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The selectivity of a sequential extraction procedure for the determination of iron oxyhydroxides and iron sulfides in lake sediments

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Abstract. A popular sequential extraction procedure (Tessier et al. 1979) designed to extract metals partitioned in various sediment phases, was evaluated for its selectivity. Amorphous FeOOH, FeS, and FeS_2 were added separately to natural lake sediments and sequentially extracted. The selectivity of the sequential procedure for the added solid phases was evaluated by determining the difference in the mass of Fe extracted from treated and control sediments. In the experiments where sulfide minerals were added, total S was measured in the residual solids in order to confirm selectivity of the method. Concentrations of total carbon remaining in the solid phase after each extraction step were also measured to determine the selectivity of the sequential procedure for carbon.

The procedure was moderately selective for Fe added as FeOOH; a mean of 77 ± 12% ($p < 0.05$) of the Fe added was extracted in the step designed to reduce Fe-Mn oxyhydroxides. In experiments where FeS was added, a mean of 69 ± 11% ($p < 0.05$) of the Fe added as FeS was extracted in the fraction designed to oxidize sulfides and organic matter. Approximately 25% of the Fe added as FeS may have been extracted prematurely. Although less precise, total S analyses confirmed that much of the FeS was extracted in the oxidation step, yielding 104 ± 87% ($p < 0.05$) of the S added as FeS. The procedure was highly selective for FeS_2; 92 ± 14% ($p < 0.05$) of the Fe added as pyrite was extracted in the sulfide extraction step. Extraction of 80 ± 54% ($p < 0.05$) of S added as pyrite confirmed that FeS_2 were selectively extracted in the sulfide extraction step. Carbon in the sediments was also selectively extracted in the oxidation step (77 ± 2.4% of total C; $p < 0.05$). The applications and limitations of sequential extraction procedures as limnological research tools are discussed in light of our results.

Introduction

Within the past decade, numerous studies have focused on the cycling and transport of trace metals and redox sensitive elements (i.e. Fe, S, Mn) in marine, riverine, and lacustrine sediments. This interest in the fate and transport of metals entering aquatic environments from both anthropogenic and natural sources has led to the development of sequential extraction procedures designed to ‘selectively’ measure metal partitioning in sediments. The selective extraction of metals bound to labile solid phases and recalcitrant mineral phases in sediments has been investigated (Tessier et al. 1979, 1980). These procedures have been developed for use as tools in geochemical exploration (Tessier et al. 1982), and more recently, to gain a better understanding about the partitioning and biogeochemical cycling of metals in acidified lake ecosystems (Tessier et al. 1989; White et al. 1989; White & Gubala 1990).

Total extractions of trace metals from sediment cores have traditionally been used to elucidate temporal trends in the deposition of anthropogenic sources of trace metals to lakes (Norton 1986; Nriagu & Rao 1987). These and other authors (Carignan 1985; White & Gubala 1990) have suggested that the use of trace metal profiles as a chronological record of metal deposition to lakes should be viewed with caution due to the potential for mobility and redistribution of trace metals within sediments.

The recent application of sequential or partial extractions to determine the partitioning of trace metals in sediment profiles has helped to underscore the significance of biogeochemical cycling and mobility of metals within the sediment column. Extraction techniques have been used to determine trace metal adsorption to Fe/Mn oxyhydroxides and organic particulates in estuarine systems (Lion et al. 1982). Trace metal binding to Fe oxyhydroxides has also been investigated in acidified lakes in Canada (Belzile & Tessier 1990; Tessier et al. 1989). Others have applied sequential extraction techniques to evaluate biogeochemical cycling of iron, trace metals, and sulfur in acidified lake ecosystems (Carignan & Tessier 1988; White et al. 1989; Gubala et al. 1990; White & Gubala 1990). Investigators have focused on the cycling of iron oxyhydroxides and iron sulfides within sediments and the role of microbial processes in controlling the accumulation and diagenesis of Fe, S and associated trace metals in lake sediments.

