INAA, XRD, SEM and TL investigation of loess/paleosoil sequences of Eastern Dobrudja, Romania

O.G. Duliu, A. Chirosca, University of Bucharest, Romania

D. Dumitraş, C. Costea, Geological Institute of Romania

L. Ţugulan, National Institute for Physics and Nuclear Engineering, Bucharest, Romania

M.V. Frontasyeva, O.A. Culicov, I.V. Zinkovskaya Joint Institute of Nuclear Research, Dubna, Russian Federation

Aims

- **A detailed investigation of the geochemistry and mineralogy of the Dobrudja loess deposits for a future absolute geochronolgy by means of radiation defects accumulation methods (Thermoluminiscence, Optical Stimulated Luminescence and Electron Paramagnetic Resonance).**

Loess

- **Loess** is an aeolian sediment formed by the accumulation of wind-blown silt consisting of quartz, feldspars, clay and carbonate, typically in the 10–50 micrometer size range.
- **Loess** It is usually homogeneous, highly porous due to quartz grains and loosely cemented by calcium carbonate.
- For this reason, **loess** is traversed by vertical capillaries that permit the sediment to fracture and form vertical bluffs.

Loess

- According to von Richthofen, **loess** formed during Pleistocene when silt outwashed from the glaciers and deposited downstream was blowed by winds and deposited far away, generating extended deposits, up to 400 m thick.
- The absence of any coeval volcanic components makes loess dating a difficult task, but at the same time a **challenge**, so that only a geochronology based on the accumulation of radiation induced defects could give confident results.

Loess

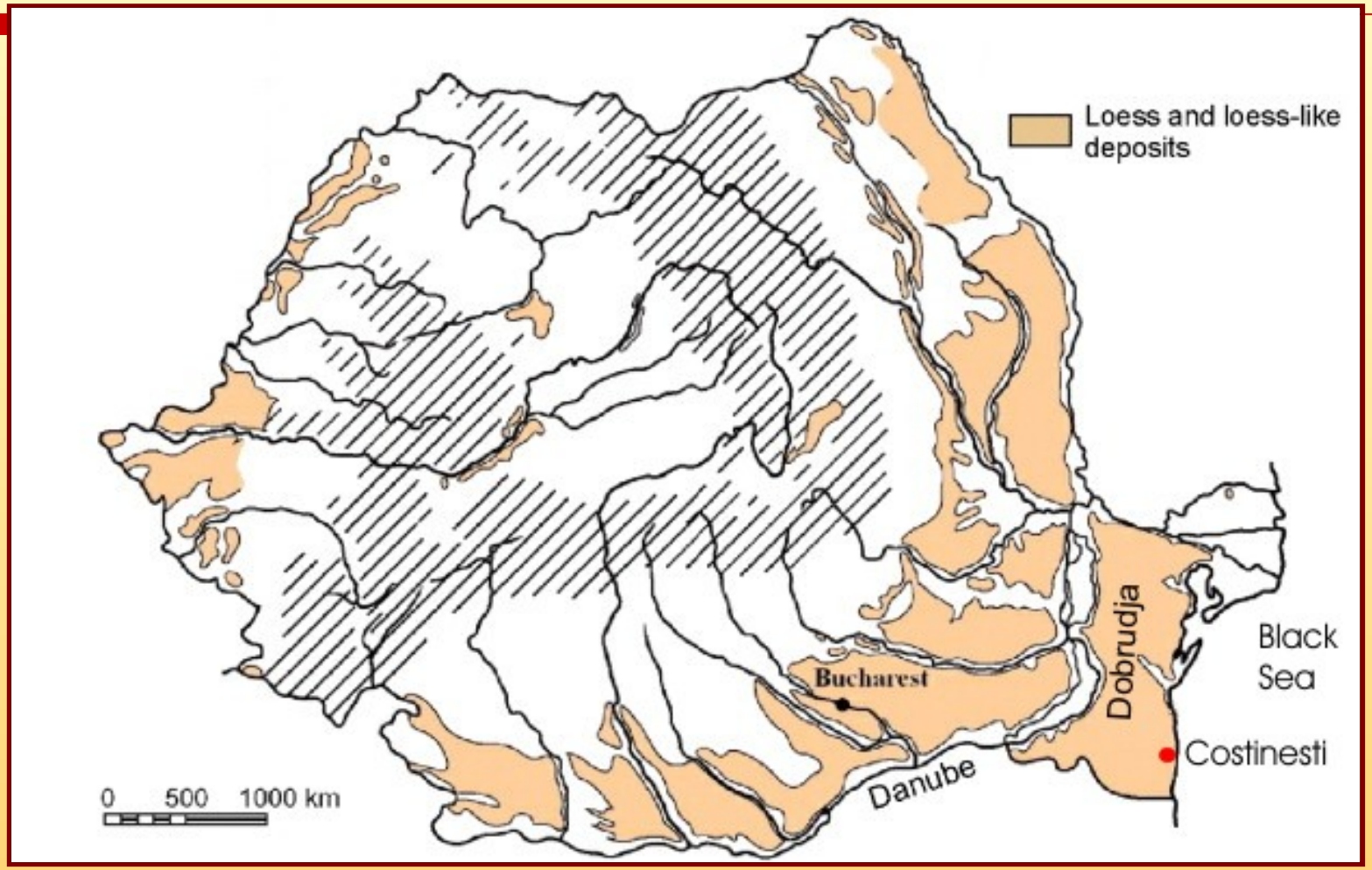
- Three different methods could be used to investigate the radiation induced centers whose content increase with the age:
  - Thermoluminiscence,
  - Optical Stimulated Luminescence
  - Electron Paramagnetic Resonance
- Each of them need an exact determination of the content of natural radioactive elements as well as a thorough study of loess deposits geochemistry and mineralogy

Loess

- Romania is a country rich in **loess** and **loessoide** deposits which could be found mainly in Romanian Plain, Southern Moldova, Western Plain end especially in Dobrudjea where loess deposits could reach 40 m thicknes.

- For this reason, in Autumn 2012 we have started a new project whose main purpose is to establish a confident, absolute geochronology of loess deposits

Loess



After: Timar-Gabor, A., Vandenberghe, A.G., Vasiliniuc, C., Panaoitu, C.E., Panaiotu, C.G., Dimofte, D. , Cosma, C. Quaternary International, 240, 62–70 (2011)

Loess

A typical loess sequence near Costinesti, South-Eastern Dobrudja

Samples

- In the South-eastern Dobrudja, at Costinesti we have extracted from the vertical cliffs and at different heights, a number of ten samples consisting of glacial loess and interglacial paleosoil.

