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ORIGINAL ARTICLE

Accumulation of potentially toxic elements in sediments in Budi Lagoon, Araucania Region, Chile

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Abstract The concentrations of Cd, Cu, Mn, Ni, Pb, Fe and Zn were determined in superficial sediments extracted from nine zones of Budi Lagoon, located in the Araucanía Region (Chile). The concentrations of these metals were determined by flame atomic absorption spectroscopy and the method was validated using certified reference material (marine sediment). The concentration ranges found for the trace elements were: Pb < 0.5; Cd < 0.2–3.9; Cu 21.8–61.9; Ni 31.2–59.4; Zn 54.5–94.8 $mgkg^{-1}$ (dry weight). The elements that registered the highest concentrations were Mn 285.4–989.8 $mgkg^{-1}$ and Fe 4.8–10.6 %. The lagoon cluster analysis of the stations was divided into three groups (Temo station with high Cu and low Mn concentrations, Bolleco, Comué, Allipén and Deume 3 stations presented highest Cd concentration, and another group Botapulli, Río Budi, Deume 2 and Deume 1 stations presented low levels of Cd). The textural characteristics of the sediment were determined (gravel, sand and mud) and the results were correlated with the concentrations of the metals in the various study zones. The sediments of Budi Lagoon presented high levels of Fe and Mn, which are of natural origin and exceed the maximum values recorded by many authors. With respect to the recorded concentrations for Cd, Cu, Ni and Zn, are within the ranges published by other authors in similar works. The Pb element was not detected. The results were subjected to statistical analysis to evaluate the correlations between the content of the elements and obtain the site of sediment.

Keywords Budi Lagoon · Sediments · Toxic metals · Atomic absorption spectrometry

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Introduction

In recent decades, the scientific community has paid increasing attention to detect the presence of certain potentially toxic elements in different kinds of matrices: water (continental and marine), sediments, aquatic organisms (fish, molluscs, algae) and foods in general (De Gregori et al. 1992; Marcovecchio 2004; Franc et al. 2005; Yilmaz et al. 2007; Samir et al. 2008; El-Hasan and Jiries 2001 Tapia et al. 2010, 2012). The reason behind this great interest in finding out the concentration of certain metals, even at trace level, in the various kinds of matrices, is due basically to the fact that over the years the degree of toxicity of certain metals for human beings has been discovered. In some cases, the chemical feature in which they are toxic or harmful for human health has been revealed, as well as the role which they act in certain enzyme processes (Jennett et al. 1980; Leonard and Lauwerys 1980; Langard and Vigander 1982; Nriega and Nieboer 1988; Páez-Ozuna 1996; Baird 2001; Barquero-Quirós et al. 2001; Bradl et al. 2005; El-Hasan et al. 2011; González et al. 2012).

The metals, unlike the majority of organic compounds, do not degrade either biologically or chemically in nature (Chiang 1988). They persist in the environment, and because of their stability may be transferred over