Following the development of sediment extraction procedures, further research has demonstrated that sediment handling and treatment prior to and during extractions can drastically alter sediment chemistry and cause repartitioning of metal phases into various sediment fractions (Campbell & Tessier 1984; Rapin et at. 1986; Kheboian & Bauer 1987; Tessier & Campbell 1988; Ajayi & Vanloon 1989; Belzile et al. 1989). In addition, it is generally recognized that the selectivity of various extractants for metals in specific phases is not absolute (Martin et al. 1987). While any specific extraction step can be moderately selective for a specific metal phase, at best the results are operationally defined. Although the reliance on sequential extraction procedures to fractionate metal phases in sediments has increased, it is acknowledged that additional research to determine the selectivity of methods for specific metal phases is needed if effective use of these techniques is to continue (Campbell & Tessier 1984; Martin et al. 1987).

Selectivity of sequential extraction procedures for specific Fe and S phases by making known additions to natural sediments has not been rigorously evaluated. In the reported experiment, we made known additions of a synthetic iron oxyhydorxide (FeOOH), iron monosulfide (FeS), and pyrite (FeS_2) to a freshwater lake sediment in order to better assess the selectivity of a popular sequential extraction procedure for these solid phases.

Methods

Sediment sampling and handling

All sediment samples were collected from Woods Lake, in the Adirondack region of New York (43°52′N, 74°58′W). The Woods Lake watershed is forested by deciduous species; soils are highly acidic and consist of shallow glacial till. Until recently, the water chemistry of Woods Lake was typical of ‘chronically’ acidic Adirondack lakes (Driscoll & Newton 1985). However, calcite additions to Woods Lake have recently been made as part of the lake acidification mitigation project (LAMP). Short-term water chemistry changes of Woods Lake in response to liming have been described by Driscoll et al. (1989).

Woods Lake sediment samples were obtained with an Eckman dredge near the maximum depth (11.5 m) of the lake. The sediment was immediately transferred to 1 L Nalgene bottles. The bottles were filled to capacity, tightly capped, and shipped to Indiana University. Because we used Eckman grab samples, the sediments were likely a mixture of oxic and anoxic layers. The sediments were stored in the bottles at 4 °C until processed (about 1 week).

Experimental

Three sets of independent experiments were conducted to evaluate the selectivity of individual extraction steps for known additions of amorphous iron oxyhydroxide, FeS, and FeS_2. The iron oxyhydroxide was synthesized following the method of Crosby et al. (1983), and was aged for one week prior to the experiment. The amorphous nature of the iron oxyhydroxide was verified by X-ray diffraction analysis and by recovery of stoichiometric amounts of Fe from the solid phase. The iron sulfides, FeS and FeS_2 (both 99.9%, 100 mesh, CAS 1317-37-9 and 12068-85-8), were obtained from Johnson Matthey/AESTAR (Seabrook, NH). The crystalline structure of the FeS was hexagonal and was formed by reaction of Fe metal with FeS_2; the FeS_2 was cubic and was formed by reaction of Fe metal with H_2S (Johnson Matthey/AESTAR, pers. comm.).

We conducted all stirring, reagent transfer, and decanting operations under $N_2(g)$ in order to reduce oxidation of sulfides and maintain comparable conditions for each experiment. In addition, reagents and rinse water for fractions F1 and F2 (see below) of the sequential extraction procedure were purged with N_2. The three experiments (one for each Fe phase added) were conducted separately, using the three distinct grab samples collected from the pelagic region of Woods Lake. Prior to adding the FeOOH, FeS, or FeS_2, the sediment was stirred for 2 h with a Phipps & Bird paddle stirrer (40 rpm) to thoroughly homogenize the grab sample. The sediment was then split into two beakers. To one beaker a known volume of FeOOH slurry, or known weight of FeS or FeS_2, was added to the sediment. The quantity of Fe added per gram dry weight of sediment (gdw) in each of the three experiments was as follows: 82 μmols Fe gdw^{-1} in the FeOOH addition experiment, and 34 μmols Fe gdw^{-1} in the FeS experiment, and 37 μmols Fe gdw^{-1} in the FeS_2 experiment. A companion beaker of sediment received no additions and served as the control in each experiment. Stirring continued for an additional 2 h after addition of the mineral phase to homogenize the sediment mixture. Triplicate subsamples of sediment from both the treatment and control beakers were measured to obtain wet and dry weights so that the results could be reported on a dry weight basis. Sediment from both beakers was then transferred to 50 ml polyallomer centrifuge tubes (approx. 0.8 g sediment dry weight/tube). Sediment porewaters were removed by centrifugation at 12,000 × *g* for 30 min. The sediment was then ready for the sequential extraction procedure. A flow diagram of the entire procedure is presented in Fig. 1.

