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Application of infrared spectroscopy and pyrolysis gas chromatography for characterisation of adhesive tapes

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ABSTRACT

Infrared spectroscopy and pyrolysis GC/MS were applied in the comparative analysis of adhesive tapes. By providing information about the polymer composition, it was possible to classify both backings and adhesives of tapes into defined chemical classes. It was found that samples of the same type (of backings and adhesives) and similar infrared spectra can in most cases be effectively differentiated using Py-GC/MS, sometimes based only on the presence of peaks of very low intensity originating from minor components. The results obtained enabled us to draw the conclusion that Py-GC/MS appears to be a valuable analytical technique for examining tapes, which is complementary to infrared spectroscopy. Identification of pyrolysis products enables discrimination of samples. Both methods also provide crucial information that is useful for identification of adhesive tapes found at the crime scene.

1. Introduction

Adhesive tape fragments are found at various crime scenes. Electrical tapes can be used in the process of wiring electronic devices to bombs, whereas duct tapes can be utilized to bind victims of violent crimes, and other tapes – to wrap packages containing drugs, explosives or other threatening material. Frequently, forensic scientists are requested to compare fragments of a tape encountered at a crime scene with fragments originating from a suspect in order to establish whether they could have come from the same roll (have a common origin). Visual investigations can provide information about physical fit, tape width, colour and morphology [1]. Infrared spectroscopy (FT-IR) [2–5] and pyrolysis gas chromatography (Py-GC-MS) [4–7] enable characterisation of organic compounds in the backing layer and adhesive layer of the tape, and Inductively Coupled Plasma Mass Spectrometry (ICP/MS) and X-ray fluorescence (XRF) indicate their elemental composition [8–10].

However, the discriminating power of these methods is limited. FT-IR is used mainly to determine the class of adhesive and backing material used [10,11]. More information about organic compounds such as polymers and additives can be obtained using Py-GC/MS [11].

During pyrolysis, macromolecules of synthetic polymers are broken down by thermal energy into smaller fragments, which can be more easily identified. Additionally, minor components of a polymer sample may be visible as separate peaks in the pyrograms obtained. This enables one to find subtle structural or compositional variations in similar matrices. It was reported that this method provides greater discrimination of adhesive tapes than IR [12]. However, the reproducibility of the relative peak intensities in chromatograms is not as good as that in infrared spectra, and small differences do not lead to clear discrimination between different tapes. Therefore, statistical methods of evaluation of the results obtained should be applied [13–15].

Some authors have found that application of High Resolution Inductively Coupled Plasma Mass Spectrometry (HR ICP-MS) enables determination of profiles of trace elements present in tapes and could help in differentiation between different batches of the same type of tape [8,9], although the generally low metal concentration of adhesive tapes can limit the sensitivity of this approach.

As a non-destructive method, infrared spectroscopy is routinely applied in the forensic examination of different polymer samples. Being sensitive to molecular structure, it provides valuable

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information about the chemical composition of such materials as paints, plastics and glues [4,11]. The information obtained is in many cases sufficient for classification of these materials [16–20]. Some authors recommend the application of the attenuated total reflection (ATR) technique in the examination and comparison of polymer samples [2,21].

Generally, tapes consist of a backing film and an adhesive layer. The backing film is primed with a primer layer for improved adherence of the adhesive layer to the backing film. A release coating is applied on the back side of the backing film. During unwinding of the tape roll, this coating assists in reducing unwind tension and preventing adhesives from the tape from sticking to the back side of the tape beneath it. So, the tape is usually structured as follows (starting at the back of the tape): release coating/backing film/primer layer/adhesive layer. In principle, all these separate layers can be investigated. In practice, however, it is difficult to isolate the very thin primer layer and the release coating. In the present study, the tape was divided into two layers only, i.e. backing and adhesive (glue), and the two layers were investigated separately.

In this paper, the FTIR method was used to characterize adhesive tapes of different colours and types, and produced by different manufacturers. A great diversity of polymer composition was observed. The tapes were distinguished on the basis of IR data—visually taking into account the location and intensity of absorption bands — as well as by the use of principle component analysis. Discrimination between tapes belonging to the same chemical group was performed using Py-GC/MS.

