



A Mamluk Copy of Fawaed El Mawaad: Investigation and Analytical Approach

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HIGHLIGHTS

- A Mamluk copy of *Fawaed El Mawaad*, dated on Friday the fourteenth of *Sha'aban* in the year eight hundred and ninety-two Hijrah.
- Polarizing microscope, FTIR-ATR, SEM-EDX, X-ray Fluorescence, Raman Spectroscopy, and technical photography were used for analysis and investigation.
- The paper leaves of the manuscript were composed of hemp fiber.
- Starch was used as a sizing material in the manuscript.
- Mixed ink (carbon and, iron-gall), and vermilion (HgS) were identified, as black ink and red ink, respectively.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article History:

Received: 24 February 2023.

Revised: 25 April 2023.

Accepted: 23 June 2023.

Available online: 23 June 2023.

Keywords:

Mamluk manuscript, investigation, hemp fiber, filler material, sizing material, black and red inks.

ABSTRACT

A Mamluk paper manuscript copy entitled *Fawaed El Mawaad* in the Library of Ain-Shams University was examined, and several analytical techniques were employed to identify the materials used in its making, and to examine the manuscript's anatomy and sewing technique. Portable digital optical microscope, polarizing microscope, scanning electron microscope with EDX (SEM/EDX), Fourier transform infrared spectroscopy (FTIR), and Raman spectroscopy were among the investigation and analytical techniques employed. Microscopic investigation revealed that hemp fiber, which is shorter than flax

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fiber, yet looks similar to it, was used to produce paper hemp. It is distinguished by transverse knots, a thick, fibrous cell wall, and a smooth surface. This was supported by EDX results that revealed the presence of calcium, which is a main element in calcium oxalate commonly found in hemp and ramie but not flax. SEM images and FTIR-ATR indicated the use of starch as a sizing material and calcium carbonate as a filler. Raman spectroscopy and X-ray Fluorescence were used to determine the types of black and red ink; mixed ink (carbon and iron-gall), and vermilion (HgS) were identified, respectively. The ink type was further confirmed with technical photography using Infrared Fluorescence (IRF) and Ultraviolet Fluorescence (UVF).

1. Introduction

The arts of the written book were of utmost importance throughout the Mamluk era. Scholars believed that the existence of a Quran manuscript in Cairo during the Mamluk era had contributed to the florescence of Mamluk manuscript creation [1], where secular illustrated books never really gained much popularity, possibly because figural imagery had declined at the height of Mamluk artistic patronage [2]. Mamluk manuscripts received care and attention in terms of their production and attention to all their elements (calligraphy, decoration, quality of paper, and binding) by the kings and princes as well as their wives [3].

Islamic manuscripts are assessed according to their calligrapher, artist, and bookbinder as well as their date of the inscription. Five factors make up the most comprehensive dating styles. Namely, the time of day or night; the day of the week; the night, day, or decade of the month; the month's or festival's name; and the year [4].

It is crucial to acquire data on the raw components in order to authenticate a manuscript and attribute it to the time period in which it was used. Islamic papers were often created using hemp and linen, both of which typically had long fibers [5]. The constituents of the local paper were listed in Arabic texts as being flax (*Kettan*) and its products such as linen and rags, hemp (*Qannab*) rope, cotton (*Qutn*), and the bast fibers of the fig tree (*Mudakh* or *Muda*) [4, 6]. Surface sizing material was used by papermakers to achieve smooth writing surfaces during the preparation process of a manuscript's components [6]. In ancient treatises, fish glue and numerous vegetable-based choices, such as rice or wheat starch, were used in paper-making recipes [5], and papermakers polished them

with stones or glass [4]. Early Arabic papers were glazed and surface sized with wheat starch, wheat flour, or starch and chalk combinations to achieve a lustrous, burnished finish and smooth writing surfaces [7]. The scaling solution formulations differed greatly among the papermakers. Salt ammonia, gum tragacanth, white lead, fish glue, and other substances were used as sizing materials. Despite the fact that a number of raw materials might be employed, egg white or starch was most typically utilized to combine with alum (hydrated potassium aluminum sulphate) during the process on both sides of the paper [6] to provide the paper with a glossier appearance and to prepare it for the application of inks from the 8th to the 18th century [8].

Calcium carbonate (CaCO₃) as an inorganic filler or calcite extracted from calcite crystals was used in the manufacturing process of paper; yet other substances like white lead and powdered cattle bone were used to improve the physical and mechanical properties of paper [9], e.g. weight of the paper, and to protect it from the effects of acidity [10, 11].

It took training and learning to create inks of various colors, produce paper in a wide range of tones and varieties, sew bindings using various techniques, and blend dyes for leather book covers [12]. There are four categories of inks mentioned in the recipes, namely carbon (soot), iron-gall (tannin), mixed (compound), and incomplete inks, whereas traditional components of both carbon and iron-gall inks were made up of mixed inks (carbon base and tanning agents) [4]. Colored inks were used to draw attention or to highlight particular passages of the text.

Most Arabic codices lacked foliation, especially the early ones where there is the commentary, which is the word that is highlight-

ed at the bottom of the page and is used as the basis for the first word on the following page [4]. The final word of the text on the b-page (verso), which is typically placed alone below the final line and repeated as the first word of the following page, is known as the "catchword" (a-page or recto) [4].

One of the distinguishing features of Islamic manuscripts are the puncturing marks (*Mastarah*), and the colophon the "tail of the text," which includes the manuscript's and the paper's publication dates [4, 13]. Ruling was employed by Muslim copyists as early as the 1st AH/7th AD century [14]. The ruling of the main area of the page is a dry point (a stylus), lead plummet (early form of a pencil) ink, a ruling board or frame (*Mastarah*), or even a fingernail [4]. However, not all manuscripts were ruled, and lay lines were often very clearly apparent in Arab/Middle Eastern laid paper, and they may have been employed by copyists to guide their hand [4]. The blind lines generated on the paper when the leaf was ruled with a dry point or a ruling board are imbedded (sunk) on one side or raised in relief on the other. The sinking line is referred to as a 'furrow', while the relief line is referred to as a 'ridge' [4].