We included a sufficient number of replicates in each experiment so that several subsamples could be removed after each extraction step of the sequential procedure for the analysis of sulfur and carbon in the residual solid phase. In this way, we could track the extraction of sulfur in both controls and treatments and compare these values to the iron extracted in the corresponding supernatants.

Analyses

The sequential extraction procedure that we used was devised by Tessier et al. (1979) and is used with minor modifications in many studies of

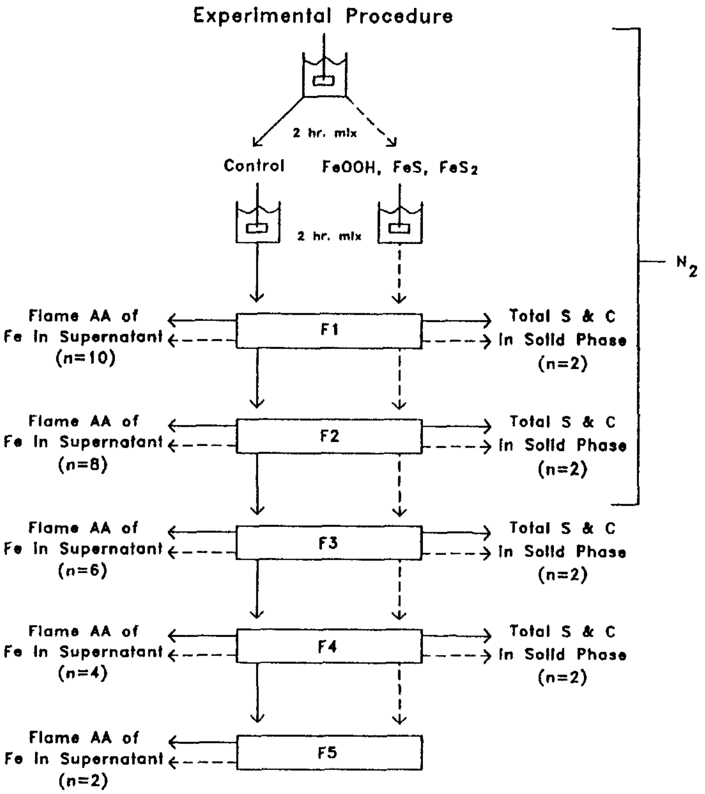

Fig. 1. Experimental design of the sequential extraction of sediments following the addition of known mineral phases to lake sediment.

lacustrine sediments. We designated the sequential fractions F1 through F5, nomenclature common to other papers published on this method. While the selectivity of the method remains in question, the procedure is designed to extract metals that are exchangeable (F1), specifically absorbed or bound to carbonates (F2), bound to Fe-Mn oxides (F3), bound to organic matter and sulfides (F4), or are bound to the mineral matrix (F5). The operational steps for extracting metals from each fraction are listed below:

F1: The sediment was extracted for 30 min. at room temperature with 8 mL 0.5 M $MgCl_2$, adjusted to pH 7.0. The samples were agitated gently on a wrist-action shaker.

F2: The sediment residue from F1 was extracted for 5 h at room temperature with 8 mL 1 M NaOAc, adjusted to pH 5.0 with acetic acid. These samples were also gently agitated on a wrist-action shaker.

F3: The residue from F2 was extracted for 6 h at 96 °C with 20 mL 0.04

M $NH_2OH \cdot HCl$ in 25% (v/v) acetic acid. The samples were periodically agitated during the course of this step.

