Characterization of a potential ilmenite reference material

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Ilmenite is a common accessory mineral in igneous and metamorphic rocks, and is a significant constituent in cumulates and high-Ti lunar basalts. Despite its importance, there is no well-characterized matrix-matched reference material available for high spatial resolution (10s to 100s micron scale) analysis using laser ablation- inductively coupled plasma mass spectrometry (LA-ICP-MS). Matrixmatching between reference material and unknowns is crucial for obtaining accurate elemental abundances in LA-ICP-MS analysis in order to minimize laser induced elemental fractionation. One criterion for establishing certified reference material is the abundant supply of the potential standard for wide distribution. In this respect, we had in our possession ~25 specimens of ilmenite megacrysts, ranging in size between 2 to 4 cm, obtained from alnöite intrusions from the island of Malaita, Solomon Islands. We report major element data obtained by electron microprobe (EMP) analysis, and trace element contents obtained by laser ablation- and solution mode-ICP-MS analysis for seven of the Malaitan ilmenite megacrysts.

LA-ICP-MS analyses were conducted on a Thermofinnegan Element2 high resolution ICP-MS, coupled to a UP213nm ND:YAG laser ablation system.. The NIST SRM 610 glass was employed as the external standard, and TiO_2 wt% contents (EMP) served as the internal standard for elemental abundance determinations using GLITTER©. For solution mode ICP-MS analysis, ilmenite megacrysts were crushed, hand-picked for purity using a binocular microscope, and dissolved in concentrated HCl for a period of ~7 days.

LA-ICP-MS results for most of the ilmenite megacrysts analyzed indicate significant variation of trace element concentrations that preclude their usefulness as reference material. The variation in elemental abundances obtained by LA-ICP-MS are smallest for Co (4-19%) and Nb (4-11%; both at 2s RSD level). Comparison between laser ablation and solution mode results indicate important discrepancies for certain elements (e.g. Mo), whereas there is very good agreement for the abundances of V, Hf, Nb, and Ta. The Malaitan ilmenite megacrysts are characterized by the presence of exsolution lamellae (average 64 wt% FeO and 22 wt TiO₂), which can account for some but not all of the discrepancy between solution and laser ablation results.

Kinetics of Cr(VI) adsorption and desorption on Montmorillonite and Kaolinite

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Chromium is a highly toxic contaminant that has been introduced into aquifers and shallow sediments and soils through a variety of anthropogenic activities. Although Cr (VI) typically migrates rapidly in aqueous solutions, it can be retarded by anion-type sorption on solids, and by subsequent reduction with organic matter and Fe-bearing solids. In this study, the kinetics of Cr (VI) adsorption and desorption are investigated as a function of ionic strength and sorbent concentration on two clay minerals.

Batch kinetics experiments are conducted using ~500 mL reactors with 10 μ M Cr (VI) in 0.1, 0.01 or 0.001 M NaNO₃ background electrolyte, under open atmospheric conditions at room temperature and pressure. Up to 30 g/L of montmorillonite (SWy-2) or kaolinite (KGa-1b) is added to the solution, which is then manually titrated to and held at a pH of 3 using small volumes of concentrated HNO₃. 20 mL aliquots of the slurry are removed periodically, prior to solid addition and then at timed intervals of 30 min to 1 day, for up to 14 days. After a minimum of 48 hours of sampling, the slurry pH is raised to ~10 using small additions of 1 to 10 M NaOH, Aliquots are removed periodically for at least two additional days with manual titration used to maintain a pH of ~10. Immediately upon removal from the slurry, each aliquot is centrifuged and filtered (0.45 μ m nylon syringe-filter), acidified with nitric acid, spiked with an internal standard (~1 ppm Y) and analyzed for Cr (VI) using UV/Vis spectrophotometry and for total Cr using ICP-OES.

In 30 g/L montmorillonite batch experiments at 0.1, 0.01, and 0.001 M NaNO₃ Cr (VI) achieves greater than 88% sorption within 2 hrs and approaches 100% adsorption within 1-2 days at pH 3, with no detectable dependence of the kinetics on ionic strength. In contrast, adsorption on 30 g/L of kaolinite occurs much more gradually, with only ~40% Cr (VI) removed from solution after 12 days at pH 3. Increasing the pH to 10 for up to 48 hrs does not result in the release of any Cr from the montmorillonite. A subsequent decrease in the pH to 3 did not result in release of measurable Cr (VI) or total Cr in 0.01 or 0.001 M NaNO₃, but up to 10% Cr (III), calculated by difference between ICP and UV/Vis data, was released in the 0.1 M NaNO₃ experiment, suggesting that reaction with FeII may have occurred.