

Article

The Characterization of Commercial and Historical Textiles Using a Combination of Micro-Chemical, Microscopic and Infrared Spectroscopic Methods

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Abstract

The aim of this study is to identify the textile materials used to make Bulgarian folk costumes and to support the process of conservation and restoration of ethnographic objects. In the 18th and 19th centuries, folk costumes were made almost exclusively of natural materials, while in the first half and middle of the 20th century, they included contemporary synthetic and regenerated cellulose materials, as well as blends of these materials with natural fibers. A series of historical textiles and contemporary industrial fabrics were studied using a variety of analytical approaches, including micro-chemical staining and solubility tests, optical microscopy, scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), and infrared (IR) spectroscopy. IR measurements were carried out in attenuated total reflectance (ATR) and external reflectance (ER) modes using a portable IR spectrometer, which enabled non-invasive analysis. The analysis revealed that the composition of the industrial fabrics and historical textiles encompassed synthetic fibers, such as polyester and polyamide, while others were made of regenerated cellulose fibers like viscose. Additionally, some textiles had a mixed composition of cotton and polyester or silk and viscose. The combined analytical approach provided reliable identification of both the synthetic and natural textile materials.

Keywords: textile fibers; modern fabrics; historical textile; ethnographic costumes; micro-chemical analysis; optical microscopy; SEM-EDS; IR



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1. Introduction

The cultural traditions of Bulgaria are vibrant and diverse, and a particularly vivid expression of this rich cultural heritage is folk costumes. Traditional Bulgarian costumes refer to the folk clothing worn by Bulgarians until the mid-20th century and they are related to the specifics of the Bulgarian people's traditional culture and way of life. The different ethnographic regions of the Balkan Peninsula where Bulgarians live have their own unique

costumes that reflect the local conditions, lifestyles, and aesthetic preferences of the people [1–6]. Today, costumes are used for artistic performances, and some artistic elements are incorporated into applied arts, arts and crafts, and less frequently, contemporary clothing.

Over four years, in the framework of the INFRAMAT project (National Research Infrastructure), extensive research was undertaken with the goal to expand the knowledge of textile materials during different periods. The purpose of these studies is to be able to determine the composition of different textile samples in order to support the process of conservation and restoration of ethnographic objects, such as traditional Bulgarian folk clothing and other movable cultural property made of textiles from the collection of the National Ethnographic Museum. Cognizance of the material used determines how the museum objects are to be preserved, maintained, exhibited, and restored. At the same time, it helps us select contemporary materials suitable for restoring historic textiles and partially reconstructing them when appropriate [7,8].

Since folk costumes from the 18th and 19th centuries were made almost exclusively of natural materials such as wool, cotton, silk, linen, and hemp, the initial focus of this study was directed mostly toward research on these materials. In the second phase of the project, we shifted our focus to more modern materials used for costumes in the first half and middle of the 20th century, such as mercerized cotton, viscose, polyester, polyamide, and acetate silk, as well as blends of these materials with natural fibers.

Our study encompassed folk costumes from Bulgaria and from the region of Gora, situated in present-day Kosovo (19 villages), with a smaller part located in Albania (nine villages). The costumes were donated to the Museum by the Society of Bulgarians- Muslims from the Gora region. It is interesting to note that the traditional clothing of the Gorani people (Figure 1) continues to be worn both in daily life and on special occasions [9]. It bears a closer resemblance to the colorful attire of Bulgarian Muslims from the Rhodope Mountains and differs significantly from typical Bulgarian folk costumes. The differences are evident in the individual components of the clothing, the rich and sparkling decorations, and the distinctive bright blue color.



(a)



(b)

Figure 1. Pictures of an unmarried woman (left) and a bride (a); a wedding dance from the village of Shishtavec, Albania (b). Reproduced with permission from V. Toncheva, *The Unknown Gora* (Original Title: *Nepoznatata Gora*); published by IIK Pod: Sofia, Bulgaria, 2012.

In this study, we have also included a number of samples of contemporary industrial fabrics and threads, which were intended for restoration purposes. For identification of the composition and the morphological features, we applied different analytical approaches—micro-chemical tests, optical microscopy, scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), and infrared (IR) spectroscopy including IR measurements in attenuated total reflectance (ATR) mode with a benchtop IR spectrometer and external reflectance (ER) mode with a portable IR spectrometer, which allowed for non-invasive characterization.

2. Background: Established Methods for Analysis of Natural and Artificial Textile Materials

2.1. Feeling Test

This test involves fiber identification by touching the fabric (Table S1, Supplementary Material). It requires much experience of handling and skilled perception. It is not easy to reliably identify and compare the fabrics made of different fiber contents. Still, for example, cotton feels good against the skin due to its softness and suppleness, while jute fiber feels stiff and harsh.

2.2. Burning and Solubility Tests

Identification of the fibers was also based on examination of the odor, residue, or how the fiber behaves when brought near a flame. Every fiber displays a specific characteristic behavior [10] (Table S2, Supplementary Material). For example, natural cellulose fibers as well as regenerated cellulose fibers, when burned, produce a smell of burning paper, whereas natural silk and wool produce a smell like burned hair. Polyester releases a slightly sweetish smell, and nylon releases a celery-like odor [11].

The solubility test is a non-technical test which involves treating the fibers with certain solvents such as H_2SO_4 , NaClO , NaOH , acetic acid, formic acid, chloroform, methylene chloride, methylene dichloride, dimethyl formamide, xylol, *m*-cresol, etc. (Table S2, Supplementary Material).

2.3. Micro-Chemical Tests

The chemical reactions most commonly used for fiber identification include (1) the Herzberg test; (2) a phloroglucinol test; and (3) a ninhydrin test.

Herzberg stain is a selective stain for non-lignin-containing fibers. Practically every vegetable fibrous material containing large quantities of lignocellulose is colored yellow. The removal of their lignocellulose content changes the staining effect from yellow to a blue or wine-red color. Cotton and linen rags as well as thoroughly bleached manila hemp are colored in a wine red [12].

When added to a fiber with a high lignin content such as flax or hemp, the colorless to pale-yellow phloroglucin reagent makes the fiber turn red. The intensity of the red color gives an indication of the amount of lignin. A bright, deep red or magenta color indicates a high lignin content in the fiber. A colorless or pale-yellow color indicates that the fiber has no or minimal lignin content [13–15].

The ninhydrin test consists of a chemical reaction that determines whether a sample contains amines or alpha-amino acids. The main reactant in this process is ninhydrin. The marker for a positive ninhydrin test is a deep blue coloration obtained in the solution [16]. This test is successfully implemented for identification of protein-based fibers [17].

2.4. Optical Microscopy

Light microscopy represents an instrumental method that is successfully used for the identification of textile fibers. This method of testing gives quick, accurate results and is easy to perform. Fibers of natural origin, i.e., cotton, wool and silk, have characteristic features. Cotton has characteristic twists in its separated fibers; wool shows characteristic scales which allow its fiber to interlock with other fibers. Flax and hemp show a bamboo-like structure, and silk has very thin fibers. Man-made fibers can be easily differentiated from natural ones. Rayon (viscose), for example, has a perfectly smooth shaft of fibers which differentiates it from cotton [18–21]. The characteristic morphological features of natural and man-made textile fibers are summarized in Table S3 (Supplementary Material).

2.5. Scanning Electron Microscopy

Scanning electron microscopy (SEM) is also used for visualization of the longitudinal and cross-sectional characteristics of textile fibers [22–28]. It requires more sophisticated instrumental equipment but provides imaging at magnifications down to several microns and information on the elemental composition, when coupled with energy-dispersive X-ray spectroscopy (EDS) analysis. SEM–EDS analysis can provide useful information about the preservation state and mineralization of historic textile fibers (gradual replacement of the original organic composition of the fibers by metal corrosion products) [29], as well as the presence of inorganic components such as mordants and other additives.

