

AN EXPERIMENTAL STUDY ON THE INFLUENCE OF POLAR AND NON-POLAR ORGANIC SOLVENTS ON THE EGG YOLK PAINTED LAYER IN WALL PAINTINGS

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ABSTRACT

This paper presents the Physicochemical effects of organic solvents on a painted layer during the cleaning process in the conservation of mural painting. An experimental study was carried out replicating mural paintings followed by using two organic solvents (Ethanol and Benzene), which were applied on Egg yolk binder mixed with Malachite. This experiment was performed by a range of complementary analytical techniques used to examine treated samples, such as FTIR (Fourier- Transform Infrared Spectroscopy) analysis, Colorimetric Measurements and the Scanning Electron Microscope (SEM). The Surface Morphology analysis by SEM confirms an increase in the roughness with a fairly even surface topography at the micro level as the grains of pigment becomes more evident and more light scattering than a saturated paint film. The FTIR analysis indicated that the amides of the secondary structure of proteins were affected by organic solvents. Colorimetric measurements showed a total color difference ΔE in the sample with benzene.

KEYWORDS

Egg yolk; Organic solvents; FTIR; Colorimetric measurements; SEM.

المخلص

يتناول هذا البحث التأثيرات الفيزيوكيميائية للمذيبات العضوية على الطبقة اللونية اثناء عملية التنظيف للصور الجدارية. حيث تم اجراء دراسة تجريبية بعمل مكعبات مماثلة لتركيب للصور الجدارية تحمل طبقة لونية من صفار البيض مخلوطا بالملاكييت وتم وضع المذيبات العضوية البنزين-الايثانول كل على حده وتم قياس التأثير باستخدام التحليل بالاشعة تحت الحمراء والتغير اللوني والميكروسكوب الالكتروني الماسح. اوضح استخدام الميكروسكوب الالكتروني الماسح ان سطح العينات اصبح اكثر خشونة وعاكسا اكثر للشعاع الساقط عليه مقارنة بالعينة القياسية، كما اوضح التحليل بالاشعة تحت الحمراء تأثر مناطق الامتصاص المميزة مثل مجموعة الأמיד وهي التي تميز التركيب البروتيني لصفار البيض. فيما يختص بالتغير اللوني فقد كانت قيمة التغير اللوني الكلي للعينة بعد إضافة البنزين أكبر من العينة مع الايثانول.

الكلمات الدالة

صفار البيض؛ المذيبات العضوية؛ الأشعة تحت الحمراء؛ التغير اللوني؛ الميكروسكوب الالكتروني الماسح.

INTRODUCTION

The response of egg tempera paint films to solvents was investigated by (Khandekar 1994), using films prepared with various pigments and exposed to light and thermal ageing regimes. Surface changes resulting from solvent immersion or swabbing were monitored using SEM, and quantities of lipid material extracted were determined using (CG) Gas Chromatography. Substantial quantities of the lipid content of these films were found to be extractable using organic solvents, particularly chlorinated solvents (Khandekar, Phenix, and Sharp 1994).

A study made by (Shaaban, 2004) used organic solvents such as Acetone, Ethanol, and Toluene mixed with water during chemical cleaning to remove soot and dust from the surface of a wall painting containing Arabic Gum as a binding medium in this operation, dimming and fading of the painted layer was observed. The study also warned against using acetone as it causes drying, cracks, embrittlement and loss of flexibility (Ali and Abd El Aal 2011, 53-59).

A study was made by (Casoli, Berzioli, and Cremonesi 2012) to investigate the properties of egg yolk films used as a binding medium and its interactions with water and organic solvents. The research used samples prepared in July 2007 and on a tempera layer made by researchers of the Smithsonian's Museum Conservation Institute in 1995. It also included a sixteenth-century panel painting from the Pinacoteca di Siena. Ethanol or acetone, isooctane and water were used to test for leaching. It was observed that the lipid components are mostly removed by the application of solvents of different polarities (from isooctane to water). The most significant amount of leaching was observed within the fresher samples. Furthermore, it was noticed that pigmented layers are less affected by the leaching phenomena than a layer without pigments, especially lipid components. Also, the unsaturated fatty acids were extracted mainly from fresh samples, with less from the 1995 tempera and nothing from the sixteenth-century painting. However, the study carried out on this painting showed that leaching is more evident for the lipid components and amino acids.(Casoli, Berzioli, and Cremonesi 2012,39).

