

Forensic Analysis Of Dyed Textile Fibres With Various Analytical Techniques: An Updated Review

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Abstract

Introduction: In forensic science, fibres are utilized to establish a link between the crime and the suspect. The samples are examined using several analytical methods to discover relationships between various clothing materials. The most typically studied forensic trace evidence in forensic laboratories are remnants of single fibres. The colour and dyes of fibre micro traces are studied using microscopic, chromatographic, and spectroscopic approaches, which play a significant part in forensic investigations.

Objectives: This study aims to identify the forensic analysis of dyed textile fibres with various analytical techniques.

Conclusion: Because of the significance of fibre clues in criminal investigation, a significant number of studies were done using analytical methods. Raman spectroscopy is being researched more and more for the forensic investigation of textile fibres, as it has a high potential for detecting and identifying dyes in fibres. In addition, FTIR can identify co-monomers, solvents and additives, allowing even more differentiation amongst acrylic fibre subclasses.

Keywords: Textile Fibres, Natural fibres, Micro Traces, Dyes, Dyed Fibres.

INTRODUCTION

Fibres are in contact with us daily, whether natural or synthetic. They are used to make clothing, upholstery for home and office furniture, and automotive upholstery; therefore, they can function as a silent witness in a criminal investigation. Forensic laboratories expertly link persons, things, or a crime scene using micro traces from pieces of single fibres. Fibres can occasionally assist in recreating the circumstances of the occurrence. In some cases, such as homicide and sexual assault, even the tiniest particle can be used as evidence. Micro traces are increasingly being used to explain occurrences like killings, sex offences etc.^[1].

Identifying or comparing micro traces in single fibre fragments would be to evaluate their physical and chemical characteristics and classify them into a particular fibre class and textile product availability. The colour of such micro traces plays a significant function in the initial phase of comparison. Evidential fibres can be judged eligible for investigation with a comparable material based on colour analysis, or they can be disregarded and considered distinct from the comparative material.

Textile fibres are chemically complicated materials that are classed as natural, regenerated and synthetic based on their origin.^[2-3]

Clothes, rugs, furniture, ropes, and bags may all be made from textile fibres. Textile fibre identification is critical because textiles are a significant worldwide commodity, a vital and valuable physical evidence in forensics.^[4-5]

Analysis and identification of fibres is typically difficult due to the broad diversity of fibres used in textiles manufacturers. ^[1] Many samples in forensics are one-of-a-kind and extremely valuable. As a result, non-invasive methods or tiny samples should be used for analysis. Otherwise, the thing being studied would be harmed. In addition, the duration of time required to complete the study might play a significant impact. As a result, straightforward approaches are frequently preferred.

Dye analysis seems a logical possibility to be used as a forensic clue besides colour comparison with optical and spectral techniques; nonetheless, dye examination has been widely overlooked at forensic crime scenes. Identification of differentiating aspects of both the fibre and the dyes used in forensic fibre comparisons is dependent on the identification of distinguishing properties of both the fibre and the dye employed. Because dyes can provide important information in forensic tests, many studies have previously been done to investigate dyes in coloured fibres using various microscopical and spectral techniques.

TYPES OF FIBRES

Various textile fibres are broadly classified into two categories. First is natural fibres, the hair-like raw material that may be converted into nonwoven textiles and can be obtained directly from animal, vegetable, or mineral sources like cotton, jute, hemp, ramble, linen, etc. The second one is synthetic fibres are manufactured fibres, the majority of which are created from petroleum-based basic materials known as petrochemicals, as shown in Fig.1. Fibres are used to make all textiles items. They can come from natural or artificial sources. They are formed up of a remote unit or polymer that is made up of several repeating monomers. Nylon, acrylics, polyurethane, and polypropylene are among them.

