

Characterization of the Mamluk painting materials in Cairo, Egypt: El-Ashraf Bersbay Madrasa (826 A.H/ 1423 A.D) a case study.

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Abstract: The painting materials in the Islamic monuments in Cairo were variable and distinguished. The accurate identification and characterization of painting materials in an art object or in a paint layer is certainly an important step in the history of art, technology and conservation. The main objective of the present paper is to examine and investigate the different painting materials (pigments, gilding, binding media and painting ground) employed in the decorated ceilings of Madrasa of Al-Ashraf Bersbay (Mamluk Jarkasy period). X-ray diffraction (X.R.D.), scanning electron microscope SEM (EDAX), light optical microscope (LOM) and fourier transform infrared spectroscopy (FTIR), together with determination of physical properties of the support (water content and density) and microbiological study, adapted for analysis, investigation and evaluation of the current status. The study revealed the severe damage of painting materials. Gypsum, ultramarine, smalt, prussian blue, red ochre, emerald green, red lead (minium) and gold leaves were identified in El-Ashraf Bersbay Madrasa decoration painting.

Keywords: ceilings, pigments, gilding, and painting ground, XRD, SEM-EDS, LOM, and FTIR.

-Introduction:

Madrasa of sultan Al-Ashraf Bersbay (826 H/1423A.D.)

Architecture was the preeminent art of the Mamluk period¹.on the other hand the Mamluk architectural decoration is distinguished not merely by its strong sense of color but also by its pervasive

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¹ Sheila S. B., and Jonathan M. B., :The art and architecture of Islam, 1250-1800, Yale University press, 1994, p. 8.

sculptural quality². The Madrasa of sultan, Al-Ashraf Bersbay is consider one of the best examples which represent this period.

This Madrasa is located on Al-Mu'izz street and is also known as "Al Ashrafiya", after sultan Al-Ashraf Bersbay. The Madrasa was completed in 831H/1427 A.D., its façade is located in the southeastern side that overlooks Al-Mu'izz street and its northern section ends with the mausoleum and the *sabil*. The entrance leads to square area where the *sabil* is located with an assembly hall facing it, and at the end, there are two doors, one leading to the *sabil* of the Madrasa, while the other leads to the courtyard of the school. The Madrasa consists of a major open courtyard surrounded by four iwans. The courtyard has four doors leading to different units of the Madrasa, such as the *Sufi* cells, the corridors and the bathrooms. The arches of the *iwans* have a Naskhi inscriptive band all around with the founder's name, sultan Al-Ashraf Bersbay, engraved on it.

The main *iwana* of the *qiblah* is distinctively larger in size (16.5 by 12.8 meters) and has a wooden ceiling, which is decorated with a beautiful paintings consisting of floral and geometric elements. The *iwana* overlooks the school courtyard through a large horse-shoe arch. The opposite *iwana* is smaller in size (11.2 by 8 meters) and has a wooden ceiling that dates back to the foundation of the school. It includes some gilded geometric ornaments and a large niche flanked by two smaller ones. The northern and the southern sides of the school have two smaller *iwans*.³

-Decoration of the wooden ceilings (techniques and materials):
Islamic painting "Paintings are composed of a wide range of organic and inorganic constituents"⁴ on the wooden ceilings is one of the finest manifestations of Islamic civilization. The paintings

² Robert, H.,: Islamic art and architecture, Times and Hudson, LTD, London, 1999, p.146.

³ Shiha, M., : The Islamic architecture in Egypt, archaeological series 5 prism publications Al - Ahram commercial press – Kalyoub-Egypt, 2001, pp.140-141.

⁴ Santos A., et al.,: Application of molecular techniques to the elucidation of the microbial community structure of antique paintings, Microb Ecol,58, 2009, pp.692–702.

were applied (*according to the Arab texts*) as follows: the wood panels are covered with a layer of chalk ground, bound with glue. The paintings are executed in a tempera technique “*tempera is painting that employs a medium that may be freely diluted with water but upon drying becomes sufficiently insoluble to allow over painting with more tempera or with oil and varnish mediums*”^{5,6}. In a civilization where learning and arts played an important role, great care taken in the manufacture of writing and painting materials.

- **Pigments:** the pigments are intensely colored and finely powdered solids used (mainly in paints) to impart color to other materials⁷. It is derived from the following categories: minerals, inorganic or artificial materials and organic, that is plant and animal sources. Different sources for pigments were mentioned in Arab texts^{8,9}.

White pigment came mainly from lead white, a basic lead carbonate ($2PbCO_3.Pb(OH)_2$), and at times bone white “*bone white is composed mainly (85–90%) of calcium phosphate mixed with calcium carbonate (13–9%), minor constituents making up the rest*”¹⁰. On the other hand, the number of white colors used in ancient Egypt was limited for long time to only two types of pigments, calcium carbonate ($CaCO_3$) obtained from the mineral “*Calcite*”, and calcium sulphate (as $CaSO_4$ or its hydrate $CaSO_4 \cdot 2$

⁵ Mayer R.,: The artist's handbook of materials and techniques, the Viking Press Inc., New York, 1991. p. 264.

⁶ Brania A.A.,: Analytical study of the decorative materials of the ceilings of the mosque of el-motaher (1744 A.D.) Cairo, Egypt, in: Egypt. J. Anal. Chem., 15, 2006, pp.200-201.

⁷ Goffe Z.,: Archaeological chemistry, John Wiley & Sons, Inc., Hoboken, New Jersey 2007, p.63.

⁸ Al-Hassan A.Y., and Hill D. R.,: Islamic technology an illustrated history, Cambridge University Press, UNESCO, Paris, 1986, 170-174.

⁹ Sheila R. Canby, S.R.,: Persian painting ,the trustees of British Museum , British Museum press , London,1993, pp.18-19.

¹⁰ Goffe Z.,: Archaeological chemistry, John Wiley & Sons, Inc., Hoboken, New Jersey 2007, p.68.

H_2O)¹¹ obtained from the mineral “Gypsum”. As for red pigments, they came mainly from cinnabar “mercuric sulfide (HgS)”, and red lead (Pb_3O_4). Red lead (also known as minium), a lead-based bright orange pigment, has been known since antiquity¹². It has been imported to Egypt by the Romans¹³. It was used widely in medieval manuscripts and has been identified on wall paintings, polychrome sculpture and panel paintings. However, the majority of red pigments used in ancient Egypt were earthen based colors containing iron oxide; especially the mineral Hematite (αFe_2O_3) which was very common. It can be applied on wood or on stone, and it can be used as well for skin-paintings which was common in some other cultures. These Fe-based colors are longer lasting and light faster than others, and are sometimes of astonishing brilliance^{14, 15, 16}. Lac, a dark red resinous incrustation deposited on certain trees by the *lac* insect was also used.

Yellow pigments were derived mainly from orpiment “arsenic trisulphide (As_2S_3) although yellow ochers “ forms of clay iron ores” were also used. Additionally, massicot “monoxide of lead (PbO)” mentioned in Arab texts as saffron, was employed together with other pigments¹⁷. Blue pigments considered as a one of the most popular pigments in Islamic decorated ceilings¹⁸, which, came

¹¹ Heywood, A.,: The use of huntite as a white pigment in ancient Egypt, in: Colour and painting in ancient Egypt, London, 2001, pp. 5-9.

¹² Gettens J. and Stout G.L.,: Painting materials, a short encyclopedia ,Dover, New York, 1991, pp.29 -133.

¹³ Katja H.,: Characterization of pigments and colors used in ancient Egyptian boat models, Spectrochimica Acta, Part B 61, 2006, pp.1224-1228.

¹⁴ Green L., : Colour transformations of ancient Egyptian pigments, in: Colour and painting in ancient Egypt, London, 2001, pp. 43-48.

¹⁵ Uda M., et al.: Yellow, red and blue pigments from ancient Egyptian palace painted walls, NIM B 161-163, 2000, pp.758-761.

¹⁶ Katja H.,: Characterization of pigments and colors used in ancient Egyptian boat models, Op cit. pp. 1224-1228.

¹⁷ -Ramadan, Z.S.,: The wooden ceilings in the ottoman period, master thesis, Islamic dept. , Faculty of Archaeology, Cairo University, 1992, pp. 25-112.

¹⁸ Brania A. A., Analytical Study of the blue Pigments in some Islamic monumental decorated Ceilings in Cairo, Egypt, J. Anal. Chem.,15, 2006, pp.189-199 .

from the mineral lapis lazuli, though *azurite* (a form of copper carbonate) was also used, together with *Prussian blue* (iron blue ferric-Ferrocyanide $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$), as were *Smalt* and *indigo* (dye). *Lapis lazuli* "ultramarine blue, sodium sulfosilicate" ($3\text{Na}_2\text{O}_3 \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 3\text{Na}_2\text{S}$ (NATURAL) and $\text{Na}_{8-10}\text{Si}_6\text{O}_{24}\text{S}_{2-4}$ (ARTIFICIAL), its present name ultramarine, derives from *azrrum ultramarinum* or *azurro oltramarino* that formerly served to distinguish it from azurite.

Ultramarine has been for centuries one of the most highly prized pigments of all traditional artists' materials due to its durability, excellent color, and its intrinsic value. It is made from the mineral lapis lazuli and archaeological evidence shows that this mineral was used as a semi-precious stone and decorative building stone from early Egyptian times. In the late sixteenth and the seventeenth century, it has been noted that there was a shortage of the other most valuable blue pigment, azurite, which must have resulted in increased demand for that already costly ultramarine, thus making it even more precious and expensive. Since the price of this extraordinary pigment was sometimes even higher than that of gold, the motivation for producing a synthetic version accelerated the quest for a more favorably-priced substitute and the first synthetic manufacturing of the ultramarine pigment succeeded in 1828. Because of their almost ten times lower price, they are being widely used in nearly all of the art works today, even though some critics claim they are less pure and less permanent than the pigment obtained from lapis lazuli¹⁹. On the other hand, azurite ($2\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$) was also used, as were smalt .

The use of smalt in painting dates back to the period between the mid fourteenth and early nineteenth centuries. On the other hand, cobalt ores were used for coloring glass in Egyptian and classical

¹⁹ Desnica V., et al.,: Multi analytical characterization of a variety of ultramarine pigments, e-PS, 1, 2004, pp. 15-21.

times .The earliest attested use of smalt as a blue pigment was in the early post-Byzantine period²⁰ ,“*smalt (Artificial in nature which is a potash silicate SiO_2 , K_2O , As_2S_3 , CoO , and Al_2O_3) was the earliest of the cobalt pigments. It is moderately fine to coarse ground potassium glass of blue color; the blue is due to small but variable amounts of cobalt added as a cobalt oxide during manufacture. Since smalt is a coarsely ground glass it can easily recognized at low magnifications. The particles show conchoidal fracture and thin sharp edges of glass splinters*”^{21, 22}. Prussian blue “*iron blue ferric-Ferrocyanide $Fe_4[Fe(CN)_6]_3$* ” and indigo were also used for this purpose.