F4: The residue from F3 was extracted for 2 h at 85 °C with 3 mL 0.02 M HNO_3 and 5 mL 30% H_2O_2, adjusted to pH 2.0 with HNO_3. After 2 h, an additional 3 mL 30% H_2O_2 (pH 2.0 with HNO_3) was added, and extraction continued at 85 °C for another 3 h. The samples were occasionally agitated during the entire procedure. The samples were then cooled, 5 mL 3.2 M NH_4OAc in 20% (v/v) HNO_3 was added, and the samples were diluted to 20 mL with deionized water. The samples were continuously agitated for 30 min on a wrist-action shaker.

F5: The residual sediment from F4 was digested with a HF-$HClO_4$ mixture. The sediment was first digested to near dryness with 10 mL HF and 2 mL $HClO_4$. A second 10 mL aliquot of HF was added along with 1 mL $HClO_4$, and the sample was evaporated to near dryness again. A final 1 mL $HClO_4$ was added and the sample was digested until white fumes appeared. The residue was dissolved in 12 M HCl and diluted to 25 mL.

After each extraction step, the sediments were centrifuged for 30 min at 12,000 × *g*. The supernatant was decanted and analyzed for iron. The sediment pellet remaining was rinsed with 8 mL deionized water and centrifuged or 30 min at 12,000 × *g*. The supernatant from this step was discarded. At this point, four tubes (two treatment and two control) containing the rinsed solids were removed from the extraction sequence to determine total sulfur and total carbon content. The sequential extraction procedure continued with the remaining subsamples (Fig. 1).

Iron from all sediment extracts and from synthetic FeOOH was analyzed by flame atomic absorption spectrophotometry; calibration was performed using matrix-matched standards. Total sulfur and carbon analyses were conducted on the dried (105 °C, 48 h) sediment pellet remaining after each extraction step using a Leco total sulfur/carbon analyzer.

Error analysis

The quantities of Fe and S extracted from the control sediments at each step in the sequence differed between the three experiments. The differences reflect the actual heterogeneity of the sediments of the pelagic region in Woods Lake and resulted from the fact that three separate dredge samples were necessary to provide sufficient sediment for our experiments.

Although Fe and S concentrations varied between experiments, the precision of Fe and S analyses from replicate samples at each step in the extraction sequence was high: relative standard deviations for Fe and S were generally $<5\%$ and $<10\%$, respectively. The sequential extraction procedure, as it is designed, is better suited for the analysis of liquid supernatants removed at the end of each extraction step. Nevertheless, we analyzed the concentration of S remaining in the solid phase after each extraction to determine the amount of S extracted in each step, and how it related to the amount of S added in the treatments. Using the S concentrations in residual solids in both the FeS and FeS_2 experiments to estimate the recovery of S introduced several sources of error not found in the straightforward analysis of Fe in the extracts. These sources of error contributed to the total error:

- Error associated with drying and weighing S in the residual solids. Oven drying can introduce inconsistent loss of some S from sediments (Amaral et al. 1989). In addition, the mass of solids remaining decreased as selective phases were attacked with each step, requiring reweighing of the subsamples and relating the S concentrations back to the original dry weight. Along with the potential loss of S during drying, the additional source of error from the second dry weight determination required propagation of both sources of error.
- A smaller sample size ($n = 2$) was necessary for the S analyses, as compared to Fe (with the exception of the fraction F5 analysis for Fe).
- Propagation of errors was larger in the calculation of percent S extracted from solid phase sediments relative to the analysis of Fe in the extracts. Calculating the difference of differences of dry mass-S contributed to large error propagation. We propagated all sources of error through our calculations of percent S and percent Fe extracted. Analytical and sampling errors were relatively minor, but propagation of these minor errors contributed to the large confidence intervals.

We believe that the sequential extraction procedure is better suited for the analysis of the liquid extract because the single analysis of liquid extract generates one source of measurement error while the determination of differences of dry mass S between two fractions compounds their respective measurement errors. Therefore, throughout our analysis of the results from the FeS and FeS_2 addition experiments, we place greater emphasis on the estimates of percent Fe extracted than on our estimates of percent S extracted.

