

**COMPARATIVE ANALYSIS OF BURIAL DEGRADATION OF
TEXTILE FIBRES IN DIFFERENT SOIL MOISTURE CONTENTS
USING ATR-FTIR SPECTROSCOPY**

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2026

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USING ATR-FTIR SPECTROSCOPY**

by

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Thesis submitted in fulfilment of the requirements

for the degree of

Bachelor of Science in Forensic Science

February 2026

ACKNOWLEDGEMENTS

First, I would like to express my deepest gratitude to my respected supervisor, Assoc. Prof. Dr. Nik Fakhuruddin bin Nik Hassan, for his invaluable guidance, constructive feedback, and encouragement throughout this research study. I sincerely appreciate his patience, dedication, and enthusiasm in guiding me throughout the journey, and I feel truly fortunate to have had him as my supervisor.

I would also like to extend my sincere appreciation to Dr. Nur Waliyuddin Hanis bin Zainal Abidin, the course coordinator of GTF411, as well as all the forensic lecturers, for their guidance, advice, and support throughout this learning journey. I am also grateful to the staff from Unit Pengurusan Makmal (UPMS) and the laboratory assistants from the Forensic Science Laboratory and Advanced Analytical Laboratory for their assistance and cooperation during the laboratory work.

I would like to thank my dear friends Cheong Yi Jing, Chee Jia Inn, Tan Zong Hong, Olivia Ong Jian Yie, Chew Serene, Camey Ho Ting Ting, and others, for being my pillars of support. Their help, both practically and emotionally, has been invaluable throughout this research journey.

Finally, I am deeply grateful to my beloved parents, Chan Boon Hai and Lim Guat Lian, and my siblings, Chan Hui Yee and Chan Hui Qi, for their unwavering support, encouragement, and belief in me. Their love and guidance gave me the confidence to overcome challenges and complete this research successfully.

Lastly, I would like to express my gratitude to everyone who has directly or indirectly contributed to my research journey. Their guidance, support, and encouragement have been invaluable in the completion of this study, and I am sincerely thankful to all of them.

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LIST OF SYMBOLS AND ABBREVIATIONS

2D	2-Dimensional
3D	3-Dimensional
ATR	Attenuated Total Reflectance
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy
DI	Deionized (usually refers to water)
GCMS	Gas Chromatography-Mass Spectrometry
HCl	Hydrogen Chloride
IR	Infrared
LDA	Linear Discriminant Analysis
p	Probability Value
PAN	Polyacrylonitrile
PC	Principal Component
PCA	Principal Component Analysis
PCA-LDA	Principal Component Analysis - Linear Discriminant Analysis
PET	Polyethylene Terephthalate
PLA	Polylactic Acid
PLS-DA	Partial Least Squares-Discriminant Analysis
PLSR	Partial Least Squares Regression
R^2	Coefficient of Determination
RMSE	Root Mean Square Error
ROS	Reactive Oxygen Species
SEM	Scanning Electron Microscopy
SIMCA	Soft Independent Modeling of Class Analogy

TGA	Thermogravimetric Analysis
TMA	Thermomechanical Analysis
UV	Ultraviolet
WHC	Water Holding Capacity
XRD	X-ray Diffraction

ABSTRAK

Bahan tekstil sering dijumpai sebagai bukti dalam penyiasatan forensik yang melibatkan kes serangan, pembunuhan, kesalahan seksual dan pengebumian tersembunyi. Walau bagaimanapun, kajian terhadap pengebumian tekstil dalam jangka pendek bagi menganggar tempoh sejak dikebumikan masih terhad. Kesan kandungan kelembapan tanah yang berbeza terhadap degradasi tekstil juga masih kurang diterokai. Kajian ini bertujuan untuk menyiasat degradasi pengebumian fiber tekstil di bawah kandungan kelembapan tanah yang berbeza sepanjang tempoh pengebumian selama enam minggu. Tiga jenis tekstil telah dikaji, termasuk fiber semula jadi (kapas dan sutera) dan sintetik (poliester). Tekstil tersebut ditanam dalam tanah dengan kandungan kelembapan yang diselaraskan kepada 0%, 20%, 40% dan 60% keupayaan pegangan air. Sampel dianalisis menggunakan stereomikroskop serta spektroskopi inframerah transformasi Fourier- jumlah pemantulan terlemah untuk memerhati perubahan morfologi dan perubahan dari segi kimia masing-masing. Teknik statistik multivariat, termasuk analisis komponen utama-analisis diskriminan linear dan regresi kuasa dua terkecil separa-analisis diskriminan, digunakan terhadap data spektrum bagi menilai kesan degradasi dan memodelkan anggaran tempoh masa sejak penanaman. Pemerhatian morfologi menunjukkan bahawa kapas mengalami degradasi yang paling ketara, diikuti oleh sutera dan poliester yang menunjukkan rintangan yang lebih tinggi dan hampir setara terhadap kesan pengebumian. Analisis ATR-FTIR yang digabungkan dengan PCA-LDA menunjukkan pengelasan sampel yang jelas mengikut jenis tekstil dengan kadar pengelasan sebanyak 99.8%. Model PLSR selanjutnya menunjukkan bahawa tempoh penanaman dapat diramalkan dengan ketepatan yang munasabah, dengan nilai R^2 antara 0.93 hingga 0.98 berdasarkan data spektrum.

