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PII: S0025-3227(07)00082-5
DOI: doi: 10.1016/j.margeo.2007.03.006
Reference: MARGO 4032

To appear in: Marine Geology

Received date: 22 September 2006
Revised date: 1 February 2007
Accepted date: 8 March 2007

Please cite this article as: Dezileau, Laurent, Pizarro, Carmen, Rubio, María Angélica, Sequential extraction of iron in marine sediments from the Chilean continental margin, Marine Geology (2007), doi: 10.1016/j.margeo.2007.03.006

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Sequential extraction of iron in marine sediments
from the Chilean continental margin

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Abstract - Iron content of the bulk marine sediment from the Chilean continental margin has been used to infer millenial-scale climate changes in southeast Pacific (Lamy, F. et al. Science; 2000). Changes of iron contents have been related to a varying contribution of Andean (Fe-rich) versus Coastal Range (Fe-poor) source rocks ultimately controlled by continental rainfall changes. However, variations observed could also be produced by changes in the proportions of two components in the sediment – a detrital component with low and constant Fe content and an authigenic phase (Fe oxide, pyrite, apatite, etc.) with high Fe content. A sequential extraction has been used to partition the total iron content in the sediment of core GeoB 7101 at the continental margin of Chile. The extraction steps included five fractions: exchangeable, bound to carbonates, bound to Fe-Mn oxides, bound to organic matter, and residual. Iron appeared to be mainly present in the residual fraction, i.e. in the detrital component. Our study indicates that total Fe or Fe/Al may be used to infer millenial-scales climate changes in the southeastern Pacific.
Keywords: Iron, uranium, Chile, continental margin, sequential extraction, paleoclimate.

Introduction

[1] On the millenial timescale, recent modeling studies indicate that the tropics and the southern high latitudes may play an active role in the initiation of rapid climate changes in the North Atlantic region (Cane et al., 1998; Knorr, and Lohmann, 2003). The understanding of both the origin and consequences of these climatic changes in the tropics and the southern high latitudes are a major challenge of current paleoclimate researchers. For this purpose, marine sediments from the Chilean continental margin have been used to infer millenial-scale changes in southeast Pacific. In order to reconstruct past continental rainfall changes and Patagonian ice sheet extent since the last glacial period, the iron content of the bulk sediment has been measured at subdecadal resolution in three deep sea cores: GeoB 3375 and GeoB 7101 localised at 27°S (Lamy et al., 1998, 2000; Dezileau et al., 2004) and cores GeoB 3313 and ODP 1233 localised at 41°S (Lamy et al., 2001, 2004). This paper concentrates on core GeoB 7101. In the region where the core was taken the continental hinterland is characterized by two main physiographic features, the Coastal Ranges, and the Andes. The Coastal Ranges with elevations generally less than 500 m consist primarily of Mesozoic calc-alkaline plutonic rocks (granites). The Andes reach 6000 m and consist primarily of Plio-Quaternary volcanic rocks, including andesitic and rhyolitic rocks. The Coastal Ranges mainly consist of iron-poor plutonic basement rocks, while the high Andes are dominated by iron-rich andesitic to rhyolitic volcanics.

[2] The paleoclimatic interpretations of the Fe and Fe/Al data on core GeoB 3375 led to the conclusion that the Late Pleistocene continental climate of Chile around latitude
27°S was characterized by alternating relatively arid and humid phases (Lamy et al., 2000). During precession minimum periods at 25°S (5-15 kyrs; 25-35 kyrs; 52-65 kyrs) the region was probably even more arid than today and perennial rivers were absent (Figure 1). For precessional maxima periods (15-25 kyrs; 40-52 kyrs; 65-75 kyrs), Lamy et al., (2000) and Dezileau et al., (2004) have suggested an increase of precipitation in the region, for example due to a northward shift of the Southern Westerlies or an El Niño Southern Oscillations-like mechanism. Precipitation was sufficient to evolve a perennial river system probably including the present dry valleys and led to significant additional sediment and Fe input originating from the northern segment of the Andes (north of 27.5°S). These results are important, and have regional and global paleoclimate implications (Lamy et al., 2000; Dezileau et al., 2004). These paleoclimatic interpretations were based on the assumption that iron was predominantly in the detrital land-derived phase. However, it is also possible that an important part of the iron is in an authigenic phase. In that case, the processes related to paleoclimatic variations have been incorrectly interpreted and need to be rectified. In this study we have determined the various sedimentary Fe-phases so as to assess which phase predominantly controls the observed total-Fe variations.

