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Case study

# Use of water - white spirit microemulsion to clean a white monochromatic painting by Gilda Azevedo

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ABSTRACT

Cleaning a painting is a process that requires knowledge of its materiality, integrity, and solvent systems. Aqueous solvent systems are a sustainable alternative, but their use must consider the potential harm that can arise in the process- such as swelling and leaching of layers underlying the dirt. Modern oil paintings made from the twentieth century onwards may be sensitive to aqueous solvents and suffer such damage. In this work, the cleaning of a white monochromatic oil painting by artist Gilda Azevedo was carried out with a microemulsified system of water in white-spirit using Tween 80 and ethanol as surfactant and cosurfactant, respectively. The microemulsified system and emulsions are the result of a ternary pseudodiagram where water, white-spirit, and surfactant/cosurfactant are carefully rationed. The emulsions obtained were characterized by measurements of conductivity and Dynamic Light Scattering. The artwork was examined by infrared absorption spectroscopy and X-ray fluorescence spectroscopy. Cleaning tests were firstly performed with emulsions and microemulsion and the removal of pigments was qualitatively analyzed by X-ray fluorescence spectroscopy. It was observed that the microemulsified system was an efficient cleaner while causing less removal of pigments compared to emulsified systems and, thus, chosen for cleaning. This cleaning efficiency was attributed to the use of water-white-spirit mixes capable of interacting with hydrophobic and hydrophilic substances in dirt. The lower pigment removal was ascribed to the dynamic percolation system and the nanometric size of the microemulsions.



## 1. Introduction

Artworks and other cultural assets are subject to the action of temperature, umidity, soil etc. [1,2]. Deterioration can make it difficult to read and appreciate the artwork, making it necessary to remove foreign or degraded materials from its surface. Cleaning is a common practice in the field of art conservation and it must be executed carefully, involving knowledge related to the materiality and integrity of the work, as well as the cleaning agents. In the case of paintings, their aesthetic unity, their technique (acrylic, oil, etc.), the presence or absence of varnish, and their conservation state amongst other characteristics determine the cleaning process. The choice of cleaning materials must be meticulous since the application of solvents and solvent systems can also undesirably damage the art itself leading to loss of gloss and surface character, paint embrittlement, etc. [3].

Given the chemical nature of works of art components, organic solvents are widely used in their cleaning. These, however, pose problems of toxicity and ecotoxicity, only avoided by replacing them with less aggressive substances as prescribed by Green Chemistry criteria [1,4]. Water is one such substitute which can either be used alone or emulsified with organic solvents, mixed with chelators as well as other components which alter its properties (e.g., pH and conductivity) [5]. In this way, aqueous solvents have become a sustainable alternative.

The unvarnished oil paintings of the XX century exemplify art to which aqueous solvents can be applied. Such artworks often contain substances such as magnesium sulfate and dicarboxylic acids which can render them water-sensitive [6]. This sensitivity can undesirably swell and leach the underlying layers. In swelling, the solvent diffuses into the paint which noticeably increases volume. Additionally, the paint layer's loses cohesion and the binder-pigment interaction is reduced. As a result, swelling makes the layer materials vulnerable to removal by the mechanical action of swabbing [3].

It is difficult for conservators to perceive leaching because of low molecular weight molecules (LMWM) are imperceptibly

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removed. This leaching and the removal of fine surface layers may damage the art work by altering its brightness and stiffening its layers thereby making it brittle, as LMWM act as plasticizers [3,4].

These two phenomena are widely studied by heritage scientists to gain a better understanding of the cleaning processes and try to make them as innocuous as possible. Swelling can be studied by techniques such as MRI-MOUSE [4]. Leaching has been studied by methods such as gas chromatography coupled to mass spectrometry (GC/MS) [3,4].

This study describes the process of cleaning dirt from a white monochromatic oil painting. The solvent system chosen was the water-in-oil microemulsion, W/O, with white-spirit as the dispersant phase. To obtain it, we started from the construction of a ternary pseudodiagram to its characterization. This type of microemulsion system had been tested and proved advantageous for combining the cleaning efficiency of white-spirit with water in order to act on the hydrophobic and hydrophilic components of the dirt [5] as well as of lowering the use of organic solvent thus reducing the system's toxicity [7,8]. Microemulsions consist of thermodynamically stable [7-9] nanometer-sized micelles of 10–100 nm [9,10] which small diameter increases contact surface and thus its efficiency [7,8].