ABSTRACT

Textile materials are commonly encountered as evidence in forensic investigations involving assault, homicide, sexual offences, and clandestine burials. However, limited research has focused on the short-term burial of textiles for estimating time since burial. The effects of different soil moisture contents on textile degradation also remain largely unexplored. This study investigated the comparative burial degradation of textile fibres under different soil moisture contents over a six-week burial period. Three textile types were examined, including natural fibres (cotton and silk) and a synthetic fibre (polyester). The textiles were buried in soils with the moisture contents were adjusted to 0%, 20%, 40%, and 60% water holding capacity (WHC). Samples were collected weekly and analysed using stereomicroscopy and attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy to evaluate morphological changes and chemical alterations, respectively. Multivariate statistical techniques, including principal component analysis-linear discriminant analysis (PCA-LDA) and partial least squares regression-discriminant analysis (PLSR-DA) were applied to the spectral data to assess degradation trends and to model to model the estimation of time since burial. Morphological observations revealed that cotton exhibited the most pronounced degradation, followed by silk and polyester showed the greater and comparable resistance to burial conditions. ATR-FTIR analysis combined with PCA showed clear separation of samples according to their types with classifications rate of 99.8%. The PLSR models further showed that burial time could be predicted with reasonable accuracy with R^2 values ranging from 0.93 to 0.98 based on the spectral data.

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Textiles are widely present in everyday life, which can be found in clothing, furniture, household items, and other industrial uses. Textiles are used in both indoor and outdoor settings all over the world. Due to their widespread, textile materials are commonly encountered as evidence in forensic investigations involving assault, homicide, sexual offences and clandestine burials.

In forensic context, textile can be considered as one of the common sources of evidence used in the reconstruction of crime (Stuart & Ueland, 2017). Textile fibres can be easily transferred between individuals, objects, and environments through physical contact during criminal activities. For example, small fragments of fabric may adhere to weapons, vehicles, or any surfaces at a crime scene. Such transfers create associative evidence that can link a suspect to a victim or location.

Textiles are valuable indicators of the circumstances surrounding an event due to the ability to retain morphological and chemical signatures. When textiles are deposited in outdoor environments and especially when buried in soil, they begin to change physically and chemically (Sülar & Devrim, 2019). Soil is a dynamic environment composed of minerals, organic matter, water, and microorganisms. These components interact to create a variable condition that influence the rate and pattern of textiles degrade. Factors such as soil moisture, microbial activity, temperature, pH, and organic content also contribute to the breakdown of fibres (Nam *et al.*, 2015; Nam *et al.*, 2024).

As degradation progresses, the rate and pattern of change are also influenced by the chemical composition of the textile fibres themselves. The analysis of recovered textile evidence based on their chemical pattern is essential to avoid misinterpretation. In order to improve forensic interpretation, research on textile degradation in burial environments is crucial.

The need for a deeper understanding is supported by advancements in analytical techniques. Instrumentation such as stereomicroscopy, ATR-FTIR spectroscopy, and multivariate statistical methods can be employed to detect molecular and structural changes on a textile. These approaches will help to identify soil-induced degradation patterns across burial conditions. By characterising these changes systematically, forensic practitioners may be able to develop more reliable frameworks of estimating how long textiles have been buried in specific environmental conditions.

1.2 Problem Statement

Textile materials can be found buried in soil environments either deliberately or unintentionally for criminal purposes. These textiles may include clothing, undergarments, and daily items. As a result, they hold significant forensic value because they can provide associative evidence that links individuals, objects, and crime scenes. Since the forensic investigations are time-critical and buried materials should, therefore, be located and interpreted as quickly as possible. However, there is limited research on the short-term burial of textile materials for estimating time since burial. As a result, the forensic significance of early-stage degradation of textile remains poorly understood. The time since burial of a textile plays a significant role in forensic reconstruction, as it influences

the extent of physical and chemical degradation. It also affects how reliably the textile can be linked to a specific event or individual.

In addition, factors such as soil composition, microbial activity, and moisture levels affect the rate and pattern of textile degradation. Previous studies have studied textile degradation under various conditions, including different seasons, soil types, or field sites (Nam *et al.*, 2015; Ueland *et al.*, 2017). Some research has focused only on specific types of textiles within a fixed burial environment (Arshad & Mujahid, 2011; Sölar & Devrim, 2019; Margariti, 2020). However, there is limited systematic research examining the effects of soil moisture on the chemical and structural degradation of textiles. Similarly, few studies have quantified how changes in textile chemistry under a specific burial condition only. This creates gaps in the understanding of degradation mechanisms that are important for forensic investigations.

