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Determination of the composition of the organic matter chemically stabilized by agricultural soil clay minerals: Spectroscopy and Density Fractionation

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The interactions between soil organic matter and clay minerals are considered important processes because of their ability to sequester C in soil for long periods of time, and hence control C in the global C cycle when present. However, differing results have been reported regarding the composition of the soil organic matter – aromatic fractions versus aliphatic fractions – associated with clay minerals. To clarify this critical issue and better understand the C sequestration process in soils, we aimed to determine the nature of the chemically bound natural organic matter on clay surfaces, and to probe the speciation and spatial distribution of C in the soil clay nanoparticles using direct spectroscopic measurements namely solid-state CP-MAS and DP-MAS 13C NMR spectroscopy, x-ray diffraction spectroscopy (XRD), and scanning transmission x-ray microscopy (STXM). We tested the hypotheses that peptides and polysaccharides are stabilized by the smectite-illite clay while the lipids and black carbon are a separate phase; and that they are evenly distributed on clay surfaces. A soil clay fraction (5.5% organic C) was isolated from the surface of a prairie soil (Mollisol) in southwestern Minnesota, characterized by a pH 6.0, 32.5% clay content, and 3.7% organic carbon, using a sonication-sedimentation-siphoning process in distilled water. Then was subjected to density separation combined with low energy ultrasonic dispersion to separate the free organic and black C (light fraction) from the chemically bound C (heavy fraction).
The XRD results indicated a dominance of interstratified smectite-illite clays in soil. The 13C-NMR spectra of the soil clay fraction suggested that polysaccharides and polypeptides are the prevailing components of the organic matter associated with the mineral clay, with only a minor component of aromatic C. The light fraction has strong alkyl C-H bands characteristic of fatty acids plus strong C-O bands characteristic of polysaccharides, including the anomeric C band centered at 105 ppm. The aromatic band at 130 ppm and the phenolic C-O band at 150 pm are strong as well indicating the presence of black carbon and lignin-derived components, contrary to the heavy fraction where they are almost absent. STXM results indicated that the proteins are abundant in the soil clay fraction, separate from lipids, and partially associated with saccharides. The black carbon constitutes a separate phase, but is amply present with lipids and lignin-derived compounds in the light fraction. We conclude that (1) the smectite-illite sheets in our soils preferentially retain peptides, and polysaccharides favoring the protection of these normally readily biodegradable fractions relative to the lignin-derived phenolic components; (2) the black carbon constitutes a major component of the light fraction, and is partially attached to the organic matter bonded with the smectitic clays; and (3) the lipids are associated with soil clay fraction as a separate phase, but are not bound to clay minerals.