



Assessment of Texapon N 70 Microemulsion Confined in PVA/ Gel for Removal of Wax Stain from Marbled Endpapers Dating Back to the 19th Century

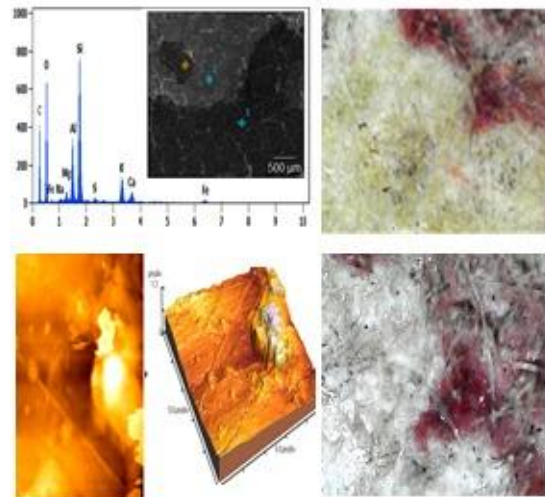
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HIGHLIGHTS

- Marbled endpapers of a printed book explaining agriculture written in French and dating back to the 19th century was used.
- Multi-analytical protocol was carried out to determine the gel cleaning performance and efficiency.
- The SEM examination of paper samples before cleaning illustrated the topography of wax layer that covered the paper surface, making it difficult to distinguish the fibers morphology.
- The results demonstrated that Texapon microemulsion could be safe and effective for removing wax stain from marbled paper.

GRAPHICAL ABSTRACT



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ABSTRACT

Wax stain on paper artwork is one of the most chronic and complicated problems during conservation treatments. Understanding changes to the marbled paper samples during cleaning with gel-based microemulsion is one of the most important issues for conservators of painted works on paper. This research aims to evaluate the effectiveness of Texapon N70 confined in PVA/ gel for removing wax stains on marbled paper and stopping wax penetration into the paper structure. Characterization procedures before and after cleaning were performed using a digital microscope, SEM microscope for morphological study, and

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FTIR spectroscopy to detect the chemical structure change. The roughness of the samples was measured using an atomic force microscope (AFM). The results of the test demonstrated that the substance could be safe and more effective for removing wax stains from marbled paper. The used gel-based Texapon microemulsion was capable of removing the wax stain from marbled paper, and the results of the test demonstrated this material's ability to safely and effectively remove wax stains from marbled paper.

1. Introduction

Ebru is the traditional Turkish art of creating colorful patterns by sprinkling and brushing color pigments onto a vessel of oily water and then transferring the patterns to paper sheet. Marbling designs have variable effects including flowers, foliage, and ornamentation, and are used for decoration in the traditional art of calligraphy. The main technique of Ebru art has been based on use of natural methods to extract colors from natural pigments which are then mixed with heavier liquid (size) made by mixing gum tragacanth or Irish moss plant (carrageenan) with water, and poured into a shallow rectangular vessel before sprinkling and brushing the colors onto a preparation of condensed liquid, where they float and form swirling patterns [1,2].

Ebru painting is an aqueous art used to produce mesmerizing works, and was popular in Turkey and Central Asia. It is a kind of art created by transferring designs and patterns onto sheets of paper, fabric, and other canvases. Ebru has a long history and can employ different types of materials compared to other decorated papers; however, the basic idea remains mostly the same across materials and techniques [3].

Cleaning of ancient paper is one of the most critical steps during conservation treatment. It is used to improve the optical qualities of a graphic work and for the removal of degradation products, external contaminants and salts, which can promote the degradation of cellulose (hydrolysis & oxidation). Nevertheless, washing treatment usually involves a substantial impact on the original morphological structure of paper and can sometimes be dangerous for water sensitive inks and pigments [4,5].

Wax stains can deteriorate or disfigure paper. Wax consists of long-chain aliphatic molecules from 20 to 50 carbon atoms. A nonpolar solvent, e.g., dodecane, may be uti-

