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Mechanical and Morphological Properties of Cellulosic Fabrics Treated with Microencapsulated Essential Oils

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Abstract: This study focused on the mechanical and morphological properties of lignocellulosic heritage textiles (cotton and hemp) made using eco-friendly salvia and thyme microencapsulated essential oils, characterized by low toxicity for humans and the environment. A morphological evaluation of the tested fabrics was performed using scanning electron microscopy. The ATR–FTIR spectra of the untreated and treated fabric samples were tested using Perkin Elmer, Spectrum 3. A tensile test of the samples was conducted based on the ISO 13934-1 using a MESDAN-Lab tensile strength tester. According to the analysis, the salvia improved the tensile strength of the fabric by 20% and 39% in the warp and weft directions, respectively. The data for the untreated cotton, untreated hemp, and fabrics treated with salvia and thyme were processed using the kernel PCA method. From the principal component analysis it was found that the textile fabrics treated with salvia coalesced close to the untreated ones. This shows that treatment with essential oils from the indicated plant does not significantly affect the tensile properties of textile fabrics. The thymol-treated textile fabrics were significantly distant from those of the control sample. In cotton textile fabrics, thymol treatment was found to have a significant effect on their tensile properties. In hemp fabrics, two types of thymol and salvia treatments were found to have a very significant effect on the tensile strength performance of the textile fabrics. The results obtained can be used in museums, universities, and ethnographic studies that own or maintain collections of heritage textiles.

Keywords: heritage textiles; microencapsulation; essential oils; salvia; thyme; morphological evaluation; tensile strength; cellulosic fibers; SEM; ATR–FTIR

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1. Introduction

Ethnographic patrimony textiles are very fragile; they have complex and often very inhomogeneous compositions, containing organic compositional material (protein or cellulosic) as well as inorganic materials (sequins, beads, metal wires, etc.). The techniques used to produce them are very different and in accordance with the historical time in which they were made, manipulated, and transported. The general state of the collection, the sensitivity inherent to the organic objects (endogenous factors), internal microclimate conditions (temperature, humidity, CO_2, brightness, biotic factors, and microorganisms), along with the external environment and anthropic factors (e.g., natural disasters, specialized human interventions such as conservation and restoration) have a significant influence on the processes of natural aging and degradation of materials from collections of ethnographic textiles. The mechanical and morphological properties of ethnographic textiles are important [1–6]. Textiles and fabrics from the past can generally be categorized into three main groups, i.e., plant-based textiles (cotton, linen, and jute), animal-based textiles (wool), and silk [7]. All three groups are vulnerable to biodeterioration and biodegradation caused by various bio-organisms and micro-organisms (fungi, molds, bacteria, and insects). It is necessary that some precautionary disinfection measures are taken for their conservation because microorganisms can irreversibly modify/destroy the weavings over time and can also have negative effects on the health of the people who are in charge of conservation and who regularly manipulate the heritage textiles, as well as visitors [8–11]. At the same time, it must be taken into account that the agents used in the conservation of cultural heritage objects should be efficient, not to cause damage to the treated materials and the environment and not be toxic to humans.

In this connection, essential oils have been known and used by humans for centuries. The term *essential oil* originates from the term *quinta essential*, used by von Hohenheim in the sixteenth century [12]. They were utilized at the time to improve health conditions and cure illnesses, and as spices as well. They are defined as concentrated hydrophobic and aromatic essences of plants that contain volatile compounds. The compounds are comprised of a mixture of different volatile terpenes as well as hydrocarbons. They are usually stored in oil glands and sacs in plants and can be evaporated at room temperature, causing different odors and smells of plants and herbs. Different extraction methods have been used throughout history to obtain essential oils from different plants, the most common of which are steam and hydro distillation [13–16]. Essential oils were proved to have antibacterial and antiviral properties [17]. With increasing international concern for both health and environmental issues, the use of disinfectant solutions formulated with essential oils (EOs) has become the focus of attention due to their bio-based and eco-friendly nature [18].

Different classification methods can be used to identify essential oils based on extraction techniques, chemical compositions, and even their aroma [19]. From a chemical point of view, the essential oils in most citrus and pine plants contain hydrocarbons containing only hydrogen and carbon atoms. Other plants such as tea, peppermint, and coriander contain hydroxyl groups (OH) that may be attached to a terpene. Some other more complicated compounds are aldehydes, cyclic aldehydes, and ketones. Some plants have phenols and phenolic ether. There are also oxides in Eucalyptus trees and esters in lavender and clary sage. Clove, aniseed, and myrtle leaf contain phenylpropanes which have carminative or anesthetic properties. Sesquiterpene lactones stimulate the immune system and act as a mucolytic [20–22]. There are different extraction methods for obtaining essential oils from different plants, each having its advantages and disadvantages. The steam-distillation of essential oils can be considered one of the oldest methods and probably the most common. This method has the advantage that it can prepare pure oil [23]. In this method, plants are put in a container that is connected to a steam-producing source. The oil sacs and glands in the plants are opened by the heat and the essential oils are released. The oil is then condensed into liquid form which in turn is separated into two parts, i.e., essential oils and aromatic water. Another method is cold pressing (or the expressed oil extraction method) which is usually used for citrus plants. Oils are forced out of the plant by means of applying mechanical pressure. The mixture of essential oils and water is then distilled to separate them. One drawback is that the cold-extracted oil may spoil in a short time. Therefore, only small quantities are processed each time [24,25]. In another extraction method called the solvent extracting method, essential oils that are sensitive to either heat or pressure are extracted. For instance, this method is used for jasmine, orange blossom, rose, tuberose, and even oak. For each essential oil, or each plant, a particular solvent is used, such as alcohol, ether, hexane, ethanol, methanol, or even petroleum [26,27]. The combination of their antibacterial properties and their natural origin make them quite a favorable option for novel compounds that have to comply with new health and environmental issues. This has even made the way for the use of essential oils as mild preservatives in the food, pharmaceutical, and cosmetic industries [28]. Nevertheless, the possible applications of essential oils are not limited to the medicinal and food industries. They have been introduced in recent decades as promising preservatives in different industries, such as the timber and textile industries [19].