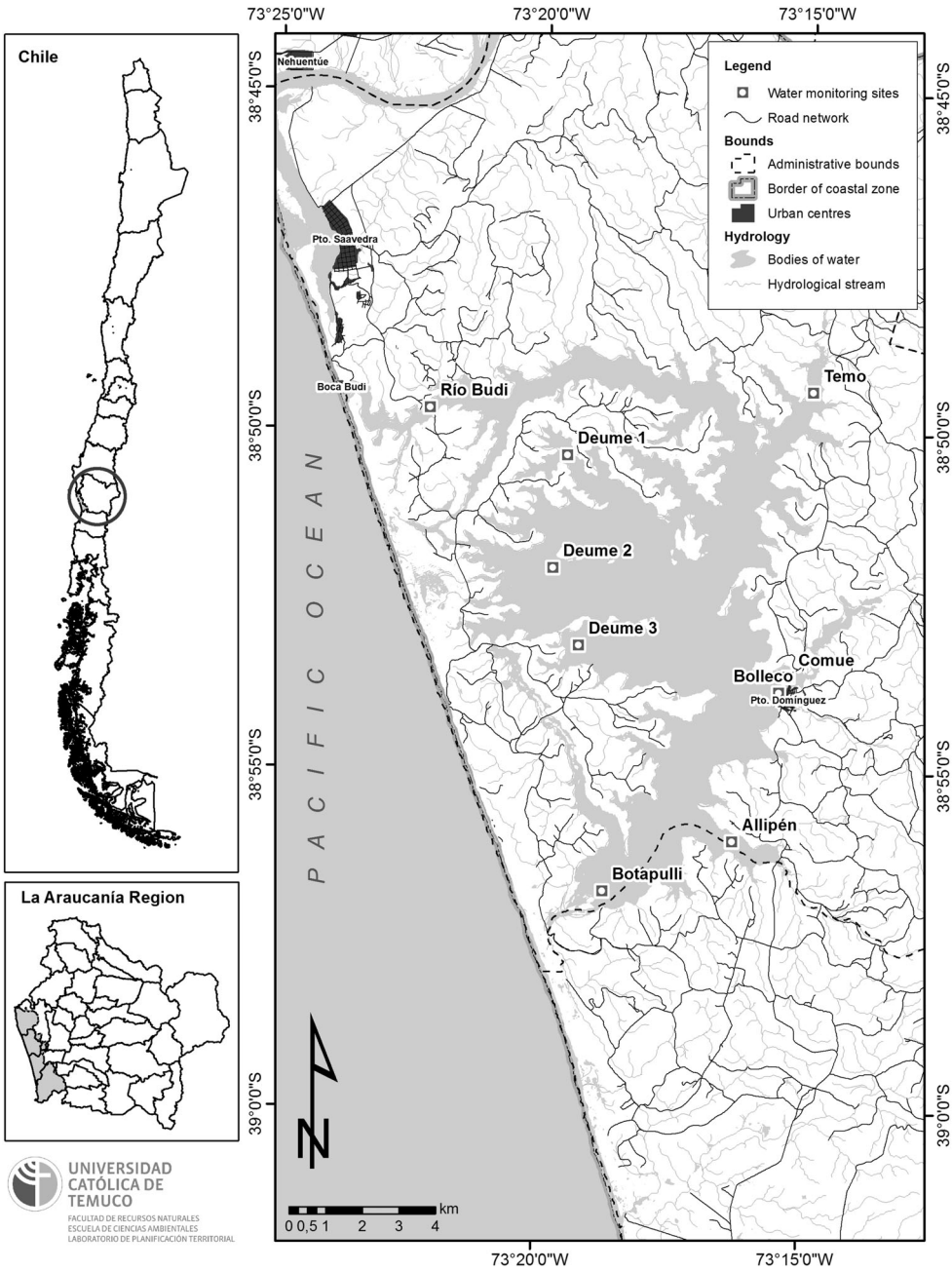

Fig. 1 Map of Budi Lagoon, location of study area and subtidal sampling stations. The *black circles* indicate the location of sampling stations in Budi Lagoon

considerable distances; therefore, they accumulate in environmental systems, and for the final sinks the metals are soils, and continental and marine sediments (Chiang 1988; Gobert et al. 1992; Sivaperumal et al. 2007).

The incorporation of potentially toxic elements into aquatic ecosystems (e.g. lagoons, rivers and estuaries) may occur through the erosion of geological strata, atmospheric deposit or human sources such as industrial, agricultural or domestic waste (Förstner and Wittman 1981; Eisler 1988; Nimmo et al. 1998; Alam et al. 2002). Metals of anthropogenic origin introduced into aquatic media are generally present in ionic or particulate forms, and are then incorporated into organic–metallic compounds or some mineral phases. They subsequently become part of the suspended matter transported in the water column, and finally decant into sediments (Kabata-Pendias and Pendias 2000).