lized to solubilize the wax. Some alkaline solutions can be employed for cleaning the surface from wax stains, which should be handled using dilute acidic solutions to neutralize the alkaline effects. Furthermore, traditional cleaning procedures utilized organic solvents. This cleaning issue can encourage disfiguration due to the unexpected action of the solvent, which has a great role in the penetration into the porous structure [6]. Conservators are familiar with different methods for wax stain removal, e.g., mechanical, solvent, solvent gel, and hydro-gel cleaning for media sensitive to water. Organic solvents that can be employed to conserve paper include N-methyl-2-pyrrolidone as a solvent for old flour paste, carbon tetrachloride and methanol (for greasy stains); hexane, toluene, and ethyl acetate for tape residues removal; 1, 1, 1-trichloroethane for wax and resins. Using solvents may result in unwanted effects, including pigment alternation, swelling of the binding media, transporting the dissolved material in the porous matrices, or lacking control in the cleaning process, causing the removal of the patina, i.e., the original layer that can be a historical part of the artifact. Furthermore, solvents can cause health issues for the operator. Using organic solvents implies many undesired issues, primarily associated with safety risks and the potential modification of the artifact. Several organic solvents are used to clean the surfaces of artworks on paper. These solvents may have a dramatic effect on paper support, causing smearing or bleeding of colorants on paper [7, 8]. It is important to use the procedure that ensures positive issues for wax removal without affecting the object or leaving harmful residues. The use of organic solvent in the form of a gel network enhances the controlled removal of wax stains with no interaction with the water sensitive supports. This nondestructive method enables the complete recurrence of artwork [9].

Researchers have recently adopted chemical and hydro gels to remove sticky tapes or old varnishes from artworks. These gels are recommended as effective tools to remove the contaminant from paper supports due to the controlled water release. Various confining systems, such as microemulsions, discussed the cleaning of paper artworks. The removal of wax stains from porous support is particularly difficult for conservators in the field. Additionally, the lack of these methods can be increased by nanostructured fluids, such as micellar solutions and microemulsions [10].

In this study, a Texapon microemulsion-confined in PVA gel was used for removing wax stains from a historical marbled paper. A multi-analytical protocol was performed to determine the gel cleaning performance and cleaning efficiency.

2. Materials and Methods

2.1. Preparation of micro emulsion confined in PVA gel

A homogenized aqueous solution of PVA with 15 % concentration was prepared by heating to 95°C with mechanical agitation for nearly three hours. After cooling to room temperature, glutaraldehyde was added as cross linking agent (10 µL) and a dilute solution of hydrochloric acid (10 µL) was added to the PVA solution and introduced to a centrifugal mixer. After that, the mixture was poured into a mold for gelation. The prepared gel was heated under 90°C for three minutes, cooled to room temperature, left to stand for two hours, and frozen at -20 °C for eight hours.

Texapon - N70, which is used for the manufacture of technical cleaning agents, contains reverse osmosis water, sodium laureth sulfate, anionic surfactant, Dioxan 1.4(ppm), sodium sulfate 0.1%, and color Hazen (20%). The pH of the microemulsion was in the range between 7 to 9. The gel matrix was immersed in texapon to upload the microemulsion.

2.2. Samples

Marbled endpapers of a printed book explaining agriculture written in French and

dating back to the 19th century were used in this study.

2.3. Cleaning process

The cleaning test was carried out by directly applying the microemulsion on stained paper. The wax removal test was performed by moving cotton swabs in a circular motion several times until the completion of the cleaning procedure and then using a cotton swab to remove swollen wax from the surface.

2.4. Methodology for the evaluation of the cleaning performance

A multi-analytical protocol was carried out to determine the gel cleaning performance, efficiency, as well as the percentage of the remaining solvent after treatment. Non-destructive methods were performed on marbled paper samples in the pre-and post-cleaning treatment.

2.4.1. Digital microscope

A professional portable mobile LCD digital microscope with MP digital zoom 100-1000 to detect the morphological changes of the treated samples due to the cleaning process was used in this study.

2.4.2. AFM microscope

Atomic Force Imaging (AFM) was performed to investigate the degradation of the fibers on the paper surface and provide qualitative and semi-quantitative data on aging and deterioration. AFM topographies of the stained paper samples were considered, and those of the original paper sample (control) were compared to the cleaned sample. The roughness of the samples was measured by an AFM autoprobe cp-research head made by Thermo microscope and run in the contact mode through the Silicon Nitride Probe model MLCT manufactured by Bruker. Moreover, ProScan 1.8 helped in controlling the scan parameters, and IP 2.1 helped in image analysis.

2.4.3. Color spectrophotometer

An Optimatch 3100® manufactured by the SDL Company at the National Institute for Standards (NIS), Egypt, was used to measure

the color changes in the treated and untreated samples. The CIE L, a, b color system was utilized for assessing the efficiency of micro-emulsion and detecting any color changes due to the cleaning procedure.

2.4.4. ATR-FTIR Spectroscopy

FTIR analysis was performed to monitor the cleaning effects and detect the presence of gel on treated samples. To determine the post-cleaning presence of wax, spectroscopic analysis was carried out on the cleaned samples. The Nicolet 350 ATR-FTIR at NIS, Egypt, was used to analyze untreated samples and the samples after cleaning procedures. The spectra were collected with the 4 cm^{-1} resolution.

