



Utilizing Different Analytical Techniques to Determine the Composition of the Paper Support of Historical Lithographic Plate from Belzoni's Atlas

Asmaa Abd El-samie Mahmoud^a, Wafika Noshy^b, Mohamed Marouf^a,
Wael S. Mohamed^c

a. Conservation Department, Faculty of Archeology, Sohag University, Egypt

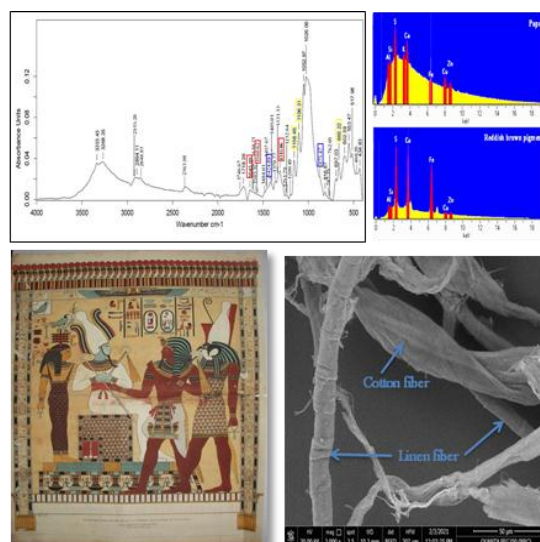
b. Conservation Department, Faculty of Archaeology, Cairo University, Giz, Egypt.

c. Polymers Department, National Research Centre, Dokki, Giza, Egypt.

HIGHLIGHTS

- Identification of the components of the rare historical papers from Atlas of Belzoni using micro-analytical methods.
- The results proved that the historical lithographic plate was printed on wove paper from Whatman paper.
- Wove paper is the predominant paper type used for fine art prints.
- Compounds such as gelatin, alum, and CaCO_3 can be identified using ATR-FTIR and XRD analyses in historical paper.

GRAPHICAL ABSTRACT



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ABSTRACT

Lithography is one of the rare historical collections that have not been sufficiently studied by researchers. This study aims to use different analytical techniques in order to identify the components of the paper support of a historical lithographic plate, which is one of the 44 printed plates in a volume entitled book "Plates illustrative of the researches and operations of G. Belzoni in Egypt and Nubia". Visual assessment, identification of the cellulose fibers by optical microscopy and

* Corresponding author: smsmmahmoud33@yahoo.com

investigation of the surface morphology by scanning electron microscope (SEM) with EDX, X-ray diffraction, and Fourier Transform Infrared Spectroscopy (ATR/FTIR) analysis were used to identify fillers and sizing materials. The results revealed that the paper used to print the lithographic plate is Whatman paper (i.e. wove paper) which is European-made. Rags (both cotton and linen fibers) have been used as a raw material in the creation of this paper. Gelatin and alum have been used as surface sizing materials in the paper support. The presence of calcium carbonate in the paper support is attributed to calcium hydroxide (lime), which was added to the rags during the beating process.

1. Introduction

Fine art prints are one of the most popular artistic forms. They consist of an original matrix that is printed on a paper support. Fine art print is an art form consisting of the production of images, usually on paper but sometimes on canvas, vellum, or other miscellaneous carriers through various techniques [1]. Whereas artistic printing (graphic art) is the art of cutting, engraving, or processing wood, metal, stone or any other material with the aim of achieving printing surfaces prepared for inking, and then printing what has been prepared by a special press or by hand pressure on paper specially prepared for that [2]. The techniques used in ancient art printing varied under the term engraving, through its multiple implementation methods such as etching, soft-ground etching, line-engraving, chalk and stipple, aquatint, mezzotint, lithography and wood engraving [3]. Lithography is the art of drawing or writing on stone [3], which was introduced at the end of the eighteenth century AD [4]. It is attributed to the German playwright Alois Senfelder (1771-1834 AD) [5,6]. It did not take long for it to develop technically and became one of the most important and most famous methods of printmaking [6]. Lithography is a chemical process that essentially is based on the fact that water repels grease. Traditionally, a lithograph is produced by drawing on a flat, polished slab of limestone with a greasy substance. The drawing is chemically treated with a mixture of gum Arabic and nitric acid, then inked and printed on a large press [7]. At the stage of printing on paper, the surface of the stone is inked, and the stone plate is fixed in the lithography machine, and printing paper is placed on top of it and through strong pressure on the stone plate, the entire drawing is transferred to the printing paper [8]. When paper is used in the lithography

