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PAPER

Detection of microscopic particles present as contaminants in latent fingerprints by means of synchrotron radiation-based Fourier transform infra-red micro-imaging

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Synchrotron radiation-based Fourier transform infra-red (SR-FTIR) micro-imaging has been developed as a rapid, direct and non-destructive technique. This method, taking advantage of the high brightness and small effective source size of synchrotron light, is capable of exploring the molecular chemistry within the microstructures of microscopic particles without their destruction at high spatial resolutions. This is in contrast to traditional "wet" chemical methods, which, during processing for analysis, often caused destruction of the original samples. In the present study, we demonstrate the potential of SR-FTIR micro-imaging as an effective way to accurately identify microscopic particles deposited within latent fingerprints. These particles are present from residual amounts of materials left on a person's fingers after handling such materials. Fingerprints contaminated with various types of powders, creams, medications and high explosive materials (3-nitrooxy-2,2-bis(nitrooxymethyl)propyl nitrate (PETN), 1,3,5-trinitro-1,3,5-triazinane (RDX), 2-methyl-1,3,5-trinitrobenzene (TNT)) deposited on various – daily used – substrates have been analysed herein without any further sample preparation. A non-destructive method for the transfer of contaminated fingerprints from hard-to-reach areas of the substrates to the place of analysis is also presented. This method could have a significant impact on forensic science and could dramatically enhance the amount of information that can be obtained from the study of fingerprints.

Introduction

Fourier transform infra-red (FTIR) spectromicroscopy is a powerful technique being utilized in many applications. It is an attractive detection technique because it is non-destructive and very often requires little or no sample preparation, offering at the same time results with a very high degree of chemical uniqueness. In 1949 the FTIR technique was used in forensics science for the first time,[1] but at that time its applications were greatly limited as a rather large sample size was required for proper analysis. Thermal IR sources, such as a Globar source, allowed scientists[2] in the early 1990s to analyze samples as small as 75 μm. Nowadays, with synchrotron sources (hundreds of times brighter than conventional thermal infra-red sources) IR light can be focused down to a 3–10 μm spot size, giving a superior signal-to-noise ratio and better diffraction characteristics than using a Globar source. Such a resolution is "diffraction limited" and is very close to what could be achieved with an ideal point source.

Synchrotron infrared light is not destructive to the samples as it does not break any bonds or change the chemical formula. According to previous studies,[3] focused synchrotron infrared light increases the temperature of biological samples by about 0.5 K. The properties of SR allow the analysis of minute samples and the observation of details that may be "invisible" using a conventional IR source.

FTIR has become an increasingly valuable forensic technique within the last few decades because of its detection sensitivity and versatility. Chemicals from a variety of sample types including blood, drugs, fibers, paints, and polymer coatings can be easily identified, giving promising results for criminal investigations.[4–13] FTIR spectromicroscopy was heavily used by our group during the analyses of explosives and post-blast residues, unambiguously identifying traces of the explosives on a variety of debris.[14,15]

In the last decade FTIR spectromicroscopy has become one of the salient techniques in the study of fingerprints which is a very important aspect of criminal investigations and forensic detection.[16–21] This is because fingerprint identification is unique, being an individual characteristic; the likelihood of two human beings having the same fingerprints is infinitesimal, a pair of

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twins may share the same genetic code, but not fingerprint patterns.

So it is true that "the fingerprint is the pillar of modern criminal identification", because its success rate outclasses even DNA identification.

The significance of fingerprints was noted in ancient times; subtle references alluding to fingerprints can be found even in the Quran and the Bible. Fingerprints were found on documents from ancient Babylonia and on clay sealings from ancient China.[22] According to the literature[23–25] the ancient Chinese were the first people who used friction ridge impressions as a means of identification.

For more than a century, investigators have been identifying suspects by studying the unique shapes of their friction ridges, taking note of such details as where the lines end, bifurcate and diverge;[23] at the same time usually forgetting that material transferred by the fingerprints may give additional opportunity for a more thorough analysis.