2.4.5. Scanning Electron Microscope with EDS Spectroscopy

SEM-EDS was employed for the elemental composition of paper samples before and after wax removal. Paper samples were examined uncoated, using a variable pressure SEM (FEL Quanta 3D 200i Edx / Thermo fisher pathfinder) with the following conditions, including low vacuum for acceleration voltage 20.0~30.0 kv by large field detector with 15~17 mm working distance.

3. Results and Discussion

3.1. Digital microscope examination

The digital microscope was used to detect the changes in the chromatic layer on marbled papers, such as fading or darkening of the pigment layer, and to register the immediate effects on the morphology of the treated samples due to the cleaning process (as shown in (Fig. 1).

Paper samples were assessed before and after cleaning. After cleaning, noticeable changes were registered; the wax was removed completely (Fig.2B & D), and the colors became brighter compared with the untreated samples, suggesting the efficiency of the cleaning procedure. The removal of wax stain from the paper might be due to the 1, 4- Dioxane and the nonionic surfactant, which represented an improvement in the surface quality.

3.2. Atomic Force Microscope (AFM)

Qualitative and quantitative assessments have been detected using Atomic Force Microscope (AFM). AFM 2D and 3D images in Fig.3 (A) show color contrast as an indication of the existence of stain material with non-uniform distribution on the surface of the paper sample, whereas the images of cleaned and control samples in Fig.3 (B, C) demonstrate the uniformity and smoothness of the surface profile which prove the cleaning efficiency. For more illustration, line profiles of the three samples are presented in Fig.4. Control and cleaned line profiles show a uniform change in height in contrast with the line profile of stained sample which cause a sudden change in height estimated by 2496.0 nm.

The effectiveness of cleaning procedure could be confirmed by quantitative measurements of surface roughness parameters [The roughness average (Ra), the root mean-squared average (Rq), and the maximum profile valley depth (Rp-v)]. Surface roughness parameters of stained, cleaned, and control samples are listed in Table 1. It was noted that the stained sample had the highest roughness parameters values because of the non-uniform distribution of wax stain on the surface [11]. Cleaned sample showed a significant decrease in surface roughness parameters values where Ra and Rq are comparable to the surface roughness parameters of the control sample. This decrease can be attributed to the success of cleaning treatment in removing the wax stain from the surface.

3.3. Change of color

From the data of lightness value (L^*) presented in Table 2, the treated samples with Texapon N70/ PVA achieved tolerable color difference. The results demonstrated that a Texapon 70 /PVA gel increased the L^* value that approached the L^* of the control, indicating the efficacy of removing wax stains by the gel, the CIE L^* value which refers to the lightness parameter confirms color enhancement of treated samples.

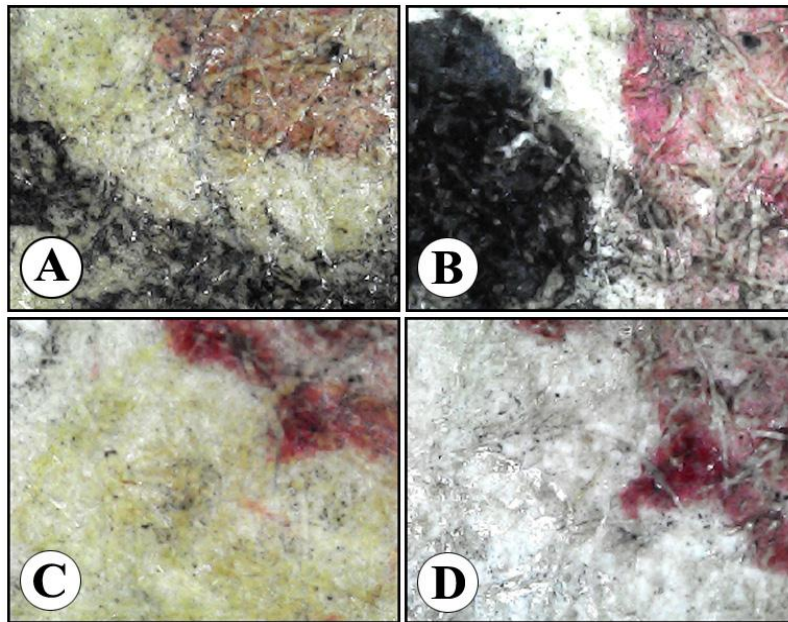


Fig. 1. Digital microscope micrographs of paper surface of marbled paper of the 19th century: (A) Stained with wax, (B) The sample after the removal of wax using PVA/microemulsion, (C) The surface before cleaning, (D) The same surface after cleaning.

