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Discovering Halite Traces on a Victim's Clothing through a Forensic Geoscience Analytical Approach: A Suspicious Case in Italy

Marcelo da Silveira Tortolero Araujo Lourenço [1,2,*], Rosa Maria Di Maggio [2,3], Chiara Germinario [1], Celestino Grifa [1], Francesco Izzo [4], Alessio Langella [4] and Mariano Mercurio [1,2]

[1] Department of Science and Technology, University of Sannio, Via F. de Sanctis, snc, 82100 Benevento, Italy; chiara.germinario@unisannio.it (C.G.); celestino.grifa@unisannio.it (C.G.); mariano.mercurio@unisannio.it (M.M.)
[2] International Union of Geological Sciences-Initiative on Forensic Geology (IUGS-IFG), Permanent Secretariat, 26 Baiwanzhuang Road, Xicheng District, Beijing 100037, China; rosamariadimaggio@gmail.com
[3] Geoscienze Forensi Italia®, Viale Mediterraneo, 77, 00122 Rome, Italy
[4] Department of Earth Sciences, Environment and Resources, University of Naples Federico II, Via Cinthia, 80126 Naples, Italy; francesco.izzo4@unina.it (F.I.); alessio.langella@unina.it (A.L.)
* Correspondence: m.lourenco@studenti.unisannio.it

Abstract: This suspect case focuses on investigating the presence of halite (NaCl) crystals on the clothing of a deceased individual to determine whether they resulted from immersion in seawater or residual absorption after immersion (i.e., the crystals were left on the clothing after contact with the victim's wet body). Thirteen clothing samples were collected from various garments worn by the victim and were subjected to optical stereomicroscopy, Scanning Electron Microscopy (SEM), coupled with Energy Dispersive Spectroscopy (EDS), and Simultaneous Thermal Analysis (STA). Optical stereomicroscopy revealed numerous white-colored, vitreous, and greasy luster microcrystals dispersed between fabric fibers, with higher concentrations observed near the hem seams and metal rivets. These microcrystals exhibited predominantly cubic and irregular morphologies. Additionally, sandy particles and organic elements, such as plant fragments and micro seashells, were detected, indicative of coastal environment exposure. SEM-EDS analysis confirmed the presence mainly of sodium and chlorine in stoichiometric ratios consistent with halite, with crystals exhibiting amorphous, needle-shaped, or cubic morphologies. Furthermore, STA analysis identified weight loss events attributed to organic decomposition and halite decomposition at high temperatures, corroborating SEM-EDS findings. The distribution and characteristics of halite crystals, along with other trace elements, support the hypothesis of immersion in seawater while wearing clothing. Specifically, the higher concentrations of halite crystals near thicker fabric portions and metal rivets suggest slower drying rates and longer evaporation times, indicative of immersion rather than residual absorption after swimming. This finding not only helps in determining the victim's exposure to seawater but also establishes a methodology for distinguishing between different sources of halite residue on clothing. Overall, the comprehensive mineralogical characterization of halite crystals on clothing samples, using best practices of forensic mineralogy, provides valuable forensic insights related to the circumstances that led to the victim's death. This approach aided investigators in reconstructing the sequence of events, enhancing the accuracy of forensic reconstructions. Moreover, this study contributes to the broader field of forensic geoscience by demonstrating the practical applications of mineralogical analysis in criminal investigations, potentially guiding future research and improving investigative techniques in similar cases.

Keywords: forensic mineralogy; halite; optical stereomicroscopy; SEM-EDS; thermal analysis

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1. Introduction

Criminalistics, one of the many aspects of forensic sciences, focuses on analyzing and comparing materials that may be exchanged among victims, suspects, and the surroundings of a crime scene [1]. Forensic comparative analysis involves the application of scientists' studies, experiences, understandings, and judgments across various fields, including physics, chemistry, biology, and others to examine forensic evidence [2]. In particular, forensic geoscientists use these skills to conduct comparative analyses of earth materials, and this has the potential to support criminal investigations [3–5]. Moreover, this contribution provided by forensic geoscientists can be used by law enforcement and courts across a range of criminal cases, primarily assisting investigators in establishing the when [6], where [6–9], and how [10] of a particular crime [5].

Given that earth materials encompass the solid constituents of the Earth, which comprise minerals, rocks, soils, various naturally occurring substances, and also man-made materials of geological origin (e.g., brick fragments, glass, mortar-based materials, ceramics, etc.), the forensic geologist is responsible for investigating these traces whenever relevant in a forensic context. Diverse examples of the use of such materials for forensic purposes mostly include comparative analyses of soils [11–15], sediments [16–18], brick, concrete, plasterboard, render [19], and even pollen [7,20–22].

Minerals are important components of most of the above-mentioned materials, sometimes providing relevant forensic information for the most diverse range of civil and criminal cases. For example, the utilization of heavy minerals in different forensic contexts is well documented, highlighting their significance in such investigations [23]. Additionally, mineralogy is used to investigate particles of man-made materials present in soils for forensic purposes [19]. Moreover, methodologies have been developed to discriminate forensic sandy soil samples based on mineralogical analyses [24].