Each ridge contains pores which are attached to sweat glands under the skin. Once the finger touches a surface, perspiration (along with oils from hair) is transferred onto that surface. The fingerprints are left on every item being touched by them mainly because of sweat. The latent fingerprint, placed on a surface, is a complex mixture of natural secretions and contaminants from the environment. Some of the residues left behind are from other surfaces or materials a person has touched – everything from everyday life such as sugar scattered on the table, pills taken, to incriminating particles of drugs or explosives.

The few micrograms of sweat and dirt (contamination) left behind in a fingerprint contain more than a physical pattern: the chemicals in the print are clues themselves, but they cannot normally be discriminated by their morphology alone as their shapes are irregular and do not provide any information about the chemical origin. The analysis of latent fingerprints is one of the very challenging tasks for forensic scientists as very often invasive methods which could destroy chemical traces present within fingerprints are required to develop a print.[26,27] New non-destructive methods providing a digital image of the fingerprint as well as chemical information about traces of contamination within fingerprints are needed to deepen investigations of fingerprints. Hence FTIR spectromicroscopy seems to be promising for using infra-red encoded information to make more accurate fingerprint identifications by determining the chemical compositions of traces present within fingerprints, which can be potentially a very valuable procedure for certain cases – for example in identifying a person accused of constructing home-made explosive devices, using a gun or handling and taking illicit drugs, by finding in his/her latent fingerprints the residues of certain ingredients.

Many techniques used nowadays in collecting trace evidence are usually invasive and destructive. The fingerprints can be destroyed irrevocably during the contamination collection, making them useless for further re-examination as forensic evidence. Using a FTIR microscope working in visual mode, particles can be carefully inspected and the area of interest can be selected prior to real analysis, IR mode enables the FTIR measurements.

In our approach we focused on the direct analysis of solid microscopic particles existing in latent fingerprints which were deposited on various substrates commonly found in everyday life, without any further sample preparation – as presented here the study shows the ability of FTIR spectromicroscopy to identify traces of contamination within fingerprints, leaving them intact for further analysis. A non-destructive method for the transfer of contaminated fingerprints from hard to reach places is also presented, giving the solution for the situation present at crime scenes, where the constituents of fingermarks left on various backgrounds cannot be identified and analyzed directly.

Special attention was paid to FTIR analysis of microscopic particles present as contaminants in latent fingerprints using synchrotron radiation as a tool for obtaining deeper and more meaningful chemical information of extremely small particles.

Experimental

All experiments were performed at the ISMI (Infrared Spectro/Microscopy) beamline at the Singapore Synchrotron Light Source. The infrared light is extracted from the edge region of dipole D1 of the compact superconducting electron storage ring Helios 2. The nominal source point is located at half the maximum field, *i.e.*, at 2.25 T. ISMI provides a state-of-the-art FTIR spectrometer and microscope to supply diffraction-limited spatial resolution to an ever-widening range of infrared spectroscopy experiments.[28] A synchrotron based source offers a versatile infrared source that covers a spectral range from 10 000 to 10 cm^{-1} and has considerable brightness advantages over conventional (thermal) IR sources, enabling experiments with high resolution and studies of extremely small samples.

All samples were analysed in transmission mode under the FTIR microscope Hyperion 2000 (Bruker Optics, Ettlingen, Germany). Slits defining the region of interest were used to analyze very small particles. Slit sizes were set to properly match the size of the analysed particles. FTIR spectra were collected by a liquid-nitrogen cooled MCT detector within the region 4000–400 cm^{-1}; 300 scans were co-added at 4 cm^{-1} spectral resolution. FTIR spectra for the background were taken in the region of substrates without fingerprint contamination in the same optical range. The ratio of sample spectra was taken against background spectra to give a transmittance (T) output. All spectra were presented in units of absorbance ($A = -\log T$) as a function of wavenumber, the number of waves per centimeter.

