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Rapid *in situ* detection of street samples of drugs of abuse on textile substrates using microRaman spectroscopy

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ABSTRACT

Trace amounts of street samples of cocaine hydrochloride and N-methyl-3,4-methylenedioxyamphetamine (MDMA) on natural and synthetic textiles were successfully detected *in situ* using confocal Raman microscopy. The presence of some excipient bands in the spectra of the drugs did not prevent the unambiguous identification of the drugs. Raman spectra of the drugs were readily obtained without significant interference from the fibre substrates. Interfering bands arising from the fibre natural or synthetic polymer structure and/or dye molecules did not overlap with the characteristic Raman bands of the drugs. If needed, interfering bands could be successfully removed by spectral subtraction. Also, Raman spectra could be acquired from drug particles trapped between the fibres of highly fluorescent textile specimens. The total acquisition time of the spectra of the drug particles was 90 s accomplished non-destructively and without detachment from their substrates. Sample preparation was not required and spectra of the drugs could be obtained non-invasively preserving the integrity of the evidential material for further analysis.

1. Introduction

Clothing from suspects and victims is valuable evidence forensically especially for serious crimes such as those involving firearm-related offences [1–3] and rape [4]. Genetic identification of individuals using blood or semen stains on clothes is also an important step in the investigation [5,6]. Smuggling cocaine using clothing impregnated with the drug has also been reported in a forensic context [7]. Forensic toxicologists have been utilizing garments belonging to drug abusers for the detection of drugs of abuse and their metabolites [8–10].

Traditionally, seized drugs are analysed by qualitative wet chemical spot tests and different chromatographic (HPLC, GC) [11,12], or hyphenated chromatographic spectroscopic techniques (GC–MS) [13]. Typically, these techniques involve removing the drug of interest from its container or environment, and detachment from its substrate via solvent washing or thermal desorption; all require destructive chemical analytical procedures and separation processes.

Raman spectroscopy has been shown to be an effective technique for the forensic screening of drugs of abuse and it has a number of distinctive advantages over other chemical analytical techniques. Raman spectroscopy produces molecular-specific spectra and, in most cases, sample preparation is minimal, permitting a non-destructive analysis of bulk or microscopic samples *in situ* without effecting the separation of the analyte from its substrate or host matrix. This is particularly important with regard to the speed of analysis, prevention of sample contamination and preservation of evidential material [14]. Unlike IR spectroscopy, water has a very weak Raman signal and so Raman spectra can be readily obtained from aqueous solutions or moist materials [15]. Raman spectroscopic techniques have been applied successfully for the identification of ecstasy [16], cocaine [17], barbiturates [18], and benzodiazepines [19]. They have also been used for the quantitative analysis of drugs of abuse in admixture with caffeine, anhydrous alpha D-glucose, mannitol, lactose, maltose, talc powder, flour, and baby formula [20–22].

The transportation, packaging, sale and use of drugs of abuse will almost inevitably cause contamination of the clothing, premises and other possessions of the persons involved [23]. Money contaminated with drugs can be directly associated with drug-related crimes and is frequently used as part of the evidence trail to establish a link between an individual and these substances; a limited number of studies have demonstrated the use of Raman microscopy for the detection and identification of illicit drugs and related substances on paper currency [24,25]. The clothing of persons involved in drug-related activities will inevitably became contaminated with these drugs resulting from microscopic particles of the drug being physically trapped between the fibres of the specimens. So, the detection of illicit drugs residues on clothing can be used as a strong indicator to establish a link between these illicit substances and individuals involved in these activities.