Green pigments were mainly derived from basic copper carbonate verdigris (zinjar) and from mineral malachite. In addition, different greens, including those with plant-like hues, were manufactured by mixing other varieties of pigments.

The pigment identification is so important for conservators and restorers’ .There are two key reasons to be concerned with pigment identification. The first is to ensure that all restoration is carried out with the original pigment and not with alternatives of similar hue. This is important because the alternatives may be liable to react with contiguous pigments with disastrous visual effects. The second is to identify any degradation products of pigments and to suggest possible treatments whereby degradation processes may be either arrested or reversed²³.

-Media: Paints thinned with water have a long history. From the earliest times artists decorated surfaces with pigments bound in adhesives “*If they were water – based, all these pigments required a*

²⁰ Daniilia S. and Minopoulou E.,: A study of smalt and red lead discolouration in *Antiphonitis* wall paintings in Cyprus Appl. Phys A.,96 ,2009, pp 701-711.

²¹ Muhlethaler B., and Thissen J.,: Identification of the materials of paintings, smalt, studies in conservation, 14, 1996, pp. 47- 61.

²² Altavilla C. and Ciliberto E.,: Decay characterization of glassy pigments: an XPS investigation of smalt paint layers Appl. Phys. A 79, 2004, pp. 309-314.

²³ Clark. R. J.H., : Pigment identification by spectroscopic means: an arts/science interface, C. R. Chimie, 5, 2002, pp. 7-20.

binding medium, which usually mixed with the pigment” i.e. (Gums “acacia or Arabic”, Glues, and egg white or yolk”). Gum Arabic was the most common binder, though glues especially fish glue and glair were employed as well. Paintings were protected with a varnish made through a typical recipe of the tenth century A.H. (Sixteenth century A.D.) which involved adding a naphtha solvent to a thick mixture of sandarac and linseed oil. The solution was applied two or three times to the surfaces being protected.

- Agents of deterioration and the decoration condition:

The decorative paintings on the Madrasa ceilings are in a very poor condition. Almost 70% of the original painting is insufficiently attached to the wooden ground. Different deterioration aspects were found on the studied painted ceilings (i.e. cracks and micro-cracks, flaking, infiltration of rainwater through the roof, biodeterioration and inadequate previous interventions).

The decoration were covered with numerous and different layers of dirt. The previous intervention is rather dark and dirty due to the accumulation of airborne deposits from different sources on the surface of the paintings. This has caused the formation of a dark film which obscure and deadens the original colors. Some areas of the paintings have been damaged or suffer from missing parts, as a result of the entry of humidity by infiltration through the much deteriorated roof. Signs of infiltration can be observed in several areas in the decoration of the ceilings. The most serious areas exist in the *qiblah iwan*. These infiltrations have caused a brown to black staining, a partial washing away of the painted decoration, and some detachment of the painting layers from the wooden support.

The previous interventions have played a role in the deterioration of the paintings. Extensive damage unfortunately was done to the painting enacted in the hope of helping to stabilize the structure and the decoration layers. Most of the decoration had already been removed and replaced with a new one. From the architectural point of view, the intervention was not successful and perhaps was

inappropriate. The painting material of the Madrasa exposed to different biogenic and abiogenic stresses under generally aggressive climatic conditions²⁴. the main degradation induced by diverse living organisms “fungi”. *Fungi are among the most active microorganisms which play an important role in the deterioration process. In general it is considered that fungi can grow at rather low water levels, while bacteria and algae grow at higher moisture*²⁵. *Growth of microorganisms on paintings and other materials may cause aesthetic and structural damage*²⁶. *The nature of the support will determine the type of degradation. The alteration mechanisms are different on organic supports and on inorganic material due to the heterotrophic nutrition of fungi. Heterotrophic microorganisms may be fulfilled by remains of polluted air and rain or animal remains and secretion*^{27, 28, 29}. *While fungi can use the organic material itself as nutrients, in the case of inorganic supports these are transformed by several metabolites which are excreted and that may react with the support in different ways*^{30, 31}. *Many of these fungi are responsible, along with other chemical and*

²⁴ Mohammadi P. and Krumbein W.E., : Biodeterioration of ancient stone materials from the Persepolis monuments (Iran) *Aerobiologia* ,24, 2008, pp. 27–33

²⁵ Petersen K.,: wall paintings: Aspects of deterioration and restoration in: conservation science heritage materials , edited by Eric May and Mark Jones, the royal society of chemistry,2006, p.247.

²⁶ Milanesi C.,:Fungal deterioration of medieval wall fresco determined by analyzing small fragments containing copper, *international biodeterioration & biodegradation* ,57 , 2006, pp. 7-13.

²⁷ Sterflinger K., Fungi: Their role in deterioration of cultural heritage, *fungi biology reviews*, 24, Issues 1-2, February-May 2010, pp. 47-55.

²⁸ Suihko, L. M., et al.,: Characterization of aerobic bacterial and fungal microbiota on surfaces of historic Scottish monuments, *Syst. Appl. Microbiol.*,30, 2007, pp. 494-508.

²⁹ Milica V.,: Role of fungi in biodeterioration process of stone in historic buildings, *zbornik matice srpske za prirodne nauke / proc. nat. sci, matica Srpska novi sad*, no 116, 2009,pp.245-251.

³⁰ Arroyo I.,: The role of fungi in the deterioration of movable and immovable cultural heritage , *e-conservation*, 2007, p.748.

³¹ Sharma K., et al.,: Fungal involvement in biodeterioration of ancient monuments: problem and prospects, *Journal of Phytology* ,3(4), 2011, pp.15-17.

biological factors, for the formation of black crusts due to the melanin in their hyphae. The hyphae of the fungus can penetrate the mineral crystals previously dissolved by enzymes. Some fungi are called endolithic because they penetrate into the substrate causing "pitting", a surface that appears to have many small holes. Furthermore old insect damage was found in the wood of the studied ceilings also.

From the point of view of the preservation of the Madrasa paintings, the work enacted can only be described as a catastrophe. Approximately 50 to 60 % of the decorations of the ceilings were destroyed during the previous interventions. The deleterious effect described is shown in figures 3-8, 46 and 48.

-Materials and methods:

Painting materials samples were collected "pigments, gilding and painting ground" from the decorated ceilings of *qiblah* (QC) and the against *qiblah* ceilings (AQC), for analysis investigation and evaluation the status. All samples of the painting materials were taken from areas of ceilings that were already damaged, to avoid disfiguring the patterns. Selected samples can be considered as representative of the painting materials .Their size was sufficient for both the preparation of cross-sections needed for LOM and SEM-EADX, and for the FTIR analysis.

-Methods of analyses and investigation:

The identification of materials used in artworks is of great importance for conservation, restoration, and comprehensive study of our historical and cultural heritage³².The analysis, in fact, gives information useful in defining the gamut of pigments available at a local, regional or even wider scale and to understand the techniques of color preparation and application. In addition, through the study of pigments, it is possible to discover the lines of communication

³² Castanys M. et al., Automatic identification of artistic pigments by raman spectroscopy using fuzzy logic and principal component analysis, Laser Chemistry, 2006, Article ID 18792, 8 p.1.

and trade exchange³³. Characterization and analysis should preferably be done using non-invasive, non-destructive methods. In some cases, micro-samples (a few milligrams) are allowed to be taken³⁴.

The methods and techniques used in the study were X-ray powder diffraction (XRD), light optical microscope (LOM), scanning electron microscopy (SEM) equipped with an EDAX microanalysis detector, fourier transform infrared spectroscopy (FTIR) and microbiological study. Most of which are non-destructive; and suitable for the determination of anionic groups, crystalline phases, structure and elemental composition which allowed us to have a complete characterization of the used pigments, materials and techniques.

X-Ray Diffraction (XRD): A Phillips X-ray diffraction equipment model pw/1840 with Ni filter, Cu radiation 1.54056 Å at 40 KV, 25mA, 0.05 /sec, (laboratories of the national research centre, Cairo).

Light Optical Microscope (L.O.M.): LOM Zeiss standard microscope was used to investigate surface samples from the pigments and the decorated plaster, (Conservation Dept. Faculty of Archaeology, Cairo Uni.).

Scanning Electron Microscope (SEM EDAX): The scanning electron microscope photographs and microanalyses were carried out by utilizing S.E.M. Philips XL 30 attached with EDAX unit, with accelerating voltage 30 K.V., magnification 10X up to 400.000X and resolution for W. (3.5nm). For the purpose of painting materials identification the elemental composition was determined using the prepared carbon coated sample.

Fourier transform infrared spectroscopy (FTIR): Fourier transform infrared spectroscopy (FTIR) was carried out using a

³³ Mazzocchin G.A., et al.,: Analysis of pigments from roman wall paintings found in Vicenza, Talanta 61, 2003, pp.565- 572.

³⁴ Mihaela C., et al.,: Degradation of lime wood painting supports e-ps, 2005, 2 , pp.19-29 .

Perkin–Elmer Spectrum one instrument (Laboratories of the national research centre, Cairo). Spectra were recorded in the 4000–100 cm^{-1} region.

Microbiological study. Isolation of fungi from the studied decoration material was carried out in the national research centre in Cairo.

- Results & Discussion:

-X-ray diffraction method (X.R.D): XRD represents a very effective tool for indisputable determination of practically all inorganic crystalline pigments and materials^{35, 36}. X-ray diffraction method (X.R.D) was adapted for analysis of the painting ground sample, (Fig.9). Powder of X.R.D., show the painting ground is composed principally of gypsum mineral (calcium sulphate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) as a major components and a small amount of calcite mineral (calcium carbonate CaCO_3) as a minor. Calcium sulphate can appear as three distinct minerals: gypsum (dihydrate), bassanite (hemihydrate) and anhydrite (anhydrous)³⁷. The presence of gypsum as a major component indicates to the on purpose using as a painting ground. Gypsum can dehydrate to a lower hydrate, the hemihydrate, and to an anhydrous phase, anhydrite. Therefore there is a possibility of the dehydration–hydration reaction playing a critical parts in the deterioration mechanism of the painting ground and the pigments itself of the decorated surfaces. On the other hand, the presence of calcium carbonate as a minor component comes from the intentional addition of gypsum to improve the work ability during surface preparation.

³⁵ Svarcova S., et al., :Micro-analytical evidence of origin and degradation of copper pigments found in Bohemian Gothic murals, anal bioanal Chem. ,395,2009, pp. 2037-2050.

³⁶ Pagès-Camagna S., et al.,: Non-destructive and in situ analysis of Egyptian wall paintings by X-ray diffraction and X-ray fluorescence portable systems, Appl Phys A ,100, 2010, 671-681.

³⁷ Charola A. Elena, et al., : Gypsum: a review of its role in the deterioration of building materials Environ Geol vol., 52, 2007 pp. 339-352.