Analysis of various chemicals found as contaminants in fingerprints deposited on various substrates

The first task was to demonstrate the ability of FTIR spectromicroscopy to directly analyze the microscopic particles present as contaminants in latent fingerprints on challenging (from the perspective of FTIR analysis) substrates – found in everyday life, without any preprocessing or pretreating of the samples.

To prepare fingerprint samples, a volunteer's clean index finger (the hands were cleaned with ordinary liquid soap, rinsed in water and dried in air prior to sample preparation in order to eliminate any contamination) was rubbed over the forehead to ensure sufficient sebaceous secretions were present for easy transfer of substances of interest – various types of powders, creams, and medications, as well as explosive materials such as PETN, TNT and RDX. Pure explosive materials were provided

by the Singapore Police Force (SPF). Then the volunteer's finger was pressed into a small amount of well-mixed powder of a selected material, whereas for another sample preparation procedure – the finger directly touched the analysed substances in their raw (solid) forms. Any excess substance was brushed away with the other hand so that no powder was noticeable on the skin. This procedure, in our opinion, could mimic the real typical amount of powder transferred with latent fingerprints. The contaminated finger was then lightly pressed onto various porous and non-porous substrates (*e.g.* polyethylene wrap, sandwich bag, banknote, Mylar foil, *etc.*). No macroscopic traces of the chemicals used were visible on the substrate surfaces.

It is important to stress that the location of the fingerprints was known before collecting FTIR spectra. Prior to analysis, contaminants in fingerprints were located by using a visual light microscope (15× infrared Schwarzschild objective working in viewing mode). Photos of areas selected for analysis were taken by a video camera connected to the microscope.

Analysis of mixtures

In the case of there being various chemicals within the latent fingerprints, there is no chance to correctly identify them by looking under a microscope at only their shape or size. FTIR imaging and, in particular, spectromicroscopy, can be considered as chemical mapping where each pixel within an image corresponds to a complete FTIR spectrum that reflects the chemical composition at the analysed spot. Two dimensional (2D) scans done under FTIR spectromicroscopy were used to prove the fact that proper identification and distinction are possible, taking into account the unique IR spectral signatures of analysed chemicals. For these types of experiments, one substance (from substances of interest mentioned earlier) transferred onto a volunteer's finger was deposited onto Mylar foil, a second person provided a new chemical. 2D scans were performed on selected areas, with slits set to 15 μm × 15 μm, 300 scans were summed up at 4 cm^{-1} spectral resolution, and the number of points collected for one scan depended on the size of the area of interest.

Analysis of fingerprints from hard-to-reach places

To simulate a real case scenario – at a crime scene where fingerprints are deposited on various surfaces and hence analysis of themselves and their contamination seem to be impossible, other experiments were conducted. Contaminated by high explosive materials, fingerprints were enhanced with forehead sebum and laid directly on several different porous and non-porous substances such as cardboard, paper, glass, door knob, table, *etc.* Then they were collected by placing the various types of foil over the area of the fingerprints deposition. Prior to lifting the fingerprints, the foil was gently smoothed over the region of interest, detached and transported for further FTIR analysis.

Results and discussion

Analysis of various chemicals found as contaminants in fingerprints deposited on various substrates

Particles present in contaminated fingerprints were scattered sparsely on the substrates as shown in Fig. 1. Usage of the system

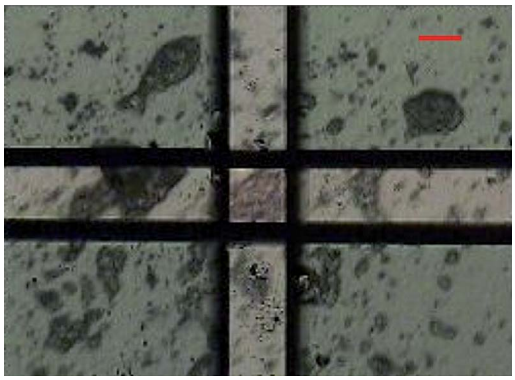

Fig. 1 Photo of aspirin particles scattered sparsely on polyethylene wrap, viewed under a Hyperion 2000 microscope (scale bar denotes 25 μm); black stripes show the vertical and horizontal slit system used for matching the size of the analysed particles.

of slits enabled the collection of FTIR spectra from spots where the particles were located.