More importantly, soil moisture is a critical factor that affects the microbial activity, oxygen availability, and chemical reactions that facilitate degradation. However, it has often been treated as a secondary variable rather than a primary focus in forensic textile research. Therefore, it is necessary to study the relationship between soil moisture and textile degradation. This can provide scientifically supported evidence for use in court and improve the interpretation of buried textile evidence in forensic investigations.

1.3 Research Objectives

1.3.1 General Objective

The objective of this study was to investigate the degradation of textile fibres (cotton, silk, and polyester) under different soil moisture levels (0%, 20%, 40%, and 60% of water-holding capacity) in a burial environment over a six-week period.

1.3.2 Specific Objectives

- a) To examine the morphological changes of cotton, silk, and polyester fibres during burial at varying soil moisture levels using stereomicroscopy.
- b) To analyse chemical changes and degradation patterns of the textile fibres across a six-week burial period using ATR-FTIR spectroscopy.
- c) To develop statistical models using identified spectral markers that can assist in estimating time since burial.

1.4 Significance of The Study

The study is of paramount importance in forensic science as it provides a clearer understanding on how textiles degrade under specific burial conditions. In addition, it improves the interpretation of degraded textile evidence by linking chemical changes observed in ATR-FTIR spectra of different textile fibres to burial duration and different soil moisture levels. With this information, forensic investigators can evaluate recovered textiles more objectively. Furthermore, the study also supports the estimation of burial duration. This information provides valuable insights into timelines and aids in reconstructing events in criminal cases.

Moreover, this study demonstrates how ATR-FTIR spectroscopy can be used practically as a sensitive and non-destructive analytical method for analysing chemical changes in textile fibres. Samples can be preserved for further analysis if chemical changes can be detected without destroying the evidence. By combining ATR-FTIR spectroscopy results with chemometric techniques, the study provides insights into the degradation mechanisms of different textile fibres. It also establishes a methodological framework that can be applied in forensic investigations in the future.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Textiles in Forensic Context

Textile materials can be classified based on their fibre composition into two main categories, which are natural fibres and synthetic fibres. Besides, there are regenerated fibres and blended textiles that combine different types of fibres to enhance the performance characteristics of the textile. Textiles usually originate from fibres and filaments, which are then spun into yarns and constructed into fabrics by woven, knitted or non-woven structures (Scholpp *et al.*, 2025).

The chemical composition and molecular structure of fibres determine their fundamental physical and mechanical properties, including tensile strength, flexibility, durability, and resistance to environmental degradation (Elfaleh *et al.*, 2023). These differences will affect the interaction of the fibres with moisture, heat, light, and microorganisms hence the degradation of the fibres under varying environmental conditions.

In forensic science, the examination of textile materials is important for fibres identification and comparison. Such analyses can help the investigators to identify the type, origin and manufacturing process of a fibre. This information is valuable as it can establish a connection by linking a textile sample to its possible source, manufacturer, or association with a particular object or individual. For example, Aljannah *et al.* (2022) conducted forensic analysis of synthetic textile fibres to establish correlations between different fabric fragments. Moreover, morphological features like fibre thickness, surface

texture, and cross-sectional shape can also help differentiate fibre types and reveal their manufacturing methods.

2.2 Textile Materials and Their Composition

Textile fibres can be broadly categorised into natural and synthetic fibres, such as cotton, wool, silk, polyester, nylon, and acrylic. Natural fibres are derived from plants, animals or insects. In contrast, synthetic fibres are man-made materials produced from polymers that do not occur naturally. They are usually manufactured from petrochemical sources through the polymerization of monomers, followed by extrusion into continuous filaments. Unlike natural fibres, synthetic fibres lack the complex mixture of biopolymers and microstructures found in plant- or animal-derived fibres (Smith & Thompson, 2017; Che Othman & Abu Hassan, 2023). This study focuses specifically on cotton, silk, and polyester due to their widespread use in clothing and their contrasting polymer compositions.

2.2.1 Natural Cellulose Fibres - Cotton

Plant-based natural fibres can be obtained from various parts of the plant such as seed, stem, bast, leaf and nut (Abdul Majid *et al.*, 2020). In general, this plant fibres consist of a complex network made up of three main biopolymers, such as cellulose, hemicellulose and lignin (Nautiyal *et al.*, 2025).

Cotton is one of the most common textile fibres used in clothing, household fabrics, and industrial materials. It is composed of about 90-95% cellulose, where it is a carbohydrate polymer made of D-glucose units linked by β -1,4 glycosidic bonds. Its

structure contains numerous hydroxyl groups (-OH) that contribute to its hydrophilic nature and mechanical strength.

