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Synchrotron FTIR Microscopy and Photoacoustic depth profiling of Polymer Laminates

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Introduction: Photoacoustic (PA-FTIR) depth profiling is a non-destructive experimental technique for probing the chemical microstructure in the vicinity of the surface. The technique is often applied to well-defined polymer laminate films. However, its applicability to finely divided laminate samples is less clear. In order to test the applicability of the PA-FTIR depth-profiling technique to finely divided polymer laminates we require a microscopic technique with sufficient spatial resolution and which will provide identical information to the PA-FTIR experiment. Synchrotron FTIR microscopy seems an ideal candidate.

The system chosen is a solid support resin used in combinatorial chemistry. The resin consists of a cross-linked polystyrene (PS) core encased in a covalently attached shell of polyethylene glycol (PEG) terminated with a functional group upon which chemistry can be performed to attach the reaction adducts for the synthesis. The PEG shell also provides a hydrophilic environment to facilitate certain classes of organic reactions. The laminate nature of the resin micro-spheres (90-120 microns in diameter) makes them ideal candidates for the depth profiling experiment.

Methods and Materials: No special sample preparation is required for the photoacoustic measurements. However, in order to make use of the synchrotron FTIR microscopy the samples must be sectioned to a thickness of about 10 microns. The beads were embedded in melted paraffin, cooled and then sectioned with a microtome at -30°C . The resulting sections were 10-12 microns thick and contained many circular beads of the right size to have been sectioned near the middle of the bead.

Results: the photoacoustic depth profiles and the synchrotron FTIR microscopy data are in full agreement. The synchrotron data confirm that outer PEG shell is 5-8 microns thick and the distinctive laminate structure seen in the photoacoustic data is the real configuration of the sample.

Conclusions: With the synchrotron FTIR microscopy data we have confirmed that the photoacoustic depth profiling experiment is a powerful tool for the analysis of all types of laminate samples, from polymer films to finely divided powders.

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