All spectra obtained by FTIR contain their own set of different peaks as a function of wavenumber [cm^{-1}]. After experiments, they were analysed using Opus 6.0 software supplied by the manufacturer of the spectrometer.[29] Analysis included a baseline correction, normalization procedures, and peak-picking analysis (qualitative analysis) as well as preliminary statistical interpretation.

The relative peak height and the spectral position in a FTIR spectrum provide a unique characterization for any molecule that responds to infrared radiation.

As an example, FTIR spectra collected for aspirin particles deposited on a 2 Singapore dollars banknote, foil from a sandwich bag, and polyethylene wrap are presented in Fig. 2. Prior to the experiments, pure (*i.e.* without contaminated fingerprints) substrates were measured and treated as backgrounds. Each substrate contains their own lines, but they were compensated well during further procedure, except for some regions where the IR radiation is blocked completely (seen in spectra as a noise in

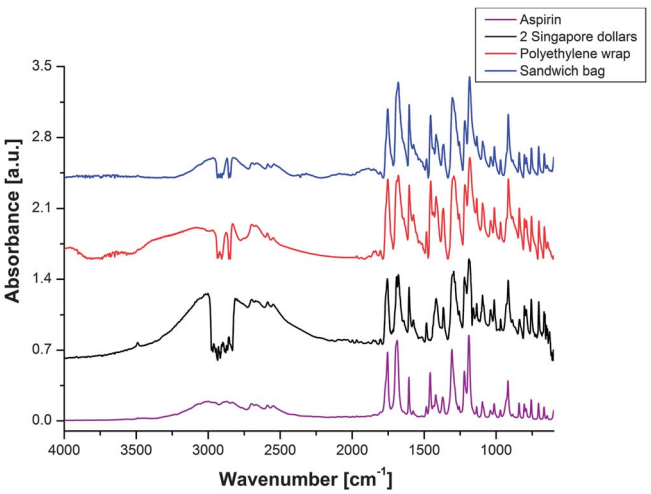

Fig. 2 FTIR spectra collected for aspirin particles found in fingerprints deposited on a two Singapore dollar banknote, polyethylene wrap and foil from a sandwich bag. For comparison a FTIR spectrum for pure aspirin is presented.

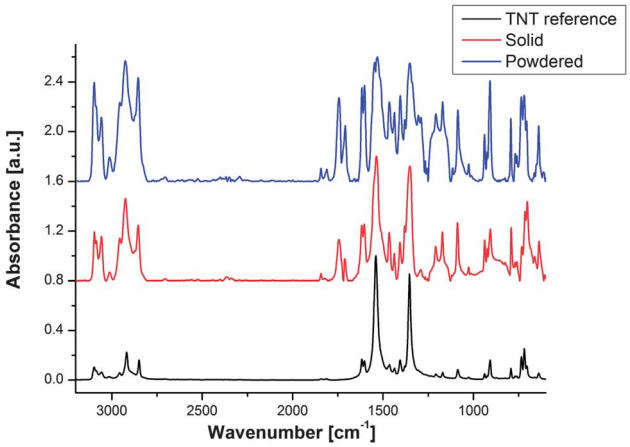

Fig. 3 FTIR spectra collected for TNT found in fingerprints deposited on Mylar foil; the red line (solid) denotes the FTIR spectrum for which samples were prepared by the direct touch of solid TNT (HQ = 0.108) whereas the blue line (powdered) presents the FTIR spectrum for a sample prepared by touching previously ground high explosives (HQ = 0.182). FTIR spectrum for pure TNT (TNT reference) is shown for comparison.

the 3000–2800 cm^{-1} region) but it does not mask the so-called "fingerprint region" of 1500–400 cm^{-1} rich in absorption bands which are highly specific to the analysed material.

