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# Illite-smectite structural changes during metamorphism in black Cambrian Alum shales from the Baltic area

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## ABSTRACT

Illite-smectite (I-S) from Cambrian black shale of both early diagenetic and anchimetamorphic grade was investigated to determine the mechanism of the clay transformation. The layer sequences, the distribution of thicknesses of coherent scattering domains (CSDs), and the three-dimensional ordering were determined by X-ray diffraction (XRD). The proportions of cis-vacant (cv) and trans-vacant (tv) 2:1 layers were determined by thermal analysis and the proportion and distribution of interlayer ammonium by XRD and by infared spectroscopy (IR). The structural formulae were determined from total chemical analysis, and Mössbauer and $^{27}$Al NMR spectroscopies, and the particle shape and size investigated by atomic force microscopy (AFM). In the early diagenetic samples, the I-S is composed of two phases, one of which contains 0.05 and the other 0.25 smectite (S) interlayers. The first phase does not change during metamorphism. In the second phase, 0.20 S are converted to tobelite (T) layers through fixation of $NH_4^+$, but the I layers are not changed. Simultaneously, the proportion of cv layers changes from 0.18 to 0.02, and the tetrahedral substitution of Al for Si is parallel to the increase in T layers. All I interlayers contain 0.75K per $O_{10}(OH)_2$. Furthermore, the metamorphism results in increasing mean thickness of CSDs from 5.1–6.8 nm for the low-diagenetic samples to 6.7–8.4 nm for the anchimetamorphic samples. We conclude that the tobelitization was accompanied by transformation of cv to tv 2:1 layers adjacent to the smectite interlayers, and formation of tv layers adjacent to the newformed tobelite interlayers in otherwise intact crystallites. This mechanism only partly resembles the tobelitization previously observed in the Upper Jurassic North Sea oil source rocks. I-S in these rocks contained tv 2:1 layers and T interlayers formed through solid-state Al for Si substitution in the tetrahedral sheet and by ammonium fixation in the corresponding interlayers. These different mechanisms are probably because the North Sea I-S originated from weathered illite, like the Cambrian high-illitic phase, whereas the Cambrian low-illitic phase undergoing the transformation originated from cv smectite of volcanic origin. The results indicate that the illitization in oil source rocks is linked to oil generation, and that it deviates from the illitization in other rocks because of the supply of ammonium formed during oil generation and the fixation of this ammonium in the former smectite interlayers.