

Analytical high resolution microscopic investigation of organic coating on co-composted biochar

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Aged and/or co-composted biochar amendment improves soil fertility by changing certain proprieties like the porosity and sorption capacity, the redox properties, water holding capacity and nutrient transformations in soil. The beneficial properties have been correlated with surface functional groups resulting from the interactions between black carbon particles, inorganic and organic matter in the soil and soil biota, manure or other compost feedstock. As a result, porous organic layer and organo-mineral phases on the biochar surfaces are formed.

This paper presents a detailed analysis of the porous layer and organo-mineral phases formed on co-composted biochar by using high resolution scanning transmission electron microscopy (STEM) and electron energy loss (EELS) as well as energy dispersive X-ray spectroscopy (EDX). The fine structure fingerprints of carbon and nitrogen edges have been used to identify the functional groups, while EDX was used to identify the mineral phases. However, in order to achieve undoubtable results a novel preparation technic of the sample has been developed. The preparation involved 3D gold sputtering on the black carbon particles in order to preserve the surface intact, embedding in resin and, ultrathin microtome cutting.

The investigation was carried out using a probe corrected Titan 3G, at a voltage of 60 kV and in cryo-condition, with an EELS energy resolution of 0.15 eV and a spatial resolution down to atomic layers.

We proved the presence of both C and N functional groups in the porous, heterogeneous and hydrophilic organic layer and organo-mineral agglomerates. The organic layer fully covered the outer surface of the black carbon piece, but also the surface of internal pores. Its thickness varied from 500-1000 nm on the outer surface down to a couple of nanometres on internal pores. The observed C functional groups have been identified to correspond to: aromatic, aromatic with side chain, ketone, aliphatic, carboxyl/amine carbon and, carbonyl while the N functional groups were: pyridine, imine, amide/peptide, pyrrole, and NO₂-/oxidised N.

The STEM analysis also revealed the formation of complex organo-mineral agglomerates involving Ca but also the redox-active Fe as iron oxide nanoparticles and P as magnesium phosphate nanoparticles.

These findings are valuable information which contributes to the understanding of biochar reactions with soil and plants as a function of agronomic practice and environmental factors.

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