INTERNATIONAL JOURNAL OF CONSERVATION SCIENCE
ISSN: 2067-533X Volume 15, Issue 1, 2024: 515-526 www.ijcs.ro
DOI: 10.36868/IJCS.2024.01.09

REMOVAL OF PRESSURE SENSITIVE TAPES FROM VINTAGE SILVER GELATIN PRINTS USING SELECTED GEL-BASED SYSTEMS: AN EXPERIMENTAL STUDY

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Abstract

Historical photographs play a significant role as records, documenting the various stages of the history of Egypt and reflecting our identity and social, cultural, and religious values. Silver gelatin prints were the dominant positive printing processes in the 20th century. One of the most common forms of damage found among photographic collections is tears, which may result from improper handling and misuse, inappropriate storage, disasters, and other factors. In the past, tears were mended using pressure-sensitive tapes by amateurs and conservators. Over time, these tapes caused damage to the photographic surfaces since they oxidised, forming stains that are difficult and sometimes impossible to remove. Recently, many gel-based systems have been developed for use in the conservation of different cultural and historical materials, particularly paper artifacts. The aim of this research is to evaluate the efficacy of Klucel G and agar, as gel systems that are favoured by conservation specialists, in removing pressure-sensitive tapes from the surfaces of silver gelatin prints, as well as to study their effect on the photographs themselves. Organic solvents were added to the gel systems, and invaluable old photographs were used for this study. After treatment, samples were subjected to artificial ageing at a temperature of 80°C and 65% RH. Evaluation of the selected treatments was carried out through visual inspection, microscopic examination, colorimetric measurements, and attenuated total reflectance Fourier transform infrared spectroscopy. Results showed that both gel-based systems with mixed organic solvents gave great results compared to the conventional methods in terms of tape removal. However, colour change was detected in the case of agar gel-treated samples. Agarose gel may be a more proper option.

***Keywords**: Silver gelatin prints; Degrading pressure sensitive tapes; Gel-based cleaning systems; Visual inspection; Microscopic examination; Colorimetric measurments; ATR-FTIR*

Introduction

Historical photographs are considered the national memory of the specific country they were created in; therefore, many governmental and even private institutions are keen to acquire and safeguard them, for they are an eyewitness to history and a true, living document of the past [1].

Silver gelatin prints are among the most popular positive photographic processes found in Egypt and worldwide. The silver gelatin process was introduced in 1874. Images are either produced by prolonged exposure of the light-sensitive materials (i.e., silver halide or silver halides) to light (i.e., printed-out silver gelatin prints) or using a developer (i.e., developed-out

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silver gelatin prints). Both types of silver gelatin prints have a similar layer structure; nevertheless, the morphology of the final image material is different [2].

A developed-out silver gelatin print consists of four components: the primary support, paper; the baryta layer; the binder layer, gelatin; and the final image material, metallic silver particles [3]. Silver particles are deposited as twisted strains known as filamentary silver in the case of developed-out silver gelatin prints [4]. The silver particles are embedded in the gelatin binder layer. The primary paper support provides physical support to the layers of the photograph. Until the early 20th century, paper was made from rags, and later, wood pulp was used. The baryta layer was introduced in 1885 to produce smooth and bright paper. It consists of barium sulphate, a white pigment, in a gelatin binder [5].

Silver gelatin prints are prone to damage by numerous internal and external factors (e.g., natural ageing, improper processing, improper levels of temperature and relative humidity, light and irradiation, air pollution, biological threats, poor handling and misuse, disasters, and others). These factors cause many forms of damage [6, 7]. Since photographs are in high demand due to their multiple applications, they suffer from the effects of improper handling and misuse [8]. Furthermore, they may also suffer from the consequences of unexpected disasters such as earthquakes. As a result, tears are among the most common forms of damage in photographic collections [9]. Previously, it was a common practice among librarians, museum curators, amateurs, and even conservators to mend tears using different types of pressure-sensitive tapes. At that time, they believed that tapes had the ability to maintain the physical stability of the torn item [10]. Pressure-sensitive tapes have a multi-layer structure composed of an adhesive (i.e., natural and synthetic rubbers, acrylic copolymers, or silicones); a backing (i.e., paper, fabric, cellophane, cellulose acetate, or oriented polypropylene); a release coat, ensuring easy unrolling of the tape; and a primer that enhances adhesion between the backing and the adhesive layer. Aged pressure-sensitive tapes can compromise the integrity and visibility of valuable images and, thus, valuable information (Fig. 1).