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In an earlier study in our laboratory, we established the use of confocal Raman microscopy for the *in situ* detection of drugs of abuse on undyed natural and synthetic fibres, and coloured textile specimens [26] from which interpretable confocal Raman spectra of drugs of abuse, namely pure samples of cocaine hydrochloride and MDMA, could be acquired without significant interference from the textile substrates. Illegal drugs are generally mixed with a variety of materials to increase the bulk of the sample prior to distribution. In such situations, the identification of drugs of abuse by Raman spectroscopy may be difficult due to several complications arising from the diluent matrix. The primary major complication is overlapping of the Raman bands of the excipients and the drugs of abuse which may impede the identification of the drugs. The second major complication lies in the presence of fluorescent contaminants that can conceal the Raman signal through background emission, thus making identification of the drugs difficult or impossible. Furthermore, Raman spectral bands or background fluorescence which may arise from the fibre polymers or dyed textiles can exacerbate the problem of identification. In the present work we will explore the application of the confocal Raman microscopy to the detection of street samples of drugs of abuse, that have been seized by security agencies, on a similar range of textile substrates. Here, we shall demonstrate that confocal Raman microscopy offers an excellent choice for the identification of trace amounts of illicit drugs in matrices of forensic relevance. This approach required no sample preparation and data could be readily obtained non-destructively and rapidly allowing for frequent sample analytical replication. The high spatial resolution of the confocal approach has allowed the Raman analysis of discrete micron-sized particles of street samples of illicit drugs trapped between the fibres of different types of clothing and demonstrated that confocal Raman microscopy can be effectively applied for acquisition of spectra of several drugs of abuse without significant interference from the clothing substrates.

2. Experimental

2.1. Samples

2.1.1. Drugs

Street samples of cocaine hydrochloride and MDMA were used in this study. Based on liquid chromatography–mass spectrometry (LC–MS), the concentrations of the samples were 26% and 41%, respectively. The samples were supplied by the Home Office Scientific Development Branch. Pure cocaine hydrochloride and MDMA samples were supplied by Sigma-Aldrich Company Ltd., United Kingdom.

2.1.2. Fibres and textiles

A set of natural and synthetic fibres was used in this study in an attempt to cover a wide range of textile materials used in real life. Natural fibres included wool, silk and cotton. Polyester fibres were used as a representative of synthetic fibres. Also, pieces of blue denim and an orange-coloured T-shirt were used in this study as representatives of dyed clothing commonly used on an everyday basis. A bundle of fibres, each about 1 cm in length, and textile pieces (~2 cm × 2 cm) were doped with each of these drugs and then presented to the spectrometer. Trapping of the drug particles between the fibres of the specimens was achieved by pressing small quantities against the fibre bundles and textile pieces and removing the surface excess of drug by gentle brushing. In all cases, the presence of drug particles on the textiles was not visible to the naked eye.

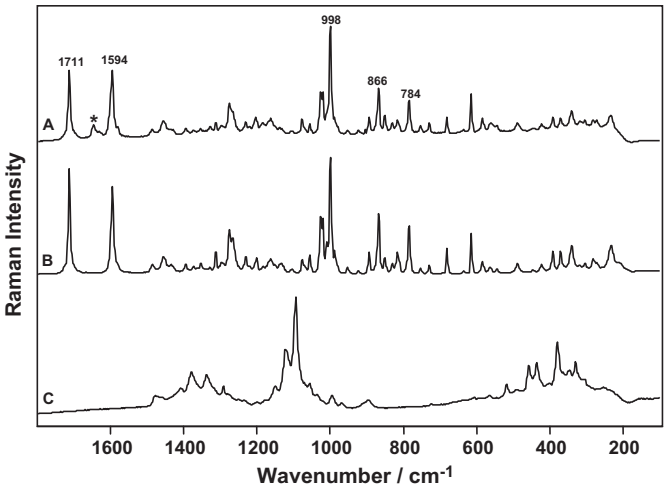

Fig. 1. Raman spectra of: A: street cocaine.HCl between cotton fibres (asterisks indicate excipient bands); B: reference cocaine.HCl; C: cotton fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

2.2. Raman spectroscopy

Reference Raman spectra of the pure drugs, fibres and textiles were obtained and used as reference spectra for comparison with the spectra of the street drugs particles trapped between the fibres of the specimens. Raman spectra were collected from drug particles with dimensions in the range 5–15 μm. Raman spectra were collected using a Renishaw *InVia* Reflex dispersive Raman microscope. The Raman scattering was excited with a 785 nm near-infrared diode laser [Renishaw HPNIR laser (Renishaw, Wotton-under-Edge, UK)] and a 50× objective lens giving a laser spot diameter of 5 μm. Spectra were obtained for a 10 s exposure of the CCD detector in the wavenumber region 1800–100 cm^{-1} using the extended scanning mode of the instrument. With 100% laser power, one accumulation was collected for the drug particles and five accumulations were collected for the fibres and textiles. The total acquisition time of the spectra of the drugs on the fibres was about 90 s. Spectral acquisition, presentation, and analysis were performed with the Renishaw WIRE (service pack 9) and GRAMS©AI version 8 (Thermo Electron Corp, Waltham, MA, USA) softwares.

