

THE EFFECT OF THE VAPORS OF CLOVE AND LAVENDER OILS ON THE CHEMICAL AND OPTICAL PROPERTIES OF SILVER GELATIN PRINTS IN WOOD FRAMES

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Abstract

Photographs made during the last one hundred years belong to a class known as silver gelatin prints. Framing has been traditionally used in displaying photographic collections. Although a number of materials were used in framing, wood was the most common material used in the creation of frames in the past. Among the many factors threatening the permanence of framed silver gelatin prints, fungi are certainly one of the most common, particularly in a humid climate. Many fungicides have been globally used to disinfect contaminated historic objects; however, this is definitely not the best approach. On this basis, the aim of this paper was to evaluate the effect of both clove and lavender oils on the optical and chemical properties of framed silver gelatin prints. The oils were provided by the National Research Center in Cairo, Egypt. Wood and photographic samples were exposed in desiccators to the selected essential oils in vapor phase for a period of 5 days. All samples were exposed post oil vapor exposure to humid heat aging conditions at a temperature of 80°C and 65% RH for a period of 5 days. Treatments were evaluated using several techniques including visual inspection, microscopic inspection, pH value measurement, colorimetric measurements, Fourier transform infrared spectroscopy and X-ray diffraction. Based on the obtained results, clove and lavender essential oil vapors are safe for use on silver gelatin prints housed in wood frames.

Keywords: Framed silver gelatin prints; Mold; Essential oils; Colorimetric measurement; pH value measurements; FTIR; XRD

Introduction

Growing concern for the preservation of photograph collections has led to a greater interest in developing appropriate conservation procedures to help prolong the lifespan of such valuable records. Photographs form an essential part of the Egyptian cultural and visual heritage for their artistic and documentary value as well as as record of the history of photography. Photographs made during the last one hundred years belong to a class known as silver gelatin prints. A silver gelatin print is composed of three layers: the primary support (i.e. paper), the baryta layer (i.e. fine particles of barium sulfate in gelatin layer) and the gelatin binder layer carrying the final image material (i.e. metallic silver particles) [1-4]. Furthermore, many photographs are a part of a housing which is designed to support and protect the artwork. Framing adds an aesthetic appeal to the photographs, acts as a support system, visually enhances the image and provides proper protection for the collections on display [5]. Although a number of materials were used in framing, wood was the most common material used in the creation of frames in the past and many silver gelatin prints were found in their original wood frames [6-8]. Original frames are considered museum objects in their own right. Accordingly, when a frame

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is known to be original or of value, it is considered significant to exclude reframing using new materials; since it conveys the spirit of the time in which it was made. Alternatively, conservation is a wiser option in terms of preserving the historic integrity of the object [9, 10].

Silver gelatin prints are one of the most vulnerable objects held by museum, archives and libraries. The multiplicity of the components which make up silver gelatin prints gives them a unique yet complex structure making them more prone to both intrinsic and extrinsic factors of deterioration [11-13]. The presence of a wood frame while advantageous for the preservation of photographs, it can also be problematic when used in an uncontrolled environmental condition; thus, presenting a new challenge for photograph conservators. The threats to framed silver gelatin prints are many; since they are very sensitive to their surrounding environment (i.e. improper temperature and relative humidity levels, light, air pollution, improper handling, poor storage and display materials, biological threats and disasters) [14, 15].

Among the many factors threatening the permanence of valuable photograph collections, fungi (i.e. mold) are certainly one of the most significant and most common, particularly in a humid climate [16]. Additionally, display of silver gelatin prints inside structures intended for their preservation such as wood frames creates new manmade environments for fungi to inhabit [17]. Paper, gelatin and wood found in framed silver gelatin prints are good sources of nourishment for fungi [18-22]. The highwater content and hygroscopic nature of such components make framed silver gelatin prints more susceptible to damage by mold [5, 18, 23]. Under favorable conditions, fungi can attack all these organic materials. For many types of mold growth, the optimum environment is over 22°C and over 70% relative humidity with poor air circulation [24]. Generally, fungi produce chromatic alterations such as stains of various colors due to mycelium growth and the release of colored metabolites [25]. However, damage can range from stains to complete destruction [23]. Mold feeds on gelatin, paper and wood extracting carbon and nitrogen through an enzyme hydrolysis reaction that weakens and lowers the mechanical strength of such materials [5, 25, 26]. Wood-decaying fungi include white rot (i.e. *Pholiota sp.*, *Fomes sp.* and *Pleurotus sp.*), brown rot (i.e. *Merulius lacrymans*, *Poria spp.* and *Coniophora puteana*) and soft rot (i.e. *Chaetomium*, *Xylaria*, *Alternaria* and *Humicola stenphylium*) [18, 23, 27]. On the other hand, the most frequent species identified in photograph collections belong to the genera *Aspergillus*, *Pencillium*, *Mucor*, *Cladosporium*, *Trichoderma*, *Phoma*, *Rhizopus*, *Epicoccum* and *Alternaria* [14, 28].