The utilization of essential oils as disinfecting agents to preserve and maintain textiles and fabrics made from natural fibers has been suggested by some researchers because of their antibacterial and antiviral properties [29–36]. In this connection, conservators traditionally used thymol vapor as a homemade disinfectant [37]. Ethylene oxide is another traditional disinfectant that has a particular advantage, i.e., it has no or very little negative effect on cellulosic materials [38]. Therefore, conservators have used it to disinfect a variety of cellulose-based materials such as textiles and clothes, and even books. Thyme-based disinfectants have also been demonstrated to have no undesirable effect on thermally aged natural fibers (linen and papyrus) [39]. Thyme extract has not only significantly improved the resistance of some natural textiles against bacteria and mold, but it has also been demonstrated to cause no statistically significant loss of strength in cotton-blended fabric [16]. The essential oils of many salvia species have also been reported to have significant bactericidal properties [40–43]. Essential oil extracted from *Salvia officinalis* L. was even reported to effectively hinder the growth of two troublesome fungi in grains and crops after being harvested, namely *Verticillium dahliae* and *Penicillium aurantiogriseum* [43]. Essential oil extracted from *Salvia majdae* L. using the hydro-distillation (HD) method was reported to have the highest antibacterial effect compared with those of oils obtained by other extraction techniques, such as steam distillation, ultrasound, and microwave hydro-distillation [44]. EOs as disinfecting agents may greatly differ in their achieved results (comprising physical, chemical, and physic–chemical processes) [36,45] depending on a variety of factors, such as the method of application, the concentration of the agent, the degree of reactiveness on the textile, etc. [45,46]. One of the recently developed EO application methods is microencapsulation. Microencapsulation has been used in many industries and products, including insect repellents, dyes, vitamins, antimicrobials, phase-changing and medical materials, e.g., antibiotics, hormones, drugs, and specifically spores (antibacterial, anti-odor, and anti-muscle pain) [47].

Conventional fabrics can have new value-added properties that can elevate them to the status of intelligent textiles through the incorporation of the microencapsulated functional agents (assuring protection from reactions with light, oxygen, moisture, etc.) [48]. A study by Specos et al. [49] reports the development and testing of two types of microcapsules (based on Arabic gum or yeast). The treated fabrics showed good results concerning the durability of the fragrance and the morphology and laundering properties of the textiles and fabrics. The microencapsulation of different aromatic EOs has been used to improve anti-microbial and insect-repellent properties in medical textiles [50,51].

Though EOs have been proven to be effective, many other unknown aspects are yet to be investigated, including their effects on the mechanical properties, short- and long-term morphological properties, and chemical properties of the treated textiles. In this connection, thyme and salvia essential oils are characterized by a low toxicity for humans and the environment [7,18,39,52]. The antimicrobial efficiency and impact on fabric strength of thyme and salvia essential oils applied to cotton and hemp fabrics have been evaluated [18,39].

Therefore, the present study is focused on the effects of the essential oils of thyme (*Thymus vulgaris*) and salvia (*Salvia officinalis*) on the mechanical and morphological properties of heritage textiles. The purpose of the study was to find out whether or not microcapsules of these active ingredients (thyme and salvia essential oils) degrade cotton and hemp fabrics.

Therefore, the mechanical and morphological properties were tested after being exposed to the saturated vapors of the active compounds. The present study aims at a multidisciplinary analysis of the diagnosis, protection, and preservation of ethnographic textiles from personal collections, but the results obtained will also benefit museum institutions that own and curate heritage textile collections.

2. Materials and Methods

2.1. Materials

The studies were carried out on two traditional elements belonging to Romanian cultural heritage: a hemp towel and a traditional shirt (ie) (Figure 1). Both cultural heritage objects are approximately 100 years old and belong to the Maramureș region, an area with strong cultural and ethnographic peculiarities, located in the north-western part of Romania, at the border with Ukraine [53–56].

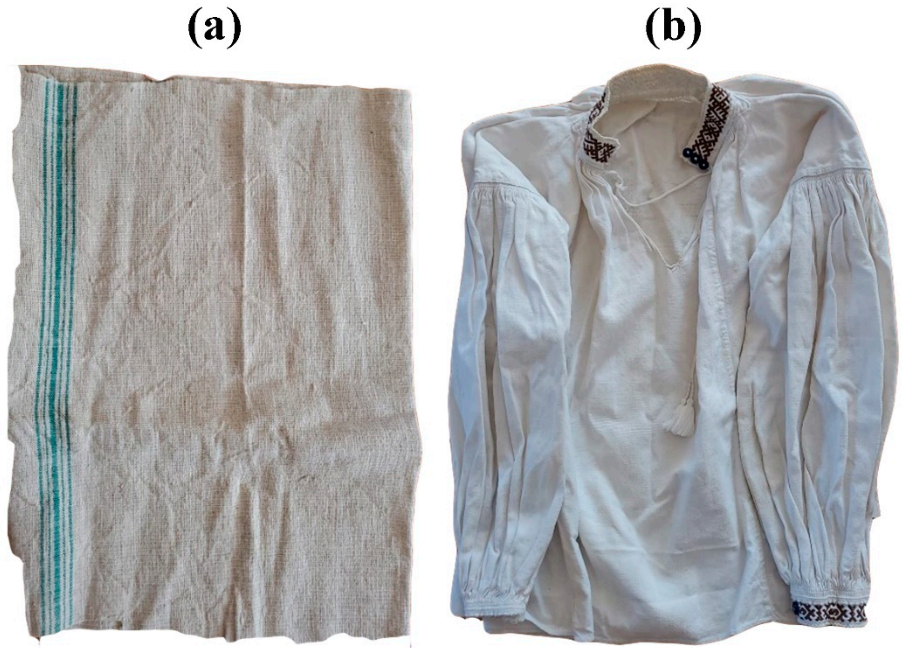

Figure 1. Studied heritage textiles. (**a**) Hemp towel; (**b**) traditional Romanian cotton shirt.