- Finally, each sample were divided into more aliquots for subsequent analysis (INAA, gamma-ray spectroscopy, XRD, SEM)

Analytical methods

- **Epithermal Neutron Activation Analysis** was performed at the Frank Laboratory of NeutronPhysics JINR Dubna Russian Federation at the IBR 2 Nuclear Reactor

- 36 elements, seven major, rock forming (Na, Mg, Al, K, Ca, Ti and Fe) and 30 trace elements (Sc, Cr, Mn, As, Br, Rb, Sr, Zr, Mo, I, Cs, Ba, La, Ce, Nd, Tb, Dy, Yb, Hf, Ta, W, Th and U) of both untreated and HCl treated loess samples to remove carbonates.

Analytical methods

- **Scanning Electron Microscopy performed at the Geological Institute of Romania**

  More information concerning microstructure of loess and authigenic calcareous concretions

- **X Ray Diffraction performed at the Geological Institute of Romania**

  Mineralogical composition as well as the existing differences between soil and paleosoil

Analytical methods

- **Electron Paramagnetic Resonance (EPR) measurements done at the University of Bucharest to evidence the presence of different paramagnetic species and to identify the radiation free radicals prone to be used in geochronology**

- **Thermoluminiscence (TL) performed at the University of Bucharest to establish the presence and nature of the TL spectrum and its stability with respect to external light**

Analytical methods

- **Gamma ray spectroscopy performed at the national Institute of Physics and Nuclear Engineering**

Radiometric determination of the content of natural radioactive elements: (^{40}K, ^{210}Pb, ^{232}Th and ^{238}U and their daughter radionuclides) in order to calculate the annual dose debits as well as to establish at which all radioactive series are in a secular equilibrium.

Results

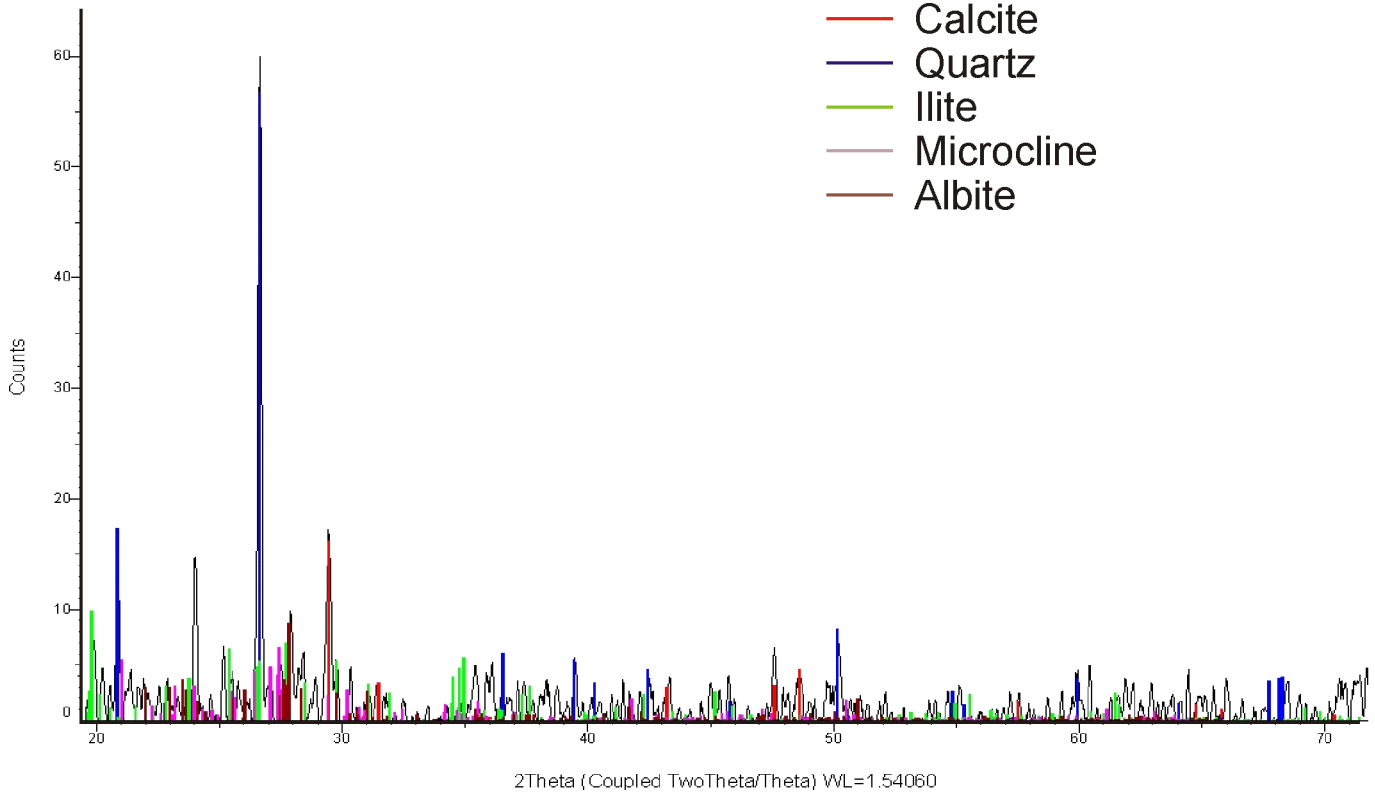
❑ X Ray diffraction

- A relative homogeneity of samples, with no differences within experimental uncertainties between soil and paleosoil, with a very low content of amorphous material.

- Experimental data showed, as expected, the dominance of quartz and calcite as well as of two types of feldspars microcline (alcali feldspar) and albite (plagioclase felspar) together with ilite and traces of ziron, monazite and chamosite.

