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Forensic Discrimination of Various Subtypes of Regenerated Cellulose Fibers in Clothing Available on the Consumer Market

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Abstract: The discrimination of five subtypes of regenerated cellulose fibers, i.e., viscose, bamboo, lyocell, modal, and cupro, from both men's and women's clothing available on the prevalent apparel market was described. The examinations were conducted using optical microscopy (in transmitted white light and polarized light), scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM–EDX), and Fourier Transform Infrared Spectroscopy (FTIR). The microscopic methods revealed characteristic features of the morphological structure of the examined fibers, enabling the identification of differences between the subtypes. As a result, the microscopic methods were found to be the most effective for identifying and distinguishing between the types of examined fibers. Although the FTIR technique did not allow for distinguishing between the fiber subcategories, it contributed to the enlargement of the IR spectra databases for regenerated cellulose fibers. Based on the findings, a general scheme of the procedure for identifying the tested fibers was proposed.

Keywords: commercial clothing; fibers identification; optical microscopy; SEM-EDX; FTIR

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1. Introduction

Man-made textile fibers modified during the manufacturing processes may consist of artificial (semi-synthetic) fibers, which are made with natural long-chain polymers that are chemically treated only partially and synthetic fibers that originate from synthetic materials due to chemical synthesis (chain-building processes). The first artificial fiber was rayon, classified as a regenerated cellulose fiber [1]. Currently, there is a boom in regenerated cellulose fibers on the clothing market. These fibers are an intelligent material that can replace cotton and other natural fibers such as silk and linen. Their softness and delicacy significantly enhance the comfort of wearing clothes, making them widely popular among customers. They are used to produce everyday garments as well as specialized medical apparel or, for example, day and night underwear designed for health-conscious consumers.

The use of cellulose fibers within the global textile market is increasing, making them one of the fastest-growing fiber groups in the textile sector. Cellulose fibers have a low ecological footprint and consist of 100% renewable carbon. They are bio-based and biodegradable, even in marine environments, where their degradation does not leave any microplastic particles—unlike synthetic fiber clothing, which is estimated to be responsible for 35% of this type of residue currently present in the world's oceans. Moreover, numerous production technologies for extracting cellulose, mainly from plants, can be applied for its recycling, offering the possibility of material circularity [2]. It is foreseen that wood pulp will be used in approximately 7% of global textile fiber production by 2025. However, cellulose fiber producers are currently looking for substitutes to wood as a raw material. The fibers can be made from residues, by-products, and side-streams of various industrial processes [2].

In turn, the task of a forensic expert in the field of textile and fiber examinations is to learn about and track new types of clothing and textile products, including fibers, appearing on the consumer market [3]. Consumer studies indicate that viscose fibers were the third most chosen type of fiber by women in the clothing they purchased in 2021, with an almost 11% share, following cotton (38%) and polyester (33%) fibers [4].

Next, it is important to assess the possibility of identifying or comparing modern fibers when they are present as evidence in a case as well as to evaluate the usefulness of the methods used in the laboratory for this purpose and for expanding existing databases [5]. The standard EN ISO 20705 method, commonly used in textile laboratories for fiber identification [6], cannot be directly applied to forensic sciences due to the specificity of the material being examined, i.e., often limited to only a few fragments of single fibers. Therefore, standards dedicated to forensic purposes have been developed [5].

The presented research aims to assess the possibility of distinguishing, from the perspective of forensic sciences, different subtypes of regenerated cellulose fibers originating from the selected clothing available on the market, which resembled the typical evidential materials. This issue is at the center of interest of fiber experts working in forensic laboratories around the world because fragments of single fibers are one of the most frequently examined physical microtraces revealed at the scenes of crime or on objects and the bodies of people who may be related to them. The discrimination of various subtypes of regenerated cellulose fibers has not yet been exhaustively analyzed in the forensic context in the available scientific literature.

2. Materials—Theoretical and Practical

2.1. Fiber Subtypes Selected for Examination

The production process of regenerated cellulose fibers consists of dissolving pure cellulose, squeezing it through a spinning nozzle, and then solidifying the cellulose fibers during coagulation or evaporation [7]. For this study, five subtypes of regenerated cellulose fibers were selected for analysis and are briefly characterized below, primarily from a utilitarian perspective.