A detailed analysis revealed that all FTIR spectra collected for various substances present as contaminants within a latent fingerprint deposited on different backgrounds contain the lines characteristic of pure substances. The positions of these lines and their relative ratios are in agreement with FTIR spectra collected for pure chemicals saved in our own database as the reference spectra, which can be seen in Fig. 2, where the FTIR spectrum for pure aspirin is presented. All important lines for aspirin, including those for C═O stretching (carbonyl groups) (1780, 1750 cm^{-1}), C–O stretching (1150, 1100 cm^{-1}), C–O–H (1150 cm^{-1}), C–O–C (1100 cm^{-1}), C–H stretching (800–500 cm^{-1}) are found in experimental data presented in Fig. 2.

This procedure has proved that FTIR spectromicroscopy can be successfully used to analyze the particles within latent fingerprints directly on various substrates without any additional sample preparation and intimate physical contact with the sampling area.

An analysis of the contaminants in latent fingerprints belonging to a suspected person using FTIR spectromicroscopy could allow the detection of traces of explosive materials, as proved in our analysis for PETN, TNT and RDX. In the first case, after using natural sebum from his/her own forehead, the volunteer directly touched the chemical and deposited the contaminated fingerprint onto Mylar foil. In the second case a person lightly touched the powdered explosive and transferred the material onto a substrate. After experiments all FTIR spectra were searched in our own database and the best matches for each spectrum were ranked in order of matching percent.

In Fig. 3 FTIR spectra collected for samples with TNT were compared with the reference spectrum. For both cases their