Controlling the surrounding environmental conditions and keeping the photograph collections clean are among the recommended practices to prevent microbial growth within the display area. However, stabilization of thermohygrometric parameters is an extremely difficult process, particularly in case of a disaster or in certain cases where the installation of control systems will either be too expensive or too complicated [29, 30]. Fungicides have been globally used to disinfect contaminated historic objects such as thymol, formaldehyde, ethylene oxide and many others [31-33]. Nevertheless, this is definitely not the best approach. The use of such toxic chemical disinfection methods is becoming increasingly restricted mainly due to their damaging effect on the environment and human health [32, 34]. This has forced researchers to look for safe and natural alternatives.

In recent years, essential oils have been recognized by scientists as having antibacterial and antifungal properties. Essential oils are volatile aromatic concentrated hydrophobic oily liquids which are obtained from various plant parts [35]. The biocide activity of extracts of sweet basil, clove, cumin, cinnamon, anise, bardock, garden cornflower, lavender, thyme and many other essential oils has been addressed in many publications [36-48]. However, among these studies, only a few evaluate their effect on the properties of cultural heritage material such as historic silver gelatin prints. On this basis, the aim of this paper was to evaluate the effect of both clove and lavender oils on the optical and chemical properties of framed silver gelatin prints.

Experimental part

Materials

Preparation of photographic samples

Fomabrom Variant IV 123 Multigrade Fiber-based paper with a semi matt surface finish was selected for the preparation of silver gelatin fiber-based test materials. Greyscale samples with 10 different densities (i.e. steps) were produced using a Kodak enlarging exposure scale for a full range of silver density (i.e. highlights, midtones and shadow values). The photographic paper was contact printed under the glass template using a Durst M670 BW photographic enlarger. The samples were exposed in the enlarger for 50 seconds. A 5 + 4 Jessop contrast filter was used. Chemicals used in processing the samples were prepared as listed in table 1. After exposure, the samples were developed in Fomatol LQN for 2:15 minutes at a temperature of 24°C and fixed in a hypo bath for a period of 10 minutes. Washing proceeded for 20 minutes. The samples were then allowed to dry, face up on a drying rack. To flatten the samples, they were placed between blotting paper and pressed in a hydraulic press for two days.

Table 1. Photographic chemical solution used in this experiment

| Chemical name | Quantity | Information |
|---------------|----------|------------------------------|
| Fomatol LQN | 250mL | 1:7 (dev:water) at 25°C |
| Hypo | 250g | 250 g in 1L of water at 25°C |

The resultant greyscale contained ten different densities/steps (i.e. 2, 3, 4, 6, 8, 12, 16, 24, 32, and 48). The study was carried out on three densities: step 2 representing the highlight areas, step 12 representing the midtones and step 48 representing the shadow areas.

Preparation of wood samples

Pine wood was used to prepare the samples representing the frame [7]. Two cubes of the size of 2 cm³ were used for this study (Fig. 1). Table 2 lists the samples used for this study.

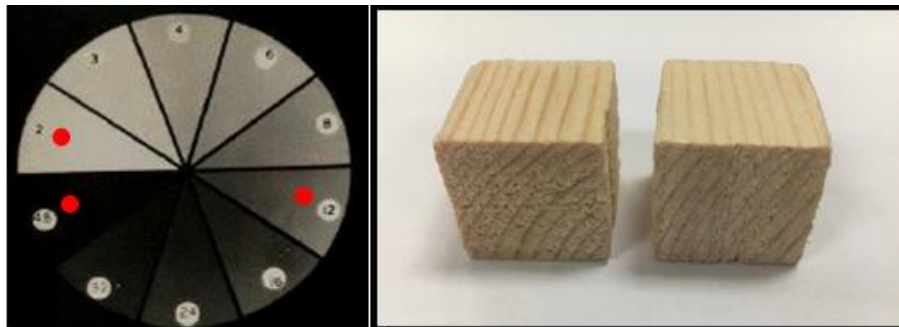


Fig. 1. Prepared samples. Photographic sample and the steps selected for testing (left) and wood sample (right)

Table 2. Test samples

| Sample No. | Quantity |
|------------|---|
| CP | Photographic sample exposed to clove oil |
| LP | Photographic sample exposed to lavender oil |
| 1 | Wood sample exposed to lavender oil |
| 2 | Wood sample exposed to clove oil |