Fig. 1. Severely degraded pressure sensitive tape staining the surface of a silver gelatin print.
https://www.archivalmethods.com/blog/filmoplast-tapes/

The main issue with pressure-sensitive tapes arises from the use of masking tape and cellophane. The adhesives in both types oxidise over time. Tape removal at the initial stages of damage is possible and relatively simple. However, with the progression of oxidation, the adhesives change in consistency and colour (i.e., become sticky and oily and start to yellow). At this stage, tape removal is possible but difficult. Eventually, adhesives turn dark brown, become hard and brittle, and lose their adhesive power [11].

Removal of pressure-sensitive tape works to extend the lifespan of photographs and enhance their physical and chemical stability. However, it represents a great challenge to conservators since some removal techniques are aggressive and may cause severe damage to photographic surfaces. The success of tape removal depends on breaking the bond between the photographic surface and the adhesive. The selected tape removal technique should meet the following criteria: effective in tape removal; does not cause harm to the photograph; user-friendly; and eco-friendly. The selection of a proper removal technique depends on the condition and type of photograph, as well as the type of tape [12-14].

There are many conventional methods used to remove tape from the surfaces of paper-based artifacts. Mechanical methods include the use of scalpels and spatulas. While these tools are simple, available, and budget-friendly, they leave behind tiny scratches that disfigure the surface [15]. There are many organic solvents that can be used in the removal process (i.e., acetone, ethyl alcohol, toluene, benzene, xylene, etc.). The use of organic solvents provides a simple, rapid, and inexpensive solution, and it ensures easy application. However, organic solvents, whether in their pure form or as a mixture, can rapidly penetrate the surfaces of photographs and cause severe damage. Additionally, they are harmful to both the user and the environment [16-18]. Previous studies have pointed out the negative effects of using solvents on paper, particularly their effect on the tensile strength and pH value [19].

Recently, many studies have been conducted to develop new methodologies for dealing with this issue. Among these methods is the use of gel-based systems [11, 20, 21]. Gels are produced using two main components: a gelling agent and a solvent. Other additives may be added, such as solvents, to enhance the efficiency of the gel [22]. Based on previous studies, gel-based systems provide a simple, effective, and non-invasive solution for many of the challenges faced by conservators, reducing possible resultant damage through the controlled release of liquid cleaning agents onto the object. They are also user- and environment-friendly and provide easy application and removal [23]. Gels are divided into two categories, depending on the nature of their bonds: i) chemical gels: the bonds linking the subunits are covalent chemical bonds; ii) physical gels: the subunits are linked by weak secondary bonds (i.e., van der Waals interactions, hydrophobic, electrostatic, and hydrogen bonds). Physical gels (e.g., polysaccharides, cellulose ethers, or polyacrylic acid-based gels) tend to leave residues on the treated surface [24, 25]. To overcome the drawback of conventional fluid gels, so-called rigid gels, such as agar-based formulations, have been used since they do not require an aftertreatment due to their physical form and their limited adhesive strength [26]. Gels can be applied as solid pads or brushed onto objects in a semi-solid state [27].

In this study, two physical gels (i.e., Klucel G and agar gel) are studied to determine their efficiency in removing pressure-sensitive tapes from the surface of silver gelatin prints and to assess their long-term effect on the optical and chemical properties of the selected photographic process. An analytical approach was conducted during this study using visual inspection, digital microscopy, colorimetric measurements, and attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR).

Materials and methods

Sample preparation

Two vintage silver gelatin prints, dating back to the 20^{th} century, were selected for this study. Pressure-sensitive adhesive was applied to several areas of the selected photographs. The application areas were marked on a Mylar template. Four areas were selected for each of the three image tones found in black and white photographs: highlights, midtones, and shadow areas. Three areas are used for studying the efficacy of the selected gel-based systems, and one is for studying their effect on the optical and chemical properties of silver gelatin prints (Fig. 2).