METHODS

Simulated samples of mural painting were prepared in the laboratory, and the mortar sheets were prepared from sand+ lime+ gypsum (3:2:1) and then covered with a thin layer of whitewash made of gypsum. A mixture of 2 mL of egg yolk binder was sufficiently blended with 2 grams of Malachite and then applied to a rectangular cube. The paint layer on the substrate was applied using a soft brush then left to dry for eight months before applying solvents.

The mixture was created using a fresh egg with the yolk, and egg white separated, followed by the addition of a few drops of vinegar and water to preserve the solution from biodeterioration and prevent it from drying rapidly. In addition, Malachite was ground to a fine powder using a mortar and pestle. The application of organic solvents (brought from the organic chemistry lab-faculty of science-Fayoum university) Ethyl-alcohol 96% and Benzene 99.8% was applied to the painted layer via a syringe dripping on a layer of cotton, followed by a layer of gauze on the surface of the sample. The sample was then covered with a Polyethylene sheet to prevent rapid evaporation of a solvent on the surface, with 6 hours intervals separating each shot for one

day. No mechanical action was applied to avoid scratching the surface and concentrate on solvent influence only.

Used Organic solvents and their Polarity

Polarity is the ability to form two opposite centers in the molecule. The concept is used in solvents to describe their dissolving capabilities or the interactive forces between solvent and solute. Because it depends on dipole moment, hydrogen bonding, entropy, and enthalpy, it is a composite property without a physical definition. The dipole moment has the most significant influence on the polar properties of solvents. Highly symmetrical molecules (e.g. benzene) and aliphatic hydrocarbons (e.g. hexane) have no dipole moment and are considered non-polar. Ketones, esters, and alcohol are examples of compounds having dipole moments (from high to medium, sequentially), and they are polar, medium polar, and dipolar liquids (Wypych 2014, 65-97). Ethyl Alcohol contains the hydroxyl group, which is polarized and can form hydrogen bonds (Torraca, 2005, 64), Table 1(Raj Joshi, Adhikari, and Cruz-Olivares 2019, 1-18).

Table 1, Physicochemical properties of Ethanol and Benzene

| Physicochemical properties | | | | | | | | |
|----------------------------|---------------------------------|-----------------------|----------------------|-----------------------|----------------|------------------------------|---------------------|--------------------|
| Solvent | Structural formula | MW (Molecular weight) | BP(°C) boiling point | MP (°C) Melting point | Density (g/mL) | solubility in water (g/100g) | Dielectric Constant | FP(°C) Flash point |
| Ethanol | C ₂ H ₆ O | 46.07 | 78.5 | -114.1 | 0.789 | Miscible | 24.6 | 13 |
| Benzene | C ₆ H ₆ | 78.11 | 80.1 | 5.5 | 0.8765 | 0.18 | 2.28 | -11 |

Benzene or Benzol; aromatic hydrocarbons (D'Andrea and Reddy 2018, 1-10). Aromatic hydrocarbons are made up of carbon-carbon and carbon-hydrogen bonds, much like all other hydrocarbons. Because the electronegativity of these atoms is quite alike and relatively low (2.1 for H, 2.5 for C), no persistent dipole is formed in hydrocarbon bonding configurations. Aromatic hydrocarbons possess a multiple, conjugated C=C with a pi-electron system, but this does not produce enough dipole for energetically desirable dipolar interactions with other molecules. In alcohols, on the other hand, carbon and/or hydrogen are bonded to an atom with substantially higher electronegativity - the oxygen atom. In alcohols (and organic compounds with other atoms of electronegativity higher than carbon or hydrogen), permanent dipoles exist, generated by the uneven distribution of bonding electrons between the more electronegative atom (O) and less electronegative atom (C, H). Due to their atomic composition, aromatic hydrocarbons are classified as "non-polar" compounds or substituents. The only noteworthy intermolecular connection that can occur is through relatively weak van der Waals interactions (VDWs) or "induced dipolar" interactions induced by temporary distortions in the electron distribution among atoms in the structure (Deruiter 2005, 1-13).