The Current European Union regulations (EC 2006) consider three metals to be dangerous for human beings: Pb, Cd and Hg; while (USFDA 1993a, b, c) also includes Cr, As and Ni. From the point of view of contamination with potentially toxic elements, Cr and Ni are good indicators for industrial contamination. The presence of Pb, Cu and Zn are generally good indicators for a variety of human

activities, domestic, agricultural or industrial (Salomons and Förstner 1984; Baisch 1988). Ni, Ag, Cd, Hg and Pb are considered to be toxic elements (Förstner and Wittman 1979); the most critical in terms of their presence in sediments are Hg and Cd. The metals that depend on the redox conditions of the sediment are Fe and Mn. The elements that normally present high concentrations in sediments are Al, Fe and Mn, up to percentage level. They are not considered to be an indicator for contamination and their possible variations are usually related to mineralogical changes (Kouadio and Trefry 1987). As a rule, contaminating metals tend to adhere to the fine particles in aquatic sediments, due to their greater relative surface area (Herut and Sandler 2006).

Budi Lagoon is one of the few Chilean coastal lagoons to present the influence of sea water. Its northern limit lies approximately 1 mile south of the Imperial River estuary, close to the mouth of the Budi River (Araucanía Region, Chile) which connects it sporadically with the sea (Stuardo and Valdovinos 1989). The area of the Budi coastal lagoon is 57.4 km^2. Signs can be seen around the lagoon of high anthropogenic intervention in recent years; the banks are covered with vegetation in some places and exposed in others, and there are sandy-muddy beaches. Sediments have high content of organic material of aquatic plant origin (Bertrán et al. 2006, 2010).

The objectives of this research were to determine the Cd, Cu, Fe, Mn, Ni, Pb and Zn content in sediments taken from nine stations around Budi Lagoon, and to correlate their concentrations with the type of sediment and the human activity carried on around the lagoon.

Materials and methods

Study area

The Budi Lake is located in the Araucanía Region, Chile (38°49′30″S, 73°23′30″W). On the basis of the characteristics of Budi Lagoon and the contributions of its principal tributaries (Budi, Temo, Allipén, Comué, Bolleco and Botapulli Rivers), nine sampling stations were established: allocated as shown in Fig. 1.

Sediment sampling

The samples were collected in January 2004 (summer). The sediments were extracted using a PVC core of (0.010 m^2), with five replications ($n = 5$) in each station. The sediment samples were identified and placed in plastic bags for subsequent laboratory analysis. At each sampling station was recorded pH and electrical conductivity (EC) of its surrounding waters using a pH-meter WTW model 330i. The measurement of these parameters was to allow possible intrusion of sea water into the lagoon to be predicted.

Chemical analysis

The sediment samples were dried at 105 °C, grinding, sieving and about (1.0 gm) of the most fine dried grains were digested with a mixture of conc. HF-HNO_3 (2:1) as the method described in (Page 1982) and preserved in a refrigerator till analysis. The analyses were done in duplicate with a control solution for each.

The reagents used were of high purity (Suprapur, Merck, Darmstadt, Germany). The standard solutions for the various metals were prepared from concentrated solutions of the metals of 1,000 mg L^{-1}, (Fisher Scientific International Company). Cleanliness of the material was fundamental to guarantee optimum results in analysis.

The metal measurements were done by Flame Atomic Absorption Spectroscopy (air/acetylene), using a Unicam spectrophotometer mod. 969 with deuterium background corrector for Cd and Pb.

Validation of the method

The method of analysis was validated using certified reference material MESS-1 (marine sediment), supplied by the National Research Council, Canada, NRC, Division of Chemistry.

Table 1 shows the results for the measurements of Cd, Cu, Pb, Ni, Mn, Zn and Fe in the certified reference material MESS–1 (marine sediment). The replications in the reference material presented good accuracy, with relative errors varying between −11.9 % (Cd) and +5.9 % (Zn) and recovery percentages from 88.1 % (Cd) to 105.9 % (Zn).