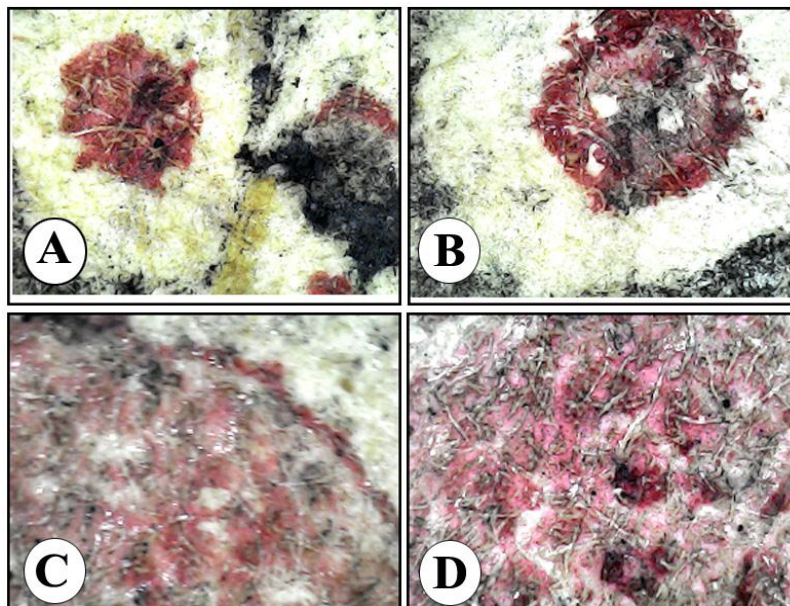


Fig. 2. Digital microscope micrographs of the chromatic layer represents wax layer deposited on paper surface: (A) Stained with wax, (B) remarkable changes were observed due to wax removing, (C) The stained sample, (D) The same spot post-treatment.

