Coupled and combined analyses for unambiguous iron-oxy hydroxides characterization: from laboratory to industrial use

- Orberger, Beate (*GEOPS, Université Paris Saclay, Université Paris Sud, Bât 504, 91405 Orsay Cedex, France*);
- Wagner, Christiane (*Sorbonne Université, CNRS-INSU, Institut des Sciences de la Terre de Paris, ISTeP UMR 7193, F-75005 Paris, France*);
- El Mendili, Yassine (*ESITC, Caen, France*);
- Chateigner, Daniel (*CRISMAT, CNRS, Caen, France*);
- Gascoin, Stéphanie (*CRISMAT, CNRS, Caen, France*);
- Pillière, Henry (*Thermofisher, Artenay, France*);
- Fialin, Michel (*Sorbonne Université, IPGP, CNRS-INSU, UMS 3455, UMR 7154, F-75005 Paris, France*);
- Rividi, Nicolas (*Sorbonne Université, IPGP, CNRS-INSU, UMS 3455, UMR 7154, F-75005 Paris, France*);
- Boudouma, Omar (*Sorbonne Université, CNRS-INSU, Institut des Sciences de la Terre de Paris, ISTeP UMR 7193, F-75005 Paris, France*);
- Wirth, Richard (*GFZPotsdam, Potsdam Germany*)

Abstract

Natural and synthetic iron oxides and iron hydroxides are important minerals for many industrial sectors (e.g. steel making, colors, pigment for coating, electronics, catalysis, soil, waste water and gas treatments, and medicine). In natural environments, such as iron ore mines or iron rich soils (laterites or bauxites), iron oxy-hydroxide associations are complex and evolve related to varying physico-chemical conditions, including. biological interactions. For efficient resource use, unambiguous multiscale characterization is indispensable. Synthetic iron oxides, produced for medical and electronic sector, needs to be failure-free pure phases, thus a continuous quality control is required. Complex iron oxy-hydroxide association can be related to various processes, topotactic transition, pseudomorphosis by substitution and alteration paramorphosis, and corrosions, leading to massive, porous, fibrous and acicular textures or poorly crystalline crusts.We present examples from iron ore deposits, where coupling of X-Ray diffraction (XRD) with scanning electron microscopy (SEM) and micro-Raman spectroscopy is a powerful tool to distinguish hematite, maghemite and magnetite at grain scale. Oxygen analyses by electron microprobe at (EMPA) fixed carbon coating thickness help to distinguish magnetite and hematite, and contribute with quantitative trace element analyses to chemically differentiate both oxides. At micro- and nano-scale, Transmission Electron Microprobe analyses coupled to X-Ray Diffraction (XRD) and Electron Energy Loss Spectroscopy (EELS) on nanometric inclusions can unambiguously identify various iron oxy-hydroxide phases. In Nickel-laterite and bauxite profiles, iron oxy-hydroxides (e.g. lepidocrocite, ferrihydrite, goethite…) are abundant and may form complex intergrowth with various types of

phyllosilicates. Part of it host valuable metals such as Nickel. Combined XRF-XRD and Raman spectroscopy allow phase mapping and differentiation at micron scale of these phases, and even detect solid solutions (e.g. Ni-rich and Ni-poor goethite; El Mendili et al., 2019). Results from coupled laboratory analyses are necessary for building up data bases. They allow calibrating recently developed combined XRF-XRD-Raman benchtop systems. For industrial applications coupled and combined analyses will increase resource efficiency, and ensure a quality control for natural and synthetic iron oxide products. Such systems are recently developed by EU projects, such as SOLSA (www.solsa-mining.com).El Mendili, Y., Chateigner, D., Orberger, B., Gascoin, S, Bardeau, JF., Petit, S., Le Guen, M., Pillière, H. (2019). Combined XRF, XRD, SEM-EDS, and Raman analyses on serpentinized harzburgite (Nickel Laterite Mine, New Caledonia): Implications for Exploration and Geometallurgy. ACS Earth and Space Chemistry. 3, 10, 2237-2249; DOI: 10.1021/acsearthspacechem.9b00014

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