2.1.1. Viscose (CV)

The first generation of cellulosic fibers, namely classic viscose fiber, is one of the most popular and is obtained through a technological process known as the viscose process. Low heat resistance and heat conductivity make viscose hygroscopic but not thermoplastic. This fiber is resistant to mildew as well as to discoloration and weakening. Viscose is used to produce clothing, underwear, and furnishings, often mixed with cotton, polyamide, or polyester fibers due to its soft character and static-free properties. Additionally, it is one of the cheapest fibers implemented in the production of rugs and carpets [8].

2.1.2. Bamboo (BB)

The bamboo fiber is a novel fiber with antibacterial and breathable properties [9–11]. It is a type of viscose fiber manufactured from cellulose pulp derived from the bamboo plant. It is very popular as a fiber for garments and is marketed as environmentally friendly due to its renewable source [9]. It absorbs water well and is easy to dye. In the textile industry, bamboo fiber can be applied on its own or in combination with other materials such as silk and cotton [10].

2.1.3. Modal (CMD or MD)

The second generation of cellulosic fibers, particularly modal—the most popular among them—is produced through the modified viscose process, resulting in a higher level of strength and lower extension at the break. This fiber can be washed and dried in a machine as it maintains its shape; however, it can absorb more water than viscose and 50% more than cotton [12]. It has a shiny, luster, and gloss structure and also has better resistance to swelling and elastic recovery [13]. Cotton is being replaced by more expensive modal in materials like bedding, outerwear, towels, sportswear, and underwear due to its softness and coziness. The modal production process requires relatively more energy, although the fibers are 100% biodegradable.

2.1.4. Lyocell (CLY)

The third generation of cellulosic fibers, i.e., lyocell, is another type of 100% cellulosic fiber that is also produced from wood pulp; however, the cellulosic molecules in this fiber are oriented alongside the axis and have a higher crystallinity and polymerization degree [14]. Lyocell is a product of a technological innovation process that is eco-friendly and devoid of toxic solvents while also being as soft as silk. Commercially known as Tencel®, it is widely produced by the company Lenzing AG (Manufacturer Lenzing AG, Lenzing, Austria) [15]. It is characterized by high strength when wet or dry and may absorb more water; nevertheless, it remains stable during washing. Similar to modal, lyocell is displacing cotton in the production of underwear and sportswear apparel (better air penetrability makes clothes airier), fashionable clothing, and nonwoven fabrics.

2.1.5. Cupro (CU or CUP)

Cupro is a regenerated cellulose fiber made from wood pulp or cotton linters. During the production process, the cellulose is dissolved in a mixed solution of copper salts and ammonia. Cupro is classified as an eco-textile and is biodegradable due to its solubility in the soil without harming the environment. It is used primarily in summer dresses and blouses and is sometimes combined with cotton to produce textured fabrics with uneven surfaces [16].

2.2. Original Material

The presented studies included ten artificial cellulose fiber samples used to manufacture clothing for both men and women available on the commercial market. The garments were made of knitted or woven fabrics, with most using only one subtype of regenerated cellulose fiber. Among them, there were two made from each type: viscose, bamboo, lyocell, modal, and cupro. The clothing items were selected from various popular, primarily international, brands produced and available in different countries around the world (Table 1).

Table 1. Studied material data.

Sample Marking	Type of Clothing	Commercial Brand	Country of Production	Fabric Type	Fabric Composition
1V1	Women's trousers	H&M	India	Knitted	100% viscose
2V2	Women's blouse	H&M	Indonesia	Woven	100% viscose
3B1	Men's socks	Gatta	Poland	Knitted	93% bamboo (6% polyamide, 1% elastane)
4B2	Women's shorts	tosh. tosh.	Poland	Woven	100% bamboo
5L1	Women's shirt	Camaïeu	Bangladesh	Knitted	100% lyocell
6L2	Men's shirt	Zara	Türkiye	Woven	100% lyocell
7M1	Women's housecoat	Oysho	Camboya	Woven	94% modal (6% elastane)
8M2	Men's socks	Zara	India	Knitted	54% modal (25% polyamide, 11% polyester, 8% metallized fiber, 2% elastane)
9C1	Women's dress	Marimekko	Romania	Woven	100% cupro
10C2	Women's skirt	Reserved	Türkiye	Woven	66% cupro (34% viscose)

3. Methods

3.1. Optical Microscopy

Observations of the fibers present in the research fabrics were performed with the use of a high-powered, bright-field, and polarized light microscope—Eclipse E600 Pol

(Nikon, Sendai, Japan)—with magnification ranging from 125× to 500×. Images were recorded using a Digital Sight DS-Fi3 high-definition color microscope camera, while the NIS Elements AR system was used for data processing (Nikon, Sendai, Japan).