Results

❑ X Ray diffraction



Results

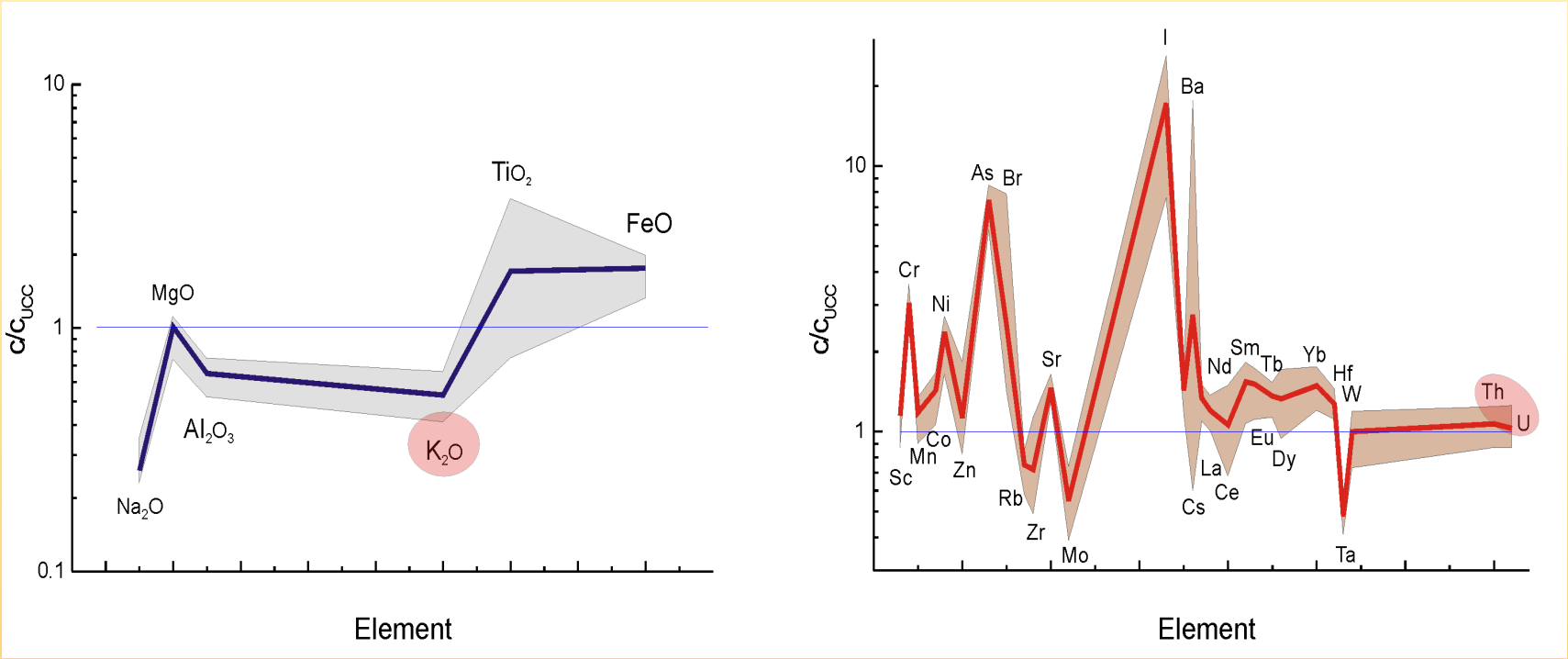
❑Epithermal Neutron Activation Analysis

- A relative homogeneity of samples, with no differences within experimental uncertainties between soil and paleosoil.

- Experimental data showed that all samples were very close to the Upper Continental Crust (UCC), but we have noticed some peculiarities regarding the REE distribution especially in the case of HCl treated and untreated samples.