The characteristics of the fabrics were tested according to the standards outlined in Table 1.

In the study phase, correlations between the structural and physical properties of the fabrics were investigated (mass per unit area, thickness, and porosity).

The measurement of the porosity of cotton and hemp fabrics using a pycnometer was explored [57].

The thyme and salvia were purchased from Lozano Essences (Esencias Lozano, S.L., Murcia, Spain). The fabrics were then treated with two essential oils microcapsules which contained salvia and thyme, respectively, and were further tested using scanning electron microscopy (SEM), attenuated total reflectance–Fourier transform infrared spectroscopy (ATR–FTIR), and a tensile test.

Table 1. Characteristics of cotton and hemp fabrics.

Testing	Standard Method	Cotton Fabric	Hemp Fabric
Warp density (ends/cm)	ISO 7211-2:1984 Textiles—Woven fabrics—Construction—Methods of analysis—Part 2: Determination of number of threads per unit length	14	8
Weft density (picks/cm)		17	8
GSM, grams per square meter (g/m^2)	ISO 3801:1977 Textiles—Woven fabrics—Determination of mass per unit length and mass per unit area	338.87 ± 19.89	367.04 ± 44.62
Yarn number (warp)	ISO 1144:2016 Textiles—Universal system for designating linear density (Tex System)	99.77 ± 26.79	284.89 ± 69.93
Yarn number (weft)		69.17 ± 7.17	109.33 ± 18.50
Fabric thickness (mm)	ISO 5084 (fabric thickness)	0.79	1.34
Yarn twist (warp) (twist per meter (tpm))	ISO 2061:2015 Textiles—Determination of twist in yarns—Direct counting method	550	110
Yarn twist (weft) (twist per meter (tpm))		760	80
Porosity (%)	* Pycnometer-based test for porosity	48.80	71.86

* A modified test method.

The salvia and thyme essential oils were encapsulated in ethyl cellulose, ethoxy content 49%, from Sigma-Aldrich (Akralab, Alicante, Spain) using a solvent evaporation method. Ethyl cellulose was chosen as a suitable shell because this polymer is used for protecting the release of different active ingredients in some pills [58], and consequently we assumed that it could be used for textiles involved in food processes. The same procedure was therefore applied for each oil: 5% w/v was solved in a 1:9 acetone/methanol solution with magnetic stirring until the solution was homogenous and 10 mL of essential oil was later added to 25 mL of ethyl cellulose solution and was further stirred at 900 rpm for 5 min. This mixture was added to 100 mL of polyvinyl alcohol (PVA) 1%, continuously stirred at 900 rpm, and kept for 8 h to let the solvent evaporate. The microcapsules were applied to the cotton and hemp fabrics by spraying the surface with the microcapsule's dispersion (the solvent for the microcapsule's dispersion was 60 g/L water). Finally, the fabrics were left to dry at room temperature.

2.2. Scanning Electron Microscopy (SEM)

The morphological structures of the untreated and treated fabrics were characterized using scanning electron microscopy (SEM, Hitachi TM3030Plus, Hitachi, Ltd., Tokyo, Japan). All the samples were prepared by cutting 1 cm × 1 cm sections from the fabric, and these were observed under 2000 magnification with an accelerating voltage of 15 kV. All the samples were mounted on stubs and sputter treated with gold before the analysis to prevent any charging from the samples. The diameter of the microcapsules was measured using the ImageJ software. Twenty measurements were taken at random places from selected SEM images.

2.3. Attenuated Total Reflectance–Fourier Transform Infrared Spectroscopy (ATR–FTIR)

The ATR–FTIR spectra of the untreated and treated fabric samples were tested using a Perkin Elmer, Spectrum 3 (PerkinElmer Inc., Waltham, MA, USA). The spectral range was 550–4000 cm^{-1}, and an average of 16 scans was used to test the fabrics. An analysis of the data was conducted by observing the shift of the spectra of the untreated and treated fabric samples.

2.4. Tensile Test

The tensile test of the samples was conducted based on the ISO 13934-1, but with several modifications. Three samples of each fabric were cut with dimensions of 6 cm × 1.5 cm. Each sample was tested using a MESDAN-Lab (Mesdan S.p.A., Puegnago del Garda, Italy) tensile strength tester. The test was carried out using a 20 N load cell with an extension rate of 100 mm/min. The results were expressed as mean ± SD, n = 3.

3. Results

3.1. Morphological Structures of Untreated Fabrics and Microcapsule-Treated Fabrics

The morphological structures of the untreated cotton and hemp and the fabrics treated with salvia and thyme are presented in Figure 2a–f. In Figure 2a, the presence of twisted ribbon-like fibers in the fabric indicates the characteristics of cotton fibers. After the fabrics were treated with salvia and thyme through the spraying process, there were numerous fine microcapsules immobilized along the twisted cotton fibers, compared with the untreated cotton fabric. The diameters of the microcapsules were in the range of 0.21 µm to 0.74 µm. The use of ethyl cellulose in the essential oil solution bound the cotton fibers, making the fibers unnoticeable in the SEM micrographs (Figure 2b,c).