For detailed high-power microscopy examination, fiber samples were placed on glass microscope slides (Menzel-Glaser, Braunschweig, Germany) with a drop of pure glycerin as the mounting medium (Merck KGaA, Darmstadt, Germany) and covered with coverslips (Menzel-Glaser, Braunschweig, Germany). Three microscopic slides of each sample were prepared and taken from different areas of the clothing. During the observations, the features of the fibers' morphological structure visible in their microscopic images were compared. The principal optical properties of fibers were examined under polarized light.

3.2. SEM-EDX

Special sample preparation was required for the SEM-EDX study. From each of the fabrics, one sample was collected, composed of a group of representative, untwisted fibers. Such samples were placed on 12 mm aluminum stubs with double-sided adhesive carbon tabs (TAAB Laboratories Equipment Ltd., Berks, UK). The specimens were covered with graphite using a JEC-530 sputter (Jeol, Tokyo, Japan) to prevent electric static charging. Observation of the fibers and their elemental analysis was performed employing a scanning electron microscope JSM-6610 LV (Jeol, Tokyo, Japan) combined with an energy dispersive X-ray spectrometer with SDD X-Max80 detector and Inca Energy 5.04 software (Oxford Instruments, High Wycombe, UK). The detector was hyphenated with SATW (super atmospheric thin window), and its resolution was 127 eV for MnKα at 10,000 counts. The applied measurement conditions were the following: accelerating voltage: 20 kV; live time: 20 s; and magnification in the range of 100–4000 times. It was found that at higher magnifications, e.g., 4000×, the electron beam concentrated on a relatively small area of the fibers quickly caused damage to their surface.

3.3. FTIR

Infrared spectroscopy was performed for all research fibers using a Nicolet iS50 FTIR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) with built-in, all-reflective diamond ATR. The spectra were obtained by collecting 32 scans ranging from 400 cm^{-1} to 4000 cm^{-1}, with a spectral resolution of 4 cm^{-1}. Later, they were pre-processed in OMNIC 9 Spectra Software (Thermo Fisher Scientific, Waltham, MA, USA) applying background correction and smoothing. Fabric samples, specifically, warp and weft threads in the woven fabrics and one thread in knitted fabrics (after removing fibers not subject to testing in mixed compositions), were verified directly by performing three repetitions of measurements on different threads of each sample. Before each measurement, a spectrum of the background was collected.

4. Results and Discussion

4.1. Optical Microscopy Analysis

Figure 1 collates selected images of fibers from each of the five examined subtypes of regenerated cellulose fibers obtained in both transmitted white and polarized light.

Based on the optical microscopy studies, conclusions can be drawn about the structure of specific subtypes of regenerated cellulose fibers (Figure 1).

The typical microscopic image of viscose fibers was one of the most distinctive and could be easily recognized when identifying their fragments. Viscose was characterized by several parallel stripes [17,18] and appeared to be composed of multiple individual fibers joined together (samples 1V1 and 2V2). Similarly, the surface of bamboo exhibited numerous striations parallel to the fiber axis. However, these strands appeared thinner in the samples examined by the authors (samples 3B1 and 4B2), and the fiber itself looked as if it was composed of a dozen or so individual, interconnected ones. The examined viscose and bamboo fibers exhibited the same behavior under crossed polars when oriented at 45°

and 315°. For colorless samples (or parts of them), the observed colors were subdued blue and brown.