2.6. IR Spectroscopy

Fourier transform infrared (FTIR) spectroscopy have been proven to be a very fast, reliable, and easy-to-handle method for analysis of textile fibers [29–41]. It can provide completely non-destructive analysis when attenuated total reflectance (ATR) or external reflectance (ER) techniques are used. Due to their specific chemical composition and, consequently, their characteristic vibrational spectra, protein, cellulose, and synthetic fibers can be successfully differentiated by type. With the exception of natural cellulose fibers, differentiation is also possible between individual representatives within the same fiber class.

The applicability of IR spectroscopy for the identification of textile materials and the study of their degradation processes has been demonstrated by the investigation of excavated textile samples from the Vesuvian Area, ancient Ainos (Enez–Turkey), Kerameikos and Nikaia (Greece) [29–33], ethnographic textiles such as traditional folklore costumes, pillowcases, and other artefacts [34–36], and modern textile materials [37–41].

3. Materials and Methods

The studied industrial textile samples and museum textile objects are presented in Figure 2. Small samples (several textile threads) were taken from the objects for micro-chemical analysis, while spectroscopic IR analysis was carried out directly on textile objects.

Microscopic observation was performed using an optical microscope (Amplival Pol, Carl Zeiss Jena, Germany, equipped with Carl Zeiss Jena plan achromat objective 874,152). The samples were examined under a general magnification of 312.5 $\times$, which was achieved by combining an ocular lens with a magnification of 12.5 $\times$ and an objective lens with a magnification of 25 $\times$.

The textile fibers were studied by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy using a JEOL JSM 6390 (Tokyo, Japan) instrument. The images were acquired with a detector for secondary electrons. The accelerating voltage was 20 kV. EDS analysis was carried out by using a 10 mm² detector with a 140 eV resolution (INCA Oxford Instruments). The surface of the samples was thinly gold-coated prior to analysis.

The chemical reagents used in the micro-chemical test included ninhydrin (Riedel-De Haën™, Seelze near Hanover, Germany) and Herzberg stain. Herzberg stain (known also as Cl-Zn-I reagent) is obtained by mixing two separate solutions (assigned as solution A and solution B). Solution A represents 20 g of zinc dichloride (S.A. Limpes, Barcelona, Spain) dissolved in 10 mL of distilled water and solution B contains 2.1 g of potassium iodine (Chimspectar, Sofia, Bulgaria) and 0.1 g of iodine (Sigma-Aldrich, Steinheim, Germany) dissolved in 5 mL of distilled water.



Figure 2. Studied industrial fabrics and museum textile objects: (a) industrial fabric 1, (b) industrial fabric 2, (c) industrial threads 3, (d) industrial fabric 4, (e) industrial fabric 5, (f) white shirt—from region Gora, (g) white skirt—from region Gora, (h) black waist-band with red braid, garment of silk dress—from Bulgaria, region Sofia, and (i) pink headscarf—from Bulgaria.

The industrial fabrics and historic textile items were studied using ATR-FTIR spectroscopy by directly fixing the fibers or the fabric to the surface of the ATR accessory and collecting the spectra in the interval $4000\text{--}600\text{ cm}^{-1}$. The spectra were recorded by accumulating 100 scans at a resolution of 2 cm^{-1} on a FTIR Bruker Invenio R FT spectrometer with a diamond crystal ATR accessory (Pike Technology, Madison, WI, USA). Air was used as a background spectrum. The ER-FTIR non-invasive study was performed on a portable IR spectrometer Bruker Alpha II in reflectance mode. The experimental parameters used were as follows: 128 scans, a resolution of 2 cm^{-1} , and a gold-covered mirror used as a reference spectrum.

The sources of reference textile fibers used are as follows: cotton—commercial product Cotton Wool made in Turkey for Avans LTD, Gabrovo, Bulgaria; flax—commercial product skirt “Trachten Redl”; silk—silk crepekin Talas; wool—commercial product wool hat; polyester—commercial product scarf “Esmara”; polyamide—commercial product swimming suit “EXTRE”; acrylics—commercial product kid’s vest “George”; hemp, jute, viscose, and polyolefin—available at the lab.

4. Results

4.1. Optical Microscopy and SEM-EDS Analysis

The samples of the fibers (several threads or small pieces cut from the textile) were initially examined by optical microscopy and afterwards by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (Figures 3 and 4).

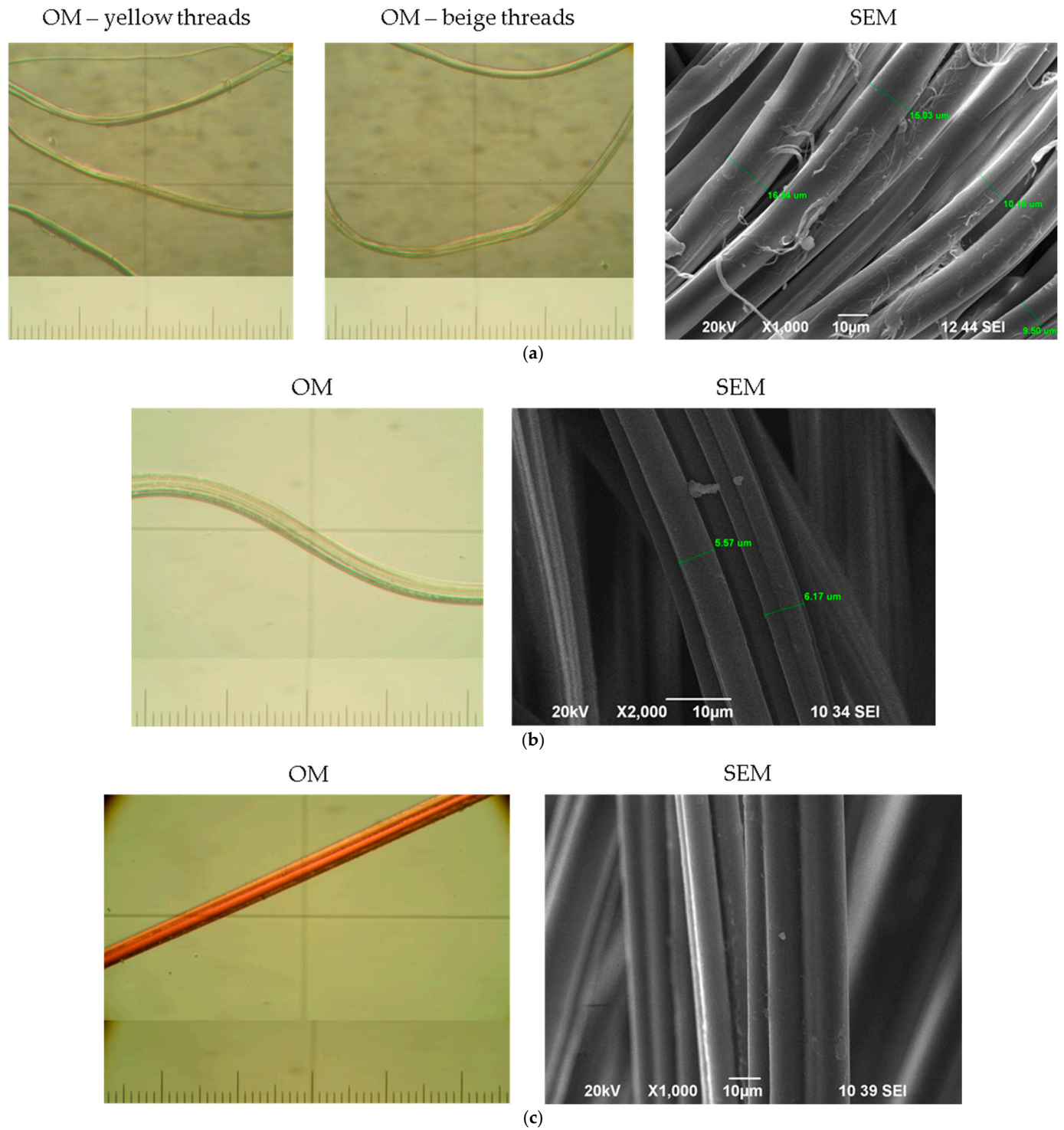


Figure 3. Cont.