Due to its well-characterised structure and high biodegradability, cotton is frequently used as a model material in textile degradation studies. For example, Li *et al.* (2010) compared the biodegradability of cotton and polyester. They found that all cotton samples were highly degraded under composting conditions, confirming that cotton is readily biodegradable. Similarly, Śmigieli-Kamińska *et al.* (2023) examined the impact of different disinfectants and sterilisers between dyed and non-dyed cotton fabrics. Their results showed that these treatments caused visible colour changes, fibre damage, and the alterations in fluorescence and spectroscopic characteristics.

2.2.2 Natural Protein Fibres - Silk

The common animal-based natural fibres are proteinaceous fibres in which they are mainly composed of protein fibre like fibroin or keratin. These fibres are more expensive than plant-based ones due to complex production and their desirable qualities.

Silk is an example of animal fibre produced by the larvae of silkworms, larvae, or spider. It is composed of the protein fibroin, which forms the structural core, as well as sericin, a gummy substance that binds the fibres together. Its smooth texture, lustre, and strength make it valuable for high-quality textiles.

Silk fibroin is a protein whose amino acid composition differs slightly between species, but its structure generally follows the same pattern. It contains hydrophobic blocks rich in glycine, alanine, and serine as well as the hydrophilic regions made up of

charged amino acids (DeFrates *et al.*, 2018). The hydrophobic segments align to form β -sheet structures which are responsible for high tensile strength in silk. On the other hand, the hydrophilic regions contribute to the overall flexibility and elasticity of the fibre.

2.2.3 Synthetic Fibres - Polyester

Polyester is the most popular kind of synthetic fibre used in textile material. It is composed of polyethylene terephthalate (PET), which is synthesised through a condensation reaction by forming ester linkages between ethylene glycol and terephthalic acid or its methyl ester (Dave *et al.*, 2019). This molecular structure contributes to its characteristic resistance to stretching, shrinking, and abrasion. In addition, the lack of hydroxyl or amide groups in its backbone makes it hydrophobic and less prone to moisture absorption.

Among the various types of synthetic fibres, polyester is the most commonly used in textile research as it is widely applicable and possesses chemical stability. Li *et al.* (2010) conducted a study with the use of polyester fabrics under industrial composting conditions. They found that these fabrics exhibited almost no biodegradation. Its strong ester linkages and hydrophobic nature limit moisture absorption and microbial attack. In general, synthetic fibres are far more resistant to environmental degradation compared to natural fibres, as they are not readily broken down by biological or chemical processes.

2.3 Degradation Mechanism of Textiles

Textiles are susceptible to degradation regardless of the environment in which they are subjected. Textiles may undergo degradation whether they are exposed to open air, buried in soil, submerged in water, or stored indoors over long periods. Degradation

of textile fibres refers to the process by which the fibre structure progressively weakens and undergoes molecular alteration. Over time, textiles will exhibit loss of strength, increase in brittleness, discolouration, or colour changes (Hayavadana & Srinivasulu, 2019). Generally, the degradation mechanisms of textiles can be grouped into three main processes, which are biological, chemical, and physical degradation.

Biodegradation refers to the process by which microorganisms, particularly bacteria and fungi break down the textile materials through the secretion of enzymes (Skilbeck *et al.*, 2025). When textiles are buried in soil, they are exposed to diverse microbial communities and the degradation is influenced by the type of microorganisms present and the fibre composition (Smith & Thompson, 2017). Several studies have investigated the biodegradation behaviour of different textile fibres under soil burial conditions. As an example, Sölar & Devrim (2019) examined the biodegradability of seven fibre types of textiles which had been buried in soil over a period of one month and four months. The findings revealed that cellulosic fibres like cotton and viscose degraded significantly, while synthetic fibres such as polyethylene terephthalate (PET) and polyacrylonitrile (PAN) exhibited minimal biodegradation.

Chemical degradation of textiles mainly occurs through hydrolysis and oxidation processes. Hydrolysis involves the chemical breakdown of polymer chains due to reactions with water, acids, alkalis, or enzymes. In damp or soil burial condition, the process begins when moisture from the surrounding environment diffuses into the polymer matrix and weaken the chemical bonds within the fibre (Fayyazbakhsh *et al.*, 2025). Cellulosic fibres such as cotton can undergo hydrolytic cleavage of glycosidic bonds in cellulose, leading to chain scission and loss of tensile strength. While the

hydrolysis of polyester breaks the ester bonds, thus shortening its polymer chains and increasing the number of carboxylic acid and hydroxyl end groups (Cionek *et al.*, 2020).