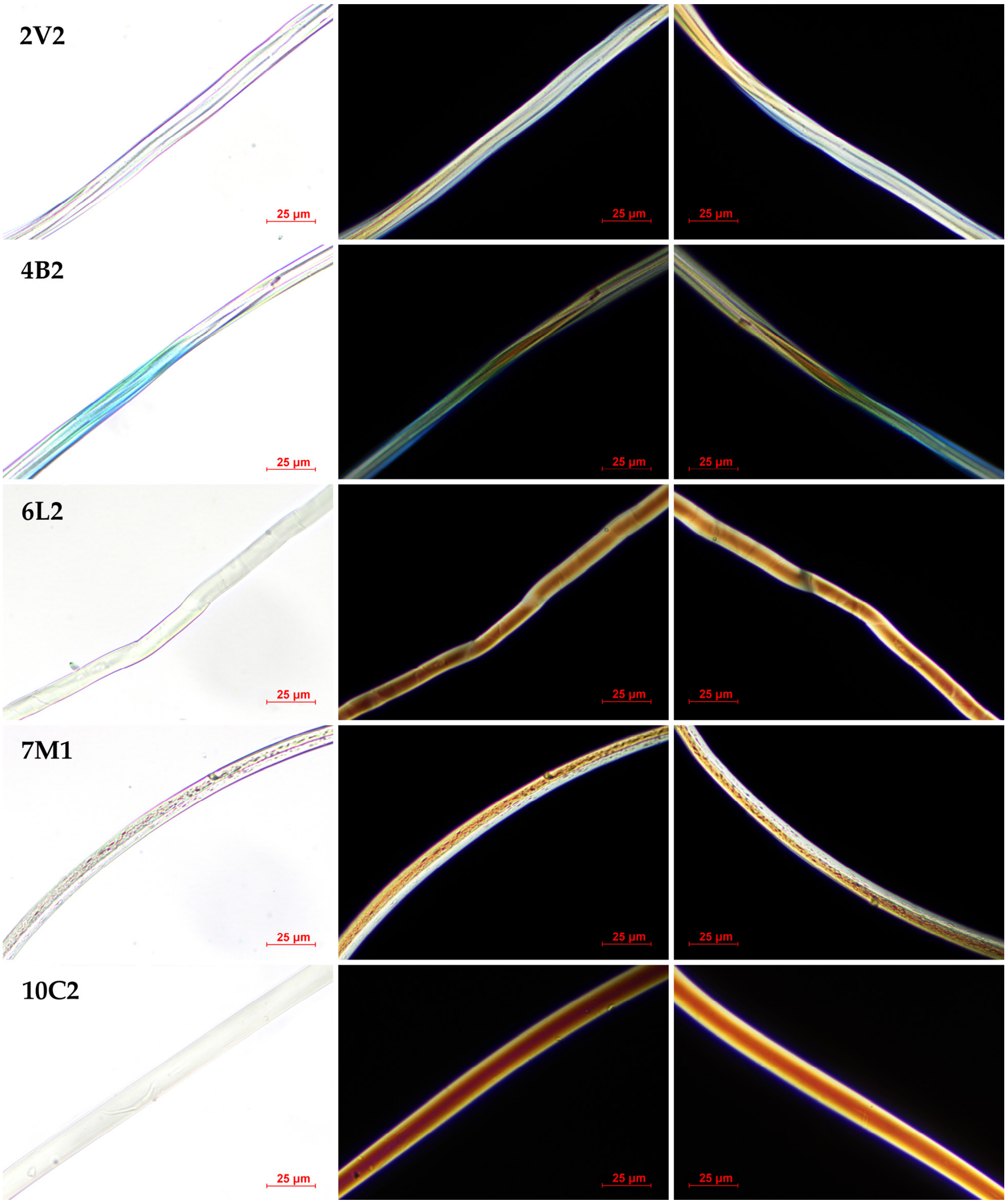


Figure 1. Selected microscopic images of examined fiber samples in transmitted white and polarized light.

For the lyocell (samples 5L1 and 6L2) and cupro fibers (samples 9C1 and 10C2), a smooth surface was observed, and in the cross-section, these fibers were round or nearly round (slightly oval). Both subtypes exhibited the same behavior under crossed polars when the fiber was oriented at 45° and 315°. The observed colors for colorless samples were mainly yellow and brown. The intensity of these colors was higher for the examined cupro fibers, and the colors themselves also seemed to be much more regular along their entire length. On the surfaces of the lyocell and cupro fibers, a fibrillation process was observed; this was a longitudinal splitting of the fiber into a bundle of microfibers of smaller diameter [3].