Essential oils

The essential oils were provided by the National Research Center (NRC) in Cairo, Egypt. Two essential oils of the plants *Syzygium aromaticum* L. (i.e. clove) and *Lavandula angustifolia* Mill. *Ssp. Angustifolia* (i.e. lavender). Clove essential oil is a distillate of flowers, stems and leaves of *Syzygium aromaticum* found in Eastern Hemisphere or *Eugenia aromaticum* and *Eugenia caryophyllata* in Western Hemisphere. The oil possesses anesthetic, antioxidant, antifungal, antiinflammatory, cytotoxic and antimicrobial properties towards pathogenic fungi, bacteria, and viruses [49]. The major component of clove oil is usually considered to be eugenol, with β -caryophyllene and lesser amounts of other components such as benzyl alcohol [50]. Lavenders are a genus of about 25 - 30 species of flowering plants in the mint family, Lamiaceae, native to the Mediterranean region south to tropical Africa and to the many regions of Asia. The genus includes annuals, herbaceous plants, shrubs, and small shrubs. Lavender has been used for centuries as an herbal remedy [51]. The major components of lavender essential oils are 1,8-cineole, cis- β -ocimene, linalool, 1-octen-3-yl acetate, camphor, linalyl acetate and lavandulyl acetate [52].

Methods

Application method

Tests were performed in two glass test-chambers (i.e. desiccators). The application procedure involved pouring 25mL of clove and lavender oils into two separate glass beakers and each was placed in a different desiccator. A petri dish with saturated magnesium sulfate solution was placed in each desiccator to maintain humidity at 80%. Two sample sets were prepared from each tested material (i.e. photographic and wood samples). One set was exposed to clove essential oil and the other was exposed to lavender essential oil. Samples were exposed to the vapors of the oils for a period of 5 days (Fig. 2).

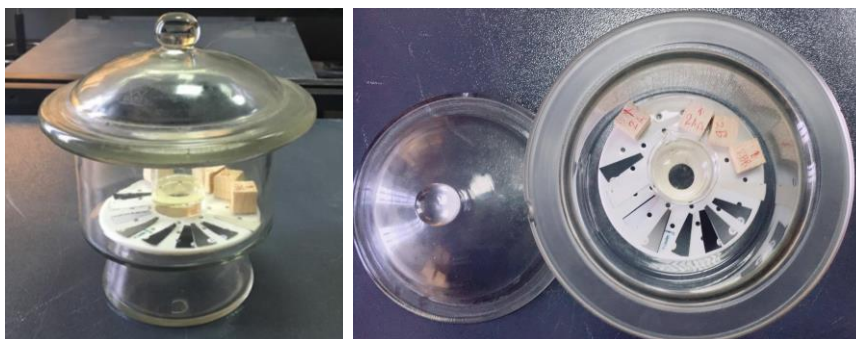


Fig. 2. Exposure of the samples to the vapors of the essential oils using a desiccator

Assessment methods

Immediate and long-term effects of the tested essential oils have been examined and evaluated. Accordingly, measurements were made before and after exposure to the vapors of clove and lavender oils and after accelerated aging. Surface, optical (i.e. CIELAB color coordinates), physio-chemical (i.e. pH value measurements) and chemical properties of the tested samples (i.e. changes in chemical structure) were determined as follows:

Surface examination by digital microscope

A SUPREYES PZ01 500X USB Digital Microscope was used to document the resultant damage forms due to the action of each essential oil before and after accelerated aging.

Colorimetric measurements

The change in color was measured using a MiniScan Model No. EZ MSEZ0693. All samples were measured in a visible region, with an interval of 10 nm using D65 light source and an observed angle of 10 degrees. The CIELAB color parameters (L^* , a^* , b^*) were used,

where L^* defines lightness and varies from 0 (black) to 100 (white); a^* represents the red/green axis, where $+a^*$ means red and $-a^*$ means green; b^* represents the yellow/blue axis, where $+b^*$ means yellow and $-b^*$ means blue. All values of L^* , a^* , and b^* were obtained before exposure, after exposure and after artificial aging. Each reading was the average of three measurements. The total color difference ΔE^* was also calculated from the following formula: $\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$ [53-58]. The analysis was carried out at the Faculty of Archaeology, Cairo University.

pH value measurements

The pH of the essential oils was evaluated using pH paper strips. Using a pipette, a drop of each oil was placed on the pH paper strips and left for a few minutes. The color of the strip was then compared with that of the standard to determine the pH value of tested oils.

Fourier transform infrared spectroscopy (FT-IR)

FT-IR spectroscopy was used to study the chemical changes post artificial aging. The FTIR instrument used is Jasco FT/IR-6100 Spectrometer in the range of $4000 - 400\text{cm}^{-1}$, in absorption mode. The analysis was carried out at the National Institute of Standards (NIS) in Cairo, Egypt.

X-ray diffraction (XRD)

Cellulose Crystallinity measurements in the wood samples were obtained by X-Ray powder diffraction using a Philips Analytical X-Ray B.V. (type PW 1840 Diffractometer, Netherland) with a Cu tube anode. Peaks were identified and measured by using Match 3 + PDF4 2017 software program.