There was a tendency to employ unequal (odd) numbers for each page, with the number of lines per codex and even every page varying greatly. The middle line may be used as "a strong organizational axis for the entire page," which proved to be a huge benefit for layout (*mise-en-page*) [4].

The aim of this study is to examine, analyze and identify the raw materials and inks used in manufacturing the dated Mamluk manuscript "*Fawaed el Mawaad*", which is currently at the Ain Shams University library. In this 15 leaf manuscript the line numbers are given as subscripts, the number of lines is 15 and each line contains almost 11-12 words.

Hopefully, the results will help in providing insight into the technological and social development of manuscript copies, because there are three other known copies of this manuscript preserved in different manuscript vaults:

- A copy in the British Museum; the rate of words in each line is about eight words, and the conclusion sheet containing the date of publication and anything indicating the completion of copies is missing.
- A copy in the vaults of the Vatican; the handwriting of this copy was not good enough to be extracted from the microfilm. It contained 17 leaves, and the fifteenth page had fallen out of it. Each side of the paper contained 19 lines and each line had 8 words. This copy was dated on the fourteenth day of the month of *Shawwal* in the year seven hundred and thirty-four AH, which means that it was written after the death of the author.
- A copy in the National Library in Paris, which included about thirty-seven leaves, and each side of the paper had about eleven lines, and each line had eight words [15].

2. Materials and Methods

2.1. Historical background of the studied manuscript

The original manuscript of "*Fawaed el Mawaad*" was written by *Jamal al-Din Abu al-Husain Yahya ibn Abd al-Azim al-Masri*, one of the Mamluk era's tramps of poets of the 7th century AH [15], who was born in *Fustat* (Egypt) in the year 601 AH / 1205 AD, and grew up there. In the book entitled "*Shadharat al Dhahab*" (which means nuggets of gold), written by the jurist historian *Ibn al-Imad al - Hanbali* (1032 AH - 1089 AH) it was mentioned that *Al-Jamal al-Din Abu al-Husain Yahya ibn Abd al-Azim al-Masri*, who was also known as *Abu al-Husain Ibn al-Jazzar* or known as "The butcher" died in the year 672 AH on the eighteenth of *Shawwal*, suffering from hemorrhoids at the age of seventy-six years or so, but in the study by *Ibrahim El Samara'ay* it is mentioned that he died in the year 679 AH (which makes it difficult to verify this point) [15, 16].

Based on "the colophon" transcribed in the 'tail of the text' the manuscript in this research study entitled "*Fawaed el Mawaad*" was dated on Friday the fourteenth of *Sha'aban* in the year eight hundred and ninety-two (892 Hijra calendar); which means

that this manuscript was rewritten after more than 200 years of the original copy, and the type of dating in the document under study is referred to as *tarkh bi-ajz al-yawm aw al-laylah* or *tarkh bi-layl al-shahr* by *Al-Qalqashand* (Fig. 1).

In the numbering of the manuscript under study, the sides of the folio were in the correct sequence (Fig. 1), and the 'catchword', which is the last word of the text on the b-page (verso), is usually placed alone below the last line and repeated as the first word of the following page (a-page or recto). The thumbnail was used to rule the major area of the sheet that was destined to receive the text, yet they were so faint that they are impossible to discern (Fig. 2).

2.2. Analysis and investigation methods

2.2.1. Portable digital light microscope

A portable digital microscope (Color CMOS Sensor, high speed DSP resolution 1000X Zoom) was used to examine the ink (red and black) and the surface of the paper manuscript.

2.2.2. Polarized Light microscope (PLM)

Fiber identification entails obtaining samples from the artefact and studying the fiber shape under 100 times or higher magnification [18]. A metallic needle was used to take micro samples, which were prepared for investigation. The fibers were removed from the paper substrates and placed on slides; a drop of water was added, and the samples were subsequently stabilized with cover slips [19] and investigated under visible and polarized light (100x, 200x) using Polarized Microscope (PLM) at the Egyptian Mineral Resources Authority laboratories (EMRA), Microscope: Leitz Orthoplan, Camera: Leica mc 190 HD.

2.2.3. Thickness Gauge

The thickness of the paper sheets was measured with a handheld Accud thickness gauge model no. 443-010-11, with an accuracy of ± 0.02 mm and a measuring range between 0-10mm.

2.2.4. Scanning electron microscopy-energy dispersive X-ray (SEM-EDX)

All samples were first coated with a thin gold film using the gold sputtering tech-

nique, and Scanning electron microscope / FEI Quanta 3D 200i EDX / Thermofisher Pathfinder operated under conditions of low vacuum for acceleration voltage 20.0 ~ 30.0 KV using large field detector with working distance 15 ~17 mm was used.

2.2.5. Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR):

Bruker model Vertex70 at the Center of Research and Conservation of Antiquities, Ministry of Tourism and Antiquities was used to determine the nature of the sizing material employed in the document by detecting the main functional groups. ATR was used to analyze the sample without preparation and without damaging the sample. The spectral range of the device is from 400 to 4000 cm^{-1} .

2.2.6. Raman spectroscopy (RS)

Senterra Bruker Raman microscope at the Center of Research and Conservation of Antiquities, Ministry of Tourism and Antiquities, scale from 45 to 4500 cm^{-1} , with condition: Nd: YAG at 785 nm, continuous laser source, power up to 100 m watt, apertures of 50 x 1000 μm , was used.

2.2.7. X-ray fluorescence (XRF)

The elemental composition of inorganic compounds used in manuscript inks (red-black) was determined using portable X-ray fluorescence (pXRF) spectroscopy.

Thermo Scientific Niton XL3t with a "GOLDD" detector. x-ray tube with a Ag anode of 50 kV and 200 μA . X-ray spot size is 3 mm in diameter, Thermo Scientific NDT program was used to obtain spectra, at the Grand Egyptian Museum Conservation Center.