Results and discussion

FeOOH additions

For extraction steps F1, F3, and F4, concentrations of Fe in the extracts of the sediments treated with amorphous FeOOH were significantly greater ($p \leqslant 0.05$) than Fe concentrations in the control samples (Fig. 2). Additional Fe extracted in fractions F1, F3, and F4 of the treatments accounted for all of the Fe added as amorphous FeOOH. The extraction step F3 of the sequential procedure recovered a mean of 63 μmols Fe gdw^{-1} of the 82 μmols amorphous FeOOH gdw^{-1} added to the sediment (recovery = 77 ± 12%; $p < 0.05$). This step, which uses a hydroxylamine hydrochloride-acetic acid mixture, is designed to selectively reduce Fe and Mn oxides to their soluble forms (Tessier et al. 1979). Approximately 10 μmols Fe gdw^{-1} of the Fe added as amorphous FeOOH (12.2 ± 8.1%; $p < 0.05$) was also extracted in fraction F4, which is designed to extract metals bound to organic fractions and sulfides. We do not attribute the additional Fe in fraction F4 to incomplete dissolution of amorphous FeOOH in fraction F3, since the molar

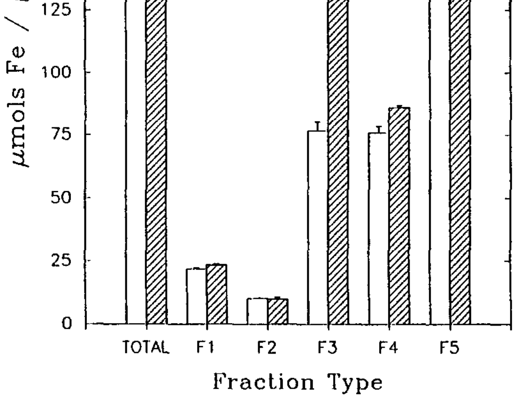

Fig. 2. The mean concentration of Fe in extracts from controls and treatments (amorphous FeOOH) at each step in the sequential procedure. Error bars represent +1 standard deviation of the mean.

ratio of hydroxylamine hydrochloride to total Fe extracted in fraction F3 was nearly 6:1. However, anoxic conditions in the sediments during the experiment may have contributed to reprecipitation of amorphous FeOOH as FeS, or sorption of reduced Fe to organic matter. It is also possible that the relatively high concentrations of Fe in extracts from step 3 could have contributed to significant carryover of Fe between fractions F3 and F4. Despite the apparent carryover of approximately 12% of amorphous FeOOH into F4, the sequential extraction step F3 was moderately selective in extracting amorphous FeOOH.

The differences in the mass of Fe extracted in F5 between controls and treatments were not significant ($p < 0.05$) (Fig. 2). Note that the majority of Fe in Woods Lake sediments (in both controls and treated samples) was extracted in the residual phase, tending to overshadow the relatively small amount of Fe added to the treated samples. The differences in extractable Fe in F5 between treated and control samples were small relative to the actual concentrations of Fe extracted in fraction F5, while the differences were large relative to the mass of Fe added as amorphous FeOOH to the treated samples.

We obtained similar results for fraction F5 in the FeS experiment and in the FeS_2 experiment (Figs 3a, 4a, respectively). It is possible that some of the Fe added in our treatments carried over to fraction F5. However, when considering only the extractions which removed a significant ($p < 0.05$) quantity of the added Fe in each of the three experiments, extracts likely to contain Fe from the added mineral phases accounted for $>90\%$ of the Fe added. Therefore, we estimate that $<10\%$ of the Fe added in any of the three treatments would have been extracted in fraction F5. As with the FeOOH experiment, the absolute amount of Fe extracted in F5 from the treated samples was large, the amount extracted from treated sediments was not significantly different ($p < 0.05$) than the amount of Fe extracted from control samples.