In forensic cases involving coastal environments, the study of salt crystallization may play an important role within investigations. Actually, the use of different analytical techniques in studying naturally formed evaporite crystals enables us to obtain information regarding their growth conditions [25] that may be forensically relevant.

Halite (NaCl) is a soft mineral (Mohs scale 2–2.5) of the halide group, typically white-colored or colorless [26], that tends to crystallize in a cubic formation. Its natural morphology can largely vary, showing, for example, fibrous prismatic and needle-shaped forms whenever it occurs as a cementing agent [27]. However, when halite forms via rapid evaporation of an aqueous solution, the regular shapes of the crystals are normally lost [28].

Usually, the precipitation of this mineral in environments such as industrial evaporation ponds, saline pans, and sabkhas is the result of the desiccation of a previous saline--water-flooded area [29]. This process is generally fast, which results in high rates of crystal nucleation and low growth rate [30]. In addition, hypothetically, the higher the evaporation rate, the larger that salt crusts with even smaller halite crystals will form [31].

Besides the relevance of halite in addressing foundational questions in earth sciences, such as its use for hydrological studies [32] and paleoclimate reconstruction of saline environments [33], there are peculiarities in its crystallization processes and potential impacts on both natural and built environments that warrant detailed investigation. For instance, the formation of salt crusts due to the upward flow of evaporating water in microporous materials, which carries soluble salts, is a significant phenomenon. When ion concentration reaches a critical level, halite crystals form, resulting in efflorescence [34]. This process is not only of scientific interest but also has practical implications in various fields.

The formation of halite efflorescence has been recognized as a crucial research topic in building engineering [35–37] and heritage conservation [38,39], due to its potential to cause damage to structures and artifacts. Additionally, the role of efflorescent halite in inhibiting soil loss through aeolian erosion [40] and its ability to trap and preserve organic materials [41] highlight its broader environmental significance. Efflorescent halite, commonly found in various environments, can have significant implications in forensic science. This study investigates the efflorescent halite precipitated on a victim's clothing within a forensic context to provide insights into its formation and implications in a particular investigation.

Optical stereomicroscopy is a fundamental step in forensic investigations, providing an initial overview of the questioned sample [42,43]. This technique helps in recognizing and comparing trace evidence, directing the investigation toward more detailed analyses [44,45]. This technique was used within the case reported by this paper to identify and document the presence and distribution of halite crystals on the clothing.

Scanning Electron Microscopy–Energy Dispersive Spectroscopy (SEM-EDS) is instrumental in forensic geoscience, offering high-resolution visualization of sample morphology and elemental composition identification. Since the 2000s, it has been widely used in soil recognition and other forensic applications, such as art analysis and predictive geolocation [6,46–52]. For this case, SEM-EDS was utilized to examine the morphology of halite crystals and to confirm their elemental composition, ensuring that the crystals were indeed halite.

Thermal analysis, including techniques like thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential scanning calorimetry (DSC), dates back to the late 19th century. These methods are valuable in forensic science for investigating the physical and chemical properties of samples under varying temperature conditions [53–55]. Within this investigation, thermal analysis helped in understanding the thermal properties and stability of the halite crystals, providing insights into their formation conditions.

The primary aim of this project is to investigate the specific crystallization processes of halite and their implications in forensic geosciences. By applying the aforementioned analytical methods, we sought to understand the conditions under which halite precipitates in the investigated case.

Specifically, the techniques were applied to obtain forensically relevant information from halite in a suspected death case in Italy, where investigators needed to reconstruct the last hours of the victim's life to determine the manner of death. The focus of this study is on how the efflorescent halite found on a victim's clothing can contribute to resolving these forensic questions. If clothes have been immersed in seawater, the fabric would have microcrystals of marine salt both the inside and outside. On the other hand, the predominant presence of sea salt inside the clothing would indicate a residual absorption that took place after swimming without them. Therefore, this paper aims at presenting and discussing the details of the analytical activities in order to understand if the presence of sea salt crystals found on a victim's clothing is due to immersion of the victim with their clothes on or simply absorption by the victim's body after immersion without clothes on.

A Brief Overview of the Case

In a town on the southern seacoast of Sardinia (Italy), early in the morning, a man was found dead after jumping from the top floor of a five-story residential building. Investigators wanted to reconstruct the last hours of the victim to understand if the victim committed suicide or was murdered. According to eyewitnesses and the victim's friends, the night before the victim had been seen at a nightclub on the beach, right in front of the sea, and late at night, he was seen walking away alone to go home. The building from which the victim jumped was about 2 km from the beach. The victim's clothes were wet; so, it was important to ascertain the presence of sea water residue on the clothes and, if so, whether the victim had been into the sea dressed.

The authors asked the prosecutor for permission to write about this case, and permission was granted with the silent assent formula. Furthermore, the case was archived, and given the analysis was performed following article nr. 360 of the Italian Code of Criminal Procedures (not-repeatable analysis), all the samples remained at the laboratories. All the photos and analysis were repeated (after case filing) with the aim of writing this article. Therefore, the photos in this article were never used in court.

