

[KN 1.2]**Chemometric Techniques for the Study of Acid-Base and Complexation Equilibria of Humic Substances****Adélio A.S.C. Machado**LAQUIPAI, Faculdade de Ciências, R.Campo Alegre 687, P4169-007 Porto, Portugal; e-mail: amachado@fc.up.pt

The work will be reviewed which was developed along the last years at the LAQUIPAI/Chemistry Department of the Faculty of Science at Oporto on the use of spectroscopic techniques (mainly synchronous fluorescence), with the treatment of data by chemometric techniques, for the study of acid-base and metal complexing properties of humic substances.

The advantages of procedures involving the repeated collection of spectra along titrations (at an increasing pH for acid-base studies or increasing metal ion concentrations for metal

complexation studies), followed by the treatment of sets of spectra by multidimensional techniques of self-modelling mixture analysis (evolving factor analysis, EFA, and SIMPLISMA) will be presented and discussed.

Results will be presented on the characterisation of natural humic substances extracted from soils as well as anthropogenic substances extracted from sludges in urban wastewater treatment plants and solid waste treatment plants.

[KN 1.3]**Analytical Speciation: Sources of Errors and Consequences on the Quality of Environmental Data****Armando da Costa Duarte**

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An overview of the role of Analytical Chemistry in speciation studies is undertaken in order to assess the basic principles which should support the analytical processes involved in obtaining experimental data on chemical speciation in the aquatic environments. The basic concepts of analytical quality control will be highlighted concerning their importance to monitor the quality of data, a finding which can be obtained by a plethora of different analytical techniques.

The experimental data obtained by different analytical techniques can provide different information about the chemical species present in environmental samples. Therefore, analytical speciation, among other possible definitions, is an operational concept and the data has to be used with caution when decisions have to be made on the quality of the environment.

The concentration levels of interest in speciation studies are in the range of ppt levels. Such a low level of detection is not pointed out as a problem for most of the advanced analytical techniques available through out a network of research laboratories. Those techniques are able to detect and quantify different chemical forms of the same element in complex environmental matrices even at levels below the ppt range. However, critical steps of the analytical process such as sampling, sample storage and sample treatment are not regarded with the same degree of care as the detection and quantification steps. Lack of proper quality control of all the steps of the

analytical process will lead to an enormous amount of data with no use for speciation and environmental decisions.

A considerable effort has been made, mainly in the European Union, and reported in technical papers and books on speciation analysis of Hg, Pb, Sn, Pb, As, Se, Cr and Al, and also on the certification of suitable materials for quality control purposes. Even an 'European Network on Speciation' has been conducted over two years (1998-2000) to 'tackle the problems related to the lack of communication between scientists, industry representatives and legislative for the possible improvement of written standards and EC regulations' [1]. Without a proper definition of the sources of errors on analytical speciation and evaluation of the consequences of these errors on the quality of environmental data, the improvements of standards and regulations will be very limited. The development of various degrees of sophistication in the quantification process will not reach a satisfactory level without proper research on the processes of sampling and extraction of analytes in environmental matrices.

Two examples will be used to illustrate some of the difficulties in analytical speciation: The presence of humic substances (a classical problem in speciation studies) and the quantification of endocrine disrupting chemicals.

[1] QUEVAUVILLER, PH. (1998): Method Performance Studies for Speciation Analysis. The Royal Society of Chemistry, pp. 228