On the other hand, oxidation occurs when textile fibres are exposed to oxygen, pollutants, or ultraviolet (UV) radiation. Reactive oxygen species (ROS) attack the polymer structure, leading to cleavage of double bonds, oxidation of hydroxyl groups, and formation of carbonyl or carboxyl compounds. In cellulose fibres, oxidation can cause the rupture of glycosidic ether linkages, resulting in yellowing and embrittlement (Smith & Thompson, 2017).

Physical degradation occurs when mechanical or environmental forces irreversibly damage the molecular and structural integrity of textile fibres (Smith & Thompson, 2017). Continuous stress, friction, or pressure can weaken fibre bonds, reduce tensile strength, and lead to thinning or fragmentation of fabrics over time. For example, Mitchell *et al.* (2012) investigated the physical degradation of modern cotton and polyester-cotton shirting fabrics in burial environments within a forensic context. Their study reported obvious signs of physical deterioration, including staining, fibre disintegration, and reduced tear strength, with the most severe damage observed in clay-rich soils and longer burial durations.

Degradation can alter textiles in several ways. In forensic investigations, it is required to assess whether a fabric or fibre remains sufficiently preserved for accurate identification and analysis. Forensic investigators often consider several key questions, such as whether the observed damage resulted from natural decay or human activity, whether the textile's condition provides insight into the decomposition process, and

whether the clothing or its size could help in identifying the victim (Mitchell *et al.*, 2012; Smith & Thompson, 2017; Maximilian *et al.*, 2020).

2.4 Burial Environment and Textile Degradation

2.4.1 Soil

Soil can be defined as the layered material that can be found on the earth surface. It has resulted from chemical and biological processes and physical organisation of minerals and organic matter (Alex & Alfred, 2024). Soil is composed of three primary particle types including sand, silt, and clay. The relative proportions of these components determine the soil's texture, which in turn influences moisture and air holding capacity of the soil (Jeffers, 2025).

Soil can be vertically structured into several layers, including the topsoil, subsoil, parent material, and bedrock (Williamson, 2023). The topsoil is the uppermost layer and is the most biologically active zone as it contains organic matter, microorganisms, and nutrients. It is easily accessible by hand or shallow digging without heavy equipment. When textiles are buried, their degradation is strongly influenced by the surrounding soil conditions. Several key factors that affect this process are discussed in the following sections.

2.4.2 The Soil Properties on Burial Degradation of Textiles

The degradation of textiles in soil environment is highly dependent on the surrounding chemical, biological, and geological conditions. In addition to these broader environmental factors, specific soil properties also play a critical role in determining the rate and extent of textile degradation during burial. Key factors such as moisture content,

microbial activity, temperature, pH, soil texture and organic matter can contribute to both the physical breakdown and chemical modification of textile fibres.

Microbial activity within the soil is one of the key properties that influences the textile degradation intensity. The presence and activity levels of microorganisms will determine how quickly the textile fibres breakdown (Arshad & Mujahid, 2011). Microbial activity also depends on several factors, such as soil moisture, nutrient availability, and organic content. Decomposition begins when microbes attach to the fibre surface, proliferate, and secrete enzymes that reduce the degree of polymerisation of the fibre polymers. This biochemical process produces smaller and more degradable products that accelerate further breakdown of the material. For example, cellulose-based fibres are especially vulnerable to this process because cellulose serves as a primary nutrient source for many microorganisms (Ueland *et al.*, 2017).

Temperature is also a significant factor that influences the rate of textiles degradation as it directly affects microbial activity. Warmer conditions promote faster microbial reproduction, which in turn increases the rate of fibre breakdown. For example, a recent study by Marasović *et al.* (2025) showed that higher soil temperatures accelerate the degradation of mulch materials. Temperature also affects the production of cellulase, the enzyme responsible for cellulose degradation. Cellulolytic microorganisms generally produce cellulase more efficiently at temperatures between 25°C and 45°C (Rumi *et al.*, 2024).

The chemical environment of the soil, such as pH also influence the degradation rate of materials. According to ISO 11721-1:2001, standard soil burial tests specify that

the pH should be maintained between 4.0 and 7.5. This range provides an optimal condition for textile samples to undergo degradation in a controlled soil environment.

The physical properties of soil such as its texture, also influence the degradation of buried textiles. Although texture may not strongly affect large-scale visual degradation, it plays an important role in regulating oxygen availability and water distribution (Arshad & Mujahid, 2011). Soils with finer particles, such as silty clay loam, can restrict water movement and gas diffusion, which creates conditions that favour anaerobic microorganisms (Lowe *et al.*, 2013). Furthermore, loam and clay soils generally exert a greater influence on textile degradation than sandy soils because they retain more moisture and support more active microbial communities (Wang *et al.*, 2022).

2.4.3 Influence of Soil Moisture on Textile Degradation

Soil moisture plays an important role in influencing the rate of textile degradation in burial environment. While the presence of microorganisms, suitable temperature, and pH are necessary for degradation, the availability of water determines how effectively those organisms can access and chemically attack the polymer structures of textile fibres.