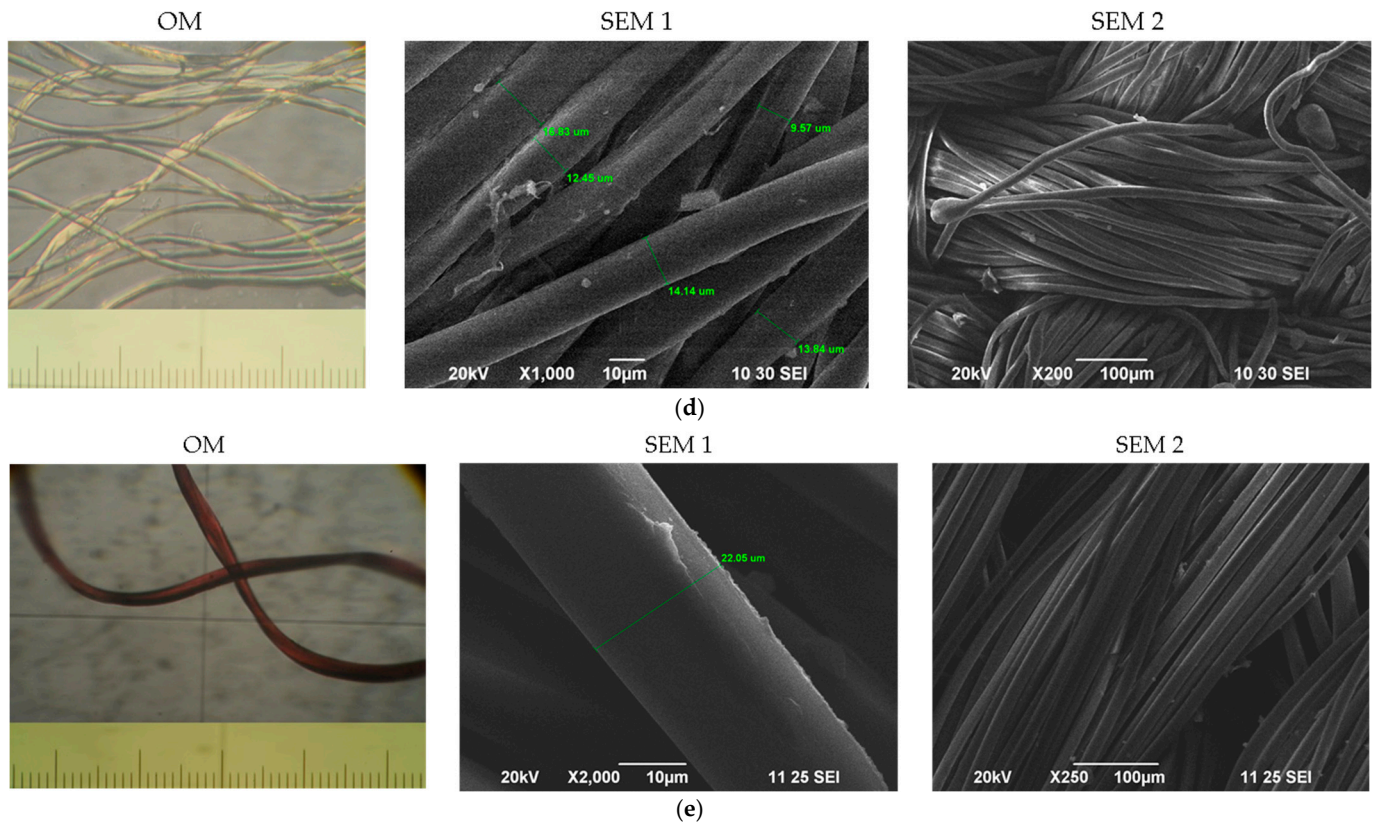


Figure 3. OM and SEM images of the studied industrial fabrics: (a) industrial fabric 1, (b) industrial fabric 2, (c) industrial threads 3, (d) industrial fabric 4, and (e) industrial fabric 5. One division on the scale bar of the OM images corresponds to 10 μm .