Table 1 Concentrations of cadmium, copper, lead, nickel, iron, manganese and zinc in certified reference material MESS-1 (marine sediment reference materials for trace elements and other constituents), from National Research Council Canada

Element	Certified concentration[a]	Observed concentration[b]	Rel. error (%)[c]	Recovery (%)
Cd	0.59 ± 0.10	0.52 ± 0.2	−11.9	88.1
Cu	25.1 ± 3.8	25.9 ± 2.4	+3.2	103.2
Pb	34.0 ± 6.1	35.8 ± 7.5	+5.3	105.3
Ni	29.5 ± 2.7	26.9 ± 4.4	−8.8	91.2
Mn	513 ± 25	496.1 ± 18	−3.3	96.7
Zn	191 ± 17	202.2 ± 19	+5.9	105.9
Fe_2O_3	4.36 ± 0.25[d]	4.04 ± 0.88[d]	−7.3	92.7

[a] The concentrations are expressed in mg kg^{-1}

[b] Mean and standard deviation of three independent measurements

[c] Accuracy of data, expressed as relative error

[d] Result expressed as % of Fe_2O_3

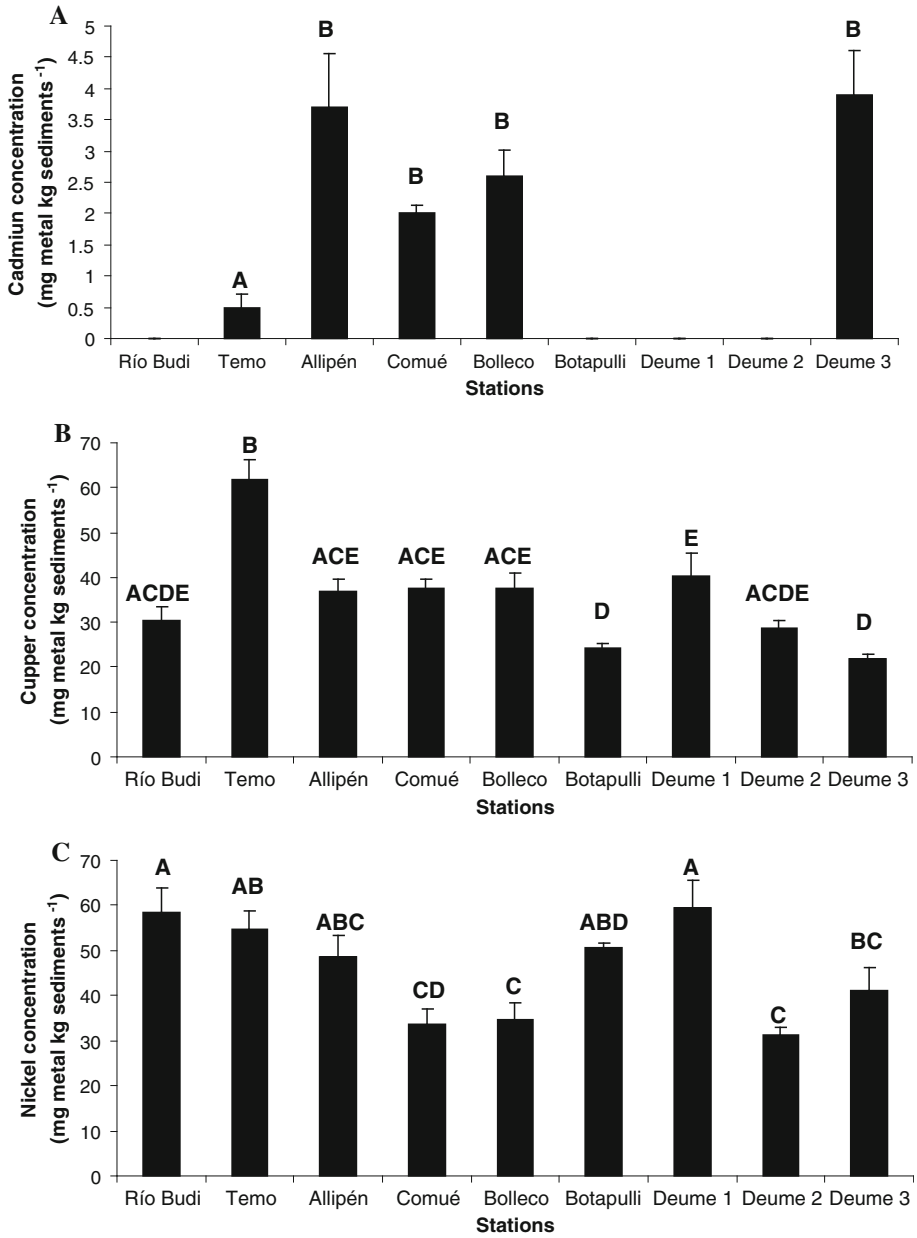

Fig. 2 Concentrations of metals **a** Cd, **b** Cu, **c** Ni (mg of metal kg of sediments^{-1}) from sediments of Budi Lagoon. Data are shown as mean ±SD. *Different letter* indicated significant differences between groups ($P < 0.05$, one-way ANOVA Tukey's test)

Statistical analysis

Once the assumptions of normality, independence and homoscedasticity had been verified (Kolmogorov–Smirnov, Lilliefors and Bartlett tests, respectively), an one-way analysis of variance (ANOVA) was carried out in which the metal concentrations (Cd, Cu, Ni, Mn, Fe and Zn) and the sampling stations were compared. A confidence level of 95 % was used to define the significance values.

In addition, the stations were compared considering the averages of the replications of the chemical data for the habitat (Cd, Cu, Ni, Mn, Fe and Zn), using ordering and classification (cluster analysis) techniques with PRIMER V.6 software (Clarke and Warwick 1994). First, the data were transformed with $\log_{10}(x + 1)$; subsequently a Euclidian distance matrix was constructed, which was grouped using the UPGMA grouping method.

Results and discussion

Sedimentological characteristics

In accordance with the results, the highest percentage of gravel was recorded at the Comué station with 9.05 %; the highest percentage of sand was at Deume 2, with 82.68 %, and of mud at Allipén with 89.61 %. Mud is the highest represented fraction in the whole lagoon, and gravels the