2.2.8. Technical photography

FUJIFILM X-T4 Mirrorless Camera, Fujifilm XF 18-135mm f4-5.6 R OIS Lens, UV Flashlight Black Light, 51 LED 395 nm Ultraviolet Blacklight, Infrared Bulbs (Red), wattage 250 watts, bulb shape/size A19 temperature, 6500 Kelvin, Infrared Fluorescence (IRF) and Ultraviolet Fluorescence (UVF).



Fig. 1. (a) The folio of the manuscript, first page includes stamps and was written in red and black ink, **(b)** Last page includes stamps and “The colophon” transcribed the ‘tail of the text’ records a date.

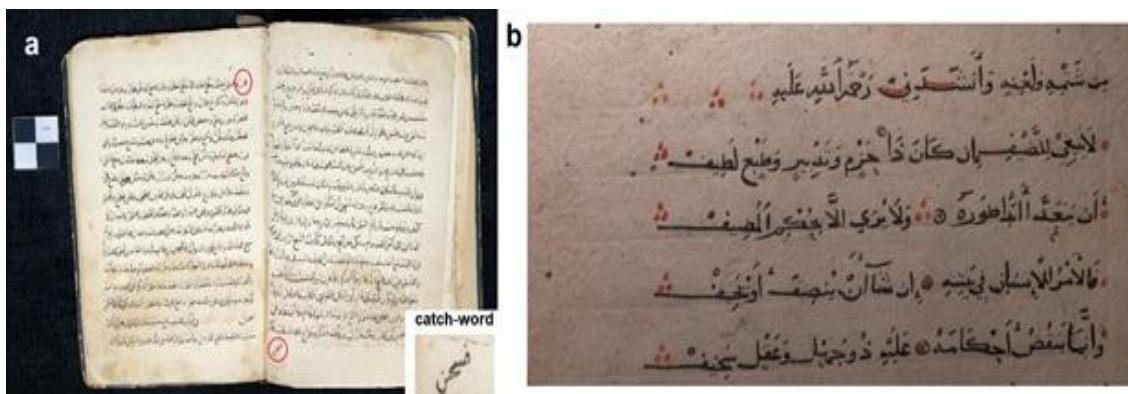


Fig. 2. (a) The 'catchword' is the last word of the text on the b-page (verso), usually placed alone below the last line and repeated as the first word of the following page (a-page or recto). (b) The ruling of the page (Mastara).

3. Results and discussion

3.1. The structure of the manuscript entitled “Fawaed el Mawaad”

3.1.1. Paper Mold

The fundamental difference between European paper and its Arab/Middle Eastern and Indian counterparts is that the former has watermarks that have been the axis of western paper research, while the latter does not [4, 13]. Hand-made and machine-made paper, on the one hand, lay and wove paper, on the other, are the four primary categories of European paper. Chain and placed lines are two characteristics of lay paper. Laid paper is a form of paper that has a pattern of lines (laid and chain) that are caused by the natural pressure exerted by the pulp in the mold [11, 14]; these lines are marks that reflect the design of the mold's mesh.

In 'Islamic' papers, the chain lines can be organized in twos, threes, and fours, or twos and threes. Papers from Iran, Syria, Egypt, Asia Minor, and even Mecca, which date from the 5th – 9th centuries, contain those organized in threes [4], which can be seen in the studied manuscript (Fig. 3), and "each set is separated by a gap roughly three times wider than the lengths separating the sets within each group". The chain lines are frequently wavy and uneven, and they can be spaced anywhere between 30 and 90 mm apart. When it comes to the set lines, they are frequently double, resembling thin stripes. Parallel rows of grass or reeds (creating laid

lines that run vertically) were bound together with horse hair lacings to create the laid mold (producing chain lines, which run horizontally) [4].

By holding a sheet against the light or using a basic gadget with a glass top lighted from underneath to examine the pattern it was possible to characterize that there are 15 lines on average per 20 millimeters in the laid paper of the manuscript flyer sheet (Fig 3), and the average thickness of the written sheet measured 0.23mm.

The paper's opaqueness (or translucency) according to the Sharp Scale of Opaqueness [17], which is given a numerical value for categorizing the translucency of paper in terms of the number of folios required before the outline of a dowel held behind the folio(s) was no longer visible when illuminated from behind with a constant light of 60 watts at a distance of approximately 15 cm. Through this test, it is clear that the light transmittance is strong in the case of one sheet (Fig 4a), but it decreases and is almost opaque in the case of two sheets or pastedowns due previous restorations (Fig 3b).

3.1.2. Sewing Structure

Sewing techniques can be found all around the Islamic world. Stitched sewing methods are an unsupported sewing structure that is completely different from link-stitch sewing structures, and resemble repair sewing structures at times. They could also be the original

