Analysis of Polymer Standards and Development of Searchable Libraries by FTIR-ATR and Py-GC/MS

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Abstract

•Evidence often encountered in forensic case, such as fibers, paints, adhesives, and tapes, contain both organic and inorganic constituents.
•Fourier Transform Infrared Spectroscopy (FTIR) is used to identify or characterize both the organic and inorganic components of a sample.
•Pyrolysis Gas Chromatography Mass Spectrometry (Py-GC/MS) provides analytical information on the organic component of samples, especially in samples where the organic component in the IR spectrum is masked by the presence of the inorganic constituent(s).
•Py-GC/MS helps detect and identify minor organic components in a multi-polymer mix.
•A FTIR library and a Py-GC/MS library were compiled to assist with the identification of forensic case samples.

Introduction

•FTIR and Py-GC/MS are two instrumental analysis techniques used to characterize and identify samples commonly encountered in forensic case samples such as fibers, paints, adhesives, and tapes [1,2] .
• FTIR is an established, non-destructive technique that can provide qualitative analysis of samples based on the absorption or transmittance of the infrared radiation as it passes through the sample[3].
• Py-GC/MS is destructive[4] since it breaks down compounds by exposing them to high heat for a very short period of time[5,6].
• Analysis using pyrolysis techniques may provide a greater discrimination power compared to infrared spectroscopy, but should be used as a complementary technique rather than used as a substitute for infrared spectroscopy[6].
• The mass spectrometer data analysis software allowed the creation of a combined mass spectrum of each sample, which was used to build the Py-GC/MS library.
• Libraries help forensic laboratories identify unknown samples.

Materials & Methods

100 polymer standards from the Scientific Polymer Products (SP^2) Polymer Sample Kit Catalog No. 205 were analyzed.

Polymers were analyzed on the PerkinElmer Spectrum One FTIR Spectrometer with Specac single reflection diamond ATR using the following parameters:
• Run from 4000cm^{-1} to 450cm^{-1}
• 8 scans with scan speed of 0.20cm/s
• Resolution of 4.00cm^{-1}

Polymers equal in volume to an equant 300um particle were analyzed using CDS Analytical, Inc. Pyroprobe 5000 series model 5150 with the PerkinElmer AutoSystem XL Gas Chromatograph and PerkinElmer Turbo Mass Gold Mass Spectrometer under the following parameters:
• Pyroprobe parameters:
  • Interface temperature of 300°C
  • Interface run time of 1 minute
  • Transfer line was set at 300°C
  • Sample was pyrolyzed at 750°C for 15 seconds with a ramp rate of 0°C

• Gas Chromatograph parameters:
  • Inlet temperature of 300°C
  • Column flow of 1.5mL/min
  • Split ratio of 50:1
  • Carrier gas was helium
  • Column used was a J+W DB-5MS (250um diameter and 30 meters long)
  • The oven temperature program:
    • Initial temperature of 40°C, held for 2 minutes
    • Temperature increased at 12°C/min until 280°C and held for 4 minutes
    • Total run time of 26 minutes

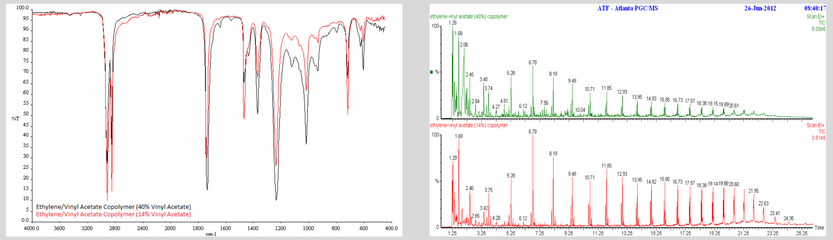

Figure 1. The IR spectra (left) and pyrogram (right) of Ethylene Vinyl Acetate Copolymers.

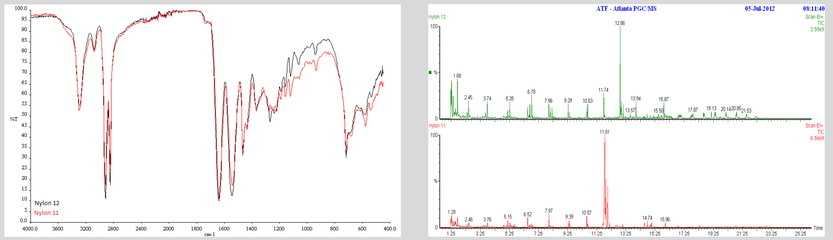

Figure 2. The IR spectra (left) and pyrogram (right) of Nylons.