Egg Yolk

Proteins are complex structures that are composed of amino acids linked together by peptide bonds. Proteins, as a class of materials, may be identified by the presence of specific bands in the infrared. The bands result from the peptide bonds which link the amino acid "building blocks" in the polymer chain. The yolk makes up about 36% of the weight of the fresh whole hen egg, the main components are lipids (about 65% of the dry matter), and the lipid-to-protein ratio is about 2:1, table 2(Chang et al. 2018).

Table 2, approximate Composition of Fresh yolk and dry yolk (%)

| Compounds | Fresh yolk | Dry yolk |
|----------------------|------------|----------|
| Water | 51.1 | - |
| Lipids | 3.6 | 62.5 |
| Proteins | 16.0 | 33.0 |
| Carbohydrates | 0.6 | 1.2 |
| Minerals | 1.7 | 3.5 |

The two egg yolk fractions, Plasma and Granules, contain the major egg yolk proteins and lipoproteins. The plasma contains the water-soluble fraction, namely the livetins, whereas the granules are mainly comprised of lipovitellins (as parts of high-density lipoprotein, HDL), phosvitins, and a small amount of low-density lipoproteins (LDL) (Lilienthal, Drotleff, and Ternes 2015, 68-79). Lipids of the yolk are exclusively associated with lipoprotein assemblies. They comprise 62% triglycerides, 33% phospholipids, and less than 5% cholesterol. Carotenoids represent less than 1% of yolk lipids and give it its color. Proteins are present as free proteins or apoproteins (included in lipoprotein assemblies). Phosphatidylcholine is an amphiphilic lipid containing 3 fatty acids as the non-polar part and choline as the polar part (Anton, Nau, and Nys 2006). Carotenoids are the natural pigments of hen egg yolk. They confer its yellow color, which can go from a very pale yellow to brilliant dark orange (Huopalahti et al. 2007, 1-113).

The yolk is a complex system with several suspended particles in a clear yellow fluid (plasma) that contains proteins and is encircled by a vitelline membrane. The main types of particles are spheres, profiles, and granules. Spheres are minor components (Mine 2008, 1-48). The yolk consists of minute spherical droplets of oil suspended in a water medium. Dried yolk consists of about one-third protein and two-thirds other compounds. These other compounds are predominantly oil. As with salad dressings that consist of oil and vinegar, they need to be continually shaken to disperse droplets of the oil in the water phase (vinegar) to form an emulsion. If let sit, the water phase and oil separate. Egg yolk is also considered a mixture of an oily phase and a water phase. An egg yolk is a stable emulsion of these two

insoluble classes of material, naturally stabilized by a compound called lecithin. As a medium, egg yolk is usually diluted with water and mixed with pigments. After it dries, it can no longer be dissolved in water, a property that distinguishes it from all other water-soluble media. Many proteins that are found in solution in water in nature, such as those in egg yolk, transform as they are heated or dried. These transformations (called denaturation) mean that once heated or dried, these proteins can no longer be put back into a solution in water (Taft and Mayer 2000, 1-11).

INVESTIGATION TECHNIQUES

FTIR (Fourier-Transform Infrared Spectroscopy) Analysis

FTIR is commonly employed to study organic binders such as proteins (Abdelaal 2019, 171-181), The amide group of proteins (the secondary structure of proteins is the amide I region between the wave numbers 1600 and 1700 cm^{-1}) (Lilienthal, Drotleff, and Ternes 2015, 68-79) and polypeptides presents characteristic vibrational modes (amide modes) that are sensitive to the protein conformation. In this technique, the irradiates the sample infrared beam (IR). Then certain wavelengths that indicate the presence of specific functional groups are usually absorbed by the sample (Mahmoud 2018, 1-6; Sh. Abdelaal 2018, 709-722). FTIR spectra were obtained in transmittance/ wavenumber mode in the mid-IR range of 4000–500 cm^{-1} (Bader and Waeel 2014, 349-366). The BRUKER OPTICS VERTEX 70 FT-IR spectrometer obtained the infrared absorption spectra of samples. The samples were prepared by mixing them with KBr (99+% chem-lab NV). The measurements were carried out at the College of Graduate Studies for Advanced Science, Beni-Suef University.