Microemulsions can be characterized by techniques such as small-angle X-ray scattering (SAXS) [7,8]. In this study, characterization included Dynamic Light Scattering (DLS) and conductivity measurements [11]. Dynamic Light Scattering is a technique that characterizes particle sizes through the scattering of light [12]. Conductivity measurements are useful to characterize microemulsions. In water emulsions, low conductivity values are expected when water is in the dispersed phase and high values for the dispersing phase. In this way, varying water concentration and measuring its conductivity values, makes it possible to know in which phase the water is located.

Water content variation also affects the structure of the liquids present in the emulsion. At low water contents, globular water-in-oil (W/O) structures can be found, while at high levels the globular structures are inverted to the oil-in-water (W/O) type. Intermediate concentration values may be more complex. One way to study intermediate structures is to use percolation theory, which encompasses the sudden phase change of the system from one state to another [11] without observing a macroscopic change [13].

The surfactant used in our formulations was Tween 80, which is a nonionic, non-toxic, and non-irritating surfactant [14] with good degrability and low aquatic toxcicity [15,16]. Tween 80 also has a lipophilic–hydrophilic balance (HLB) value of 15, which is within the recommended range of 12–20 for aqueous cleaning [2].

The aggressiveness of the applied cleaning system was also verified by measuring the leached materials. As swelling and leaching are interconnected phenomena [4], it is expected that the action of the solvent on the dirt-covered layer may be accompanied by the removal of medium, additives, pigments, and fillers. This action can be indirectly observed through the detection of any of these components in the cleaning swabs using chemical means. Nowadays, portable X-ray fluorescence (XRF) equipment is commonly used in conservation given how easy it is to handle, its fast data collection, and its accurate spectra-interpretation software [17-19]. These advantages make it an auxiliary tool during the cleaning process when there are metal-based pigments and fillers present in the pictorial layers. It can reasonably and qualitatively detect metals from these kinds of removed pigments and fillers in swabs. However, XRF is limited because it does not provide information on leached organic matter, including medium, organic pigments, etc. Also, pigment can be loosely held in the swab or even loosely on the surface of paints. The technique can be used as long as the analyst is aware of these limitations. Arkarazo et al. [20] used a Raman spectroscopy technique with XRF to monitor the cleaning of a mural painting and a stone altarpiece. The removal of S compounds, such as $CaSO_4$, was observed by XRF in the cleaning of Michelangelo's sculpture of David and several frescoes in Italy [21]. Based on these experiments and aware of XRF limitations, we used this technique to measure the removal of metal-based pigments and fillers in the layer underlying the dirt.

## 2. Research aim

Characterize a microemulsified system with water and white spirit using conductivity measurements, observing its percolation behavior, and studying its use for cleaning a 20th-century oil painting.

## 3. Materials and methods

The ternary pseudodiagram of phases was obtained by titration of a mixture of Tween 80 /ethanol (tested in three ratios: 1:1, 2:1, and 2:3) and white spirit (AkzoNobel – Coral. 0 - 100 % hydrodesulfurized heavy naphtha (petroleum) and ≤ 0.1 % benzene) with distilled water. Three emulsions (EM1, EM2 and EM3) were prepared with a simple mixture components at room temperature [5]. The components of each emulsion were added sequentially after being measured in water pipettes, 2:1 mixture of Tween 80/Ethanol and white-spirit as per Table 1 ratios. An AZ 8650 conductivity meter was used to calculate conductivity *(σ)* at 27.0 °C. The volumetric fraction of water $\varphi$, was calculated using the formula below [22]:

$$\varphi = \frac{V_{water}}{V_{water} + V_{white\ spirit} + V_{surfactant} + V_{cosurfactant}}$$

The micelles diameters were measured by Dynamic Light Scattering (DLS) using a Zeta Sizer 3000 device. All cleaning tests were performed by a conservator. They were carried out after the paint was softly brushed, either with swabs soaked in distilled water or in one of the emulsified systems: EM1, EM2, and EM3. The swabs were gently applied and rolled over the surface three times for about 10 s each. The swabs used in cleaning were analyzed by XRF at two points: first, after emulsion immersion (control); second, after cleaning the screen surface. All tests were performed thrice in different areas. XRF measurements were taken with a Bruker - XRF TRACER IIIV portable spectrometer. The infrared spectrum of the paint layer was measured with a Bomem MB 100 spectrometer using a micro diamond cell, in the range of 4000–400 $cm^{-1}$ with a 4 $cm^{-1}$ resolution.

