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The use of quantitative mineralogy linked to palynological studies in palaeoenvironmental reconstruction: the case study of the "Lagoa Campestre" lake, Salitre, Minas Gerais, Brazil

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Abstract Comparison between the minerals (determined by FTIR spectroscopy) and pollen content of lacustrine sediments from the Salitre area (Brazil) shows that the variations of the mineral content are indicators of regional or local environmental changes. An opposite trend between the forest cover and the detrital content of sediments is observed during the Pleistocene lacustrine episode. FTIR Spectroscopy allows the detection of a sedimentary hiatus outlined by a large change in the mineral content; it also emphasizes the abrupt nature of some changes of the vegetation cover which, without adequate datings, could have been misinterpreted as a sedimentary hiatus.

Keywords: Quantitative Mineralogy, Late Quaternary, Palaeoenvironment, Pollen, Lacustrine Sediments, Brazil.

Résumé **Utilisation de la minéralogie quantitative couplée à l'analyse palynologique dans les reconstructions paléoenvironnementales : l'exemple du lac « Lagoa Campestre » de Salitre, Minas Gerais, Brésil**

La comparaison des contenus minéraux (déterminés par spectroscopie IRTF) et polliniques de sédiments lacustres de la région de Salitre (Brésil) montre que les variations des teneurs en phases minérales répercutent des changements régionaux ou locaux de l'environnement. Une évolution inverse entre couvert forestier et charge détritique des sédiments est observée pendant la phase lacustre pleistocène. La méthode utilisée permet la détection d'un hiatus sédimentaire marqué par un changement brutal du contenu minéral, ainsi que l'évaluation du caractère abrupt de certains changements du couvert végétal qui, en l'absence de datation, pourraient être confondus avec un hiatus sédimentaire.

Mots-clés : Minéralogie quantitative, Quaternaire récent, Paléoenvironnement, Pollen, Sédiments lacustres, Brésil.

Version française abrégée

L'ANALYSE minérale quantitative par spectroscopie infrarouge à transformée de Fourier (IRTF) a été appliquée à l'étude de 53 échantillons prélevés sur une carotte de sondage de 6 m de long extraite des sédiments de la « Lagoa Campestre » de Salitre (Brésil) (fig. 1). Seules ont été dosées les phases essentielles : quartz, kaolinite, gibbsite et anatase, ainsi que la silice amorphe essentiellement exprimée sous forme de phytolithes. Comparée à d'autres méthodes telles que la DRX ou l'analyse de la susceptibilité magnétique, l'IRTF s'est avérée une méthode de choix pour ce genre de sédiments riches en matières

Note

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organiques (**fig. 2**) (Bertaux *et al.*, 1995). Antérieurement et sur cette même carotte, des analyses polliniques avaient été effectuées sur des échantillons collectés au pas de 2 ou 4 cm entre 15 et 575 cm, ainsi que 14 datations radiocarbone donnant des âges s'étageant entre 3 060 BP, à 17 cm, et 32 030 BP à 200 cm (Ledru, 1993).

D'une façon générale, la quantification ainsi réalisée montre que :

– Deux séquences sédimentaires, I et II (**fig. 2**), séparées par un hiatus à 164 cm (28 740-16 800 BP) se succèdent dans le dépôt. Chacune débute par une sédimentation organo-minérale à la base (Ia et IIa, **fig. 2**) et se termine par des dépôts purement organiques (Ib) ou presque (IIb) au sommet.

– Les phases identifiées sont identiques tout au long de la carotte, mais présentent des proportions relatives variables. Ces phases sont présentes dans les sols ferrallitiques du bassin versant, ce qui témoigne de leur caractère détritique.

1) LA SÉDIMENTATION DU PLEISTOCÈNE TARDIF

Durant cette période, on observe deux épisodes où les algues (*Pediastrum, Botryococcus*, Ledru *et al.*, 1995), indicatrices de l'environnement lacustre local, sont abondantes (**fig. 2**). Le premier, entre 550 et 480 cm, est caractérisé par une faible teneur en pollens d'arbre. Le second, entre 320 et 210 cm, se produit dans une période de fort développement de la forêt. Or, dans chacun de ces deux cas, les apports détritiques évoluent en sens inverse des teneurs en pollens d'arbre. Ceci montre que la teneur en phases minérales du sédiment est un indicateur de l'environnement régional : quand le couvert végétal est faible, l'érosion et le transport des minéraux des sols environnants vers la dépression sont favorisés. Ce type de dynamique sédimentaire, bien connu en milieu tempéré, est donc ici mis en évidence dans un contexte tropical où la composante organique du sédiment est très importante. Cette dynamique concerne également les phytolithes qui, durant le Pleistocène tardif, doivent donc être considérées comme des composants détritiques.