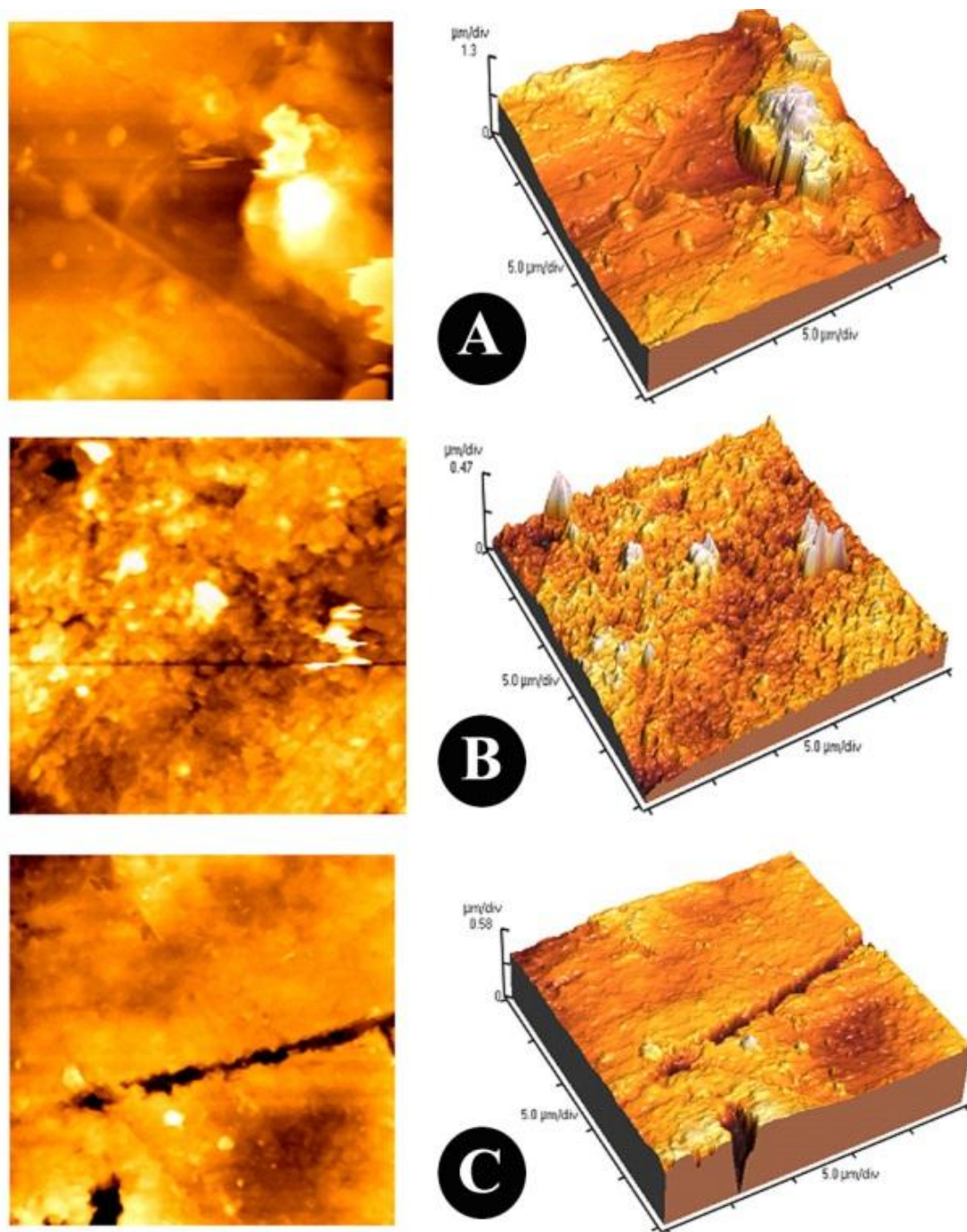


Fig. 3. 2D and 3D Atomic Force Microscope images (A), (B) and (C) of stained, cleaned, and control samples, respectively.

Concerning the CIE a^* value (red to green parameter), the color tended to be green, which became more in stained samples. The

cleaned sample had less green than the control sample.

Table 1. Surface roughness parameters (Ra, Rq, and Rp-v). of control stained and cleaned samples.

Sample	Ra nm	Rq nm	Rp-v (of line profile) nm
Control	42.9	65.9	97.5
Stained	473.4	592.4	2496.0
Cleaned	54.4	71.9	243.0

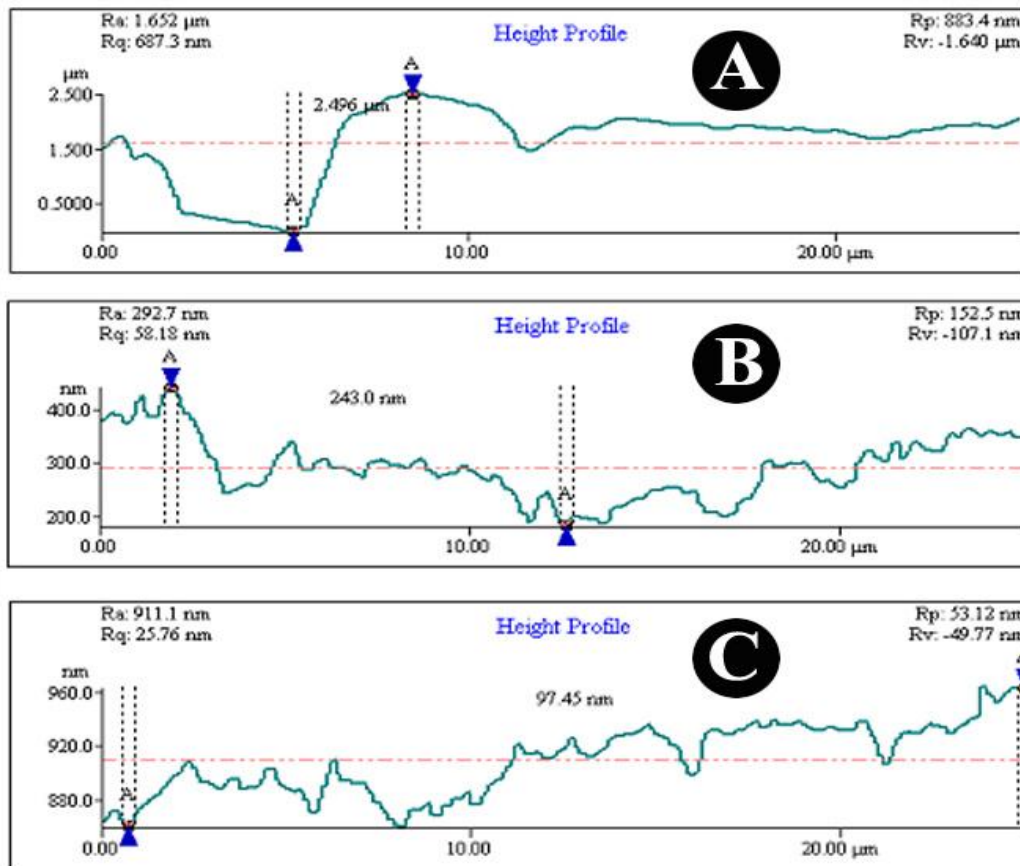


Fig. 4. Line profile (A), (B), and (C) of stained, cleaned and control samples respectively.