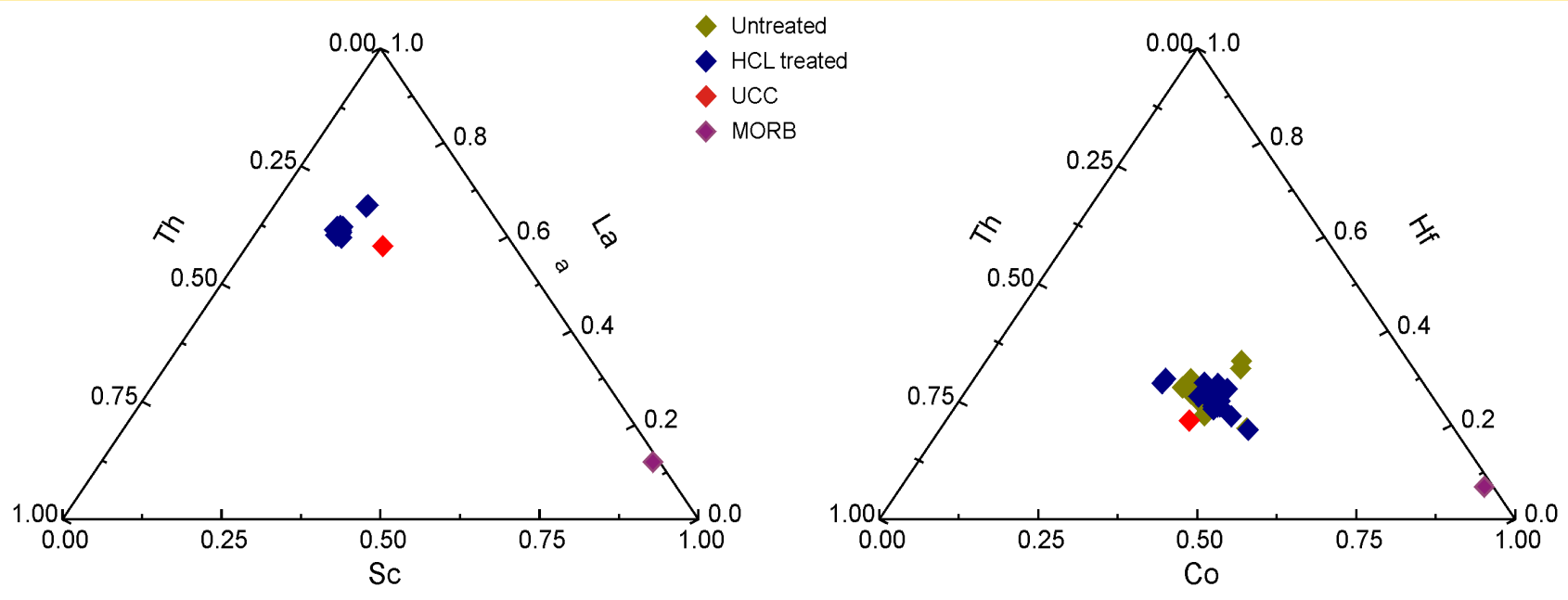
Results

❑Epithermal Neutron Activation Analysis



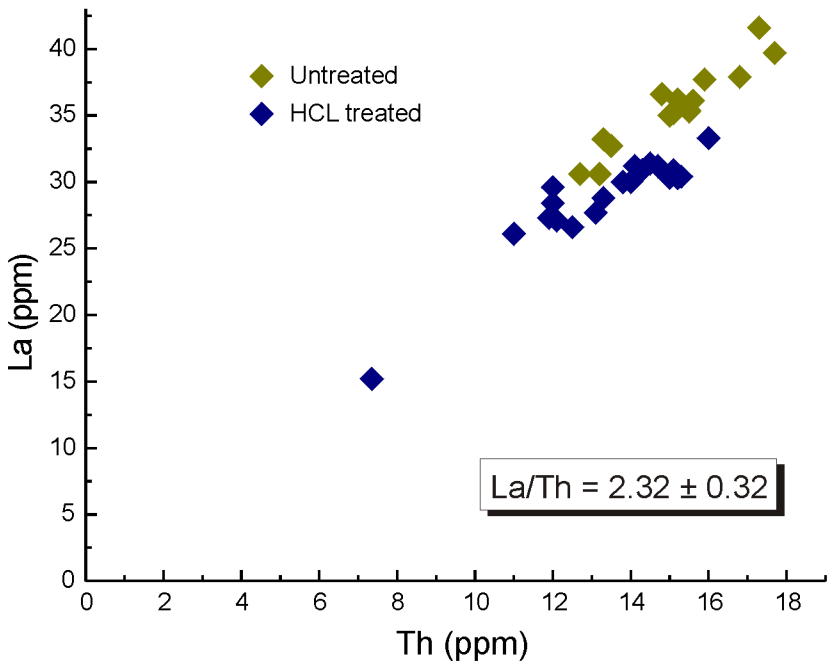
Results

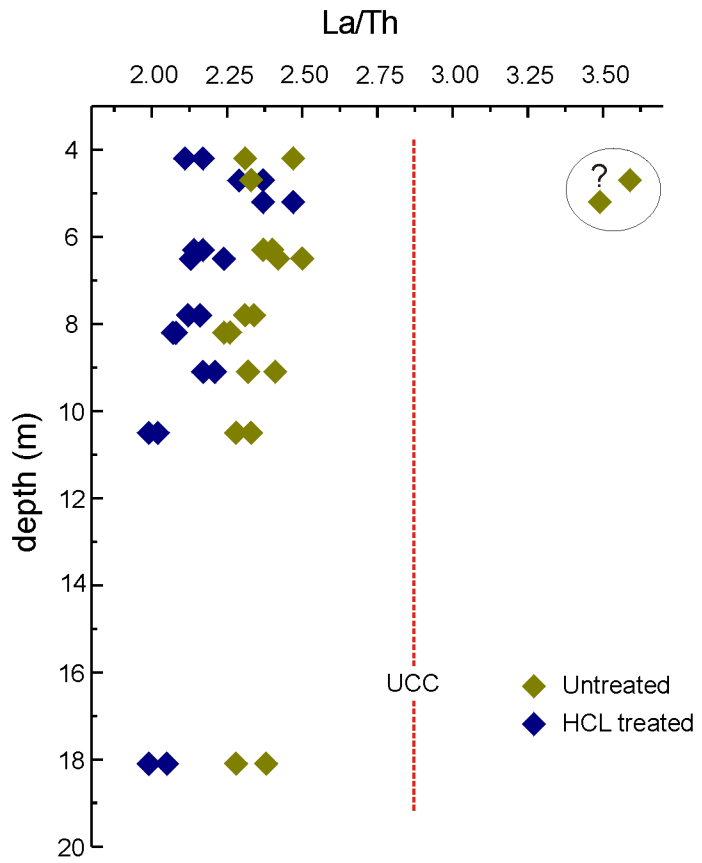
❑Epithermal Neutron Activation Analysis



Results

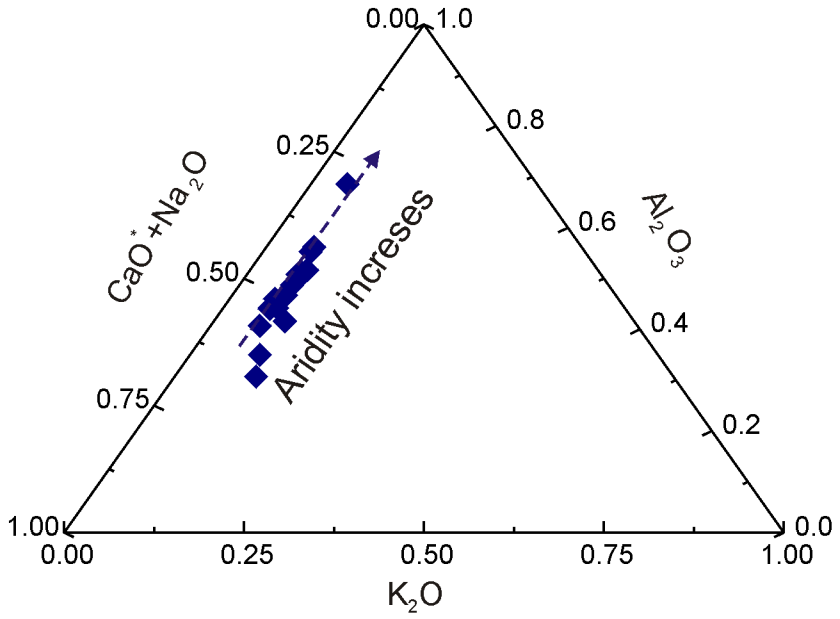
- **Epithermal Neutron Activation Analysis**