Since surfactants present in microemulsions are not volatile, residues of these can remain on paints after cleaning. Therefore, surfaces must be rinsed to remove them. To study the amount of residual Tween 80, attenuated total reflection (ATR) infrared spectroscopy tests were conducted on surfaces of white oil paints to evaluate the presence of residues after rinsing. The equipment used was a Bruker Alpha spectrometer equipped with ATR. Spectra were collected in the range of 4000 – 600 $cm^{-1}$ with a resolution of 4 $cm^{-1}$. Due to the size of the painting object of this cleaning case study, we were unable to analyze it directly on the infrared equipment. Therefore, we conducted a study on facsimiles of artificially aged white paints - zinc white and titanium white from the Winsor & Newton brand - on canvas. The paints were aged for 2 weeks at 50 °C in an oven. Spectra were collected in three situations: aged pure paints, aged paints after cleaning with microemulsion and not rinsed, and aged paints rinsed after cleaning with microemulsion. Rinsing was performed with a swab soaked in white spirit. The swab was rolled over the surface for 10 s and followed by a dry swab. This rinsing process was conducted three times.

**Table 1**
Volumetric percentage content of the components of EM1, EM2 and EM3 emulsions, mean micelle diameter with percentage distribution and polydispersity.

| EMULSION | COMPONENTS /% | | | | WATER: WHITE-SPIRIT RATIO | Micelle diameter/nm (Percentage Distribution /%) | | | Polydispersity |
|---|---|---|---|---|---|---|---|---|---|
| | Water | White-spirit | Tween 80 | Ethanol | | | | | |
| EM1 | 20.0 | 20.0 | 40.0 | 20.0 | 1:1 | 589.7 (39.1) | 5319 (28.2) | 106.4 (20.8) | 0.90 |
| EM2 | 30.0 | 10.0 | 40.0 | 20.0 | 3:1 | 549.77 (57.1) | 12.14 (30.6) | 49.377 (12.2) | 1.00 |
| EM3 | 8.2 | 24.6 | 44.8 | 22.4 | 1:3 | 61.27 (58.9) | 3.38 (32.4) | 19.03 (8.7) | 0.32 |

## 4. Results and discussion

### 4.1. Ternary pseudodiagram of phases

Fig. 1 shows the ternary pseudodiagram of phases for the studied system using different surfactant/cosurfactant ratios.

The initial ratio proposed for surfactant and cosurfactant was 1:1, obtaining a 1-phase region above the curve and a 2-phase region below it. Microemulsified systems are usually found in the 1-phase region. To better understand this system's behavior, curves were obtained in the 2:1 and 2:3 surfactant/cosurfactant ratios. The new curves also tells whether it is possible to increase the area of 1 phase in the pseudodiagram which widens the amount of microemulsions possible to obtain. An increased surfactant content in a 2:1 ratio, enlarged the 1-phase region. The 2:3 ratio led to a reduced 1-phase region. Finally, the 2:1 ratio was selected for the formulations since it was the one with the largest region in phase 1.

Three water/ white-spirit solvents ratios were formulated: 1:1, 3:1, and 1:3. This variation's aim was to determine the cleaning system's optimal conditions according to the solvents. The final surfactant/cosurfactant proportions in Table 1 were obtained by adjusting the initial ratios according to the pseudodiagram's information.

### 4.2. Dynamic Light Scattering measurements

The DLS measurements showed a polymodal pattern of the curves with three significant distributions in terms of percentages, represented in Table 1. We observed that 100 % of the EM3 system's population has a diameter <100 nm, with only 42.8 % of this population in EM2 and, an even lower 20.8 % population in the diameter <100 nm range for EM1. Therefore, according to the microemulsion size criterion of 10 – 100 nm [8,9], only the EM3 emulsion was characterized as a microemulsion.

System EM3 presented the lowest polydispersity (PDI) value (0.32) against the others very high values (Table 1). The polydispersity value is associated with the uniformity of distribution of the sample particle diameters. Values lower than 0.05 show greater uniformity in this type of distribution (monodisperse) while greater than 0.7 indicate very low uniformity [23-25]. The EM3 emulsion is not monodisperse given its PDI value > 0.05, but it is not high polydisperse either since it's <0.7 [24,25]. Microemulsions are predicted to be stable for >30 days [26] which has been experimentally confirmed.

### 4.3. Conductivity measurements

Conductivity measurements are useful for studying microemulsions, mainly through the observation of the percolation phenomenon [27]. Basically, there is a static model and a dynamic percolation model. In the former, higher water contents lead to an increase in the volume of W/O globules until they connect into tubular structures that form bicontinuous channels. The sequential rise in water content increases the amount and connections between the tubular structures to a point from which the oil phase is secreted into globules and the water enters the dispersing phase. In the dynamic model, moving globules are considered to collide and fuse for a while and then break apart. During fusion, the charge is transferred between globules. The sequential rise in water content increases the number of globules, squeezing them together into collisions which result in an increased charges conduction due to the higher number of fusions [11]. From a certain concentration value, the number of globules is high enough for the water to become dispersant and the oil dispersed.