2. Materials and Methods

The clothes worn by the victim (Figure 1) at the time of death were carefully examined to search the possible occurrence of seawater residue. Once the presence of salt crystals and sand grains was confirmed (Figure 1A,B), attention turned to the identification of any biological traces or other clues useful for reconstructing the events that preceded death.

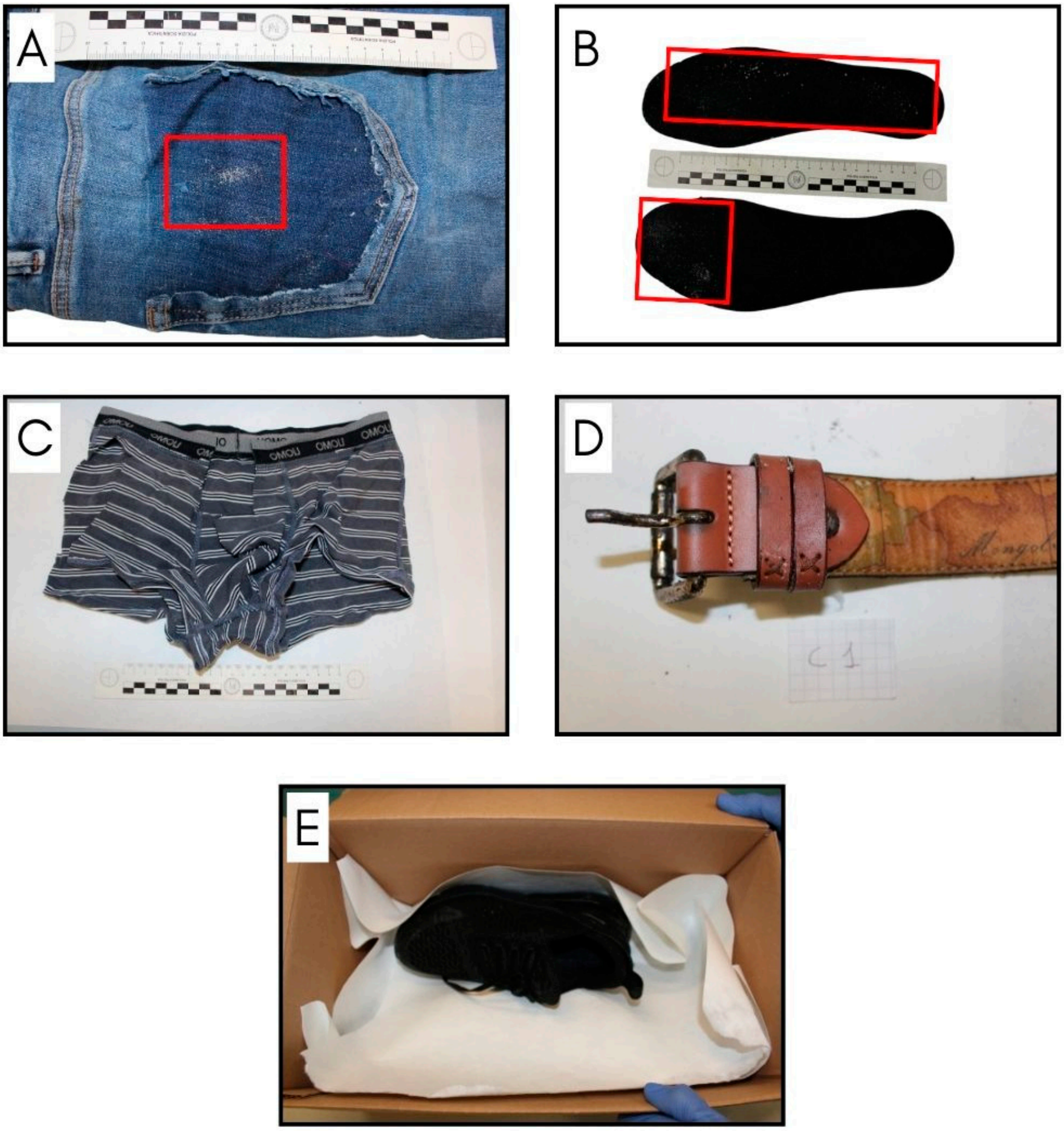

Figure 1. Victim's clothing: (**A**) trousers, on which there are visible traces of sand; (**B**) insoles of the victim's shoe, with traces of sand; (**C**) briefs; (**D**) belt; (**E**) sneaker.

Thirteen samples were collected from the victim's clothes (see Table 1), two of which were from his briefs (samples S1 and S2), seven from his denim trousers (samples P1, P2, P3, P4, P5, P6, and P7), one from the leather belt of his trousers (sample C1), one from his shirt (sample M1), and two from the insoles of his right and left shoes (samples SC1 and SC2, respectively).

Table 1. Detailed information of the thirteen collected samples from the victim's clothes.