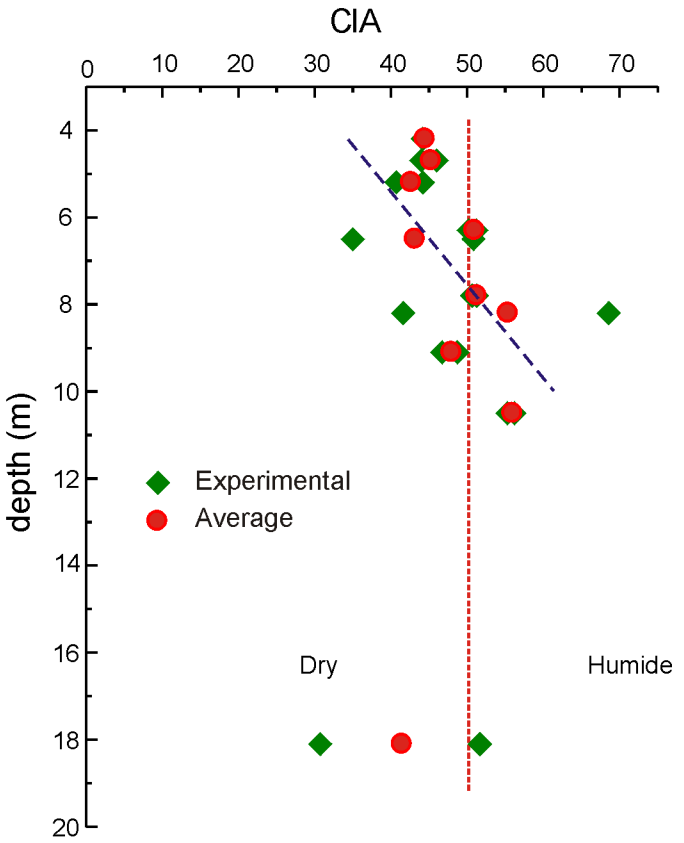




Results

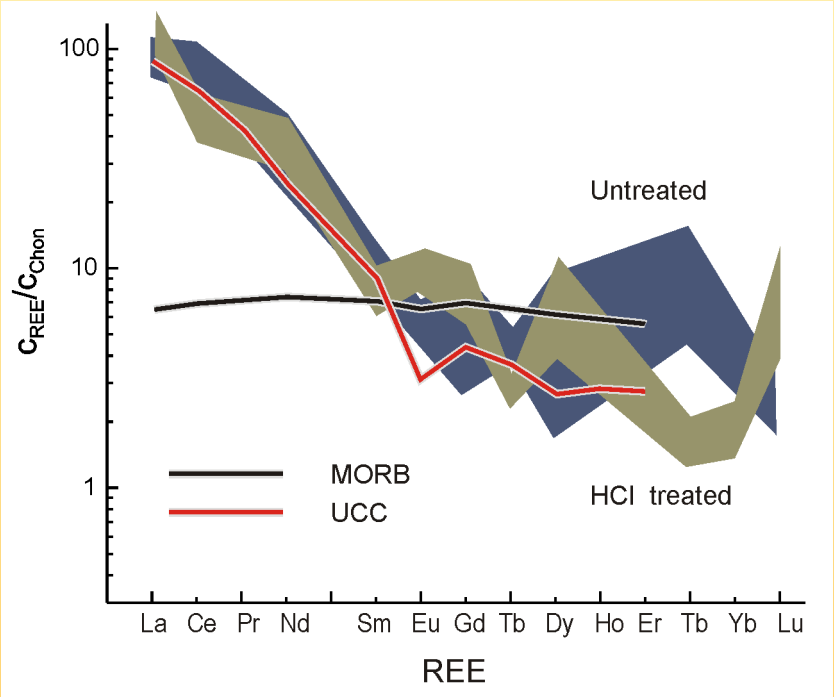
- **Epithermal Neutron Activation Analysis**





Results

❑Epithermal Neutron Activation Analysis



Results

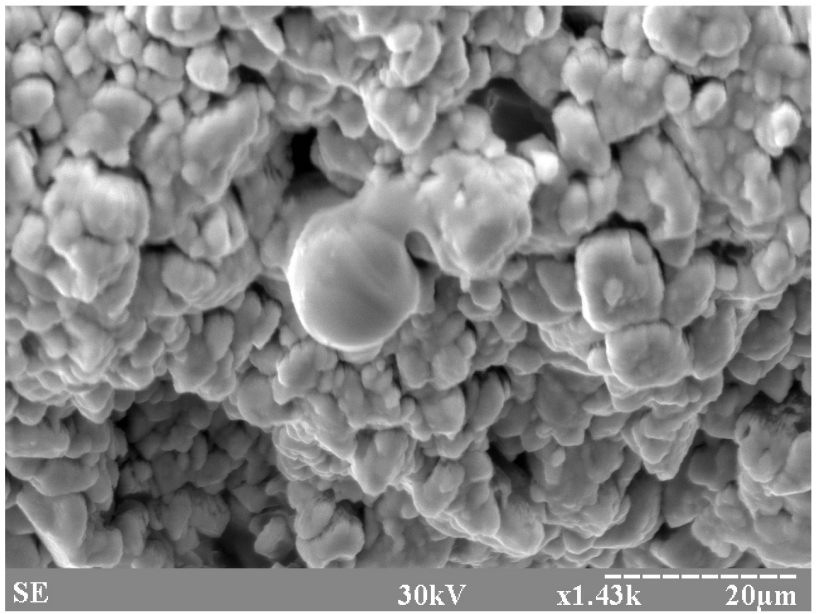
❑Scanning Electron Microscopy

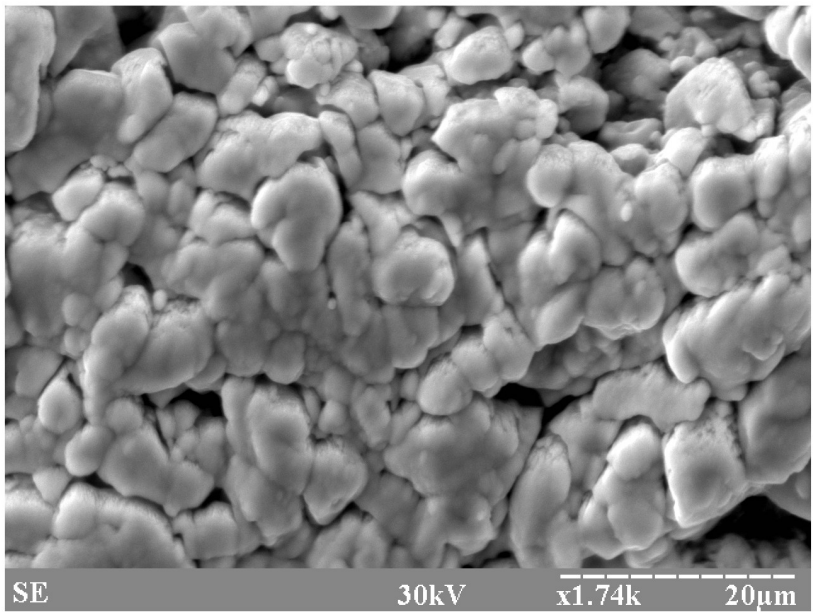
At micrometric levels, we have evidenced differences between loess and paleosoil on one hand and calcareous concretions

