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Degradation patterns of natural and synthetic textiles on a soil surface during summer and winter seasons studied using ATR-FTIR spectroscopy

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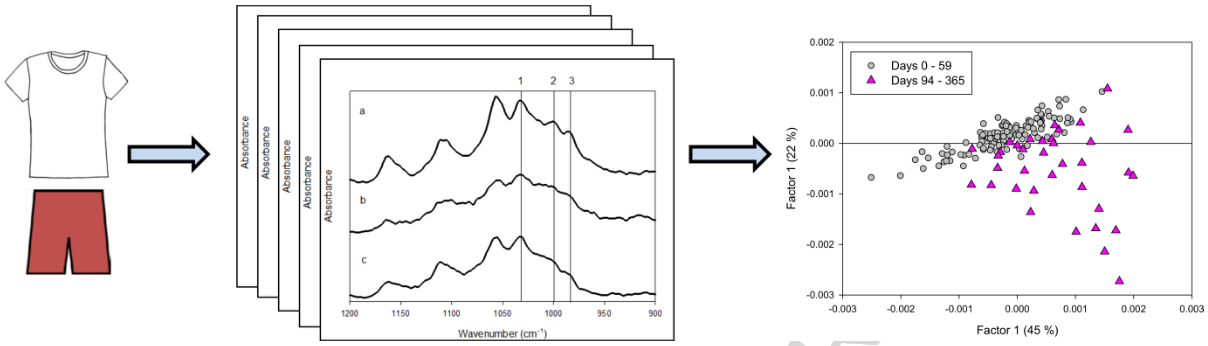
Abstract

Textiles are a valuable source of forensic evidence and the nature and condition of textiles collected from a crime scene can assist investigators in determining the nature of the death and aid in the identification of the victim. Until now, much of the knowledge of textile degradation in forensic contexts has been based on the visual inspection of material collected from soil environments. The purpose of the current study was to investigate the potential of a more quantitative approach to the understanding of forensic textile degradation through the application of infrared spectroscopy. Degradation patterns of natural and synthetic textile materials as they were subjected to a natural outdoor environment in Australia were investigated. Cotton, polyester and polyester – cotton blend textiles were placed on a soil surface during the summer and winter seasons and were analysed over periods 1 and 1.5 years, respectively, and examined using attenuated total reflectance (ATR) spectroscopy. Statistical analysis of the spectral data obtained for the cotton material correlated with visual degradation and a difference in the onset of degradation between the summer and winter season was revealed. The synthetic material did not show any signs of degradation either visually or statistically throughout the experimental period and highlighted the importance of material type in terms of preservation. The cotton section from the polyester – cotton blend samples was found to behave in a similar manner to that of the 100 % cotton samples, however principal component analysis (PCA) demonstrated that the degradation patterns were less distinct in both the summer and winter trial for the blend samples. These findings indicated that the presence of the synthetic material may have inhibited the degradation of the natural material. The use of statistics to analyse the spectral data obtained for textiles of forensic interest provides a better foundation for the interpretation of the data obtained using ATR-FTIR spectroscopy, and has provided insight into textile degradation processes relevant to a soil environment.

Graphical abstract:



Highlights:

- An infrared spectroscopic approach for monitoring forensic textile degradation demonstrated
- The use of chemometrics successfully demonstrated degradation patterns
- Cotton textile degradation in a soil environment was seasonally dependent
- Presence of synthetic material in the blend textile causes a delay in degradation

Keywords: textile; forensic; infrared spectroscopy; cotton; polyester; principal component analysis

1. Introduction

Textiles are a common source of forensically important evidence and can provide insight into a crime event. In the field of forensic taphonomy, the nature and condition of textiles collected from a crime scene can assist investigators in determining the nature of the death and aid in the identification of the victim. The ability to determine how long a piece of clothing has been left in a soil environment can be crucial in certain circumstances. These include times when textiles located at a scene are the only sources of evidence, in the absence of remains or when remains have reached skeletonization and conventional taphonomic methods can no longer be used to estimate post-mortem interval.

The nature and timing of textile degradation in a soil environment has long been of interest for forensic and material purposes. Natural textiles, such as those made from cotton, have been found to degrade rapidly in a soil environment. Cotton is one of the most widely used natural textile fibres and is mainly composed of cellulose [1], making it highly vulnerable to degradation [2]. Traditionally, natural plant based fibres like cotton were used in clothing, however, the development of synthetic polymer fibres during the 20th century resulted in greater variation in the material used in clothing production. Synthetic fibres include materials such as polyamide, elastane, polyacrylic fibres to mention a few. Polyesters, which are compounds of high molecular weight with repeated ester bonds in the main chain [3, 4], is another one of these synthetic materials. Polyester accounts for about 60% of synthetic fibre production with poly(ethylene terephthalate) (PET) representing the major form, it was chosen as the representative for synthetic clothing in the present study. In contrast to natural textiles, synthetic material displays a much higher resistance to degradation in soils. In addition to the inherent properties of the materials used, there is an array of environmental factors that will influence if a textile will degrade in a soil environment, and if so, how rapidly this will progress. These factors include the presence of insects and larger scavengers that might mechanically damage the clothing through tearing and biting, the presence of microorganisms in the soil, the soil properties such as pH, moisture and conductivity, and the ambient temperature [2, 3, 5]. Overall the degree to which these factors influence the textile degradation process is highly dependent on the textile properties.

Biodegradation is a major source of deterioration of textiles in soil environments. Mechanically, the microorganisms break down the textile by penetrating, branching out and growing within the fibres. Chemically, the microorganisms will secrete enzymes that are capable of attacking the molecular structures of the textiles, most often through hydrolysis [6]. There are several biodegradation mechanisms since some microorganisms thrive in an oxygen-rich (aerobic) environment, whereas others will prosper in an anaerobic situation. The availability and type of microorganisms present will also be dependent on the environmental conditions mentioned above. Previously it has been found that natural textiles are more easily and rapidly affected by degradation from microorganisms than synthetic fibres [4, 5]. However, these results are often drawn purely from visual observations and little statistical analysis has been carried out on such data. Providing statistical data of degradation patterns will help develop a more reliable timeline of degradation of clothing materials.

