Forensic examination of textile fibres by pyrolysis GC/MS

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Introduction

Clothing may be made from a wide variety of fibres, both natural (such as wool or silk) and synthetic (such as nylon and polyesters). Polyamides (like nylon), polyethylene and poly-vinyl-chloride (PVC) are popular and are the most utilized materials in the textile industries of occidental countries. Textiles fibres can be simply transferred from a crime scene to a person and from a person to a crime scene because of their small heights: it is called the "Locard exchange principle" (coming from the Dc Edmond Locard of Lyon). Forensic scientists find textile fibres in crime scenes, and have to analyse this type of sample for evidence. The current analysis of fibers uses Infrared methods, observations with optic microscopes and SEM. A Pyrolysis-GC/MS optimized method fibre analysation isn't used in the chemical section of Lyon, yet this process could be very interesting to corroborate other analyses (like IR or MEB).

Experimental conditions

Analytical pyrolysis is the process that uses heat energy to break molecular bonds in large molecules, thereby creating smaller volatile compounds. These compounds can then be analyzed by gas chromatography with mass spectrometry. Since PVC and Polyethylene are chemically quite different, the analysis of these two fibres by Py-GC/MS is a simple task. Moreover, synthetic fibres are suitable for pyrolysis analysis, because they are bigger than natural fibres and produce a large variety of compounds by pyrolysis. Consequently, analysation of "man made" fibres is more rewarding than analysation of natural fibres.

Instrumentation:

Samples were pyrolyzed in a quartz tube using CDS Analytical Model 2500 pysolysis autosampler interfaced to an HP 6890 Agilent Gas Chromatograph associated with an HP5973 mass spectrometer detector.

Pyrolysis conditions: temperature 750°C for 15 seconds, with a "clean to vent" (mod of pyrolysis room cleaning).

Gas Chromatography conditions: the carrier gas was helium gas, and column was a HP5MS column (30m x 0.25mm and e_f 0.25µm). We used the "splitless" method for 1 min.

GC program of temperature: initial temperature 40°C for 2 min, ramp 10°C/min and final temperature 290°C for 1 min.

Detector: MSD with Scan range of [33-550uma].

Results

Figure 1 shows a pyrogram generated from a piece of PVC thread heated at 750°C for 15 seconds. The compounds are eluted between 4 to 21 minutes. Once PVC was degraded it generated product aromatics including benzene, benzoic acid, toluene and naphthalene. The Polyethylene, on the other hand, was degraded into organic compounds such as alcohols, alkenes and diolefines. Figure 2 shows a pyrogram of a polyethylene fibre in which compounds were eluted three by three. One alcohol was eluted between the diolfine and the corresponding alkene. Figure 3, shows the pyrogram of a cotton thread in which we can see that peaks are not perfectly separated, and their heights are rather small.

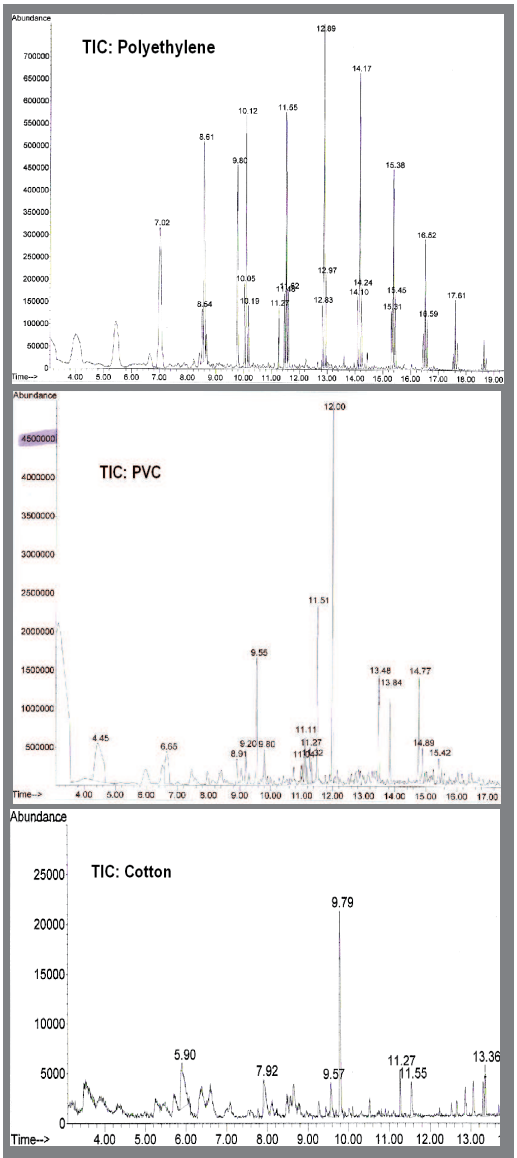

Fig 1, 2 and 3: Pyrograms (TICs) of Polyethylene, PVC, and Cotton fibers.

When a textile fibre is constituted by more than one type of fibre, each type when pyrolyzed produces its characteristic chromatographic peaks, so that the nature of the blended material may be elucidated.

Conclusion

Since synthetic fibres produce characteristic pyrograms, these fibres are easy to compare and analyse. In contradiction, natural fibres had to be analyzed with other methods like the IR and microscopic methods.

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