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Titel

Sample pre-treatment effects on identification of smectite by X-ray diffraction

Abstract

Analysing the mineral assemblage of soils and their clay fractions requires extensive pre-treatment, such as removal of carbonates, iron oxides, and organic matter, as well as dispersion procedures. These pre-treatments affect the properties of target minerals, and thus, hamper their identification. Smectites, being expandable and of small particle size, are most prone to undesired changes upon pre-treatments.

We studied the changes in X-ray diffraction patterns of smectite-dominated soil material (topsoil and subsoil of vertisols) in response to sequential pre-treatments. The sequence started with the removal of carbonates (by HCl), followed by the removal of iron oxides (dithionite-citrate-bicarbonate (DCB) method, without heating) and organic matter by H₂O₂ or NaOCl (at pH 8). Last, the samples were dispersed either in sodium pyrophosphate or sodium pyrophosphate in combination with sonication. Untreated material and material from each treatment step was magnesium-saturated, and then freeze dried. Textured specimen were analysed by X-ray diffraction.

The pre-treatments had small effects of the position of the 001 reflection, with a tendency to larger d spacing with increasing numbers of pre-treatments. This suggests increasing accessibility of the interlayer spaces, probably by increasing dispersion of the samples. Also, the width of the 001 reflection was little affected by the different treatments. Pre-treatment of soil samples with HCl for removal of carbonates, with DCB for iron oxide removal, and with H₂O₂ for organic matter removal had either some positive or only little negative effects on the intensity of 001 reflection of smectite. Dispersion with pyrophosphate, especially in combination with sonication, strongly decreased the 001 reflection. Treatment with NaOCl resulted in a more complete removal of organic matter than H₂O₂, however, at the cost of a pronounced drop the intensity of the 001 reflection. In summary, all pre-treatments of soil samples for X-ray analyses have the potential to hamper the identification of smectite. The effect of the removal carbonate, iron oxides, and organic matter is little; these treatments may even support the identification in some samples. Dispersion with pyrophosphate or by sonication and the treatment with NaOCl may impair the identification of smectite in textured specimen.