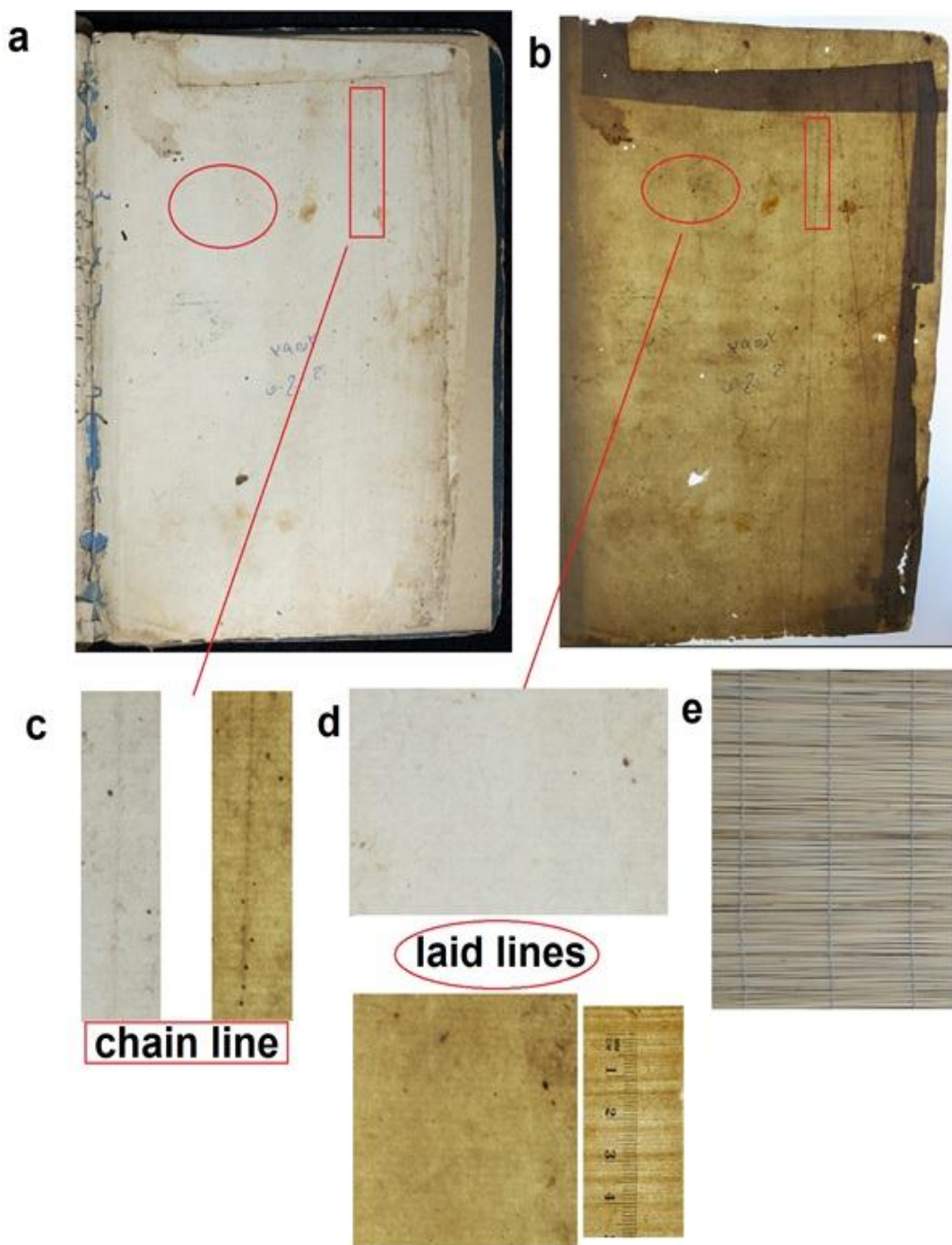


Fig. 3. (a) The flyer page of the manuscript (b) The flyer page on a lighted glass top, (c) Chain line, (d) Laid lines with 15 placed lines per 20 mm on average, (e) Traditional mat used for paper production.



Fig. 4. (a) Measuring opaqueness, showing the light transmittance in the case of one paper sheet (b) The manuscript is sewn with a stabbed sewing (red arrows), perpendicular to the spine. The small holes in the center (red circle) bear witness of a former link-stitch on two stations (c) The manuscript with a stabbed sewing parallel to the spine (d) Modern pastedowns or endpapers, which indicates that these materials were most likely included when the volume was last bound or rebound.

sewing structure; however, they do not have to be from the same time period as the manuscript [1, 14]. The thread (or string, or leather lace) goes through transverse holes in the text block, very close to the spine, in punctured formations as seen in the manuscript under study (Fig 4 b, c). By examining both the binding and the flyer of the manuscript and the type of stitching used it is possible to deduce that they are recent papers, and also the stitching is modern and not from the original manuscript (Fig 4 d).

3.2. Identification of fibers

Flax, hemp, nettle, ramie (an Asian nettle variant) and jute are classified as bast fibers. Hemp fiber is similar to flax fiber in appearance, but is shorter [20]. Hemp, which is thought to have originated in Northern India and Persia, is a bast fiber that is extracted from the plant by retting in water, then sepa-

rating and washing the fibers from the plant's residual components [20]. Its fiber is longer, has a higher degree of lignification than flax fiber and is a good raw material for pulping [21].

Transverse knots, a thick fibrous cell wall, and a smooth surface, which are all characteristics of hemp fiber [20], were identified in the manuscript during investigation using polarized light microscope (Fig. 5a, b) and SEM-EDX (Fig. 6a, b, c).

3.3. Identification of the fillers

Hemp fibers contain 68–78.3% cellulose, 5.5–16.1% hemicellulose, 0.8–2.5% pectin, 2.9–3.3% lignin and some fats and waxes, in addition to calcium oxalate crystals, which are present in ramie and hemp but not in flax [22, 23].