Sample	Material	Description
S1	Briefs	Fabric taken from the left rear area, close to the crotch, where a light-colored trace is visible.
S2	Briefs	Elastic fabric taken from the left rear area.
P1	Trousers	External front side: oxidized metal rivet of the right pocket.
P2	Trousers	External front side: oxidized metal rivet of the left pocket.
P3	Trousers	External front side: fabric around the left pocket.
P4	Trousers	External front side: middle-right leg fabric.
P5	Trousers	External back side: right back pocket fabric.
P6	Trousers	External back side: left back pocket fabric.
P7	Trousers	Internal front side: right leg fabric below the pocket.
C1	Belt	Belt loop close to the buckle.
M1	T-shirt	Fabric from the back of the crew neck.
SC1	Shoe	Inner insole of the right shoe.
SC2	Shoe	Inner insole of the left shoe.

The trace sand and mineral encrustations present in all the samples described in Table 1 were analyzed under optical stereomicroscopy using the facilities of SCAR Labs srl ENTERPRISE (Italy). SEM-EDS and thermogravimetric analysis/differential scanning calorimetry (TGA/DSC), coupled with evolved gas analysis, were carried out at the Laboratory of Mineralogy and Petrography of the Department of Science and Technology (DST) of the Sannio University (Benevento, Italy) to identify the potential presence of salt crystals recrystallized on the cloths.

Such techniques were selected as each one is able to highlight some specific aspects that will hereafter be detailed and that all contribute to achieving the main goal of this investigation.

2.1. Optical Stereomicroscope

The thirteen samples were examined using reflected light at different magnifications (from 12× to 120×) to identify the possible occurrence of salt crystals, sand, organic remains (vegetable and animals), or any other trace of anthropic origin on the clothes. The instrument used was a Nikon SMZ1000 stereoscopic microscope working in a 12×–250× range of magnifications, interfaced with a Nikon DS-Fi1 camera (Nikon, Tokyo, Japan) and NIS Element F3.0 software.

2.2. SEM-EDS

SEM-EDS analyses were performed in single targets on the selected crystals' surfaces from seven of the thirteen samples (S1, P1, P3, P5, C1, M1, and SC2), previously selected based on the evidence emerging from stereoscopic microscope observations. The instrument used for this analysis was a Zeiss EVO 15 HD VPSEM (Zeiss, Oberkochen, Germany), operating at 12 kV accelerating voltage to record images, coupled with an Instruments Microanalysis Unit with Xmax 80 EDS detector (standard details used for EDS calibrations are reported in [56]).

Samples were mounted on stubs of 12.5 mm in diameter and then gold-coated with a Q150R ES Sputter Coater (Quorum Technologies, Lewes, UK).

2.3. Simultaneous Thermal Analyses (STA)

Thermogravimetry and differential scanning calorimetry were performed simultaneously using a NETZSCH STA 449 F3 Jupiter system (Netzsch, Selb, Germany), following a heating ramp of 10 °C/min for an interval temperature between 40 and 1050 °C in a pure air atmosphere (flow rate 60 mL/min). Furthermore, Evolved Gas Analysis (EGA) was carried out using the Fourier-transform infrared spectroscopy (FTIR) technique with Bruker's Tensor 27 system (Bruker, Billerica, MA, USA), connected to the STA system through a transfer line heated to 200 °C. The spectra were acquired using 32 scans per minute and a resolution of 8 cm^{-1} in an IR range between 4000 and 600 cm^{-1}. The data from the TGA/DSC analysis coupled with FTIR(EGA) carried out on a representative portion of the P3 sample (~12 mg) were acquired and elaborated with Proteus 6.1.0. and Opus 7.0 software, respectively.

3. Results and Discussion

3.1. Optical Stereomicroscope

All the fabrics examined under the stereomicroscope presented numerous white or colorless, vitreous-to-greasy luster, and translucent microcrystals. Such microcrystals, dispersed between the fibers such as in the samples P4 and M1 (see Figure 2A,B) but homogeneously distributed throughout the examined clothes, mostly occurring with irregular morphology (samples SC1 and P3, respectively—Figures 2C and 2D). Furthermore, the individual fibers were surrounded by microcrystalline encrustations with the same characteristics described above.