Accelerated Aging

After exposure, samples went through accelerated aging to evaluate the long-term effects of both oils on wood and photographic samples. Treated samples were artificially aged at a temperature of 80°C and 65% RH for 5 days, which is equivalent to aging of paper under normal conditions for 25 years. The aging process was in conformance with the ISO 5630-3:1996 standard [59, 60]. This procedure was performed in a Binder dry oven with digital indicator, model no. 92403000002000 at the National Institute of Standards (NIS) in Cairo, Egypt.

Results and discussion

Surface examination by digital microscope

Photographic samples

Visually speaking, very slight change in color was observed in the highlights and midtones of clove and lavender-treated samples after exposure to essential oil vapors and after artificial aging (Fig. 3).

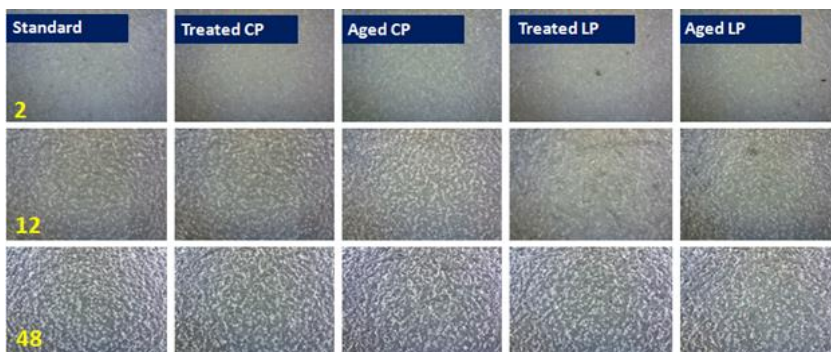


Fig. 3. Microscopic inspection of photographic samples after exposure to the vapors of clove and lavender oils and artificial aging

Wood samples

Very slight change in color was observed in the wood samples of clove and lavender-treated samples after exposure to the vapors of the essential oils and after artificial aging (Fig. 4).

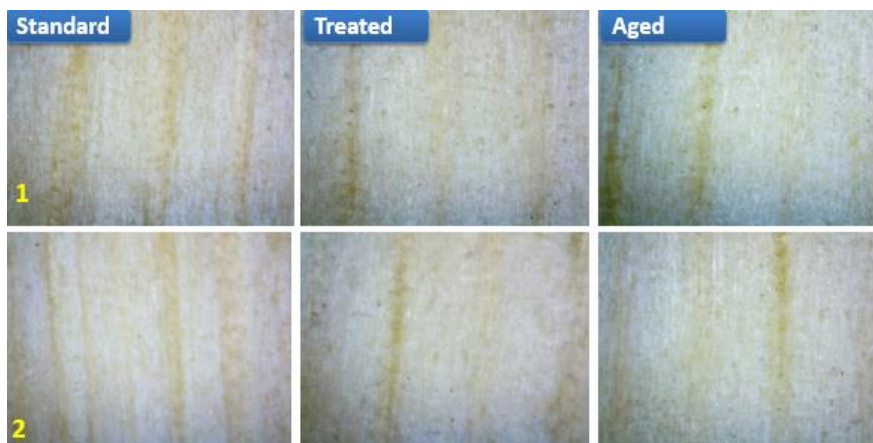


Fig. 4. Microscopic inspection of wood samples after exposure to the vapors of clove and lavender oils and artificial aging.

Colorimetric measurements

The total color difference (ΔE^*) is a value useful as an indicator of the difference between the sample and the reference. According to DIN EN ISO (super ceded by BS EN ISO 4628-1:2004), evaluation of ΔE^* is as follows:

- 0 – 1: color difference is not visible.
- 1-3: few people can recognize the difference.
- 3- 5: 66% of people can recognize the difference.
- > 5 everyone can recognize the difference [61, 62].

In literature, chromatic variation of 2-3 can be considered noticeable by human eye; however, it is clearly lower than the threshold limit required (i.e. $\Delta E^* = 5$) for the maintenance and restoration of historical surfaces [63].

Photographic samples

The obtained results for the clove and lavender treated samples show no to minimal change for all three color coordinates (i.e. L^* , a^* and b^*) in treated and aged treated samples. However, the highlights and midtones represented in step 2 and step 12 respectively appear to have been the most affected (Fig. 5). Results are given in table 3. All samples showed total color change below 5 (i.e. $\Delta E^* < 5$) excluding aged clove-treated sample CP12 and lavender-treated sample LP12 showing ΔE^* values of 5.01 and 5.49, respectively (Fig. 6).