Fig. 2. Shows the selected silver gelatin prints and gel application areas and the selected areas for measuring the optical and chemical properties before treatment, after treatment, and artificial ageing

Gel-based systems

Agar from B & V Laboratory Chemicals and Klucel G from CTS were used for the preparation of the gel-based systems. Agar is composed of polysaccharides and extracted from red algae species [28], while Klucel G is a non-ionic adhesive, hydroxypropylcellulose, which is soluble in water and alcohol [29].

Preparation of the gel-based systems

Agar-based gel was prepared by placing 2g of agar into a closed vessel filled with 100ml of distilled water. The vessel was then heated to 85ºC using a Bunsen burner. The resultant sol-gel was placed in three non-stick molds. For each mould, a measured amount of a solvent (i.e., ethyl alcohol, toluene, or a mixture of toluene and acetone) was added to the agarose sol-gel and stirred in. The agarose-based formulations were left to cool down into a rigid gel form of 0.5cm thickness [30, 31]. 3% of Kluce G in distilled water was prepared and poured into three molds. Similarily, a measured amount of one of the previously mentioned solvents was added to each mould [32] (Fig. 3).

Gel application

The resultant rigid agar gel was cut into the desired size in the form of pads (i.e., 1×1cm). Using the Mylar template, the pads were applied to the selected area and covered with a piece of Mylar and a piece of Plexiglas to provide some pressure. The contact time was 30 minutes for all samples. Klucel G gel was applied using a brush since it is a non-rigid gel. The contact time was also 30 minutes for all samples (Fig. 4) and (Fig. 5) [28]. Table 1 shows the numbering systems used for the treated areas in each photograph.

Fig. 3. Gel-based systems preparation steps

Table 1. Numbering systems for the treated areas

Sample number	Treatment	Image Tone	Sample number	Treatment	Image Tone
	First photograph			**Second photograph**	
Kg1	Klucel G + ethyl alcohol	Midtones	Mg1	Agar + ethyl alcohol	Dmax
Kg2	Klucel G + toluene	Midtones	Mg2	Agar + toulene	Dmax
Kg3	Klucel G + toluene and acetone	Midtones	Mg3	Agar + toluene and acetone	Dmax
Kg4	Klucel G + ethyl alcohol	Dmin	Mg4	Agar + ethyl alcohol	Dmin
Kg5	Klucel G + toluene	Dmin	Mg5	Agar + toulene	Dmin
Kg6	Klucel G + toluene and acetone	Dmin	Mg6	Agar + toluene and acetone	Dmin
Kg7	Klucel G + ethyl alcohol	Dmax	Mg7	Agar + ethyl alcohol	Midtones
Kg8	Klucel G + toluene	Dmax	Mg8	Agar + toulene	Midtones
Kg9	Klucel G + toluene and acetone	Dmax	Mg9	Agar + toluene and acetone	Midtones
Kg10	Standard	Dmin	Mg10	Standard	Dmax
Kg11	Standard	Midtones	Mg11	Standard	Dmin
Kg12	Standard	Dmax	Mg12	Standard	Midtones