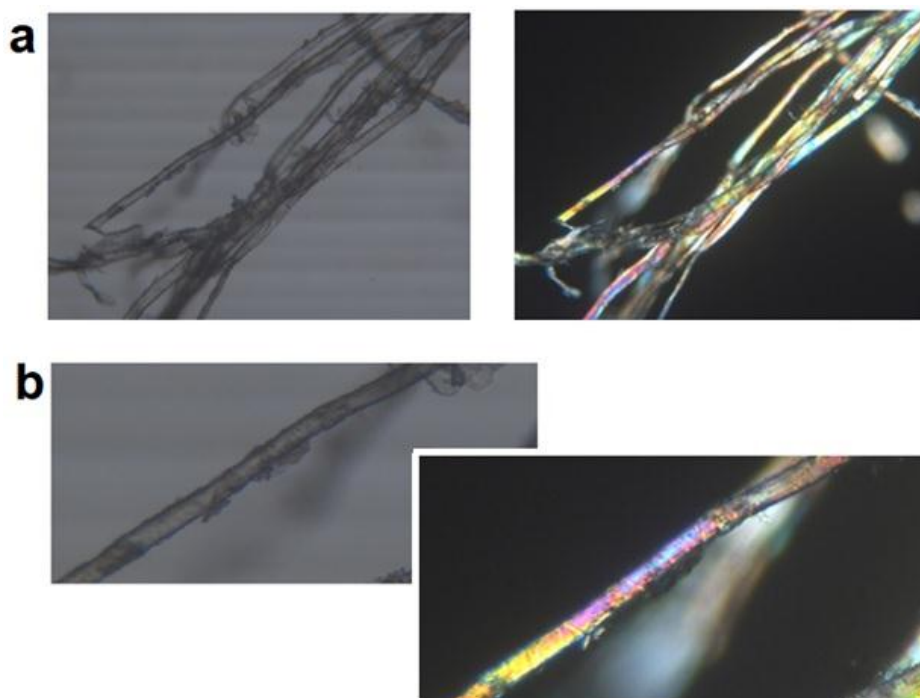


Fig. 5. (a) Fibers of manuscript in polarized light, 100x, the image observed suggests the use of hemp or linen fibers (b) Hemp fiber.

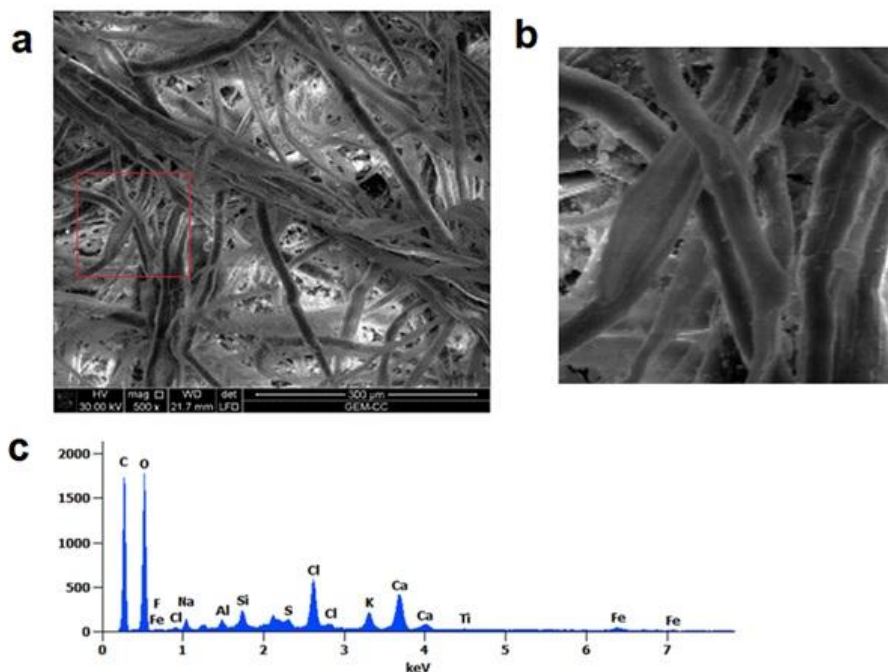


Fig. 6. (a) SEM image of hemp fiber, which is similar to flax fiber in appearance, but is shorter. Transverse knots, a thick fibrous cell wall, and a smooth surface are all characteristics of hemp fiber. (b) Previous Fig. magnified, (c) EDX analysis revealed the presence of Si, Al, Cl, K, O, Fe and Ca.

EDX spectra of the paper sample showed that it consisted of C (5.89) intensity, O (5.91) intensity, Ca (11.92) intensity and Cl intensity (10.47), and during the investigation of the samples the fillers in the paper were noticed. Si, Al, and Fe were impurities found on the surface of the paper, which is supported by their small percentage in the

analysis. The investigated and analyzed samples had a relatively high calcium content (Fig 7); which may be attributed to either the calcium oxalate present in hemp or the usage of calcium carbonate as a filler during the manufacturing process of paper as mentioned in references [24, 25].

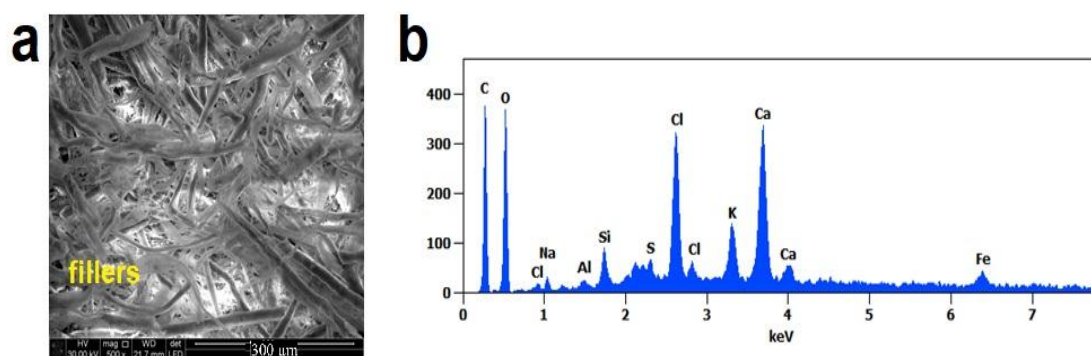


Fig 7: (a) Fillers in the paper that are similar in shape of the calcite crystal (b) EDS X-ray analysis confirm presence of calcium (Ca), which is referred to the use of calcite (calcium carbonate) as filler material in paper making process in references [9, 10, 24].

3.4. Identification of the sizing material

Sizing on both sides of the paper, and the leaf itself form three sandwich like layers that make up a folio. The sizing layer and paper fibers can be easily distinguished using SEM (Fig 8).

