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Selectivity and Efficiency of the Acido-soluble Extraction in Sequential Extraction Procedure

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Abstract: In order to estimate heavy metals partitioning in soil or sediments, to evaluate their mobility and so their bioavailability, a 7-steps sequential chemical extraction procedure had been developed. But as the efficiency and selectivity of the acido-soluble extraction was now controversial, the objective of this complementary study was to show that this fraction is actually essentially composed of carbonates (as calcite or dolomite), but phosphates (as apatites) are few solubilized during this extraction step.

Key words: Chemical extraction procedure, apatite, efficiency

Introduction

Sequential extraction procedures, which are expensive and time consuming, have the advantage of characterizing the different labile fractions. So, they are one of the most useful tools for solid speciation of particulate elements, to study the origin, the fate, the biological and physicochemical availability and transport of sorbed elements (Gaiero *et al.*, 2003; Da Silva *et al.*, 2002; Leleyter and Probst, 1999; Ma and Uren, 1998; Baruah *et al.*, 1996; Lebourg *et al.*, 1996; Ure *et al.*, 1995; Shuman, 1985; Tessier *et al.*, 1979). The elements present in the exchangeable fraction are weakly bound and in equilibrium with the composition of the dissolved phase. Elements present in the 'acido-soluble' fraction are very sensitive to pH variations, whereas elements scavenged in the reducible or oxidable fractions are very sensitive to redox conditions and to microbiological activity.

The major problems linked to sequential extraction procedure are the lack of selectivity and of efficiency of each step of the procedure (Forstner and Kersten, 1988; Nirel and Morel, 1990; Kheboian and Bauer, 1987; Kersten and Forstner, 1989). Especially the efficiency of the acido-soluble extraction was controversial. It is why the previous study (Leleyter and Probst, 1999) has been now completed in order to show that this fraction is actually essentially composed of carbonates (as calcite or dolomite), whereas phosphates (as apatites) are few solubilized during this extraction step.

Materials and Methods

All the samples were leached by an optimized chemical extraction procedure (Table 1; Leleyter and Probst, 1999). This procedure dissolves selectively and efficiently all the chemical constituents of the river sediments and soils (Leleyter and Probst, 1999; Aubert *et al.*, 2004) which can be affected by changes in physico-chemical conditions, in the following order: elements dissolved by water (F1), really exchangeable elements (F2), elements bound to acido-soluble fraction (F3), reducible fraction (manganese oxides (F4a), amorphous iron oxides (F4b) and crystalline iron oxides (F4c)) and oxidable fraction (F5) which represents sulfides and organic materials. The Non-Residual Fraction (NRF) or "labile fraction" of the studied sample is the sum of all the previous leached fractions.

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Table 1: Protocol of the 7-steps sequential extraction procedure (Leleyter and Probst, 1999)

	Fractions	Reagent	Time	pH
F1	Evaporitic	H ₂ O	30 min	5.7
F2	exchangeable	1M Mg(NO ₃) ₂	2 h	5.0
F3	Acido-soluble	1M NaOAc/HOAc	5 h	4.5
F4a	Reducible (Mn)	0.1 M NH ₂ OH HCl	30 min	3.5
F4b	Reducible (Afe)	0.2 M (NH ₄) ₂ C ₂ O ₄	4 h	3.0
F4c	Reducible (Cfe)	+ 0.2 M H ₂ C ₂ O ₄		
F5	Oxidable	0.2 M (NH ₄) ₂ C ₂ O ₄	30 min	2.3
		+ 0.1 M C ₆ H ₈ O ₆		
		35% H ₂ O ₂ /HNO ₃	5 h	2.0
		then 3.2 M NH ₄ OAc		

Results and Discussion

Selectivity of the Sequential Extraction Procedure Used

Several tests on pure natural and synthetic minerals (such as clay minerals, calcite, dolomite, Mn-Oxides, Fe-Oxides and organic matter) have allowed to verify the repeatability, selectivity and efficiency of the used procedure (Leleyter and Probst, 1999).

Efficiency and Selectivity of the Acido-soluble Fraction

By using the chemical extraction procedure proposed by Leleyter and Probst (1999), Aubert *et al.* (2004) suggested that phosphate minerals like apatite were dissolved during the acido-soluble step, as they find a maximum of 5% of the total Ca leached from their soils in this step, despite that these samples presented no trace of carbonates.

Previous study (Leleyter and Probst, 1999) already showed that the extractant solution is efficient to dissolve all carbonates present in sediments (if the studied 1 g sample contain less than 50% of carbonates). Dolomite could be also solubilized during the sequential extraction procedure too (Fig. 1). Regarding apatite, complementary investigations have been performed to elucidate this point. The results are presented below (Fig. 1). Synthetic apatite (from ACROS Organics, Ca₃(PO₄)₂), has been leached by the used 7-steps procedure. The results obtained suggest that a maximum of 25% of Ca could be solubilized if the initial sample contains 10% of apatite. These results indicate that apatite is more stable than carbonates (as dolomite or calcite) but could be partially solubilized during the acido-soluble step of this sequential extractions procedure.

Moreover in order to study the selectivity of the acido-soluble step, we also investigated apatite stability during the 7 steps sequential extraction procedure (Table 2). The low percentage of Ca measured in the leachates indicate that apatites are stable during these extractions (except the acido-soluble fraction).

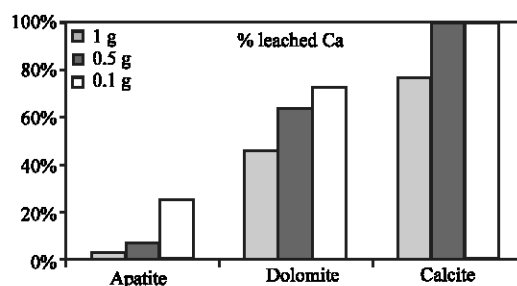


Fig. 1: Efficiency of carbonate dissolution with 10 mL of 1M NaOAc at pH = 4.50 (F3 extraction) versus the initial weight of material

Table 2: Efficiency of apatite dissolution according to % leached Ca

Fraction	F1	F2	F3	F4a	F4b	F4c	F5	NRF
Apatite 1 g	0	2	3	1	0	0	4	10
Apatite 0.5 g	0	3	7	2	0	0	1	13
Apatite 0.1 g	1	5	25	8	0	0	4	43

Conclusions

This study shows that the acido-soluble fraction is actually essentially composed of carbonates (calcite or dolomite), but that phosphates (as apatites) could also be partially solubilized during this extraction step.

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