Construction of a Low Cost Photoacoustic Spectrometer for Characterization of Materials

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Summary: During the past few years, another optical technique has been developed to study those materials, which cannot be studied, by the conventional transmission or reflection techniques. The present technique called *Photoacoustic spectroscopy* or PAS is different from the conventional techniques chiefly in that the interaction of the incident energy of the photons with the materials under investigation is studied not through subsequent detection and analysis of some of the photons, but rather through a direct measure of the energy absorbed by the material. The aim of this presentation is to highlight the construction of a simple *Photoacoustic spectrometer* which can easily be constructed even in high school and college laboratories with the available low cost but efficient components and use it for characterization of solid (opaque or transparent), liquid and gas samples under investigations. The essential parts of the photoacoustic spectrometer designed in the laboratory (MADURAI - PA SPECTROMETER), consists of three main components.The total cost comes around 900 Euros. It is an affordable cost for researchers working with paucity of funds and facilities and many constraints especially in the developing countries. In the next few years we aim to study material characterization using MADURAI –PA SPECTROMETER.

Keywords: low cost spectrometer; photoacoustics; photoacoustic spectrometer

Introduction

Optical spectroscopy has been a scientific tool for over a century and it has proven invaluable in studies on reasonably clear media such as solutions and crystals and on specularly reflective surfaces. There are, however several instances where conventional transmission spectroscopy is inadequate even for the case of clear transparent materials. Such situations arise when one is attempting to measure very weak absorption. In

 DOI: 10.1002/masy.200550438

addition to weakly absorbing materials there are a great many non-gaseous substances, both organic and inorganic, that are not readily amenable to the conventional transmission or reflection modes of optical spectroscopy. These are usually light scattering materials; such as powders, amorphous solids, gels, smears, suspensions and nanoparticles. To fill this gap, photoacoustic spectroscopy has come into existence. Photoacoustics (PA) is well known in research but it is less known to Physics students at undergraduate and postgraduate levels. In this article a simple and low-cost experimental design of the photo acoustic spectrometer is explained which can be easily designed in any developing laboratories.

The principle of photoacoustic is the generation of acoustic energy from modulated light energy. The resulting energy propagates away from the source as acoustic waves. That is, the photoacoustic effect is the generation of acoustic waves in a sample resulting from the absorption of photons. The sample may be solid, liquid, gas, powder, gel, thin films or nanoparticles. Alexander Graham Bell [1] discovered the photoacoustic phenomena in the year 1880. The PA effect is represented pictorially in Figure 1.

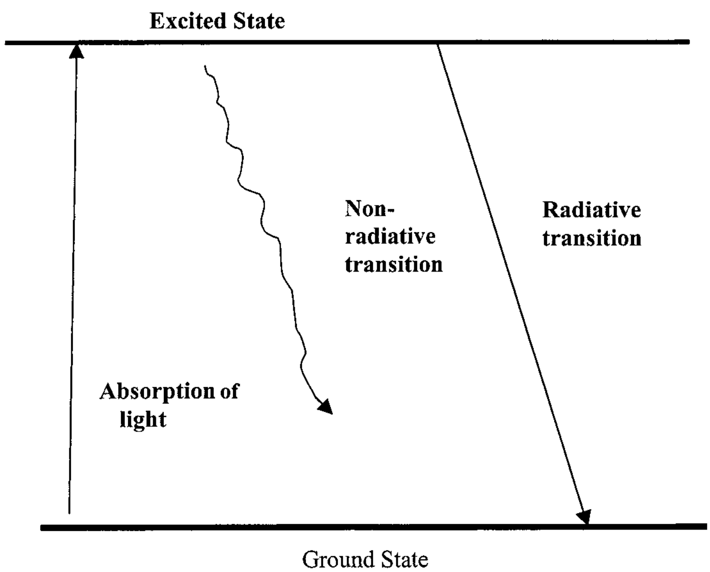

Figure 1. Origin of the photoacoustic spectrum.

Experimental

Construction of PA spectrometer

All the parts of the PA spectrometer were designed and assembled in School of Physics of Madurai Kamaraj University. The only readymade instrument was the digital storage oscilloscope, which was used to make measurements. The block diagram of the so-called 'MADURAI-PA SPECTROMETER' is shown in Figure 2.