Un changement abrupt de végétation est mis en évidence dans la zone Ib (**fig. 2**) : les algues disparaissent et la forêt semi-décidue régresse au profit d'un développement de Myrtaceae. Sur la base des caractéristiques sédimentologiques et spectroscopiques (absence de minéraux et identité de composition des constituants organiques, **fig. 4**) du sédiment en dessous et au-dessus de ce changement, l'hypothèse d'une variation régionale de l'environnement, sans hiatus sédimentaire, est la plus plausible.

Le hiatus sédimentaire révélé par les âges ^{14}C (28 740 ans BP à 170 cm et 16 800 ans BP à 157 cm), marquant la limite entre les deux séquences sédimentaires (zones Ib et IIa), est très clairement souligné par la brutale augmentation des teneurs en phases minérales, y compris les phytolithes, alors qu'il est peu lisible sur l'analyse palynologique.

2) LA SÉDIMENTATION HOLOCÈNE

Cette période (niveau IIb) est marquée par l'absence de minéraux détritiques. Les phytolithes, seule phase minérale présente, évoluent parallèlement aux pollens d'arbre (**fig. 2**), donc à l'opposé de ce qui se passe au Pleistocène tardif. Une diminution de la fréquence des pollens d'arbre vers 5 500-4 500 ans BP, due à une augmentation de la saison sèche (Ledru, 1993), n'est pas accompagnée d'un retour des détritiques. Ces observations suggèrent que la dépression ne fonctionne plus comme un milieu lacustre et que le milieu palustre actuel s'est mis en place au début de l'Holocène.

En conclusion, l'étude des sédiments de la carotte LC3 montre que l'analyse minéralogique quantitative par spectroscopie IRTF est un bon outil pour compléter l'interprétation des informations sur les paléo-environnements archivées dans les sédiments lacustres. Dans le cas du site de Salitre, cette méthode :

1) améliore la détection de changements régionaux du couvert végétal, lorsqu'ils induisent des variations dans le transport et la sédimentation des phases détritiques ;

2) permet la détection d'un hiatus sédimentaire marqué par le changement brutal du contenu minéral ;

3) permet d'évaluer le caractère abrupt de certains changements du couvert végétal qui, en l'absence de datations, ne doivent pas être confondus avec un hiatus sédimentaire.

Outre son intérêt comme outil de corrélation en sédimentologie, la minéralogie quantitative s'avère être une méthode utile pour affiner les interprétations paléo-environnementales bâties à partir des données palynologiques.

INTRODUCTION

In the study of lacustrine sediments, semi-quantitative mineralogical data are usually obtained from X-ray diffraction analyses of clay fractions (Tiercelin *et al.*, 1987) or bulk sediments (Dean, 1993), or from measurements of bulk magnetic properties (magnetic susceptibility or anhysteretic remanent magnetization (Sprowl and Banerjee, 1993)). Such methods give only rough estimates of crystallized-mineral content. Moreover, conventional sedimentological tools such as grain size studies are difficult to apply on recent lacustrine sediments because the fine mineral fraction is usually mixed with an abundant organic fraction.