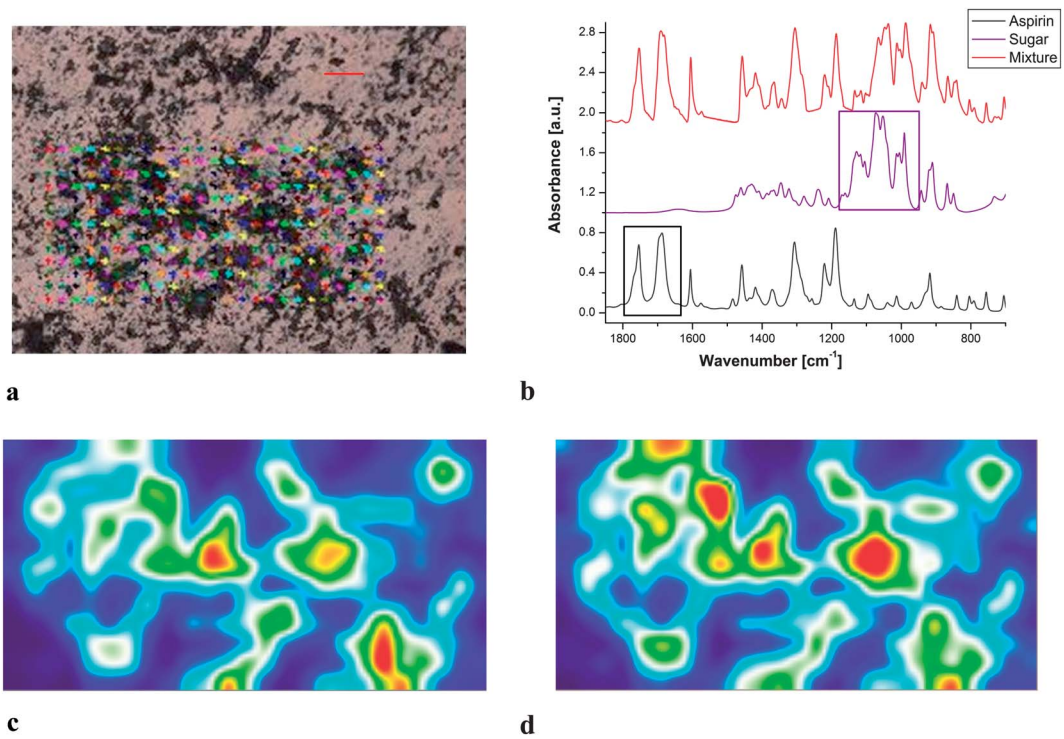

Fig. 4 Analysis of mixtures. (a) A bright-field image of part of a fingerprint contaminated by the mixture (aspirin and sugar) (scale bar denotes 100 μm). The coloured points denote the points from the 2D scan selected for analysis. (b) FTIR spectra collected from various spots of the fingerprint contaminated by sugar and aspirin; an example of spectra identified as pure aspirin, sugar and a mixture of both ingredients. Distribution of aspirin (c) and sugar (d) within the fingerprint deposited on Mylar foil. 2D maps were prepared by calculating the area characteristic for sugar or aspirin lines marked on the respective FTIR spectra (b).

FTIR spectra contain characteristics of TNT lines, thus revealing its chemical signature, but very often these lines are overlapped by another one, probably coming from the fingers residue. Spectra identification was also done by using the IDENT method available in the OPUS software. The aim of an IDENT analysis was to determine the differences between a test spectrum and the reference spectra of the library. The results are presented as Hit Quality (HQ) values within the range from 0 denoting a perfect match to 2 denoting no match.

In this way a preliminary quantitative assessment of the presence of traces of high explosive material in the spectra of particles found in latent fingerprints was performed. Low values of HQ (around 0) for the FTIR spectra depicted in Fig. 3 reveal a high similarity of these spectra to the FTIR spectrum for pure TNT.

Analysis of mixtures

As was mentioned earlier, several mixtures consisting of two various chemicals were prepared and 2D scans were performed on selected areas. One of them, a mixture of aspirin and sugar, is presented in Fig. 4a. Selecting random spectra from the different points shows that some of them have lines belonging only to aspirin or only to sugar, whereas some of them show evidence for being a superposition of the spectra of sugar and aspirin (Fig. 4b).

By calculating the area under all the lines characteristic of sugar or aspirin separately and presenting them in the form of a 2D map it is possible to measure their local distribution. Bearing in mind that in FTIR spectra, lines characteristic for aspirin and sugar can coexist in the same region, a new image based on the integrated area between 1190 and 970 cm^{-1} for sugar and 1813–1635 cm^{-1} for aspirin which correlate to the strongest lines for aspirin and sugar, was prepared. As can be seen in Fig. 4, this method of presentation enables one to observe the local distribution maxima for either aspirin (Fig. 4c) or sugar (Fig. 4d). This experiment proves that proper identification and distinction in the analysis of mixtures of substances is possible, taking into account their unique IR spectral signatures.

Analysis of fingerprints from hard-to-reach places

In the case of samples lifted from different porous and non-porous substances before being analysed in transmission mode, Mylar foil was found to be an appropriate medium to transport the contaminants of trace amounts of explosive material fingerprints and to make the FTIR experiments possible. Prior to sample analysis, a background for clean Mylar foil was collected, allowing for complete compensation of its own chemical composition in the final spectrum.

Fig. 5 shows FTIR spectra collected for materials taken from cardboard and doors having almost all lines belonging to the chemical signature of PETN. However, due to the rather indirect way of sample collection, in these spectra one can also notice lines characteristic for residues coming from other substances. They may influence the line shapes belonging only to PETN, but they do not mask identification of PETN within this spectrum. Calculated Hit Quality values for the spectra presented in Fig. 5 are 0.356 and 0.436 for samples collected from a door and cardboard respectively.