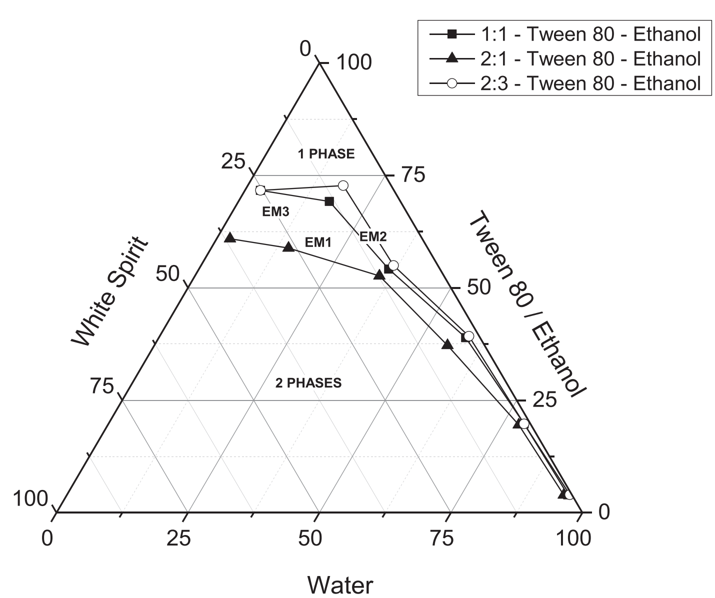


**Fig. 1.** Pseudodiagram of phases of the water, white-spirit, Tween 80 and ethanol system with three surfactant/ co-surfactant ratios.

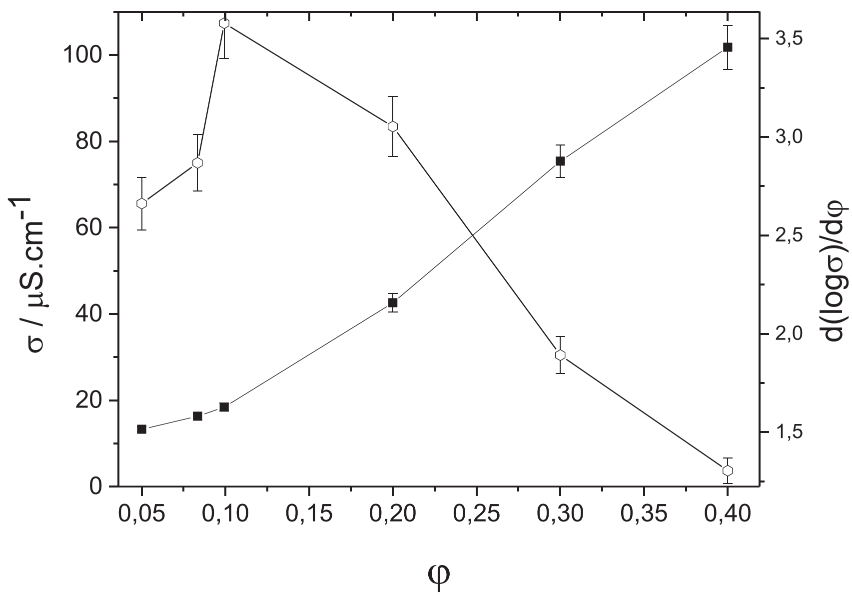


**Fig. 2.** Curves of $\sigma$ vs $\varphi$ and $d(\log\sigma)/d\varphi$ vs $\varphi$.

In both models, it is possible to experimentally construct a conductivity curve $(\sigma)$ as a function of the volumetric fraction of water$(\varphi)$. If the curve is exponential, split into two sections, and if the second section becomes more pronounced along the increasing water concentration, it becomes possible to deduce its percolation model by applying power laws. The value of the critical volumetric fraction $(\varphi_c)$ from which the curve becomes steeper is called the percolation threshold and each stretch obeys one of the power laws described in Eqs. (1) and (2) [22].

$$\sigma = A(\varphi_c - \varphi)^{-s} \; if \; \varphi_c > \varphi \tag{1}$$

$$\sigma = B(\varphi - \varphi_c)^{t} \; if \; \varphi_c < \varphi \tag{2}$$

Where: $A$ and $B$ are free parameters; "*s*" and "*t*" are the critical exponents; $\varphi$ is the volumetric fraction of water; $\varphi_c$ is the percolation threshold (critical volumetric fraction). Fig. 2 shows the curve of $\sigma$ vs $\varphi$ for the system studied.