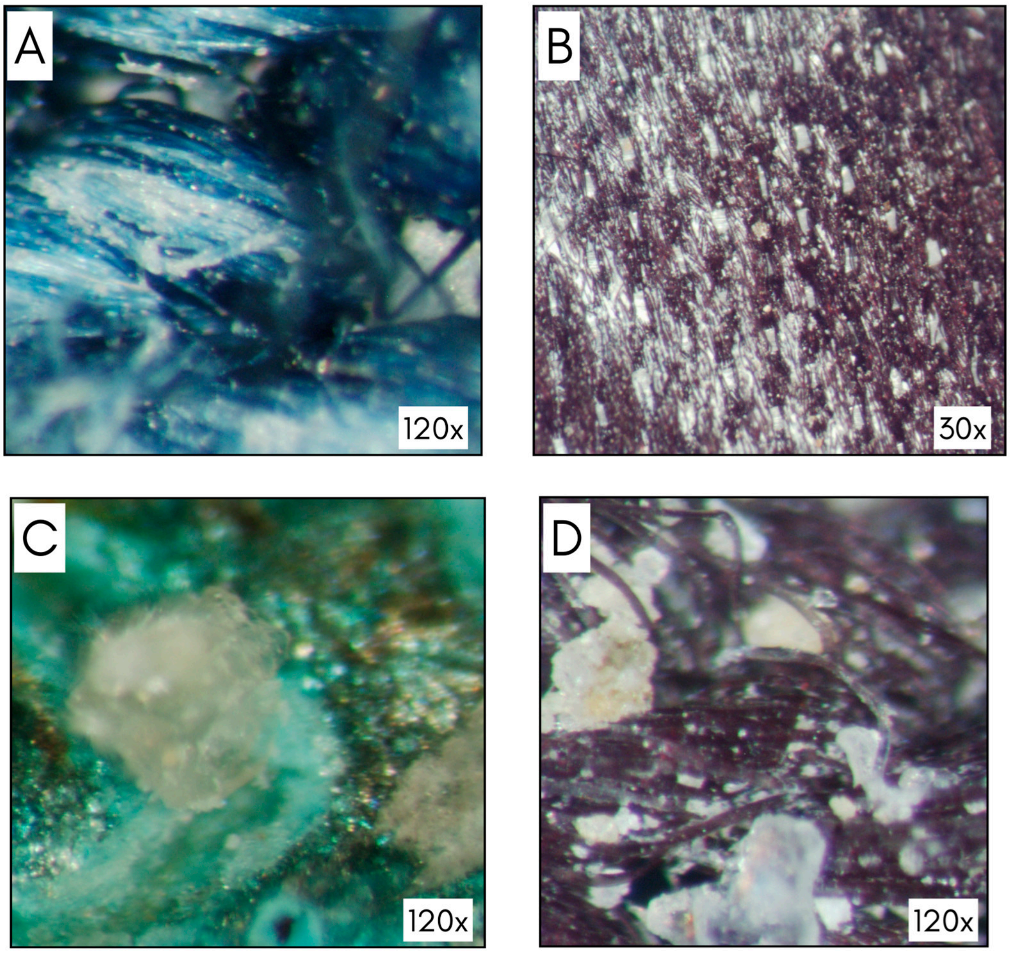

Figure 2. Microphotographs taken under a stereomicroscope showing white microcrystal(s) (**A**) between denim fibers (sample P4); (**B**) on the t-shirt neck tag (sample M1); (**C**) on the oxidized rivet (sample P1); (**D**) on the inner sole (sample SC1).

Larger crystals were observed near the hem seams of the pockets, where the fabric is thicker, and near the rivets, where the fabric is covered by the rivets themselves. Assuming that the clothing was wet (immersed in seawater), these portions of fabric would probably have dried more slowly in the air compared to other areas. This slower evaporation rate would allow for greater growth of salt crystals precipitated during evaporation. The relationship between slower drying rates and increased crystal size is well documented in the literature; slower evaporation allows for extended growth periods, resulting in larger crystals [30–32]. This observation is supported by research indicating that lower evaporation rates favor the growth phase over nucleation, leading to fewer but larger crystals [33]. In this study, the distribution and size of halite crystals are consistent with these findings, demonstrating how varying evaporation rates influence crystal growth and nucleation.

The rivets (samples P1 and P2) show a high stage of oxidation (Figure 3), presumably due to the contact with seawater. Furthermore, the rivets have numerous white, vitreous or greasy, as well as translucent microcrystals. Such microcrystals are arranged especially along their circumference, between the rivet itself and the fabric (sample P1, Figure 3A), and inside the words 'Original Denim' engraved on its surface (Figure 3B–D).