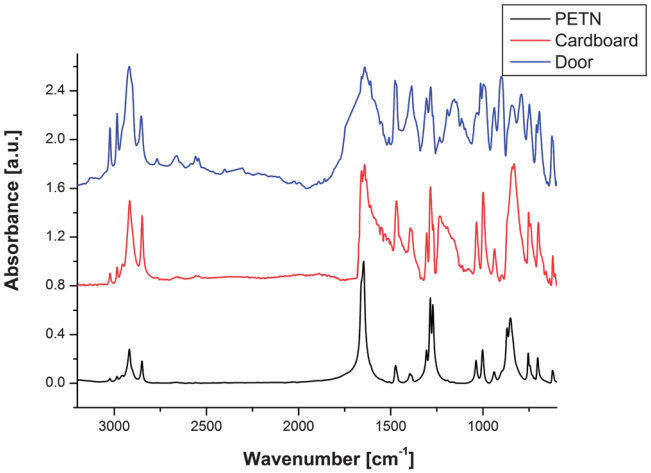

Fig. 5 FTIR spectra collected for PETN traces found on cardboard and a glass door; as a reference FTIR spectrum for pure PETN is presented in the lower part of this figure.

As shown, Mylar-lifted samples provide a means of obtaining fingerprints from inaccessible objects and a means of analyzing them using FTIR spectromicroscopy.

Advantage of synchrotron radiation over Globar source

Frequently, analysis of particles in latent fingerprints is limited by their size – only particles bigger than 20 μm in diameter can be easily analysed by FTIR spectromicroscopy working with a Globar or any other conventional IR source. IR light based on synchrotron radiation can be focused down to a few μm spot size and superior signal-to-noise ratio capability and better diffraction characteristics than using a Globar source can be obtained. Fig. 6 depicts the comparison of FTIR spectra collected for a small RDX particle found within fingerprints measured under the same experimental conditions (slits set to 4 μm by 4 μm) and over the same period of time using SR or laboratory (Globar)

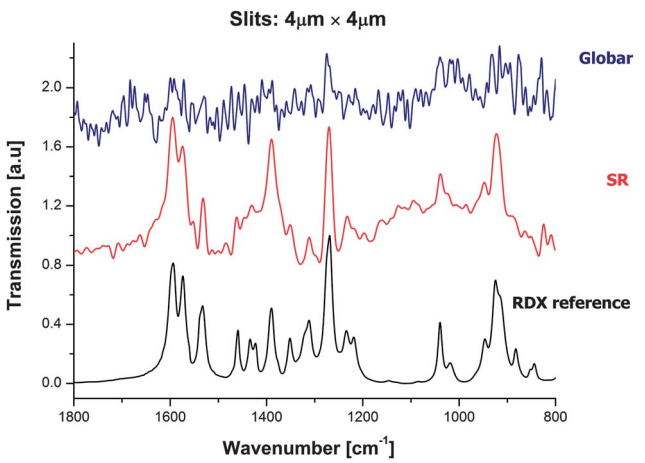

Fig. 6 FTIR spectra collected for a small RDX particle found within fingerprints measured under the same experimental conditions (in transmission mode, slits set to 4 μm by 4 μm) and over the same period of time using SR (red line), or laboratory (Globar) (dark blue line), source respectively. The black line represents FTIR spectrum for pure RDX (reference spectrum).

source respectively. In both cases, 300 scans were co-added at 4 cm^{-1} resolution. The improvement in brightness at the synchrotron enhances the signal-to-noise ratio for small samples. For analysis of traces smaller than 5 μm, only the use of SR guarantees the collection of FTIR spectra with well-resolved peaks while spectra taken using a Globar source have a low signal-to-noise ratio which leads to loss of all details about peaks belonging to the analysed substance. The Hit Quality value calculated for the FTIR spectrum collected using SR is 0.364, allowing positive classification of this result as a RDX FTIR spectrum.

In the case of a FTIR spectrum obtained using a Globar IR source, the Hit Quality value was 1.156, showing the lack of agreement between the analysed spectrum and the FTIR spectrum for RDX.