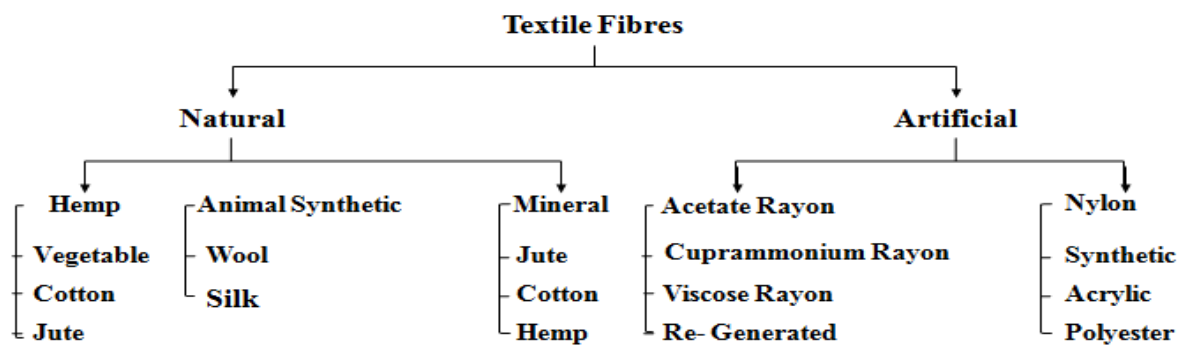


Figure 1: Classification of textile fibres.

A dye is a substance that is utilized to alter or fix the colour of an object. In forensic cases, dyes are crucial for the identification and analysis of fibres. Fig.2. shows textile dyes and its various categories.

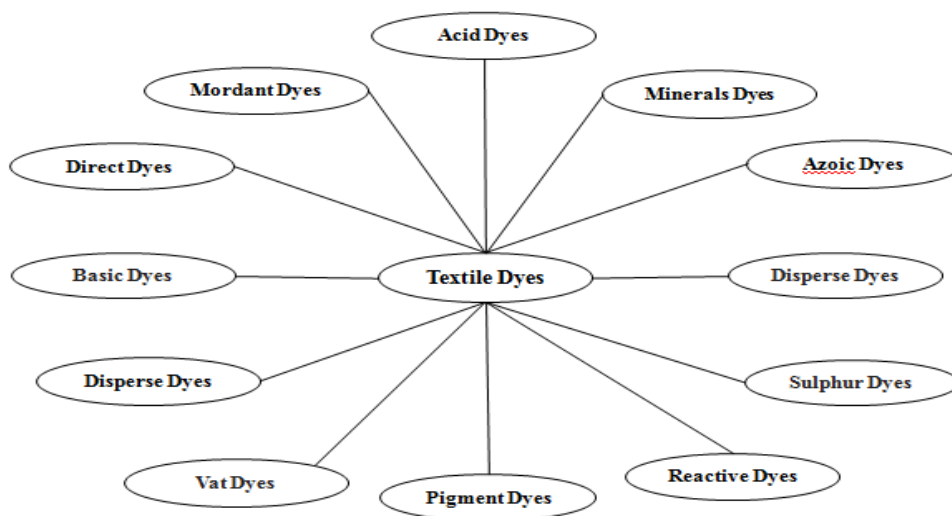


Figure 2: Different types of Textile Fibres Dyes that are commonly found.

FORENSIC ANALYSIS OF FIBRES

Microscopy, mass spectrometry, chromatography and spectroscopy are the principle methods for classify and contrasting textile fibres as per their types and subgroups.

1. MICROSCOPY

Microscopy has always been and will remain to be an important part of fibre analysis^[6]. In forensic laboratories, infrared micro spectroscopy is commonly used to describe and compare single fibres^[7]. Fiber classes and subclasses are distinguished using specific spectral properties^[8]. Finally, scanning electron microscopy with energy dispersive spectroscopy could also be utilised to evaluateanelement components of fibres, as well as inorganic compounds derived from manufacturing process wastes, additives, or environmental pollutants^[9].

Fig.3. shows the analytical techniques that are being used to analyse dyed fibres.