The percolation threshold can be estimated numerically by obtaining the curve of $(d(\log\sigma)/d\varphi)$ vs $\varphi$. A curve peak corresponds to the $\varphi_c$ value which indicates the phenomenon of percolation. In the curve studied, $\varphi c = 0.10$ indicates percolation in systems containing alcohols with a carbon number lower than 6, as expected [22].

At the percolation threshold, there is a change of globules to bicontinuous tubular structures (static model) or the continuity of globules in greater numbers, greater frequency of collisions and greater load transfer (dynamic model). To distinguish the static from the dynamic model, we must determine the critical exponents "*s*" and "*t*". This can be done by linearizing the sections of the $\sigma$ vs curve $\varphi$ when applying the logarithm to Eqs. (1) and (2), thus obtaining Eqs. (3) and (4).

$$\log \sigma = -s\log(\varphi_c - \varphi) + \log A \; if \; \varphi_c > \varphi \tag{3}$$

$$\log\sigma = +t \; \log \; (\varphi - \varphi_c) + \; \log B \; if \; \varphi_c < \varphi \tag{4}$$

By plotting log $\sigma$ vs log $(\varphi c - \varphi)$ to $\varphi_c > \varphi$ and log $\sigma$ vs log $(\varphi\text{-}\varphi_c)$ to $\varphi > \varphi_c$, then performing the linear regression, "*s*" and "*t*" values are given by the slopes of the curves obtained.

Guettari [11] argues that the exponent "*s*" ranges from 0.77 to 1.60 and "*t*" from 0.61 to 1.77 for the static model. Li [27] puts these ranges at 0.50 to 0.70 for "*s*" in the static model and approximately 1.9 for "*t*" in both models. Mehta [28] cites the range of "*s*" between 0.7 and 1.6 for the static model. The experimental values obtained from the critical exponents were $s = 0.18$ and $t = 0.76$. Considering the aforementioned authors, "*s*" was found far from all ranges thus indicating that the dynamic model applies to a W/O type microemulsion EM3.

### *4.4. Cleaning tests*

This paper's focus is a 1970 untitled abstract painting by the artist Gilda Azevedo (Fig. 3). This work belongs to the Friends of Culture Collection, Federal University of Minas Gerais (UFMG) in Brazil.

The 110 × 80 × 1.8 cm mixed media work, consists of oil paint and applications of colored spectacle lenses on a plywood-type panel support. The components of the paint layer detected by the infrared (IR) spectrum were: oil (2923, 2852, 1730 $cm^{-1}$), carboxylate (1614, 1538 $cm^{-1}$), kaolin (3686, 3614, 1022, 1004, 910 $cm^{-1}$) and calcium carbonate (1798, 1403, 874 $cm^{-1}$) [29,30]. The XRF spectrum indicated the presence of Zn, Ti and Ca attributed to the white pigments of zinc and titanium and calcium carbonate charge. The attribution of these white pigments in the IR spectrum is ambiguous since they have bands below 700 $cm^{-1}$ that may have been superimposed. The carboxylate band at 1538 $cm^{-1}$ is thin and can be attributed to Zn carboxylate [31] as this element has been characterized by XRF. The XRF also detected the presence of Fe - attributed to dirt.

All cleaning tests were performed with swabs soaked in distilled water or in one of the emulsified systems. In none of the swabs was white color observed (a visual indicator of leaching) which could have been hindered by the difficulty of spotting the work's whiteness in white cotton swabs.

It is important to control conductivity values when using water as solvent and consider the osmosis phenomenon during which the solvent moves from more dilute media (hypotonic) to more concentrated media (hypertonic). Oil paint layers generally have conductivity values between 50 – 300 $\mu S.\ cm^{-1}$ [2]. The distilled water used for cleaning had a conductivity of 133.2 $\mu S.\ cm^{-1}$ and pH = 6 thus well within the range allowing it to behave as close to an isotonic solution as possible with the oil layer. According to Wolbers [2], "solutions that are 'isotonic' (i.e. the same 'tonicity' or ion concentration) to painted surfaces to be cleaned generally cause the least swelling and therefore the least damaging effects