process, it must be pre-moistened, as this process makes it softer and more likely to attract ink and this method allows less ink and less pressure to be used in the printing process [9, 10]. Lithographic papers should also have the following basic properties: flexibility, surface toughness, and a high degree of ink absorption. The flexibility of the paper is necessary due to the movement of pressure needed to print the drawing on the paper, so the paper must be very flexible. The outer surface of the paper must also be somewhat durable so that it can resist nicking (pulling) of its fibers by the printing ink. Lithographic inks have a heavier body and greater viscosity, and therefore exert a drag on the surface fibers of the paper; if the paper is too smooth, the fibers will separate and stick to the surface of the stone when removing the paper, deforming the paper surface. Lithographic papers must also be ink absorbent, mainly for aesthetic reasons. The printed image must appear embedded in the paper rather than appearing on its top surface, and ink deposits must appear smooth and matte [11]. Paper is one of the most versatile materials and since its invention has been the ideal support for writing. Paper consists of fibrous raw materials that give the body of the paper, in addition to fillers and sizing materials in order to improve the properties of the paper and make it more suitable for writing and printing process [12]. Different fibers have different chemical and physical properties [13]. Therefore knowing the fiber type in the paper support of rare fine art prints can help to select appropriate conservation treatment and techniques. The additives were used to improve the properties of the paper and make it appropriate for various printing processes, such as fillers or sizing materials. Due to a lack of research that dealt with fine art prints (such as lithographic plates) with

studies in the field of conservation in general; this study aims to use different analytical techniques identify the type of paper used in fine art prints and the materials included in its composition by examining and analyzing the paper support of one of the historical lithographic plates.

2. Historical background

The investigated plate is one of the plates in The Giovanni Belzoni's atlas, which includes 44 plates under the title "Plates illustrative of the researches and operations of G. Belzoni in Egypt and Nubia". This historical atlas is preserved in the Heritage Library at the Faculty of Archaeology - Sohag University - Egypt. It dates back to the nineteenth century (1820-1822).

Giovanni Battista Belzoni (1778-1823) was an Italian traveler and archaeologist, born in Padua, Italy. He traveled to Egypt in 1815, where he worked as a collector of antiquities for the British Museum. Belzoni left Egypt for England in 1819, and in the following year he published his book known as "Narrative of the Operations and Recent Discoveries within the Pyramids, Temples, Tombs and Excavations in Egypt and Nubia" [14-16], attached with it is an atlas volume of the printed plates under study.

2.1. Description of the plate

This study focuses on the plate No. 19 in the atlas volume, which was printed using the lithography method, it is a copy of one of the wall paintings in the tomb of Seti I in Luxor, known as KV17 (Fig.1a). Seti I tomb (KV17) was discovered in 1817 by Giovanni Battista Belzoni, and since that time, it is known as the most famous burial in the Valley of the Kings (Beban el Molouk) in Luxor, Egypt. It contains the most beautiful wall decoration and writings [17]. The plate (Fig.1b) consists of four figures; the god Osiris sitting on his throne, receiving the homages of a hero (the Pharaoh Seti I), who is introduced by a hawk-headed deity (the god Horus). Behind Osiris stands Hathor. The whole group is surrounded by hieroglyphics and enclosed in a frame richly adorned with symbolical figures. The winged globe is

above, with the wings spread over all and a line of serpents crowns the whole [18, 19].

This plate was drawn by Belzoni and lithographed by Charles Joseph Hullmandel. The plate is folded inside the volume; its dimensions are 57.5 cm × 46.1 cm. It was hand-colored after printing (Fig.1b).

3. Materials and Methods

Different analytical techniques were used to give information about the type and composition of the paper support of historical lithographic plate. A visual examination gives initial information about the type of paper, and will detect the presence of watermarks in the paper support. Optical microscopy (OM) and scanning electron microscopy (SEM) were used to detect the morphology of the paper fibers (i.e. type of fibers). Energy dispersive X-ray spectroscopy (EDX) was applied to detect the elements in the paper support. Attenuated total reflection-Fourier transforms infrared spectroscopy (ATR-FTIR) and X-ray diffraction spectroscopy (XRD) were employed to give information about the additives in the paper support.

3.1. Sampling

Micro-samples (100 µg) of the paper were collected from the plate studied for analysis in order to achieve appropriate results concerning the components of the plate. Since the lithographic plate is hand-colored, a micro-sample of brick red color (as it is the dominant color in the plate) was taken for elemental analysis using EDX, in order to verify the original elements in the paper support, and distinguish them from the extraneous elements of colored materials.