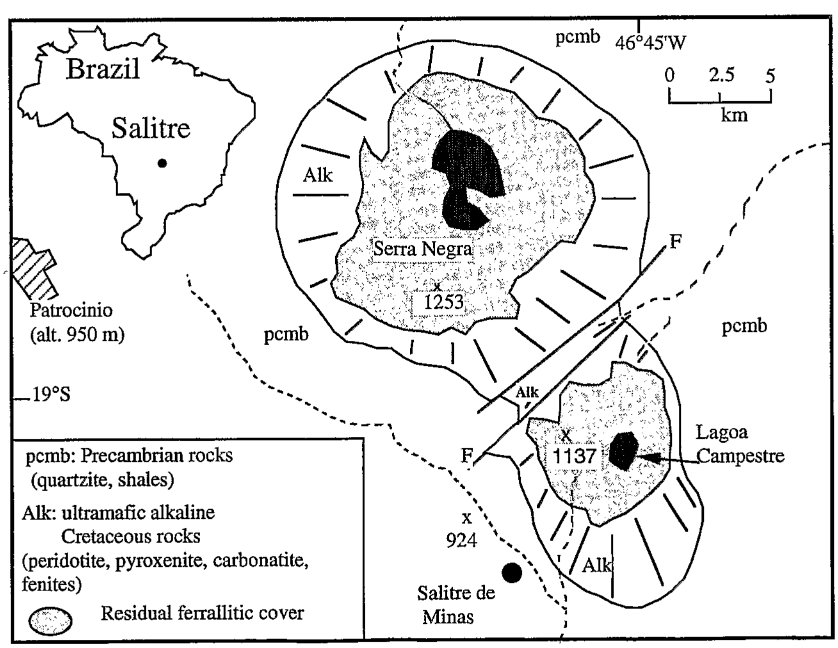
Fourier transform infra-red (FTIR) spectroscopy has been demonstrated to be a truly quantitative method in mineralogy (Fröhlich, 1981, 1989; Herbert *et al.*, 1992), and a first application on lacustrine sediment was made at Carajas (Brazil) by Sifeddine *et al.* (1994). The results were very promising, and yielded a record of mineralogical signals along each core; such data can be compared to the fluctuations of other biological (pollen, diatoms, etc.) or geochemical (Sondag *et al.*, 1993) markers. The recent development of FTIR spectroscopy provides a new method to obtain quantitative data on organic-rich sediments containing amorphous silica (Bertaux *et al.*, 1995), and permits a comparison with fluctuations of tree pollen recorded in a core from Brazil. Our aim was to improve the understanding of Quaternary palaeoenvironmental changes in this area.

MATERIALS AND METHOD

The Salitre ultramafic-alkaline complex (West of Minas Gerais State, Brazil) has a central, doline-shaped, depression (Lagoa Campestre) partially filled with lacustrine and palustrine sediments (**fig. 1**, Sondag *et al.*, 1993). A thick, loose latosolic cover (hematite, gœthite, gibbsite, kaolinite, magnetite, maghemite, quartz, anatase, crandallite group phosphates) surrounds the depression, overlying a thick saprolite (50-100 m). Due to the particular mineralogy of the bedrock, where perovskite, calzirtite and apatite are often essential minerals, these alterites have high contents in TiO_2 (up to 30%), REE (up to 5% ΣR_2O_3), ZrO_2 (up to 1%) and Nb_2O_5 (up to 1%). A 6-m depth core (LC3) was drilled in this depression. Fourteen radiocarbon dates were obtained on separated organic fragments (wood and vegetal fibres) in the upper part of the core; the oldest indicates an age of 32,000 a BP at 210 cm depth (Ledru, 1993). A high organic matter content characterizes most of the core, as indicated by high LOI and C contents (**fig. 2**). The mineral fraction consists of a mixture of quartz, amorphous

Fig. 1 Location, geomorphology and geology of the Salitre area, Brazil.

Localisation, géomorphologie et géologie de la région de Salitre, Brésil.