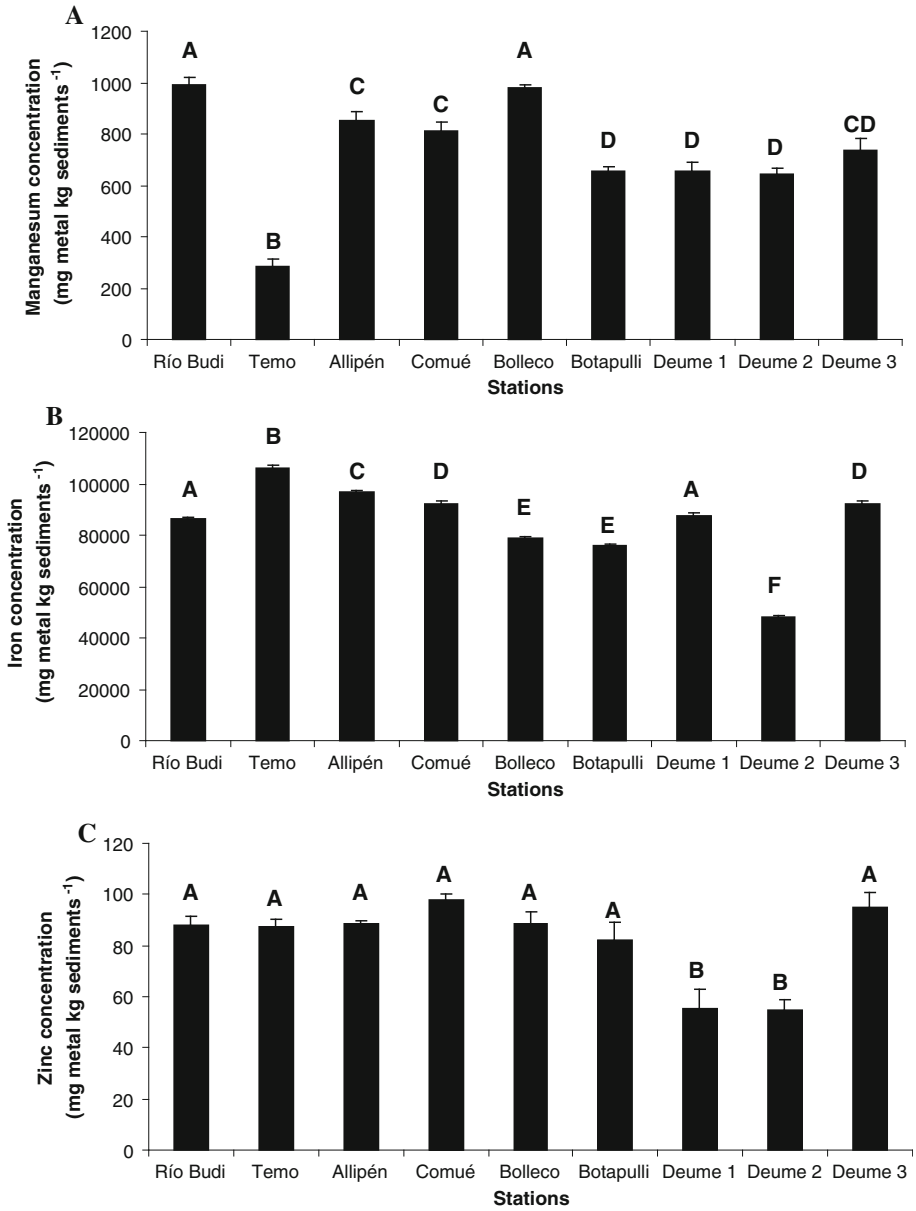

Fig. 3 Concentrations of metals **a** Mn, **b** Fe, **c** Zn (mg of metal kg of sediments^{-1}) from sediments of Budi Lagoon. Data are shown as mean ± SD. *Different letter* indicated significant differences between groups ($P < 0.05$, one-way ANOVA Tukey's test)

lowest. The highest percentages of organic matter were found in the mud fraction at the Allipén and Temo stations, with 87.35 and 84.29 %, respectively. According to Bertrán et al. (2010), the gravel, sand and mud fractions are all represented in Budi Lagoon, but in different percentages depending on the location of the stations.

Potentially toxic elements in sediments

Figures 2 and 3 show the concentrations of Cd, Cu, Ni, Mn, Fe and Zn in sediments from nine sampling station of the lagoon: Río Budi, Temo, Allipén, Comué, Bolleco, Botapulli, Deume 1, Deume 2 and Deume 3. The Pb element was not detected (<0.5 mg kg^{-1}). Under cluster analysis of the stations, the lagoon was divided into three groups. The first to separate was the Temo station with high Cu and low Mn concentrations (minimum Euclidian distance 0.39), and the other two groups differentiated mainly for Cd concentration, where Bolleco, Comué, Allipén and Deume 3 stations presented the highest concentrations and another group presented low levels of Cd (Botapulli, Río Budi, Deume 2 and Deume 1 stations) (Fig. 4).

According to the results obtained, the points presenting the lowest conductivity are Temo, Bolleco and Comué with values ranging between 0.17 and 2.77 mS cm^{-1}, and pH between 6.87 and 7.99. On the other hand, the Río Budi, Deume 3, Deume 2, Allipén, Botapulli and Deume 1 stations present high conductivity with values between 7.06