FeS additions

Iron attributed to the addition of 34 μmols FeS gdw^{-1} of sediment was extracted in concentrations significantly greater ($p < 0.05$) than the controls in fractions F1, F3 and F4 (Fig. 3a). Based on the analysis of Fe concentrations in extracts, the mean differences in Fe concentrations between treatments and controls were 3.0, 5.6, and 23 μmols Fe gdw^{-1} for fractions F1, F3 and F4, respectively. Relative to the 34 μmols Fe gdw^{-1} added as FeS, 8.7 ± 8.4%, 17 ± 11%, and 69 ± 11% of the treatment amount of Fe ($p < 0.05$) was recovered in fractions F1, F3, and F4, respectively. Analyses of total S, while less precise than Fe, corroborate our findings for Fe. Note that the differences in concentrations of total S in residual solids between controls and treatments remained relatively constant until F4, where the difference was eliminated (Fig. 3b). We attribute the loss in S to selective extraction of the added FeS. Compared to the 34 μmols S gdw^{-1} added as FeS, an average of 35 μmols S gdw^{-1} (104 ±87%; $p < 0.05$) was extracted in F4. Differences between S extracted from controls and that extracted from treatments in F1, F2, and F3 were not significant ($p < 0.05$).

Based primarily upon our Fe results, we suspect that significant amounts of FeS were extracted in fractions F1, F3 and F4 (Fig. 3). Other researchers have reported progressive extraction of FeS and acid volatile sulfides (AVS) from sediments (FeS is often a major component of AVS in sediment). Rapin & Förstner (1983) performed sequential extractions of synthetic amorphous FeS in absence of a sediment matrix, and found

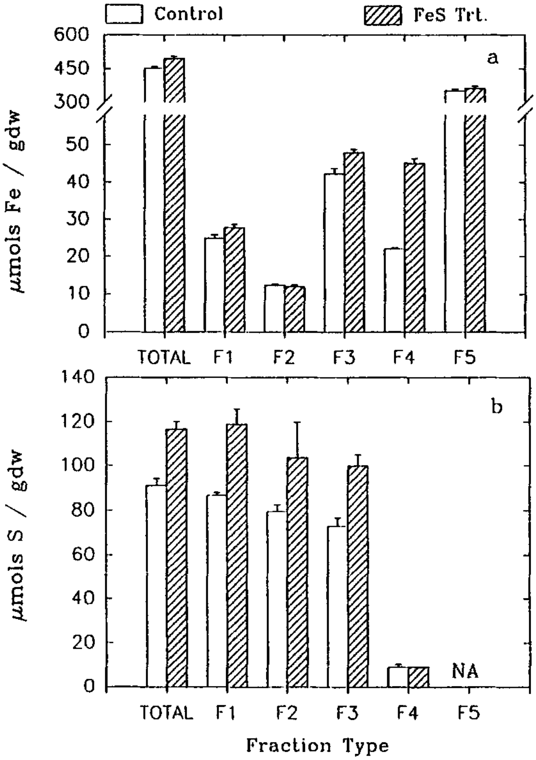

Fig. 3. The mean concentration of (*a*) Fe in extracts, and (*b*) S in remaining solids, from controls and treatments (FeS) at each step in the sequential procedure. Error bars represent +1 standard deviation of the mean.

that 60% of the Fe was extracted in fraction F4 as expected, and 20% was extracted in each of fractions F2 and F5. Rapin et al. (1986) demonstrated that over 94% of the acid volatile sulfides (AVS) in anoxic sediments from two lakes was extracted in fraction F3 when the sediments were handled anaerobically. However, when the sediments were handled aerobically, over 72% of the AVS was extracted in steps F1 and F2. Both our results and those of Rapin et al. show significant extraction of sulfides in extraction steps which precede oxidation (F1, F2, and F3 vs. F4). However, the data of Rapin et al. indicate that the majority of the sulfide was extracted in fractions F1, F2, and F3, while the majority of FeS in our experiment was removed in fraction F4. A hexagonal, crystallized form of FeS was used in our experiments, resulting in greater preservation of FeS through earlier steps. Steps F1, F2, and F3 use reagents which are not strong oxidizing agents, while extraction F4 uses a strong oxidizing mixture of HNO_3 and H_2O_2. Thus, the discrepancy between the two studies may reflect differences in the cystallinity of the FeS extracted.