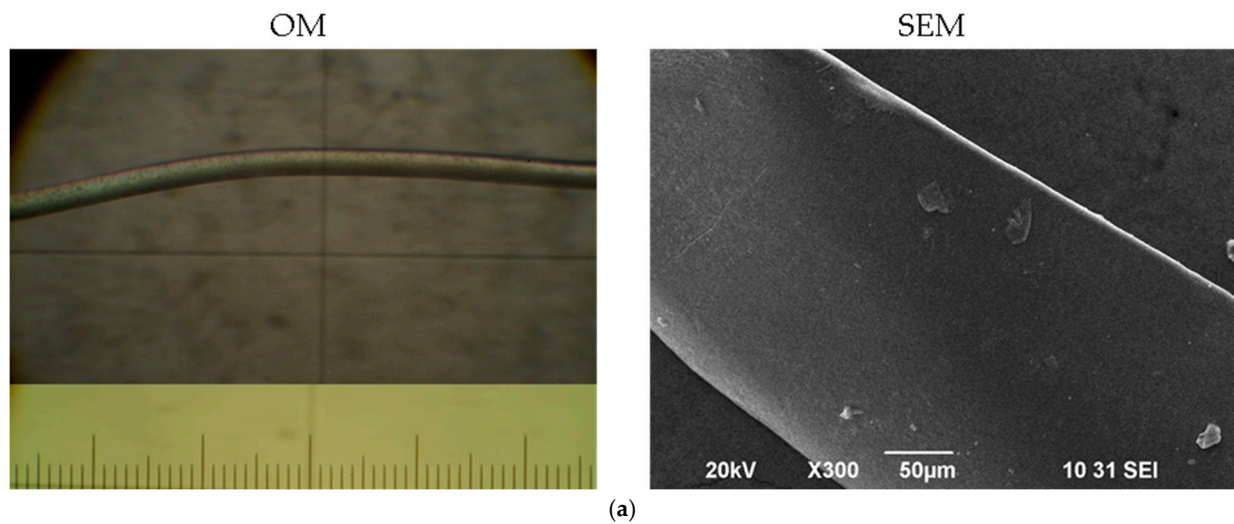
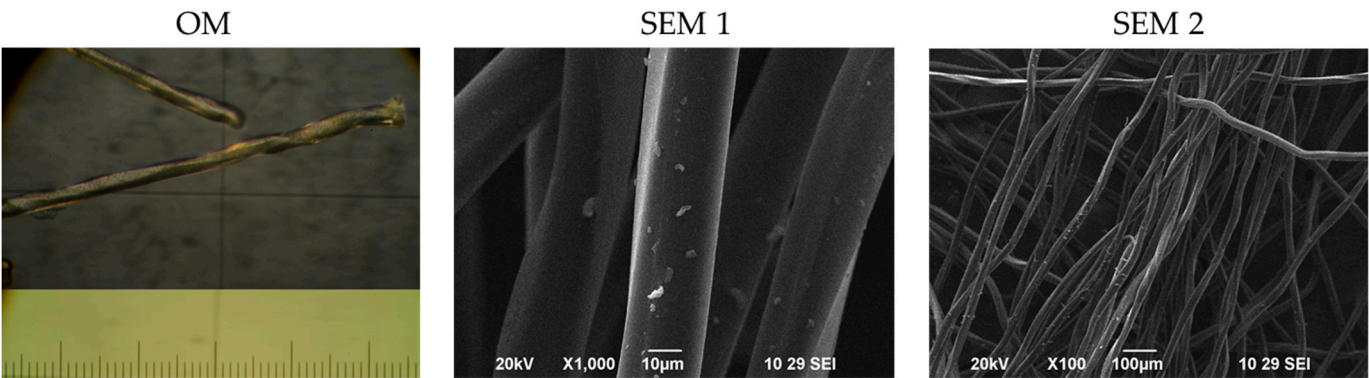
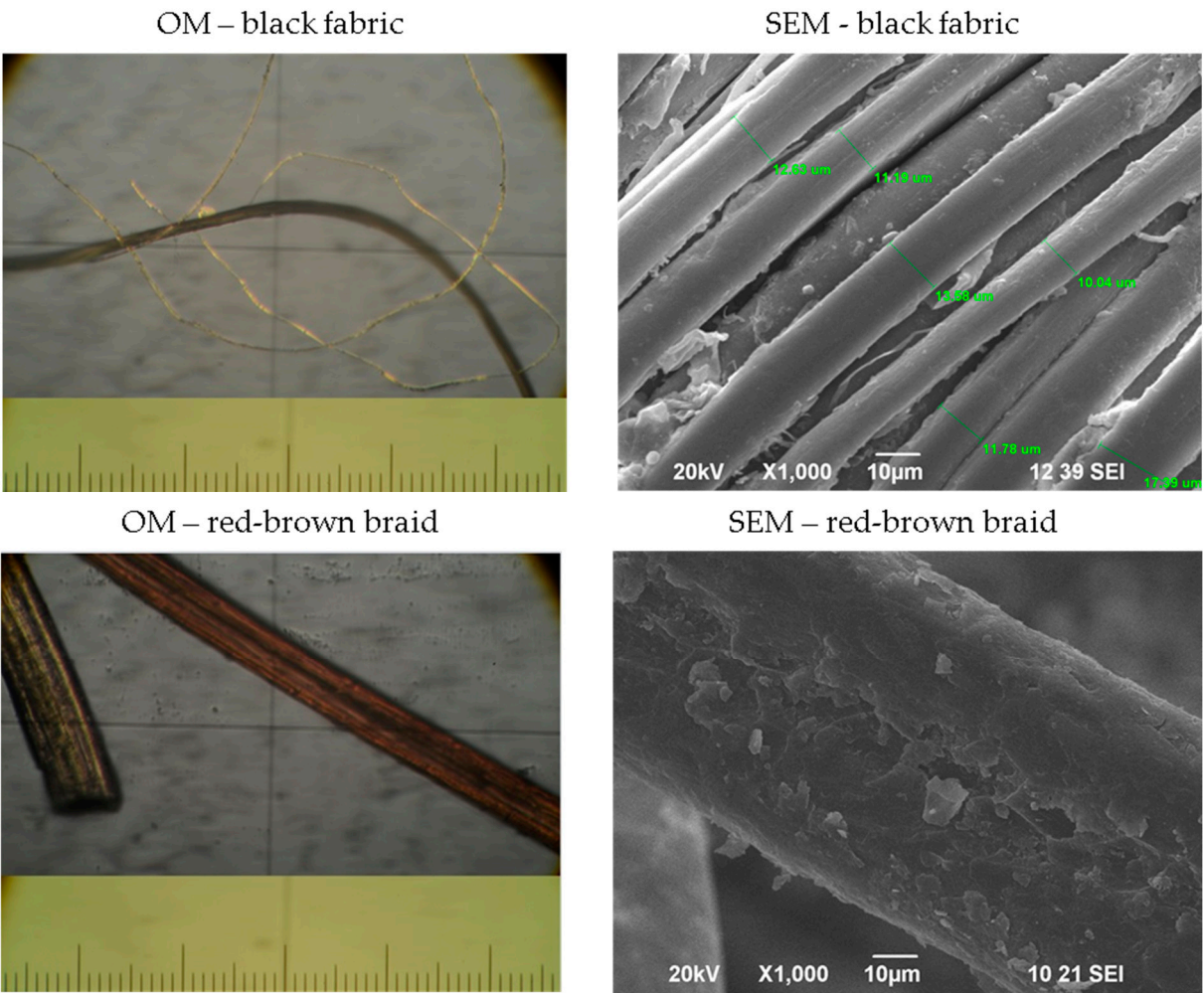


Figure 4. Cont.



(b)



(c)

Figure 4. Cont.

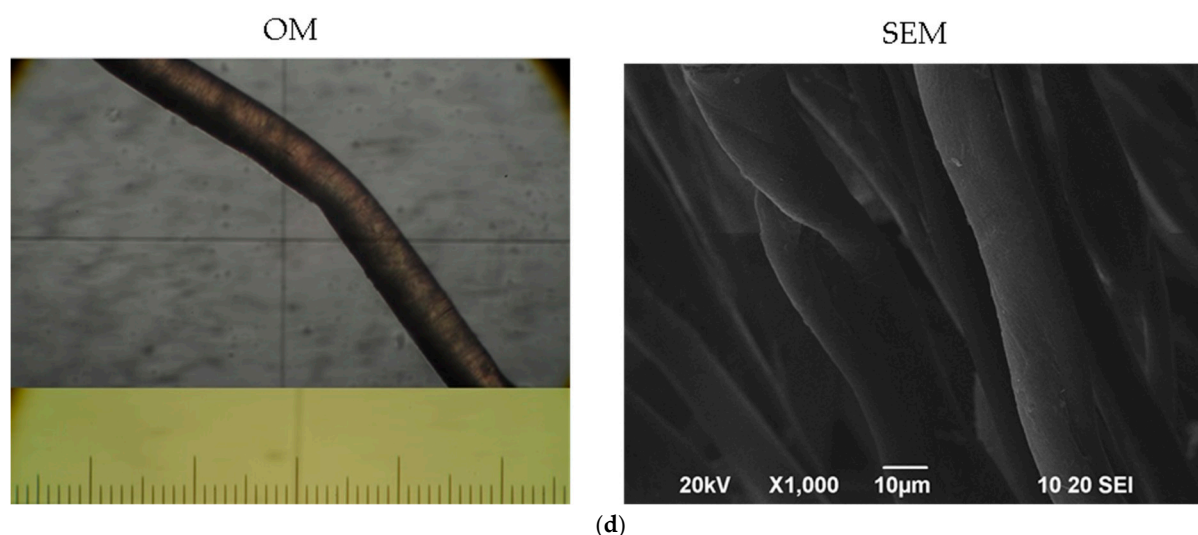


Figure 4. OM and SEM images of the studied historical textiles: (a) white shirt from Gora, (b) white skirt from Gora, (c) black waist-band with red braid from Bulgaria, region Sofia, and (d) pink headscarf from Bulgaria. One division on the scale bar of the OM images corresponds to 10 μm .