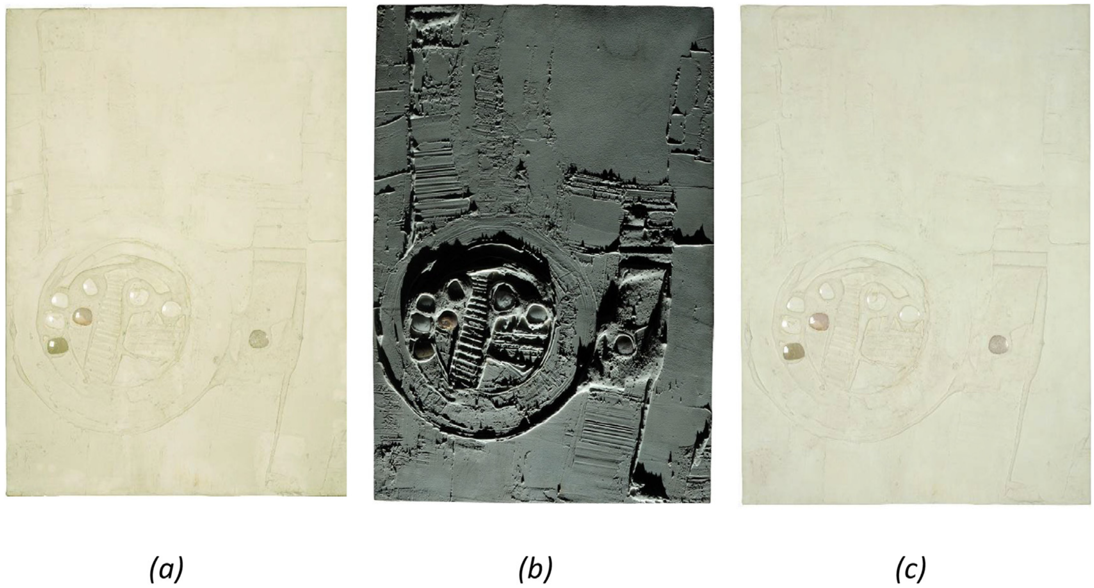


**Fig. 3.** "Untitled work", 1970. Gilda Azevedo. *(a)* Before cleaning. *(b)* Photo with grazing light highlighting the brushstrokes. *(c)* After cleaning. Credit: Claudio Nadalim. Edition: Viviane Xavier.

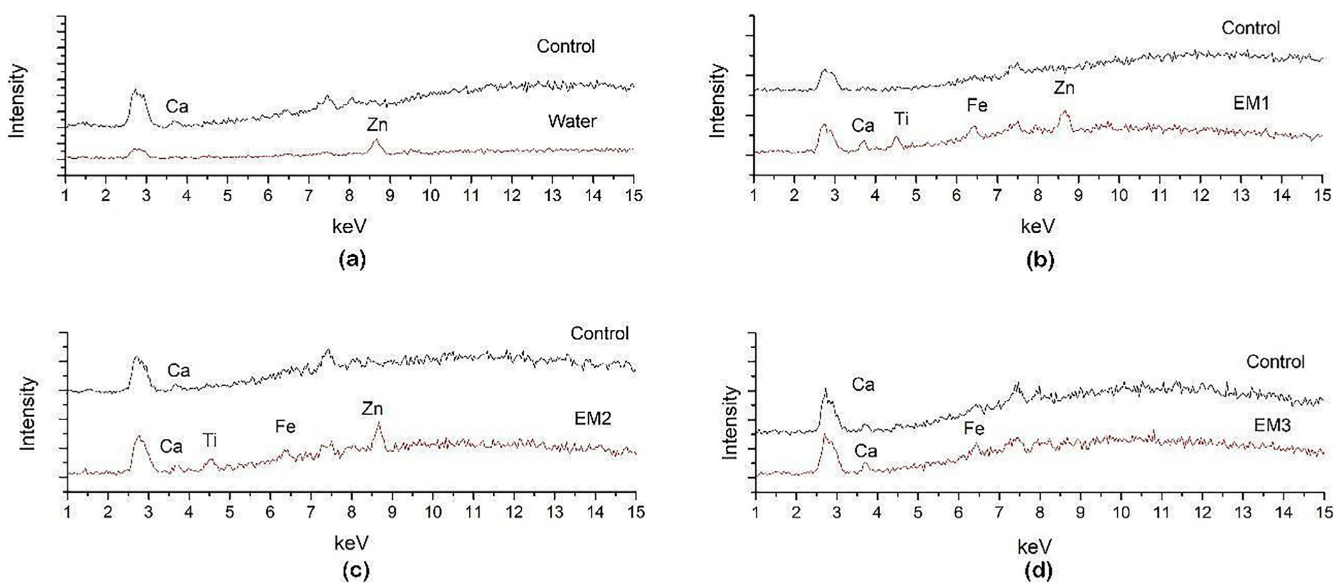


**Fig. 4.** X-ray fluorescence spectra obtained from the cleaning test swabs. Each sub-item compares the control, swab with water or emulsion, and swab after cleaning. *(a)* Test with distilled water, *(b)* test with EM1, *(c)* test with EM2, and *(d)* test with EM3.