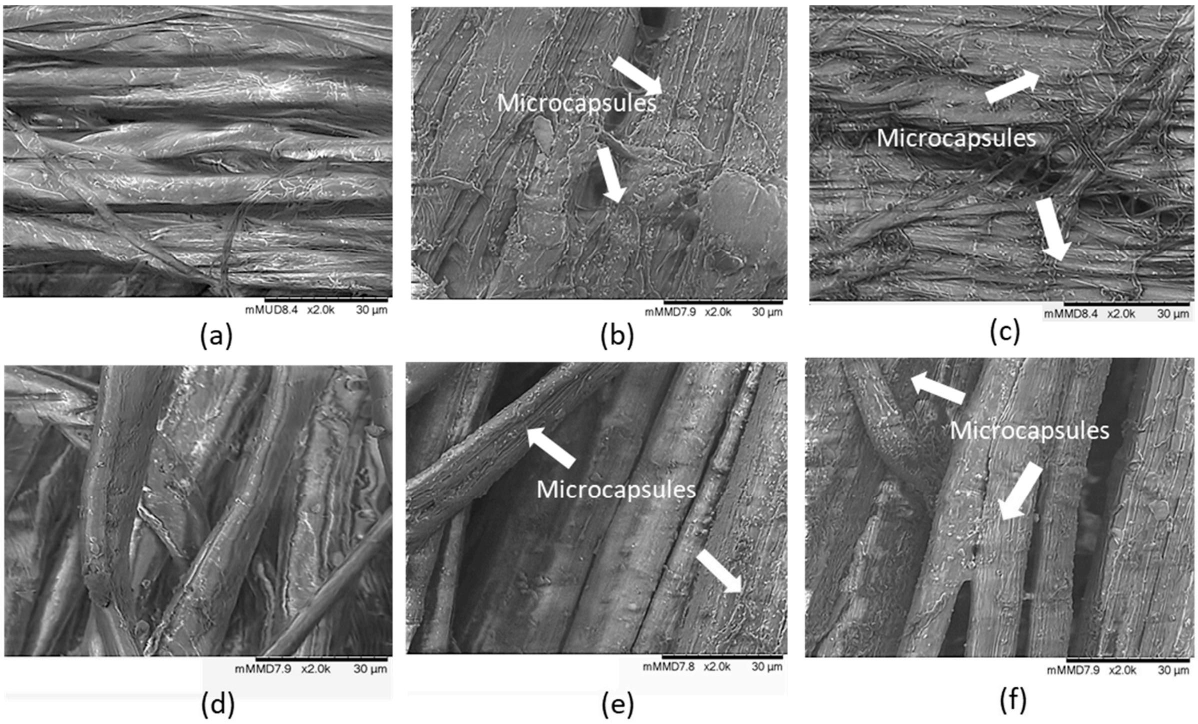

Figure 2. Morphological structures of (**a**) untreated cotton, (**b**) cotton treated with salvia, (**c**) cotton treated with thyme, (**d**) untreated hemp, (**e**) hemp treated with salvia and (**f**) hemp treated with thyme.

As shown in Figure 2d–f, the microcapsules also affected the morphological structure of the hemp fabric. The presence of ethyl cellulose in the fine capsules promoted adhesion between fibers, causing the fibers to adhere along their length. In addition, the deposition of the fine microcapsules was clearly observed on the fabric.

These microcapsules were also found to affect the total porosity of the fabrics (Figure 3). Lower porosity of the cotton and hemp fabrics was associated with the diffusion of microcapsules in the fabrics.

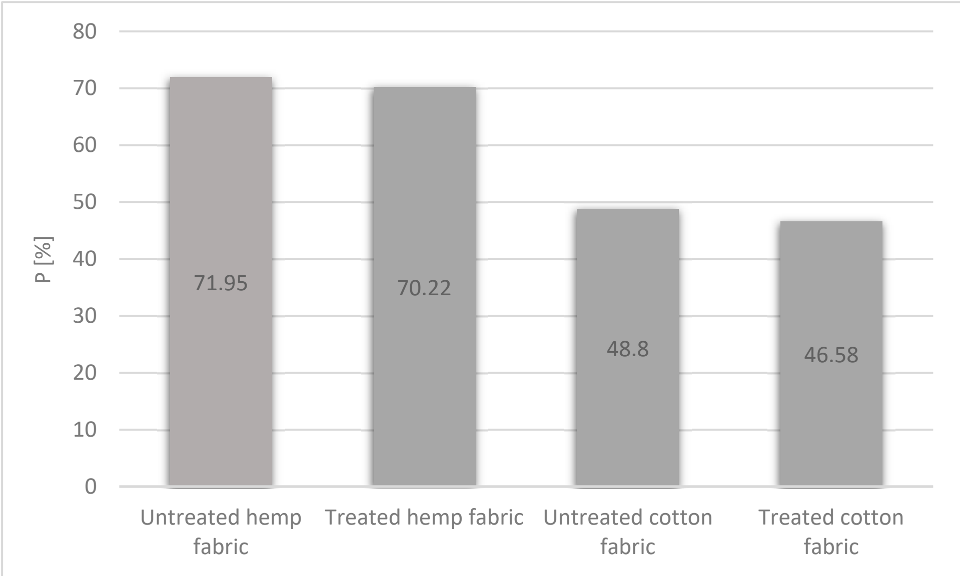

Figure 3. Comparative histogram of porosity values before and after applying the microcapsules.

Additionally, the big difference between the porosity values of the hemp fabric and the cotton fabric is due to the following facts:

- the fineness and nature of the threads of the two fabrics is different;
- the tread counts (yarn numbers) of the two fabrics are different. Cotton has a lower warp and weft yarn than hemp (by about 37% and 65%, respectively);
- there are major differences between the thicknesses of the two types of fabrics.

This result is in agreement with earlier research in which the the authors claimed that a fabric thicknesses affect the fabric's porosity [59].