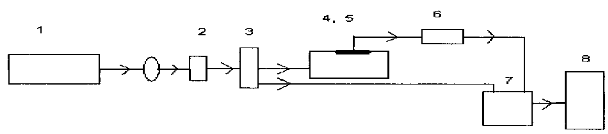

Figure 2. Block diagram of Madurai PA spectrometer; 1.Source; 2.Monochromator; 3. Chopper; 4. PA cell; 5. Microphone; 6. Pre-amplifier; 7. Lock-in amplifier; 8. Digital Storage Oscilloscope.

Source

The source is a 1000 watt tungsten halogen lamp [2] with the reflecting mirror and condensing lens arrangement, which can be moved, collimated the beam of light back and forth for convenience of focusing. A sturdy transformer was assembled to operate tungsten halogen lamp at 12 amperes a.c current and was tested for its performance.

Monochromator

Since the cost of monochromator is high we employed a simple procedure. Since it was intended to carry out the experiments in the visible region of the electromagnetic spectrum, we used colored filter papers (highly transparent) violet, indigo, blue, green, yellow, orange and red. The appropriate filter paper was pasted to a lens holder, which will act as a monochromator when white light passes through it. Care was taken to place the colored filter paper intact and clean.

Chopper

We have made the beam chopper from thick aluminum sheets. The aluminum sheets were cut accordingly so that it will convenient to vary the frequency of the chopper. Hence we cut out three blades of suitable dimensions. The shape of the chopper was that of a fan blade except that the blades are plane instead of twisted. Like this we have designed the different shopper with four blades and two blades. The number of blades increases the frequency of the chopping. This chopping blade with proper arrangements for rotations is connected to an a.c motor. The operating voltage for the a.c motor can be varied from 0-230V. Change in the motor speed changes the chopping frequency. This can be adjusted by changing the power supply voltage, which is variable from 0V to 230V. According to the required experimental frequency a proper chopper blade was selected from the 3 chopping blades. The chopper was placed near or at the focus of the lens. The shape of one of the chopping blades is shown in Figure.3.

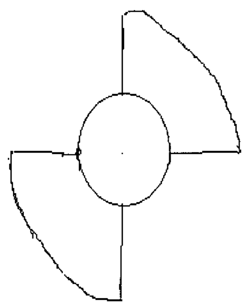

Figure 3. Two segments chopper blade.

Photoacoustic cell

The photoacoustic cell is one of the most important components that require skillful designing suitable for obtaining PA spectra according to the nature of the sample (solid, liquid, gas). For solid samples we have designed a PA cell as shown in figure 4. The PA cell is made up of a flint glass funnel whose larger diameter is 10 cm. A funnel of high thermal conductivity material such as metal may result in a weaker signal. Therefore glass funnel was used in our experiment. A cylindrical glass vessel to which liquid can be circulated and the sample in the cell can be kept at any desired temperature by connecting inlet and outlet to the constant temperature bath envelops the whole funnel. The interior of the glass funnel is coated with the lampblack. When opaque layer of carbon has been deposited over the entire interior of the funnel it acts as a backing material. The large opening of a funnel is sealed with watch glass using best adhesive paste. In the curved watch glass the convex side should be placed inside the funnel to reduce volume of the shield chamber and subsequently focusing of the beam. A thin sample holder made of brass/aluminum is placed inside the stem of the glass funnel and in front of the holder a sensitive microphone is placed perpendicular to a sample holder but without touching it. Thin electrical connecting wires are taken from the microphone without disturbing the airtight arrangement of the experimental PA cell setup. One of the detectors namely the microphone is placed inside the funnel which was inevitable. All the other detector devices are assembled in a separate chassis.