The aim of the study was to examine the degradation patterns of a natural and a synthetic textile material as they were subjected to a natural outdoor environment in Australia. In addition, samples placed on the surface during two different seasons were investigated in order to determine seasonal variability in the textile degradation timeline. The samples were investigated using attenuated total reflectance (ATR) – Fourier transform infrared (FTIR) spectroscopy, a well-established technique for the examination of textile samples. ATR-FTIR spectroscopy is also beneficial as it requires minimal to no sample preparation prior to spectral measurements, it is also a non-destructive technique, which is desired should further analysis using other techniques be required [7]. In order to provide baseline data on textile degradation of both natural and synthetic material, a substantial dataset is needed, which prompts the use of statistical tools in order to encompass all factors. In a previous study, the applicability of multivariate analysis to ATR spectra collected for cotton exposed to decomposition fluid has been demonstrated [8]. A recent study by Peets, Leito, Pelt and Vahur [9] demonstrated the potential of the coupling of spectral data with chemometric analysis for the classification of a range of textile samples.

The current study aims to provide a statistical approach to the analysis of ATR-FTIR spectra of textile degradation patterns exposed to differing climatic conditions using chemometric analysis. The proposed technique and data processing has the ability to aid law enforcement agencies by obtaining more and novel information from textile evidence than what was possible without the use of multivariate statistical analysis. This additional and more objective information can increase the prosecution rate of forensic cases where textile samples are obtained and could increase the importance of such evidence in court.

2. Materials and methods

2.1. Field site

Textile samples were placed outside in an open eucalypt woodland in Western Sydney. The soil conditions at the site have been previously reported [8]. Two trials were established, with trial one commencing during the Australian summer (January), and the second trial commencing in the Australian winter (July). The first study (summer) was carried out from January 2013 to January 2014, during this time period a high of 47 °C was recorded at the start of the trial, while the winter trial was carried out from July 2014 to January 2015 where the lowest temperature reached was 2 °C. The relative humidity for the trial periods was 77.7 %RH and 76.0 %RH for summer and winter, respectively.

Three replicates of 100% cotton t-shirts (alpha ♂, KMart, Australia), three replicates of 100% polyester briefs (alpha ♂, Kmart, Australia) and three replicates of a polyester – cotton blend socks (Tiny Little Wonders, Kmart, Australia), all brand new at the start of the trials, were placed directly on the soil surface and allowed to degrade naturally during each trial. In order to prevent removal by birds or displacement of the textiles due to strong winds etc. a wire mesh cage was created. The cages were placed over the textiles and were not in direct contact with the clothing items placed below.

2.2. Sample collection and storage

Three 5×5 cm swatches of the textiles were collected from each of the sites using sterilised scissors. Sections of dimensions 1×1 cm were prepared for ATR-FTIR analysis. Sampling occurred on days 0, 2, 4, 6, 8, 10, 14, 17, 21, 24, 31, 48, 59, 94, 149, 184, 212, 268, and 365 for the summer trial, and on days 0, 3, 6, 9, 13, 20, 37, 51, 65, 79, 100, 140, 210, 269, 325, 381, 443, 499 and 565 for the winter trial. Prior to sample collection the visual conditions of the textiles were noted and photographs were taken. The samples were packaged and stored at – 18 °C following the procedure outlined in [8].

2.3. ATR-FTIR spectroscopy

A Nicolet Magna-IR 6700 spectrometer (Thermo Scientific, USA) coupled with a liquid nitrogen cooled mercury-cadmium-telluride (MCT) detector was used for the collection of spectra. An ATR accessory with a germanium crystal and a 45° angle of incidence was employed, with specimens placed directly onto the crystal for analysis. Spectra a spectral resolution of 4 cm^{-1} were recorded over a range of 4000 – 550 cm^{-1} and averaged over a minimum of 128 scans. OMNIC software (Version 8.1.11, Thermo Scientific, USA) was used for spectral analysis.

2.4. Statistical analysis

Microsoft Excel 2010, SigmaPlot (version 11.0, Systat Software Inc., USA) and Past (version 3.14, Hammer and Harper, Norway) were used for the initial analysis of the results. One-way analysis of variance (ANOVA) statistical test was used to compare the absorbance ratios of selected FTIR bands with a significance level of 0.05. After this initial analysis the spectra were directly imported into Unscrambler X (version 10.3, CAMO, Norway). Prior to analysis the second derivative was obtained using a Savitzky-Golay algorithm. A region of interest in the spectrum was then selected. For cotton samples this region was selected from wavenumber 950 to 1200 cm^{-1}, whereas for the polyester the regions selected were 1800-1600 cm^{-1} and 1300-950 cm^{-1}, in order to encompass the regions where degradation is suspected to occur. Initial trials using the full spectral region for the cotton and polyester samples also confirmed that changes between the spectra over time would result from these regions. Multiplicative scatter correction was then performed in order to compensate for additive effects in the spectral data. Finally, principal component analysis (PCA) was applied to visualise and to interpret the spectral groupings.

3. Results and discussion

3.1. Visual observations

After approximately 5 months on the soil during the summer season, the cotton samples showed signs of visual damage due to the presence of small holes in the neck region (work previously reported by the authors in a parallel study [8]). Following this, the cotton samples continued to degrade visually. On the final day of sampling (i.e. day 365) for the summer trial the t-shirts were visibly damaged in certain regions and the fabric markedly thinner (Fig. 1b). Yellowing of the fabric also occurred, which is attributed to the formation of chromophores that are able to absorb ultraviolet radiation [10]. At the beginning of the winter trial there was severe wet weather and wind, this caused the fabric to become wet, and dead organic matter covered the t-shirt. The leaves and twigs caused the t-shirt to display orange spots on the white fabric. After 140 days the t-shirt displayed a clear yellow discolouration (Fig. 1c), which could have resulted from early degradation by microorganisms [4, 5]. On day 269 the first signs of visual damage to the textile were observed, again in the form of small holes in certain regions of the t-shirt. On this day, parts of the t-shirt were covered in soil and several ant hills were found around the edges of the clothing. The damage continued to develop as seen with the summer trial until day 565 when several holes were observed and the edges of the damaged area were clearly frayed and weakened.