3. Results and discussion

3.1. Drugs on undyed natural fibres

A confocal Raman microscope spectrum obtained from particles of seized cocaine hydrochloride trapped between cotton fibres is shown in Fig. 1A. A direct comparison of this spectrum with the reference spectrum of cocaine hydrochloride reveals that the identity of cocaine hydrochloride is readily established. The Raman spectrum of cocaine hydrochloride has several distinguishing features that can be used to identify the drug; such as the benzoate ester (C=O) stretch at 1711 cm^{-1}, the aromatic ring (C=C) stretch at 1594 cm^{-1}, the aromatic ring breathing mode at 998 cm^{-1}, the pyrrolidine ring (C–C) stretch at 866 cm^{-1}, and the piperidine ring (C–C) stretch at 784 cm^{-1} [27]. In Fig. 1A, the additional small peak adjacent to the aromatic ring (C=C) stretch at 1594 cm^{-1} is due to an adulterant present in the sample. This adulterant presented no difficulty in determining the identity of cocaine hydrochloride. It is also observed that no peak in the spectrum can be assigned to the cotton fibre substrate. Also, Figs. 2 and 3 show the Raman spectra collected from particles of seized cocaine hydrochloride trapped between the fibres of silk and wool, respectively. Apart from the

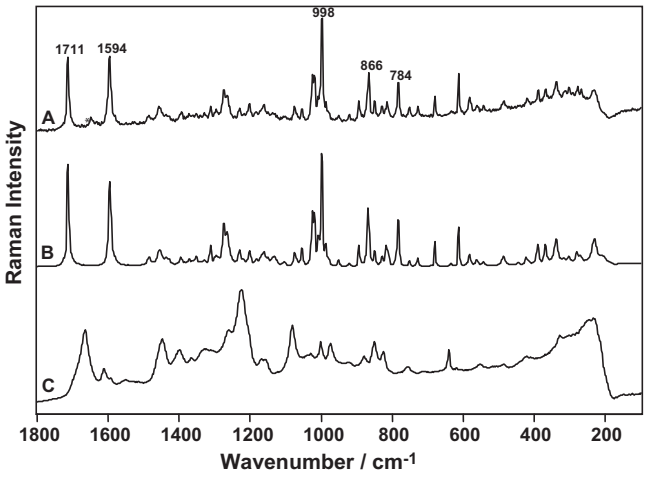

Fig. 2. Raman spectra of: A: street cocaine.HCl between silk fibres (asterisks indicate excipient bands); B, reference cocaine.HCl C: silk fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

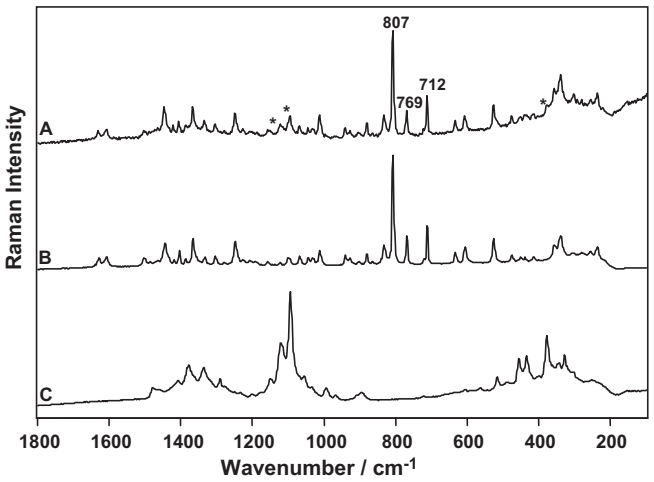

Fig. 4. Raman spectra of: A, street MDMA between cotton fibres (asterisks indicate cotton bands); B: reference MDMA; C: cotton fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

bands arising from the adulterant (marked with asterisks in the figures), no significant bands can be attributable to the silk or wool substrate. In each case, the characteristic signature bands of cocaine hydrochloride can be clearly identified.