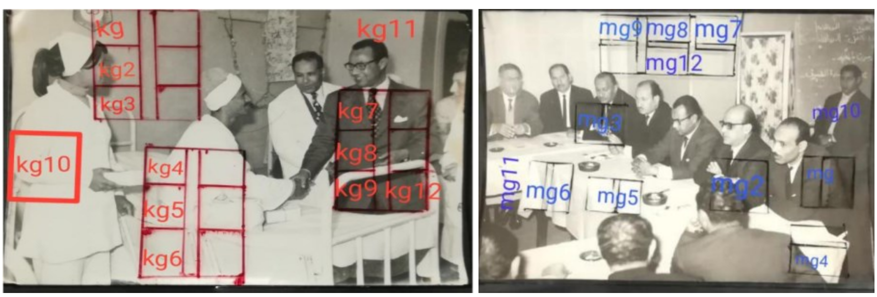

Fig. 4. Treatement application areas

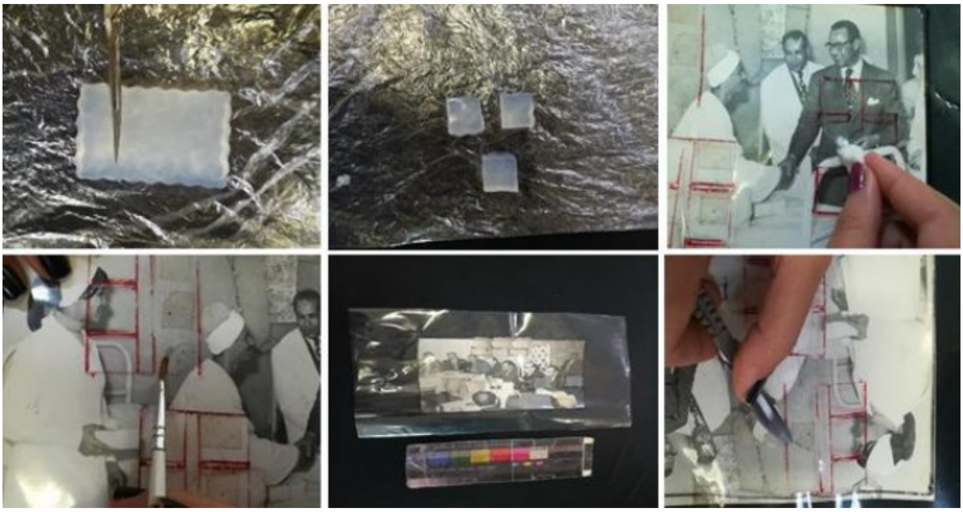

Fig. 5. The application of the gel-based systems on the photographic prints

Artificial aging

After treatment, the silver gelatin prints were aged at 80°C and 65% RH for 5 days, which is equivalent to the ageing of paper under natural conditions for 25 years. The ageing procedure was in accordance with the ISO 5630-3:1996 standard [33]. This process was performed in a BINDER drying oven with digital indicator, model no. 92403000002000, at the National Institute of Standards (NIS) in Cairo, Egypt.

Assessment methods

The efficiency of the gel-based systems in adhesive removal and their effects on the optical and chemical properties of silver gelatin prints were evaluated by visual inspection, microscopic inspection, colorimetric measurements, and attenuated Fourier transform infrared spectroscopy (ATR-FTIR).

Visual inspection

The visual changes resulting from the use of the tested gel-based systems were examined and documented post-artificial ageing.

Microscopic inspection

A ROHS Digital USB Microscope 1000X was used to monitor the optical changes resulting from the use of gel-based systems post-artificial ageing.

Colorimetric measurements

The change in colour was measured using a MiniScan Model No. EZ MSEZ0693. All samples were measured in a visible region with an interval of 10nm using a D65 light source and an observed angle of 10 degrees. The CIELAB colour parameters (L* a* b*) were used to express colour change, 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 -a* means blue [34]. All values of L*, a* and b* were obtained before treatment and after treatment and artificial ageing.

These data were used to calculate the total colour difference parameter (i.e., ΔE*). Each reading was the average of three measurements. This analysis was carried out at the Conservation Department, Faculty of Archaeology, Cairo University, Egypt.

Attenuated Total reflectance Fourier Transform Infrared (ATR-FTIR)

Spectra were obtained by using a Nicolet 380 FT-IR Spectrometer, in the frequency range of 4000–400 cm^{-1}. The ATR accessory was a Thermo Scientific ™ Performer Plate ZnSe Crystal with an angle of incidence of 45°. The diamond has an active area of 1 mm in diameter, and the depth of each scan was approximately 2 microns below the surface. No preparation of the samples was necessary. The analysis was performed at the National Institute for Standards in Cairo, Egypt.

Results and discussion

Visual inspection showed no changes in all treated aged samples.

Microscopic inspection

Surface inspection showed good results for all aged-treated samples in both photographic prints. However, samples treated with Klucel G gave better results, particularly sample Kg1 that was treated with Klucel G in ethanol. Klucel G gel was removed after 30 minutes, and the remaining adhesive was efficiently mechanically removed using a scalpel. On the other hand, samples treated with agar gel pads prepared with ethyl alcohol (i.e., Mg1, Mg4, and Mg7) gave the best results (Fig. 6).