The soil moisture content in open-field or burial environments is largely influenced by weather conditions and seasonal precipitation. Since environmental factors cause moisture levels to fluctuate, experimental studies will regulate water content relative to the soil's water holding capacity (WHC). WHC refers to the maximum amount of water a soil can retain (Jessica *et al.*, 2023). It is determined by properties such as the soil texture. Soils with higher WHC can hold more water and therefore support greater microbial activity. In standardized soil burial tests, moisture is often maintained at a

specified percentage of WHC, commonly around $60 \pm 5\%$ to provide optimal conditions for microbial growth.

The effect of soil moisture on textile degradation varies according to the chemical composition and water affinity of the fibres. Water participates directly in the primary chemical reaction known as hydrolysis that breaks down most textile polymers. Cellulosic fibres with hydrophilic nature are highly susceptible to degradation in the presence of moisture (Sülar & Devrim, 2019). Proteinaceous fibres can also absorb water due to the presence of hydrophilic amino acid residues but generally to a lesser extent than cellulose. In contrast, most synthetic fibres are less hydrophilic and absorb little moisture, which makes them more resistant to moisture related degradation. Overall, soil moisture significantly influences textile degradation by affecting both microbial activity and chemical breakdown, with natural fibres being more susceptible than synthetic ones.

2.4.4 Forensic Significance of Buried Textile Evidence

Textiles are frequently encountered as trace evidence in forensic investigations, as they can establish connection between victims, suspects, and crime scenes. When these materials are buried, soil environmental conditions can alter their physical and chemical properties. Therefore, understanding how textile fibres degrade in soil is essential for interpreting their evidential value.

Analytical techniques usually allow forensic scientists to evaluate both macroscopic changes and molecular alterations in buried fibres. While most previous research has focused on textiles recovered with decomposing remains, there is limited work examining textiles buried independently for the purpose of estimating burial time.

For example, Lowe *et al.* (2013) demonstrated that chemical profiles obtained through gas chromatography-mass spectrometry (GC-MS) and attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) could be correlated with stages of body decomposition. Their findings showed that changes in lipids retained within the textile reflected the progression of tissue decay.

Such findings highlight the broader forensic potential of analysing buried textiles. By characterising how their chemical signatures evolve over time, it becomes possible to estimate the time since burial. It is a factor that can assist in reconstructing events and strengthening investigative interpretations. Overall, buried textile evidence represents a valuable but is still not fully explored in forensic casework.

2.5 Analytical Techniques in Degradation of Textiles

2.5.1 Non-Forensic Application

Major studies on the burial degradation of textiles in soil environment have been well established within the fields of materials science, environmental engineering and textile conservation. These studies aim to evaluate the rate and mechanisms of degradation by examining the physical, chemical, and mechanical changes that occur in fibres when exposed to environmental stressors. Therefore, various analytical techniques have been employed to characterise the extent of deterioration and to understand the behaviour of textile during burial degradation.

An earlier comprehensive study on textile biodegradation was conducted by Arshad and Mujahid (2011). They examined the natural degradation process of both natural and synthetic fibres, including cotton, linen, jute, wool, and polyester. The

research aimed to determine how microbial and environmental factors influence the rate of decomposition, thereby contributing to sustainable waste management strategies. The study employed visual observation, optical microscopy, and scanning electron microscopy (SEM) to assess physical damage such as fibre splitting, erosion, and surface roughening. FTIR spectroscopy was used to monitor chemical changes in fibres by detecting alterations in their characteristic absorption bands. Furthermore, thermogravimetric analysis (TGA) was applied to quantify thermal stability loss associated with microbial degradation. However, since TGA is a destructive technique, it has been used less frequently in later studies that prioritise sample preservation for further analysis.

Sülar and Devrim (2019) expanded on this work by studying seven fibre types, including both biodegradable (cotton, viscose, modal, Tencel) and synthetic (polylactic acid (PLA), polyethylene terephthalate (PET), and polyacrylonitrile (PAN)) materials, over one and four month soil burial periods. Their study combined weight loss analysis, SEM, and FTIR spectroscopy with measurements of soil total organic carbon and microbial content. They found significant chemical and structural degradation in cellulosic fibres, indicated by reduced O-H and C-O peaks in FTIR spectra. In contrast, synthetic polymers showed minimal change which confirms their resistance to biodegradation.

Margariti (2020) investigated the effects of microorganisms on cellulosic and proteinaceous fibres (cotton, linen, hemp, silk, and wool) using a simulated soil burial experiment. The study aimed to document specific degradation patterns of each fibre for conservation purposes. SEM was primarily used to observe the physical characteristic

changes of the fibres. For example, diagonal splits in cotton, fibrillation in bast fibres, and scale loss in wool were observed. Silk showed minimal deterioration due to its crystalline fibroin structure.