The OM observation yielded evidence that some of the textile samples contained fibers of rather uniform appearance and morphology—industrial fabrics 2, 3, and 5 (Figure 3)—and the textile in the shirt, the waist-band, and the headscarf (Figure 4), while the others were composed of two or more types of fibers. SEM analysis supported these morphological observations and provided information on the thickness of the fibers.

4.2. Solubility Testing

The fibers from the studied textile samples were treated with several of the solvents listed in Table S2—5% NaOH, 70% H_2SO_4 , m-cresol, and formic acid. Yet the most distinctive results were obtained when the samples were exposed to the action of 70% H_2SO_4 . The results of the test are summarized in Table 1. It was observed that when treated with 70% H_2SO_4 , the natural fibers (cotton, hemp, flax, wool, and silk), regenerated fibers (viscose, cellulose acetate), and some of the synthetic fibers (polyacrylic and polyamide) quickly dissolved. The other types of synthetic fibers—polyester and polyethylene—did not react to the acid.

Table 1. Solubility test with 70% H_2SO_4 and micro-chemical tests of industrial and historical textile samples.

Sample	Solubility in 70% H_2SO_4	Micro-Chemical Tests	
		Ninhydrin	Herzberg
industrial fabric 1 (yellow threads)	Dissolves	Positive	Negative
industrial fabric 1 (beige threads)	Does not dissolve	Negative	Positive
industrial fabric 2	Dissolves	-	Positive
industrial threads 3	Dissolves	-	Not detected ^a
industrial fabric 4	Dissolves partially	Negative	Positive
industrial fabric 5	Does not dissolve	Negative	-
white shirt from Gora	Dissolves	Positive	-
white skirt from Gora	Do not dissolve	Negative	-
black waist-band with red braid from Bulgaria (black threads)	Dissolves	Positive	-
black waist-band with red braid from Bulgaria (red threads)	Dissolves	Negative	Positive
pink headscarf from Bulgaria	Dissolves	Positive	-

^a the color was not detected due to interference with the original color of the sample.

The fibers in three of the studied samples—industrial fabric 1 (beige threads), industrial fabric 5, and the skirt from Gora, did not dissolve in 70% H_2SO_4 , indicating the presence of synthetic polyester or polyethylene material. The fibers in industrial fabric 4 dissolved partially, suggesting that part of the fibers are of polyester or polyethylene origin.

4.3. Micro-Chemical Testing

After consideration of the solubility test results, all fibers were subjected to the ninhydrin test and/or Herzberg test for further identification (Table 1).

The ninhydrin test (deep blue coloration) was used as a selective tool for identification of fibers containing proteins, alpha-amino acids, or synthetic amide-based materials. Based on this test, the studied samples were divided into two groups—with a positive result, i.e., containing protein or synthetic material with amide groups (industrial fabric 1 (yellow fibers); the shirt from Gora; the black threads of the waist-band from Bulgaria; and the pink headscarf from Bulgaria), and with a negative result, i.e., not containing protein or amide-based material (all other studied fabrics and historical textile objects).

Positive Herzberg staining (wine-red coloration), i.e., cellulose content, was observed in industrial fabric 1 (beige threads), industrial fabric 4, and the red threads of the waist-band from Bulgaria.

4.4. IR Measurements

The ER-FTIR measurement provided good differentiation of the polyester and cellulose acetate fibers from the other types of fibers. However, it was much more difficult to differentiate between natural protein and synthetic polyamide materials, as well as between natural and regenerated cellulose fibers.

The ATR-FTIR measurements enabled very accurate differentiation of the textile material, including those materials with similar spectra and/or a mixture of materials. Notably, although characterized by absorption bands at close wavenumbers, silk, wool, and polyamide were differentiated successfully due to the particular band positions and different band shapes observed in the ATR-FTIR spectra. The ATR-FTIR spectra of regenerated cellulose fibers were distinct from those of natural cellulose fibers.

The ATR-FTIR and ER-FTIR spectra of the studied historical textile are presented in Figure 5. The measured IR spectra of the industrial fabrics are provided in Figures S1–S12, compared with relevant reference materials in the Supplementary Materials. The IR frequencies observed in the ATR-FTIR spectra of all the studied fabrics and historical textiles are in very good agreement with earlier published data [32,40]. The most characteristic ATR and ER bands are described together with their assignment to vibrational modes in Tables S4 and S5 in the Supplementary Materials. In Figures S13–S20, we provide a comparison of the IR spectra of the studied historical textiles with reference materials.

As shown in Figure 5, the measured ER-FTIR spectra were more complex than the ATR spectra, making direct comparison difficult. It is known that reflected IR signals can appear distorted—for example, they can appear as inverted peaks accompanied by pseudopeaks (artificial peaks at the edges of the band) or have a derivative-like shape [41–46]. Additionally, the intensity ratios of the bands may differ considerably from those of the ATR spectra due to the influence of the physical and optical properties of the sample surface [41–46]. Although pseudopeaks can be used for identification based on spectral comparison, it should be kept in mind that their appearance is not always constant since it is influenced by the sample's reflectivity and may change as a result of surface treatments or aging. A recent study reported in detail the characteristic ER-FTIR bands that might be used for the identification of natural and synthetic textile fibers in the extended spectral region from 7500 to 375 cm^{-1} as well as for their assignment to vibrational modes [41]. The ER-FTIR

spectra measured by us show good correspondence with the reported spectral features, although some of the bands are not so well resolved.