FeS_2 additions

There were no significant differences in Fe concentrations between FeS_2-treated and control sediments in fractions F1, F2, F3 and F5, while the differences in F4 were significant ($p < 0.05$). Note that we measured a mean difference between treatments and controls of 34 μmols Fe gdw^{-1} in fraction F4 (Fig. 4a). Recall that our treatment amounted to 37 μmols FeS_2 gdw^{-1} of sediment. Thus, essentially all of the Fe added with the FeS_2 treatment was extracted in F4 (92 ± 14%; $p < 0.05$). Similarly, the analyses of total S in the FeS_2 experiment indicated that a significant amount of S attributed to the FeS_2 treatments was extracted in fraction F4 (80 ± 54%; $p < 0.05$) (Fig. 4b). Our results suggest that, in anoxic lake sediments, the HNO_3-H_2O_2 mixture effectively oxidized FeS_2 in the time allotted for this step, and that the sequential extraction procedure is highly selective for this mineral phase.

Tessier et al. (1979) observed that approximately 58% of the sulfide in two riverine sediments was extracted in fraction F4. Since no sulfide was extracted in F3 in their experiments, they state that this implies the sulfide removed in F4 was well crystallized pyrite or organosulfur compounds. By using known additions of selected mineral phases, our results confirm these findings, showing that while 17% of the Fe added as FeS was extracted in fraction F3, there was no significant extraction of Fe as FeS_2 in the same fraction. This indicates that well crystallized FeS_2, unlike the more amorphous FeS, is resistant to leaching and oxidation in steps prior to F4.

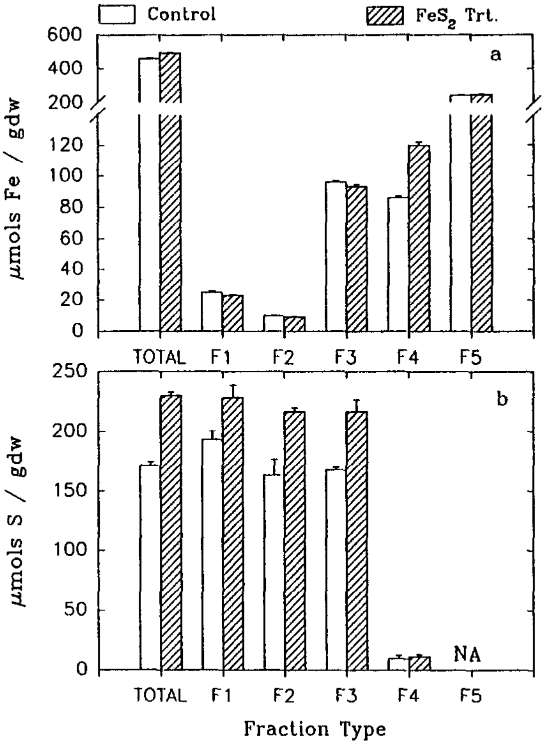

Fig. 4. The mean concentration of (*a*) Fe in extracts, and (*b*) S in remaining solids, from controls and treatments (FeS_2) at each step in the sequential procedure. Error bars represent +1 standard deviation of the mean.

Total carbon analyses

In the FeOOH, FeS, and FeS_2 addition experiments, we compared total carbon removed from controls and treated samples and found no significant differences. A representative example is the total carbon analysis for the FeS_2 experiment (Fig. 5). Total carbon was not significantly removed by the fraction F1 extraction in either control or treated samples. The negative values for percent carbon removed in fraction F2 indicated that significant quantities of total carbon (13% of the unextracted samples) were added to the sediment in the fraction F2 extraction. Tessier et al. (1979) obtained similar results, finding that fraction F2 had organic carbon concentrations that were approximately 17% greater than the total organic carbon content of the unextracted samples, although no confidence limits were reported. The higher carbon content of residual solids following the F2 extraction are likely a result of the extractant in fraction