Table 3. Measured $L^*a^*b^*$ values for photographic samples post treatment and post aging

| Clove-treated samples | L^* | a^* | b^* | Lavender-treated samples | L^* | a^* | b^* |
|-----------------------|-------|-------|-------|--------------------------|-------|-------|-------|
| Standard 2 (S2) | 75.66 | 1.74 | 8.18 | Standard 2 (S2) | 75.66 | 1.74 | 8.18 |
| Treated CP2 | 73.16 | 1.56 | 7.57 | Treated LP2 | 71.47 | 1.87 | 8.18 |
| Aged treated CP2 | 79.64 | 1.68 | 8.68 | Aged treated LP2 | 71.01 | 1.85 | 8.38 |
| Standard 12 (S12) | 43.35 | 0.54 | 2.99 | Standard 12 (S12) | 43.35 | 0.54 | 2.99 |
| Treated CP12 | 43.81 | 0.51 | 2.81 | Treated LP12 | 45.61 | 0.73 | 2.26 |
| Aged treated CP12 | 48.32 | 0.63 | 3.59 | Aged treated LP12 | 48.73 | 0.69 | 4.08 |
| Standard 48 (S48) | 17.23 | -0.05 | 0.16 | Standard 48 (S48) | 17.23 | -0.05 | 0.16 |
| Treated CP48 | 16.45 | 0.03 | 0.26 | Treated LP48 | 16.56 | 0.01 | 0.27 |
| Aged treated CP48 | 19.82 | 0.05 | 0.22 | Aged treated LP48 | 17.35 | 0.08 | 0.326 |

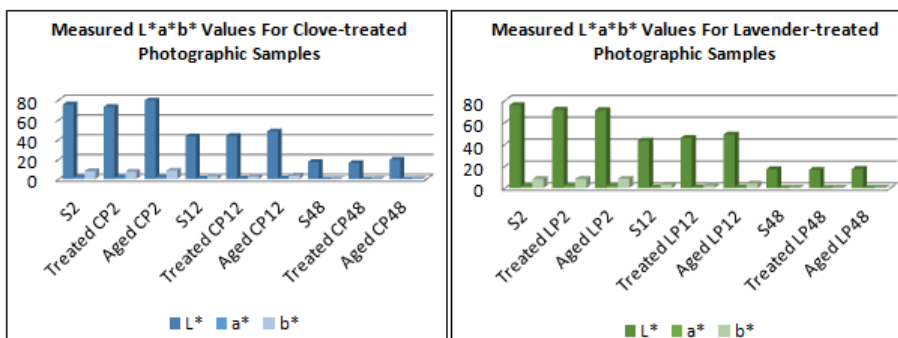


Fig. 5. Measured L*a*b* values for clove-treated photographic samples (left) and lavender-treated photographic samples (right)

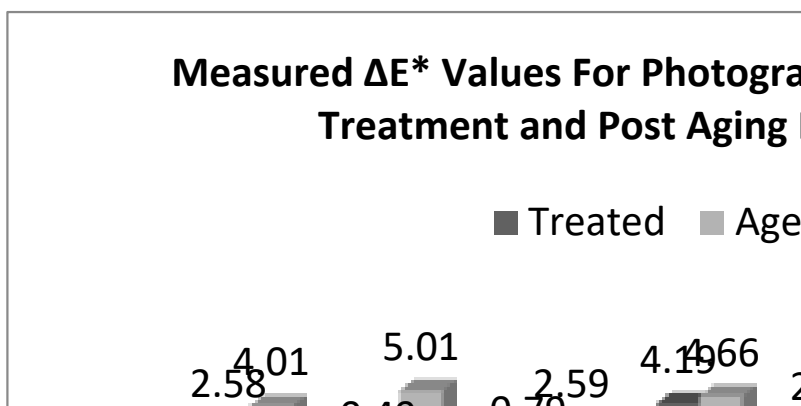


Fig. 6. Measured ΔE^* values for photographic samples post treatment and post aging

Wood samples

The results for the clove and lavender treated samples show slight change for all samples. Results are given in Table 4. All samples showed total color change below 4 (i.e. $\Delta E^* < 4$) (Fig. 7).

pH value measurements

The measured pH values for both essential oils were around 7.

Fourier transform infrared spectroscopy (FT-IR)

Photographic samples

FT-IR Spectra for the photographic samples show the absorption bands characteristic of gelatin, of these, the amide I band ($1600 - 1700\text{cm}^{-1}$) and amide II ($1500 - 1600\text{cm}^{-1}$) are the most prominent vibrational bands of protein backbone [64]. Amide I is presided by the C=O stretching vibrations of the peptide linkage (70 – 85%) [65] Amide II is a combination of several types of vibrations within the peptide group; it originates from the in-plane N-H bending (40 – 60%), along with both the C-N stretching vibrations (18 – 40%) and C-C stretching vibrations (about 10%) [661, 67].