SEM (Scanning Electron Microscopy)

This method allows for the investigation of the surface morphology (El Aal 2012, 79-89) before and after applying solvents using the SEM Model QUANTA 250 FEG (Field Emission Gun). The scanning electron microscope is then attached to an EDX Unit (Energy Dispersive X-ray Analyses), with an accelerating voltage of 30 KV, with a magnification of 14x up to 1000000 and resolution for Gun.1n), FEI Company, Netherlands. The Egyptian Mineral Resources Authority, Central Laboratories Sector.

Colorimetric Measurement

CIE Color Systems, the CIE, or Commission Internationale de l'Eclairage translated as the International Commission on Illumination), is responsible for international photometry and colourimetry recommendations. In this system, L*axis (+L* for light, - L* for dark) represents the lightness, a*(+ a* is for red, - a* for green), and b*(+ b* for yellow, and - b* for blue) the chromaticity coordinates (Abdelaal and Sandu 2019, 459-474; Ali and Abd El Aal 2011, 53-59; (Orabi 2020)). The color strength (K/S) value was measured for green color at 620 nm. The K/S values for samples are then compared to a standard sample at the same wavelength; the higher values represent darker and more saturated colors. The coordinate

values (CIE L*a*b*) were measured before and after the treatment. The color difference is calculated using the L*a*b* value for each object (sample) color using this equation:

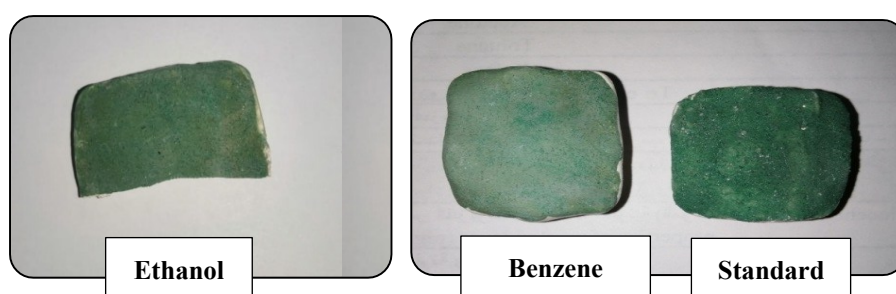
$$\Delta E = \sqrt{\{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2\}} \text{ (Teklemedhin 2018, 538-542; S. Abdelaal 2018, 429-438).}$$

This technique was applied to detect color changes in the painted layer after applying organic solvents. The measurement was made using Spectrophotometer OPTIMATCH 3100 –SDL company- in the National Institute of Standards (NIS) Cairo.

RESULTS AND DISCUSSION

Visual Observation

This primary phase depends mainly on naked-eye observation of the aspects of deterioration caused by applying organic solvents to the painted layer. The most easily distinguished samples were those with egg yolk, specifically after the addition of Benzene, with less damage caused by Ethanol. Fading and erosion of the surface appeared obviously, figure 1.



4. Figure 1, Visual observation of Egg yolk + Malachite sample after addition of Benzene – Ethanol, images show more fading, and loss of luster compared to standard samples

Organic binders lose water during the application, and the tertiary and quaternary structures alter through a rearrangement of the internal bonds between functional groups (Volpi 2017,13). Egg yolk is considered a proteinaceous binder characterized by the presence of N-H stretch about 3355 cm^{-1} . Amide I occurs near 1639 cm^{-1} , C-N-H bending band of amide II 1546 cm^{-1} , and C-H bending vibration at 1456 cm^{-1} in stair-step-type intensities indicated in Table 3. The amide I band, which mainly probes stretching vibrations of the C=O bonds in the peptide backbone, has been a sensitive and popular band in studying protein secondary structure. Each type of secondary structure correlates to a slightly different C=O stretching frequency in the amide I band region of the spectrum due to its unique molecular geometry and hydrogen bond pattern. Amide II also provides information on the vibrational bands of the protein backbone. Though it derives primarily from in-plane N-H bending (40–60% of the potential energy) and C-N stretching (18–40%), resulting in less sensitivity and specificity for protein conformational changes compared to the amide I band (Sadat and Joye 2020, 2-16).