-Scanning electron microscope S.E.M (EDAX): scanning electron microscope (SEM) photomicrographs and microanalyses of the studied samples are as follows, (table no.1)

-White pigment with deteriorated painting ground (QC): result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big quantity of Ca and S (representing gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ as a main component and small amount of chalk CaCO_3), significant amounts of Na, and traces of Si, P, Cl and (representing the accompanied impurities) (fig. 10,11).

-Brown pigment with deteriorated painting ground (AQC): result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big quantity of Pb (representing red lead "Minium" Pb_3O_4 as a main component), significant amounts of Cl, and traces of Al, Si, K, P, and Ca (representing the accompanied impurities). This kind of pigments has the tendency to darken in water color and wall paintings. The formation of lead dioxide is the cause of this darkening. As for the transformation of red lead from orange Pb_3O_4 to black PbO_2 , it is assumed that red lead degradation may have been induced not only by the effect of temperature, light and humidity but also by the presence of chlorine salts. The darkening of lead tetroxide, however, may not be a simple matter of alteration to plattnerite. Moreover, exposure to light, rain and atmospheric carbon dioxide can lead to the formation of the basic lead carbonate (lead white), which gives a chalky surface³⁸. Minium is liable to discolor in the presence of hydrogen sulphide or reacts when mixed with sulphide containing pigments leading to the formation of black lead sulphide (Fig.14, 15). On the other hand, atmospheric pollutants (SO_2 , CO_2) together with water condensation cause fading of red pigment. Red led pigment can also transform into both

³⁸ Daniilia, S., and Minopoulou E.: A study of smalt and red lead discolouration in *Antiphonitis* wall paintings in Cyprus Op.cit , pp 701-711.

cerussite (lead carbonate $PbCO_3$) and anglesite (lead sulphate $PbSO_4$).

Table no.1 shows the SEM (EDAX) samples and the obtained results.

Kind of sample.	Location	SEM (EDAX) Results of pigments.	Fig. no.
-White pigment with DPG.	(QC)	Gypsum ($CaSO_4 \cdot 2H_2O$) +some impurities.	10,11
-Brown pigment with DPG.	(AQC)	Red lead "Minium" (Pb_3O_4) + some impurities	14,15
-Red Pigment with DPG.	(AQC)	Hematite (αFe_2O_3 red ochre) + some impurities	18,19
-Red Pigment on canvas with DPG.	(AQC)	Hematite (αFe_2O_3 red ochre) + some impurities	20,21
-Blue Pigment on canvas with DPG.	(QC)	Prussian. Blue, (Iron blue ferric- Ferro cyanide $Fe_4 [Fe (CN)_6]_3$) + some impurities.	22,23
-Blue Pigment on canvas with DPG.	(AQC)	Smalt ($SiO_2, K_2O, As_2S_3, CoO, Al_2O_3$ and Prussian. Blue, (Iron blue ferric- Ferro cyanide $Fe_4 [Fe (CN)_6]_3$, + some impurities.	25, 26
-Blue Pigment with DPG.	(AQC)	Ultramarine (sodium sulfosilicate $Na_6-8 Al_6 Si_6 O_{24} S_2-4$) and Prussian. Blue, (Iron blue ferric- Ferro cyanide $Fe_4 [Fe (CN)_6]_3$.	29-31
-Green Pigment with DPG.	(QC)	Copper (II)-acetoarsenite or emerald green $Cu(CH_3COO)_2 \cdot 3 Cu(AsO_2)_2$ + some impurities.	33-34
- Brown pigment with DPG.	(QC)	Red lead "Minium" (Pb_3O_4) is the main component+ some impurities	38-39
- Gilding with DPG.	(QC)	Gold (Au) is the main component + some impurities.	41,42
- Gilding with DPG.	(AQC)	Gold (Au) is the main component +some impurities.	44,45
-Painting and gilding grounds.	(QC)	Gypsum ($CaSO_4 \cdot 2H_2O$) and calcite ($CaCO_3$) are the main components + some impurities.	49- 51

N.B., DPG is a deteriorated painting ground.

-Red Pigment with deteriorated painting ground (AQC): result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big quantity of Ca, S, and Ba representing gypsum and barium sulfate as a painting ground and a major component; this is due to the severe damage of the red pigment represented in Fe (red

ochre "hematite $\alpha\text{Fe}_2\text{O}_3$ "). The pigment is scattered on the surface sample and therefore has a very bad hiding power. On the other hand, significant amounts of Si and Cl, and traces of Al, K, and Na (representing the accompanied impurities have been detected as well (fig.18,19).

-*Red Pigment on canvas with deteriorated painting ground (AQC):* The result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big quantity of Ca, S and Ba representing gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ as a main component. Significant amounts of Fe (red ochre "hematite $\alpha\text{Fe}_2\text{O}_3$ as a deteriorated pigment"), and traces of Si and Cl, (representing the accompanied impurities) were also detected (fig. 20,21).

- *Blue Pigment on canvas with deteriorated painting ground (QC):* result of SEM (EDAX) microanalyses and the attached photomicrograph revealed the presence of a big quantity of Ca, and S representing gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ as a main component of the painting ground. Moreover significant amounts of Al and Si and traces of Na Cl, K, and Mg representing the accompanied impurities were detected as well. On the other hand, Fe is scattered on the sample surface representing Prussian blue (iron blue ferric- ferro cyanide $\text{Fe}_4 [\text{Fe} (\text{CN})_6]_3$) in a very bad condition (fig. 22, 23).

-*Blue Pigment on canvas with deteriorated painting ground (AQC):* result of SEM (EDAX) microanalyses and the attached photomicrograph revealed the presence of Si, Al, K, As, Co representing the known blue pigment smalt (SiO_2 , K_2O , As_2S_3 , CoO , Al_2O_3). This pigment is a coarsely ground glass which can easily be recognized at low magnifications. The particles show conchoidal fracture and thin sharp edges of glass splinters³⁹. On the other hand, the detection of Fe indicates the presence of prussian blue (iron blue ferric- ferro cyanide $\text{Fe}_4 [\text{Fe} (\text{CN})_6]_3$, which was added to smalt to improve the color, (fig. 25, 26).

³⁹ Muhlethaler, B., and Thissen, J.: Identification of the materials of paintings, Op. cit, pp. 47:61.

-*Blue Pigment with deteriorated painting ground (AQC)*: result of SEM (EDAX) microanalyses and the attached photomicrograph revealed the presence of a big quantity of Ca, S representing gypsum as a painting ground. Significant amounts of Si and Al and traces of Na, Mg, Cl, K and Fe were also detected. The previous elemental composition and the morphological feature are due to the presence of ultramarine blue pigment ($\text{Na}_6\text{Al}_6\text{Si}_6\text{O}_{24}\text{S}_2\text{S}_4$). From the inorganic blue colors, only ultramarine contains sulphur in the crystals and for this reasons its color is blue⁴⁰. The variety of the elements and especially the presence of S and Cl lead us to the conclusion that the pigment is natural ultramarine.

On the other hand, Fe indicates the presence of Prussian blue; iron blue ferric-ferrocyanide $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$ was added intentionally (fig. 29-31).

-*Green Pigment with deteriorated painting ground (QC)*: result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big quantity of Ca and S (representing gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (painting ground), significant amounts of As, and Cu (Copper(II)-acetoarsenite $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 3\text{Cu}(\text{AsO}_2)_2$ ⁴¹) and traces of Si, P, Al, Mg, Ba and Fe and representing the accompanied impurities (fig. 33:34). On the other hand the artificial pigment Copper (II)-acetoarsenite or emerald green “*The name Emerald green comes from Latin smaragdus = precious stone*”, was first produced commercially by the firm of *Wilhelm Sattler at Schweinfurt*, Germany in 1814. That means it is not from the original painting of El-Ashraf Bersbay Madrasa, which dates back to 1423. Based on the previous result, it can assume that the pigment

⁴⁰ Zorba T., et al.,: Technique and palette of XIIIth century painting in the monastery of Mileseva, Appl. Phys. A 83, 2006, pp.719-725 .

⁴¹ This copper aceto-arsenite pigment was first produced commercially by the firm of Wilhelm Sattler at Schweinfurt, Germany in 1814. Justus Von Liebig and Andre Braconot separately published papers on its method of manufacture. Von Liebig's paper "Sur une couleur verte" was published in 1823 in Annales de chimie XXIII (pp. 412-3). Verdigris (or acetic acid) was dissolved in vinegar and warmed. A watery solution of white arsenic was added to it so that a dirty green solution was formed. To correct the color, fresh vinegar was added to dissolve the solid particles. The solution was then boiled and bright blue-green sediment was obtained. It was then separated from the liquid, washed and dried on low heat and ground in thirty-percent linseed oil. The pigment was considered a good drier.

is a new one (new intervention). This previous interpretation was confirmed by LOM examination (fig. 35-37).

-Brown pigment with deteriorated painting ground (QC): The result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big quantity of Pb representing red lead “Minium” Pb_3O_4 as a main component, significant amounts of Cl, Ca and K and traces of Si, and Na representing the accompanied impurities. This kind of pigments has the tendency to darken as previously mentioned during the interpretation of the brown pigment of *AQC* (fig. 38, 39).

-Gilding with deteriorated painting ground (QC): The result of SEM (EDAX) microanalyses and the attached photomicrograph shows on one hand, gold leaves (Au) is the main component plus Ca, Al and Si as accompanied impurities, (fig. 14,42). Gilding is the technique of applying a thin sheet, most commonly gold, over a firm support. This practice comprises a multitude of different methods, which can be carried out on a great variety of substrate materials Gilding is easily differentiated from cheaper decorative alternatives, such as gold paint. Gilding appears as a solid surface whereas paint will appear more granular, streaky and dull in appearance by comparison. Whilst gold leaf does not tarnish, paint oxidizes and consequently becomes a green-brown color on ageing^{42, 43}.

-Gilding with deteriorated painting ground (AQC): result of SEM (EDAX) microanalyses and the attached photomicrograph shows gold leaves (Au) is the main component plus Ca as impurities, (fig. 44, 45).

-Painting and gilding grounds (QC): result of SEM (EDAX) microanalyses and the attached photomicrograph revealed a big

⁴² Olga K. and, Russell F.H.,: Microscopic, mass spectrometric and spectroscopic characterization of the mordants used for gilding on wall paintings from three post-Byzantine monasteries in Thessalia, Greece, *Microchemical Journal* ,94, 2010, pp. 83-89.

⁴³ Moses J.: *Gilding techniques care, and maintenance*, published by Technical Conservation, Research and Education Group, Edinburgh January, 2007.

quantity of Ca, and S representing gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and calcite CaCO_3 as the main components of the painting ground, traces of Na, Al, Si, Mg and Fe representing the accompanied impurities (fig. 49- 51), which confirm the result obtained by XRD analyses.