Similar results were obtained from MDMA particles trapped between the fibres of cotton, silk, and wool specimens. In each instance, trapping of the drug particles between the substrate fibres presented no difficulty in determining the identity of the drug as MDMA. The Raman spectrum of MDMA has several diagnostic features that can be used to identify the drug, such as the δ (OCCO) symm (sub. catechol) mode at 807 cm^{-1}, the aryl C–H wag mode at 769 cm^{-1} and the δ (CCC) mode at 712 cm^{-1} [16,28]. A confocal Raman microscope spectrum obtained from an MDMA particle trapped between cotton fibres contains three bands attributable to the cotton substrate (Fig. 4A); the glycosidic (COC) symmetric stretch at 1120 cm^{-1}, the glycosidic (COC) asymmetric stretch at 1094 cm^{-1} and the ring δ (CCC) mode at 378 cm^{-1} [29]. These bands do not overlap with MDMA characteristic bands which can be easily identified. It is also observed that no significant bands can be assigned to the cutting agents which can be attributed to the ability of Raman microscopy to acquire Raman spectra from individual drug crystals in heterogeneous mixtures. In Fig. 5A, the spectrum obtained from an MDMA particle trapped between silk fibres contains some bands arising from the silk substrate and the cutting agents in the sample. The strongest spectral features from the substrate are assigned to the amide I (C=O) stretch at 1664 cm^{-1} and the (CN) stretch at 1227 cm^{-1} [30]. The presence of these bands does not prevent the identification of the drug which can be readily identified by the diagnostic bands at 807, 769, and 712 cm^{-1}. Similarly, the spectrum obtained from MDMA particles trapped between wool fibres (Fig. 6A) contains some bands assigned to the wool substrate; the amide I ν (C=O) mode at 1654 cm^{-1}, and the δ (CH_2) mode at 1445 cm^{-1} [31]. Again, these bands do not overlap with the diagnostic bands of MDMA and presented no difficulty in determining the identity of the drug. It is also noted that no significant band can be assigned to the cutting agents as the microscopic collection of data enabled the acquisition of selective Raman spectra from individual drug crystals in heterogeneous sample.

We have also observed that the Raman spectra collected from MDMA particles trapped between cotton, silk and wool fibres have very similar band positions but have pronounced differences in the relative intensities of the strongest MDMA band at 807 cm^{-1} to the weaker bands at 769 and 712 cm^{-1}. We attribute this to the presence of differently hydrated forms of MDMA in our seized sample

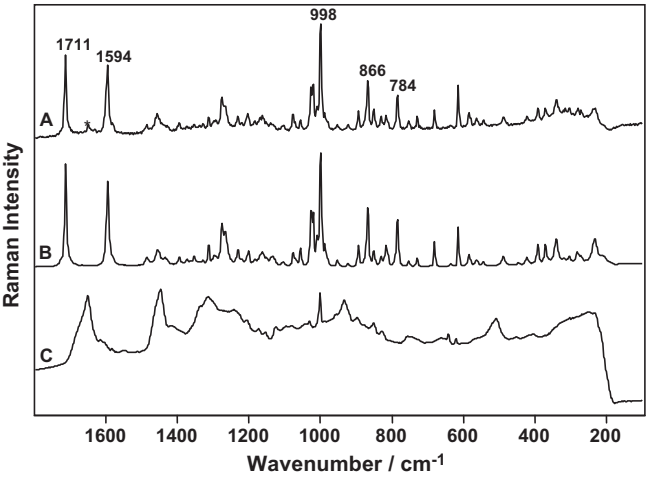

Fig. 3. Raman spectra of: A: street cocaine.HCl between wool fibres (asterisks indicate excipient bands); B: reference cocaine.HCl; C: wool fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

