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Application of the alkylammonium method to determine the layer charge of the expandable component of mixed-layer Chlorite/Smectite

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The layer charge characteristics (amount, tetrahedral or octahedral character and distribution) of the expandable component of mixed-layer chlorite/smectite (C/S) of diagenetic marly samples (R1 stage of smectite illitization) have been analyzed in detail using the alkylammonium method. The obtained data have been used to investigate the diagenetic evolution of the Upper Cretaceous materials from the Alava Block (Basque Cantabrian Basin). The application of the procedure has been validated using the rapid estimation of the layer charge with the two commercially available alkylammonium chlorides (Nc: 12 and 18). The sample used to verify the methodology is a nearly pure corrensite caused by contact metamorphism and hydrothermal alteration of marly rocks associated with subvolcanic bodies during Middle Jurassic in Priego de Córdoba area (SE Spain) of the Subbetic zone.

For the diagenetic samples, conventional XRD methods show a heterogeneous clay mineral suite with R1 order mixed-layer illite/smectite, illite, kaolinite, chlorite and small amounts of mixed-layer C/S.

Despite the small proportion of C/S and the numerous coexisting clay minerals, the alkylammonium method has allowed us to study the nature of the smectitic component of C/S. The treatment of the <2 and <0.5μm fractions with the complete series of alkylammonium ions (Nc: 6 to 18) before and after Li saturation shows layer charge values of 0.41 eq/O10(OH)2, calculated from MTB transitions. A significant homogenity in the distribution of charge in the layers has also been observed and a tetrahedral location deduced from the small variations shown after Li saturation. The XRD patterns obtained from the <0.5μm have not allowed layer charge calculations, since the C/S content is clearly smaller in this fraction.

The diagenetic samples have also been studied by TEM after intercalation of alkylammonium ions (Nc: 8 and 14) in the interlayers. The treatment with the short alkyl chain shows a bilayer alkylammonium-ion arrangement, represented by spacings of 31Å (14Å Chl + 17Å Sm). The saturation with the long alkyl chain shows spacings of over 31Å indicating a BTP transition. Lattice fringe images show 50:50 C/S mixed layer crystals with large coherent domains (800 to 1500Å thick) and no significant changes in the spacing both along and between expandable layers of the packets, in agreement with the XRD results. This homogenity of the smectite layers could be related to the thickness of the mixed-layer C/S packets and it explains its concentration mainly in <2μm fraction. Chemical data of AEM analyses of the mixed-layer C/S have confirmed both the calculated layer charges and the dioctahedral nature of the expandable component.

Due to the excellent agreement found between the data obtained by three different techniques and the validation of the methodology with a nearly pure corrensite, we conclude that alkylammonium method can be successfully applied to characterize the expandable component of mixed-layer C/S, which has been scarcely referred until now.