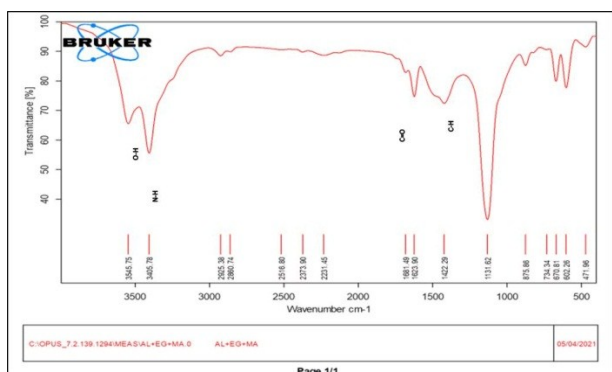


Figure 2, FTIR spectrum of Egg yolk +Malachite after addition of Ethanol

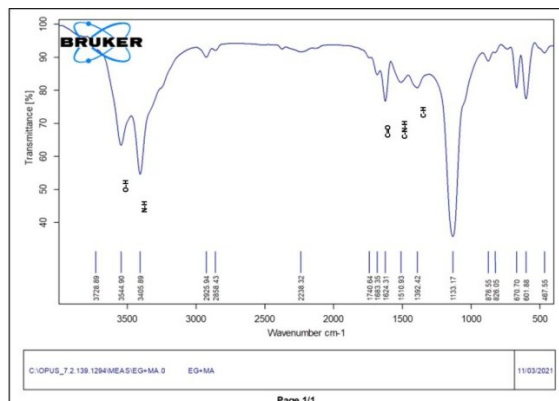


Figure 3, FTIR spectrum of mixture of Egg yolk +Malachite before addition of organic solvent

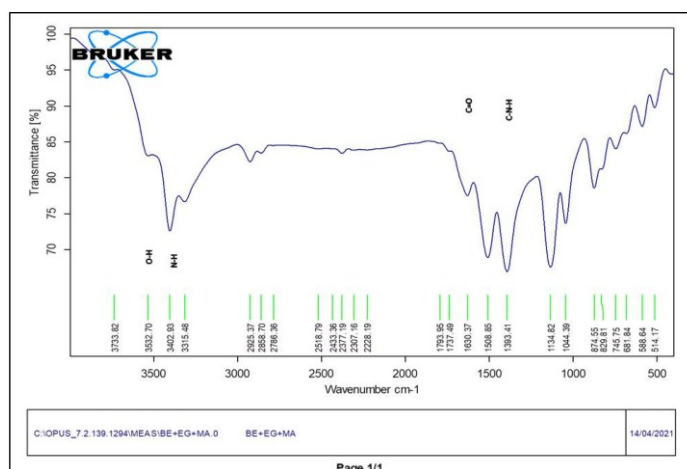


Figure 4, FTIR spectrum of Egg yolk+ Malachite after addition of Benzene

Table 3, Characteristic wavenumbers of Egg Yolk

| Egg Yolk | | | | | |
|------------------------------|---|--|--------------------------------|-----------------------------|--------------------------------|
| Wave number cm^{-1} | 3355 | 2925-2858 | 1639 | 1546 | 1456 |
| Functional group | O-H stretching vibration of water molecules, and also from the N-H stretching vibrations of peptide bonds (amide A band) because the sample was in a liquid state | stretching band C-H group in Lipid molecules | C=O stretching band of Amide I | C-N-H bending band Amide II | C-H bending bands of amide III |

FTIR results showed that the addition of Ethanol caused the disappearance of the band at 1510 cm^{-1} of amide II and a major shift in the band at 1456 cm^{-1} to 1422 cm^{-1} of C-H. FTIR spectrum of the sample with Benzene showed a change in the band at 3544 to 3532 cm^{-1} of O-H, 1624 cm^{-1} to 1630 cm^{-1} of amide I (the secondary structure of proteins), also an absence in the band at 1456 cm^{-1} assigned to amide III, table 4.