3.2. ATR–FTIR of Untreated Fabrics and Microcapsule-Treated Fabrics

Figure 4a shows the absorption spectrum for both essential oils, thyme (grey) and salvia (orange). Absorption peaks ranging from 3200 cm^{-1} to 3300 cm^{-1} (O-H stretching) can be clearly observed, indicating the presence of a hydroxyl group for thyme as carvacrol and thymol are some of the constituents. On the other side, salvia only exhibited a slight band. However, both show a band on 1650 cm^{-1} and 1744 cm^{-1} which cannot be achieved on the cotton or hemp spectra without treatment (blue lines for Figure 4b,c).

The FTIR spectra for the cotton and hemp samples (Figure 4b,c, respectively) exhibited transmittance peaks ranging from 3200 cm^{-1} to 3300 cm^{-1} (O-H stretching), indicating the presence of a hydroxyl group in the cellulose structures of the cotton and hemp fabrics. In addition, the absorption peak at 2913–2916 cm^{-1} was attributed to C-H stretching. Both the cotton and hemp treated with microcapsules of thyme and salvia (Figure 4b,c, respectively) seem to show a shoulder in the region around 1643 cm^{-1}. This band is not observed in cellulosic fabrics, and consequently it can be attributed to the treatment with essential oil microcapsules. Furthermore, this should not be attributed to the microcapsule shells as these are comprised of ethylcellulose, whose components, OH and CH, are similar to those observed on cellulosic fibers. The presence of the pick centered around 1643 cm^{-1} indicates the presence of the essential oils on the treated fabric. As a conclusion from the FTIR study, we can confirm that the treatment successfully applies the essential oils to the fabrics.

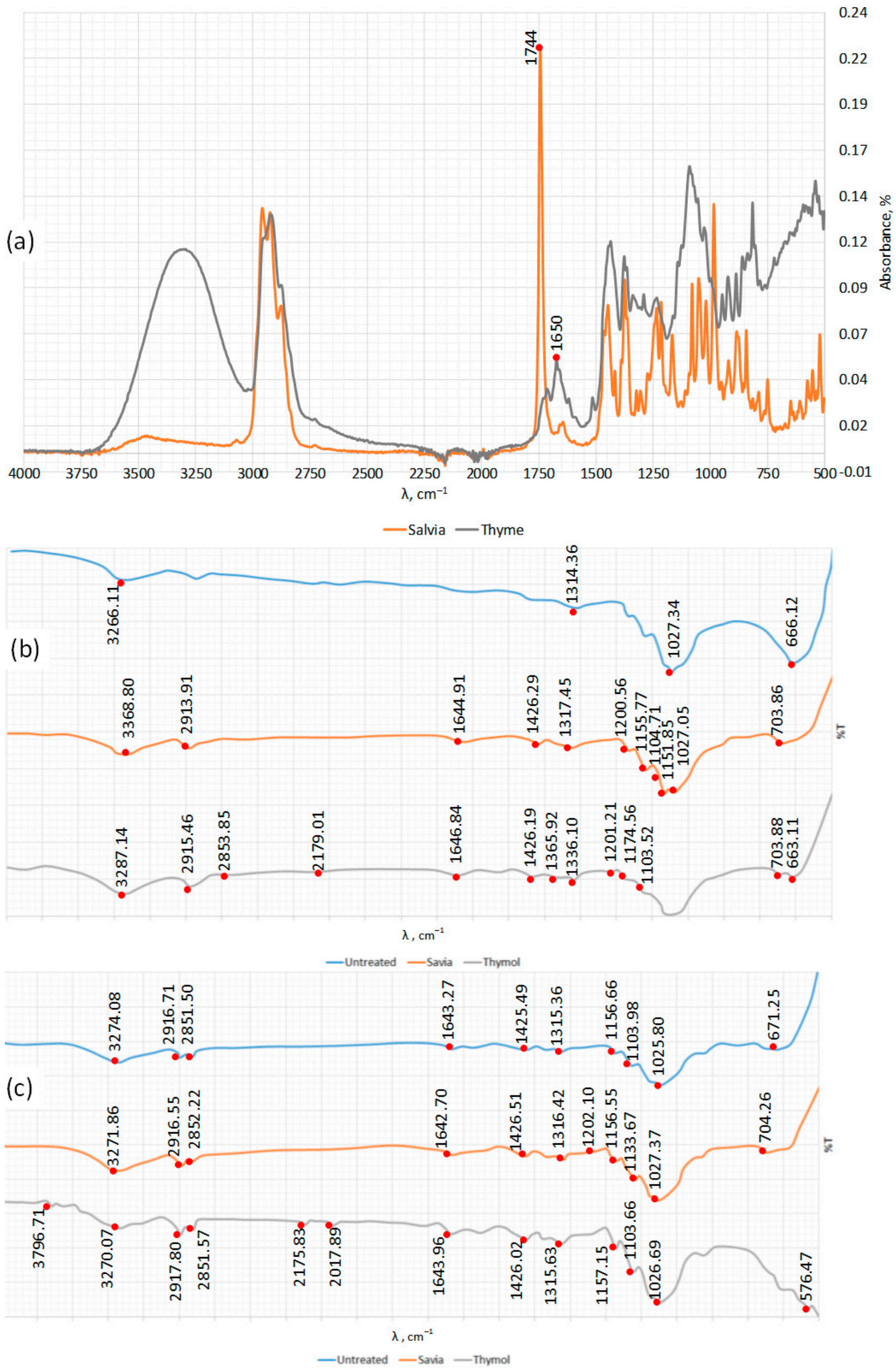

Figure 4. IR spectral for: (**a**) essential oils; (**b**) untreated and microcapsule-coated cotton fabrics; (**c**) untreated and microcapsule-coated hemp fabrics.