on the films". The pH value of 6 falls within the recommended pH range for oil cleaning, 5.5 - 8.5. Wolbers [2] also discusses that the oil layer tends to swell in aqueous cleaning solutions at pH values "close to the pKas of the free fatty acids present in the films (palmitic or stearic acid pKa s= 4.5)" whereas higher pH levels above 8.5 cause saponification, which triggers the formation of filmogenic materials [2].

Cleaning with distilled water proved to be efficient without visually observing the removal of the pictorial layer, but the phenomena of swelling and leaching may occur without being visually perceptible. The XRF spectrum (Fig. 4a) indicated the removal of Zn. Using only XRF, it is not possible to determine the compound containing Zn. It is likely derived from the white pigment zinc (ZnO) or zinc carboxylate. As previously discussed, the infrared spectrum showed a band at 1537 $cm^{-1}$, which was attributed to a crystalline form of this carboxylate [32]. Finally, the Zn compound presence for its removal in cleaning may be due to the decreased interaction of the pigment with the binder as a result of swelling and also due to the swab's mechanical action [33]. Equally important, this spectrum also detected Fe traces which were attributed to dirt.

In the tests carried out with the EM1 and EM2 emulsions, a greater removal of elements was observed compared to that of distilled water. In addition to the Zn and Fe, Ti was also detected, originating from $TiO_2$ (Figs. 4b and 4c). Ca signals were also spotted, but since this is also present in the spectrum of the swab with emulsion prior to cleaning, those cannot be attributed solely to $CaCO_3$ removal. The higher efficiency in removing materials, when compared to neat water, can be explained by the detergency effect caused by the presence of surfactant and the fast action of the microemulsion [1].

In the tests performed with microemulsion EM3, Zn and Ti traces were undetected, indicating that there was no significant removal of the uppermost layer (Fig. 4d). The presence of Ca was