Any change in the FTIR spectrum after any chemical reaction/process indicates that the process/reaction has altered or modified the chemical interactions inside the samples. If new

chemical bonds are produced, we will find new peaks originating. If the peaks have vanished, it indicates that a particular chemical bond no longer exists in the sample. Furthermore, shifting of the peaks may also propose that the molecular environment (the strength of the chemical bonds) may change too **Invalid source specified.**, figures 2,3,4,5.

For an accurate and complete description of the processes involved in solvent cleaning, the following procedures need to be considered: paint layer swelling, solvent diffusion and Capillary penetration, evaporation, leaching and chemical reactions inside the paint layer. The extent to which any of these processes occur depends on the amount of solvent that is applied (varying with application method) and the rate of solvent transport inside the paint. The rate of solvent transport inside the paint depends on the condition of the paint layer and is influenced by factors such as the presence of (micro) cracks or soluble extractable materials. Two forms of solvent penetration—diffusion and capillary penetration are the basis of interaction with paint layers: Capillary penetration is the transport of bulk liquid solvent into the cracks and pores in the paint and ground. Diffusion is the transport on a molecular level of solvent through the solid organic phase of the paint. The first effect occurs rapidly and is mainly influenced by paint porosity and solvent surface tension. On the other hand, diffusion is influenced by the increased surface access created by the first effect. Solvent sensitivity occurs in cases where the binding medium has such a low degree of polymerization and a high degree of oxidation (due to network degradation or inherently low cross-linking) that minor mechanical action removes pigment particles (Baij et al. 2020, 1-23).

Organic solvents modify the native structure of proteins by disrupting hydrophobic interactions between the non-polar side chains of amino acids. Relatively high concentrations of these solvents are essential to unfold the well-arranged structure of polypeptide chains (Asakura, Adachi, and Schwartz 1978, 6423-6425). The characteristic physical feature of lipids, to be precise, their water insolubility, derives from the chemical structure of part of the lipid molecule, which is termed as Hydrophobic. The hydrophobic moiety is the hydrocarbon chain of (the fatty acid). The most plentiful membrane lipids are the phospholipids. These contain a polar head group and two hydrophobic hydrocarbon tails. The tails are generally fatty acids (Gurr, Harwood, and Frayn 2002, 215-263).

The lipids which exist in egg yolk contain the same characteristics as minor molecules in a drying oil. A plasticizer action can then be hypothesized, and accordingly, structural damage can affect the outcome if the lipids are eliminated by using solvents through the cleaning process. Complexity increases as the medium begins to age and undergoes chemical alterations as a result of the oxidation processes and interactions amongst the diverse components, with lipid oxidation and related radical reactions, cross-linking, and chain scission being the major reactions. Soon after the egg tempera application, lipids can be extracted with numerous solvents such as alcohols, ketones, and aromatic and chlorinated hydrocarbons. Subsequently, the film experiences contraction and presents superficial alterations. Also, pigmented layers are less affected by leaching phenomena than pigment-free layers, particularly for lipid components which could be linked to a physical, morphological

factor when the pigment is dispersed in it, the binding medium is obviously reduced. Furthermore, The cations could bind to fatty acids, forming metal soaps and making the fatty acids less sensitive to leaching (Casoli, Berzioli, and Cremonesi 2012, 40-44).

Table 4, FTIR results of Egg yolk with Malachite before and after the addition of Organic solvents

| EGG YOLK+MALACHITE | | | | | |
|---|----------------------|--------------------------|----------------------|--------------------------|----------------------|
| Standard FTIR (KBr)/ Cm ⁻¹ | | EtOH | | Benzene | |
| Wave number assignment | Absorbance Intensity | Changement in wavenumber | Absorbance Intensity | Changement in wavenumber | Absorbance Intensity |
| 3544 of O-H | 63.4 | 3545 | 65.6 | 3532 | 83.1 |
| 3405 of N-H groups | 54.6 | 3405 | 55.6 | 3402 | 72.6 |
| 2925 | 90 | 2925 | 88.5 | 2925 | 82.2 |
| 2858 | 92 | 2860 | 89.9 | 2858 | 83.4 |
| belong to C-H group in Lipid molecules | | | | | |
| 1624 of amide I peptide bonds of proteins | 76.7 | 1623 | 74.7 | 1630 | 77.5 |
| 1510 C-N-H of amide II | 82.4 | - | - | 1508 | 68.9 |
| 1456 C-H of amide III | - | 1422 | 72.5 | - | - |
| 1392 | 80.8 | - | - | 1393 | 66.9 |
| 876 | 88.9 | 875 | 85.3 | 874 | 78.6 |
| 826 of carbonate (CO ₃) ²⁻ characteristic of Malachite | 91.4 | - | - | 829 | 81.2 |