Regarding the CIE b^* values (yellow to blue parameter), the treated samples tended to be near blue except for the control sample. The color difference values (ΔE) recorded after the stain removal 5.1, which proved that the microemulsion treatment caused a perceptible difference detected by human eye. According to the previous studies that discussed the measurement of color change, it could be concluded that the measurement of lightness could give a perfect indication of the behavior of the paper in artificial aging,

denoting the performance of the cleaning treatments [11, 12].

Table 2. L^* , a^* , b^* and ΔE values before and after cleaning

Samples	L^*	a^*	b^*	ΔE
Stained	56.58	1.19	7.08	8.6
Treated	61.52	0.53	6.19	5.06
Control	64.76	1.69	9.91	----

3.4. ATR-FTIR Spectroscopy

FTIR spectra were collected before and after the cleaning process (Fig. 4). The spectra revealed characteristic absorption bands related to cellulose molecule; O-H stretching band, C-H stretching vibration band, C=O stretching band and C-O stretching band. There was no change in the chemical structure of the paper after cleaning; typical characteristic bands were detected in the untreated and treated samples. The bands at 1420 cm^{-1} corresponded to the OH bending of the cellulose molecule, and those at 1050 cm^{-1} and 1150 cm^{-1} corresponded to $\text{C}_2\text{O}_2\text{H}$ stretching [13, 14]. Moreover, calcium carbonate was detected at around 1400 cm^{-1} and 875 cm^{-1} . The ATR-FTIR revealed all the signals attributed to the aliphatic hydrocarbon chains related to waxy stains. The characteristic bands of wax could be noticed at 2912 cm^{-1} , 2840 cm^{-1} , 1474 cm^{-1} , 1465 cm^{-1} , and CH_2 stretching at 731 and 721 cm^{-1} . In addition, the absorption bands could be detected at 1730 cm^{-1} and 1172 cm^{-1} [15, 16]. These results were caused by the presence of carboxyl groups of fatty acids in beeswax's

chemical composition. After cleaning, the results illustrated that the substance efficiently removed the stains in all treated samples. Thus, the characteristic bands of wax and carbonyl group stretching at 1742 cm^{-1} disappeared. The difference between the pre-and post-cleaning spectra appeared in the position of the bands relevant to the carbonyl group (C=O). These could be due to the hydrolysis of the hydrocarbon esters, which were converted to carboxylic acids. The spectral region detected between 1700 and 1550 cm^{-1} is attributed to the absorption bands of cellulose byproducts; containing aldehydes and carbonyl groups (C=O)[17]. The spectra of stained sample showing the intensity of the bands in the 1700 to 1550 cm^{-1} are considerably smaller compared with untreated samples [18, 19]. An increase in the OH stretching region is noted, which proved the efficiency of Texapon for removing of wax stain. Additionally, the bands related to cellulosic material were better identifiable.

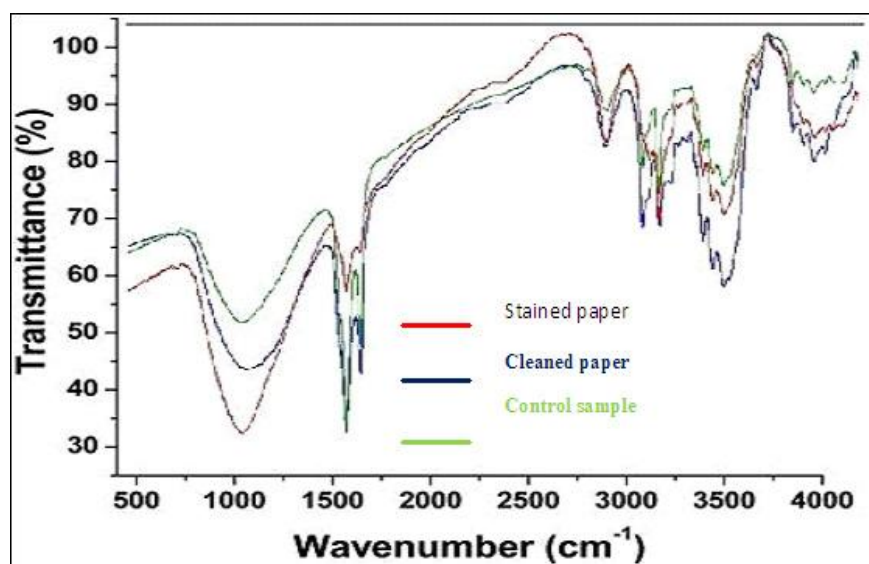


Fig. 5. Pre- and post-cleaning ATR-FTIR spectra of the paper sample; the characteristic bands of wax are noticed at 2912 cm^{-1} , 2840 cm^{-1} , and 1474 cm^{-1} .