Overall, even though these studies were non-forensic in application, they demonstrate that microscopy analysis and FTIR spectroscopy are the preferred analytical methods for investigating textile degradation in burial environments. These techniques provide both qualitative and quantitative insights into morphological and chemical changes.

2.5.2 Forensic Application

Beyond non-forensic applications, the analysis of burial degradation can also be applied in forensic contexts. For forensic purposes, a combination of analytical methodologies is required to assess the physical and chemical changes associated with textile degradation. According to Smith & Thompson (2017), forensic textile examination commonly employs various microscopy techniques, such as visual inspection, light microscopy, and SEM, which are crucial for examining fibre morphology and identifying specific damage patterns. Chemically, ATR-FTIR spectroscopy is used to detect changes in functional groups. Spectroscopic methods such as X-ray diffraction (XRD) also help determine variations in polymer crystallinity and orientation. In addition, thermal analysis methods such as thermogravimetric analysis (TGA) and thermomechanical analysis (TMA) are used to quantify fibre loss and dimensional changes resulting from degradation.

While there are limited studies focusing solely on textile degradation in isolation within a forensic context, most research has examined the degradation of textiles in association with other variables. For instance, Lowe *et al.* (2013) employed ATR-FTIR and GC-MS to chemically analyse textiles recovered from buried pig carcasses. The purpose of using ATR-FTIR in this study was to qualitatively profile lipid degradation by-products, such as triglycerides and fatty acids, retained in the textiles. GC-MS was used to quantify specific fatty acids, such as palmitic, stearic, and oleic acids. This provided a more sensitive measure of decomposition progression and time since burial. However, this study focused mainly on examining the relationship between these by-products and the stage of soft tissue decomposition rather than on the degradation of the textiles themselves.

A study more closely related to textile degradation was conducted by Ueland *et al.* (2017). The degradation of cotton, polyester, and blended textiles exposed to soil surfaces were investigated during different seasons. ATR-FTIR was used to monitor the degradation behaviour of the fibres. Principal component analysis (PCA) was applied to the spectral data to reveal distinct clustering between early and late degradation stages. The use of statistical analysis confirmed that the onset of degradation depended on temperature, which occurred earlier during summer trials than in winter.

Besides, Aljannahi *et al.* (2022) focused on the forensic discrimination of synthetic textile fibres which aimed to link suspects, victims, and crime scenes through trace evidence. ATR-FTIR spectroscopy was employed to obtain chemical profiles of 138 synthetic fibre samples, including nylon, polyester, acrylic, and rayon. To manage the complex spectral data, advanced chemometric techniques were applied, such as PCA for

visualising natural clustering and soft independent modelling by class analogy (SIMCA) for classification. This robust approach achieved a 97.1% correct classification rate. It clearly indicates the potential of integrating spectroscopy with multivariate statistics for reliable and objective fibre discrimination in forensic investigations.

Overall, although research on textile degradation in forensic contexts is still limited, existing studies indicate that physical alterations can be examined using microscopy, while chemical changes can be profiled using ATR-FTIR spectroscopy. In addition, multivariate analysis can be applied to better interpret complex data. This approach supports reliable conclusions about degradation patterns for forensic applications.

2.6 Stereomicroscope

A stereomicroscope is a widely used optical instrument in forensic investigation for examining the surface morphology of samples with low magnifications (Ijjas Fahi *et al.*, 2024). It provides a three-dimensional view of the sample, which allows researchers to observe texture, colour changes, surface damage, and other physical alterations of the samples. Typically, a stereomicroscope consists of a pair of eyepieces and objective lenses, with magnification can be adjusted using the magnification changer knob. The total magnification is calculated by multiplying the eyepiece magnification by the objective lens magnification.

In forensic textile degradation studies, stereomicroscopy is useful for assessing macroscopic changes in fibres caused by environmental factors such as burial. For example, Ziogo *et al.* (2023) used a stereomicroscope to perform qualitative analyses of

textile damage. Their study examined pre-existing and new damage on cotton, synthetic, and blended fabrics exposed to pig carcass decomposition and associated insect activity.

Cotton, silk, and polyester fibres will exhibit different morphological features that can be tracked over time. For example, natural fibres like cotton may show fraying or surface roughening as indicators of degradation. In contrast, synthetic fibres such as polyester generally retain a smoother surface and are more resistant to environmental deterioration (Arshad & Mujahid, 2011). These observations provide initial qualitative information on the degradation state of fibres, which can be correlated with subsequent chemical analyses such as ATR-FTIR.