A distinct difference in the rate of the textile degradation of the cotton t-shirt was observed between the summer trials and the winter trial, with the summer trials experiencing visual damage from day 149 onward for the summer trial whereas the winter trial did not display any signs of cotton degradation until day 269.

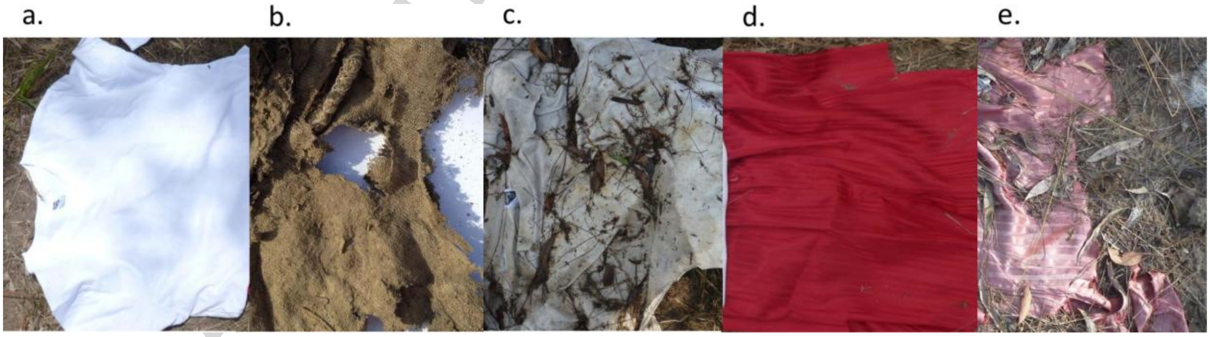


Fig. 1. Visual appearance of the cotton t-shirt after a) 0 days, b) 365 days from summer, c) 140 days from winter, and the visual appearance of the polyester briefs after d) 0 days and e) 365 days from summer.

The polyester samples showed no visual degradation in either of the two trials, despite having the winter trial continue beyond a year and a half, other than fading of the colour of the boxer shorts due to sunlight as can be observed in Fig. 1e.

During the summer trial there were several ant hills formed around or on top of the polyester – cotton blend socks; these caused the socks to be buried. By the final day of sampling vegetation was growing in-between some of the sock samples, which were visibly thinner and frailer. Despite the close contact with soil both below and on top of the socks, there was no complete structural damage to any of the replicates. The polyester – cotton blend socks appeared to be relatively intact throughout most of the winter trial. On day 13, orange discolouration could be seen, most likely due to the dead leaves that were lying on top of the textile. By day 210 the textile appeared to be markedly weaker and more transparent. Vegetation growth occurring between the fibres in the socks was also observed.

3.2. ATR-FTIR spectroscopic analysis

A number of bands in the infrared section of cotton were initially investigated to determine their potential for monitoring degradation of the clothing items (Table 1).

Table 1. Assignments of the main bands observed in the FTIR spectrum of cotton.

Spectral region (cm^{-1})	Wavenumber (cm^{-1})	Assignment
3500-2800	3500- 3000	O-H stretching band
	2920	CH_2 asymmetrical stretching
	2850	CH_2 symmetrical stretching
1700-1200	1640	O-H bending
	1430	CH_2 scissoring
	1335	C-H bending
	1314	CH_2 rocking
1200-800	1110	C-O-C stretching
	1056	C-O stretch
	1033	C-O stretch
	1001	C-O stretch
	985	C-O stretch

A preliminary visual analysis was carried out by inspection of the spectra from the earlier days compared to those from the later sampling days. A visual trend was observed with some of the IR bands in the samples from the summer trial: initially the 985, 1001 and 1033 cm^{-1} bands were very distinct. However, the 985 and 1001 cm^{-1} bands disappeared in the later sampling days (Fig. 2) and merged with the 1033 cm^{-1} band. It has been established that one mechanism by which cotton degrades is through hydrolysis [11] and the preliminary findings support this as the 985, 1001 and 1033 cm^{-1} bands are all in the region of the spectrum where C-O stretching occurs. A potential region where changes might occur during the cotton degradation has thus been identified. A similar disappearance of the 985 cm^{-1} band was observed in the cotton control samples from the winter trial, however, this only occurred towards the very end of the trial (sampling days 499 and 565). Another apparent change was found in the band at 1110 cm^{-1}, this band represent C-O-C stretching, initially this band was of medium intensity (Fig. 2a), before decreasing on day 184, the intensity of the band then increased again in the later sampling days.