In the images of modal fibers (samples 7M1 and 8M2), a smooth surface with few but deep striations and a slightly twisted structure was observed. Modal exhibited the same behavior using crossed polars when the fiber was oriented at 45° and 315°. The observed colors for colorless samples were mainly yellow and white.

4.2. SEM-EDX Analysis

The morphological features were observed using secondary electron imaging (SEI) at different magnifications. Ultimately, 1000× magnification was chosen to obtain more detail for identifying and characterizing the structure of two neighboring fibers. In Figure 2, examples of SEI images of two neighboring fibers as well as single ones are shown for each subtype of the analyzed regenerated cellulose fibers.

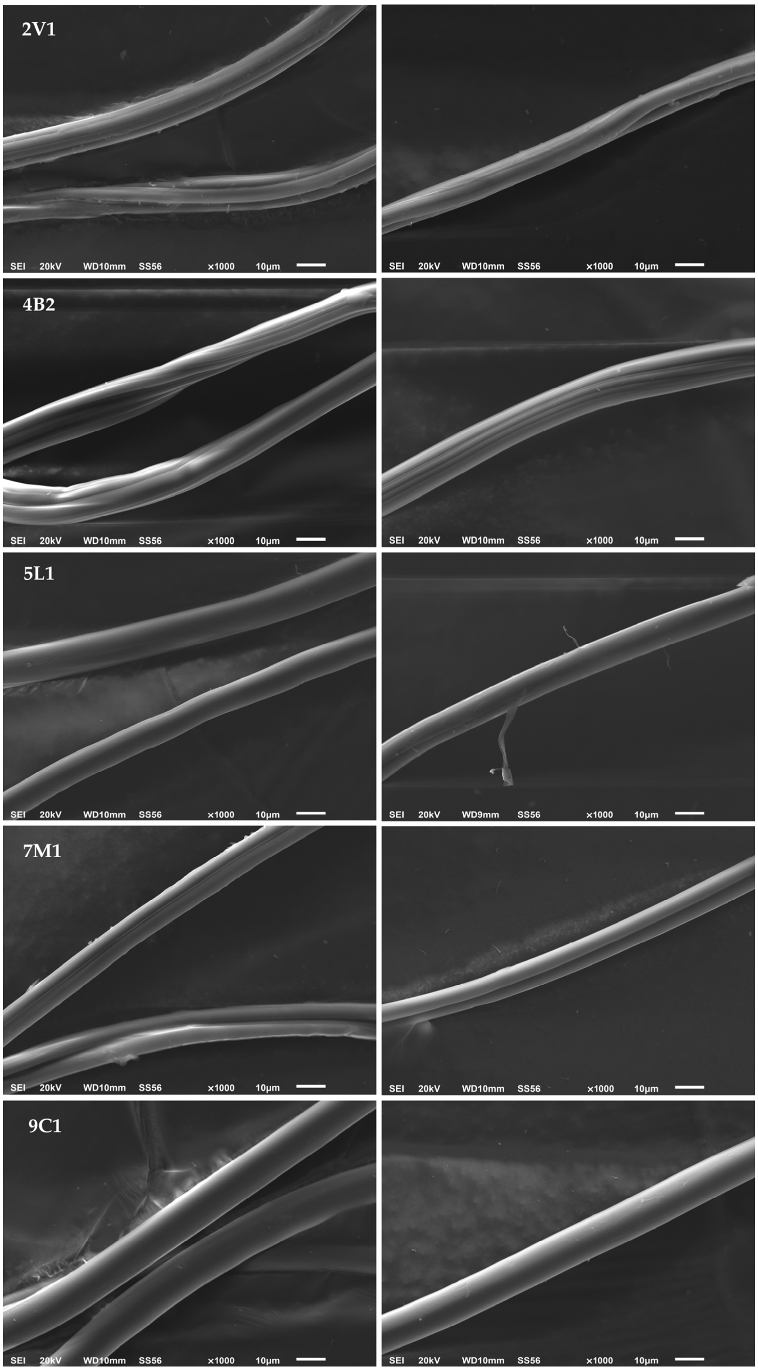


Figure 2. Selected SEM images of examined samples at 1000× microscope magnification.

