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Review Article

PHOTOACOUSTIC SPECTROSCOPY- A REVIEW

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Abstract:

Photoacoustic spectroscopy in simple words is the measurement of the effect of absorbed electromagnetic energy (particularly of light) on matter by means of acoustic detection. It is based on the photoacoustic effect. A photoacoustic spectrum of a sample can be recorded by measuring the sound at different wavelengths of the light. This spectrum can be used to identify the absorbing components of the sample. The photoacoustic effect can be used to study solids, liquids and gases.

The application of the photoacoustic effect in the infrared region is a new technique; with the development of highly-sensitive FTIR instruments, this technique is being widely researched for the analysis of solid samples. Advances in FTIR photoacoustic (PA) spectroscopy have significantly extended the scope and utility of the PA technique in the areas of: microparticle spectroscopy, compositional determinations via factor analysis, coal surface oxidation measurements, spectroscopy of highly opaque samples, and PA detector technology. This review article includes complete study regarding the principle, instrumentation, working along with the advancements & future prospects of photoacoustic spectroscopy.

Key Words: FTIR, PA, Acoustic detection, Microparticle Spectroscopy

Introduction:

Photoacoustic spectroscopy is the measurement of the effect of absorbed electromagnetic energy (particularly of light) on matter by means of acoustic detection. The absorbed energy from the light causes local heating, generating a thermal expansion which creates a pressure wave or sound. Later, it was discovered that materials exposed to the non-visible portions of the solar spectrum (i.e., the infrared and the ultraviolet) can also produce sounds. A photoacoustic spectrum of a sample can be recorded by measuring the sound at different wavelengths of the light. This spectrum can be used to identify the absorbing components of the sample. The photoacoustic effect can be used to study solids, liquids and gases.

Principle: Photoacoustic spectroscopy measurement is based on the photoacoustic effect. The photoacoustic effect was discovered by Alexander Graham Bell in 1880. This is the phenomenon whereby, when intermittent light is irradiated onto a substance, the substance emits acoustic waves of the same frequency as the light pulse frequency. It took several decades from that time until the photoacoustic effect was subsequently applied as a measurement technique. With the development of highly-sensitive microphones and other advances in electronics, research progressed into the measurement of gas samples, in particular. The application of the photoacoustic effect in the infrared region is a new technique. With the development of highly-sensitive FTIR instruments, this technique is being widely researched for the analysis of solid samples.

The solid sample to be measured is placed in a sealed vessel to which small microphone is attached. When a modulated infrared light beam is absorbed by the sample, heat is generated due to the incident light. This heat causes pressure changes in the surrounding gaseous layer, which can be detected by the high-sensitivity microphone. The signals from the microphone are acoustic interference waves.

Instrumentation:

A. Radiation source

Radiation source can be output from a laser, a monochromator furnishing radiations in UV, IR, or a FT-IR spectrometer (tungsten lamp, carbon arc lamp, high pressure xenon lamp, Nernst glower and lasers.) All radiation must be pulsed at an acoustical frequency 50-1200Hz. PA cell is filled with transparent gas often air or helium and cell volume is kept small, less than $1cm^3$ in order to preserve the strength of the acoustical signal. In commercial photoacoustic spectrometers, incoherent sources such as lamps are employed in combinations with filters or interferometers. Devices equipped with a small light bulb, with either a chopper or direct current modulation as modulated source and appropriate filters to avoid absorption interferences with other species, are used as compact gas sensors, e.g. for indoor CO2 monitoring.

B. Modulation Schemes

Modulation schemes can be classified into the modulation of the incident radiation and modulation of the sample absorption itself. The first technique includes the most widely used amplitude modulation (AM) of continuous radiation by mechanical choppers, electro-optic or acousto-optic modulators as well the modulation of the source emission itself by current modulation or pulsed excitation. In comparison to amplitude modulation (AM), frequency modulation (FM) or wavelength modulation (WM) of the radiation may improve the detection sensitivity by eliminating the continuum background caused by a wavelength independent absorption, e.g. absorption by cell windows, known as window heating. This type of modulation is obviously most effective for absorbers with narrow line width and most easily performed with radiation sources whose wavelength can rapidly be tuned with a few wave numbers.11-13

C. Photoacoustic cell

The Photoacoustic cell serves as a container for the sample under study and for the microphone or other device for the detection of the generated acoustic wave. An optimum design of the Photoacoustic cell represents a crucial point when background noise ultimately limits the detection sensitivity. In particular, for trace gas application many cell configurations have been presented including acoustically resonant and non-resonant cells, single and multipass cells, as well as cell placed intracavity. Nonresonant cells of small volume are mostly employed for solids samples with modulated excitation or for liquids and gaseous samples with pulsed laser excitation.