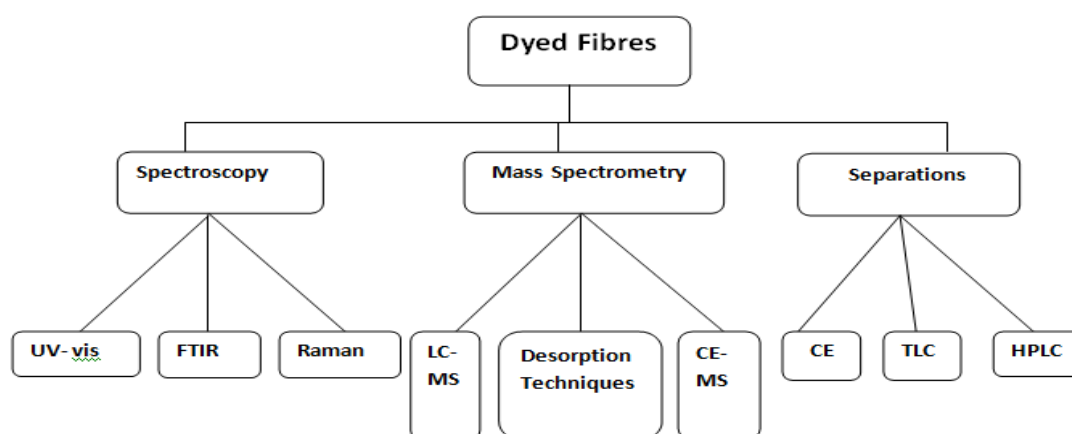


Figure 3: Analytical methods that have been used to examine dyed fibres.

For example, based on evident visual changes in hue, a simple inspection of fibre colour under a microscope can rule out that twin fibres are coming from an identical source. Target fibre investigations employing movies and vehicle seats, for example, have revealed that using fluorescence microscopy, the frequency of apparent fits with green cotton targeted fibres utilizing white optical microscopy was drastically reduced.^[10]

2. UV- VISIBLE MICROSPECTROPHOTOMETRY (MSP)

The application of MSP to fibre analysis has been addressed in detail^[11]. This technique is appropriate for objective observations of the colour of numerous types of trace evidence^[12]. The use of an instrumental technique like MSP is essential since it is repeatable, non - destructive, and requires minimal sample preparation, unlike other dye extraction procedures.

MSP techniques require the examination of many fibres per item (five for manufactured fibres and 10 for natural fibres). Because the dye is linked to a generally stable chemical environment, synthetic fibres usually have a homogeneous chemical makeup when examined with MSP. Cotton and other natural fibres include a variety of chemical constituents that are not evenly distributed throughout the strand^[13-14].

For decades, researchers have investigated the application of MSP for the study of different dye fibres, with a particular focus on natural fibres like wool^[15] and cotton^[13-14], Where colour is the primary distinguishing feature, for instance, it has been demonstrated that MSP can distinguish clearly among cotton fibres of the same dye, with blue cotton fibre showing the most vital discrimination, followed by red cotton fibres.^[13-14] Although early research suggested that black fibres were challenging to distinguish, modern micro spectrophotometry tests of various black cellulose fibres revealed that it might not be the example, especially in separating sulphur, volatile and substantive dyes.^[16] Other blocks of colour (described as "a set of fibre pieces of the same general kind with the same subjective colour") have been studied in MSP. Types of red, blue, pink or brown cotton fibres are identified in these investigations based on their optical characteristics, notably in the UV range.^[17-18]

MSP and thin layer chromatography (TLC) pairing is another well-established method and research topic^[14-19-20]. Wiggins recently tested the capacity of comparing microscopy, UV range MSP and TLC to distinguish reactive colourants in cotton and wool fibre^[21]. MSP spectra are frequently adjusted and visually compared to one another. There have been some reports of efforts to build more advanced techniques.^[12-22] and Adolf and Dunlop provide a thorough description of fibre colour measurements using colorimetry^[11]. It has been established that CCC can be used to explain MSP findings for single textile fibres^[13-23]. Wiggins started investigated the value of estimating the absorbance spectra's first derivatives^[24].

3. INFRARED AND RAMAN SPECTROSCOPY

While infrared microscopy is widely used to determine the composition of fibres^[7-25], it is not widely used to identify fibre dyes. This is due to the intrinsically low level of dye prevalent in most fabrics, as well as the infrared absorbance's lack of sensitivity to components that make up less than 5% of a sample. There have been more detailed reports on the contributions of recognised dyes to this section of the infrared spectrum of acrylic fibres^[26]. Although surface finishes can be identified using ATR (attenuated total reflectance) IR microspectroscopy^[27]. Infrared diffuse reflectance characterizing fibre dyes has been more successfully accomplished using Fourier transform spectroscopy (DRIFTS). Because DRIFT breaks Beer's law, measurements of reflectance at low concentrations are much more sensitive than those of transmission^[28]. DRIFTS were used to distinguish dye colour and reactive dye condition on cotton^[29-30]. A thermospray connector for HPLC has also been created^[31], allowing for DRIFTS detection of the dyes contained in the wastewater.

Raman spectroscopy has gained traction as a fibre analysis tool^[7-32] and the EFG has designated it as a priority research topic^[33]. While techniques like MSP allow for non-destructive colour measurement, little information about the dye is collected. In both natural and synthetic fibres, Raman microprobe spectroscopy has been utilised to characterise dyes^[34-35].