The SEM examinations confirmed the consistency of the shape characteristic of individual fiber subtypes observed using optical microscopy. During the testing of the samples, the cross-sectional shapes of each subtype were determined by comparison with literature data, and the observations were consistent with these findings [19].

Based on the X-ray spectra obtained during the elemental analysis, significant peaks for carbon (C) and oxygen (O) were observed. Thus, it can be established that the examined regenerated cellulose fibers primarily contained organic compounds. Although some sodium (Na), silicon (Si), sulfur (S), and calcium (Ca) were detected in some areas of the fibers from samples 2V2 and 6M2, their content was not significant. It is assumed that they may have originated from textile finishing processes or impurities, such as dust, present in the air, which may have settled on the samples' surface from the environment. This could occur since the samples were prepared in a laboratory, in an open system, and the original clothing was exposed to external factors such as transport from the manufacturer, storage, display in stores, and handling by numerous customers before purchase. For this reason, they resembled typical evidential materials.

4.3. FTIR Analysis

No significant differences were found between the spectra obtained from different parts of the same samples. Therefore, the spectra of each examined regenerated cellulose fiber (samples 1V1-10C2) were compared with one another, and selected examples are presented in Figure 3.

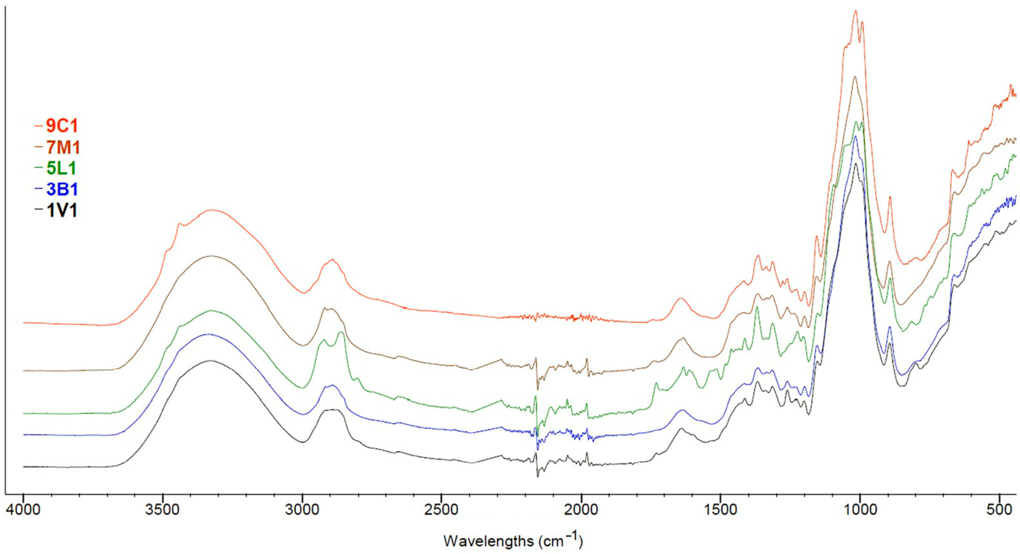
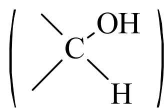


Figure 3. Selected ATR-FTIR spectra of examined samples.

Due to the high similarity between the spectra, only one of them (sample 9C1) was selected for a detailed analysis of individual bands and characteristic bonds (Table 2). This analysis remained in agreement with the literature data [20–25]. The FTIR spectra indicated that cupro fibers were predominantly composed of crystalline cellulose II, as evidenced by the distinct band at 894 cm^{-1} [20,21].

Concluding the results of the analysis carried out using the FTIR technique, it should be underlined that this technique allows for determining the type of the primary component (regenerated cellulose) of the examined fibers. However, taking into account the obtained results, it was not possible to distinguish between the individual subtypes of regenerated cellulose fibers. Furthermore, the commercial FTIR databases existing in forensic laboratories identified each of the subtypes as "viscose" or "rayon" fibers.

Table 2. The most important absorption bands present in the spectra of cupro fiber samples (9C1) together with their presumed origin.