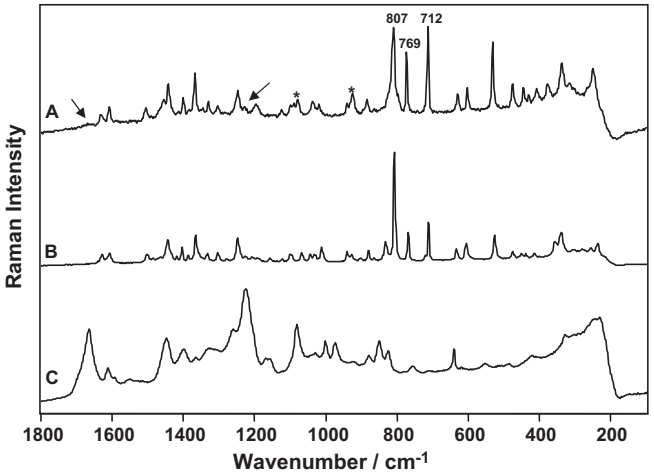

Fig. 5. Raman spectra of: A, street MDMA between silk fibres (asterisks indicate excipient bands and arrows indicate silk bands); B, reference MDMA; C, silk fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

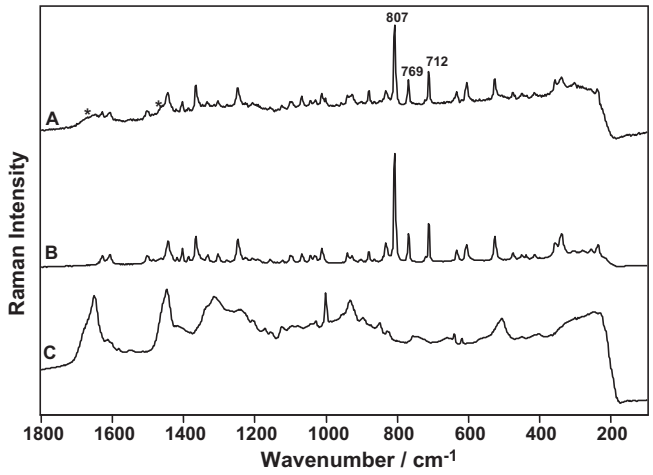

Fig. 6. Raman spectra of: A, street MDMA between wool fibres (asterisks indicate wool bands); B: reference MDMA; C: wool fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

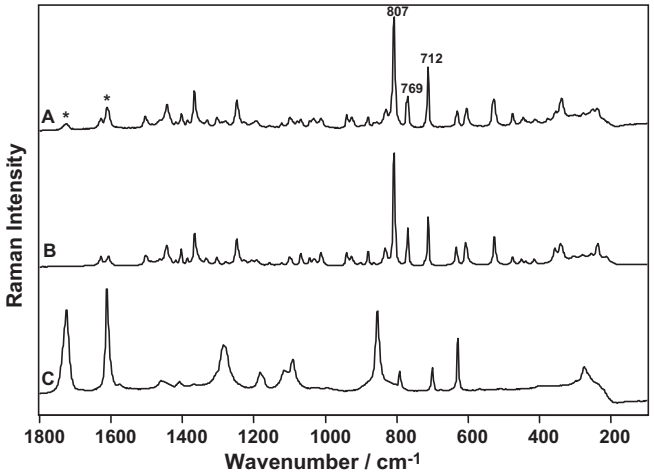

Fig. 8. Raman spectra of: A: street MDMA between polyester fibres (asterisks indicate polyester bands); B: reference MDMA; C: polyester fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

which indicates that MDMA has been manufactured in different ways and with different drug feedstocks. These data are useful for tracing of drug distribution networks and agree with the conclusions reported by Bell et al. [16].

3.2. Drugs on undyed synthetic fibres

A spectrum obtained from a particle of seized cocaine hydrochloride trapped between polyester fibres is shown in Fig. 7A. In addition to the band arising from the excipients at 1648, the resulting spectrum contains two bands attributable to the polyester fibres; the (C=O) symmetric stretch at 1725 cm^{-1} and the aromatic ring stretch at 1610 cm^{-1} [32,33]. The polyester band at 1725 cm^{-1} appears as a shoulder on the cocaine hydrochloride benzoate ester (C=O) stretch at 1711 cm^{-1} and the polyester band at 1610 overlaps with the cocaine hydrochloride aromatic ring (C=C) stretch at 1594 cm^{-1}. On subtraction of the polyester bands, the resulting difference spectrum (Fig. 7B) agrees well with the reference spectrum of cocaine hydrochloride. A spectrum collected from a particle of seized MDMA trapped between polyester fibres (Fig. 8) contains two bands assigned to the polyester substrate; the (C=O) symmetric stretch at 1725 cm^{-1} and the aromatic ring stretch at 1610 cm^{-1} [32,33]. The presence of these bands does not interfere with the identification of the drug which can be unambiguously identified by its characteristic bands at 807 cm^{-1}, 769 cm^{-1} and 712 cm^{-1}.