Raman for fibre analysis is still being optimised. According to research, near infrared radiation excitation (785 and 830nm) performed the best terms of spectra purity, sample depreciation and collecting speed^[36]. The EFG then carried out a substantial joint investigation employing spectrometers from seven various suppliers and eight fibre laser^[37]. Three different yet known dyes were found in 9 dyes and 3 test. The results demonstrated that regardless of the instrument employed, the spectrum of similar dyes obtained at same wavelength was reliable.

4. HIGH- PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) AND CAPILLARY ELECTROPHORESIS (CE):

Griffin and Spears describe how HPLC has several advantages over Thin Layer Chromatography and MSP for fibre dye analysis^[38]. When attempting to evaluate highly absorbing (dark hue) fibres, for example, Micro Spectrophotometer is limited. TLC also necessitates vast amounts of dye and varied eluent methods for different dye classes. When designing an HPLC system for fibre dye analysis, various practical considerations must be made, including the dye's general chemical nature, suitable extracted solvents, and sample degradation. The systematic employment of multi wavelength detectors enables for the data of entire Ultra violet spectrum of each dye in mixtures, peak purification assessment, and spectral database generation^[38].

The absence of repeatable migration periods, insufficient sensitivity, and the difficulty to differentiate non-ionizable dyes thwarted early efforts to create stringent capillary electrophoresis procedures for fibre dye ^[39]. Natural dyes like flavanols and anthraquinones, for example, were isolated from modern and ancient fur samples and evaluated by micellar electro kinetic chromatography^[40].

Sample-induced isotachopheresis with MEKC (micellar electro kinetic chromatography) has been proven as a related approach. This boosted sensitivity considerably and reduced sample sizes to single fibres^[41]. Finally, nonaqueous CE has showed potential for basic dye, despite the fact that it has not yet been applied to fibres^[42].

5. MASS SPECTROMETRY

Combining HPLC and CE separation efficiency with mass spectrometry (MS) sensitivity and specificity is a natural combination of instrumental procedures. The use of thermospray high-performance liquid chromatography–mass spectrometry (TSP-HPLC-MS) to detect and quantify dyes in diverse matrices was described in the first report in this domain^[43]. Some writers had already studied the examination of fibre dye using high performance liquid chromatography connected to a MS through an electro spray ionisation origin since then ^[44-45]. The chromatographic extraction of dye of known colour, the UV-visible absorption analyzer worked well as a monitor. LC-MS with electro spray ionisation may discriminate between dye sets that would otherwise be indistinguishable using UV–visible methods, as well as inject dyed molecules into MS with little breakdown^[46]. In reality two pairs of fibres were indistinguishable using microspectrophotometry and can only be identified using LC-MS due to changes in dye separation and identification.

Capillary electrophoresis-mass spectrometry was effectively useful to discriminating of coloured fibres^[47]. CE can also be combined with a MS. Acrylate, polyamide cotton as well as synthetic fibres are used in this study. For CE analysis, basic dye requires a low pH, while dispersion dyes were water insoluble. The resolution of the mass spectrometer permitted fibres as minute as 2mm to be effectively studied ^[47].mass spectrometer was included as a detection tool, or eleven mordant dyes from plants or insects become break by lower detection limits^[48]. According to present study improvements in mass spectrometry have concentrated on non destructive, less intrusive ionisation techniques that maintain sensitivity and specificity ^[49].

Analytical Techniques used for identifying and analysing textile fibre dyes:

S. No.	Name of Fibre	Classification	Sub Classification	Analytical Tool	Conditions	Results	RRef.
1.	Cotton	Natural	Vegetable	UV-Vis MSP.	R: 400-850 nm M:50x Re: 5 nm	It was observed that Dyed fiber spectrum with yellow and red dyes contribution to the overall color, but less than the blue dye. Vis-MSP results: Synazol gold yellow: max. Band at 422 nm. Synazolred: max: max. Band at 529 and 567 nm. SumifixNavy: max: max. Band at 652 nm. Morocan Blue: max.Bands at 629 y 564 nm.	[50]
					R: 240-760 nm M: x 40	Reactive dye: active in the UV-region below 300 nm. Vinyl sulphone dyes shows two peaks in the lower UV range and four peaks between 350-500 nm. Reactive Black 5 and Reactive Orange 72: two peaks in the lower UV range and three peaks between 350-500nm. Greenish blue grey fibers: slight differences between 400-530 nm. Direct dyes: major transmission peak below 400 nm. In the range below 400 nm there is much spectral information is found.	[51]
					Transmittance mode. R: 240-780 nm	RY145 (at 5%): signals at 290 and 430 nm. RB221 (at 5%): signals at 280, 580 and 630 nm. RY145 and RB221 LOD: 0.5%. For mixtures at 90:10 only the major dye was detected. Both dyes were detected for mixtures at 1.5-3.5% and 2.5-2.5%. Different dyes can show identical signals; therefore, a good characterization needs several laser sources.	[52]
				Raman Spectroscopy	E: 514, 633, 785 nm R: 150–2500 cm ⁻¹ T: 10 s Ac: 5A:100x Rs: 4 cm ⁻¹	E. 514 nm: only detection of cotton bands. E. 633 nm in 1.0% dyed fibers: bands from dye and one low intensity band from cotton. E. 633 nm in 4% dyed fibers: no bands from cotton. E. 785 nm: band from glass slide analyzing fibers dyed with bifunctionalreactives placed on a microscope slide. Fibers dyed At 1.0%: comparable intensities between the highest bands from cotton and the highest bands from dye. Fibers dyed at4%: minor intensity for cotton. Intensity varied according to the dye concentration in the range from 330–520 cm ⁻¹ .	[53]

					E: 514.5, 632.8, 685, 785,830 nm R: 2100–400 cm ⁻¹ LP: 1.125, 2.25, 4.5, 14, 17, 300 M Ac: 10. A: 50x T: 10–120 s Rs: 8 cm ⁻¹ and 1.5–2.5 cm ⁻¹	Ex. 632.8 nm: good results for the major dye component with little acquisition time and no spectral degradation. Only some bands were observed between 1700–1000cm ⁻¹ from Moroccan Blue dyed cotton fiber. The band at 1400 cm ⁻¹ disappeared and band shape at 1300 cm ⁻¹ changed when moving from visible to NIR lasers. Ex. at 785 and 830 nm provided excellent spectra for dyes and dyed fiber.	Click on tap here to enter text.
				FTIR Spectroscopy	E: 785 nm Rs: 4 cm ⁻¹ Ac: 256	Bands with consistency shape: 170-1250 cm ⁻¹ . Differences in the range 1250-1500 cm ⁻¹ , caused by a signal originated from glass slide.	Click on tap here to enter text.
2.	Wool	Natural	Vegetable	UV-Vis MSP.	R: 240–760 nm	Small intra-variation of the transmittance intensity in woollen fibers. Large variation for different areas in the same textile attributed to the dye concentration and not to maxima and minima wavelengths or minute shoulder bands.	[54]
				Raman Spectroscopy	E: 514.5, 632.8, 785,830 nm M: 50x, 100x R: 200–2000 cm ⁻¹ T: 10 s	E. 785 and 830 nm: cotton and wool signals. Raman analysis is recommended after UV-Vis MSP	[55]
3.	Silk	Natural	Vegetable	FTIR Spectroscopy	ATR mode R: 400-4000 cm ⁻¹ A: 32 Rs: 4 cm ⁻¹	Band at 2363 cm ⁻¹ , attributed to CO ₂ . Band at 1595 cm ⁻¹ , assigned to CO stretching vibrations and justified to a product of bleaching.	[56]

4.	Polyester	Artificial	Synthetic	UV-Vis MSP	R:380–730 nm M: 7.1–115x	Most pigmented polyester fibers showed atypical band spectral trace associated with carbon black pigment or with an additional absorption at approximately 677 nm.	[57]
				Raman Spectroscopy	E: 514.5, 785 nm LP: 0.5, 100 mW M: 50x, 100x	Excitations lasers at 514 and 785 nm detected indigo spectrum in blue molten ends of fibers.	[58]
				FTIR Spectroscopy	ATR mode R: 400–4000 cm^{-1} A: 32Rs: 4 cm	Stretching bends for CDO and C-O	[59]
5.	Acrylic	Artificial	Synthetic	UV-Vis MSP	R:380–730 nm M: 7.1–115x	Spectral differences among grey pigmented three distinct bands at approximately 568, 606 and 677 nm, a doublet at 528 and 583 nm and a mixture of the previous two with dominant absorptions at 583 and 677 nm. The last was the one identified in pigmented fibers	[60]
				Raman Spectroscopy	E: 514.5, 632.8, 785, 830 nm M: 50x, 100x R: 200–2000 cm^{-1} T: 10 s	E. 633 nm: no signal from acrylic.	Click or tap here to enter text.
6	Polyacrylonitrile	Artificial	Synthetic	Raman Spectroscopy	E: 514.5, 632.8 nm LP: 8–12 mW T: 10–90 s Rs: 1.5–2.5 cm^{-1} M: 50x	Main pigment component from a coloration mixture was identified by Raman. Acrylic fibers showed low intensity bands in the spectral fingerprint region from 400–1700 cm^{-1}	[61]

