeter or differential scanning calorimetry coupled with thermogravimetry (TG-DSC). In this latter case, two different procedures were used that differ on the introduction of the (n-hexane) molecules that are to be adsorbed. The results obtained by the (standard) static microcalorimeter method and the TG-DSC method were compared. This comparison showed the heats of adsorption obtained by TG-DSC are differential heats of adsorption, and it showed the range of adsorption. Characterizing the texture of pillared clays, especially microporosity, is important for monitoring the intercalation process and for determining potential applications of these materials. Correlations between the obtained heats of adsorption and the dimensions of micropores suggest that TG-DSC is a semi-quantitative method for characterizing micropores in aluminum-pillared clays.

Key Words: Adsorption • Calorimetry • Microporous Materials • Pillared Clays • Textural Characterization • TG-DSC

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