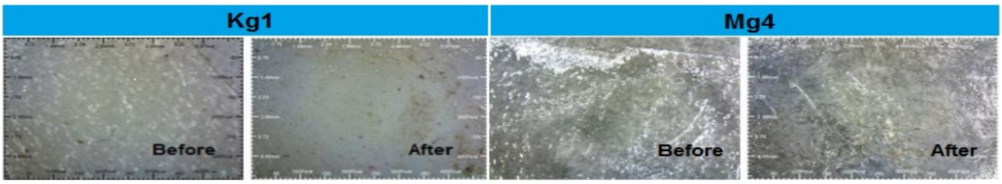

Fig. 6. Microscopic inspection of the aged-treated photographic surfaces, samples Kg1 and Mg4, using a USB digital microscope

Colorimetric measurements

The total colour difference (ΔE*) is a value useful as an indicator of the difference between the sample and the reference. In literature, chromatic variation of 2-3 can be considered noticeable by the human eye; however, it is clearly lower than the threshold limit required (ΔE* = 5) for the maintenance and restoration of historical surfaces [35]. The test results were illustrated using the Colour Math application (Fig. 7).

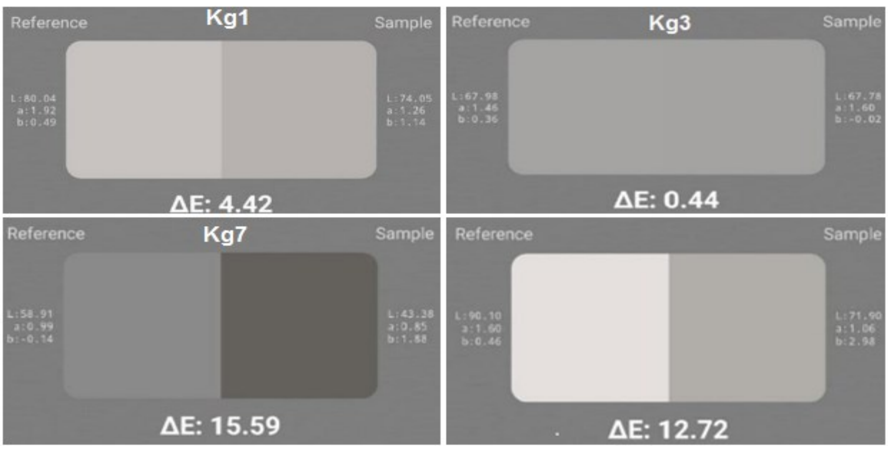

Fig. 7. Illustrations of color change due to the application of selected gel-based systems and artificial aging using Color Math application

Results listed in table 2 showed minimal change (i.e., ΔE* below 5) for all aged, treated samples, excluding samples Kg7 (i.e., ΔE* = 15.59), Mg9 (i.e., ΔE* = 12.72), Mg2 (i.e., ΔE* = 22.84), and Mg5 (i.e., ΔE* = 17.33).

Table 2. Colorimetric measurements (L*, a*, b* and ΔE*) for tested samples

	L*	a*	b*	ΔE*	L*	a*	b*	ΔE*
Sample	**Kg1**				**Kg2**			
Untreated	80.04	1.92	0.49	**4.42**	67.01	1.49	0.36	**3.62**
Aged treated	74.05	1.26	1.14		71.64	1.41	0.55	
Sample	**Kg3**				**Kg4**			
Untreated	67.78	1.60	-0.02	**0.44**	78.38	1.56	-0.47	**4.04**
Aged treated	67.98	1.41	0.36		84.25	1.51	0.05	
Sample	**Kg5**				**Kg6**			
Untreated	79.44	1.39	-0.42	**3.90**	80.32	1.95	-0.21	**1.78**
Aged treated	85.13	1.26	0.20		82.31	1.51	0.72	
Sample	**Kg7**				**Kg8**			
Untreated	58.91	0.99	-0.14	**15.59**	45.17	0.51	1.59	**3.98**
Aged treated	43.38	0.85	1.88		40.87	0.62	2.06	
Sample	**Kg9**				**Mg9**			
Untreated	41.13	0.70	1.57	**1.51**	90.10	1.60	0.46	**12.72**
Aged treated	39.47	0.55	1.88		71.90	1.06	2.98	
Sample	**Mg2**				**Mg5**			
Untreated	58.41	1.19	1.56	**22.84**	98.31	0.98	0.35	**17.33**
Aged treated	34.91	0.53	1.16		72.12	1.36	2.72	

Attenuated Total reflectance Fourier Transform Infrared (ATR-FTIR)

Results show the two most prominent absorption bands of gelatin: the amide I band (1600–1700cm^{-1}) and amide II (1500–1600cm^{-1}). Amide I is presided over by the C=O stretching vibrations of the peptide linkage (70-85%) 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 (18-0%) and C-C stretching vibrations (about 10%). Slight changes have been observed in the OH stretching band, the amide I band, and the amide II band. Some samples showed an increase in the intensity of the OH stretching band, indicating the occurrence of protein hydrolysis [36] (Fig. 8).