ATR-FTIR analysis determined the nature of the sizing substance employed in the document (Fig. 8). The broad peak at 3320 cm^{-1} is due to OH stretching vibration and is assigned to the hydroxyl groups in cellulose, while the band at 2900 cm^{-1} is related to CH_2 stretching vibration of the CH_3 group and is assigned to the hydrocarbon group in cellulose, hemicellulose, and lignin of the paper. The band at 1642 cm^{-1} is due to cellulose oxidation and the formation of carbonyl and carboxyl groups, assigned to the stretching vibration of -C=O [26]. The FTIR spectrum of starch typically shows at bands around $2900\text{-}3000\text{ cm}^{-1}$ (C-H stretching), $1100\text{-}1150$

EDX spot measurements of the sizing showed that the intensity of C is (26.58), O (20.86), Cl (22.49), Ca (12.66), which can be attributed to the usage of starch across the book.

3.5. Identification of the black and red ink

3.5.1. Black ink

Throughout the ages and in most geographical regions, carbon-black ink was the most frequent writing medium [13]. Raman analysis make it possible to distinguish between a variety of inks. Iron-gall ink is distinguished by the peaks at roughly 1472 and 572 cm^{-1} , carbon black ink by the characteristic bands at 1334 and 1588 cm^{-1} , and iron sulphate by the peak at 1454 cm^{-1} [28-32].

The data obtained from XRF analysis of the black ink indicated the presence of Si: 0.098%, Cl: 0.101%, Fe: 0.23%, S: 0.057%, P 0.14%.

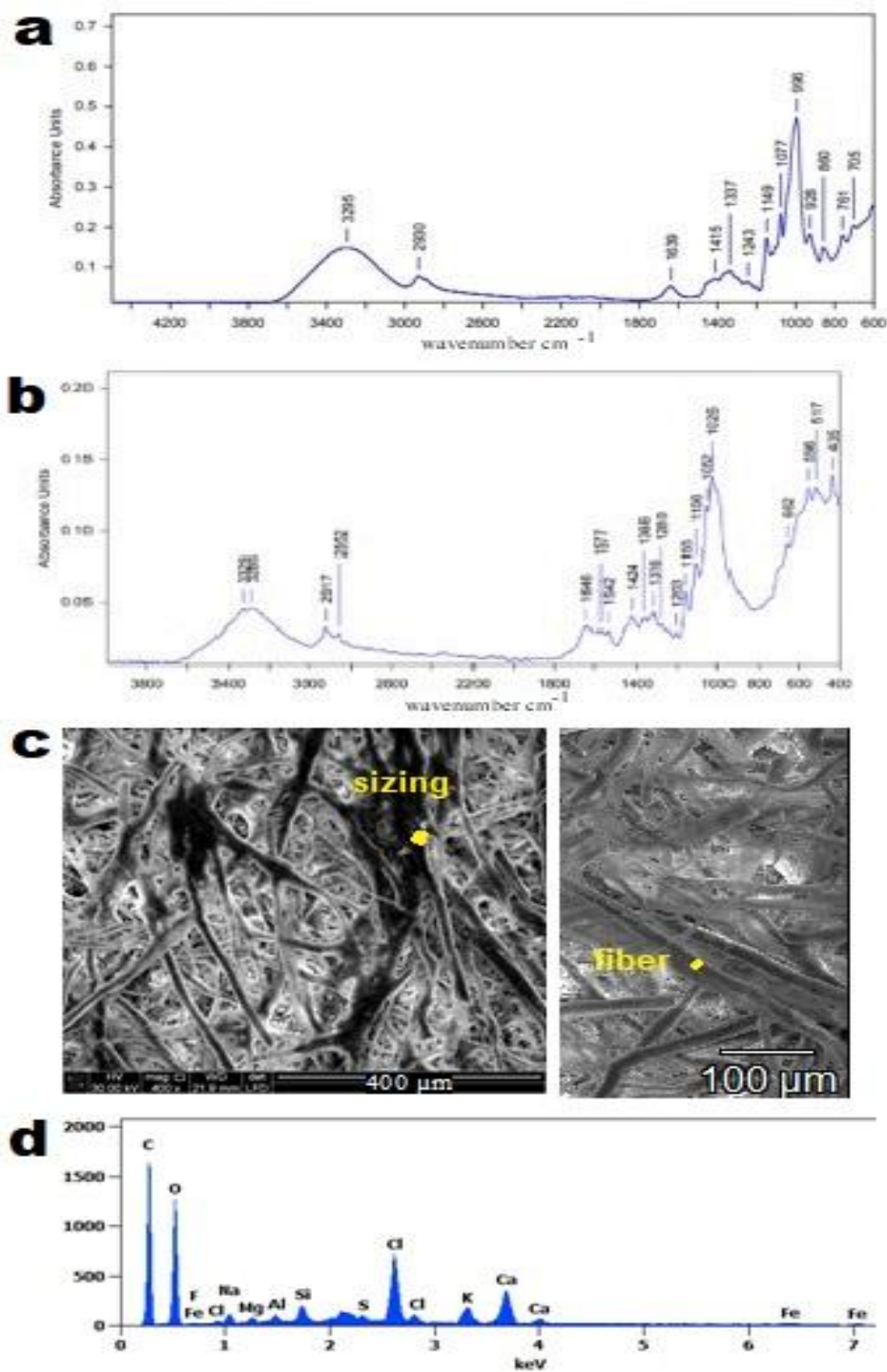


Fig. 8. (a) ATR/FTIR spectra of starch standard, (b) ATR-FTIR spectra of paper sample, (c) SEM, the size layer and paper fibers could be easily differentiated (D) EDX measurements of paper sample.

The obtained Raman spectrum of the ink sample shows three characteristic bands in the region between 1334–1592 cm^{-1} , as well as a less intense broad band at 473 cm^{-1} , in addition to a band at 984 cm^{-1} which indicates the presence of phosphorus element as a result of the apatite which comes from the burning of animal bones, and is evidence of black bone (Fig. 9). The co-presence of iron and sulphur in the ink place this ink in the true category of mixed inks.