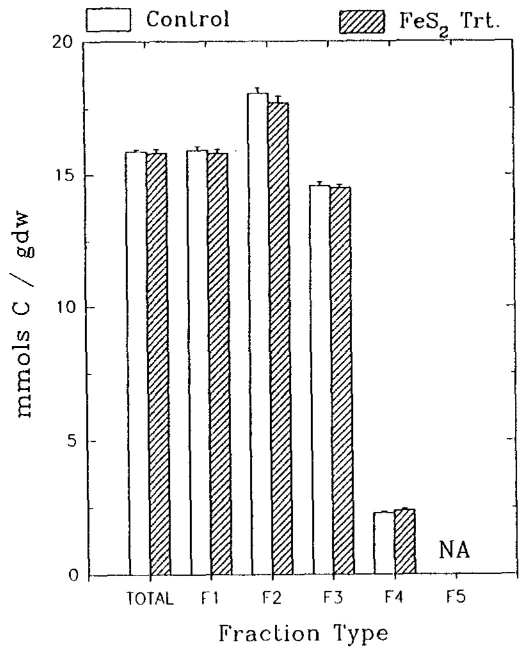

Fig. 5. The mean concentration of total carbon in remaining solids from controls and treatments (FeS_2) at each step in the sequential procedure. Error bars represent +1 standard deviation of the mean.

F2, 1.0 M sodium acetate brought to pH 5.0 with acetic acid. While about 8% of the initial concentration of total carbon was removed in the F3 extraction step, the largest percentage (77 ± 2.4%; $p < 0.05$) was removed in fraction F4, the step designed to oxidize organic matter with HNO_3 and H_2O_2. These results also compare favorably with those of Tessier et al. (1979), who extracted approximately 83% of the organic carbon in their untreated sediments in fraction F4. This confirms that fraction F4 is effective at fully oxidizing the organic matrix of these freshwater sediments.

Summary and conclusions

Sequential extraction procedures will continue to serve as important tools for the environmental scientist. These procedures however cannot be employed indiscriminately without an awareness of the limitations imposed by factors such as sediment handling and treatment. In addition, experiments such as ours that are designed to shed light on the selectivity of extraction procedures for specific phases must keep these limitations in mind. Our results emphasize that specific sequential extraction procedures are inherently better designed for the analysis of liquid extracts. The analysis of remaining solid phases introduces additional sources of error, which add to imprecision of the results.

The selectivity of the Tessier procedure for Fe oxyhydroxides indicates that when sediments are handled and treated properly, the procedure can be used effectively. In conjunction with water column and porewater chemistry data, sequential extraction of Fe oxyhydroxides can be employed to gain a more complete understanding of biogeochemical cycles of Fe and other species occurring within sediments. These techniques have recently been used successfully to investigate spatial differences in the biogeochemical cycling of redox-sensitive Fe within sediment profiles (White et al. 1989), and the effects of Fe oxyhydroxides on the distribution and sorption of trace metals within oxic sediment layers (Tessier et al. 1989; Belzile & Tessier 1990; Gubala et al. 1990).

The limitations placed on the use of sequential extractions for the determination of iron sulfides are more restrictive. For example, our results and those of Tessier et al. (1979) indicate that the relative structure (crystalline vs. amorphous) of FeS will determine whether FeS is extracted primarily in fraction F4 or progressively in early extraction steps (e.g. F1—F3).

FeS_2, while more selectively extracted in fraction F4 than FeS, is subject to similar restrictions because Fe-organic matter complexes and organic sulfur complexes are also selectively extracted in fraction F4. However, complementary approaches utilizing sulfur speciation techniques to identify downcore formation of Fe sulfides suggest that Fe extracted in fraction F4 from anaerobic sediment layers may be derived primarily from FeS_2 (White et al. 1989).

Sequential extraction procedures can be used as effective conformational tools when other sediment chemical parameters are measured. Physical characteristics of the sediments must also be considered when using sequential extraction procedures in order to ensure that extractants are added in concentrations high enough to completely solubilize the specific phase of interest. Additional research on a variety of sediment types (e.g. high organic matter content vs. inorganic sediments), as well as on both aerobic and anaerobic sediment layers exhibiting differing redox conditions needs to be conducted to further define the utility and limitations of sequential extraction procedures.

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