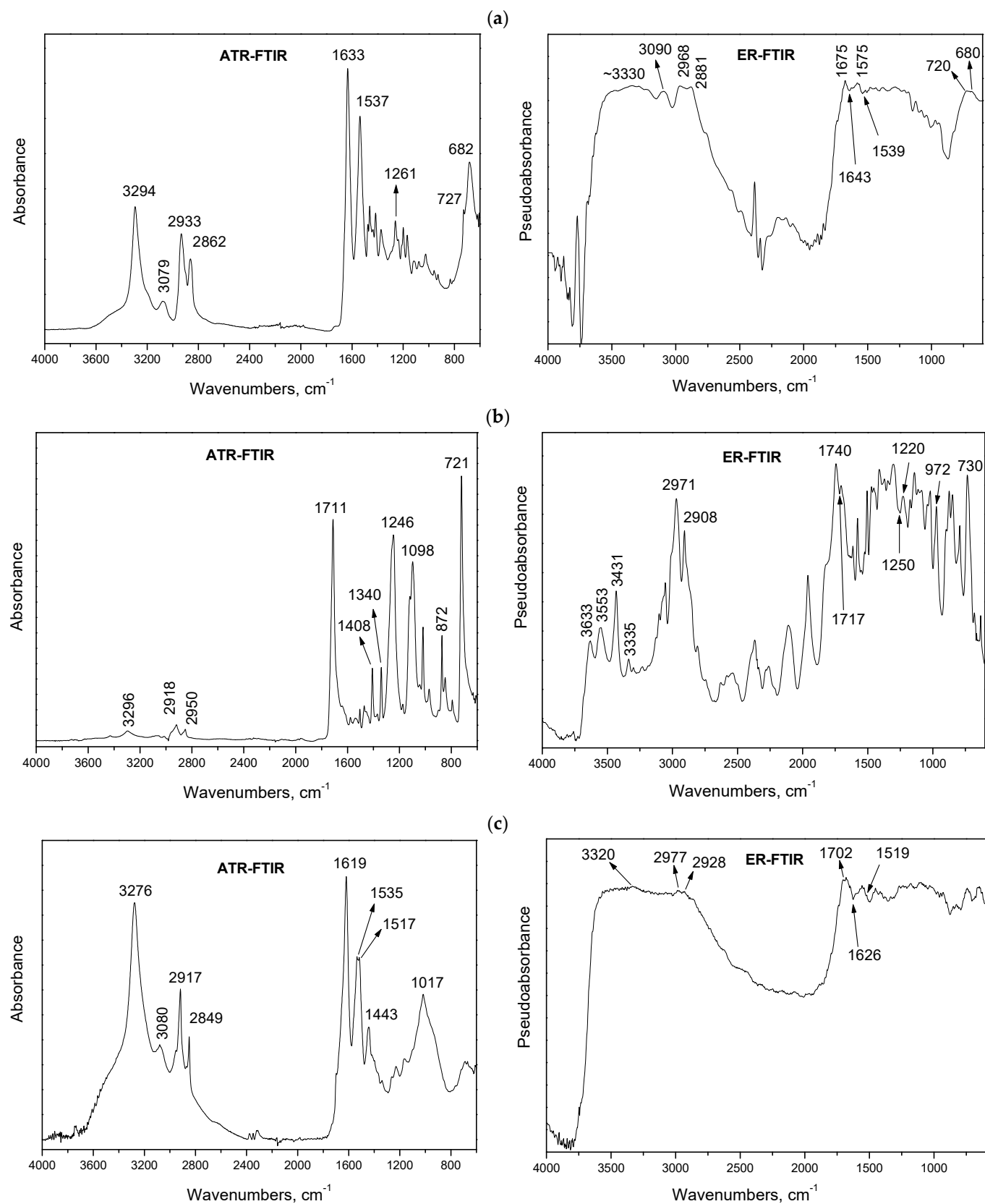


Figure 5. Cont.

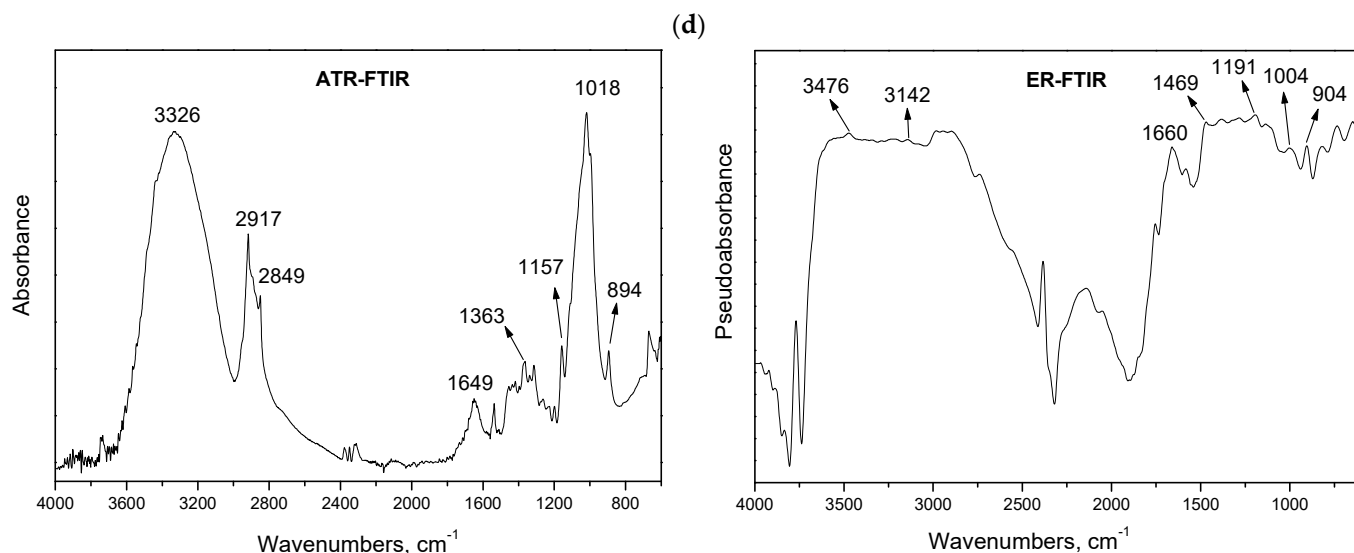


Figure 5. The selected IR spectra of the studied historical textiles: (a) white shirt from Gora, (b) white skirt from Gora, (c) waist-band from Bulgaria, region Sofia—black fabric, and (d) waist-band from Bulgaria, region Sofia—red braid.

5. Discussion

Microscopic examination of industrial fabric 1 revealed that threads of two colors were present—yellow and beige. OM examination of the yellow threads showed that they were composed of three differently appearing fibers—very thin and smooth ones (resembling silk), striated and smooth ones (resembling viscose), and smooth and rod-like ones (looking like synthetic fibers, Figure 3a). In the SEM analysis, thinner and smooth fibers of 9–10 μm were observed as well as thicker ones—with a thickness of 15–16 μm (Figure 3a). The solubility test showed that the fibers in the yellow threads dissolve in 70% H_2SO_4 , therefore suggesting that they do not contain polyester or polyethylene material. The micro-chemical testing demonstrated a positive ninhydrin reaction for protein or amide-based materials. The ER-FTIR study of the yellow commercial fabric indicated best similarity to the spectrum of reference silk based on the appearance of a pseudopeak at 1703 cm^{-1} and inverted peaks at 1628 and 1507 cm^{-1} (Figure S1). Furthermore, the ATR-FTIR spectrum of the fabric showed very good correspondence to the natural silk—with characteristic peaks at 3279 , 1618 , 1514 , 1441 , and 1230 cm^{-1} (Figure S2); therefore, it was concluded that the main composition of the yellow commercial fabric is silk (Table 2).

Some of the beige fibers showed similar rod-like morphology resembling that of the synthetic fibers (Figure 3a). Among these fibers, there were some with a different appearance—striated and smooth ones that according to the information in Table S3 resemble viscose or cellulose acetate. Some of the beige fibers gave positive results on the Herzberg test, indicating that they may contain cellulose. Yet other beige fibers gave negative results to the ninhydrin reaction, indicating that they do not contain any protein or amide-based material. Insolubility of the fibers in 70% sulfuric acid was observed, most probably because acid-insoluble threads prevailed. In accordance with that, the ATR-FTIR spectrum of the beige threads (Figure S3) revealed IR bands mainly of polyester, with most characteristic bands at 1711 , 1246 , 1097 , and 722 cm^{-1} . IR bands for silk and viscose were also observed (Figure S3). Hence, it could be concluded that the beige threads are composed mainly of polyester fibers with a smaller content of silk and viscose fibers.

Table 2. Identified materials in industrial fabrics and historical textiles.