3.5.2. Red ink

The use of colored inks predates the coming of Islam and was prompted by a desire to draw out or highlight certain features of the text [4], and red ink can be found already in early fragments of the Qur'an from the end of the 1st AH/7th AD century. The obtained Raman spectrum of the red pigment in the book under study shows three characteristic bands in the region between 253 and 344 cm^{-1} (Fig. 10). XRF spectrum of the red pigment obtained by the portable instrument detected Fe 0.24 %, P 0.24%, S 1.83%, Sc 0.20% Hg 0.24%. The concentration of the Hg element in all red pigments used in writing and decoration of the manuscripts' results were similar to previously published research [33]. Light red HgS vermilion is one of the earliest synthetic pigments that has been around since antiquity, is in bright and opaque colors, and a high purity color is distinguished by durability [6, 31, 32]. Cinnabar, a naturally occurring material, was also frequently utilized by ancient scribes because of its accessibility and ease of manufacture. Since they are both chemically equivalent, structural and spectroscopic methods cannot differentiate between cinnabar and vermilion [30].

3.6. Technical photography Infrared Fluorescence (IRF) and Ultraviolet Fluorescence (UVF)

Technical photography is one of the effective methods for concluding the process of identification, which offers substantial advantages to researchers and therefore sampling and analysis over a vast area is not required [34]. Any significant change of opaci-

ty can be detected by comparing the visible and near-infrared micrographs in any text examined, revealing the presence of carbon in every ink [10, 29, 34].

By using the IRF approach, carbon black ink created the black hue, confirming the aforementioned results, and eliminating the probability of the use of iron-gall which produces a vivid red color.

Natural-based inks have a high infrared absorption, making them opaque to IR radiation. Additionally, such inks do not produce UV fluorescence, as in the case of the orange strip or the red/orange letters in the text. These pigments also had a weak infrared and significant UV absorption, which indicates the use of vermilion in the case of the red ink (Table 1, Fig. 11).

4. Conclusion

The current study focused on the use of optical and polarized light microscopy methodology for fiber characterization of paper samples from a Mamluk copy of the *Fawaed el Mawaad* manuscript dating back to 892 AH. The qualitative and quantitative investigation of writing materials using X-ray fluorescence analysis (XRF) proved to be a suitable method for determination of the elemental concentrations of the writing materials. SEM-EDX, ATR/FTIR and Raman spectroscopy were used to confirm the type of fibers, and identify the fillers, sizing materials and inks, and to obtain information about the chemical composition of the paper used in the Mamluk manuscript, which was written with a mixed ink and a red vermilion ink on paper made of hemp fibers. Infrared Fluorescence (IRF) and Ultraviolet Fluorescence (UVF) technical photography were used for further confirmation of the ink types.

It would be interesting to compare in future the type of paper and inks used in the three aforementioned copies of this book in the British Museum, National Library in Paris and the Vatican vaults; and may be the analysis can help in dating the two copies that are missing the page in which the date was written.