3.3. Tensile Strength of Untreated Fabrics and Microcapsule-Treated Fabrics

Figure 5 depicts the tensile strength of the untreated cotton fabrics as well as that of the fabrics treated with the two different essential oils, salvia and thyme. The essential oils acted differently on the tensile strength of the cotton fabric. In the warp direction, the presence of the salvia essential oil on the fabric was found to increase the tensile strength of the fabric by 20%, while in the weft direction, the tensile strength of the fabric also increased by 39%.

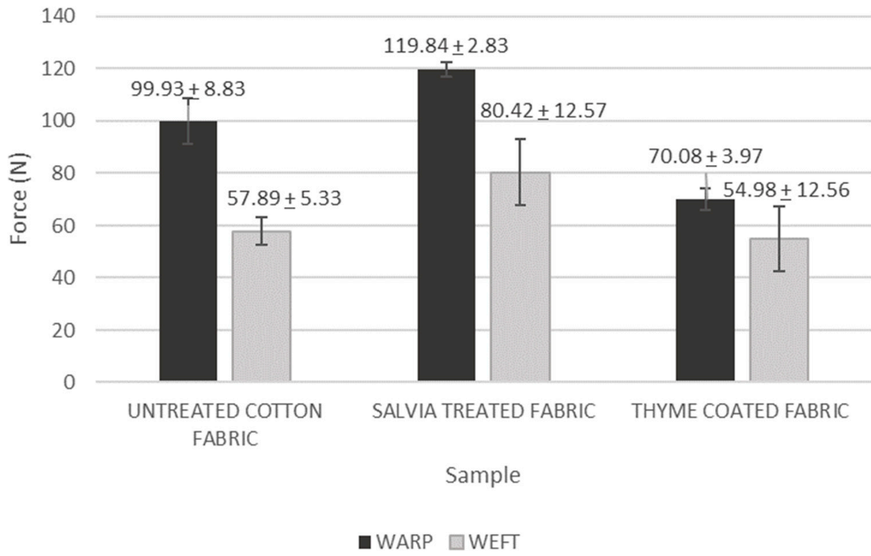

Figure 5. Tensile strength of untreated cotton fabrics and microcapsule-treated fabrics. The values are statistically significant at $p < 0.05$.

However, the thyme essential oil reduced the tensile strength of the fabric by 29.9%. A high amount of the thyme microcapsules was expected to reduce the movement of the fibres in the fabric. As a result, the fabric broke instantly when a high force was applied to the fabric. The results agree with several studies [60–62] in which the authors observed that the addition of microcapsules or nanoparticles to the fabric reduced the tensile strength of the fabric. In addition, the treated cotton fabrics also showed a reduction in tensile strength in the weft direction.

The effects of the salvia and thyme essential oils on the hemp fabric were also investigated in this study. As illustrated in Figure 6, the deposition of the salvia and thyme microcapsules reduced the tensile strength of the fabrics. In the warp direction, the tensile strength of the hemp fabrics decreased by up to 36% for salvia and 40% for thyme. These microcapsules limit the movement of fibers in the fabric, causing the fabric to break instantly at a higher load. However, in the weft direction, the fabrics treated with the salvia and thyme microcapsules demonstrated similar tensile strengths, ranging from 65.18 N to 69.69 N.

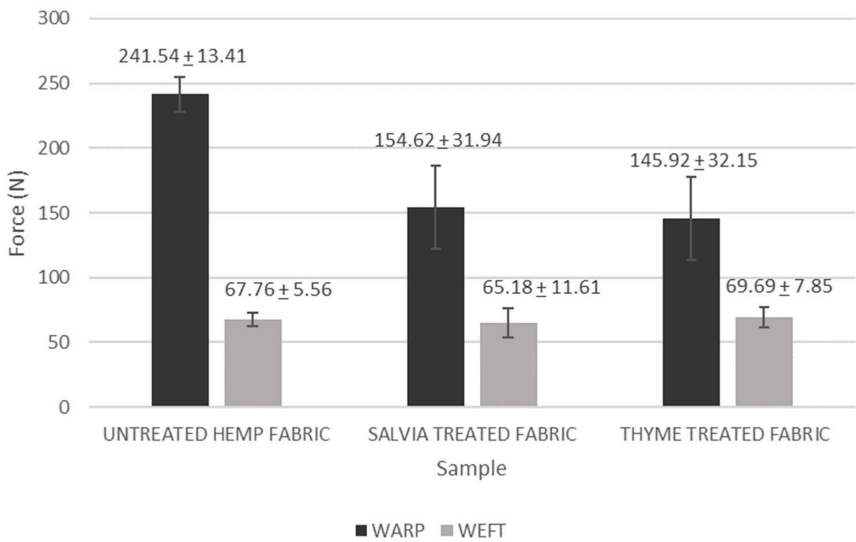

Figure 6. Tensile strength of untreated hemp fabrics and microcapsule-treated fabrics. The values are statistically significant at $p < 0.05$.

3.4. Principal Component Analysis (PCA) of Data

The data for the untreated cotton and hemp and the fabrics treated with salvia and thyme were processed using the kernel PCA method.

In classical PCA, the data are computed and projected into a new feature space by computing the covariance matrix. Data volume reduction is only achieved with linear projection. In the kernel PCA method, the projection of the data into the new feature space is calculated using a kernel function [63].

The kernel function used in the present work is a polynomial of the second degree. In general, it is described by the equation:

$$k\,(x,\,y) = (x^T\,y + C)^d \tag{1}$$

where k is a kernel function, x and y are coordinates of the kernel function, C is a constant, and d is a power factor.

Vectors were composed of a total of five features describing the textile fabrics used, which were treated with preservatives of natural origin. Before processing, the experimental data were normalized to the interval 0–1. Table 2 shows the normalized values of the used vectors of the features describing the textile fabrics.