Samples	Identified Material	Methods Used					
		OM	SEM	Sol	MCh	ATR-IR	Refl-IR
Industrial textiles							
industrial fabric 1 (yellow threads)	Silk	Silk Regenerated Synthetic	Synthetic	No polyester or polyethylene fibers	Protein and/or polyamide	Silk	Silk
industrial fabric 1 (beige threads)	Polyester Viscose Silk	Regenerated Synthetic	Synthetic	Polyester or polyethylene fibers	Cellulose	Polyester Viscose Silk	Polyester Viscose Silk
industrial fabric 2	Viscose	Regenerated	Synthetic or regenerated	No polyester or polyethylene fibers	Cellulose	Viscose	Viscose
industrial threads 3	Cellulose acetate	Regenerated	Synthetic or regenerated	No polyester or polyethylene fibers	Cellulose	Cellulose acetate	Cellulose acetate
industrial fabric 4	Polyester Cotton	Synthetic Cotton	Synthetic Cotton	Polyester or polyethylene, other natural, semisynthetic or synthetic fibers	Cellulose	Polyester Cotton	Polyester Cotton
industrial fabric 5	Polyester	Synthetic	Synthetic	Polyester or polyethylene fibers	No polyamide	Polyester	Polyester
Historic textiles							
shirt from Gora	Polyamide	Synthetic	Synthetic	No polyester or polyethylene fibers	Polyamide	Polyamide	Polyamide
skirt from Gora	Polyester	Synthetic	Synthetic	Polyester or polyethylene fibers	No polyamide	Polyester	Polyester
waist-band from Bulgaria (black threads)	Silk Viscose	Silk Synthetic	Silk	No polyester or polyethylene fibers	Protein and/or polyamide	Silk Viscose	Silk Viscose
waist-band from Bulgaria (brown-red threads)	Viscose	Regenerated	Synthetic	No polyester or polyethylene fibers	Cellulose	Viscose	Viscose
pink headscarf from Bulgaria	Polyamide	Synthetic	Synthetic	No polyester or polyethylene fibers	Polyamide	Polyamide	-

Microscope examination of industrial fabric 2 and industrial threads 3 revealed striated and smooth fibers. According to the SEM analysis, the thickness of the fibers in industrial fabric 2 is between 5 and 7 μm (Figure 3b). Fibers from industrial fabric 2 and threads 3 were subjected to Herzberg testing. The fibers in industrial fabric 2 gave a positive result to the Herzberg test for cellulose composition. The characteristic wine-red coloration could not be detected for the fibers in threads 3, which might be due to their original red color. Because of the distinct morphology of the fibers in both cases, which is not typical for protein and amide-based threads (like wool, silk, and polyamide), the ninhydrin test was not conducted.

The ATR-FTIR spectra of industrial fabric 2 showed a broad IR band at 3326 cm^{-1} and an intense multiplet band centered at 1018 cm^{-1} , which are both typical of viscose (Figure S4). In the ER-FTIR spectrum, the absorption between 3600 and 3100 cm^{-1} did not reveal a well-resolved maximum, but showed peaks at 1650 , 1158 , and 1021 cm^{-1} (Figure S5). In accordance with previous observations [41], the latter two peaks appeared inverted.

For industrial threads 3, the ATR spectrum revealed strong IR bands at 1735 , 1649 , 1364 , 1216 , and 1031 cm^{-1} , which indicates cellulose acetate composition (Figure S6). The ER-FTIR bands showed well-resolved shapes and very good matching to the positions of the observed ATR bands (Table S4), although most of them were inverted: 1739 (inv), 1433 , 1365 (inv), 1221 (inv), 1032 (inv), and 902 cm^{-1} . As a result, several pseudopeaks were also observed at $17,767$, 1707 , 1409 , and 1348 cm^{-1} (Figure S7).

OM examination of the fibers from industrial fabric 4 revealed two types of fibers of different morphology—the first ones were smooth and rod-like and the others were

flat and twisted (Figure 3d). SEM analysis showed that the thickness of the textile fibers varies between 9 and 17 μm . When comparing the appearance with the information in Table S3, it could be suggested that smooth and rod-like fibers are synthetic fibers and the flat and twisted ones are cotton. The sample reacted positively on the Herzberg test, indicating cellulose contents as expected for cotton. The ninhydrin test gave a negative result, showing that the sample did not contain amide-based material; hence, polyamide was ruled out as a possible material in the synthetic fibers in the sample. Partial solubility of the sample in 70% sulfuric acid was observed in accordance with the fact that besides cotton, which is soluble in 70% sulfuric acid, the sample contained acid-insoluble fibers such as polyester or polyethylene. Most of the IR peaks in the ATR-FTIR spectrum of gray industrial fabric 4 matched those of the reference polyester—with strong characteristic peaks at 1713, 1244, 1098, 1017, and 721 cm^{-1} —while others, 3335, 3298, 2908, 2851, 1097, and 670 cm^{-1} , were attributed to cotton (Figure S8). The ER-FTIR spectrum showed stronger resemblance to the polyester spectrum (Figure S9). Most of the ER-FTIR bands were observed at approximately the same positions. The band at 1717 cm^{-1} appeared inverted and accompanied by pseudopeaks at 1740 and 1705 cm^{-1} (Table S4). The peak at 1250 cm^{-1} was also found to be an inverted peak in accordance with reported data [41]. The presence of cotton in the fabric was evidenced mainly by the presence of broad absorption in the region 3600–3100 cm^{-1} .

OM and SEM examinations of industrial fabric 5 displayed smooth and rod-like fibers, suggesting synthetic origin (Figure 3e). SEM analysis demonstrated very uniform appearance of the textile fibers, which were considerably thicker—above 20 μm (Figure 3). Since the morphology of the fibers was uniform and different from that of regenerated cellulose fibers, the sample was subjected only to the ninhydrin test in order to check whether it contained amide-based material. A negative result was obtained, indicating that the fibers are not made of polyamide. This result was complemented by the solubility testing of the sample—it did not dissolve in 70% sulfuric acid; therefore, it was concluded that industrial fabric 5 is composed either of polyethylene or polyester. Indeed, the ATR- and ER-FTIR spectroscopic measurements of the whole fabric showed excellent correspondence with the spectrum of polyester (Figures S10 and S11). ATR-FTIR characteristic peaks were observed at 2967, 2907, 2851, 1713, 1504, 1470, 1409, 1338, 1240, 1095, 1016, 969, and 722 cm^{-1} . The ER-FTIR spectrum of industrial fabric 5 matched almost completely not only the reference polyester spectrum but also that of industrial fabric 4 in the region below 2000 cm^{-1} (Figure S11).

When observed under OM, the fibers from the white shirt from Gora revealed a smooth and rod-like shape, making them appear synthetic (Figure 4a). The fibers were very thick (above 100 μm), as evidenced by the SEM analysis. The fibers in the sample dissolved in 70% sulfuric acid and gave a positive result on the ninhydrin test, implying that the sample is a synthetic amide-based material. The characteristic peaks at 3285, 3066, 2932, 2860, 1634, 1535, 1460, 1412, 1261, and 679 cm^{-1} observed in the ATR-FTIR spectrum of the shirt fabric (Figures 5a and S12), as well as the excellent match of the ER-FTIR spectrum to that of the reference polyamide (Figures 5a and S13), support the conclusion that the composition is polyamide. The characteristic peaks in the region 3600–2800 cm^{-1} appeared at slightly higher frequencies in the ER-FTIR spectrum compared to the ATR bands (Table S5). On the other hand, the positions of the peaks for amide I and amide II closely matched those of the ATR bands—1643 and 1539 cm^{-1} . The two peaks appeared inverted and accompanied by pseudopeaks at 1675 and 1575 cm^{-1} . Notably, the amide I and amide II bands in the ER-FTIR spectrum of the white shirt are shifted to a higher frequency by more than 20 cm^{-1} compared to the ER-FTIR spectrum of silk. This makes differentiating between polyamide and silk reliable, despite their ER-FTIR spectra appearing similar overall.

OM examination of the fibers in the white skirt from Gora also revealed smooth, regular, and rod-like structures that resemble synthetic fibers (Figure 4b). SEM analysis evidenced that textile fibers had a uniform morphology. The ninhydrin test was negative and the insolubility of the fibers in 70% sulfuric acid pointed to synthetic polyester or polyethylene fibers. The ATR- and ER-FTIR spectra confirmed the polyester composition of the textile (Figures 5b, S14 and S15). No significant differences were found when compared with the reference spectra and industrial fabric 5, which was also of pure polyester.