3.3. Drugs on dyed textiles

The previous results were obtained from drug particles trapped between the fibres of undyed natural and synthetic textiles. Real world clothing is often dyed and it is necessary to determine to what extent the presence of the dye will affect the Raman spectra collected from drug particles trapped between dyed clothing. Functional group features in dye molecules may overlap with the diagnostic bands of the drugs, making identification of the drugs difficult or impossible. Furthermore, Raman signal intensity of the drugs of abuse may be swamped by fluorescence interference which may be generated from the dye molecules.

A confocal Raman microscope spectrum collected from a particle of seized cocaine hydrochloride trapped between blue-dyed denim fibres is shown in Fig. 9A. Although the presence of slight background fluorescence and the strongest denim band (mostly

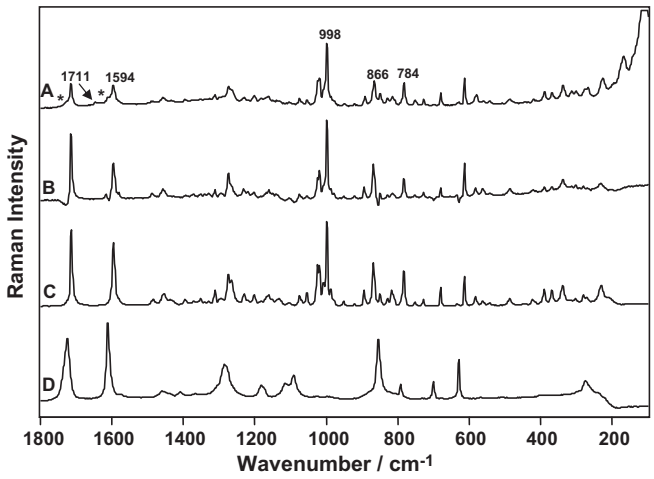

Fig. 7. Raman spectra of: A: street cocaine.HCl between polyester fibres (arrow indicates exipient bands and asterisks indicate polyester bands); B, difference spectrum (A–D); C: reference cocaine HCl; D: polyester fibres 785 nm, 10 s exposure (1 accumulation for A and C, 5 accumulations for D).

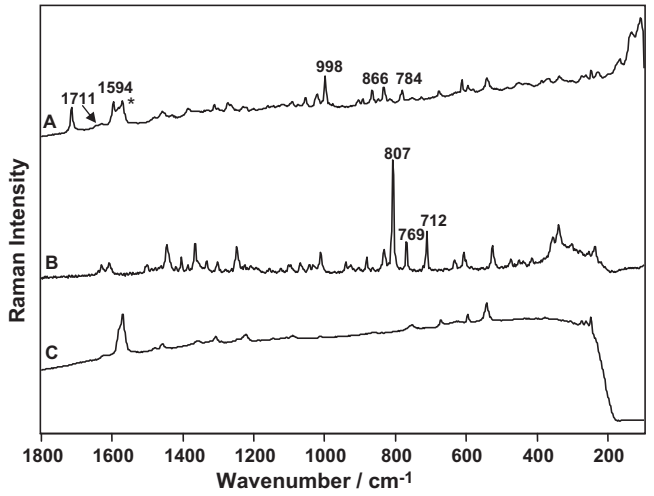

Fig. 9. Raman spectra of: A: street cocaine HCl between denim fibres (asterisks indicate denim band and arrows indicate excipient bands); B: street MDMA between denim fibres; C: denim fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