3.5. Scanning Electron Microscope with EDS Spectroscopy

SEM examination enables us to evaluate clearly the effects of cleaning procedures on the paper surfaces. The SEM observations of paper samples before cleaning illustrated the topography of wax layer that covered the paper surface, so it was difficult to distinguish the fibers morphology, and the stained paper sample showing topographic variations on the paper surface, where the surface was covered with a rough layer of wax stain Fig. 5. The topography of the surface after wax removal became more representative and a remarkable improvement was observed on the paper surface due to wax removal Fig. 6.

SEM/EDS analysis was used to reveal the elemental composition of marbled paper. It was carried out before and after cleaning in order to detect the changes in the paper structure. The EDX spectrum demonstrated the high contents of Al (up to 5 wt %) and Si (up to 4 wt %) and the presence of Fe and S (up to 1.4 & 1.3 wt %). The paper sample contained K, Mg, and Na in small percent, along with high C and O contents. The presence of salts could be due to paper-making processes, such as adding fillers or sizing materials, while minerals were related to the chromatic layer. Aluminum and silicon ions probably came from kaolin-based paper fillers; sodium and magnesium ions from cellulose pulp preparation water; and the ferric ion came probably from rust contamination present in the water. During the manufacturing process, additives, such as ion salts, e.g. clay or carbonate salts, were used as fillers. Additionally, sizing substances or traces, like minerals in the case of chromatic area, were added to the paper to obtain certain patterns [20, 21,22]. The presence of metal salts might be due to many reasons, resulting from the use of contaminated water with heavy metal or the use of rusty molds.

The major element detected in the stained sample was carbon, which arises from the presence of wax layer on the paper surface that clearly indicates the main composition of beeswax, besides a small ratio of oxygen (O), aluminum (Al), and silicon (Si) Fig.7 A&B [23,24,25]. A significant difference was observed between the elemental compo-

sition when comparing the results for the stained sample and the sample after removing the wax layer, and the EDX results became more similar to the elemental composition for paper composition which proves the performance of cleaning procedure.

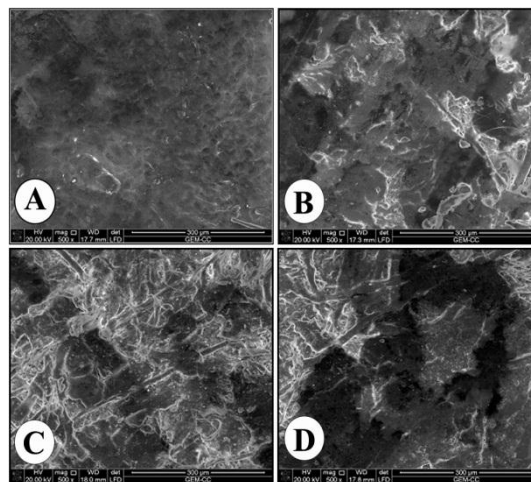


Fig. 6. SEM micrographs of a paper sample, A, B show stained paper sample showing topographic variations on the surface, where the surface was covered with a rough layer of wax stain, while C, D show the surface after wax removal, a remarkable improvement can be observed on the paper surface.

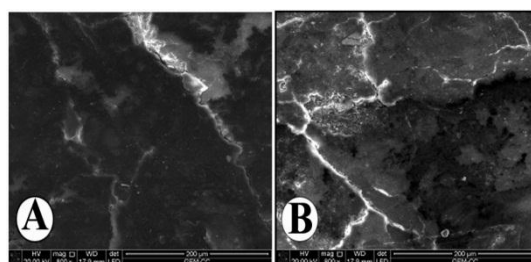


Fig. 7. SEM micrographs (A & B) show the chromatic layer that appeared with fine cracks, two shades are observed which indicate light and dark colors.