2. Materials and methods

[3] We have analyzed variations of iron contents through time in the eastern South Pacific, as recorded in sediments from one gravity core (GeoB 7101) retrieved from the continental slope off northern Chile (27°28’S, 71°15’W, 1956 m water depth). Core GeoB7101 was sampled at 10 cm intervals using 10-ml syringes. Two samples series were taken. The first one was designated for uranium analysis (Table 1) and the second one for Fe concentration measurements (Table 1). The dry bulk density of the sediments
was measured by weighting a fixed sample volume before and after freeze-drying. Uranium and thorium concentrations were measured by α-spectrometry following separation on anion exchange column and deposition onto aluminium foil (Dezileau et al., 2000). The analytical uncertainties for \(^{232}\text{Th}\) and \(^{238}\text{U}\) concentrations due to counting statistics were respectively 5% and 6%.

**Estimation of authigenic uranium content**

[4] The total uranium-238 measured in the samples consists of two components: uranium present in detrital minerals (\(^{238}\text{U}_{\text{d}}\)) and authigenic uranium (\(^{238}\text{U}_{\text{auth}}\)) derived from seawater.

[5] The authigenic uranium (\(^{238}\text{U}_{\text{auth}}\)) contents of the samples were calculated using the relation:

\[
^{238}\text{U}_{\text{auth}} = ^{238}\text{U}_{\text{T}} - 0.75 \times (^{232}\text{Th})_s
\]

where \(^{238}\text{U}_{\text{T}}\) is the measured total uranium content and where \((^{232}\text{Th} \times 0.75)_s\) represents the estimate of the mean activity of detrital \(^{238}\text{U}\) in dpm g\(^{-1}\).

**Estimation of iron content**

[6] Lamy et al. (2000) have previously measured the total iron content using a profiling X-ray fluorescence scanner on gravity core GeoB 3375 (same location as the core GeoB 7101). These iron contents were measured at high resolution (1 cm intervals). For the present work, we measured iron by Flamed Atomic Absorption (Perkin Elmer, model 2380) with a low resolution (25 cm intervals) and used an
analytical procedure involving chemical extractions to partition the total iron in different fractions.

[7] Aliquots of 0.100 g of dry sediment were digested using HF and aqua regia (10:1.5) in PARR bombs treated at 110°C during 12 h. The solutions were treated with boric acid (ultra pure grade quality, Merck), diluted to 100 mL, and stored in plastic bottle at 5°C prior to chemical analysis. Concentrations were averaged from the analyses of two replicate samples. The detection limit (DL) was 0.02 ppm corresponding to 20 ppm in the solid phase.

[8] The analytical procedure for the determination of total concentration was checked using certified reference materials of lakes sediments NMCRM (Resource Technology Corporation). Precision was better than 10% (Pizarro et al., 2003).

[9] A 2 g portions of the sediments was treated by the modified Tessier sequential chemical extraction as described below, producing five fractions:

(1) Exchangeable fraction: the sediment was extracted at room temperature for 1 hour with 6.0 mL of 1.0 M MgCl$_2$ (pH=7) during continuous shaking.

(2) Associated with carbonate fraction: the residue from (1) was treated at room temperature with 6.0 mL of 1.0 M NaOAc/HOAc (pH=5) during continuous shaking.

(3) Associated with Fe-Mn Oxides fraction: the residue from (2) was extracted with 15.0 mL of 0.04 M NH$_2$OH.HCl in 25 % acetic acid; the experiments were performed at 96 ±2 ºC during 6 hours continuous shaking.