Figure 7 shows the results obtained from processing the data with the kernel PCA method. For the cotton textiles, there was an overlap between the data for the untreated textiles and those treated with salvia. The data for thyme-treated cotton textile fabrics are clearly distinguished and combined in a separate group. These results show that salvia treatment does not significantly affect the main characteristics of cotton textile fabrics. Thyme treatment, on the other hand, produced a significant deviation in the characteristics of the cotton fabrics after treatment.

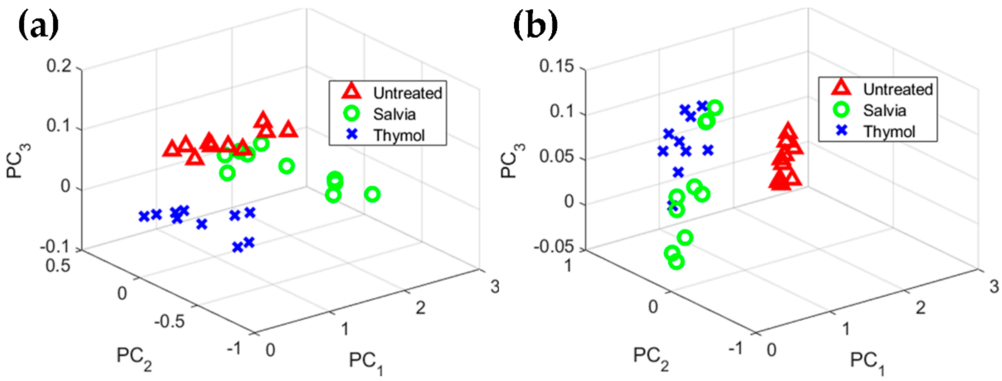

Figure 7. Separation of data with kernel PCA. (**a**) Cotton fabrics; (**b**) hemp fabrics.

A clear separation of the data set was observed for the untreated textile fabrics and the hemp. After treatment with salvia and thyme, the data for these textile fabrics overlapped. This indicates that the treatment of textile fabrics leads to a uniform change in their characteristics. The characteristics of the treated textile fabrics clearly differ from those of the untreated fabrics.

From the above analyses, it can be concluded that the treatment of cotton textile fabrics with salvia does not lead to significant changes in their main characteristics. On the other hand, treatment with both salvia and thyme resulted in significant deviations in the basic characteristics of hemp textile fabrics. The kernel PCA results complement those obtained from previous analyses. The influence of the treatment of both types of fabrics with preservatives of natural origin has been confirmed.

Table 2. Normalized values of feature vectors.

	Fabric					Cotton				
						Hemp				
Paramer Treatment	**TS Warp**	**TS Weft**	**Weight**	**Warp Yarn Size**	**Weft Yarn Size**	**TS Warp**	**TS Weft**	**Weight**	**Warp Yarn Size**	**Weft Yarn Size**
U	0.26	0.13	0.06	0.21	0.09	0.49	0.18	0.11	0.17	0.14
U	0.32	0.14	0.06	0.32	0.12	0.55	0.23	0.21	0.37	0.25
U	0.27	0.14	0.10	0.42	0.16	0.51	0.21	0.21	0.31	0.28
U	0.28	0.13	0.07	0.34	0.14	0.54	0.24	0.12	0.26	0.24
U	0.28	0.14	0.09	0.21	0.18	0.51	0.24	0.15	0.22	0.15
U	0.22	0.07	0.03	0.20	0.05	0.49	0.18	0.01	0.02	0.00
U	0.26	0.10	0.02	0.07	0.01	0.48	0.16	0.02	0.06	0.05
U	0.24	0.10	0.05	0.19	0.03	0.46	0.15	0.00	0.17	0.03
U	0.24	0.12	0.00	0.11	0.01	0.47	0.12	0.00	0.12	0.09
U	0.25	0.07	0.05	0.02	0.01	0.47	0.14	0.01	0.06	0.11
S	0.43	0.37	0.06	0.21	0.09	0.15	0.15	0.11	0.17	0.14
S	0.44	0.44	0.07	0.32	0.14	0.27	0.29	0.21	0.25	0.25
S	0.43	0.46	0.07	0.29	0.16	0.21	0.19	0.15	0.37	0.28
S	0.43	0.43	0.10	0.31	0.18	0.23	0.25	0.19	0.20	0.27
S	0.44	0.50	0.10	0.41	0.15	0.22	0.28	0.17	0.23	0.17
S	0.42	0.30	0.02	0.18	0.06	0.10	0.04	0.08	0.11	0.14
S	0.42	0.32	0.03	0.12	0.00	0.06	0.07	0.09	0.04	0.09
S	0.42	0.34	0.05	0.00	0.05	0.06	0.12	0.10	0.11	0.04
S	0.41	0.28	0.01	0.10	0.07	0.03	0.03	0.10	0.10	0.00
S	0.41	0.28	0.02	0.04	0.06	0.05	0.00	0.11	0.08	0.04
T	0.02	0.10	0.06	0.21	0.09	0.12	0.21	0.11	0.17	0.14
T	0.03	0.12	0.09	0.41	0.11	0.23	0.25	0.18	0.30	0.19
T	0.04	0.19	0.08	0.31	0.18	0.13	0.27	0.11	0.18	0.22
T	0.02	0.10	0.09	0.35	0.11	0.15	0.28	0.14	0.22	0.14
T	0.04	0.19	0.11	0.25	0.18	0.20	0.28	0.17	0.18	0.22
T	0.01	0.01	0.04	0.01	0.00	0.05	0.18	0.00	0.00	0.07
T	0.01	0.00	0.01	0.06	0.08	0.01	0.21	0.07	0.01	0.00
T	0.02	0.05	0.01	0.19	0.04	0.00	0.19	0.03	0.13	0.14
T	0.00	0.03	0.06	0.15	0.07	0.05	0.14	0.02	0.04	0.03
T	0.00	0.04	0.00	0.17	0.00	0.04	0.11	0.05	0.16	0.11

U: untreated; S: salvia; T: thyme; TS: tensile strength.

