Scanning Transmission X-ray Microscopy analysis of the composition and distribution of the organic matter chemically stabilized by soil clay minerals

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Chemical interactions with mineral surfaces are important for stabilization of organic matter (OM) in soil, and hence for its preservation against microbial biodegradation. To better discern the mechanism of stabilization of C in high Fe smectite-illite soil clay nanoparticles, we used multi-element scanning transmission x-ray microscopy (STXM-NEXAFS) to determine the nature of the chemically bound natural OM and to visualize the spatial distribution of C and N and the association with Al, Si, and Fe in intact soil clay nanoparticles. Solid-state ^{13}C NMR spectroscopy was used to characterize natural OM associated with clay minerals. The soil clay fraction containing 5.5% organic C was isolated by sedimentationfrom the surface of a prairie soil in Minnesota (pH 6, 32.5% clay, and 3.7% organic C). The clay was subjected to density separation in sodium polytungstate combined with low energy ultrasonic dispersion to separate the free and physically protected OM and black C from the chemically bound C, resulting in three fractions: light, medium, and heavy. The CP-MAS ^{13}C-NMR spectrum of the whole soil clay suggested that polysaccharides and polypeptides are the prevailing organic components, with only a minor component of aromatic C, but the DP-MAS ^{13}C NMR data suggested black C to be predominant. The STXM results showed that proteins and polysaccharides are abundant in the soil clay and the black C constitutes distinct particles Iron was identified to occur as Fe(III) associated with Al and Si in the clay. The results suggest that (1) the smectite-illite sheets in the soil preferentially retain peptides, and polysaccharides favoring the protection of these normally readily biodegradable fractions relative to the lignin-derived phenolic components that are incorporated within the light fraction; (2) black C is contained in the light fraction as a separate phase, and is likely partially associated with OM bonded with the smectitic clays; and (3) lipids are associated with organic-clay nanoparticles as a separate phase.