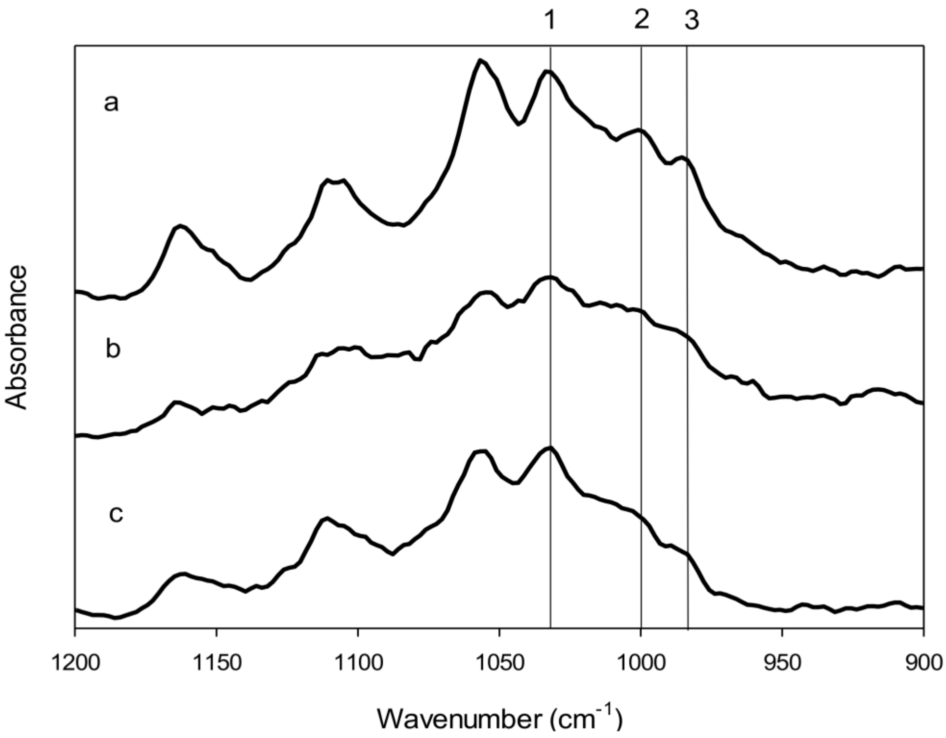


Fig. 2. FTIR region of interest for the degradation of cotton textile samples. 1. 1033 cm^{-1}, 2. 1001 cm^{-1} and 985 cm^{-1}. Typical spectra of the cotton collected from the site on a) day 0, b) day 184 and c) day 268 here represented by data from T1.

Polyester is believed to degrade through hydrolysis, resulting with a cleavage of the ester bonds; with this in mind certain bands of interest were selected for further analysis (Table 2). Hydrolysis is thought to occur in the amorphous regions of the polymer [12]. The likely result during hydrolysis of PET is an increase in carboxylic acid and alcoholic end groups, in addition to an increase in smaller chain fragments most likely through reverse esterification [13].

Table 2. Assignments of the main bands observed in the FTIR spectrum of polyester.

Wavenumber (cm^{-1})	Assignment
720	C-H aromatic ring wagging
1090	C-O stretching
1245	Ester C-O stretching
1715	C=O stretching

The regions of the spectrum where changes were predicted to occur during polyester degradation were visually compared between the sampling days (Fig 3). No visual differences were observed between any of the sampling days for either trail. This initial spectroscopic analysis is, therefore, consistent with the visual changes observed with the polyester samples and confirms that degradation did not occur throughout both trials.

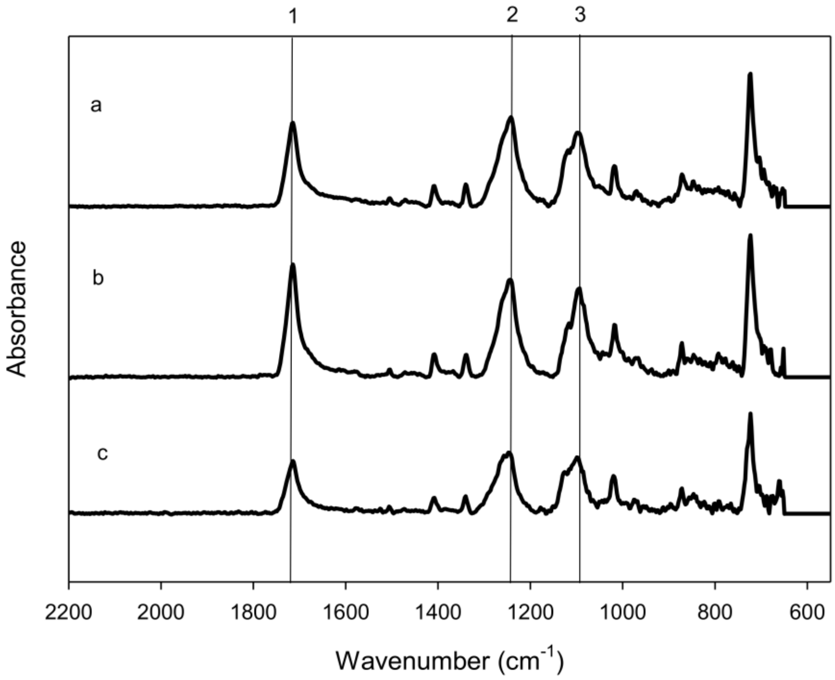


Fig. 3. FTIR region of interest for the degradation of polyester textile samples. 1. 1715 cm^{-1}, 2. 1245 cm^{-1} and 1090 cm^{-1}. Typical spectra of the polyester samples collected from the sites on a) day 0, b) day 184 and c) day 365.

As the socks employed in this study were made from a blend of cotton and polyester, all the aforementioned bands were investigated when analysing the sock samples. The ATR-FTIR regions representing both fibre types were able to be detected and distinguished. The polyester – cotton blend infrared spectra were separated into separate cotton and polyester spectra, and the two groups were processed separately. There were no visual changes in the spectra for the polyester component of the sock samples. This is similar to the results obtained with the 100 % polyester samples. In the cotton section of the spectra, however, a similar trend as that observed for the 100 % cotton samples was observed, where the cotton section of the sock samples from both summer and winter trial saw a loss of the 985 cm^{-1} band in the later samples (days 149 – 499).

3.2.1 Initial statistical analysis

In order to conduct statistical analysis, the bands of interest were normalised to the most intense band to avoid any instrument variability between the analysis days [14]. The 985 and 1001 cm^{-1} bands were normalised using the 1033 cm^{-1} band. The 1033 cm^{-1} band was selected as it is always present in the samples at a very significant absorbance level. The 1090 cm^{-1} and 1245 cm^{-1} polyester bands were both normalised using the 1715 cm^{-1} band for the initial statistical analysis.

The three surface deposit sites were treated as different sites initially, rather than replicates. However, upon visual inspection they did not appear to be different for both cotton, polyester and polyester – cotton blend, a one-way ANOVA ($p = 0.05$) confirmed that there was no significant difference between the sites with $p = 0.402$, $p = 0.912$ and $p = 0.765$, respectively. This allowed the three sites to be treated as true replicates, thus increasing the replicate number for each sampling day and the statistical importance of the findings. All subsequent analysis was done using this assumption.