(4) Associated with organic matter and sulphur fraction: portions of 2.3 mL of 0.02 M HNO$_3$ and 3.8 mL of 30 % H$_2$O$_2$ (pH=2) were added to the residue from (3) and the mixture was heated at 85 ± 2 ºC for 2 hours with occasional shaking; then a second 2.3 mL aliquot of 30 % H$_2$O$_2$ (pH=2) was added and heated at 85 ± 2 ºC for 3 hours with
shaking. After cooling, 3.8 mL of 3.2 M NH$_4$OAc in 20 % HNO$_3$ (v/v) was added and
the sample was diluted to 20.0 mL and shacked continuously for 30 minutes.

(5) Residual fraction: The residue from (4) was digested using HF and aqua requia
(10:1.5) in PARR bomb digestor at 110 ºC during 4 hours. The resultant mixture was
treated with boric acid and diluted to 100.0 mL.

[10] All reagents were Merck Suprapure grade. Double deionized water was used to
prepare the stock solutions. Extractions were carried out directly in polyethylene tubes.
Residues were separated from supernatant by centrifugation (Sorvall Econospin) during
45 min.

3. Results and discussion

3.1 The Fe/Al ratio and authigenic uranium

[11] Lamy et al., (2000) used the Fe/Al ratio in gravity core GeoB 3375 to estimate
past changes in terrigenous sediment provenance (Figure 1). In that study, the authors
interpreted changes in the Fe/Al ratio of the bulk sediment as a terrigenous signal,
which is determined by relative contributions from two source areas, i.e. the Andes and
the Coastal Ranges. Observed variations could also be produced by changes in the ratios
of the two components in the sediment – a detrital component with a low and constant
Fe/Al ratio (0.4) and a variable amount of an authigenic phase (Fe oxide, pyrite, apatite,
etc.) with a high Fe/Al ratio. Such an interpretation is the usual explanation for changes
of iron concentrations in open ocean sediment composition (Chester, 2003).

[12] Past redox conditions may be evaluated using the sedimentary authigenic
uranium content (Klinkhammer, and Palmer, 1991). In oxic pore water, U is present as a
soluble carbonate complex. When conditions become sufficiently reducing to initiate
sulphate reduction, U is reduced to an insoluble form and precipitates. The U added to
the sediment by this process (referred to as authigenic U) accumulates at a rate
dependent on the level of oxygen in bottom water and the flux of organic carbon. The
authigenic U content may therefore be used to constrain past changes in the redox
conditions.

[13] In core GeoB 7101, the authigenic uranium content varies by more than a factor
of 8 (Figure 1). This variation is in antiphase with that of the Fe/Al ratio during the last
100,000 years. Higher values of authigenic uranium reflecting low oxygen
environments are associated to lower values of the Fe/Al ratio and vice versa. The
higher Fe/Al ratios might, therefore, be related to authigenic-Fe-oxhydroxides.
Consequently, variations in sedimentary Fe/Al may be related to climatically modulated
variations in terrigenous provenance or in sedimentary diagenetic conditions.
Determining the Fe-speciation in sediment of core GeoB-7101 off Chile will permit to
discriminate between these two hypotheses.

3.2 Iron content: Total and sequential fractions

[14] The total iron contents in all sediment samples were in the range 22882-34330
ppm expressed as μg g⁻¹ dry sediment (Table 1) with an average value of 28780 ppm
(N=14). Total iron content is similar for the two techniques used (X-ray fluorescence
scanner by Lamy et al., (2001) and Flamed Atomic Absorption (Perkin Elmer, model
2380, this work).

[15] The determination of iron speciation based on the modified Tessier sequential
chemical extraction technique indicates that iron occurs mainly (about 90 wt%, Figure 2
and table 1) in the residual fraction (fraction 5). The iron associated with organic matter
and sulphur fractions (fraction 4) was in the range 206-1572 ppm. The iron associated
with oxides (fraction 3) shows a negative correlation with authigenic uranium, \((r=-0.73, N=11\) and \(p=0.01\), if depth 18 cm is not taken into account, figure not shown). Therefore, this fraction could be a good candidate to explain this variation in antiphase of the iron with the authigenic uranium. However, the iron associated with oxides (fraction 3) was found in the range of 665-1512 ppm and may not explain the ups and downs in Fe/Al ratios. In the exchangeable and carbonate fractions (fractions 1 and 2, respectively) the iron content is < 5.3 ppm.

