Recommendations for isolation of humin fraction from soil material

Jerzy Weber¹, Elżbieta Jamroz¹, Andrzej Kocowicz¹, Magdalena Debicka¹, Jakub Bekier¹, Maria Jerzykiewicz², Aleksandra Ukalska-Jaruga², Lilla Mielnik³, Romualda Bejgier³, and Irmina Ćwieląg-Piąscecka¹

¹Wroclaw University of Environmental and Life Sciences, Institute of Soil Science and Environmental Protection, Wroclaw, Poland (jerzyweber@gmail.com)
²Institute of Soil Science and Plant Cultivation – State Research Institute, Pulawy, Poland
³West Pomeranian University of Technology in Szczecin, Department of Bioengineering, Physics and Agrophysics Lab, Szczecin, Poland
⁴University of Wroclaw, Faculty of Chemistry, Wroclaw, Poland (maria.jerzykiewicz@chem.uni.wroc.pl)

Methods of isolation of the humin fraction can be divided into two main groups: (1) extraction of humic (HA) and fulvic (FA) acids followed by extraction of humin with different organic solvents, and (2) extraction of HA and FA followed by removal of soil mineral fraction. To isolate the large amounts of humin necessary to study the interactions of this fraction with pesticides, we examined some modifications of the latter method.

The first step was to separate HA and FA according to a modified IHSS method (Swift 1996). HA and FA were extracted with 0.1 M NaOH with a 5:1 ratio of extractant to soil. 20 hours shaking was found to be more effective, but 4 hours shaking provided the advantage of being able to extract twice a day, which ultimately shortened the procedure time.

The HA and FA free residue was then digested to remove mineral components. We used several (up to 8 weeks) digestions with 10% HF/HCl as higher concentrations of HF can result in structural alteration of the organic compounds (Hayes et al. 2017). While HF/HCl treatment can lead to hydrolysis and loss of polysaccharide and protein materials (Stevenson 1994), the advantage of using HF is the removal of paramagnetic compounds (such as Fe), which facilitates the use of spectroscopic techniques to characterize humin. In contrast to the procedures for only increasing the concentration of organic matter (Schmidt et al. 1997), the sample was digested until the mineral fraction not complexed with humin was completely digested. We tested different modes of mineral fraction digestion in 10% HF/HCl using polyethylene centrifuge bottles. Occasional shaking once a day had the same effect as continuous shaking. It takes 6 weeks to digest 200 g of pure sand in a 1000 cm³ bottle, when the HF/HCl was weekly replaced. After replacing HF/HCl every 2 weeks, the digestion time of the same material increased to 8 weeks.

After treatment with HF/HCl, the residue was rinsed with 10% HCl to remove secondary minerals. The residue was washed with distilled water until the neutral pH and then dialyzed to a negative Cl⁻ test with AgNO₃. Then the humin fraction was freeze dried.
Literature


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