Initial analysis of the 985 cm^{-1}/1033 cm^{-1} ratio did not produce any significant results within the cotton control samples. However, an interesting trend was seen with the 1001 cm^{-1}/1033 cm^{-1} band ratio for the cotton samples from the summer trial (Fig. 4). It is evident that the band ratio generally increased over time. A one-way ANOVA ($p = 0.05$) determined that the later sampling days (from day 149) were statistically different ($p < 0.05$) from the earlier sampling days (up to day 21). Thus, this normalised band might be a potential indicator of cotton degradation. The winter trial produced similar results, with the 985 cm^{-1} band not showing any significant differences, and the 1001 cm^{-1} band being significantly different when comparing the days from 325 until 565 with the earlier sampling days using a one-way ANOVA ($p = 0.05$). Again, this observation indicates that the 1001 cm^{-1} band might be of particular interest in the mapping of cotton textile degradation.

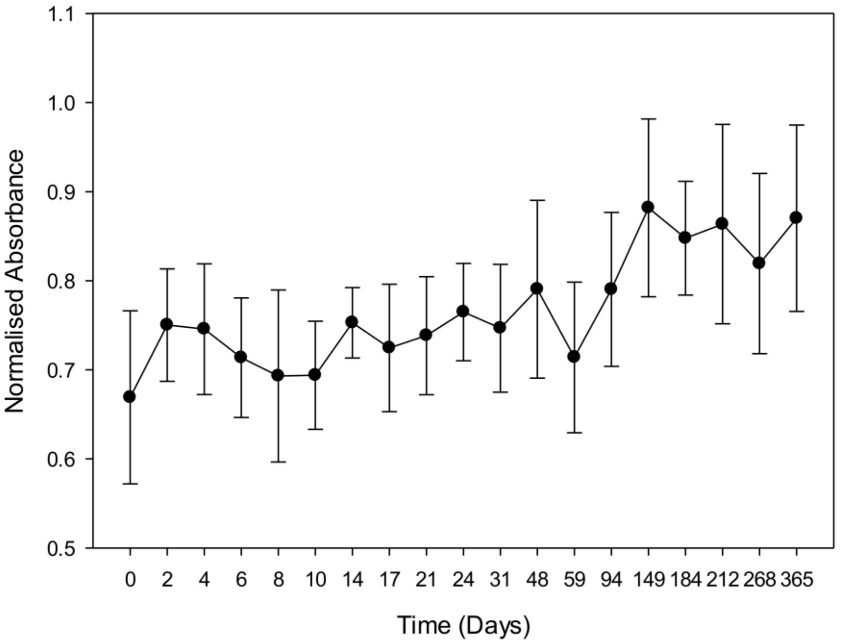


Fig. 4. Quantity of $A_{(1001/1033)}$ as a function of time. The error bars represent one standard deviation based on 9 replicates.

The 1001 cm^{-1} band was found to be statistically different for both summer and winter during their respective trials. Attempting to graph and compare the occurrence of this potential degradation of the 1001 cm^{-1} band in both the summer and winter trials was carried out using accumulated degree days (which account for the difference in temperature between the summer and winter trials [15]). This difference was found to occur around comparable accumulated degree days (ADD) values, showing that the spectroscopically recorded textile damage was temperature dependent (Fig. 5). Caution should however be taken when reading these results as due to variation in the IR spectra the error bars were found to be substantial on some of the sampling days, however an overall trend could still be observed manually and graphically.

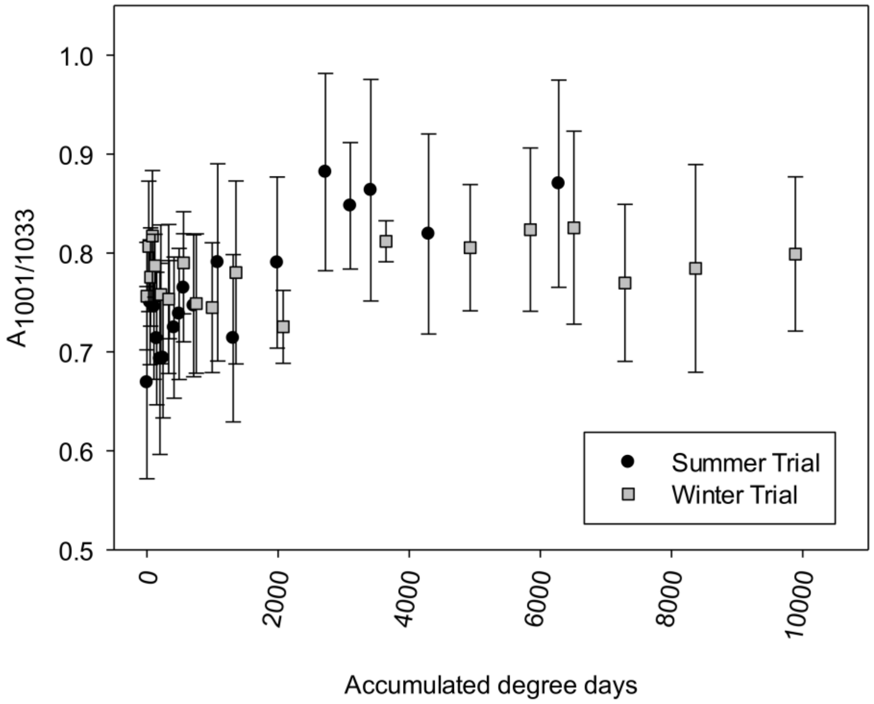


Fig. 5. Quantity of $A_{(1001/1033)}$ from the cotton samples from the summer trial (black circles) and from cotton samples from the winter trial (grey square) as a function of accumulated degree days. The error bars represent one standard deviation based on 9 replicates.

Both normalised polyester bands remained unchanged over the course of both trials (Fig. 6). This demonstrates that the polyester did not degrade over the sampling period. The initial statistical analysis confirmed the lack of any visual changes in the polyester samples. There were no initial statistical trends observed with the blend samples, for either the cotton or the polyester sections of the material during both seasons.