- **Light Optical Microscope (LOM.):** LOM was used on one hand for examination of the characteristic samples and thin cross-section of pigments, gilding and painting ground. On the other hand, it was also used for the decorative wood ceilings determination. Zeiss standard microscope was used for the collected sample examination. Samples were covered with linseed oil to facilitate viewing the grain size distribution. The results in general, revealed the presence of a thick superficial layer of deterioration aspects and fading of the studied pigments samples. Widespread loss of pigments together with gilding has been detected in the background of the paintings. The under paint in the background of the paintings is preserved, whereas the upper layer of the pigments is almost lost. Initially, this finding was ascertained by close observation of the paint and gilded surfaces and examination of the corresponding cross sections.

The cross-section revealed also the previous intervention which was done by adding new pigments to the ceilings (over painting). The previous intervention was so clear in the green pigments, which cover the blue, on the *qiblah* ceiling (QC). Some pigments (the red lead) have taken on a brownish hue as superficial layer (transformation of red lead, because of directly exposed to adverse environmental conditions.). On one hand from the investigation of the gold samples the occurrence of four different layers was discovered. The first layer is a metallic layer; the second intermediate layer probably is an organic layer, with an average 8 μm thickness. The third, a deteriorated preparation layer for gilding. The fourth one is the deteriorated wooden support (insect infected...etc).

.On the other hand, the surface of the gilded samples generally shows an extensive net of "craquelure" (Fig.12, 13, 16, 17, 24, 27, 28, 32, 35:37, 40, 43, 47.).

For the determination of the decorative wood ceilings, a representative wooden sample was prepared by making longitudinal l, cross and flat – surface sections. The result of examination revealed that *Pinus Leucodermis Ant. (Pinaceae)* was used (water content of the studied wooden sample 14.8 “the standard from 12-15%⁴⁴”, and density 0.47 gr.cm.³ respectively).

-Fourier transforms infrared spectroscopy (FTIR): In the analysis of painting materials, infrared spectrometry is adapted for analyses of many pigments, binders and varnishes. Many organic compounds with similar chemical composition and structures have similar pattern of in the IR range. This is true in the case of protein containing binders, such as glue, egg white (glair) and yolk. Thus, this instrumental technique is useful for the identification of the general class of a binder, but not usually for specific binder identification,^{45, 46} The most modern generation of infrared spectrometers is called “Fourier transform”. The binding media in three samples (pigments and gilding) from both *qiblah* and against *qiblah* ceilings were characterized using FTIR (*FTIR advantage, is the capability of identifying both organic and inorganic compounds*). Since the percentage of organic material existing in each sample is extremely small, the interpretation of the results of FTIR analysis is very complex. FT-IR results revealed that the binders are severely deteriorated protein compounds (in the course of time, the varied environmental aggressions are conducive to

⁴⁴ Roger. M ., : Moisture Properties, in : Handbook of wood chemistry and wood composites, Part II, C.R.C press, 2005.

⁴⁵Van den Berg, K. J., et al.,: Darkening and surface degradation in 19th-and early 20th-century paintings: an analytical study. In: 13th-Triennial Meeting Rio de Janeiro 22-27 September, 2002, p.469.

⁴⁶Stanley Taft, Jr. w. and Mayer W. J., : The science of paintings, Springer, New York, 2000 , pp.171- 173

deterioration) indicating to the glue “*The characteristic absorption bands of glue 3400-3200 cm⁻¹ , N-H stretching band, 3100-2800 cm⁻¹, C-H stretching bands 1660-1 600 cm⁻¹ , C=O stretching band 1565-1500 cm⁻¹ , C-N-H bending band 1480-1300 cm⁻¹ and C-H bending band*”. On the other hand, absorption bands on the three samples indicate the presence of both calcium sulfate (CaSO₄) with S–O stretching in the 1200-1050 cm⁻¹ region^{47, 48}, 1140-1080 cm⁻¹ asymmetric SO₄ stretching band 3700-3200 cm⁻¹ anti symmetric and symmetric O–H stretching bands. Weak C–H stretching bands are observable, in both B and C samples, around 3000–2800 cm⁻¹ and could indicate the presence of organic materials. The presence of an amide I carbonyl stretching band around 1650 cm⁻¹, which would indicate proteins, is difficult to ascertain due to the O–H bending of gypsum (CaSO₄·2H₂O). On the other hand, the presence of calcium sulphate and carbonate content as preparation layer and surface accumulations is reflected in an important band in the FTIR spectrum, caused by sulphate and carbonate groups. This very strong band could be overlapping others^{49, 50} which are precisely the interval where the absorption of some characteristic functional groups occurs that would unquestionably contribute to a greater understanding of the nature of the existing organic medium. The interpretation of some of the bands of the spectra and consequently any conclusion as to the nature of the organic medium was very difficult, (Fig.52 A-C).

These results indicate a mixture of gypsum and calcium carbonate as main components of the studied three samples. While the

⁴⁷ Michele R. et al., : Infrared Spectroscopy in Conservation science, The Getty Conservation Institute 1999, p.117.

⁴⁸ Brania A. A., :Gilding in Islamic monumental decorated ceilings in Cairo: analytical study, journal of Arab Archaeologists, vol. 9, 2009, pp.1-15.

⁴⁹ Bouchard M., et al.,: Micro-FTIR and micro-raman study of paints used by Sam francis, e-PS,6. 2009, pp. 27-37.

⁵⁰ Martin C. et al.,: Stratigraphic analysis of organic materials in wall painting samples using micro-FTIR attenuated total reflectance and a novel sample preparation technique, Anal Bioanal Chem,392, 2008 pp. 77-86.

presence of organic materials is suspected, absorption bands from original inorganic components preclude firm conclusions regarding the nature of the binding medium within the pigments and the painting ground layers.

- **Microbiological study** (Isolation of Fungi): sample of decorated and deteriorated wood was taken from the decoration. The sample was placed on malt agar and nutrient agar in Petri dishes. The Petri dish kept in an incubator at 30:37°C with 80% R.H. After isolation, the micro-organisms were identified by their morphological characteristics as *Aspergillus sp. (Aspergillus Unguis)*. It occurs in and upon the greatest variety of substrate, including cotton textiles, leather, dairy products and other protein-rich substrate .It is very common in many types, subject to moist conditions⁵¹. These fungi are not only responsible for color changes and formation of colored spots and dark crusts on the surfaces (yellowish or blue- green patches), but also they are highly destructive to the wood and the painting due to their chemical and physical properties “The ability of fungi to produce pigments and organic acids is crucial for the discoloration and degradation⁵² “.

Fungicides testes: Two fungicides selected for the experimental study as follows – mercuric chloride ($HgCl_2$) 0.5gm / L 0.05 % and PCP “pentachlorophenol” (20 gm / L) 2 %. The first was the best for inhibition (fig.53, 54).

-Conclusion: The present study has shown that the Madrasa of Sultan Al-Ashraf Bersbay is considering as one of the best examples, representing the distinguished Mamluk architectural decoration in Cairo. The Madrasa consists of a major open courtyard surrounded by four iwans. The decorations (paintings) are

⁵¹ Moubasher, A.H.,: Soil fungi in Qatar and other Arab countries, scientific and applied research center, university of Qatar, 1993.

⁵² Gupta, S.P. and , Sharma K., Biodeterioration and preservation of Sita Devi temple,Deorbija, Chhattisgarh, India international journal of conservation science, 2, Issue 2, 2011pp. 89-94

executed in a tempera technique. On the other hand water gilding was also used for decoration according to the Arab texts and the analyses. The decorative paintings and gilding on the Madrasa ceilings are in a very poor condition. Almost 70% of the original painting is insufficiently attached to the wooden ground. Different deterioration aspects were found on the studied painted ceilings (i.e. cracks and micro-cracks, flaking, infiltration of rainwater through the roof, biodeterioration and inadequate previous interventions). Most of the decoration had already been removed and replaced with a new one. The analyses and investigation of the decorative material revealed gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and a small amount of calcite (calcium carbonate CaCO_3) are the painting ground components. - Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) was also used as a white pigment. For the blue pigments in the Madrasa, the SEM-EDAX revealed, Smalt (SiO_2 , K_2O , As_2S_3 , CoO , Al_2O_3) is the main blue pigment. Ultramarine ($\text{Na}_{6-8} \text{Al}_6 \text{Si}_6 \text{O}_{24} \text{S}_{2-4}$) was used sometimes. On the other hand Prussian blue ($\text{Fe}_4 [\text{Fe}(\text{CN})_6]_3$) is used as a pigment alone and mixed sometimes with both of Smalt and Ultramarine (as a previous intervention). Minium (Pb_3O_4) and Red ochre ($\alpha\text{Fe}_2\text{O}_3$) were used as red and brown pigment. Copper (II)-acetoarsenite or emerald green $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 3 \text{Cu}(\text{AsO}_2)_2$ was used as a green pigment, which cover the blue pigments (over painting from previous intervention). Gold leaves were used for water gilding in the Madrasa. On the other hand much deteriorated glue is the main binder for the pigments and for the water gilding process. *Pinus Leucodermis Ant. (Pinaceae)* was the kind of used wood (Water content 14.8 % and Density 0.47 gr.cm³). *Aspergillus ungius* was detected from the microbiological study for the deteriorated decorated wood. For inhibition, mercuric chloride HgCl_2 0.5 %, was the best, compared with PCP "Pentachloro phenol" 2 %.



Fig.1 shows the main façade of Al- Ashraf Bersbay Madrasa .



Fig.2 shows plan Al- Ashraf Bersbay Madrasa ,” notice the main two iwans “.



Fig. 3 Shows the AQC., decoration of Al- Ashraf Bersbay Madrasa.

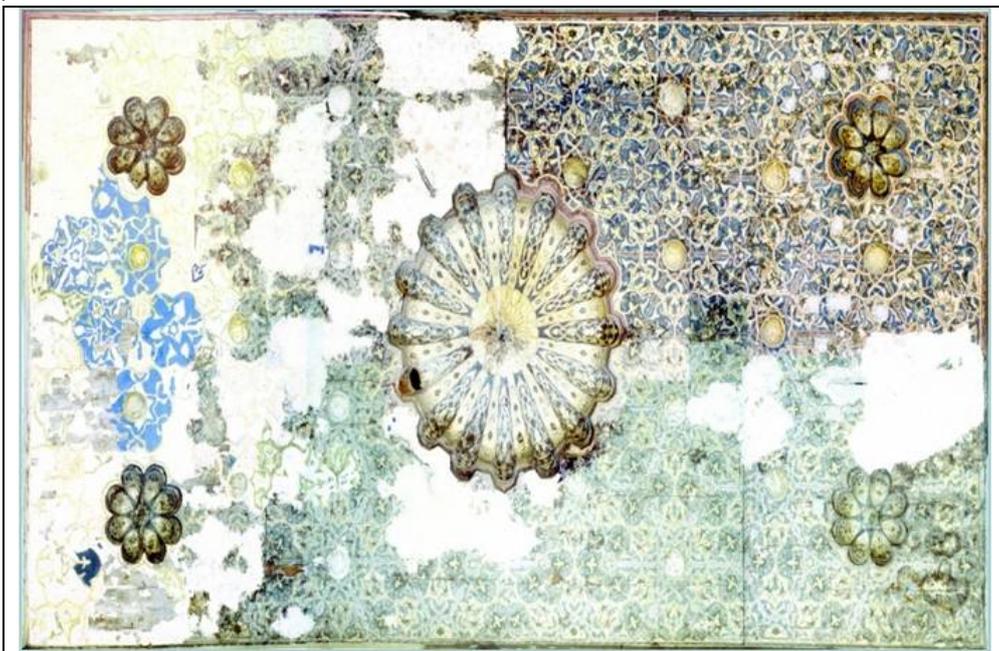


Fig. 4 Shows the AQC., decoration of Al- Ashraf Bersbay Madrasa. "notice its dramatic status because of the previous intervention etc..".

