Stabilization of Iron (II) Species in Humic Based Nanocomposite as Shown by XAFS and Mössbauer Spectroscopy

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Iron is a mineral nutrient presenting in plants and animals, it is crucial to living beings because it takes part in electron transporting and biosynthesis processes in cells. Iron deficiency is the most common nutrient deficiency in the world. Since iron can mainly be obtained from the foods, it is vital to consume iron rich meat and vegetables so that the body can absorb the iron from the diet. Bioavailibility of iron forms Fe (II) is higher than Fe (III). Humic substances are perspective matrix for bringing active compounds in living systems. Thus to obtain humic based samples containing Fe (II) is an issue of the day.

Goal of this research was to synthesis humic-based compounds containing stable iron (II) and to characterize redox speciation and local neighborhood of Fe atoms using XAFS and Mössbauer spectroscopy. Iron humate was obtained from commercially available potassium humate and iron (II) sulfate at pH 10 with stirring under pH control. Iron content was determined using spectrophotometer in the form of complex with o-phenantroline after oxidative digestion. Total iron content was 11 mass%. Results of EXAFS analysis and first shell approximation are given in the Figure 1.

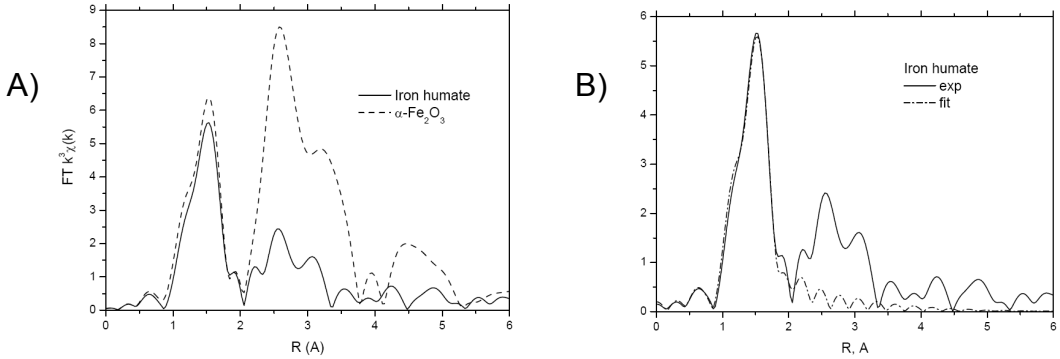

Figure 1. FT of EXAFS spectra of iron humate in comparison with standard α-Fe_2O_3 (A) and first shell approximation of local neighborhood of Fe (B).

The parameters of the first shell were R_{Fe-O} 1.975±0.015Å, N_O 4.8±0.9, σ^2_O 0.011±0.002, E_O -3.1±2.3 with fit quality R_f 0.65%. Results of EXAFS have shown that iron is surrounded with oxygen atoms in slightly distorted octahedral coordination and has Fe-O distance and structure similar to iron (III) oxide. No traces of iron (II) was found using X-ray adsorption spectroscopy. Redox speciation of iron was characterized using Mössbauer spectroscopy; parameters are given in the Table 1.

Table 1. Mössbauer parameters of iron humate at 5 K

Iron species	δ, mm/s	Δ, mm/s	H, kOe	S,%	G, mm/s
Fe (III)	0.46	-0.07	481	50	1.04
	0.43	-0.11	412	23	1.14
	0.51	0.65	-	12	0.70
Fe (II)	1.26	2.74	-	16	0.47

As shown by Mössbauer spectroscopy iron in the sample is presented with Fe (III) and Fe (II), about 75 % of total iron consist of superparamagnetic iron (III) oxides nanoparticles and about 16 % of total iron is stabilized in the form of Fe (II) which is expected to be bioavailable for plants and animals.

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