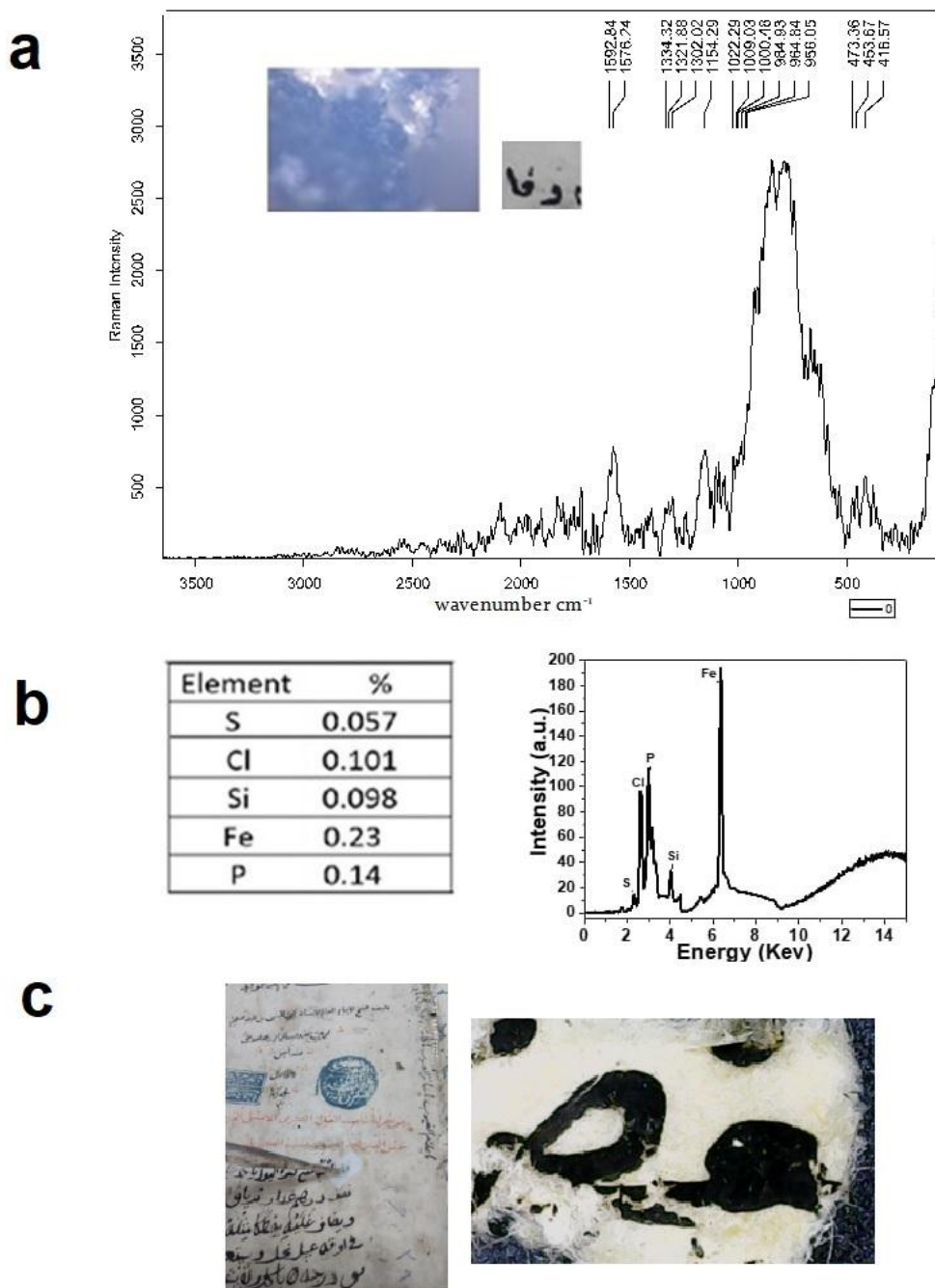


Fig (9) (a) Raman spectrum shows carbon black ink characterized by strong bands at 1334 and 1588 cm^{-1} , (b) The data obtained from XRF of the black ink (c) The agglomeration and caking off the ink on the paper surface while undergoing the ink test.

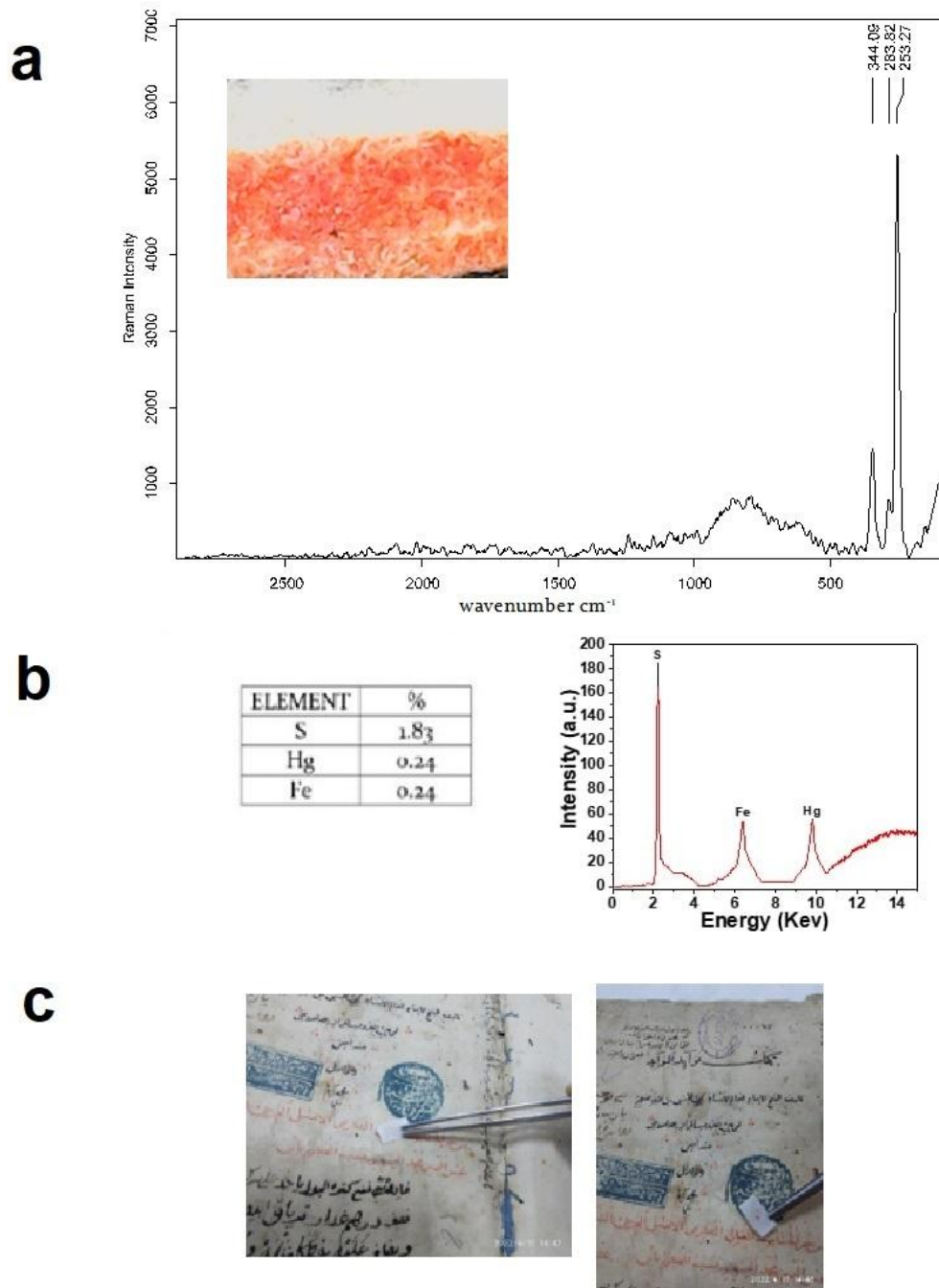


Fig 10: (a) Raman spectra shows characteristic bands in the region between peak 253 and 344. (b) XRF spectrum of the red pigment obtained by the portable instrument indicates the presence of Hg and Fe (c) The extent of penetration of ink from the surface of the paper



Fig. 11: Technical photography of two different sheets in the manuscript

Table 1. Results of technical photography along with identified elements based on EDX.

| Color | Identified element | Technical photography observations | | | Result |
|------------|--------------------|------------------------------------|-----------|--------------|------------|
| | | VIS | UV | IR | |
| Black ink | C | Black | Dark | Dark | Mixed Ink |
| Orange/red | Hg | Orange/red | Grey-blue | Total fading | Vermillion |

It is also about high time to put up a call for an extensive search in Egyptian libraries such as the Egyptian National Library and archives, the Library of Alexandria, Al Azhar Library, Rifa'a Al-Tahtawi Library and the heritage libraries in some Egyptian Universities [35] to do a concise search and documentation of their books, because there may be a high probability that other copies of *Fawaed el Mawaad* written at different intervals could be found.

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