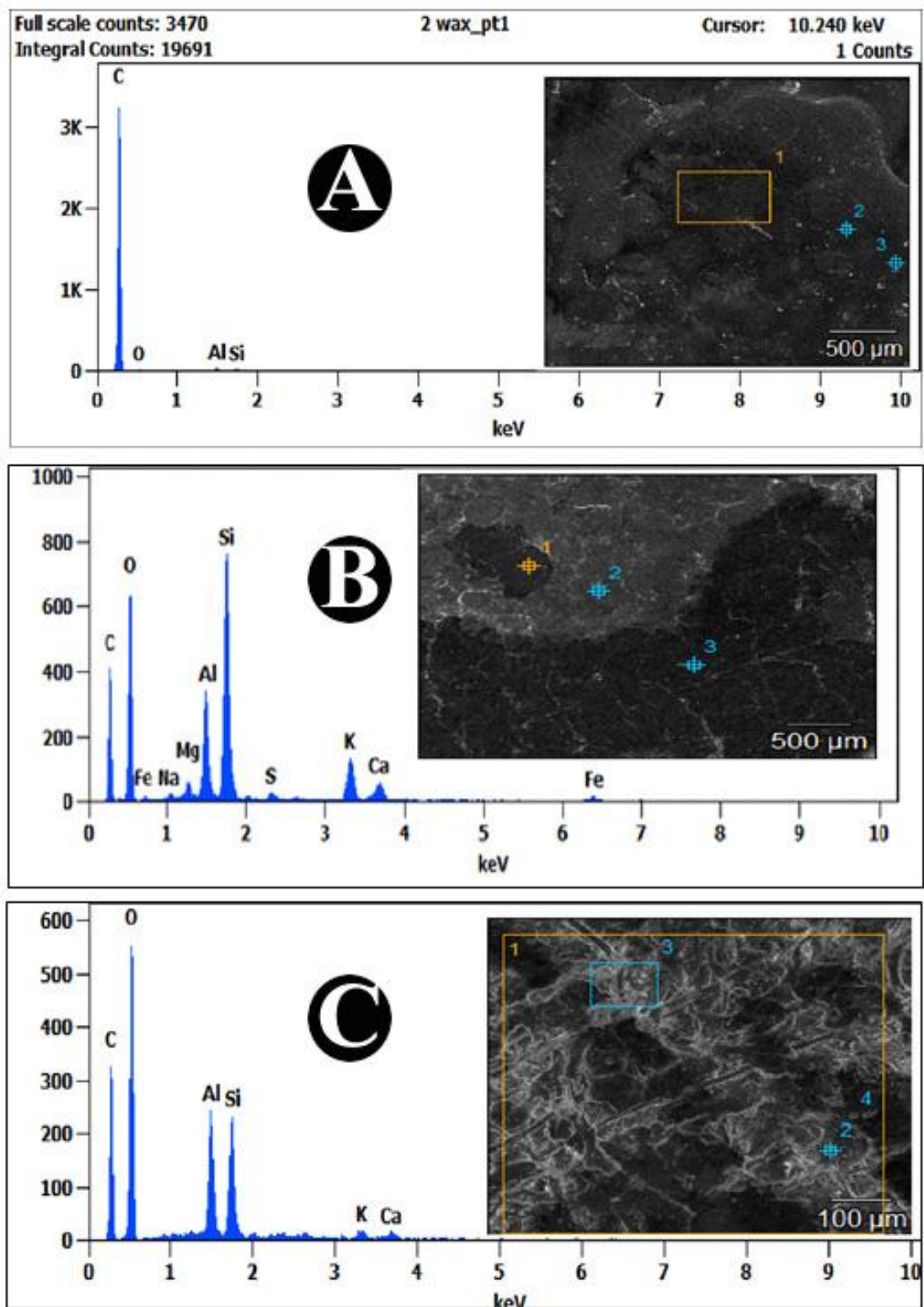


Fig. 8. EDX spectra of marbled paper sample dated back to 19th century (A) represents the stained sample, while (B & C) illustrate the cleaned sample.

4. Conclusion

The study aimed to evaluate a new micro-emulsion confined in poly vinyl alcohol (PVA) gel for the surface cleaning of marbled paper containing solvent and water-sensitive pigments.

ATR-FTIR and SEM investigations allowed monitoring the efficacy of the PVA gel/microemulsion residues after cleaning treatment. AFM imaging of paper samples after the cleaning procedure showed an improvement in the surface topography compared with the stained sample, which represented high roughness parameters. The diffusion of the microemulsion in the gel matrix during cleaning test is granted by the osmotic balance in the network.

Cleaning procedure on marbled paper showed that Texapon allowed swelling of the wax layer, which can be removed using gentle cleaning with cotton swabs. The results of the test demonstrated that the substance could be considered safe and effective for removing wax stains from marbled paper.

The results revealed the good performance of Texapon 70 based PVA gel for the removal of wax stains, avoiding the problems of wax redistribution, and ensuring safety for artworks and the conservators.

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