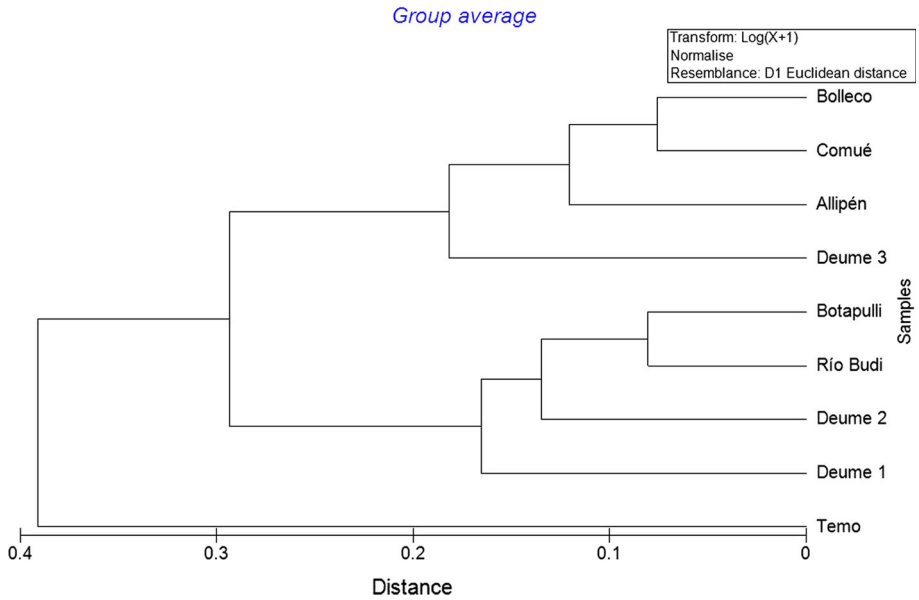

Fig. 4 Cluster analyses of sediments, per stations of Budi Lagoon. Euclidean distance matrix, which was grouped using UPGMA clustering method

Table 2 pH and conductivity values of the waters surrounding sediments taken from the sampling stations of Budi Lagoon, Araucanía Region, Chile

Parameters	Stations								
	Río Budi	Temo	Allipén	Comué	Bolleco	Botapulli	Deume 1	Deume 2	Deume 3
pH	8.97	6.87	8.84	7.99	7.30	8.65	8.55	9.28	8.82
Conductivity (mS cm^{-1})	7.06	0.17	6.23	2.77	1.11	6.16	4.47	6.39	6.45

and 4.47 mS cm^{-1}, and pH between 8.55 and 9.28, indicating that there is a greater influence from the intrusion of sea water at these points (Fig. 1 and Table 2) (Bertrán et al. 2006, 2010).

The results obtained for metal levels clearly indicate that the order of concentrations at all the stations was: Fe > Mn > Zn > Ni > Cu > Cd > Pb. This agrees with the reports of other authors (Mendil and Uluozlu 2007; Romano et al. 2012) in similar environments (Table 3).

The concentrations of the metals at the various sampling points are similar to those recorded in other places: Venice Lagoon Porto Marghera, Italy (Bellucci et al. 2002), Sakumo and Kpeshie Lagoons, Ghana(Narayana and Priju 2007), Coastal Lagoons in Central Vietnam (Romano et al. 2012), etc. (Table 3), However, it should be noted that much higher concentrations of Mn and Fe were recorded in Budi Lagoon than in other places (Maanan et al. 2004; Samir et al. 2008), with values for Mn ranging between 285 and 989 mg kg^{-1}, and for Fe between 48,186.1 and 105,883.5 mg kg^{-1} (Fig. 3). The concentrations recorded for Cd, Cu, Ni and Zn were very similar to those reported by Rigollet et al. (2004), (Narayana and Priju 2007) and Klake et al. (2012). The Pb element was not detected in any of the sampling stations (<0.5 mg kg^{-1}).

Moreover, there was little variation between the sampling stations in the concentrations of Zn, Ni, Cu and Cd, while the greatest differences were found for Fe and Mn. The highest value for Fe was found at the Temo station, 105,883.5 mg kg^{-1}, when compared with 48,186.1 mg kg^{-1} at Deume 2. The highest value for Mn was recorded at Río Budi with 989.8 mg kg^{-1}, and the lowest at Temo with 285.4 mg kg^{-1}. The high concentration of Fe and Mn present in the sediments may be attributed to that both elements are essential components of clay minerals, which are among the principal soil constituents in coastal lagoons and lake (Carrol 1958; Hamed 1998).