4. Discussion

This work is a comprehensive assessment of the changes in textile fabrics after treatment with microencapsulated essential oils. These results complement to a significant extent known methods for the analysis of textile fabrics from historical textiles.

Stan et al. [64] investigated cotton and wool textile fabrics treated with sage or rose essential oils. This work is presented with the results of a treatment of historical textiles, but treated with microencapsulated salvia and thymol essential oils.

In this work, knowledge gaps related to the application of microencapsulated essential oils for the treatment of historical textiles are addressed. In this way, we implemented the methods of Mehta et al. [51]. The authors recommend that research not only be done on the therapeutic benefits of essential oils applied to textiles, but also on their preservative properties. This task has been successfully carried out in our work on textile fabrics from Romanian historical textiles. In this way, a synergy is achieved between the relevant fields of knowledge, i.e., technological studies of essential oils and the technical field related to their application for the conservation of historical textiles.

According to Drábková et al. [65], like silver nanoparticles, essential oils are not yet widely used in the disinfection and preservation of cultural heritage sites. These essential oils are often used in the food and cosmetic industries. It is characteristic of essential oils that they have an inhibitory effect against viruses, bacteria, fungi, and insects. Essential oils contain a number of chemical compounds that include hydrocarbons, aldehydes, ketones, phenols, phenolic ethers, alcohols, and esters. For this reason, the mechanisms of action on textile fabrics have not been sufficiently studied. Due to this lack of research, the nature of the molecular interaction between the fiber and the oil has not been clearly established. What has been demonstrated is the fact that microcapsules are not reactive with fibers, and that it is necessary to add some kind of binder if they need to be fixed [66] to increase their presence during fabric use and maintenance. In this case, as the fabrics are not supposed to be worn, there is no necessity to include additional chemicals which can produce side effects. Regarding the oil behavior, the encapsulation objective is to extend the release of the oil, protect it from external factors which can degrade it, and prevent leakage on the fabric. Furthermore, a direct application of oil on the fabric can directly imply a change in color as the oils are not transparent and can produce a yellowish or greenish color. Some publications have demonstrated that even when the oils are encapsulated there is still an antibacterial effect [67], and this biocide effect will help to improve the conservation of valuable fabrics without altering the color. Both salvia and thyme show an antimicrobial and antibacterial effect, and according to the results we observed, there are no significant differences in their behavior. However, there was a modification of the properties of the cotton samples treated with thyme which was not observed for in those treated with salvia. Both salvia and thyme influenced the properties of the hemp, though the influence of the salvia was lower. This information is valuable can be used to decide which one is the most suitable.

Rezić [68] describes the historical scientific achievements of the chemical, anthropological, and conservation–restoration communities. The author points out that the composition of microencapsulated essential oils gives them potential for application in the conservation of historical textiles. This method is better than methods which use substances of chemical origin. This thesis is demonstrated in this work.

Negi et al. [69] indicate that essential oils offer antimicrobial properties. They are applicable in various fields, including the preservation of textiles. A complex method for the analysis of historical textiles is proposed, which can be extended to research related to the antimicrobial properties of microencapsulated essential oils in historical textiles.

The tests carried out, together with the other mentioned tests, are encouraging and should be intensified and continued on a large scale in Europe and all over the world to test the possibility of eco-friendly EOs as valid alternatives to synthetic preservatives (for biocidal activity, etc.), because they benefit from a low degree of toxicity to humans and the environment.

5. Conclusions

In this work, a multidisciplinary study was made related to the diagnosis, protection, and storage of ethnographic textiles from personal collections. The study focused on the influence of eco-friendly, microencapsulated sage and thyme essential oils, which show low toxicity to humans and the environment, on the mechanical and morphological properties of lignocellulosic heritage textiles, cotton, and hemp.

It was found that the morphological evaluation of the test textile samples could be successfully performed using scanning electron microscopy. Additionally, the combination of ethyl cellulose and the essential oil solution was shown to bind the cotton fibers, making the fibers invisible in SEM micrographs. Qualitative ATR–FTIR spectral analysis showed that the treatment of cotton and hemp textile fabrics has a significant effect on the composition and properties of the textile fabrics. This is because after the treatment, new chemical compounds appear in the textile fabrics, which significantly change the properties of the textile fabrics of the two investigated materials.

Through a standard test method and a specialized statistical analysis of the experimental data, it was proven that the treatment of hemp and cotton textile fabrics with salvia enhances the tensile quality of the fabric by 20% and 39% in the warp and weft directions, respectively.

From the principal component analysis, it was found that the textile fabrics treated with salvia clustered close to the untreated ones. This shows that treatment with essential oils from the indicated plant does not have a significant effect on the tensile properties of textile fabrics. On the other hand, the thymol-treated textile fabrics were significantly removed from those of the control sample. From this it can be concluded that thymol treatment has a significant effect on their tensile properties of cotton textile fabrics. In hemp fabrics, the thymol and salvia treatments had a highly significant effect on the tensile performance of the textile fabrics, although the influence of the salvia was lower than that of the thyme. The results obtained from the PCA confirmed those obtained using standard test methods for determining the tensile strength of textile fabrics.

The results of this work improve upon and complement those of the available literature. Results are presented, as well as a methodology for the analysis of empirical data related to the cultural heritage of Romania. Such studies had not yet been undertaken for textile fabrics from Romania. The results obtained have the potential to be used in museums, universities, and ethnographic studies that own and maintain textile heritage collections.

To better understand the shelf-life of heritage textiles, future investigations on the temperature, humidity, luminosity, and contamination by microorganisms are recommended.

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