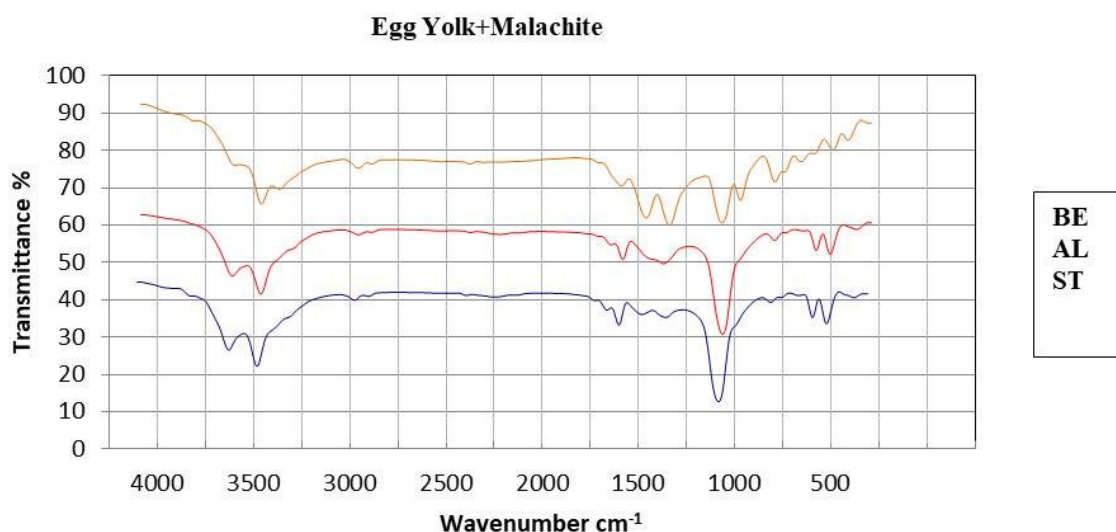


Figure 5, FTIR spectrum of Malachite (MA) +Egg Yolk (EG) starting with standard (ST) at the bottom followed by sample after addition of organic solvents ;Ethanol (AL) ,Benzene (BE).

Colorimetric measurements

The Brightness (L value) increased in the sample after the addition of Benzene (64.91) more than in the sample with Ethanol (57.39) which differed slightly from the standard sample (57.3), which appeared in fading of color.

- The **a** value (+red – green) in samples after adding organic solvents gave less green hue (all results were negative). The **a** value decreased with Ethanol more than with Benzene.
- The **b** value (+yellow – blue) In samples after adding organic solvents gave a yellow hue (the values were all positive) which decreased with Ethanol and increased with Benzene.
- The K/S value of the sample after adding Benzene was reduced more than adding Ethanol compared to the standard sample. Furthermore, the color difference ΔE between the standard sample and the Ethanol sample showed less color difference ΔE 2.47, and the color difference between Standard and Benzene samples showed more color difference ΔE 7.9, table 5.

Table 5, Colorimetric Measurement of a mixture of Egg Yolk Binder with Pigments after Applying Organic Solvents

| MALACHITE | | STANDARD | +ETHANOL | +BENZENE |
|---------------|------------|----------|----------|----------|
| + EGG YOLK | L | 57.3 | 57.39 | 64.91 |
| | a | -10.01 | -7.55 | -9.72 |
| | b | 6.23 | 6.07 | 8.33 |
| | K/S 620nm | 1.5459 | 1.3976 | 0.8885 |
| | ΔE | - | 2.47 | 7.9 |