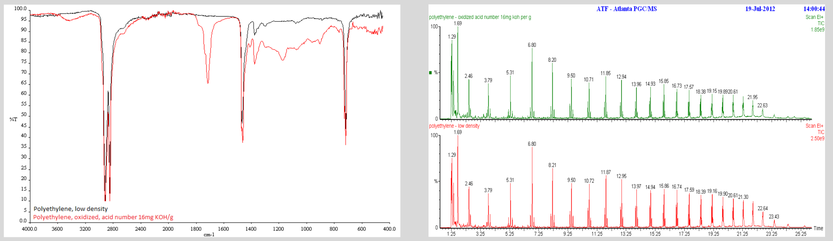

Figure 3. The IR spectra (left) and pyrogram (right) of Polyethylenes.

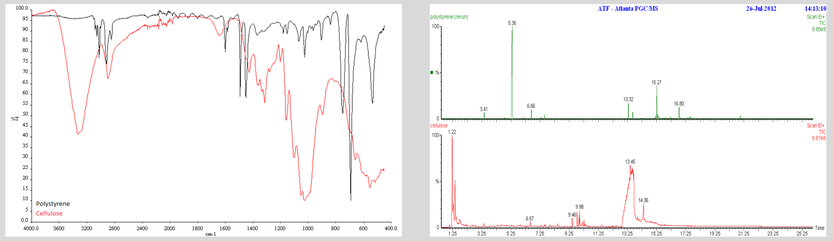

Figure 4. The IR spectra (left) and pyrogram (right) of Polystyrene and Cellulose

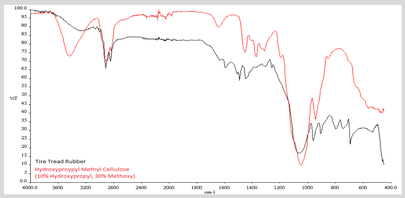

Figure 5. The IR spectra of tire tread rubber and hydroxypropyl methyl cellulose, the matching compound found with a library search.

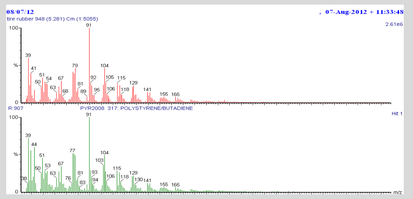

Figure 6. The combined mass spectrum match of the tire tread rubber when a library search was performed.

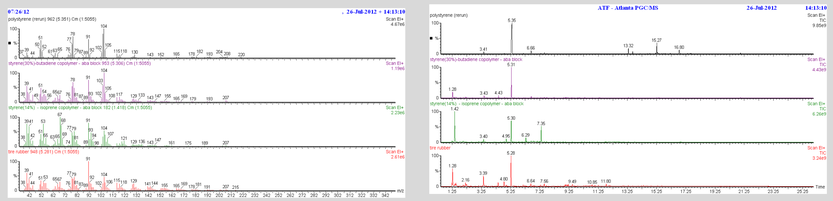

Figure 7. The pyrograms (right) and combined mass spectra (left) of tire tread rubber, polystyrene, styrene/butadiene copolymer, and styrene/isoprene copolymer.

Materials & Methods continued

Mass Spectrometer parameters:
  • Electron ionization mode
  • Set to detect a mass range of 33 – 350
  • Scans were collected from 0.5 minutes to 26 minutes every 0.2 seconds

Results

• FTIR and Py-GC/MS should be used as complementary techniques.
• FTIR library was created and shared with other ATF laboratories.
• Pyrograms can be used as a reference for comparisons.
• Combined mass spectra were created and used to build a searchable library.
• Libraries are useful tools for forensic laboratories.

Discussion & Conclusion

• FTIR and Py-GC/MS are two instrumental analysis techniques that can be used complementary to each other.
• The libraries created are useful tools in assisting with the identification of forensic case samples, but in no way replaces visual analyses.
• A combined mass spectrum eliminates problems caused by chromatographic conditions.
• For future work, FTIR and Py-GC/MS analysis of other polymer standards and known samples to add to the libraries will make the libraries a very valuable tool for forensic laboratories.
• Duplicate or triplicate runs of each standard will also ensure the spectra and pyrograms are reproducible and representative of the standard material.

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