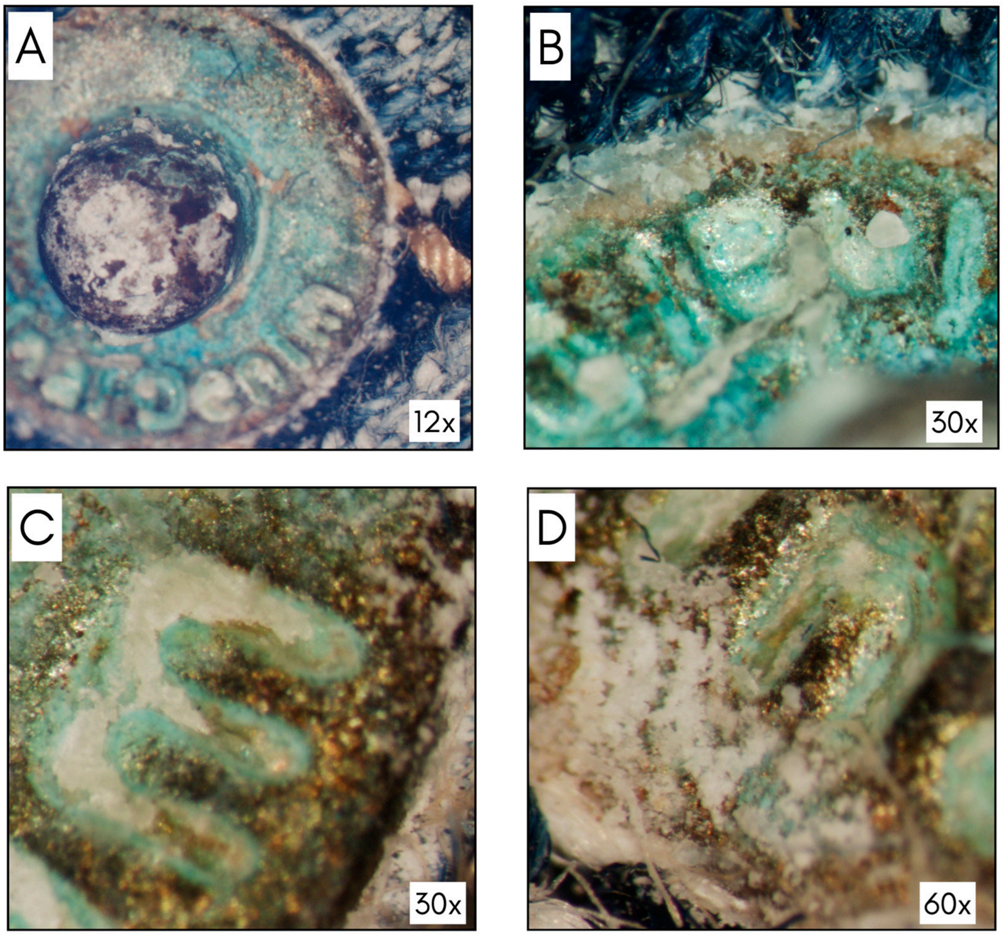

Figure 3. Microphotographs taken under a stereomicroscope, showing (**A**) white microcrystals arranged on the fabric in contact with the rivet (sample P1, 30×); (**B**) 'Original Denim' engraved on the surface of the right rivet (sample P1, 12×); (**C**) white microcrystals inside the letters engraved on the right rivet (sample P1, 120×); (**D**) white microcrystals near the letters engraved on the right rivet (sample P1, 60×).

Observations under a stereomicroscope also enabled us to detect the presence of numerous sandy particles and some organic elements, such as plant fragments and micro fragments of seashells, which are essentially compatible with marine coastal environments. The results from this technique indicated the need for further analyses through more detailed methods on the most representative areas of the clothing.

3.2. SEM-EDS

A SEM analysis allowed us to carry out a detailed morphological study of the samples. It showed that the single fibers of all the fabric samples were affected by extensive encrustations formed by amorphous (sample P3, Figure 4G,H), needle-shaped (samples P1 and C1, Figure 4D,E), or cubic crystals (samples S1 and SC2, Figure 4A,B).