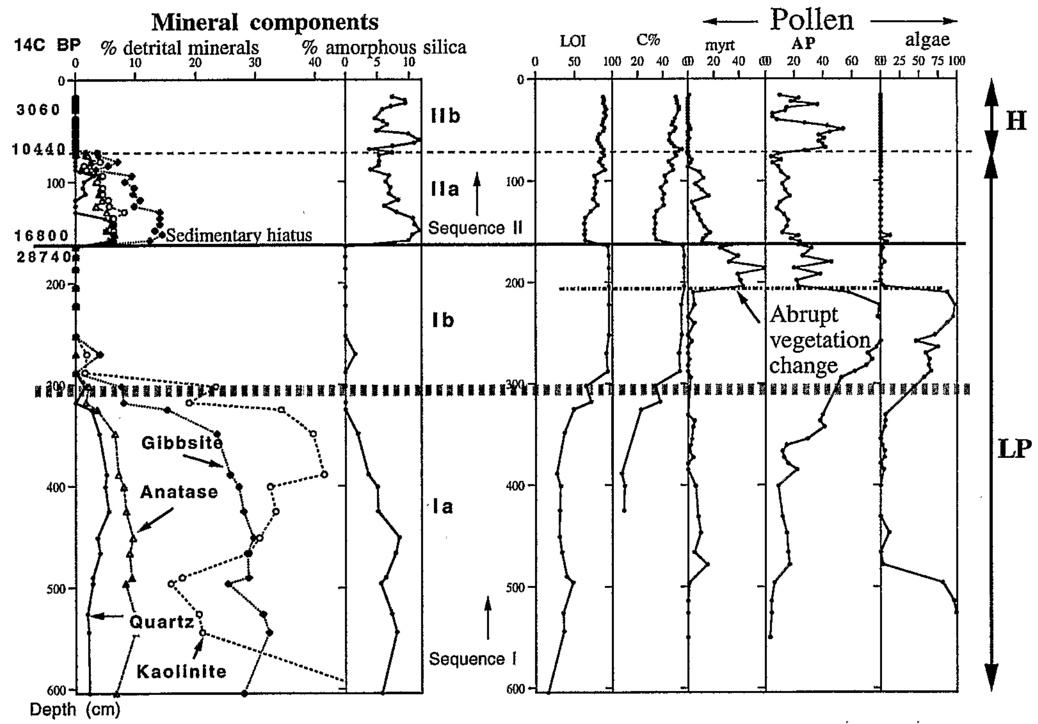


Fig. 2 Variations of mineral and pollen contents along the LC3 core.
H = Holocene; LP = Late Pleistocene;
AP = arboreal pollen except Myrtaceae.

Variations des teneurs en minéraux et pollens le long de la carotte LC3.
H = Holocène ; LP = Pleistocène tardif ;
AP = pollens d'arbre sauf Myrtaceae.

silica (phytoliths and sometimes sponge spicules), gibbsite, kaolinite, anatase (weathered perovskite), a minor amount of phosphates of the crandallite group, and scarce heavy minerals (ilmenite, calzirtite, zircon, baddeleyite, barite). The low iron content of the sediments despite the ferralitic environment can be explained by the strong solubilization of the iron-bearing phases observed in the reducing conditions of the peat.

The quantitative determination by FTIR spectroscopy of the main constituents (quartz, amorphous silica, kaolinite, gibbsite, anatase) of the LC3 sediment was performed following the method described by Bertaux *et al.* (1995). 53 samples were analyzed using the KBr-disc method. Figure 3 presents the fit between the FTIR quantitative determinations and the chemical analysis. One can see the very good agreement between the chemically and the FTIR determined SiO_2 (SiO_2 quartz + SiO_2 amorphous silica + SiO_2 kaolinite). For TiO_2 and Al_2O_3, the agreement is also quite good. The slight discrepancies observed for TiO_2 can be explained by the low definition of the IR spectral signature of anatase. For Al_2O_3 the small discrepancies can be attributed to contamination from the aluminium coring tube, enhancing the chemical signal, and to difference in crystallinity between the natural gibbsite and the synthetic one used for calibration.

Pollen analysis of the LC3 core was performed on samples collected every 2 or 4 cm between 15 and 575 cm depth, depending on the sediment characteristics (Ledru, 1993; Ledru *et al.*, 1995). The sediment contains no pollen below 575 cm.

RESULTS AND DISCUSSION

The overall variations of the mineral content of the salitre LC3 core (**fig. 2**) show that:

– qualitatively, the mineral fraction remains identical throughout the core. The main phases are kaolinite, quartz, gibbsite, anatase and amorphous silica. The variations are due to changes in the relative proportion of the mineral phases.

– Two main sedimentary sequences are identified, both characterized by the presence of detrital minerals at their base, and of a pure (sequence Ib) or almost pure (sequence IIb) organic sediment at their top.

The comparison between mineralogical and palynological data will be used here as a clue to understanding the link between the environmental changes and the resulting mineral composition of the sediments.

1) Late Pleistocene sedimentation

All the mineral phases present the same pattern of variations between 70 and 600 cm and are present in the soils surrounding the depression.

Mineral phases are indicators of regional environmental variations

As indicated by the palynological data in figure 2, two episodes are characterized by the presence of algae (*Pediastrum, Botryococcus*, Ledru *et al.*, 1995) in the depression, at depths between 550 and 480 cm and between 320 and 210 cm respectively. Algae are indicators of the local environment during sedimentation. They demonstrate that a sufficient water level was present to allow their growth. During the first algae episode (level Ia), pollen indicate that no forest was present around the Salitre depression, and we observe that detrital minerals were transported to the sedimentation area. During the second alga episode (in level Ib), a large amount of arboreal pollen indicates that a semideciduous forest covered the region, and there was no detrital flux to the depression. This clearly illustrates that the mineral phases of the sediment are regional environmental indicators. As shown in figure 2, this opposite trend between arboreal pollen and detrital minerals is observed all along the core: in levels Ia and IIa, a low content in arboreal pollen corresponds to the presence of detrital minerals; in levels Ib and IIb, the highest forest development is observed together with absence of detrital minerals in the sediment. This kind of sedimentary dynamics, where a weaker vegetation cover results in stronger erosion and transport of minerals from soils, which is well known in temperate countries, is shown here in a tropical context where the organic sedimentation is by far more important.