With respect to degradation, hydrolysis of gelatin appears as an increase in amide I band intensity and the OH stretching band. Oxidation of gelatin results in the formation of carbonyl compounds and this can be seen as a slight shoulder on the amide I band or as an increase in the area of the amide I band [68]. Increase in amide I band intensity is related to an increase in

random coil at expense of the ordered secondary structure [69]. Cellulose oxidation results in the increase of the intensity of the amide I band [70].

Table 4. Measured $L^*a^*b^*$ values for wood samples post treatment and post aging

| Samples | L^* | a^* | b^* | ΔE^* |
|----------------|-------|-------|-------|--------------|
| Standard | 77.57 | 5.62 | 20 | ----- |
| Aged Wood | 79.91 | 4.33 | 19.33 | 2.09 |
| Treated 1 | 74.36 | 4.81 | 18.58 | 2.52 |
| Aged treated 1 | 73.06 | 5.28 | 20.62 | 3.33 |
| Treated 2 | 75.44 | 4.33 | 18.65 | 2.05 |
| Aged treated 2 | 72.83 | 4.92 | 20.07 | 3.52 |

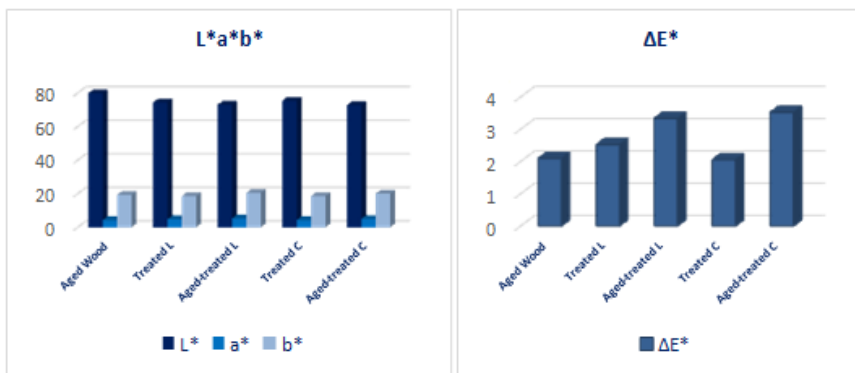


Fig. 7. Measured $L^*a^*b^*$ values for clove-treated wood samples (left) and measured ΔE^* values for wood samples post treatment and post aging (right)

Obtained results reveal that no to insignificant change in the position or intensity of the hydroxyl group (A), amide I (B) and amide II (C) have occurred for all investigated samples, whether treated with clove or lavender essential oil vapors (Figs. 8-10).

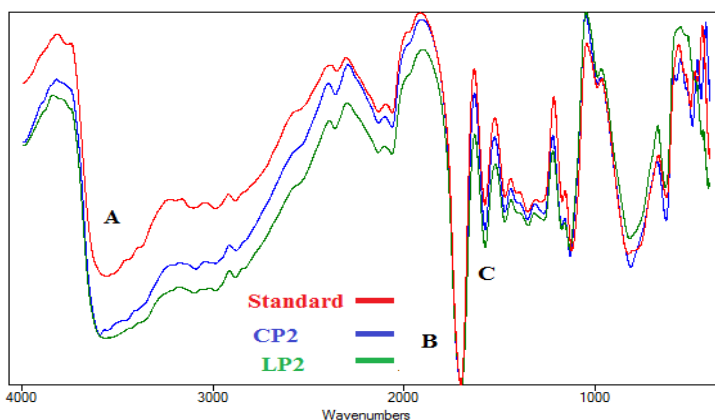


Fig. 8. FT-IR spectra representing a comparison between the standard highlight sample and the equivalent samples after exposure to clove oil and lavender oil vapors

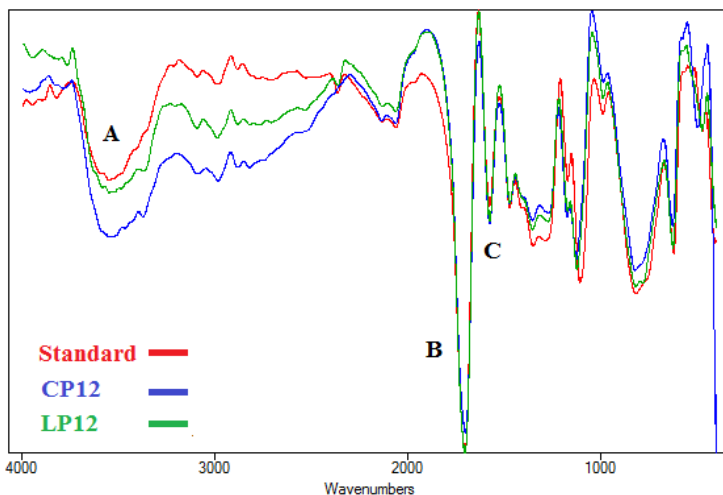


Fig. 9. FT-IR spectra representing a comparison between the standard midtone sample and the equivalent samples after exposure to clove oil and lavender oil vapors