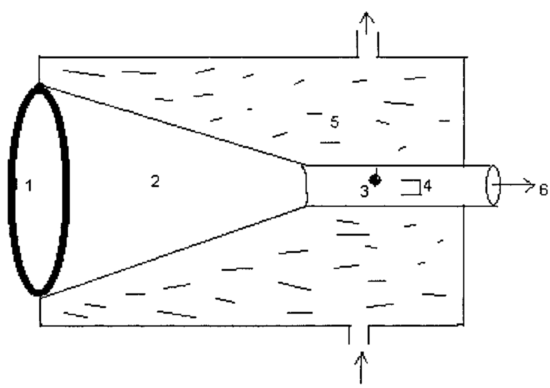

Figure 4. PA cell for solid samples; 1. curved watch glass; 2. lampblack coating; 3. microphone; 4. sample holder; 5. thermostat; 6. PA signal to detectors.

For liquid samples we have designed a unique PA cell as shown in figure 5. The PA cell is made up of a small glass funnel. In the center of the stem of the funnel and air tight compartment is drilled to insert a quartz container to hold the liquid sample. The microphone can be placed over the sample without touching it. The interior of the cell is coated with lampblack except the region containing the quartz cell. The entire is made airtight. The large opening of the funnel is sealed with the clean watch glass. For gaseous

samples we designed a innovative PA cell as shown in Figure 6. The cell is cylindrical in shape and there is provision for heating the gaseous sample at any desired temperature by externally circulating liquid system. For PA students of gaseous samples the entire cell should fill the gas and no sample holder is required. The interior of the cell is uniformly coated with lampblack. One of the ends of the cylindrical cell is completely sealed and is blackened inside with lampblack and the microphone is sealed inside the glass vessel.
The other end of the cylindrical glass vessel has an opening and a sliding door made of quartz glass will be sealing the entire cell.

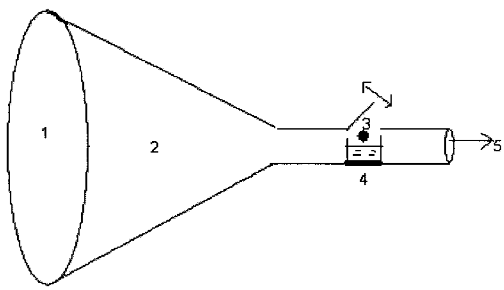

Figure 5. PA cell for liquid samples; 1.curved watch glass; 2. lampblack coating; 3. microphone; 4. quartz sample holder; 5. PA signal to detectors.

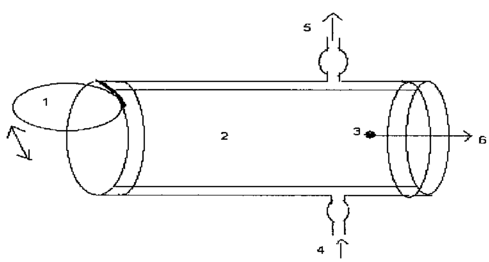

Figure 6. PA cell for gaseous samples; 1.quartz glass window; 2. lampblack coating; 3. microphone; 4. thermostat inlet; 5. thermostat outlet; 6. PA signal to detectors.

Detector

In all the three cells the two terminals of the microphone are projecting outside so that connections can be made easily without disturbing the PA cell. The microphone is connected to an external bias of 2V d.c supply. The other end of the microphone is used as input for the amplifier, which is designed with an IC 741 chip. A very simple and low cost IC 741 operational amplifier with negative feedback is used and the gain is maintained to be 100. This amplifier circuit is shown in figure 7. The power supplies that are needed for this amplifier are constructed with IC 7812 and IC 7912. The lock-in amplifier set up is made p of indigenous electronic components so that it selects only the audio signal, which is at the chopping frequency. A reference signal from the chopper is given to the lock-in amplifier. In the same lock-in amplifier set up which is kept in an iron chassis three digital displays are incorporated to measure chopping frequency, photoacoustic signal amplitude and phase of the photoacoustic signal. Provisions are made to give the output to the digital storage oscilloscope to trace the waveform and to check the PA signal amplitude and phase read by the digital displays are one and the same. In our experiment we used the digital panel meter to find the chopping frequency. The digital storage oscilloscope (DSO, 20MHz, GOULD) measured the PA signal amplitude and phase.