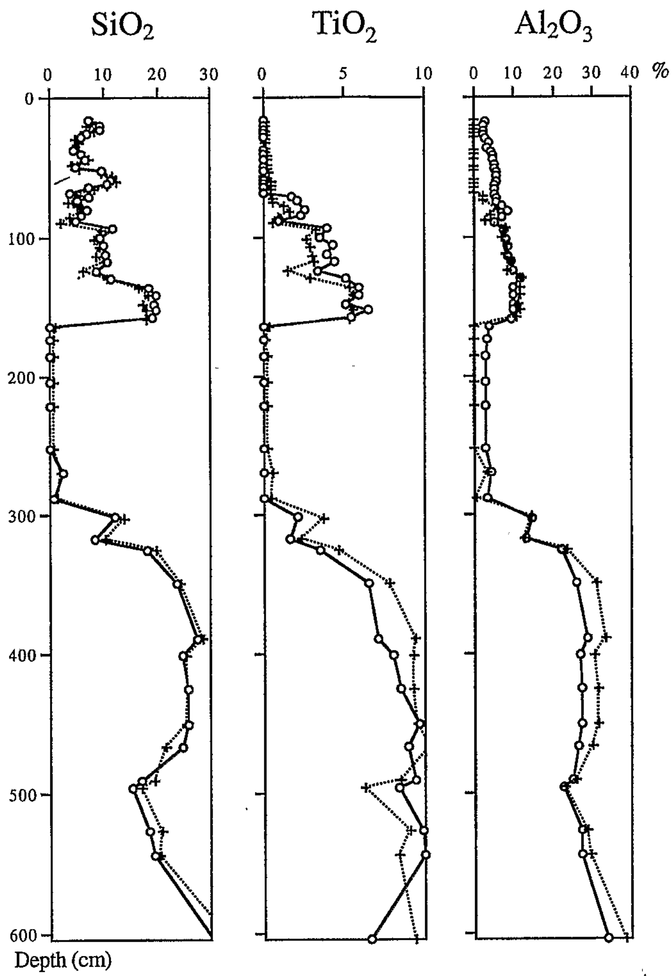


Fig. 3 Comparison of the chemically (+) and the FTIR (o) determined SiO_2, TiO_2 and Al_2O_3. FTIR SiO_2 = SiO_2 quartz + SiO_2 amorphous silica + SiO_2 kaolinite; FTIR Al_2O_3 = Al_2O_3 kaolinite + Al_2O_3 Gibbsite; FTIR TiO_2 = TiO_2 anatase.

Comparaison des teneurs en SiO_2, TiO_2 et Al_2O_3 déterminées chimiquement et minéralogiquement par spectrométrie IRTF.

Phytoliths

Amorphous silica in the sediment mainly consists of phytoliths. Phytoliths represent the accumulation of monomeric silicic acid in epidermal tissues of leaves and needles in the form of various sand- or silt-sized incrustations (Bartoli and Wilding, 1980). Between 70 and 600 cm, their proportion in the sediment varies according to the evolution of the detrital minerals. Microscopic observations indicate that all the shapes found in the soils are also found in the sediment. This suggests that phytoliths are themselves mainly trans-

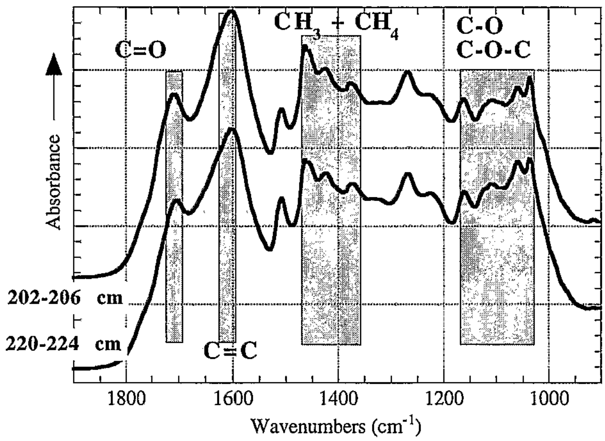


Fig. 4 IR spectra of sediments close to the change of vegetation in level Ib. Shaded areas correspond to characteristic absorption bands assigned to some functional groups.