Evidences in calcareous concertinos of some microspherules, whose nature is still unknown

Results

❑Scanning Electron Microscopy

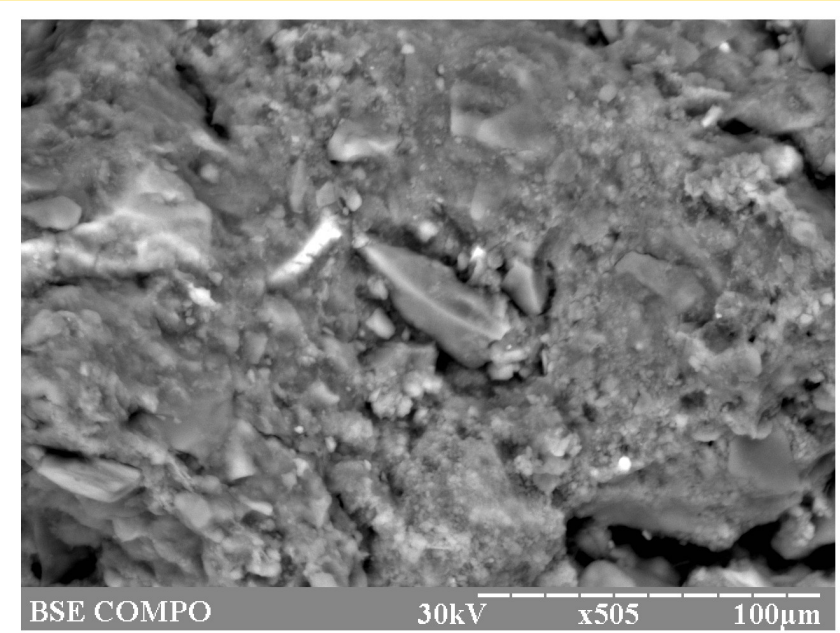


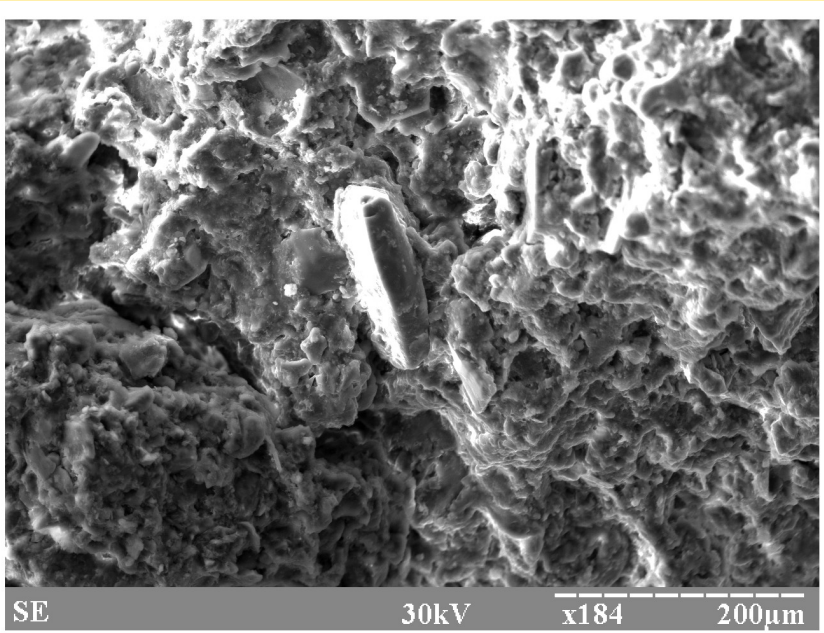


calcareous concretions

Results

❑Scanning Electron Microscopy





loess at different magnifications

Results

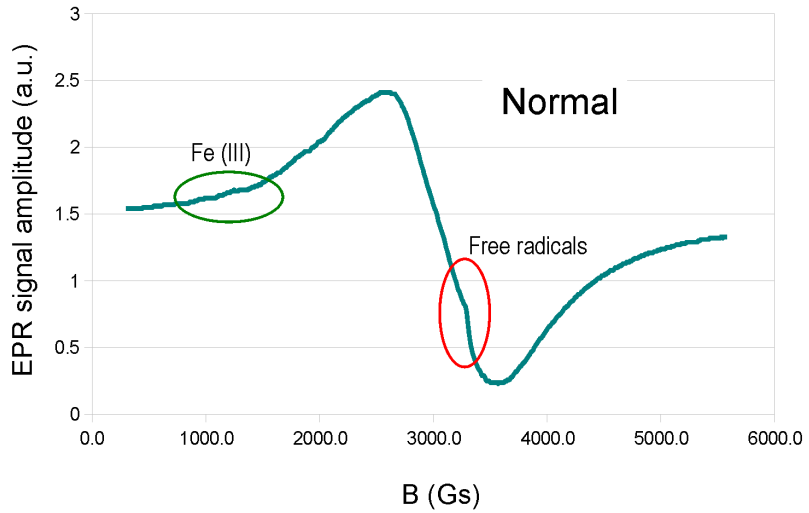
- **Electron Paramagnetic Resonance (EPR)**

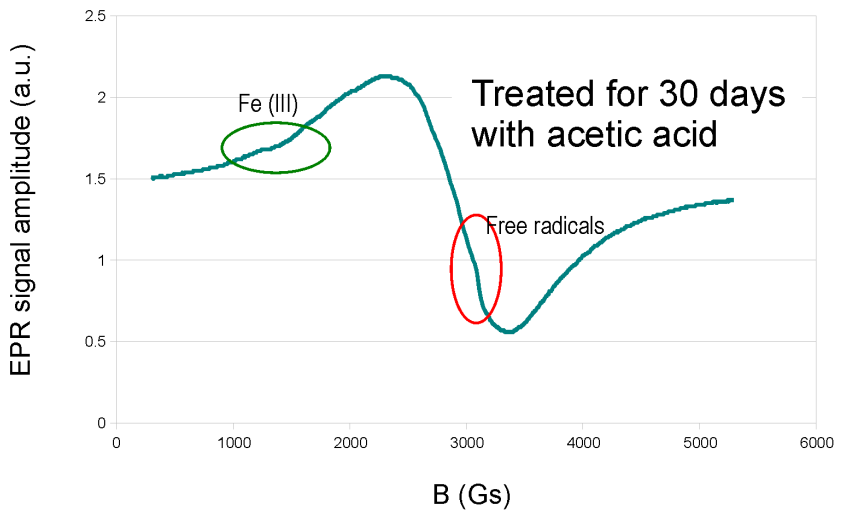
EPR spectra evidenced the predominance of Fe aggregates correlated with the presence of magnetite.