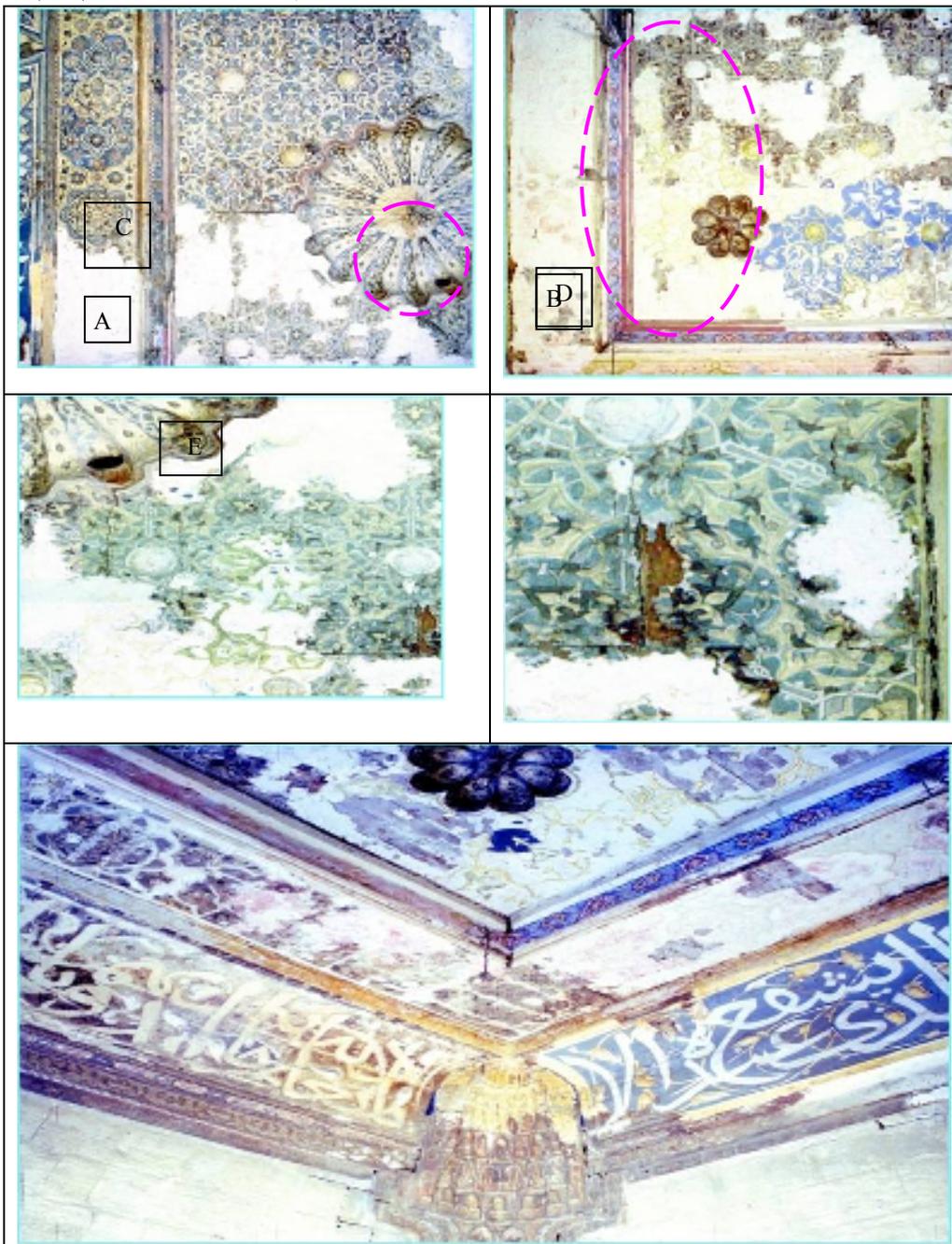


Fig. 5 A- E. Shows details from the AQC., decoration of Al- Ashraf Bersbay Madrasa, “notice the previous intervention and the very bad need of restoration “.

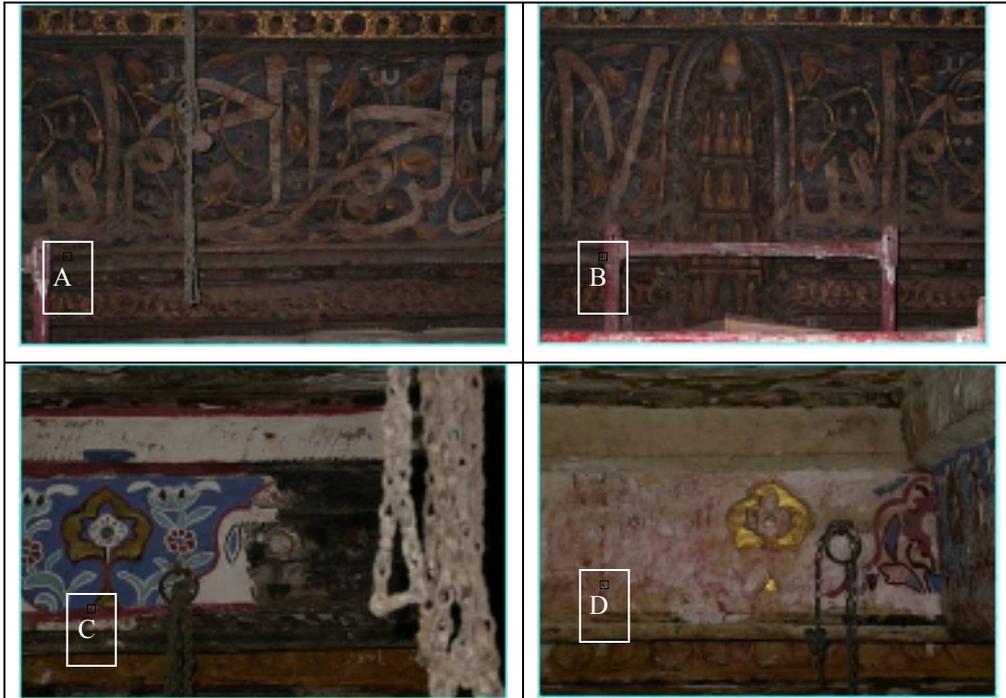


Fig. 6 Shows details from the AQC, decoration of El- Ashraf Bersbay Madrasa, “notice the unfinished previous intervention and the dramatic status “.



Fig. 7 A- D Shows the QC, decoration of Al Ashraf- Bersbay Madrasa, “notice the severe deterioration “.



Fig.8 A, B., Shows, details from the Q.C. decoration of Al Ashraf Bersbay Madrasa and its very bad status.

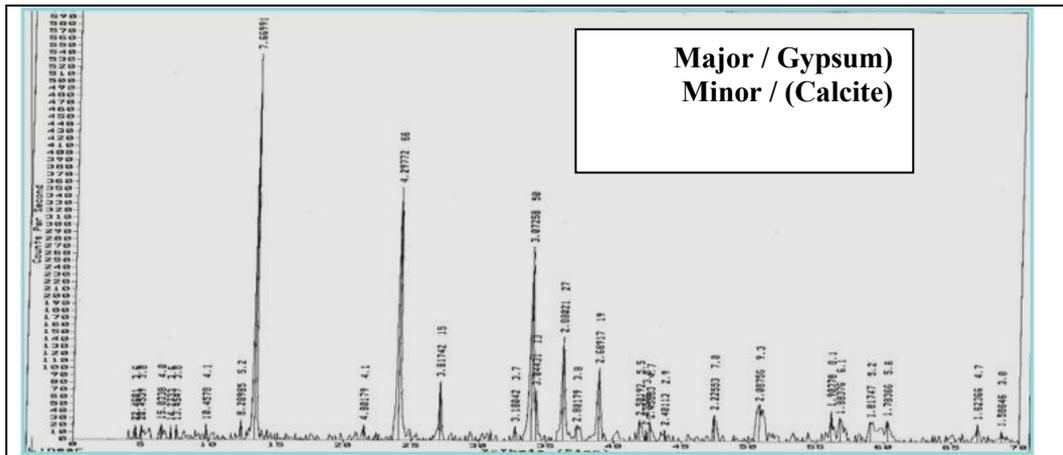


Fig. 9 Shows X.R.D. pattern of the painting ground sample from the and its components”QC.”,

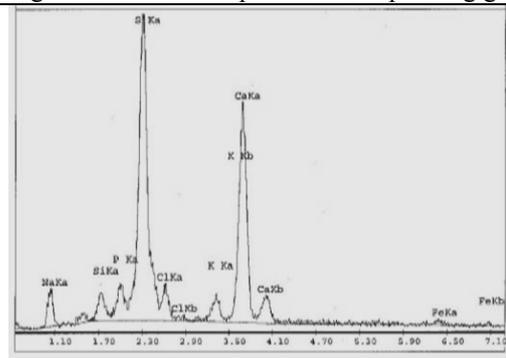


Fig.10 -SEM (EDAX) microanalyses of white pigment with painting ground, “gypsum “,QC.

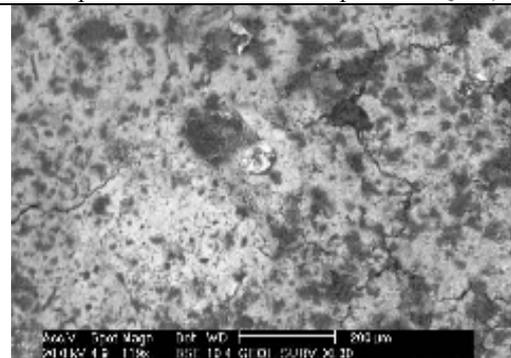


Fig.11- SEM Photomicrograph of white pigment with painting ground “QC.”