[16] The residual fraction consists mainly of primary and secondary minerals (Gleyzes et al., 2002). Its destruction is made by digestion with hydrofluoric acid and aqua requia. The iron present in this fraction could be associated to crystalline oxides structures. The hydroxylamine hydrochloride (NH\(_2\)OH.HCl) in acetic acid 25% (fraction 3) mainly removes amorphous iron and aluminium oxides that should correspond to nodules, concretions, cement between particles, or simply as a coating on particles. In both fractions (3 and 5), the iron is not expected to be released in solution over a reasonable time span under the conditions of pH and Eh normally encountered in nature (Tessier et al., 1979). Our results show that iron is principally present in the residual fraction, i.e. in the detrital component (Figure 2). Changes in the iron contents are therefore mainly related to a varying contribution of Andean (Fe-rich) versus Coastal Range (Fe-poor) source rocks ultimately controlled by changes in continental rainfall. These results confirm that iron content can be used to infer millenial-scales climate changes in southeast Pacific.

4. Conclusion

[17] An analytical procedure involving chemical extractions was used to study the distribution of particulate iron into five fractions in the deep sea core GeoB 7101: exchangeable, bound to carbonates, bound to Fe-Mn oxides, bound to organic matter,
and residual iron. The iron speciation indicates that iron was mainly found (about 90 wt%) in the residual fraction (fraction 5), i.e. in the detrital component. The authigenic phase is negligible and may not explain the precessional variation in Fe/Al ratios. Changes in the iron contents are therefore related to a varying contribution of Andean (Fe-rich) versus Coastal Range (Fe-poor) source rocks ultimately controlled by continental rainfall changes. These results confirm the use of Fe to infer millenial-scales climate changes in Chilean continental margin.

Acknowledgements:

We thank the captain and crew of R/V Sonne and Dierk Hebbeln for logistic support. We thank Louis Briqueu for useful comments and discussions and Frank Lamy kindly provided the Fe/Al data of core GeoB 3375. This study was in part supported by FONDECYT (Chile) 1040032 and 1040272 projects.

References


Lamy, F., Hebbeln, D. & Wefer, G., 1999. High resolution marine record of climatic change in mid-latitude Chile during the last 28,000 years based on terrigenous sediment parameters. Quaternary Research, 51: 83-93.


**Figure captions**

**Figure 1.**
Comparison of geochemical data for the last 100,000 years. (a) Terrigenous particle origin can be determined by using the Fe/Al ratio. (b) The authigenic uranium content (dpm/g; see methods). (c)
January insolation calculated at 25°S.

Figure 2.
Comparison between total iron content (Lamy et al., 2000) and residual iron (F5) with depth (cm).

Table 1.
$^{232}$Th and $^{238}$U activities and the five fractions in the deep sea core GeoB 7101: exchangeable, bound to carbonates, bound to Fe-Mn oxides, bound to organic matter, and residual iron (LD=1.1 µg/g).
<table>
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<th>Depth (cm)</th>
<th>Age (kyr)</th>
<th>232Th (dpm/g)</th>
<th>238U (dpm/g)</th>
<th>Iron ppm (Lamy et al., 2001)</th>
<th>Iron ppm This work</th>
<th>F1</th>
<th>F2</th>
<th>F3</th>
<th>F4</th>
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<td>3.85 ± 0.20</td>
<td>28111</td>
<td>34039</td>
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<td>9</td>
<td>1028</td>
<td>492</td>
<td>32529</td>
<td>96</td>
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<tr>
<td>393</td>
<td>92.3</td>
<td>/ ± /</td>
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<tr>
<td>403</td>
<td>95.0</td>
<td>1.77 ± 0.08</td>
<td>2.75 ± 0.13</td>
<td>33803</td>
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<tr>
<td>409</td>
<td>96.4</td>
<td>1.83 ± 0.11</td>
<td>2.45 ± 0.10</td>
<td>31965</td>
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</table>
[The image shows a graph plotting iron content (ppm) against depth (cm). The graph includes two data sets: total iron and iron from the F5 fraction. The y-axis represents iron content ranging from 10,000 to 40,000 ppm, while the x-axis represents depth ranging from 0 to 450 cm.]