D. Detection Sensors

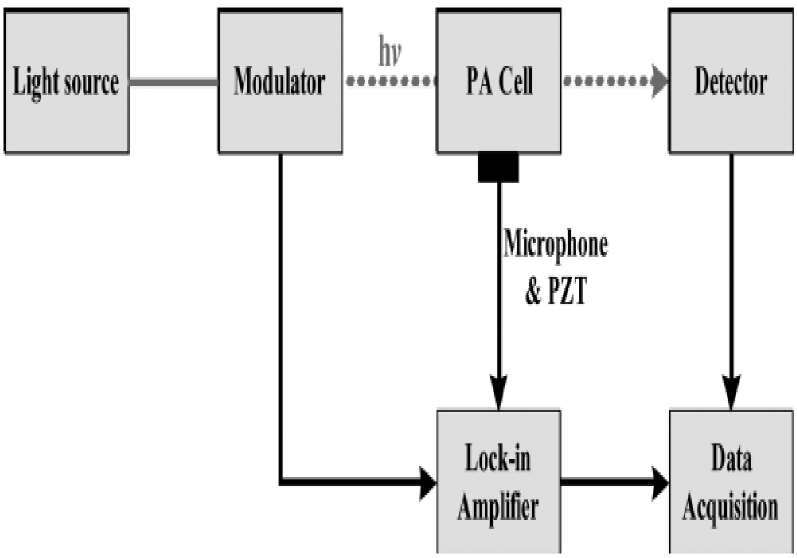
The acoustic disturbances generated in the sample are detected by some kind of pressure sensor. In contact with liquid or solid samples these are piezoelectric devices such as lead zirconate titanate (PZT), LiNbO3 or quartz crystals. These sensors offer fast response times and are thus ideally adapted for pulsed photoacoustics.

Single and Multiple Wavelength PAS:

PA Spectroscopy can be divided into two broad categories:

The first can be described as single-wavelength spectroscopy, since only one wavelength of light impinges on the sample of interest. Signal generation in a gas-microphone cell can be used to illustrate this technique. Three steps can be identified. First, modulated radia-tion from a laser or other suitable source impinges on the condensed-phase sample; second, the absorbed radiation is converted to heat by radiationless processes; and third, the heat generated within the sample is transferred to its cooler surroundings. Periodic heating of the boundary layer of carrier gas adjacent to the warm surface creates a pressure (acoustic) wave that is detected by the transducer (microphone). This experiment can be extended to include measurement of wave-length (wavenumber) dependence of optical absorption by systematically changing the wavelength of the incident radiation to build up a PA spectrum. Sequential observation of PA signals can be effected by selecting different lines from a multiple-wavelength laser or by use of an optical filter, such as a grating mono-chromator, in conjunction with broad-band radiation. These techniques were used to obtain PA infrared spectra by several research groups, particularly in the 1970s and early 1980s when PA spectroscopy enjoyed a rapid increase in popularity. Currently, multi-line gas (CO2 and CO) and solid-state mid- and near-infrared lasers are used for specific PA applications, an important example being trace gas detection.

The second category is multiple-wavelength (multiplex) PA spectroscopy, as practiced with Fourier Transform infrared (FT-IR) spectrometers. Most readers already know, of course, that these spectrometers have attained very wide accept-ance in analytical, research, and teaching laboratories during the last four decades. In the present context, the most important attribute of an FT-IR spectrometer is its capability for simultaneous measurements at a range of wavelengths; spectral coverage is determined mainly by the optical characteristics of the beamsplitter, the window material in the sample accessory, and the detector. An optical detector is not required in conventional PA FT-IR spectroscopy, and the accessible wave-length interval depends only on the beamsplitter and the window fitted on the gas-microphone cell. This technique has been used extensively for about three decades and is the source of the majority of the literature discussed in this book. Signal generation in the PA FT-IR experiment can be described in terms similar to those in the previous paragraph. Modulation is provided by the moving mirror in the interferometer or by use of an external device such as a chopper.



Applications:

One of the important capabilities of using FTIR photoacoustic spectroscopy has been the ability to evaluate samples in their in situ state by infrared spectroscopy, which can be used to detect and quantify chemical functional groups and thus chemical substances. This is particularly useful for biological samples that can be evaluated without crushing to powder or subjecting to chemical treatments. Seashells, bone and such samples have been investigated.[Using photoacoustic spectroscopy has helped evaluate molecular interactions in bone with osteogenesis imperfecta .