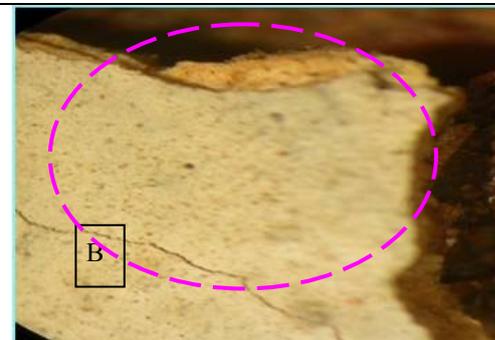
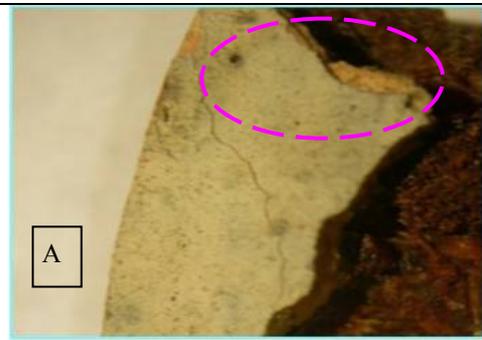


Fig.12-A,B, LOM photomicrograph shows white pigment with deteriorated painting ground, magnification X86&160.QC, “Gypsum”, QC.

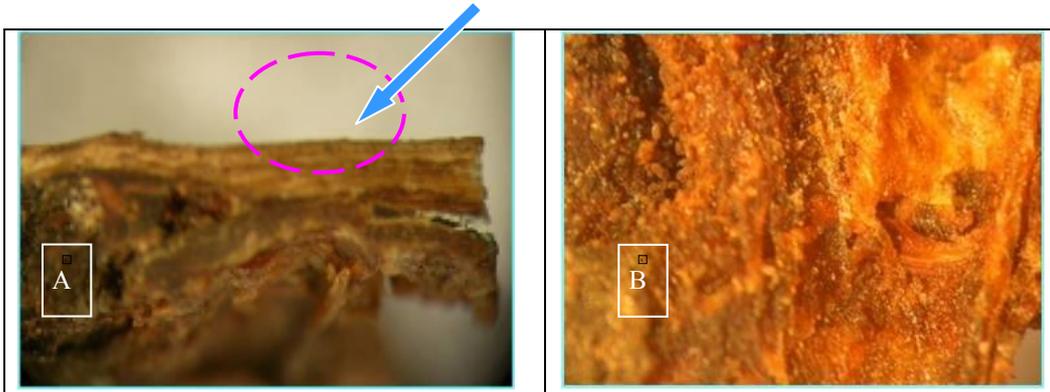


Fig.13- A,B, LOM photomicrograph shows cross section of the white pigment with deteriorated painting ground, magnification X 90,136,"Gypsum", QC.

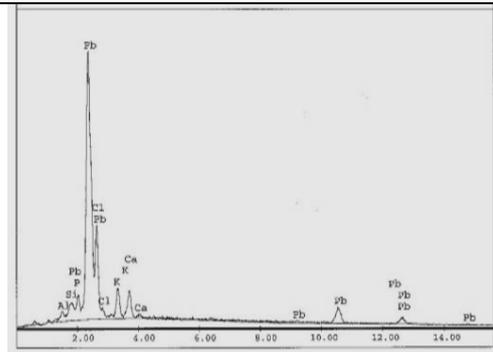


Fig.14 -SEM (EDAX) microanalyses of brown pigment with deteriorated painting ground, "Minium Pb_3O_4 ", AQC ,

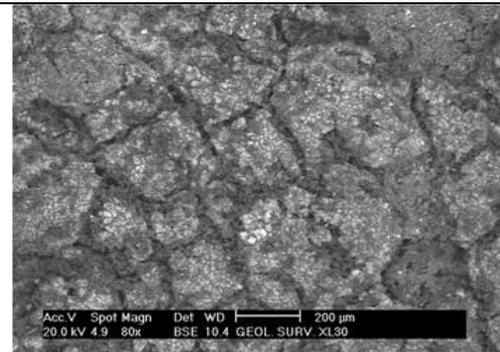


Fig.15- SEM Photomicrograph of brown pigment with deteriorated painting ground," Minium Pb_3O_4 ", AQC ,

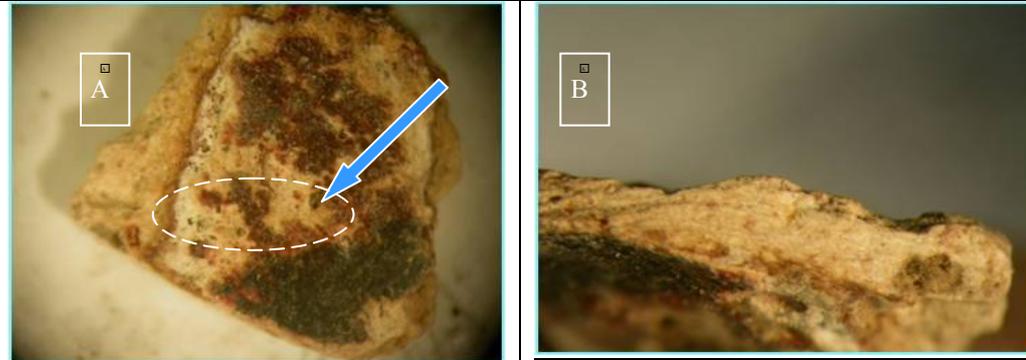


Fig.16 A,B- LOM photomicrograph shows deteriorated BR. Pigment, with painting ground magnification X90&115, "Minium Pb_3O_4 ", AQC.

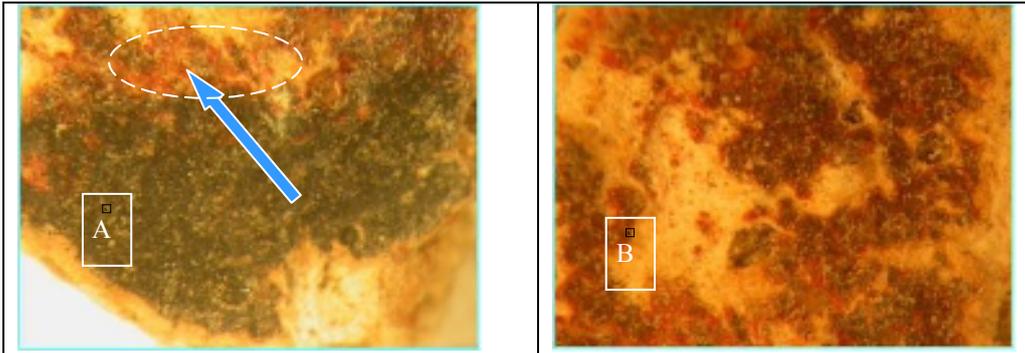


Fig.17- A,B.- LOM details from the previous shows brown pigment with painting ground magnification X150, “ Minium Pb_3O_4 , AQC, “Notice Minium alteration and the missed pigment. “

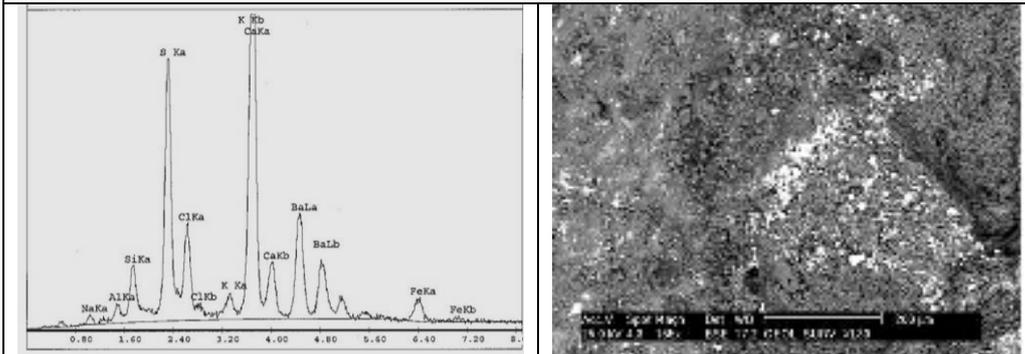


Fig.18- -SEM (EDAX) microanalyses of red pigment with painting ground shows iron is clear “hematite” AQC.

Fig.19- SEM photomicrograph of red pigment with painting ground shows scattered hematite grains AQC.

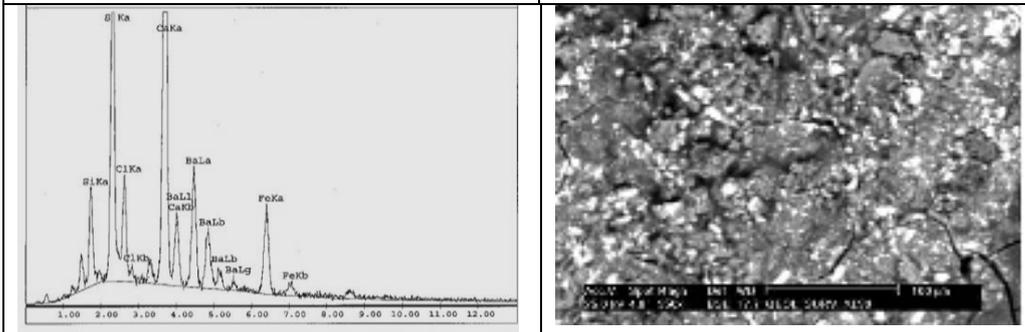


Fig.20 -SEM (EDAX) microanalyses of red pigment on canvas with painting ground shows hematite is a clear component, AQC.

Fig.21- SEM photomicrograph of red pigment on canvas with painting ground shows scattered hematite grains, AQC.

<p>Fig.22- -SEM (EDAX) microanalyses of blue pigment on canvas with painting ground shows Prus. blue iron blue ferric- ferro cyanide $Fe_4 [Fe(CN)_6]_3 \cdot 3QC.$, QC.</p>	<p>Fig.23- SEM photomicrograph of blue pigment on canvas with painting ground QC.</p>
	<p>Fig.24- LOM photomicrograph shows deteriorated blue pigment, Prussian blue scattered on the painting ground magnification X180, QC.</p>
<p>Fig.25 -SEM (EDAX) microanalyses of blue pigment on canvas with painting ground shows blue pigment, AQC .</p>	<p>Fig.26- SEM photomicrograph of blue pigment on canvas with painting ground. The particles show conchoidal fracture and thin sharp edges of glass splinters, AQC.</p>

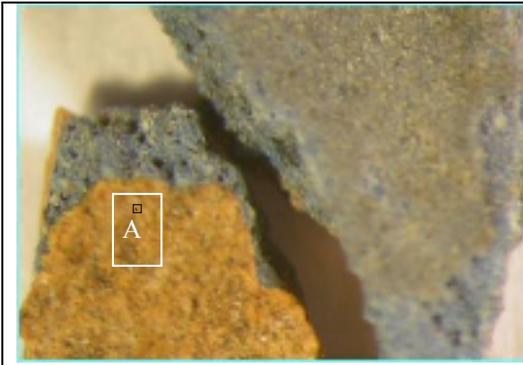


Fig.27- LOM photomicrograph shows deteriorated blue pigment with painting ground, X220, AQC.