Spectres IR de sédiments au niveau du changement de végétation dans la zone Ib. Les surfaces ombrées correspondent à des bandes d'absorption caractéristiques de certains groupes fonctionnels.

ported to the area of sedimentation during the late Pleistocene.

Abrupt change of vegetation in the Ib level

The most abrupt change outlined by the pollen occurs inside level Ib (**fig. 2**). Above 220 cm, this level is characterized by the disappearance of algae pollen, a decrease of the pollen indicative of the semi-deciduous forest and an increase in *Myrtaceae* pollen. This abrupt change can be interpreted either by a strong development of Myrtaceae inside the depression, partially masking pollen transportation from the surrounding area, or by a change in the nature of the forest around the "Lagoa Campestre" depression. However, the lack of mineral constituents arriving in the depression shows that the forest was still important regionally. A sedimentary hiatus does not provide a satisfactory explanation for this change of vegetation: IR spectra of samples just below (220-224 cm) and above (202-206 cm) the change are very similar, as demonstrated by the absorbance of organic matter constituents (**fig. 4**); absorption bands assigned to some fundamental organic functional groups (C=O, C=C, CH_2 + CH_3, C–O, C–O–C) do not vary, as would be expected in the case of an emersion and a subsequent evolution (oxidation) of some organic constituents (Durig *et al.*, 1988; Landais and Rochdi, 1993). This similarity also suggests that the local conditions of sedimentation did not vary, and therefore supports the interpretation of a regional environmental change. Here FTIR spectroscopy improves the pollen interpretation by suggesting an abrupt regional change of the vegetation rather than a sedimentary hiatus as an explanation for the observed patterns.

The sedimentary hiatus

The mineral content of the sediments outlines the hiatus detected by the ^{14}C dating: the sediment was dated at 28,740 yrs BP at 170 cm, and at 16,800 yrs BP at 157 cm. The hiatus corresponds to a sharp limit between sequences I and II at 164 cm and represents the onset of the very arid conditions corresponding to the Last Glacial Maximum in most areas of South America. There is a very clear change in the mineral content of the sediments below and above this limit while the only change revealed by the pollen assemblage is a decrease of the *Myrtaceae.* In a similar case and in the absence of dating, the mineral signal record could thus be useful to detect a hiatus of sedimentation which would not be clearly revealed by the palynological study.

2) Holocene sedimentation

Level IIb is characterized by the absence of detrital minerals and the presence of phytoliths as unique mineral constituent of the sediments. During this period, pollen indicate the presence of forest (*Araucaria* or mesophytic semideciduous forest) except for the interval 5,500-4,500 yr BP when total tree pollen frequency became low due to a strong moisture stress induced by a longer dry season (Ledru, 1993). This decrease in the arboreal pollen (AP) content was not accompanied by a return of detrital sediments. Moreover, the fluctuations of the phytolith content during the Holocene follow the variations in AP, in contrast with what we observe in all other parts of the core. These observations reinforce the hypothesis that during the Holocene the "Lagoa Campestre" depression moved from a lacustrine to a palustrine (peaty) environment, where no transport of detrital minerals occurred. In this case, mineralogy helps in assessing changes in local environment.

CONCLUSION

Data analysis from the LC3 core of the "Lagoa Campestre" depression shows that quantitative mineralogy using FTIR spectroscopy is a valuable tool for obtaining palaeoenvironmental information from lacustrine sediments. It provides a method to detect: (i) regional changes of the vegetation cover related to variation in the transport and sedimentation of detrital phases; (ii) sedimentary hiatus through sharp variations of the mineral contents; (iii) abrupt changes of the vegetation cover which, in the absence of ^{14}C dating could otherwise be misinterpreted as the consequence of a sedimentary hiatus. In the case of Salitre, quantitative mineralogy clearly identifies the change from a lacustrine to a palustrine environment.

In conclusion, we propose to use quantitative mineralogy for palaeoenvironmental reconstructions, as a fast and convenient method to correlate vegetation changes, already detected by the pollen analysis of a core, with mineral content variations measured elsewhere in the same sedimentary basin. This new method could replace replicate palynological studies, and could reinforce environmental interpretations from pollen data.

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