2.7 Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) Spectroscopy

Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy is a widely used analytical technique for characterising the chemical composition of textile fibres based on their functional group (Peets *et al.*, 2017; Paz & Sousa, 2024). The technique works by directing infrared (IR) radiation through an attenuated total reflectance (ATR) crystal onto the sample surface. As the IR light undergoes total internal reflection within the crystal, a small portion of the energy penetrates a few micrometres into the sample as an evanescent wave. Functional groups in the sample absorb specific IR frequencies according to their characteristic vibrations. The remaining reflected light is collected by a detector. The detector generates a spectrum with distinct peaks, which provides a molecular fingerprint of the sample.

ATR-FTIR provides chemical information regarding the presence, intensity, and changes of characteristic absorption bands. It allows the detection of polymer degradation through the changes in functional groups, peak intensity, band shifting, or the appearance of new peaks related to degradation. Chemical changes from degradation, such as bond cleavage, hydrolysis, oxidation, or cross-linking can appear in the IR spectra. This makes ATR-FTIR as a reliable tool for assessing the chemical stability of different fibre types under various burial conditions.

Different classes of textile fibres exhibit different degradation patterns that can be detected using ATR-FTIR. Cotton, a cellulosic fibre, is highly susceptible to environmental degradation due to its chemical composition. During degradation, the characteristic peaks associated with O-H stretching at around 3300-3330 cm^{-1} and C-H stretching at around 2900 cm^{-1} decrease in intensity as these bonds break due to microbial or hydrolytic attack on the cellulose chains (Sülar & Devrim, 2019). Furthermore, the C-O stretching region is especially sensitive to hydrolytic breakdown, with bands at 985 and 1001 cm^{-1} often diminishing or merging with the 1033 cm^{-1} band over time. The ratio of the 1001 to 1033 cm^{-1} band has been identified as a potential indicator of the extent of cotton degradation (Ueland *et al.*, 2017).

Silk exhibits remarkable preservation in burial environments compared to both cellulosic and other proteinaceous fibres (Margariti, 2020). This durability is largely attributed to the tightly packed β -sheet crystallites in its supramolecular structure. ATR-FTIR degradation analysis of silk focuses on the Amide I band at 1621 cm^{-1} and Amide II band at 1514 cm^{-1} , which correspond to the C=O stretching of peptide bonds and the coupled N-H bending and C-N stretching vibrations, respectively (Koperska *et al.*, 2015).

Degradation causes structural transformations from highly ordered β -sheet domains toward less structured β -turns and disordered conformations (Akyuz, 2019). As degradation progresses, the Amide II band decreases in intensity relative to Amide I. This results in a higher Amide I to Amide II ratio, which can serve as an indicator of silk fibroin degradation. However, these observations are primarily based on archaeological and historical silk. The study on short-term degradation under controlled burial conditions remains limited.

In addition, polyester, a synthetic textile fibre is relatively resistant to burial degradation compared to natural fibres. Its chemical stability is characterized by the preservation of the C=O stretching vibration, which appears as a sharp peak near 1700 cm^{-1} . This band represents the ester functional group of polyester. Theoretically, polyester is expected to degrade through hydrolysis, which involves the cleavage of ester bonds (Ueland *et al.*, 2017). This process typically occurs in the amorphous regions of the polymer. According to Ueland *et al.* (2017), such degradation would result in an increase in carboxylic acid and alcoholic end groups. However, their ATR-FTIR analysis on polyester textile confirms that the C-O stretching and C=O stretching bands remain almost unchanged, indicating the high resistance of synthetic materials.

Overall, ATR-FTIR provides a non-destructive and sensitive method for monitoring chemical changes in textile fibres. The spectral “molecular fingerprint” allows the identification of chemical structures and the detection of changes resulting from environmental degradation.

2.8 Chemometrics

After collecting experimental data, a standardised and systematic approach is required to analyse the large volume of information generated. In many cases, manual examination is often difficult, time-consuming, and insufficient as subtle changes are not easily detected by visual inspection alone. To address these challenges, chemometrics has become an essential tool in analytical and forensic studies.

Chemometrics was first established as a discipline in 1971 by Svante Wold and Bruce Kowalski (Otto, 2016). Since then, it has become an important tool in forensic science for enhancing the interpretation and optimisation of analytical techniques (Sauzier *et al.*, 2021). It applies statistical and mathematical methods to chemical datasets, including complex spectral data obtained from ATR-FTIR analysis. This approach allows researchers to manage large datasets, uncover hidden patterns, and identify meaningful correlations that would otherwise remain unnoticed. In the context of textile degradation and forensic fibre analysis, chemometric techniques help improve data interpretation by providing more objective results.

2.8.1 Principal Component Analysis (PCA)

Principal component analysis (PCA) is one of the most widely used chemometric techniques to visualise and interpret relationships within multivariate datasets. It works by reducing the dimensionality of large datasets while preserving the most significant sources of variation (Greenacre *et al.*, 2022). In ATR-FTIR analysis, the spectra obtained contains hundreds of absorbance values, which makes direct interpretation difficult. PCA addresses this by transforming the correlated spectral variables into a smaller set of