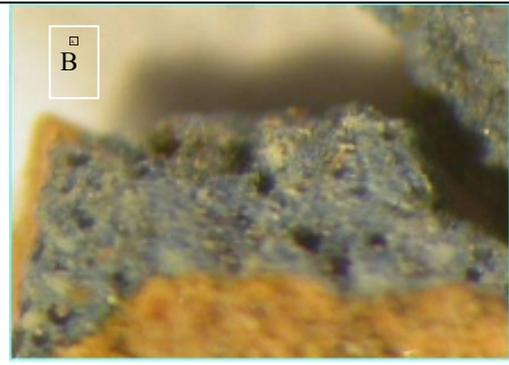


Fig.28- LOM photomicrograph shows details from fig.26 of blue pigment, Small X280.

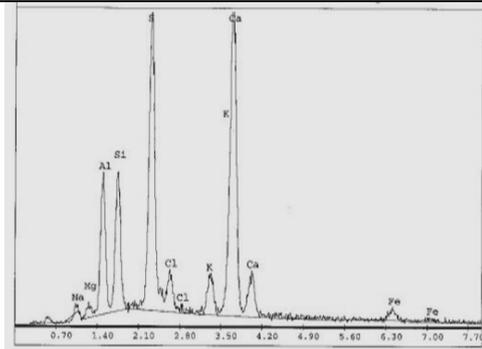


Fig.29 -SEM (EDAX) microanalyses of blue pigment, ultramarine plus prus. blue, AQC.

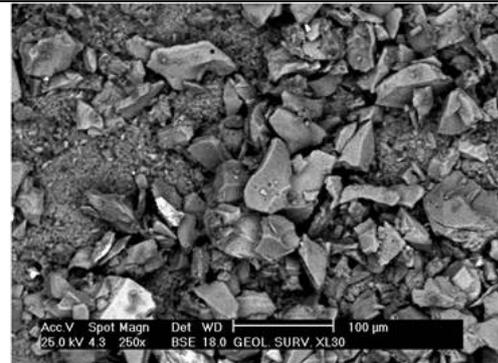


Fig.30- SEM photomicrograph of blue pigment ultramarine plus prus. blue, sharp splinters can be seen, AQC.

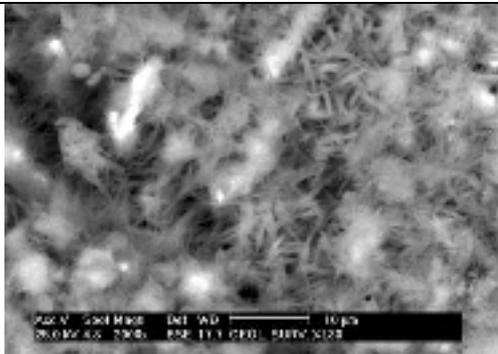


Fig.31- SEM photomicrograph of the painting ground of the blue pigment ultramarine shows the well known needle of gypsum AQC.

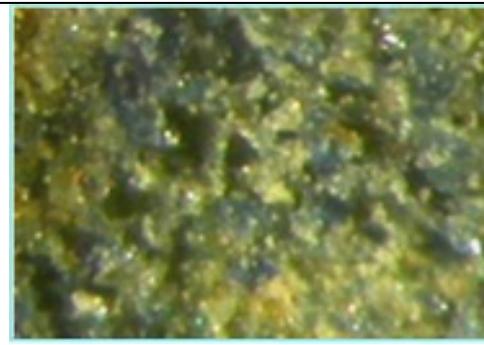


Fig.32- LOM photomicrograph shows deteriorated ultramarine blue pigment scattered on the ground. X 280, AQC.

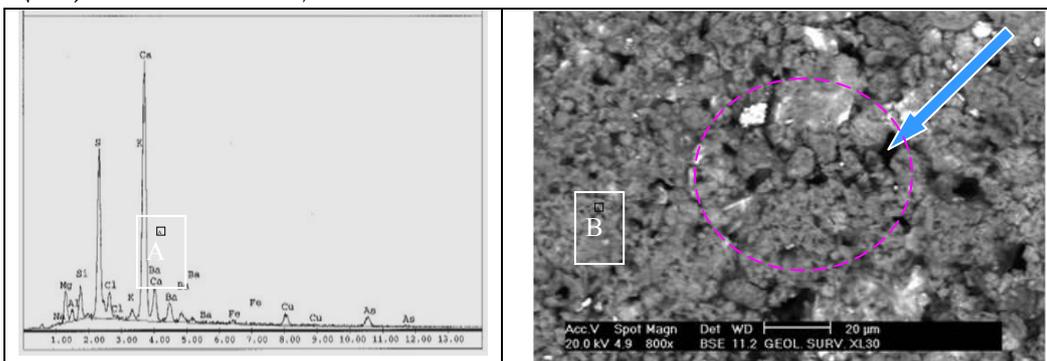


Fig.33- SEM (EDAX) microanalyses of green pigment Copper(II)-acetoarsenite is the main components QC.

Fig.34- SEM photomicrograph of the green pigment QC.

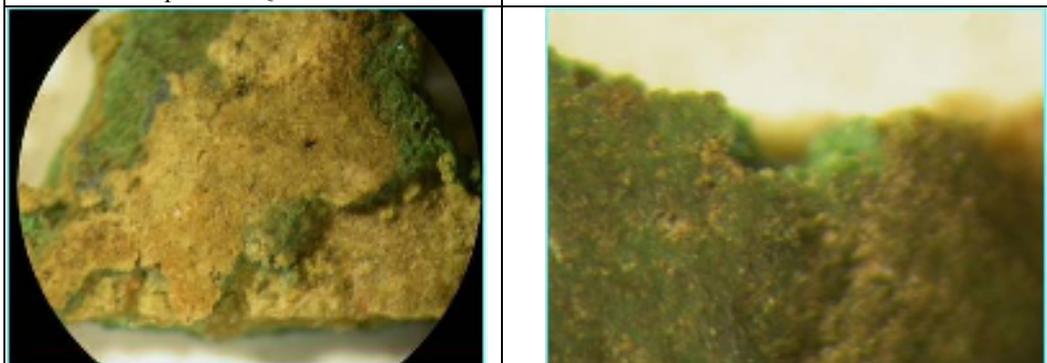


Fig.35- A,B., LOM photomicrograph shows deteriorated green pigment, shows green earth with deteriorated painting ground from both sides.X170& 300, QC.

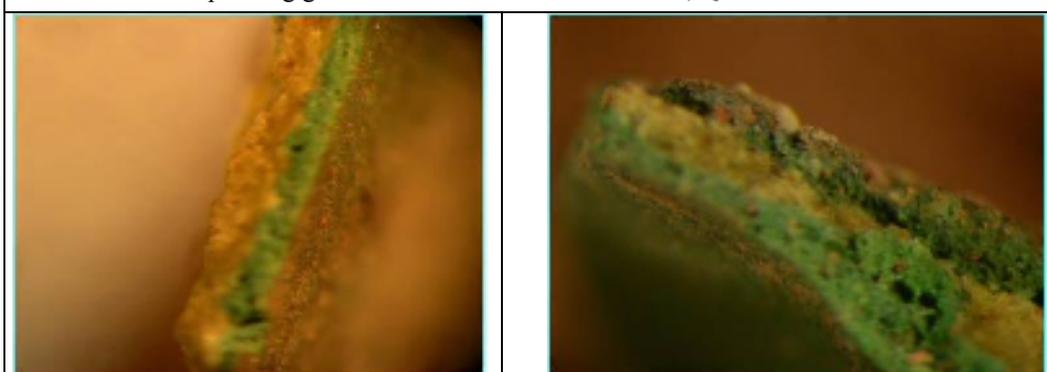
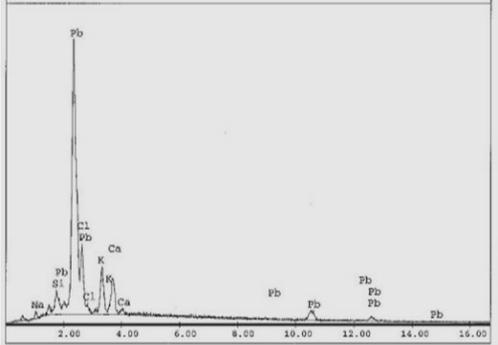
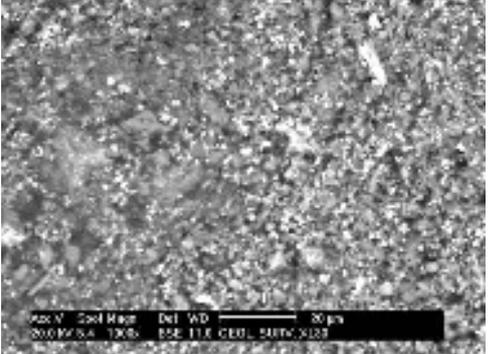
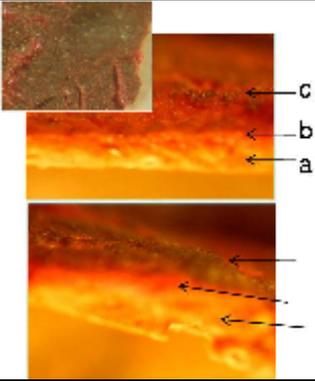
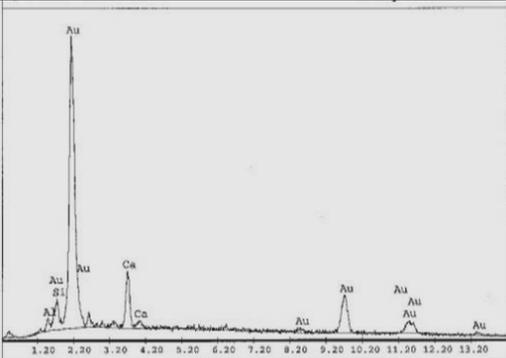
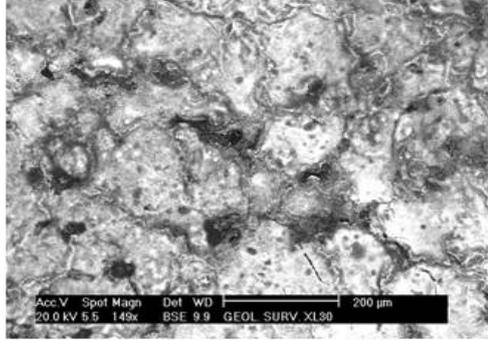
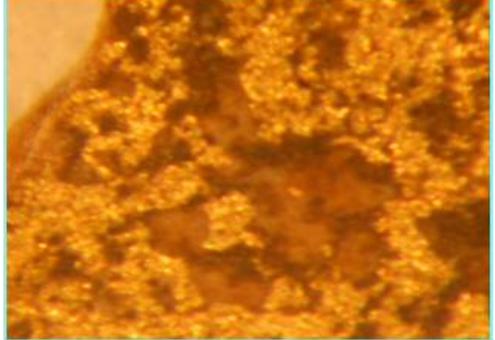
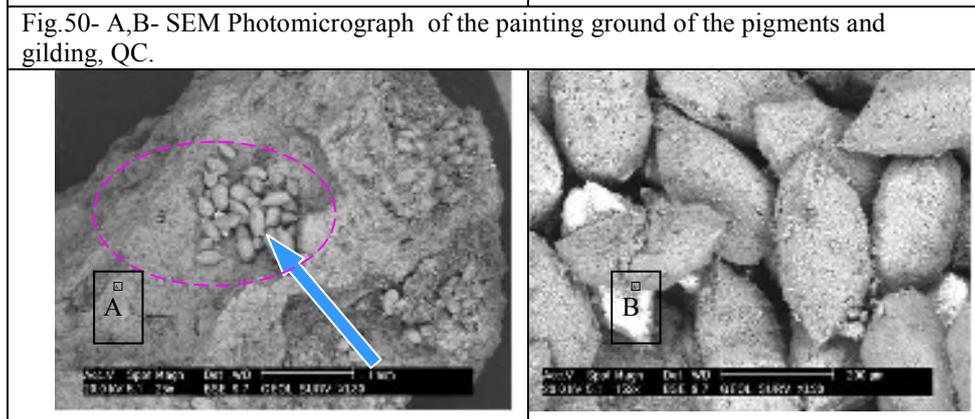
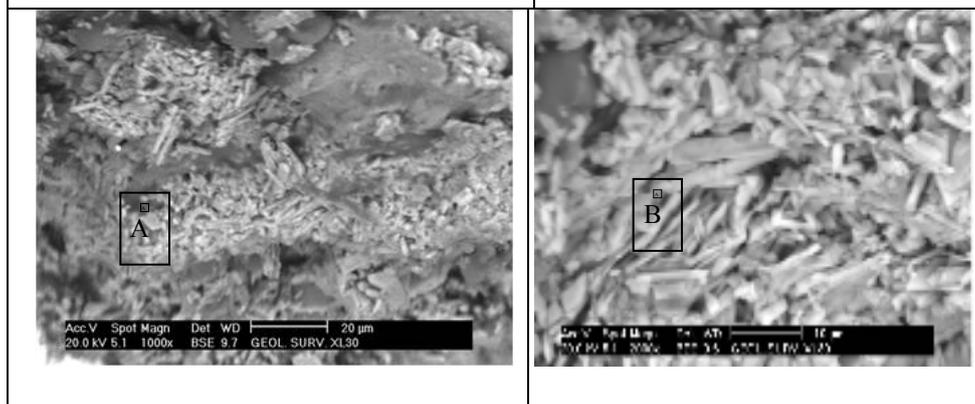
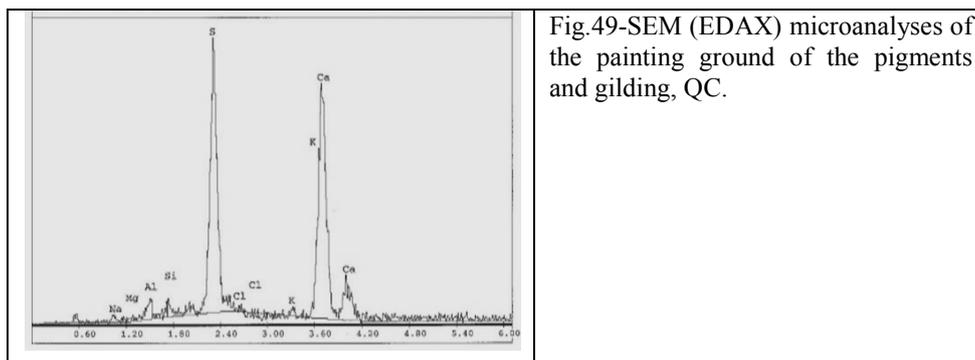