We have also identified very week signals due to the presence of Fe^{3+}iron in some crystalline matrix (quartz or feldspars) as well of the free radicals non associated with the carbonate fraction.

Results

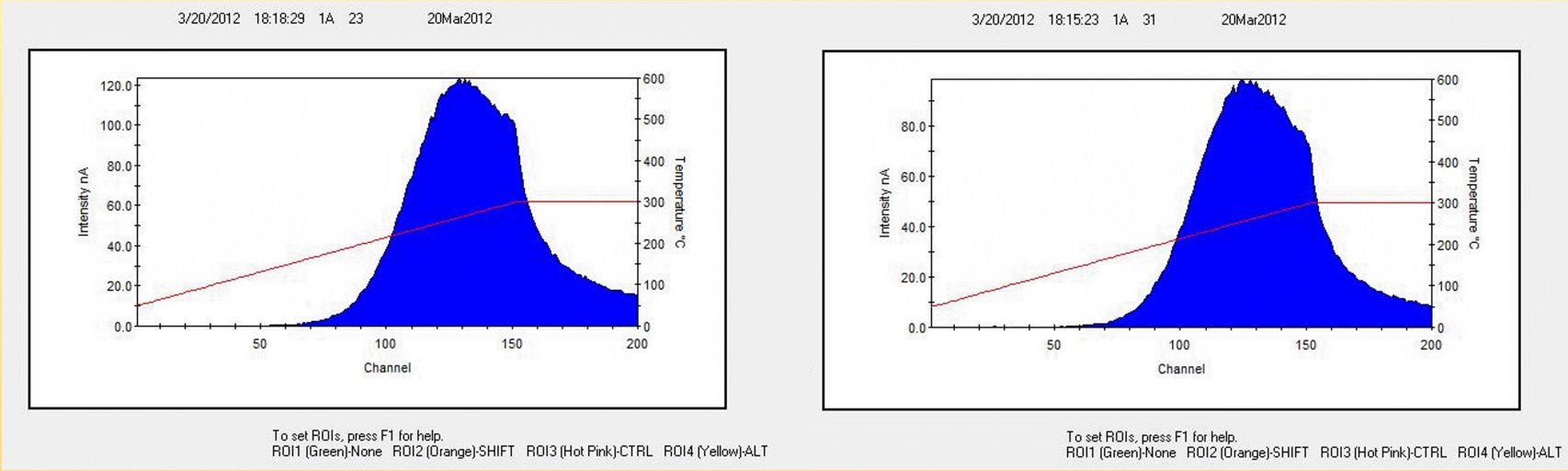
❑Electron Paramagnetic Resonance (EPR)





Results

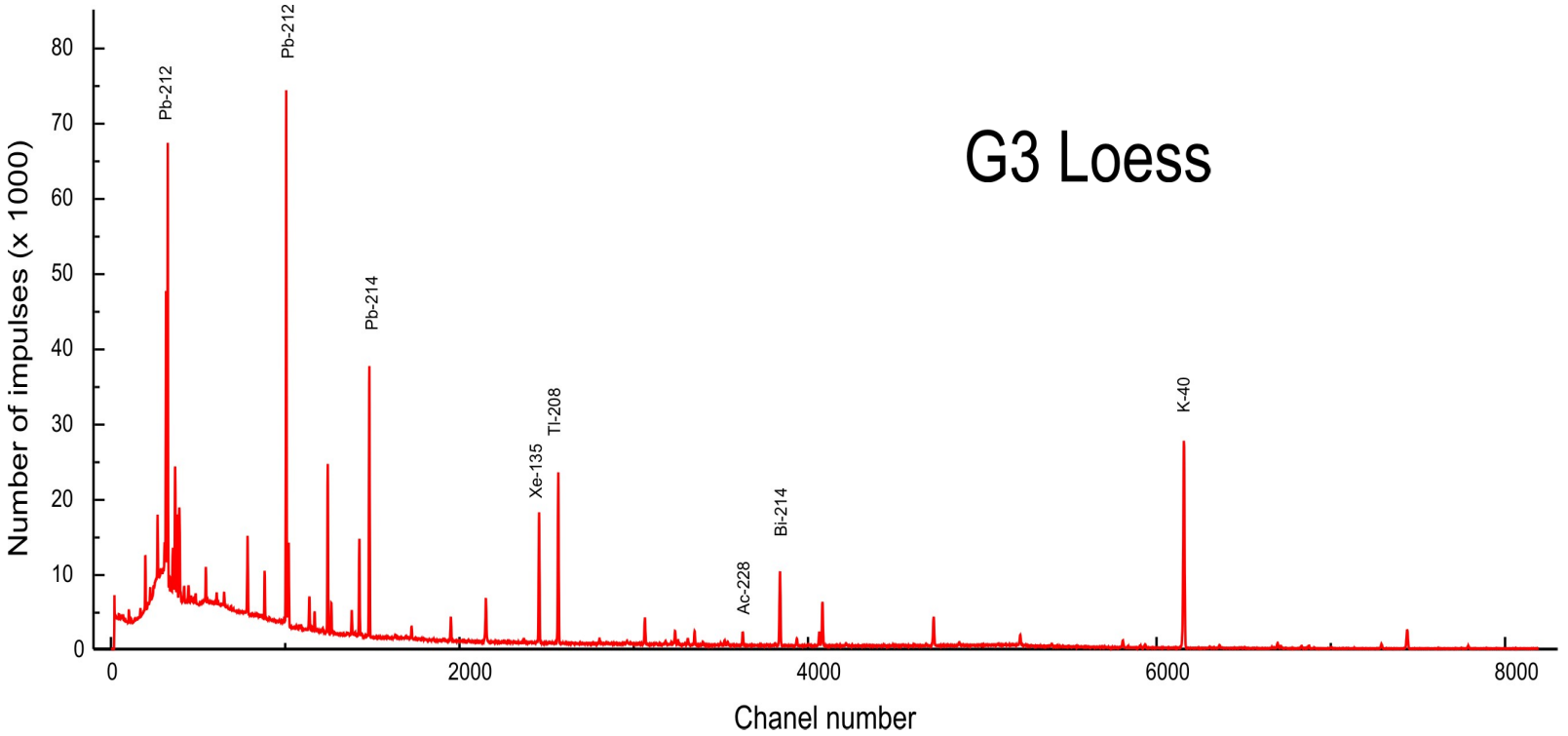
❑Thermoluminescence (TL)