				FTIR Spectroscopy	Transmission mode. M: 15x Rs: 4 cm ⁻¹ Ac: 512 R: 650–4000 cm	DMF: 1670 cm ⁻¹ (Main band).DMF: 1400, 1380, 1090, and 660 cm ⁻¹ (Secondary bands).have an extra affinity for basic dyes increasing the band at 1045 cm ⁻¹	Click o tap here to enter text.
7.	Nylon	Artificial	Synthetic	UV-Vis MSP	R:380–730 nm M: 7.1–115x	Spectral differences among grey pigmented three distinct bands at approximately 568, 606 and 677 nm, a doublet at 528 and 583 nm and a mixture of the previous two with dominant absorptions at 583 and 677 nm.	Click o tap here to enter text.
				Raman Spectroscopy	E: 780 nm R: 125–4000 cm ⁻¹ T: 50 s A: 50x	The specific band at 1281 cm ⁻¹ for nylon 6 was the major difference respect to nylon 6.6 and nylon 6.12. The band at 1298 cm ⁻¹ for nylon 6 and nylon 6.12 was shifted to 1308 cm ⁻¹ for nylon 6. Nylon 6.6 and nylon 6.12 had three Bands between 1126–1062cm ⁻¹ , while nylon 6 only presented one. Nylon 6.6 had a band at 1235 cm ⁻¹ that allowed differentiating it respect to nylon 6. C-C-Ostretching near 940 cm ⁻¹ appeared at 932 cm ⁻¹ for nylon6, at 350 cm ⁻¹ for nylon 6.6 and 948 cm ⁻¹ for nylon 6.12.Acrylic and modacrylic present C-H bend bands at 1367–1320 cm ⁻¹	[62]

UV-Vis MSP: microspectrophotometry in the ultraviolet visible range; RS: Raman spectroscopy; FTIR: Fourier transformed Infrared spectroscopy;; R: recorded spectral region; T: acquisition time; Ac: n° of accumulations; M: magnification microscope, R: recorded spectral region; T: acquisition time; Ac: no. of accumulations; M: magnification microscope; N.A: not available; ATR:attenuated total reflectance; UV: ultraviolet; E: laser excitation; SR: spectral resolution; LP: laser.

CONCLUSION

Because of the significance of fibre clue in criminal investigations, there was a great amount of study done by utilizing analytical methods. The general chemical category of the fibre is obviously important and extra information may be gleaned by investigating the numerous dyes used to colour textile fibres. This one is also true, that the fibre is natural source. The suggested method for identifying the class and subclass of the PMC is FTIR spectroscopy, which has a larger capacity for studying polymeric substrates than dyes. FTIR can identify co-monomers, solvents, and additives, allowing for even more differentiation amongst acrylic fibre subclasses. FTIR provides forensic scientists with a wide range of options for studying fibres along with its dyes and associated alteration product innovations, such as elemental composition, microstructures, fibre topologies, interface characterization, and attributes of both fibres and related composites of dyes. As this analytical technique doesn't interfere with the sample and change its integrity after examination also, therefore it is a Non-destructive technique which is very essential in forensic examination of evidence. Also, it provides rapid results without investing more time compared to other techniques and is been famous for its reliability quality of analysis.

Also, another spectroscopic technique that is Raman spectroscopy is being researched more and more for forensic investigations of textile fibres, as it has a clear high potential for detecting and identifying dyes in fibres. Raman spectroscopy was used to successfully classify the polymeric content of undyed natural and synthetic fibres. Undyed fibres or those with very little dye uptake can produce moderately strong bands. However, as the dye concentration rises, the intensity of these bands decreases.

CONFLICT OF INTEREST

Declared None

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