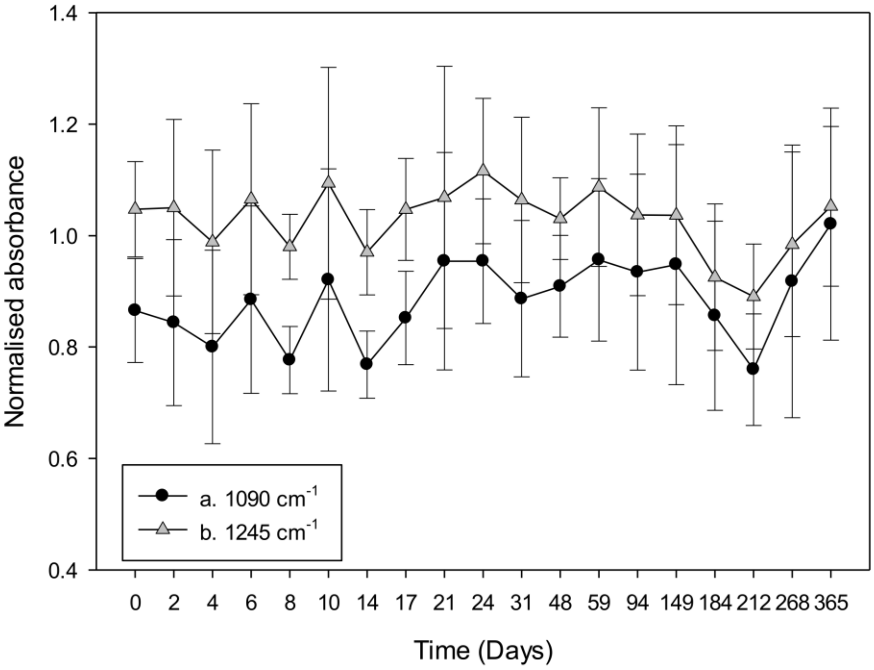


Fig. 6. Quantity of $A_{(1090/1715)}$ as a function of time (black circle) from T1 and the quantity of $A_{(1245/1715)}$ as a function of time (grey triangle) from T1. The error bars represent one standard deviation based on 9 replicates.

3.2.2 Principal component analysis

Although the initial statistical approach helped determine certain bands of interest for the different textile types, especially for the cotton samples, additional multivariate analysis was performed. The multivariate approach was selected as spectral data has a large amount of influences that needs to be accounted for statistically. Multivariate analysis also helps visually demonstrate any differences between the samples over time and any groupings of samples.

In order to further validate the importance of the 1001 cm^{-1} band and determine if the 1033 cm^{-1} band is also changing and, thus, playing a part in the change observed for the 1001 cm^{-1} normalised band, PCA was carried out on the cotton data from the summer and winter trials (Fig. 7).

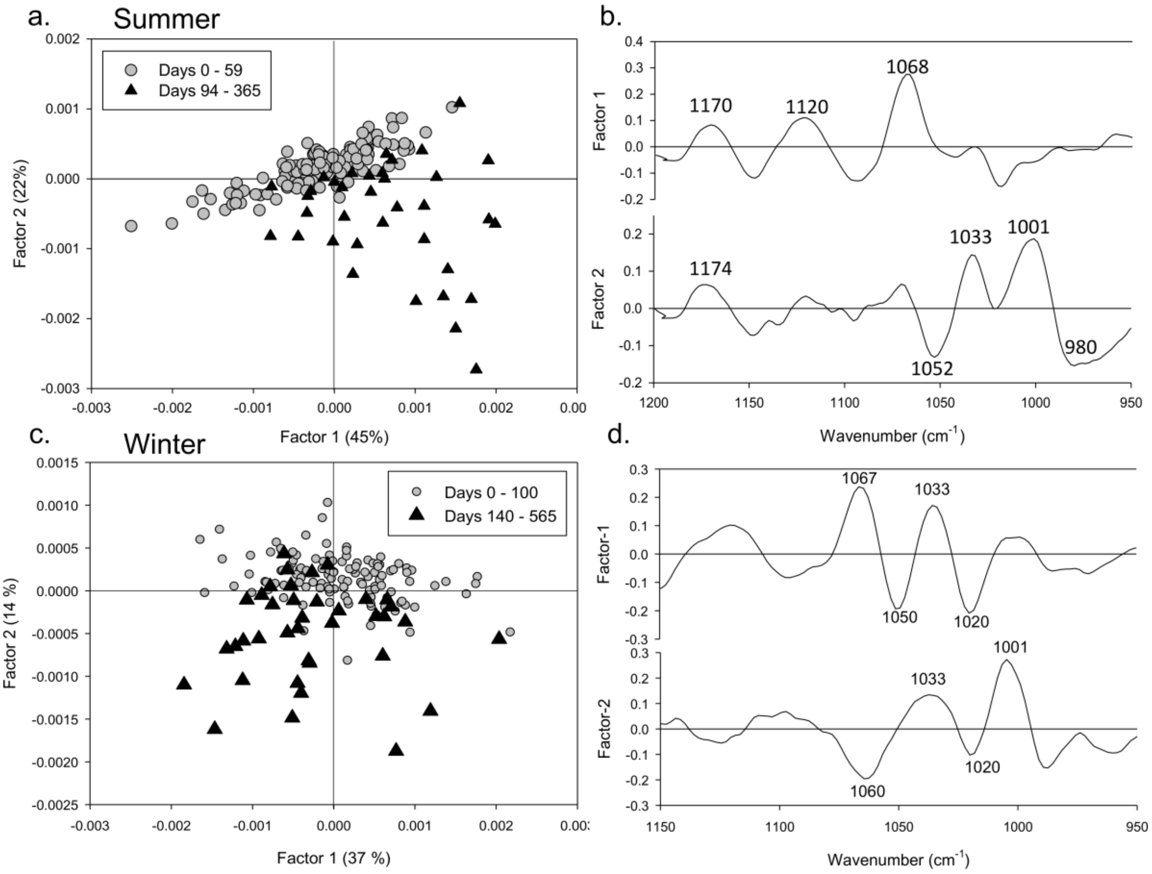


Fig. 7. a) PCA for the cotton spectra from the summer trial. PCA plot showing later sampling days (black triangles) and earlier sampling days (grey circles). b) The loadings plot shows the influence on the PCA groupings from bands in the spectra. c) PCA for the cotton spectra from the winter trial. With later sampling days (black triangles) and earlier sampling days (grey circles). d) The loadings plot shows the influence on the PCA groupings from bands in the spectra.

