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Agriculture Intensive dans les Iles de la Caraibe : enjeux, contraintes et perspectives
Intensive Agriculture in the Caribbean Islands : stakes, constraints and prospects
Agricultura Intensiva en la Islas del Caribe : posturas, coacciones y perspectivas

THE MODERN ANALYTICAL AND REGULATORY LABORATORY

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ABSTRACT

The recent developments in the technology of the instrumental analysis have led to faster and cheaper analysis with a decrease in the detection limits and an increase in the specifity and sensitivity. At the research level modern spectroscopic and chromatographic methods are becoming increasingly important as tools for probing complex, heterogeneous systems as well as for analysing trace components. Because of the reduction in size, automatization and simplification of these instruments all these methods can be also used routinely for ensuring the quality of raw material and products. In this article a number of selected methods will be briefly described, illustrating the diversity of their applications.

SPECTROSCOPIC METHODS

Vibrational spectroscopy: Infrared and Raman spectroscopies

Both technics involve transitions between vibrational energy levels. In the case of small molecules, from the absorption infrared spectra, atomic groups may be identified and quantified. The influence of organic solvents may be also studied through intermolecular interactions and for example hydrogen bonding may be evidenced. For large molecules, spectra exhibit complex patterns, specially in the near-infrared region, rich in information but very difficult to elucidate without the use of large data bank. One of the major problem of infrared

analysis is the presence of water, which is an intense absorber, giving rise to large disturbing bands and reducing the penetration depth of the infrared radiation. Nevertheless the development of Fourier-transform spectroscopy associated with special sample preparation techniques allow now a wide range of quantitative determination in routine as well as the structural determination of the conformation of carbohydrates or proteins. Among the latest development the use of fiber optics combined with Fourier-tranform appears potentialy interesting for on-line measurements. Most of the Fourier-transform infrared instruments cover a very broad spectral range -from 50 to 15,000 cm^{-1}, and are announced for use in routine analytical application as well as in the research lab.

In Raman spectroscopy it is the exploitation of the shifts in frequencies present in radiation scattered perpendicular to the incident polarized visible or near infrared laser radiation which gives structural information. Raman is very useful in solution and may be used in combination with infrared data to achieve more complete srtructural determinations.

Ultraviolet and Visible spectroscopies

Ultraviolet and visible spectra are a measure of the energy involved in electronic transitions. Electronic spectra recorded mainly on liquid samples, consist of broad bands very sensitive to intra and inter molecular interactions. Both spectroscopies cannot give direct information concerning the structure but even a small alteration in the spectrum may be used as a measure of concentration or as indicators of purity. Influence of pH or, for instance change, in the ionization of phenolic groups are easily detected by this way.

Nuclear Magnetic resonance

Nuclear magnetic resonance (NMR) involve the energy transitions resulting from the interaction of an external magnetic field with the spin of nuclei. Nuclear magnetic resonance absorption can occur only for those nuclei which have non-zero spin such as ^{1}H, ^{2}H, ^{13}C, ^{14}N, ^{17}O and ^{31}P. NMR is finding increasing application in a

great number of scientific domains. The most familiar application of NMR is to obtain spectra of molecules in the liquid state in order to determine their chemical nature and abundance. The apparition of higher stable magnetic fields and greater electronic and computational facilities have led recently to available commercial materials for high-resolution solid-state and solution-state NMR. High-resolution solution state NMR allows to establish and quantify structural features and to investigate solution conformations using through-bond and through-space ^{1}H two dimensional correlation. The combined techniques of high power decoupling, rapid spinning at the "magic angle" and cross polarisation from abundant ^{1}H nuclei to dilute ^{13}C nuclei have revolutionised high resolution NMR spectroscopy of organic solids. This method has proved particularly powerful in the study of starches and cereal proteins and may be used to explore factors such as crystallinity. NMR can be performed also to probe "physical" properties related to molecular motion. Relaxation time measurements allows to determine solid/liquid ratios, with typical applications in the oils and fats industry, and also to examine subtle changes in motion. Finally NMR imaging is an important non-invasive method mainly used in the clinical field but promised to new developments in other scientific domains.

Mass spectrometry

Mass spectrometry (MS) provide useful qualitative or quantitative data on analytes isolated from matrices in quantities ranging from nanomoles to femtomoles. Every year MS devices continue to shrink in size, even as they become more intelligent, more automated and more sensitive in detecting molecules of interest. To improve sample purity, before the ionization of the molecules, a variety of chromatographic techniques such as gas chromatography (GC/MS) or high performance liquid chromatography (LC/MS) or are now combined with mass spectrometry. Mass spectrometry was devoted firstly to volatile organic compouds but the new methods of ionization introduced allow to study nonvolatile compounds up to 20,000 atomic mass unit. The latest development which is time-of-flight instrument (TOF MS) is annouced to offer a mass range of more than 200,000 atomic mass unit with picomole sensitivity. GC/MS is still the most important technique used in flavour analysis. Toxicants and

contaminants of raw materials or food products such as pesticide residues are sucessfully analyse by this way. Naturally-occuring bioactive molecules are also determined by MS.

CHROMATOGRAPHY

Gas chromatography and high performance liquid chromatography

Gas chromatography (GC) and high performance liquid chromatography (HPLC) are the most frequently used separation techniques to solve analytical problems. GC or HPLC allow a very rapid determination at a lower cost than most of the spectroscopic methods. The major drawbacks remain i) sample preparation which may be very time consuming and ii) the sensitivity of the detection. Although the concepts of chromatography remain the same, there are important differences between GC and HPLC. In GC, the two major variables used to achieve a separation are the type of stationary phase and the column temperature while in LC, it is the type of the stationary phase as well as the type of the mobile phase. It is the mobile phase which gives LC its great flexibility but LC is much less flexible than GC in its detection systems. GC methodology is very well developed for small organic compounds such as pesticides while LC is suitable for larger molecules even for polymers. In conclusion HPLC and GC complement one another and should be employed in that manner.

Size exclusion chromatography (SEC)

The SEC technique is a liquid chromatographic technique utilized for separation of polymer mixtures. In this technique, the hydrodynamic radius of the polymer determines separation profiles. Larger molecules elute first because they spend less time in the intertitial pores of the column. In principle there is little or no interaction between the polymers and the resin surface so the choice of the resin used for a given polymer is of prime importance. Calibration techniques are available for flexible linear polymer using the intrinsic viscosity of a set of standards of known molecular weight and narrow molecular weight distribution but all these techniques are very

dependent of the solvent used. An absolute determination of the molecular weight may be achieved with on-line multi-angle laser light scattering detection (MALLS).

REFERENCES

A very large literature is devoted to these techniques so I will just mentionned some recent books more specialised on carbohydrate and protein analytical chemistry with some applications in food science.

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