2. Materials and methods

Both the glue and the backing of 50 adhesive tapes of various kinds (packaging tape, duct tape, and insulation tape) (Table 1) were examined with the use of infrared spectrometry. All tapes were purchased from supermarkets and stores at different times.

Infrared measurements were performed using an FTS 40Pro Fourier-transform infrared spectrometer (BioRad/Digilab), which was equipped with a water-cooled high temperature ceramic source (MIR) and coupled with a UMA 500 microscope equipped with an MCT detector. IR spectra in the mid-infrared range were recorded at a resolution of 4 cm^{-1}. Each spectrum represented a collection of 512 scans. The samples were placed directly on a KBr plate on the microscope stage of the spectrometer and measured by the transmission technique in an infrared beam. For the purposes of analysis, a small amount of glue was scraped off using a scalpel, then the rest of the glue was removed using an organic solvent and a small piece of clean backing was cut off.

For the Py-GC/MS analysis, a TurboMass Gold System (Perkin Elmer Instruments) was applied. Samples were pyrolysed with the use of a CDS 2000 pyrolyser (Analytix, USA). The GC program was: 40 °C held for 2 min; ramped 10 °C · min^{-1} to 300 °C; 300 °C maintained for 2 min; increased by 30 °C · min^{-1} to 320 °C; 320 °C maintained for 3 min. An RTx-35MS capillary column (30 m × 0.25 mm × 0.25 μm) was used. The stationary phase consisted of 35% diphenyl-polysiloxane and 65% dimethyl-polysiloxane. The carrier gas was helium. The electron ionisation temperature of the transferline was 240 °C, and the temperature of the ion source in MS was 180 °C. The scan range was 35–500 m/z. Pyrolysis was performed at 400 °C and 750 °C without derivatisation. Pyrograms of samples were compared and the presence or absence of peaks, their retention times and relative intensities were taken into account. An assessment of the repeatability of the measurements was performed for 3 different fragments of each of the three chosen tapes. It was found that the repeatability was satisfactory — very good for retention time and fair for peak heights.

3. Results and discussion

On the basis of the obtained IR spectra, adhesive tapes were divided into several groups of different composition of backing and glue (Table 2). Each group was named after the main polymer present in the samples. IR spectra were assessed visually in terms of the number, location and intensity of particular bands.

Table 1
Examined tapes.

No.	Tape	Colour	No.	Tape	Colour
1	**Prefecta**	colourless	26	**Tesaflex 53947**	black
2	**Painto**	grey	27	**Tesa perfect**	green
3	**Advance AT169**	red	28	**Grand**	colourless
4	**Advance AT175**	green	29	**Grand**	white
5	**Advance AT4**	brown	30	**Scotch 508**	colourless
6	**Advance AT6152**	white	31	**Scotch magic 810**	white
7	**Advance AT30**	colourless	32	**Scotch 550**	colourless
8	**Advance AT6102**	black	33	**Scotch Crystal 600**	colourless
9	**Advance AT272**	silver	34	**Scotch Removable 811**	white
10	**Advance AT202**	yellow	35	**Stick'n**	white
11	**Bizon**	green	36	**Scotch**	colourless
12	**Global System**	colourless	37	**Scley 914**	black-yellow
13	**Global System**	grey	38	**Goldtape**	grey
14	**Lobos**	colourless	39	**Goldtape**	brown
15	**Herlitz**	colourless	40	**Goldtape**	yellow
16	**Tesco**	brown	41	**Tape International**	yellow
17	**Tesco**	colourless	42	**Global point**	yellow
18	**Tesa- Fixing**	white	43	**Dalpo**	grey
19	**-**	red-white	44	**K2**	grey
20	**Blue delphin tapes**	yellow	45	**Diall**	colourless
21	**Blue delphin tapes**	yellow	46	**Tape International**	yellow
22	**Blue delphin tapes**	grey	47	**Purs**	black
23	**Tesa signal**	red-white	48	**Euro-tape silent**	colourless
24	**Tesaflex 53947**	blue	49	**Euro-tape silent**	brown
25	**Tesa- Fixing**	white	50	**Euro-tape**	yellow