

## INVESTIGATION OF THE MECHANISM OF APATITE/CARBONATES SEPARATION IN ACIDIC FLOTATION PROCESS USING *IN SITU* RAMAN SPECTROSCOPY

Lev O. Filippov, Université de Lorraine, Laboratoire GeoRessources UMR 7359 CNRS, ENSG, 2 rue du Doyen Roubault, BP10162, F54518 Vandœuvre-lès-Nancy, France  
lev.filippov@univ-lorraine.fr

Oumar B. Kaba, Université de Lorraine, Laboratoire GeoRessources UMR 7359 CNRS, ENSG, 2 rue du Doyen Roubault BP10162, F54518 Vandœuvre-lès-Nancy, France

Inna V. Filippova, Université de Lorraine, Laboratoire GeoRessources, UMR 7359 CNRS, ENSG, 2 rue du Doyen Roubault BP10162, F54518 Vandœuvre-lès-Nancy, France

Key Words: flotation carbonated hydroxyfluorapatite; calcite, phosphoric acid; Raman spectroscopy.

The separation of phosphate from carbonate gangue using flotation is extremely complex due to the similarities in surface properties. The acidic flotation process of carbonates at pH 4.5-5.0 followed by a direct phosphate flotation uses phosphoric acid as depressor of apatite during carbonate flotation. The mechanism of apatite depression is rather hypothetical because of lack of data on the surface speciation in such a complex mineral suspension. A real-time method for *in situ* control of surface compounds on the minerals (carbonated hydroxylapatite and calcite) using a submerged Raman analyzer probe is proposed in this work to collect the data on the nature and kinetics of phase formed. The carbonated hydroxyfluorapatite (C-HFap) and calcite reactivity in phosphoric acid at concentrations between 0 and 1 M) during 15 minutes was studied. The Raman spectra collected *in situ* and *ex situ* showed the peak at  $1085\text{ cm}^{-1}$  of calcium carbonate at  $10^{-3}$  and  $10^{-1}$  M on the surface of hydroxyfluorapatite that disappears at high acid concentration. Kinetics studies performed at  $4.6 (\pm 0.1)$  pH range during 15 minutes showed calcium carbonate formation after 2 minutes of conditioning and that disappears after three minutes (Figure 1). The infrared spectra of C-HFap in presence of phosphoric acid showed at  $10^{-3}$  and  $10^{-1}$  M new peak at  $713\text{ cm}^{-1}$  that belongs to calcium carbonate vibrations which seems to be due to C-HFap surface conversion as observed on XRD patterns. In flotation of phosphates from carbonate minerals in acid media, implications of such conversion surface phenomena favours separation of these

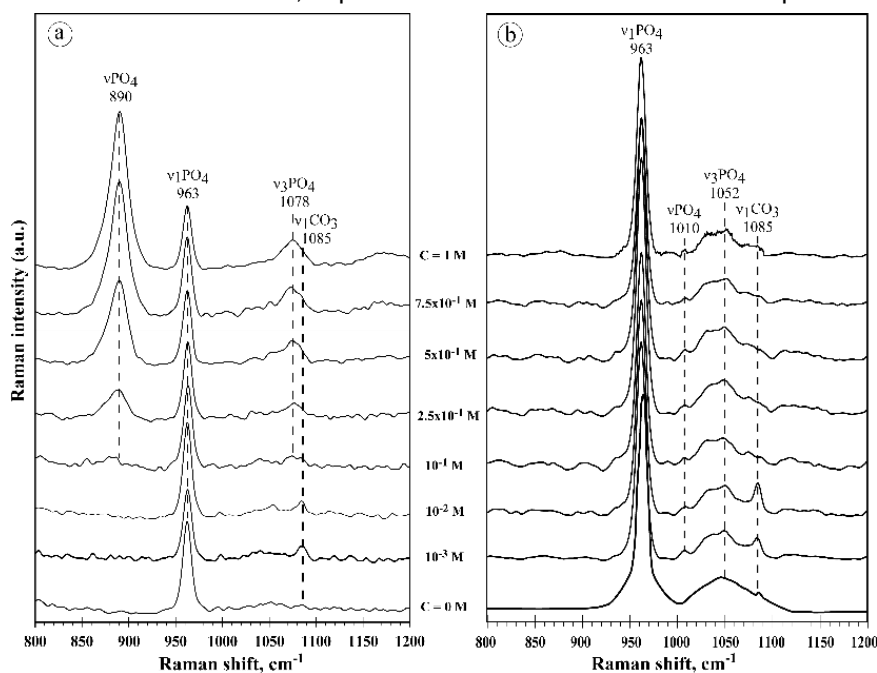


Figure 1 – Raman spectra of C-HFap after 15 min of contact with solutions of phosphoric acid of different concentrations measured (a) *in-situ* and (b) after filtration and drying (800 – 1200  $\text{cm}^{-1}$  region).

minerals by preventing C-HFap dissolution and collectors adsorption on its surface. The Raman and infrared spectra show the characteristic carbonate and phosphate peaks on the calcite surface attributed to a new phosphate phase which intensity decreases and increases, respectively with increasing phosphoric acid concentration and reaction time. A similar trend was observed with increasing acid concentration in the XRD analysis of calcite which also revealed that this new phosphate phase is dicalcium phosphate dihydrate or brushite. Fine sheets of calcium phosphate particles were observed by SEM analysis covering the calcite particles at a high phosphoric acid concentration.

The stability of calcium phosphate compounds on the surface of calcite and apatite is dependent on the initial acid concentration and stable pH values. Preferential fatty acid collector adsorption on the carbonates is attributed to the differential kinetics of hydrophilic calcium phosphates formation on the mineral surfaces. The observed mechanisms were used to adjust the reagent regimes of the flotation at various pH values performed on a sedimentary ore samples.