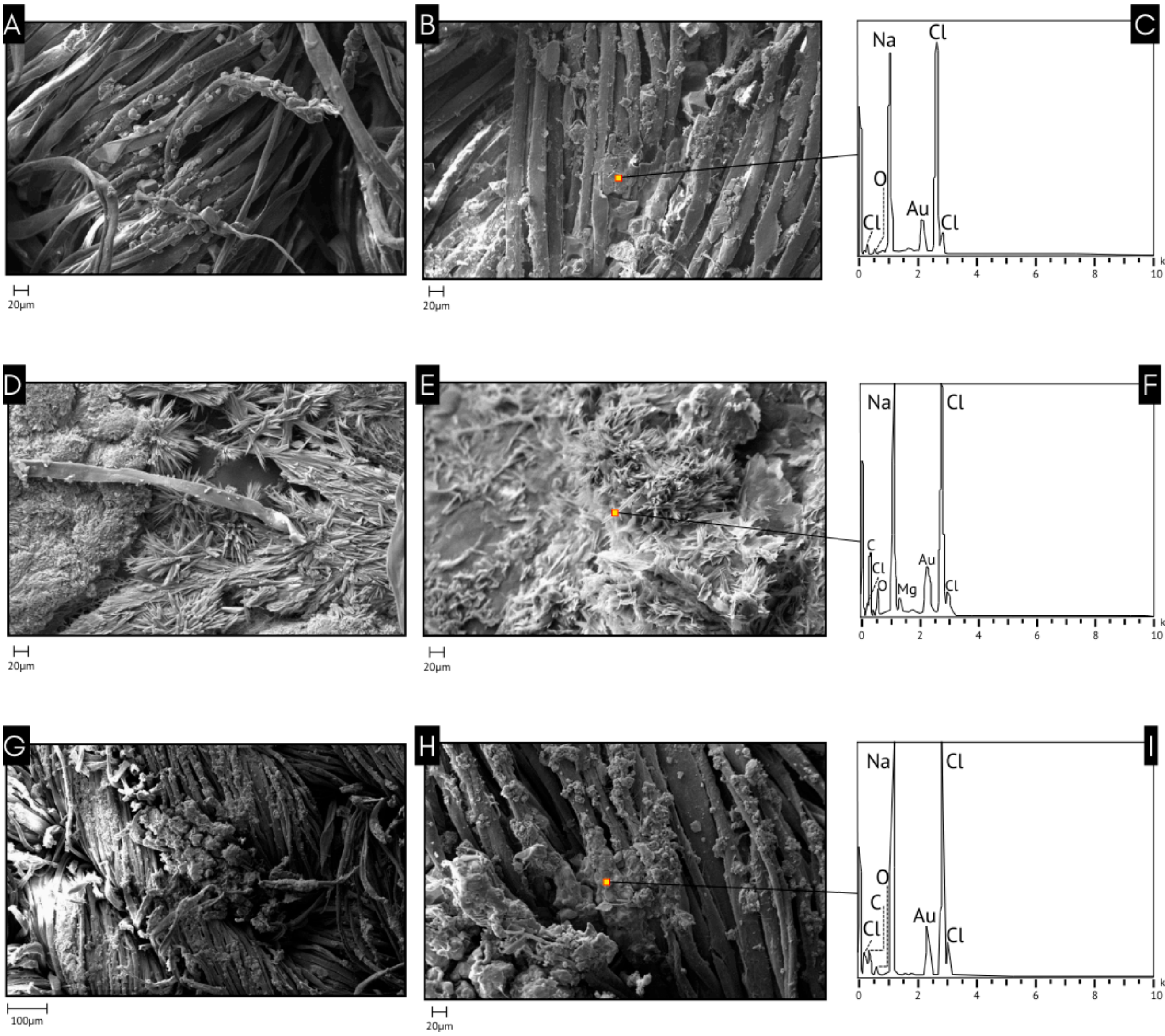

Figure 4. Cubic crystals observed in sample S1 (**A**) and sample SC2 (**B**), with its respective EDS spectrum (**C**); needle-shaped crystals observed in sample P1 (**D**) and sample C1 (**E**), with its respective EDS spectrum (**F**); shapeless crystals observed in sample P3 (**G**,**H**), with its respective EDS spectrum (**I**).

Moreover, X-ray microanalysis performed with EDS enabled identification of the chemical elements present in the encrustations within the fibers. Sodium (Na) and chlorine (Cl) in almost-identical stoichiometric ratios in the analyzed spots on the surface of different crystals were predominant, although traces of Mg, O, and C (likely due to the fibers on which the crystals grew) were detected (Figure 4C,F,I). This compositional evidence clearly suggests the presence of halite, the main mineralogical phase of sea salt, with varying precipitation and evaporation rates likely determining the different shapes identified.

3.3. STA

The TG curves and their first derivatives (DTG) (Figure 5A) highlighted for sample P3 six weight loss and related thermal events at 123 °C, 238 °C, 302 °C, 422 °C, and 466 °C. Such events result in an overall weight loss of approximately 92% and are generally attributable to decomposition and combustion processes typical of polymeric organic substances (fabric). These processes are characterized by a succession of endothermic (252 °C) and exothermic (325 °C, 425 °C, 453 °C, and 534 °C) effects (Figure 5A).