All samples showed a robust TL signal, sensitive to external light in both normal and acetic acid dissolved samples, showing that TL signal is mainly due to siliceous fractions

Results

- **Gamma ray spectroscopy**



Results

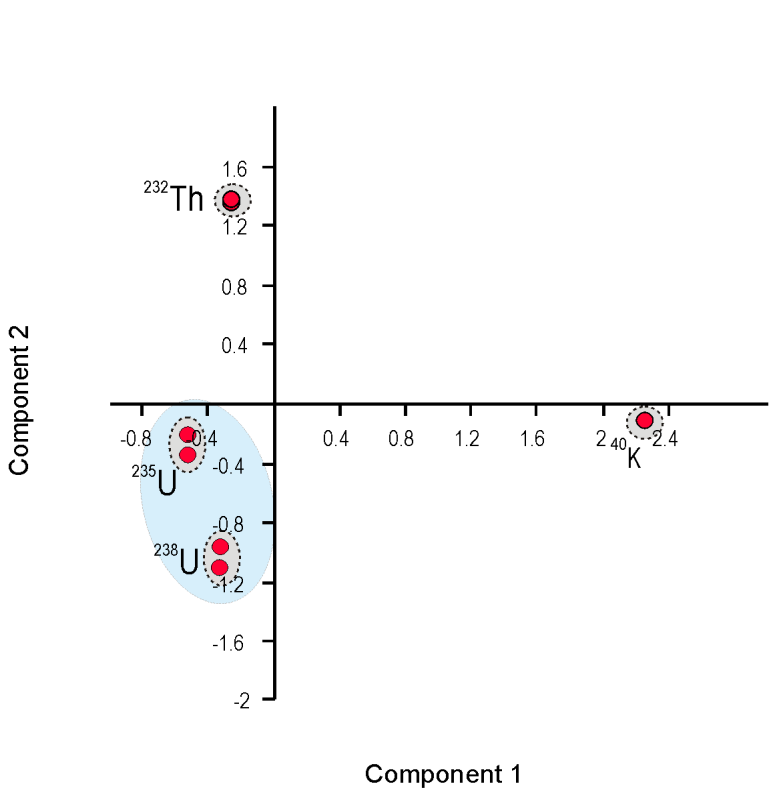
Gamma ray spectroscopy

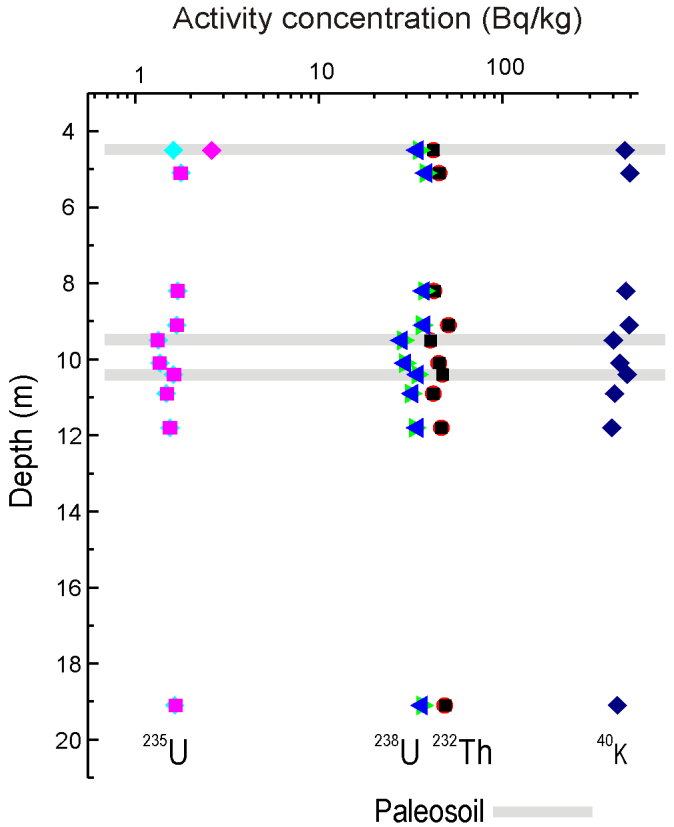
No significant differences between loess and paleosoil samples regarding the distribution of natural radioactive elements which gave for the annual dose debit a value of about 3.2 mGy/y, and a value of paleodose for the most recent loess (about 11 ky) of 37 Gy.

This value is very useful in establishing the procedure of determining the paleodose by dose additive method.

Results

Gamma ray spectroscopy





Concluding remarks

A relative homogeneity of loess and paleosoil samples concerning both major and trace elements, results confirmed by ENAA, XRD, and gamma ray spectrometry.

Trace elements distribution confirmed a close resemblance of loess and paleosoil samples to UCC.

Concluding remarks

All samples present both EPR and TL signals prone to be used for an absolute age determination.

Preliminary data sowed that these signal are associated to siliceous fraction, which indicate the experimental procedure to be followed, *i.e.* separation of different mineral fraction by selective acid digestion and use of heavy liquids.

Acknowledgment

- Cooperation Protocol 4077 – 4 - 11/13 between the Joint Institute of Nuclear Research, Dubna, Russian Federation and the University of Bucharest, Romania

- Two of the authors, L.T and A.C are Ph.D. students of the University of Bucharest

Thanks

Thank you for attention !