In the last twenty years, very low cost instruments for applications such as leakage detection and for the control of carbon dioxide concentration have been developed and commercialized. Typically, low cost thermal sources are used which are modulated electronically. Diffusion through semi-permeable disks instead of valves for gas exchange, low-cost microphones, and proprietary signal processing with digital signal processors have brought down the costs of these systems. The future of low-cost applications of photoacoustic spectroscopy may be the realization of fully integrated micromachined photoacoustic instruments.

Recently, the photoacoustic approach has been utilized to quantitatively measure macromolecules, such as proteins. The photoacoustic immunoassay labels and detects target proteins using nanoparticles that can generate strong acoustic signals. The photoacoustics-based protein analysis has also been applied for point-of-care testings. Photoacoustic spectroscopy also has many military applications. One such application is the detection toxic chemical agents. The sensitivity of photoacoustic spectroscopy makes it an ideal analysis technique for detecting trace chemicals associated with chemical attacks.

PAS sensors may be applied in industry, security (nerve agent and explosives detection), and medicine (breath analysis).

Advancements in Instrumentation:

Advances in FTIR photoacoustic (PA) spectroscopy have significantly extended the scope and utility of the PA technique in the areas of: microparticle spectroscopy, compositional determinations via factor analysis, coal surface oxidation measurements, spectroscopy of highly opaque samples, and PA detector technology. A method is reported for measuring FTIR spectra of single particles in the tens of Am size range which uses a tungsten needle to pick up particles and hold them in the sample chamber of the PA detector. The tungsten needle is initially mounted on a micromanipulator and particle pick-up is performed under a microscope. The needle and sample are then transferred directly to the PA detector sample holder which positions the particle in the IR beam. No sample alignment or thinning are necessary. Compositional determinations of kaolinite and quartz in coal have been performed using the Perkin-Elmer CIRCOM factor analysis program. The IR spectra were collected by DRIFTS and PA methods using synthesized samples of known compositions for the learning set and unknown test samples. The PA spectra yielded slightly better correlations. Coal surface oxidation was studied using a calibrated UV irradiation of coal to generate carbonyl species, thereby gauging the freshness of coal surfaces by how much carbonyl is formed by the UV exposure. FT-IR-PA difference spectra are used to measure the increase in carbonyl. UV generated carbonyl is found to increase with surface freshness. This method avoids the need of a "fresh coal standard" which is difficult to reproduce. The method's probe depth is based on the decay length of UV rather than IR photons in coal resulting in an increase in surface specificity. The linearity of FT-IR-PA spectra as a function of absorbance has traditionally not been maintained at the peaks of strong bands in opaque samples. This leads to peak truncation and reduced spectral contrast. A method to extend linearity using the magnitude and phase information of the PA signal is reported based on the Rosencwaig-Gersho Theory of PA signal generations. Spectra of polymer slabs demonstrate the utility of this approach for enhancing spectral contrast. Developments are reported in PA detector technology which increase the scope of applications that commercial PA detectors can be used for.

Future Prospects of PAS:

The combination of the relative simplicity and high sensitivity of the technique suggests a bright future for PAS in the oil analysis industry. With the ever-improving availability of diode lasers and high intensity flash lamps (required as pulsed excitation sources for a low-cost, small-scale PAS-based oil analysis instrument), with the development of specialty PAS cells and instrumentation, the opportunities are excellent for not only a lab-based instrument, but also small-scale, portable and even on-line PAS-based sensors and instruments.

Just like with FTIR, any molecular species that shows a characteristic absorption peak has the potential for detection using PAS.

There may be opportunities to extend this PAS-based system to cover not just water, but other contaminants such as glycol and fuel, lube degradation by-products of oxidation and nitration, and even oil additives such as hindered phenols and ZDDP. Eventually, one may be able to buy a portable PAS-based diagnostic instrument, similar in size and cost to a portable particle counter that allows the detection of all these parameters and more - all at the press of a button!

Conclusion

This article provides a comprehensive review of the research topics most pertinent to the recent advancement of the high-sensitivity laser spectroscopic techniques based on a PA effect and their applications. The review brings together contributions from the researchers in the field of infrared PAS, covering various aspects of PAS technology from fundamental principles to applications and addresses the most important progress of PAS. The future prospects of PAS technique are also discussed. Over several decades, PAS has been theoretically and technically developed with the establishment of many newly derived PAS techniques to improve the performance or overcome the problems of the common PAS techniques. Currently, with the wide application in atmospheric and environmental monitoring and biochemical and biomedical analysis, we believe that PAS techniques have great potential in analytical chemistry and other disciplines in the coming years

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