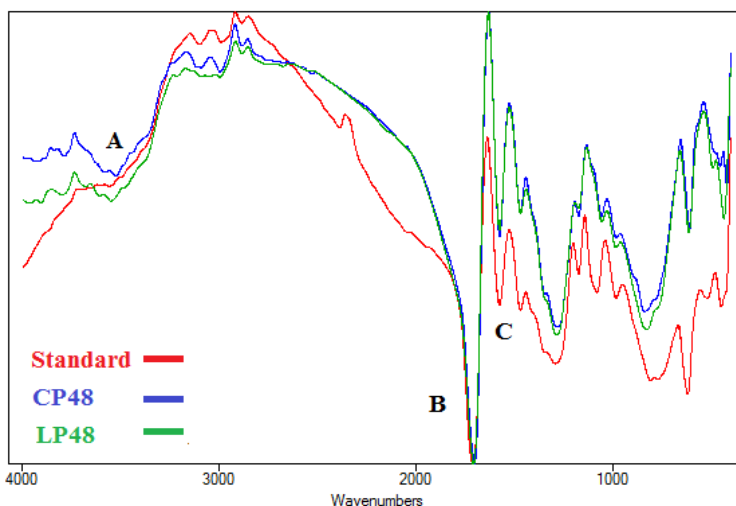


Fig. 10. FT-IR spectra representing a comparison between the standard shadow sample and the equivalent samples after exposure to clove oil and lavender oil vapors

Wood samples

FT-IR spectra representing a comparison between the standard sample and the equivalent samples after exposure to lavender oil vapors (Fig. 11) and clove oil vapors (Fig. 12) showed slight changes, most changes occurred in the chemical compounds in the area around (1000-1800 cm^{-1}). There are four major differences between the standard sample and the treated sample after aging. Increase of the intensity band of unconjugated C=O stretching (B) around (1730 cm^{-1}), explained by the existence of C=O Ester stretching from lavender oil, increase of the intensity band of C=O stretching (C) around (1650 cm^{-1}) characteristic of cellulose oxidation [71], decrease of CH bending band (D) around (1425 cm^{-1}), explained by the change of cellulose Crystallinity [72], and finally, a decrease of C-O-C stretching bands (E) around (1160 cm^{-1}), indicating an advanced breaking of cellulose chains and the occurrence of depolymerisation [73].

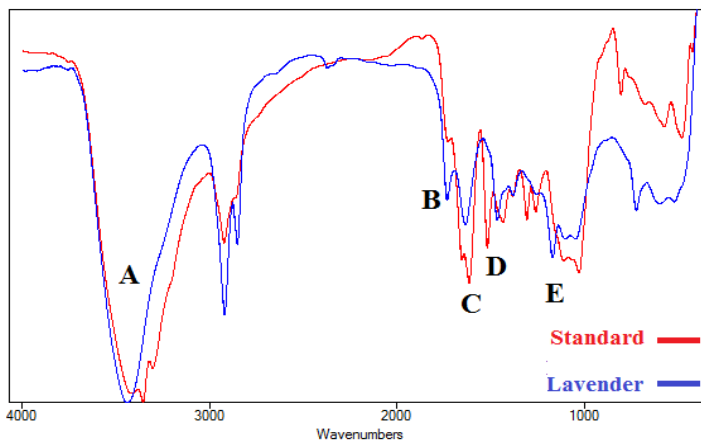


Fig. 11. FT-IR spectra representing a comparison between the standard sample and the equivalent samples after exposure to lavender oil vapors

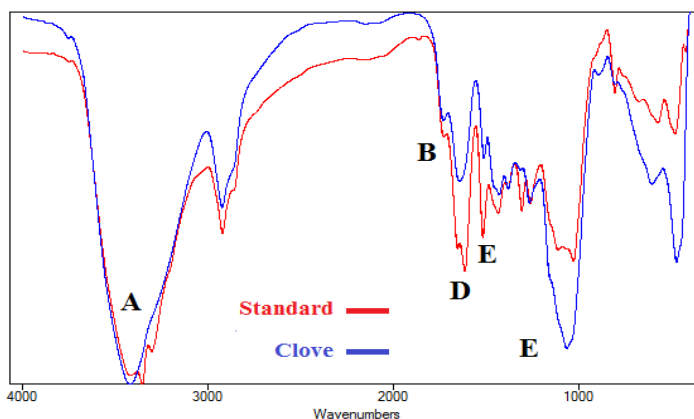


Fig. 12. FT-IR spectra representing a comparison between the standard sample and the equivalent samples after exposure to clove oil vapors

X-ray diffraction (XRD)

X-ray diffraction was used to measure Crystallinity index of cellulose in wood samples according to Segal equation:

$$CI (\%) = 100 \times \frac{I_{002} - I_{am}}{I_{002}}$$

where: CI - expresses the crystallinity index of cellulose, I₀₀₂ - express the maximum intensity of the crystallinity peak at (2θ = 22-24°) and I_{am} - represents the intensity of diffraction of the non-crystalline cellulose at (2θ = 18°) [74-77].