The PCA plot of the summer trial (Fig. 7a) demonstrated that the samples could be visualised as two separate groups, denoted as early sampling days (days 0 – 59) and late sampling days (days 94 – 365). The early sampling days grouped well together, whereas the later sampling days were spread out from this original group (Fig. 7a). The later sampling days could were not clustered together, but were identified due to their separation from the main group (early sampling days). The later sampling points were all separating from the clustered early days on the negative y-axis. From the loadings plot it was determined that this was due to a decrease in both the 1001 and the 1033 cm^{-1} bands based on the factor 2 (y-axis) loadings plot (Fig. 7b). This means that a change in the spectral region where hydrolysis occurs was observed, demonstrating that textile degradation is happening in the later sampling days. The later sampling days also exhibited an increase in the ~1052 cm^{-1} band corresponding to C-O stretching. Following the analysis of the winter trial, the same pattern was observed (Fig. 7c). The early sampling days were again grouped along the y-axis, with the later sampling days spreading out towards negative y-values (Fig. 7c). The loadings plot demonstrated that the 1001 cm^{-1} and 1033 cm^{-1} bands were again a factor in the separation along the y-axis (Fig. 7d).

A difference in the timing of the degradation was obtained between seasons; this can be demonstrated by comparing the summer trial with the winter trial as the separation in the data set from winter did not occur until day 140, compared to day 94 in summer. In order to determine if this delay in degradation observed was due to the temperature difference the ADD values at the time of the data separation were compared. The summer trial was found to be in the “early sampling days” group until ADD 1315, and samples beyond ADD 1968 were classified in the later sampling days. Similar ADD values were obtained in the winter study, with the end of the early sampling days extending to and including ADD 1357. The later sampling days grouping started on ADD 2078. Thus, comparing the ADD values that account for temperature demonstrated that the difference in ambient temperature was likely to be responsible for the delay in degradation observed in winter, which was also observed during the initial statistical analysis (Fig. 5). The difference in temperature between the two seasons is likely to have resulted in differences in microbiological activity between the summer and winter seasons as microbial communities are significantly affected by temperature [16-18]. The difference in pH between the two trials may also be a factor in the delayed onset of textile degradation observed in winter. The soil at the summer trial site was found to have a relatively stable pH around 5 throughout the study (data not shown), whereas the soil at the winter sites had pH values that were found to be a lot more variable and would fluctuate between pH 5 and 7. This fluctuation in pH might prove non – conducive for certain bacteria and fungi. The lower pH experienced in summer might also be due to the fact that cotton is highly vulnerable to hydrolysis in acidic environments and hydrolysis may have been initiated earlier in summer than during winter.

Similarly, PCA was conducted on the spectral data from all the polyester samples, despite the lack of any trends during the visual and initial data analysis. Fig. 8a shows the PCA plot obtained using the samples from the summer trial; no trends were observed. Identical results were found for the winter trial. The loadings plot (Fig. 8b) shows that the bands influencing the data sets were comparable to the ones selected during the initial spectral analysis.

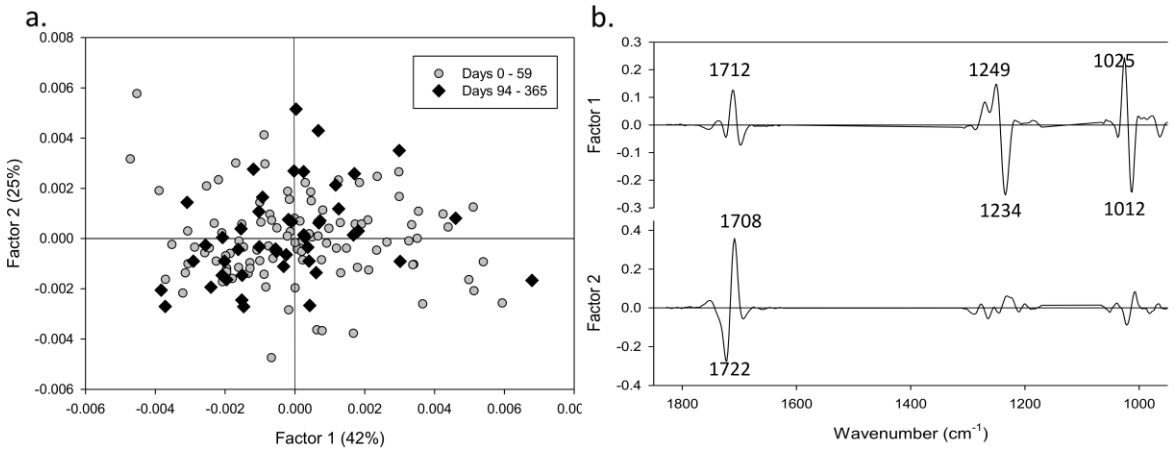


Fig. 8. a) PCA for the polyester spectra. The PCA plot demonstrates that the later sampling days (black triangles) could not be separated from the earlier sampling days (grey circles). b) The loadings plot demonstrates the influence on the PCA groupings from ester bands in the spectra.

The polyester PCA plots from all trials further validated that there was no difference in the spectral data for the polyester samples, and degradation was not observed. This corresponds to what is known from literature, as polyester and similar man-made textiles have been reported to degrade very slowly under natural conditions [3, 6, 19].

The cotton infrared region from the socks analysed during the summer trial demonstrated a similar trend to that found for the 100 % cotton control samples, however, the changes were observed to a lesser extent (Fig. 9a). The same trend is observed for the data from the winter trial (Fig. 9c). The fact that the separation between the undamaged and degraded textile samples was less distinct in the polyester – cotton blend samples compared to the 100 % cotton samples may indicate that the presence of the synthetic material inhibits the cotton degradation. Again, a delay in the degradation was observed between the seasons, as was found with the 100 % cotton samples. The polyester IR region of the polyester –

cotton blend samples did not exhibit any trends as predicted and further validated that polyester did not degrade during the time frame of any of the trials.