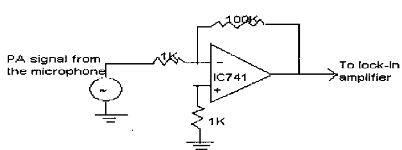

Figure 7. Circuit diagram of pre-amplifier.

Results and Discussion

To check the performance of the constructed "MADURAI - PA SPECTROMETER", we conducted experiments with a known solid sample, liquid sample and gaseous sample using the three different cells. The results that were obtained are encouraging and fall within an error of ±3% from the other references.

Solid sample

By keeping the wavelength fixed (green color filter was used) the depth profile analysis was carried out and the corresponding PA signal is measured by varying the chopping frequency. The sample that was tested was solid poly(methyl acrylate) synthesized in our laboratory. The characteristic frequency f_c was found out. This frequency is that point at which the sample goes from thermally thin to thermally thick state [3]. The thermal diffusivity of the sample was calculated and found to be $1.9 \times 10^{-6} m^2 sec^{-1}$. Jothi Rajan et al. [4] have reported for the same PMA sample with a sophisticated and very costly photoacoustic spectrometer (EG&G MODELS) the diffusivity value of $1.8 \times 10^{-6} m^2 sec^{-1}$. Thus the indigenously designed low cost set up is in no way inferior to the one that was imported.

Liquid sample

To study the thermal diffusivity of polyaniline in N-methylpyrrolidone (NMP) we used the liquid cell and followed the depth profile analysis and found the characteristic frequency. Hence we found thermal diffusivity to be $25.6 \times 10^{-6} m^2 sec^{-1}$. Pilla et al. [5] have reported the thermal diffusivity value for polyvinylacetate /polyaniline solution by thermal measurements as $1.05 \times 10^{-7} m^2 sec^{-1}$. This measurement is also fairly good and acceptable within the error limits.

Gaseous sample

We used the gas PA cell to study the photochemical deexcitation of NO_2 gas at a pressure of 10 torr. Here we followed the wavelength scanning method. The chopping frequency is kept fixed and the wavelength is changed and accordingly the PA signal amplitude is measured. Harshbarger and Robin [6] have got a similar type of spectra at a chopping frequency of 510 Hz. In our experiment the chopping frequency was fixed at 525 Hz. The spectra are compared are shown in figure 8. These are in good agreement with our results within the acceptable error limits.

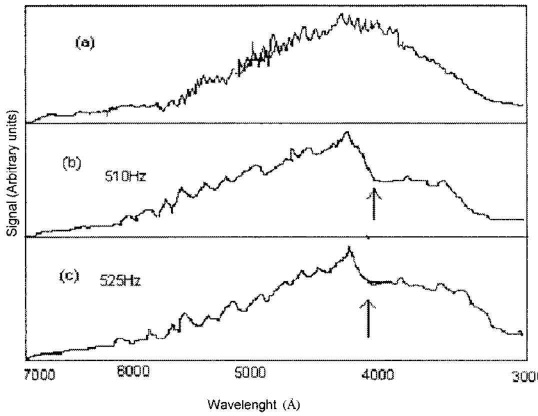

Figure 8. Comparison of the (a) optical and (b) photoacoustic spectra of NO_2 at 10 torr (c) photoacoustic spectra of NO_2 at 10 torr in the present work.

Conclusions

The indigenously and economically designed MADURAI - PA SPECTROMETER will be used for our future studies on nanocomposites of organic and inorganic polymer and biological samples. Since the PA technique is a nondestructive testing and evaluating tool with maximum facilities for obtaining best results of rare samples we intend using it and the same also fits within the limits of our funding. This technique gives fairly accurate results as any other conventional methods. This simple spectrometer can be constructed in college laboratories and number of innovative experiments can be carried out. The total cost of the entire set up came around 900 Euros.

In future we aim at using the photopyroelectric nature of the same phenomena. The designing of the low cost photopyroelectric spectrometer is in progress in our laboratory with the limited funding. We intend testing biological samples also by this technique and explore the various causes for the spread of viruses between human and animals.

Acknowledgements

One of the authors M.A.Jothi Rajan acknowledges the University Grants Commission of India for awarding FIP fellowship to carryout this work.

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