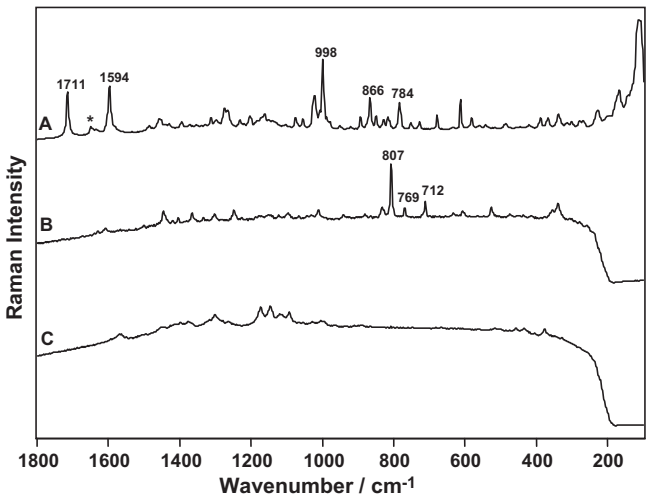

Fig. 10. Raman spectra of: A: street cocaine HCl between orange T-shirt fibres (asterisks indicate excipient bands); B: street MDMA between orange T-shirt fibres; C: orange T-shirt fibres 785 nm (10 s exposure, 1 accumulation for A and B, 5 accumulations for C).

from the blue indigo dye) at 1570 cm^{-1} overlaps with the cocaine hydrochloride aromatic ring (C═C) stretch at 1594 cm^{-1}, the other characteristic bands of the drug are clearly identified. Similarly, a spectrum obtained from a particle of seized MDMA trapped between the denim fibres (Fig. 9B) shows the key signature bands of the drug. No significant bands can be assigned to the cutting agents or the fibre substrate.

Fig. 10 shows the spectra obtained from particles of seized cocaine hydrochloride and MDMA trapped between the fibres of an orange-dyed T-shirt, respectively. The Raman spectrum of the T-shirt contains few cotton bands superimposed on a significant emission background which can conceal the Raman features of the drugs of abuse. In Fig. 10A, in addition to the excipient band at 1646 cm^{-1}, a broad fluorescence background can be seen in the spectrum of cocaine hydrochloride. However, despite this the Raman spectrum of cocaine hydrochloride was usually clearly visible above the background and the characteristic Raman bands of the drug are clearly observed. Similarly, a broad fluorescence background is seen in the Raman spectrum collected from a particle of seized MDMA trapped between the fibres of the orange-coloured T-shirt (Fig. 10B). No significant bands can be assigned to the excipients or the substrate and the characteristic Raman bands of MDMA can be clearly seen above this background fluorescence.

Selective Raman spectra could be acquired from drug particles trapped between the textile fibres with dimensions in the range 5–15 μm. In general, the spectra are of a high quality with a good signal/noise ratio and no appreciable background arising from fluorescence even from dyed fibres. This dramatic reduction of fluorescence background can be attributed to the use of near-infrared excitation source at 785 nm to acquire the Raman spectra. It was possible to focus the laser radiation and collect the Raman scattering from individual drug particles in heterogeneous mixtures. The appearance of spectral lines arising from the excipients was dependent on the relative amounts of the drugs and the excipients in the illuminated confocal volume. In all cases, the drugs of abuse were stronger Raman scatterers than the excipients and the presence of some excipient bands presented no difficulty in determining the identity of the drugs. Similarly, interference from the textiles, including background fluorescence, was overcome by careful focusing of the laser beam and the resulting spectra allow ready differentiation from interference from the textile substrate bands. In few instances, some sampling of the fibres occurred resulting in the appearance of fibres bands in the spectra of the drugs. In all cases the presence of these bands does not prevent the identification of the drugs of abuse .If necessary, interfering bands could be removed by spectral subtraction. This discrimination is attributed to the high spatial resolution of the confocal approach which interrogates the radiation originating from a small volume of the sample coincident with the illuminated spot, and efficiently eliminates the contributions from out-of-focus regions.

4. Conclusions

Interference-free Raman spectra could be acquired from particles of street samples of drugs of abuse on different types of textiles using confocal Raman microscopy. Raman bands arising from the excipients did not interfere with the identification of the drugs as these bands did not overlap with the characteristic features of the drugs. Although the presence of some bands arising from the fibre polymers and/or dyes occur in the Raman spectra, the drugs could be unambiguously identified by their characteristic Raman features. If necessary, overlapping bands could be removed by spectral subtraction. Furthermore, Raman spectra could be acquired from drug particles embedded within highly fluorescent textile specimens. Sample preparation or pre-treatment was not required and molecular specific Raman spectra could be acquired non-invasively *in situ* retaining the evidential material for further analysis .The total acquisition time of the spectra of the drugs was 90 s.

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