A MULTIANALYTICAL APPROACH FOR STUDY OF FRESH NANOSIZED PRECIPITATES FROM AMD AT ZLATÉ HORY, CZECH REPUBLIC

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One sample of fresh precipitates deposited from acid seepage of complex ore-mine tailing impoundment at Zlaté Hory, Czech Republic was studied by numerous techniques, including: ICP-ES and ICP/MS, X-ray powder diffraction (XRD), Mössbauer spectroscopy, FTIR and diffuse reflectance spectroscopy (DRS), voltametry of microparticles (VMP), thermogravimetry, differential scanning calorimetry (DSC), atomic force microscopy (AFM), high-resolution transmission electron microscopy (HRTEM) with energy dispersive X-ray (EDX) and selected area electron diffraction (SAED), and BET surface area measurements, to monitor their phase composition, structural and magnetic properties, particle size, morphology and surface area. Chemical analysis shows prevailing presence (up to 96 %) of hydrous ferric oxides, with the general formula $Fe_2O_3 \quad nH_2O$. rystalline phases, recognized by XRD analyses, are represented by subordiante, minor crystalline goethite, whereas very broad peaks in XRD diagrams indicate predominance of amorphous to poorly crystalline phases in the studied precipitates. Occurrence of mostly 2-line ferrihydrite is evident from XRD, FTIR spectra, VMP and particularly from low-temperature Mössbauer spectroscopy at 5K, which also indicates a size of the superparamagnetic particles well below 10 nm. Blocking temperature (T_B) of these phases is below T = 42 K, but higher than 25 K. At 5 K, the Mössbauer spectra do not show any change in the external magnetic field (4T) indicative of extremely low crystallinity of fine particles. Low ordering of structure is recorded also in DRS. HRTEM revealed globular aggregates about 150 nm in diameter rimmed by acicular irregular crystals suggestive of goethite. Isometric, ~ 5 nm sized particles ascribed to ferrihyhrite are evident from HRTEM (particularly from SAED of aggregates) and from AFM. EDX spectra indicate that Si, Ca and S are the most abundant elements adsorbed on the surface of particles. These natural nanoparticles have high surface area – up to 220 m^2/g. The origin of these phases is consistent with geochemical models of precipitation of mineral phases from acid mine drainage and supports the validity of these models. The acquired data help to better interpret and understand processes taking place on acid mine drainage sites and are of high environmental relevance. Thermogravimetry, DSC and subsequently XRD equipped with a heating cell shows that the thermal transformation of these precipitates to maghemite occurs above 270 °C. This suggests the possible use of these natural materials as an inexpensive precursor material for magnetic nanoparticles, which are important for many industrial and environmental applications.

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