Absorbance Range	Presumed Origin of the Bands
3326 cm^{-1}	O–H, hydroxyl groups contained in cellulose, and water
2892 cm^{-1}	C–H, stretching vibrations
1644 cm^{-1}	O–H of water absorbed
1365 cm^{-1}	CH_2 groups of cellulose, symmetrical bending
1336 and 1313 cm^{-1}	C–H bending vibrations, CH_2 wagging vibration
1156 cm^{-1}	C–O–C asymmetric stretching
1016 cm^{-1}	C–H and C–O present in polysaccharides, stretching vibrations
894 cm^{-1}	 ring vibrations, C–O–C vibrations

5. Recommended Method of Identification of Regenerated Cellulose Fiber Subtypes in Forensic Sciences

Fiber research conducted in forensic sciences has its specificity and, despite the availability of many research methods and techniques, only a select few can be used in practice. This is partly due to the limitation of research material, e.g., to several fragments of single fibers, which additionally cannot be destroyed during examinations as they constitute evidence in a criminal case.

Therefore, based on the results of experimental research on various subtypes of regenerated cellulose fibers present in commercially available clothing, the role of optical microscopy examinations, in both transmitted white and polarized light, as the most important part of their identification and discrimination should be emphasized. These non-destructive examinations allow for the differentiation of typical viscose and bamboo fibers from modal, lyocell, and cupro fibers. Subtle differences observed between viscose and bamboo fibers during the presented study can also be utilized in laboratory practice. Microscopic images of lyocell, modal, and cupro fibers in transmitted white and polarized light exhibit many characteristic features, facilitating their differentiation. The proposed method has limitations, i.e., if the same textile products contain cupro and lyocell or viscose and bamboo fibers, the possibility of distinguishing them would be restricted. However, the authors of the publication could not find clothing with this composition on the market.

If the results of the optical microscopy examination suggest that the fibers likely belong to one of the five discussed regenerated cellulose subtypes, their chemical composition should be confirmed using FTIR methods (ATR-FTIR or micro-FTIR in the case of identifying single fibers), which are classified as non-destructive testing techniques. Analyzing the obtained spectra may lead to the conclusion that the detected bonds may be characteristic of regenerated cellulose.

Complementary examinations can be performed using the SEM-EDX method, which offers highly detailed SEI images at very high magnification; however, it does not allow for further examination of fibers already analyzed, such as color comparisons. An additional advantage of this method is its ability to closely observe fiber ends and even their cross-sections. In particular, cross-sectional analysis can provide valuable insights that may help distinguish between the analyzed fibers. However, it often necessitates specialized equipment, such as a microtome to avoid distorting the fiber ends and to ensure proper sample preparation. The preparation of cross-sections from limited amounts of fibers, especially in forensic examinations, poses a risk of destruction to the evidence (single fibers). In light of this, these examinations should only be recommended if a sufficient number of evidential fibers is available.

Although SEM-EDX cannot permit differentiation of the tested fibers, and the application of high-energy electron beams may cause damage on their surface, such damage to textile fibers is rare in laboratory practice.

6. Conclusions

This publication focused on identifying and distinguishing different subtypes of regenerated cellulose fibers, primarily from a forensic perspective. Summarizing the considerations presented earlier and the obtained results, a general scheme for the identification of regenerated cellulose fibers is recommended.

Finally, the authors of this work aimed to systematize the methods employed during the investigation, along with the amount of information provided, to facilitate the distinction between different subtypes of regenerated cellulose fibers.

The key methods that provided detailed information about the structure of the fibers were optical microscopic techniques, including both transmitted white and polarized light. These techniques enabled the recognition of characteristic features in the morphological structure of the examined subtypes of the regenerated cellulose fibers.

Complementary imaging using the SEM-EDX technique typically provides a more detailed analysis of fiber surfaces and ends at higher magnifications.

In this context, the FTIR technique may have limited usefulness because it primarily offers general information about the types of bonds and functional groups present in the main components. Nevertheless, the application of FTIR can effectively distinguish between fibers of different origins and identify natural plant fibers as opposed to regenerated cellulose fibers. Additionally, these studies can help expand the limited commercial databases of FTIR spectra for regenerated cellulose fibers, which are becoming more common in the consumer market.

The analysis of the chemical composition using SEM-EDX provides elemental analysis of the fiber surface and may also be useful for detecting specific elements or potential impurities in them. However, the potentially destructive nature of this method must be taken into account when conducting a forensic analysis of the five subtypes of regenerated cellulose fibers examined in this publication.

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