The highest concentrations of metals tend to be associated with fine sedimentary particles, in which case the highest levels of metals would be concentrated in mud. This agrees with the findings of Bertrán et al. (2010) that the Temo and Allipén stations present the highest percentages of mud (87 and 89 %, respectively) and organic matter (84 and 87 %, respectively), due to their larger relative area and the characteristic composition of fine particles. Both phyllosilicates and organic matter have a chemical affinity for oligo-elements and organic contaminants, and are concentrated in mud (loam clay). The majority of other minerals, including feldspars and heavy

Table 3 Selected references of metal concentrations (mg kg^{-1} d.w.) from sediments from different geographical areas (mean values and ranges)

Sites	Cd	Cu	Pb	Ni	Zn	Mn	Fe	References
Budi Lagoon, Araucanía Region, Chile	n.d.–3.9	21.8–61.9	n.d	31.2–59.4	54.5–94.8	285.0–989.0	8,186.0–105,883.0	Present study
Lakes in Tokat, Turkey	–	3.7–8.2	3.1–7.0	38.0–55.4	23.9–38.9	76.7–232.0	1,718.0–2,138.0	(Mendil and Uluozlu 2007)
Lakes in Egypt: Edku	1.5	36.80	37.10	–	344.5	1,390.0	6,250.0	Samir et al. (2008)
Boroller	4.6	47.50	13.10	–	217.0	850.0	11,000.0	Samir et al. (2008)
Manzala	84.8	315.40	134.60	–	432.0	419.0	33,390.0	Samir et al. (2008)
Coastal Lagoons in Central Vietnam	0.03–0.23	6.58–28.60	3.70–33.80	3.7–64.6	31.0–78.0	142.0–1,499.0	13,332.0–77,579.0	Romano et al. (2012)
Vembanad Lake, India	1.0–16.0	8.0–50.0	–	30.0–118.0	36.0–858.0	–	–	(Narayana and Priju 2007)
Lagoon in Ghana: Sakumo	n.d	42.9–84.7	6.5–54.0	–	46.4–81.0	280.0–655.0	–	Klake et al. (2012)
Kpeshie	n.d	20.8–52.0	n.d–54.2	–	4.6–85.3	41.0–123.0	–	Klake et al. (2012)
Venice Lagoon, Porto Marghera, Italy	0.2–5.0	–	38.0–114.0	–	101.0–1,115.0	–	–	(Bellucci et al. 2002)
Sidi Moussa lagoon Atlantic Moroccan Coast	–	20.0–42.0	–	18.0–43.0	19.0–73.0	–	11,000.0–45,000.0	Maanan et al. (2004)
Thau lagoon. France	0.21–0.47	12.2–27.2	8.2–20.9	4.91–14.60	23.8–51.5	154.0–218.0	4,613.0–8,058.0	Rigollet et al. (2004)
Venice lagoon, Italy	0.20–0.94	4.4–21.7	5.2–7.7	10.2–15.0	48.3–95.7	313.0–355.0	12,646.0–16,128.0	Rigollet et al. (2004)

minerals, are found in the coarse loam fractions, while the sand fraction is composed mainly of carbonate minerals and/or silica (Herut and Sandler 2006).

Conclusion

According to the results obtained, it may be concluded that the concentrations of Fe and Mn in samples of sediment collected in different areas of Lake Budi, Araucanía Region (Chile) are greater than those recorded in the literature in sediment coming from different geographical areas. High concentrations of Fe and Mn in the sediments which are attributable to these elements are essential components of clay minerals that are present in soils of coastal lakes.

The concentration levels of Cd, Cu, Ni and Zn detected in the sediments of Lake Budi are similar to those reported in the literature and their limited presence is naturally occurring and not attributable to human activity. The Pb element was not detected in the sediments of Lake Budi (<0.5 mg kg^{-1}).

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