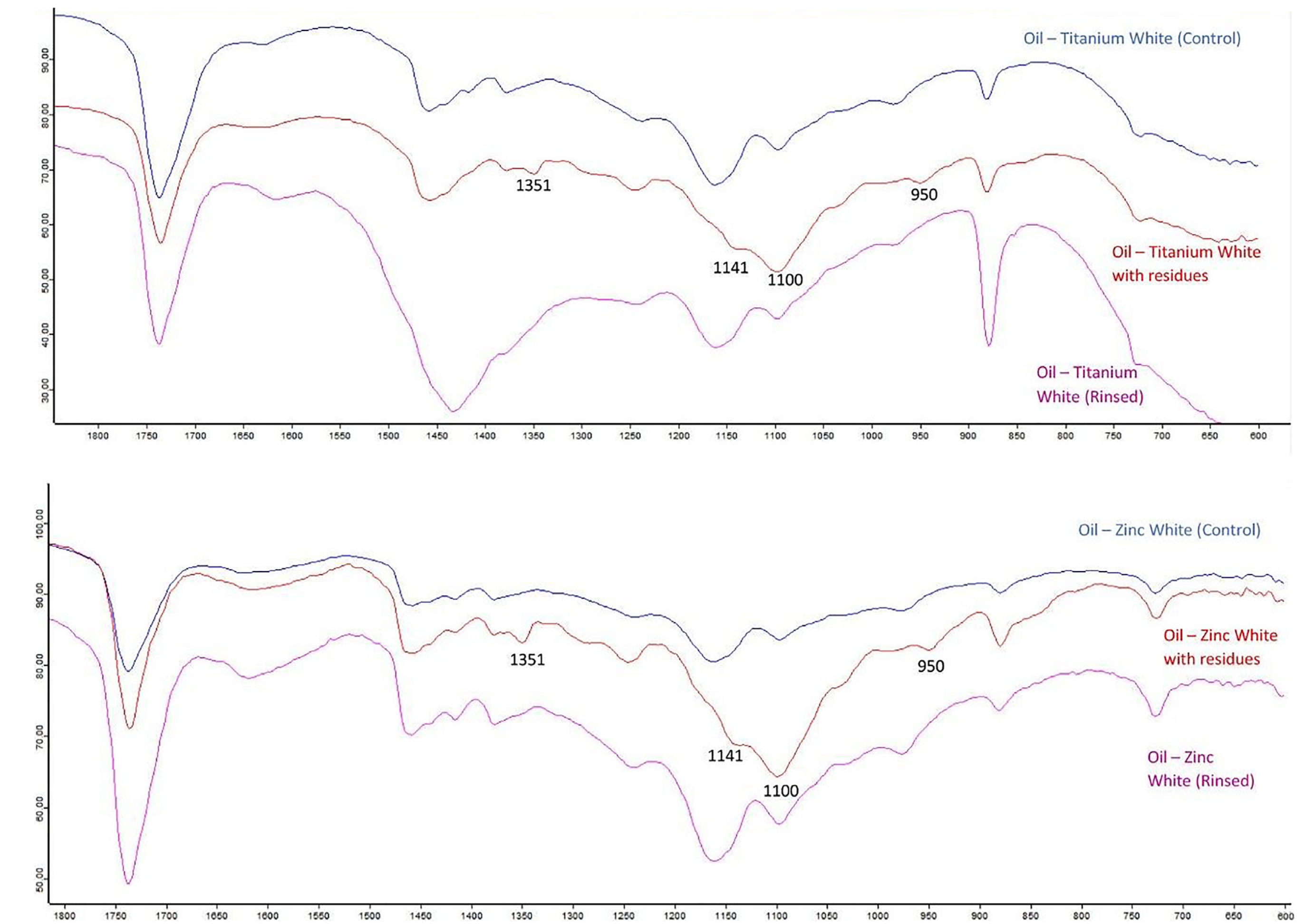


**Fig. 5.** Comparison of the spectra of two thermally aged white paints, titanium white and zinc white, cleaned with the EM3 microemulsion. Each box in the figure contains three spectra: the first spectrum shows the paint before cleaning, the second spectrum shows the paint after cleaning with EM3 microemulsion without removal, and the third spectrum shows the paint after rinsing.

common both in the control sample as well as post cleaning. Traces of Fe were attributed to dirt and its removal therefore desirable. This is the formulation with the lowest water content of the three.

In light of the test results, we opted to clean the artwork with an EM3 system- embedded swab. Rolling swabs were applied once with the microemulsion. After application, residues were removed with a rolling dry swab, followed by rinsing with a rolling swab soaked in white spirit, and then with a dry swab. This rinsing process was carried out three times. Fig. 3c shows the final result.

A study of the residues remaining after rinsing was conducted on facsimiles as described in the methodology. The analysis of the collected infrared spectrum (Fig. 5), where there was no rinsing of the microemulsion, showed two strong bands related to Tween 80 at 1141 and 1100 $cm^{-1}$. In the spectrum of the paint layer with the unrinsed surfactant, these bands overlap with the band at 1163 $cm^{-1}$ of the paint, causing a shoulder. In the spectrum of the rinsed paint, the band at 1163 $cm^{-1}$ is again visible. Additionally, a band around 1100 $cm^{-1}$ is present in the paint spectrum, but its relative intensity compared to the band at 1163 $cm^{-1}$, without interference from Tween 80, differs.

The bands at 1351 and 950 $cm^{-1}$ related to Tween 80 are also absent in the spectrum of the rinsed paint. Finally, it is important to highlight that three rinses were performed due to the high surfactant content in the microemulsion, 44.8 %. It is recommended that the microemulsion contains much lower amounts of surfactant to reduce the need for rinsing [5], since extensive rinsing can harm the paint by causing more leaching and lixiviation. The analysis of a ternary pseudodiagram allows for the selection of formulations with lower surfactant levels.

## 5. Conclusion

The outcome suggests that the differences in cleaning results of the three emulsions are due to the W/O microemulsified system in EM3. This microemulsion behavior was already stated in the literature. According to Baglioni and Casini [7,8], in microemulsified systems, the surfactant restricts the wetting action to the nano-scale area. Its smaller size increases its interfacial area, thereby enhancing its cleaning efficiency compared to other emulsions [7,8]. Also, according to Ormsby [5], this restriction of action is due to the nanoscale contact degree of the aqueous phase due to the dynamic nature of microemulsions. The studied microemulsified system behaved as described in the literature. It enhances control over the cleaning process, thereby minimizing the removal of pigment from the uppermost surface of the paint.

Conductivity studies can be easily conducted and have proven to be of great relevance in the study of obtaining microemulsions. Knowledge about percolation provided insights into the W/O microemulsion. Additionally, it indicated that the obtained microemulsion follows the dynamic model, thus supporting explanation about the cleaning efficiency.

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