Conclusions

This study has shown that FTIR spectromicroscopy is a rapid, powerful and nondestructive tool for identifying small traces of contaminants such as various types of chemicals, including drugs and high explosives, from the latent fingerprints, leaving them intact for further biometric analysis. Based on the performed experiments it is concluded that fingerprint information could be useful in identifying or confirming a suspect's involvement in a crime. While fingerprints are unique to an individual, there is more information present than just the fingerprint pattern. FTIR microspectroscopic analysis can elicit chemical information left behind with the fingerprint. Usage of mid-IR spectroscopy presented in this paper is based on molecular absorptions that are characteristic for specific chemical composition of materials being analysed. The non-invasive nature of this technique also allows the subsequent use of samples for further analysis. The straightforward chemical imaging is not restricted by additional sample preprocessing, pretreating or the addition of nanoparticles. Direct analysis of even challenging (from the point of FTIR analysis) substrates, such as a banknote containing not only paper but various inks, foils, and fibres, provided accurate and positive results. Using Mylar foil as a lifting medium allowed for the analysis of the contaminated fingerprints deposited earlier on various non-porous and porous substrates (cardboard, door, table) – that were difficult to access. The method also preserves the integrity of samples, allowing for further analysis.

The traces of various chemicals in fingerprints cannot be classified by their own morphology but since the mid-IR region is capable of discriminating between different molecules, the data collected can be used to visualize chemical differences across the sample, providing a spatial distribution of the component materials, *ipso facto* FTIR spectra can successfully differentiate various substances by identification of individual constituents. Direct identification of analysed chemicals was possible by searching their spectra in an existing database in order to find the matching spectra of a known substance.

To meet the increasing demand for the analysis of minute samples, synchrotron radiation was used in the experiments, making possible the accurate analysis of very small particles of 4 μm diameter in which spectra contain well resolved peaks, greatly improving positive identification of the chemicals present. Synchrotron radiation is not heavily used in forensic sciences, but definitely these facilities should be employed in case of highly important and difficult to analyse samples.

Of course the experiment results presented here should be treated as a preliminary approach to broad forensic topics – they were done in a controlled way – the place of latent fingerprint deposition as well as substances being the source of contamination were known *a priori*. More work on less controlled experiments with more realistic cases is needed before applying this method to routine police investigation, but in this case a rich database and powerful algorithms could be necessary for positive identification of various chemical traces left among ridges in the latent fingerprints.

Additionally, a FPA (Focal Plane Array) detector could greatly improve this type of experiment, allowing for the identification of not only traces of contaminants in the latent fingerprints but also for reconstruction with sufficient detail of the latent fingerprint itself, allowing for biometric analysis in order to identify a suspect who had committed a crime. By using the FPA detector a grid of spectra is obtained in approximately the same amount of time that is required to acquire one spectrum with a single-element detector. By simultaneously acquiring thousands of spectra within minutes, FPA detectors provide information about the identification and concentration of specific compounds and their distribution in the measured field of view.

Fingerprint analysis, based on the unique pattern of human ridges, is the oldest method used in forensic science for personal identification of people involved in a crime scene. We believe that the preliminary results presented here have proved that FTIR spectromicroscopy can be treated as an invaluable tool for deeper insight into fingerprint analysis providing a plethora of chemical, reliable, unambiguous information inaccessible by any other techniques. Additionally, FTIR spectromicroscopy could be heavily used as a very trustworthy technique in other forensic analysis as this method offers high spatial resolution and high sensitivity, which are absolutely necessary for crime scene investigations offering many examples of vibrationally active samples such as blood smears, body fluids and tissues.

Identification of compounds in the samples must be done unambiguously so that the evidence could stand up in a court of law.

We believe that FTIR spectromicroscopy together with proper statistical evaluation will become one of the workhorses of contemporary forensic science delivering reliable data in a fast manner without compromising the integrity of the evidence.

Acknowledgements

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