The waist-band from Bulgaria, in the region of Sofia, is made of black fabric with a brown-red braid. OM examination of the fibers in the black fabric showed two types of fibers that differed in their morphology. The first ones were smooth and thin like natural silk, and the others were smooth, regular, and rod-like, resembling synthetic fibers (Figure 4c). Microscopic examination of the fibers in the brown-red braid showed the striated and smooth morphology characteristic of viscose (Figure 4c). Since the fibers in the black fabric dissolved completely in 70% sulfuric acid and the microscopic observation showed fibers with a morphology typical of silk, the sample was subjected to the ninhydrin test. It reacted positively, which indicated that the thin and smooth fibers were presumably silk. As the other type of fibers observed with OM and SEM had a morphology close to that of synthetic fibers, and the fibers showed acid solubility, it could be suggested that the composition of the second type of fibers is viscose. The ATR-FTIR spectrum revealed a predominant composition of silk and a smaller proportion of viscose. Most of the IR bands in the ATR-FTIR spectrum corresponded to silk—3276, 3080, 1697, 1618, 1517, 1443, and 1230 cm^{-1} , while the maximum at 1017 cm^{-1} was attributed to viscose (Figures 5c and S16). Similarly to the reference silk and industrial fabric 1, the ER-FTIR spectrum of the black fabric of the waist-band showed inverted peaks at 1626 and 1519 cm^{-1} , as well as a pseudopeak at 1702 cm^{-1} (Figures 5c and S17). Despite being rather weak, the rest of the ER-FTIR peaks also matched the reported characteristic positions of silk well [41]. In contrast to the ATR-FTIR spectrum, the spectral features for viscose were not well expressed.

The fibers from the red braid also dissolved in 70% sulfuric acid, but their OM and SEM study displayed morphology characteristics of viscose. Therefore, a Herzberg test was carried out as a next step and gave a positive result, suggesting a cellulose-based material. The ATR- and ER-FTIR spectroscopic measurements directly on the braid (Figures 5d, S18 and S19) completely matched the IR spectra of the reference viscose and showed very similar frequencies to the spectrum of industrial fabric 2. The differentiation of the ATR-FTIR spectra of the red braid and industrial fabric 2 from those of natural cellulosic fibers is very straight forth due to the rounded shape of the absorption at 3327 cm^{-1} and the multiplet peak centered at 1018 cm^{-1} , which in cotton and flax spectra appear with maxima at 1056 and 1030 cm^{-1} [40]. Differentiation only by the ER-FTIR spectra seems much more complicated.

OM examination of the pink headscarf from Bulgaria revealed only smooth and rod-like fibers—a morphology characteristic of synthetic fibers (Figure 4d). The fibers in the sample were soluble in 70% sulfuric acid and gave a positive result on the ninhydrin test, suggesting a polyamide-based material. The ATR-FTIR spectrum of the pink headscarf fabric manifested peaks at 3294, 3068, 2932, 2861, 1634, 1537, 1462, 1416, 1261, and 1119 cm^{-1} , confirming the polyamide composition (Figure S20).

The identified textile fibers are summarized in Table 2 along with the analytical techniques supporting their presence.

Based on the above results, it can be concluded that OM enabled quick preliminary classification by distinguishing natural textile fibers (cotton and silk in the present studies) from regenerated cellulose fibers (viscose and cellulose acetate) and synthetic fibers (polyester and polyamide). The solubility test enabled the identification of polyester, whereas the

micro-chemical reactions helped to separate the fibers into protein/amide-based and cellulosic fibers. SEM-EDS analysis provided examination at a greater magnification and allowed for the measurement of the fibers' thickness. When necessary, this method can also provide information on the elemental composition of textiles. In view of the required analytical equipment, OM microscopy combined with the micro-chemical tests can be considered a relatively accessible method for identifying most natural fibers, as well as quickly differentiating regenerated and synthetic fibers. This method can be regarded as micro-invasive because only a few millimeter-long textile threads are needed for both the OM examination and the micro-chemical analysis. However, for more precise identification, the ATR-FTIR spectroscopy method is mandatory—it is particularly effective in distinguishing all types of fibers in a non-destructive way without sampling. ER-FTIR measurements offered completely non-invasive analysis and successfully identified the fibers but required much more tedious examination of the spectral information.

The analysis of gray industrial fabric 4 and purple industrial fabric 5 was directed at helping practice and thus had special added value. It had to be ascertained that these fabrics were made of material which would be compatible with the cloth of the original ethnographic object and thus usable in the envisaged restoration of a dzhube (a kind of long sleeveless overcoat) from North Macedonia (Figure 6).



Figure 6. Picture of (a) dzhube from North Macedonia, (b) detail of dzhube along with studied gray and purple industrial fabrics 4 and 5 intended for its restoration.

The composition of the gray fabric had been stated by the seller as “pure cotton”, but after being examined organoleptically and subjected to the burning test, it demonstrated the presence of impurities. Therefore, it was important to determine what kind they were and which material predominated. The subsequent analysis showed that besides cotton, there were small quantities of polyester. As for the purple fabric, the information from the seller was corroborated: it was indeed a polyester-based fabric. The gathered information indicated that both fabrics are compatible with the originals from the museum and shall be used for strengthening and lining.

Determining the exact material of the studied museum objects also helped to establish a proper method for maintenance. As it was found, the shirt and the scarf are made of polyamide, while the skirt is made of polyester; therefore, all three would be at risk if ironed at too high a temperature. Moreover, the information on composition will guide the

choice of appropriate methods, reagents, and detergents for removing spots and general chemical cleaning.

6. Conclusions

The analytical investigation successfully identified the types of textile fibers used in the studied Bulgarian folk costumes and contemporary industrial fabrics. A comprehensive range of analytical methods was applied, including optical and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), solubility testing, micro-chemical staining, and ATR-IR and ER-IR spectroscopy. The results clearly demonstrate the evolution of textile materials across different historical periods. Unlike traditional folk costumes, which were made almost exclusively from natural fibers such as cotton, wool, and silk, the costumes from the early to mid-20th century were distinguished by the use of synthetic and regenerated fibers, such as polyester, polyamide, viscose, and cellulose acetate, as well as mixtures of natural and synthetic materials.

The applied analytical techniques provided reliable identification of both natural and synthetic fiber types. Optical microscopic analysis offered a fast preliminary classification, micro-chemical tests confirmed the presence of specific fiber types through characteristic reactions, while IR spectroscopy proved particularly effective for distinguishing between fibers with similar characteristics. An additional advantage of the IR analysis was the possibility of non-destructive and non-invasive analysis. SEM-EDS microscopy allowed for examination at the micrometer scale and for measurement of physical characteristics such as fiber thickness.

This study offers valuable information about the textile composition of traditional Bulgarian clothing and contributes to documenting the materials used over time.

The findings are highly relevant for the conservation and restoration of ethnographic artifacts, enabling restorers to select appropriate materials that are compatible with the original composition of the textiles, as well as proper methods of maintenance and cleaning.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/app15179367/s1>: Table S1. Feeling characteristics of textile fibers; Table S2. Burning characteristics and solubility test of reference textile fibers; Table S3. Microscopic appearance of textile fibers; Figures S1–S20. Comparison of IR spectra of studied industrial fabrics and historical textiles with reference materials; Table S4. ATR- and ER-FTIR data for the studied industrial fabrics; Table S5. ATR- and ER-FTIR data for the studied historical textiles.

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Abbreviations

The following abbreviations are used in this manuscript:

OM	Optical microscopy
SEM	Scanning electron microscopy
EDS	Energy-dispersive X-ray spectroscopy
FTIR	Fourier transform infrared
ATR	Attenuated total reflectance
ER	External reflectance
IR	Infrared

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