Fig.36- LOM photomicrograph shows cross section of the green pigment with deteriorated painting ground,X220, QC.

Fig.37- LOM photomicrograph shows cross section of the green pigment. *Notice, the pigment with deteriorated painting ground covered another blue pigment (over painting),X300, QC.*

	
<p>Fig.38-SEM (EDAX) microanalyses of new brown pigment shows. minium Pb_3O_4 is the main component. QC</p>	<p>Fig.39-SEM and photomicrograph, of new brown pigment, QC.</p>
	
<p>Fig.40- LOM photomicrograph shows the deteriorated brown pigment, a, painting ground, b, minium, and c, minium alteration.</p>	<p>Fig.41 -SEM (EDAX) microanalyses and of gilding showing gold is the main components.. QC.</p>
	
<p>Fig.42-SEM and photomicrograph, of Gilding showing the deteriorated gold leaf, QC.</p>	<p>Fig.43- LOM photomicrograph shows fragments of gilding on a deteriorated painting ground.X180.. QC.</p>



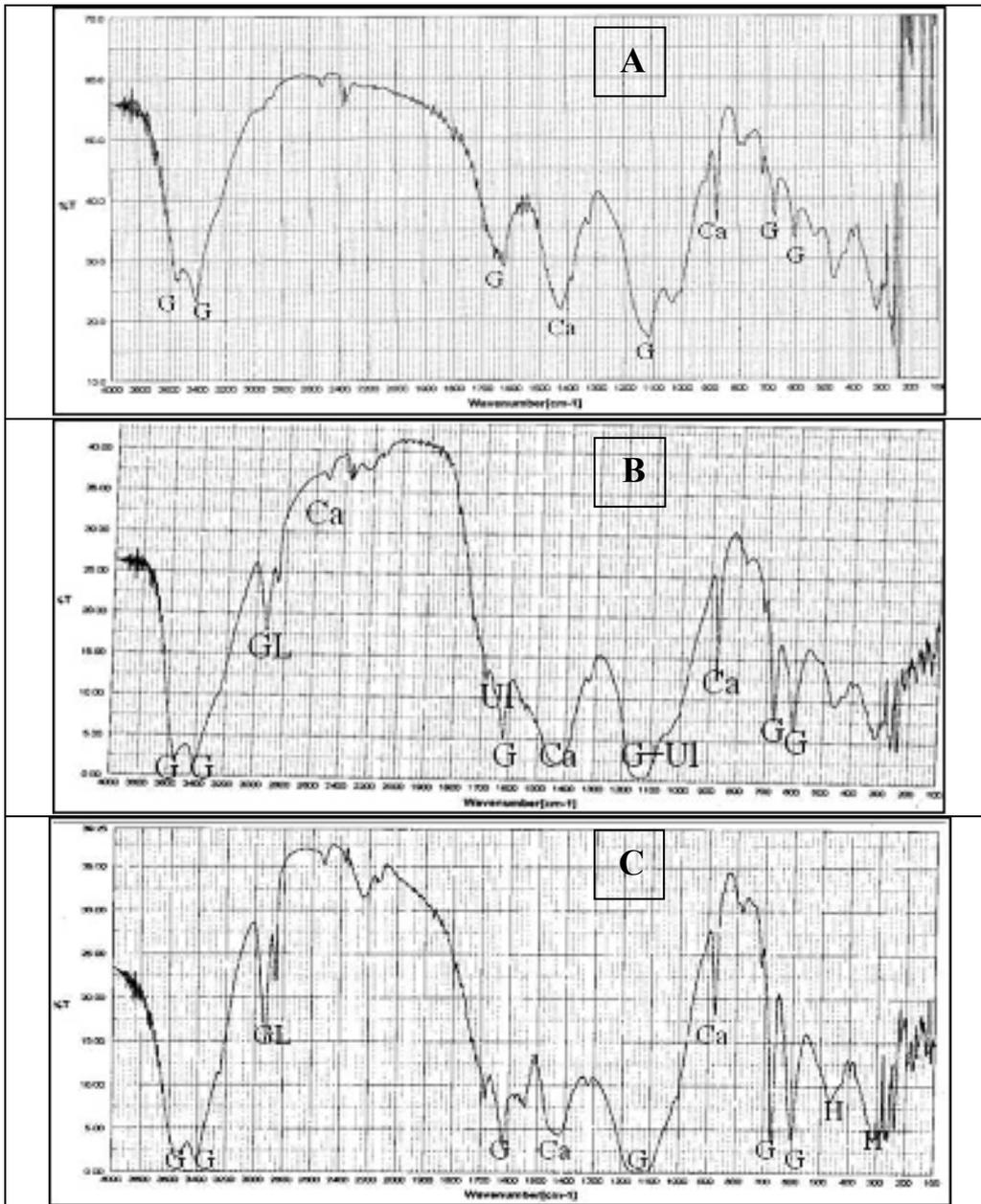
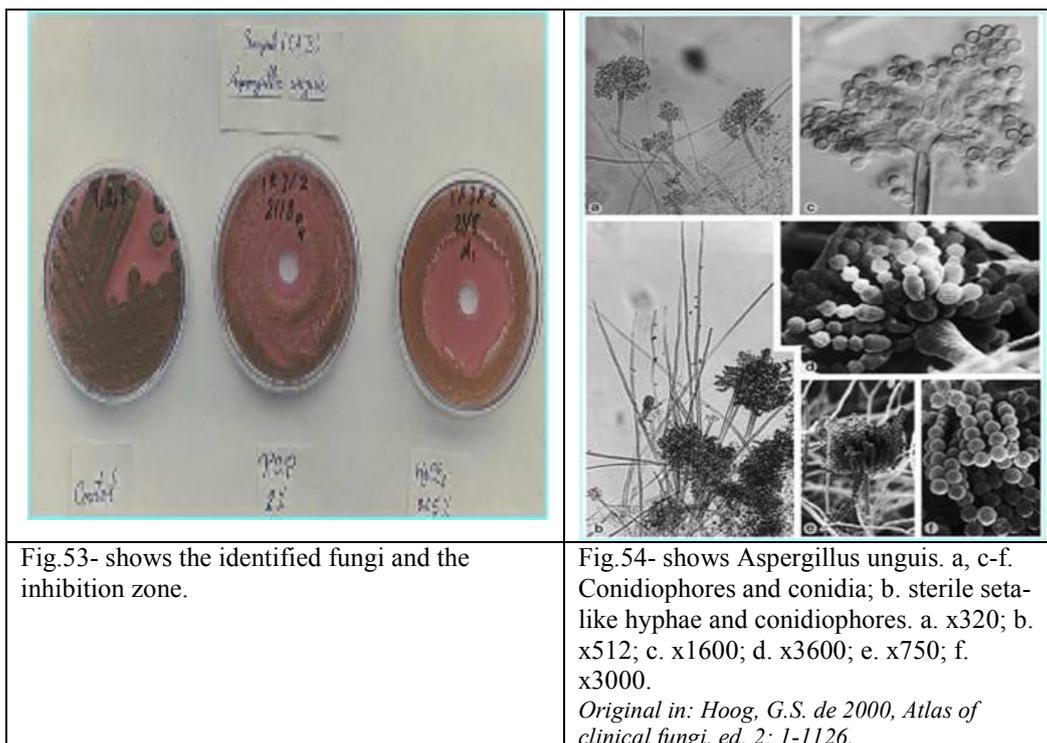


Fig.52 A-C FTIR spectra of the white pigment (A), blue(B) and gilding (C) (G: Gypsum, Ca. :Calcite, Ul. :Ultramarine and H.: Hematite.



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