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The use of mass spectrometry and spectroscopic techniques to study sulfur containing biomolecules

G Simões[1], A R Milosavljević[2], R Bernini[1,4], C Nicolas[2], F N Rodrigues[1,4], C Miron[2], A Giuliani[2,3], C S C Castro[5], G G B de Souza[1]
[1]Instituto de Química, UniversidadeFederal do Rio de Janeiro, Brazil
[2]Synchrotron SOLEIL, L'Orme des Merisiers, Saint Aubin, France
[3]UAR 1008 CEPIA, France
[4]Instituto Federal de Educação, Ciência e Tecnologia do Rio de Janeiro, Maracanã,Brasil
[5]Instituto Nacional de Metrologia, Qualidade e Tecnologia – Inmetro, Brasil

In recent years, there has been a large interest in the study of biological systems, namely, amino acids, peptides, and proteins, using synchrotron-based spectroscopic techniques, such as near-edge X-ray absorption fine structure (NEXAFS) or related X-ray photoelectron and X-ray emission spectroscopies. Most of the X-ray spectroscopic investigations of biologically relevant molecules, such as amino acids and their polymers, have been performed on thin organic films and liquids [1].The association of mass spectrometry and spectroscopic techniques has allowed for the investigation of the effects of radiation damage insulfur containing molecules. We have performed a NEXAFS (S1s) and mass spectrometry study of solid samples of cysteine, cystine and insulin irradiated with 0.8 keV electrons. The measured mass spectra point out to processes of desulfurization, deamination, decarbonylation and decarboxylation in the irradiated biomolecules [2].In another study, inner-shell measurements of insulin were performed by coupling a linear ion trap mass spectrometer, equipped with an ESI source at the french synchrotron radiation facility SOLEIL. Theelectrosprayed insulin ions were injected, mass selected, stored in the trap, and irradiated during a well-defined period.The near-edge X-ray ion yield spectra of the 6+ charge state insulin precursorwere recorded as a function of the photon energy, in the vicinity of the C1s edge.

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grazielisimoes@iq.ufrj.br

Sample preparation in MALDI ToF Mass Spectrometry

Clemens Schwarzinger
Johannes Kepler University Linz, Austria

Polymer mass spectrometry, especially MALDI-ToF MS, has gained a lot of attention in the last years because of the high amount of information that can be gained, such as molar mass distribution, repeat units, end groups, etc. But care must be taken when it comes to sample preparation, especially when the simple and fast "dried droplet" technique is used.As most people have already experienced dried droplet preparation tends to from rings of higher concentration at the outer rim, the so called "coffee rings", which results in inhomogeneous distribution of the compounds and therefore questionable results. We will show that several rather simples steps can be used to circumvent this phenomenon and how to produce reliable high quality data using dried droplets, as there are the use of ionic liquids as matrices or higher matrix concentration. The results were monitored with imaging techniques such as FTIR or mass spectrometric imaging to understand the processes necessary for a perfect sample preparation.When it comes to copolymers things are getting even worse. In this case it is mostly necessary to separate the polymer into fractions either by precipitation or by SEC. We have found that a modified Electrospray interface coupled to the SEC is a very efficient and elegant way yielding best results in terms of polymer separation and MALDI sample preparation.

Clemens.schwarzinger@jku.at