Comparison between the cellulose crystallinity of the standard sample and the aged-treated sample with lavender oil shows a slight decrease in crystallinity index of aged-treated sample (71.2%) compared to standard sample (73.2%). It also shows a slight decrease in the crystallinity index of aged-treated with clove oil (69.1%) compared to standard sample (73.2%) (Fig. 13).

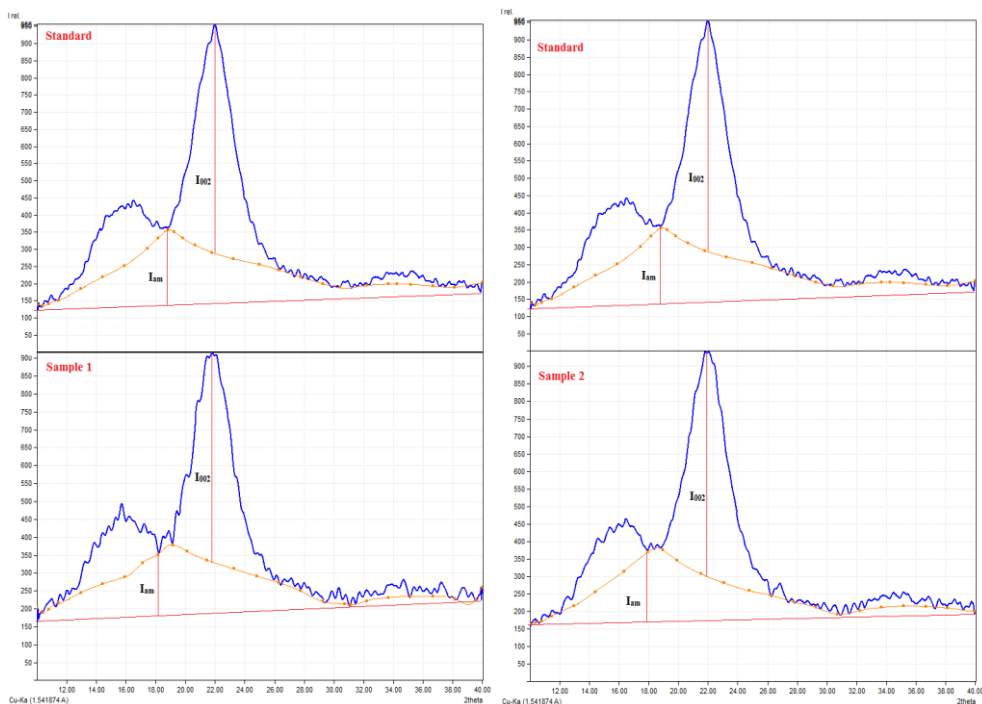


Fig. 13. X-ray diffraction pattern of standard sample and aged-treated sample (left) and X-ray diffraction pattern of standard sample and aged-treated sample (right)

Results are shown in Table 5.

Table 5. Crystalline index for wood samples posts treatment and post aging.

| Samples | I_{002} | I_{am} | crystalline index (CI) |
|----------|-----------|----------|------------------------|
| Standard | 668 | 179 | 73.2% |
| Sample 1 | 578 | 167 | 71.2% |
| Sample 2 | 643 | 199 | 69.1% |

Conclusions

The multiplicity of the components which makes up silver gelatin prints gives them a complex structure making them one of the most vulnerable objects held by museum, archives and libraries. The presence of a wood frame while advantageous for the preservation of photographs, it can also be problematic when used in an uncontrolled environmental condition; thus, presenting a new challenge for photograph conservators. Mold is a major issue for photographic collections due to their organic nature. In search for a safe and natural method to control mold growth, many scientists have studied the biocide activity of many essential oils, among which are clove and lavender essential oils. However, the possible use of these biocides in the control of biodeterioration of photographic collections has not been addressed. The significance of the study is that it evaluates the effect of both essential oils on the chemical, structural and aesthetic characteristics of silver gelatin prints and wood. Both tested clove and lavender essential oils used in vapor state have caused no to minimal change in the surface characteristics of the photographic and wood samples. Minimal color change has been observed and in all cases the change was within the permitted limit according to the international standards. With respect to the chemical changes which were studied using FT-IR spectroscopy, no to insignificant changes have been detected. Comparison between the cellulose crystallinity of the standard sample and the aged-treated samples with lavender and clove oils showed a

slight decrease in crystallinity index of aged-treated samples. Based on the obtained results, clove and lavender essential oil vapors are eco-friendly and effective alternatives to the globally used toxic fungicides and are safe for use on silver gelatin prints housed in wood frames.

Acknowledgments

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