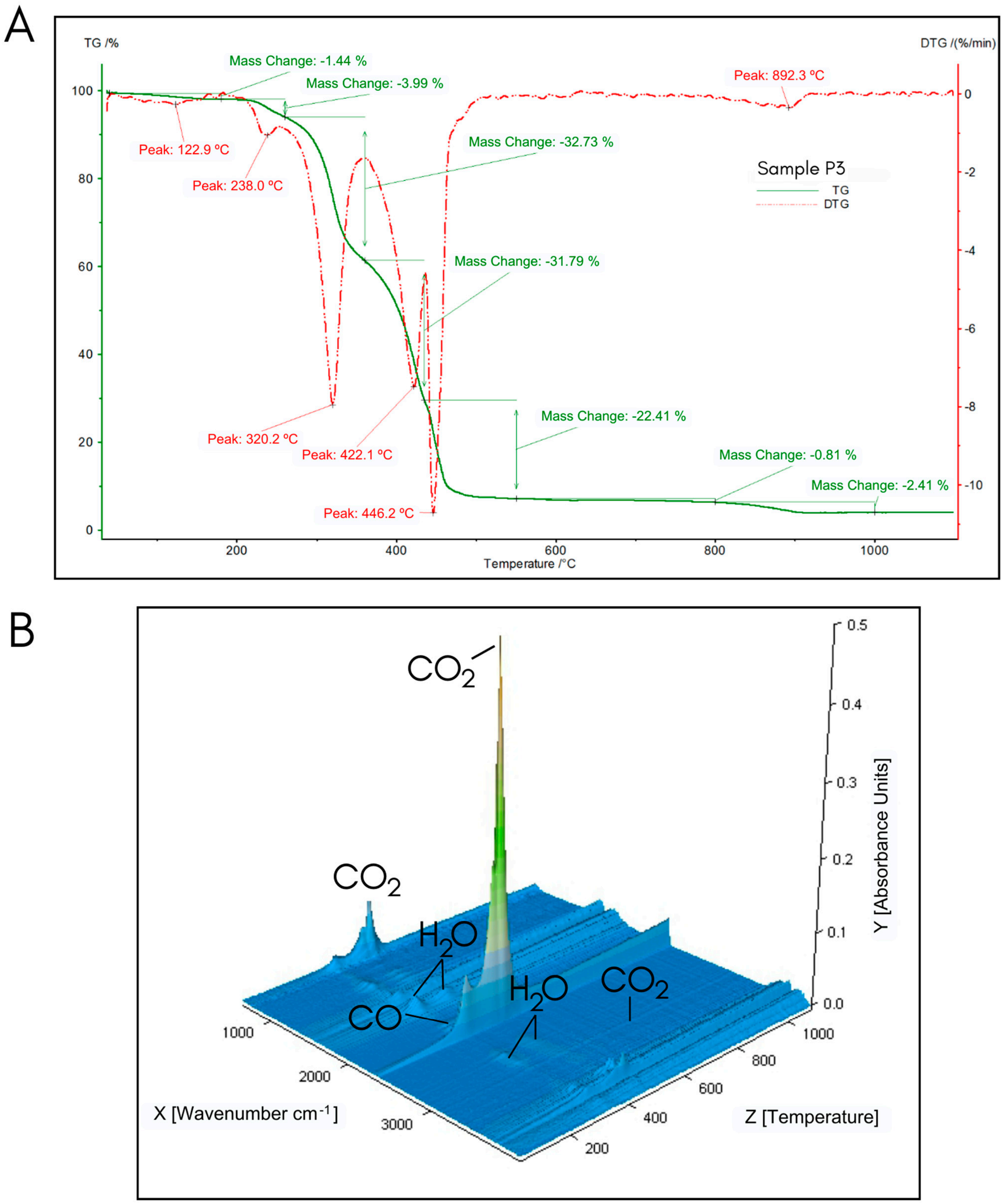

Figure 5. (**A**) TG, DSC, and DTG curves of sample P3; (**B**) 3D plot of FTIR-EGA for sample P3.

The endothermic stage might be associated with the initial decomposition of polymers, which require energy to break. On the other hand, the exothermic stages might indicate combustion and decomposition processes of the material. Combustion, if present, would be an exothermic process, where the polymer breaks down into smaller components, releasing energy in the form of heat. The gases emitted during these stages are mainly carbon dioxide (CO_2), water vapor, and traces of carbon monoxide (CO) (Figure 5B).

At the relatively low temperature of 325 °C, the primary products are carbon dioxide (CO_2) and water vapor (H_2O), with traces of carbon monoxide (CO). At 425 °C, the same gases (CO_2 and H_2O) continue to be released, alongside traces of CO. At higher temperatures, such as 453 °C and 534 °C, the quantities of CO_2 and H_2O decrease as the decomposition products become more volatile. Above this thermal range, a further weight loss (~2.4%) occurs at 892 °C, likely associated with the thermal decomposition of halite [54].

4. Conclusions

The set of analyses allowed the detection of numerous sandy particles, organic elements such as algae fragments typical of coastal flora, and micro fragments of seashells on the victim's clothing. Despite the numerous seawater residue materials found on the clothing (briefs, jeans, belt, shirt, and sneakers) at the time of death, the halite crystals on both the external and internal surfaces of the fabric, as well as on the metal rivets of the jeans, provide the most crucial evidence for clarifying doubts in this forensic investigation.

These traces are consistent with the fact that halite, composed of Cl and Na in a stoichiometric ratio of 1:1, is the main constituent of sea salt (99.5%) and recrystallizes through seawater evaporation [57–59]. Halite crystals were homogeneously distributed throughout the examined fabrics, presenting different shapes due to the different evaporation rates. Higher concentrations of crystals were observed in thicker areas of the fabric, such as the pocket edges and seams, or where the fabrics were covered by metal rivets, likely due to slower drying rates in these regions, resulting in more time for halite to precipitate during evaporation.

The presence of halite on the external surface of the clothing fabric suggests the victim was immersed in seawater while wearing the clothing, rather than the clothing absorbing seawater after being worn post-swimming (residual absorption). Therefore, the forensic mineralogy approach successfully addressed the prosecutor's question about how the clothes came into contact with seawater, providing substantial answers to resolve the case.

The methods employed in this study, including optical stereomicroscopy, SEM-EDS, and thermal analysis, proved to be robust. Optical stereomicroscopy provided an essential initial overview, guiding the more detailed analysis. SEM-EDS enabled precise visualization of the halite crystals' morphology and elemental composition, while thermal analysis offered insights into the physical and chemical properties of the samples under varying conditions. Each technique uniquely contributed to a comprehensive understanding of the evidence, highlighting their utility in forensic geoscience.

This work not only demonstrates the effectiveness of these analytical methods in a specific forensic context but also indicates their broader applications. The ability to accurately characterize and analyze halite crystals can extend to various forensic scenarios, such as determining the provenance of materials and assessing environmental conditions at crime scenes.

Furthermore, this study highlights the importance of interdisciplinary approaches in forensic investigations, combining geoscience with advanced analytical techniques to solve complex cases. By refining these methods and demonstrating their practical application, this work paves the way for future research and development in forensic geoscience, ultimately enhancing our ability to uncover and interpret critical evidence in diverse forensic contexts.

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