The Surface Morphology by SEM

The grains of pigment become more evident and more light scattering than a saturated paint film. This shows not only that binders are being removed but also that large amounts of polymer chains are being extracted, which directly affects the overall properties of the paint layer. The results were evaluated according to the level shown in the illustration in the standards ((Phi Hung et al. 2018, 2525-2518; Doménech-Carbó et al. 2013, 125-134; Orabi 2020, 238-249). The usage of Organic solvents results in the weakening of the binding medium (because of Leaching, which describes the extraction of soluble components of the organic binder and, in contrast to swelling, is less immediately tangible, it has a potential risk in the long term. As a consequence of the extraction of low-molecular-weight components, which show plasticizing properties in the film, the primary risks of leaching are the embrittlement of the film and the alterations of the optical properties of the paint surface, increasing mattness) (Volpi 2017,19), and long exposure times to them have been shown to affect the polymer lattice. This effect should be considered when considering the probable repeated exposures resulting from successive cleaning interventions over time, especially if combined with mechanical action, e.g., swabbing. The solubilization of polymer chains and additives severely affected the mechanical properties of the paint films, making them far more brittle and thus more susceptible to damage and loss (Phi Hung et al. 2018, 2525-2518; Doménech-Carbó et al. 2013, 125-134; (Osman 2019) figure 6.

-SEM images of Malachite +Egg Yolk samples with Ethanol-Benzene, MA (Malachite) – Al (Ethanol)-BE (Benzene) - EG (Egg yolk).

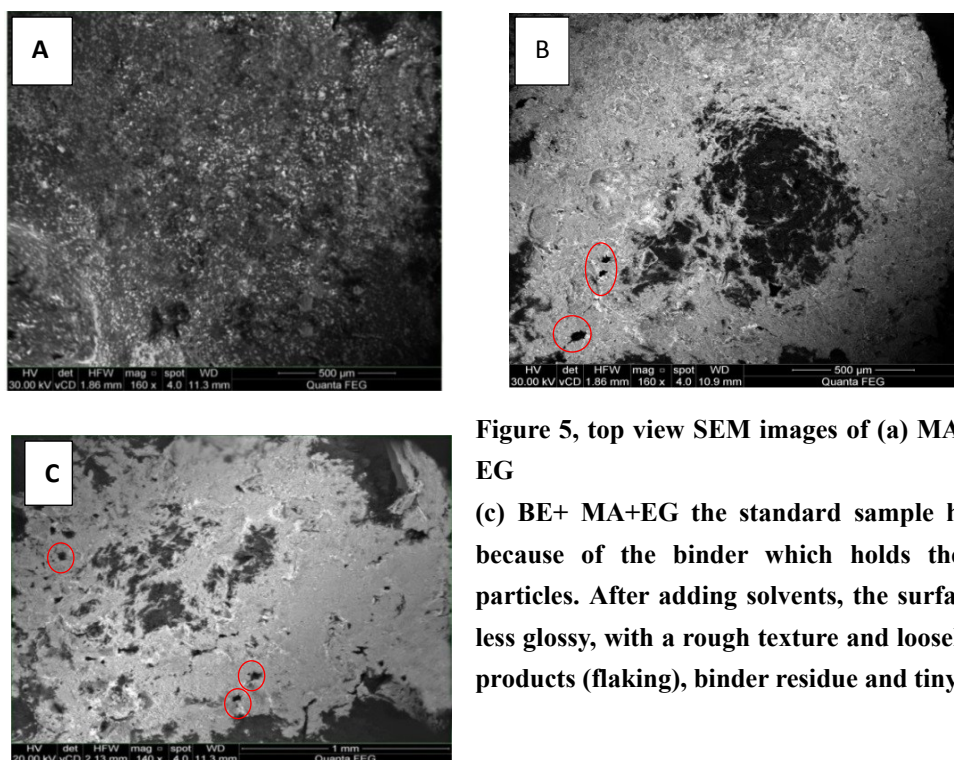


Figure 5, top view SEM images of (a) MA + EG, (b) AL+MA+EG

(c) BE+ MA+EG the standard sample has a unified surface because of the binder which holds the dispersed pigment particles. After adding solvents, the surface of the sample was less glossy, with a rough texture and loosely adherent corrosion products (flaking), binder residue and tiny pores appeared.

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