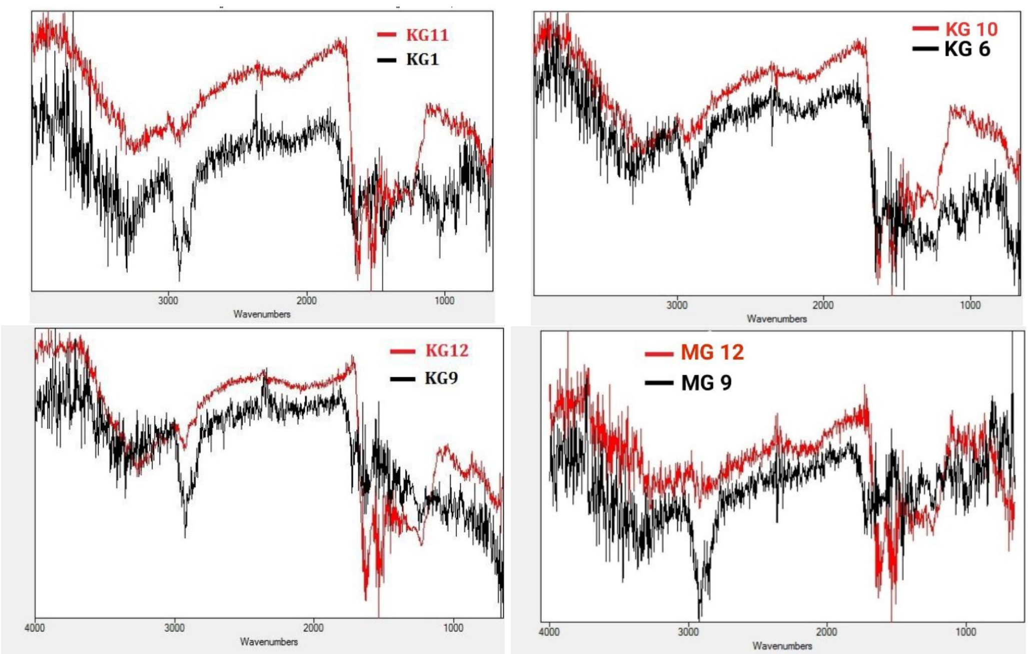

Fig. 8. ATR-FTIR spectra of aged gel-treated samples compared to the control samples

Conclusions

Based on the results of this study, both gel-based systems with mixed organic solvents gave great results compared to conventional methods in terms of tape removal. However, colour change was noticeable in the case of the aged agar gel-treated samples, while colour change was within the allowable limit in the case of the aged Klucel G gel-treated samples. In terms of preserving the colour of photographic surfaces, agarose gel is may be a more proper option since it is obtained from agar through a complex purification process. Agarose gel has been tested on albumen prints, a very sensitive photographic process, and great results have been yielded. ATR-FTIR analysis results showed a minor change in the chemical properties of the aged-treated samples compared to the control samples. Nevertheless, being a non-rigid gel, Klucel G may promote microbiological damage in the presence of conditions that are favourable for the growth of microorganisms. More studies are required to cover this area. Other types of gel-based systems such as Carbopol, gellan gum, and nanorestore gel incorporating different additives (i.e., green solvents, enzymes, nano materials) may be included in future studies.

Acknowledgments

The authors would like to thank the anonymous reviewers for the time they spent on the reading of our manuscripts and for their valuable and constructive comments. We are grateful to Prof. Mahmoud Morsy and Mrs. Rasha Sadek at the National Institute of Standards (NIS) in Cairo, Egypt for their assistance in the ATR-FTIR spectroscopy. We also thank Prof. Mona Fouad Ali at the Faculty of Archaeology, Cairo University, Egypt for the invaluable advice.

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Received: March 28, 2023
Accepted: January 24, 2024