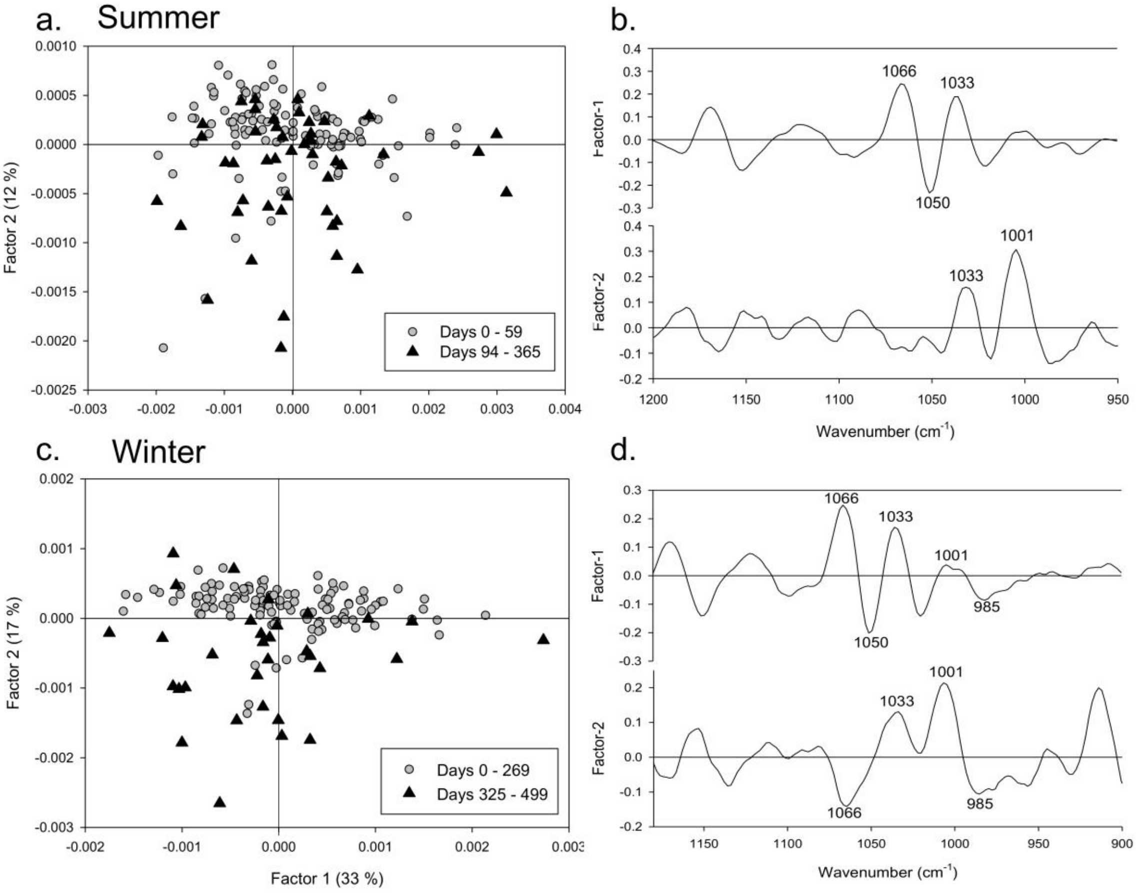


Fig. 9. a) PCA for the cotton section of the polyester – cotton blend samples from the control sites from the summer trial showing later sampling days (black triangle); and the earlier sampling days (grey circle). b) The loadings plot shows the influence on the PCA groupings from cotton bands in the spectra. c) PCA for the cotton section of the polyester – cotton blend samples from the control sites from the winter trial showing later sampling days (black triangle); and the earlier sampling days (grey circle). d) The loadings plot showing the influence on the PCA groupings from cotton bands in the spectra.

4. Conclusions

Analysis of the infrared spectra of textiles of forensic interest has been demonstrated as an effective means of monitoring degradation of the materials placed on a soil surface.

The 100% cotton samples showed a great degree of visual degradation. The synthetic polyester material was in a relatively good state visually apart from some discolouration. The polyester – cotton blend was found degrade somewhere in between as predicted, with sections of the socks demonstrating some degradation through the apparent decreased strength and general appearance of the fabric.

An inspection of the cotton IR bands in addition to initial statistical analysis saw a disappearance of several C-O stretching bands. The use of more advanced statistical software enabled the spectral data to be analysed using multivariate analysis and chemometrics. PCA of the 100 % cotton data demonstrated a clear separation between the earlier sampling days and the later sampling days where degradation had occurred. The separation in the data was also determined to be related to the C-O stretching regions of the cotton spectrum. This pattern of degradation was determined to occur at a later stage in the winter trial, as the separation in the data set from winter did not occur until day 140, compared to day 94 for the summer trial. This indicated that seasonal variation is a factor in textile degradation.

Contrary to the natural textile samples, the synthetic material did not show any signs of degradation either visually or statistically throughout the surface trials. The cotton section from the polyester – cotton blend samples were found to be very similar to the 100 % cotton samples, however the PCA results demonstrated that the degradation patterns were less distinct in both the summer and winter trial for the blend samples. These findings indicated that the presence of the synthetic material might have inhibited the degradation of the natural material.

The use of statistics to analyse the spectral data obtained for textiles of forensic interest provides a better and more objective foundation for the interpretation of the data obtained using ATR-FTIR spectroscopy, and has provided insight into textile degradation processes relevant to a soil environment. The textiles used in the current study were all identical in their respective compositions and were untreated. Pre-treatment of textile samples (especially cotton textiles) could potentially affect the rate of biodegradation by inhibiting microbial activity and would therefore be an avenue of further research once the appropriate replicate data has been created for untreated textile samples. Additional studies into textile degradation in different soil compositions should also be conducted.

The addition of objective information about the degradation age of textile samples to law enforcement agencies could benefit the time-since-death estimation as an additional piece of evidence along with the conventional decomposition stages of the remains.

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