3.2. Visual examination

The technological features of paper support of lithographic plate were examined on a light box. At this stage, the following features were examined:

- papermaking sieve type (woven or laid) attached to the mold,
- Watermark on the paper to determine the time period of the paper and to identify the location of its manufacture [20].



Fig. 1. The lithographic plate studied: (a) Relief from the Tomb of Seti I Pillared chamber F, rear (southwest) wall Seti with Horus before Osiris and Hathor, Lady of the West and (b) lithographic plate.

3.3. Optical Microscope

A Celestron – LCD digital microscope II with a built-in 5MP digital camera was used to identify the cellulose fibers used in the manufacture of paper pulp. The micro-sample collected from the plate was immersed in distilled water in a small beaker, covered, and boiled for about 20 minutes on a hot plate. The residual fibers were taken from the beaker and transferred to a 20 ml test tube with fresh distilled water. The test tube was shaken vigorously until the paper fragments were completely separated into individual fibers. A portion of about 0.5 ml of a uniform mixture of water-suspended fibers was placed on a glass slide. The water on the slide was allowed to partially evaporate until the fibers were barely suspended and then the fibers were distributed evenly on the slide with a probe needle. [13, 20, 21]

3.4. SEM-EDX

A scanning electron microscope (JEOL-JSM-5400LV) was used for the investigation of the surface morphology of the paper [22], and fine gold coater (JEOL-JFC- 1100E) was used.

The elements found in the paper support were determined by the use of EDX analysis, link ISIS Oxford. The quantitative method used was ZAF. The results obtained from EDX were automatically normalized to 100%. SEM-EDX was performed at the Scanning Electron Microscope Laboratory, the central Laboratory unit, Assiut University, Egypt. Some samples were investigated by ESEM (Quanta FEG 250, FEI Company, Netherland) at the Scanning Electron Microscope Unit, National Research Centre, Dokki, Giza, Egypt. These samples were prepared by fixing them on stubs with double-sided cellophane tape and examined without gold coating [23].

3.5. X-ray diffraction

XRD is a valuable technique for studying paper samples. Paper is composed of a matrix of cellulose with a variety of inorganic components. The inorganic components of paper can be investigated using XRD by detecting peaks caused by the crystalline structure of inorganic compounds such as calcium carbonate, alum, etc. [24]. The measurement was performed by a Bruker D8 advance

equipment at the X-ray diffraction Unit, Faculty of Science, Sohag University, Egypt.

3.6. ATR-FTIR Spectroscopy

FTIR analysis was used to identify the sizing materials in the paper support, in addition to the fillers. FTIR measurements were carried out using ATR-FTIR Spectrometer (Bruker Alpha) at the Microchemical Analysis Unit, Faculty of Science, Sohag University, Egypt. The instrument was configured with ATR sample cell including a diamond crystal with a scanning depth up to 2 micrometers. The lithographic plate was gently and carefully placed in contact with the surface of the crystal then locked in place with a “clutch-type” lever before measuring absorbance. No sample preparation is needed and the only requirement being that the sample is in intimate contact with the crystal surface. The spectrum was recorded in absorbance mode over the spectral range $4000\text{--}400\text{ cm}^{-1}$ with a resolution of 4 cm^{-1} .

4. Results and Discussion

4.1. Visual assessment

By examining the paper (i.e plate no.19) (Fig.2a) using the light box, it was found that the paper was woven type, where there are no wire marks. The paper was also manufactured by James Whatman Paper Mill in 1821 according to the watermark found on the paper (Fig.2b and 2c). Whatman paper is a type of wove paper developed by elder James Whatman in the mid-1750 to suit the artistic printing processes [25]; it was developed by using a woven screen. This type of paper is characterized by its smooth and uniform surface [26], which was made from rags and then strengthened by immersing it in a gelatin solution after manufacture [25]. Early European papers (before the mid-nineteenth century) were created from old rags (linen and/or cotton) [27]. When technology moved along the Silk Road, from the Islamic world to Europe, rags were clearly employed as a key source of fibers. Rags were treated with alkaline solutions by hand papermakers in Europe to aid in their conversion to tractable fibers for papermaking [28]. Gelatin was added as a sizing agent [27],

where it was extracted at that time from parchment, scrolls and pieces (trimmings from limed and de-haired hides when shaping prior to tanning), among other sources. Then alum was added (this was potash alum and not the pure aluminum sulphate used in modern times) [25] to paper as a precipitating agent during gelatin sizing [29]. The addition of alum (aluminum potassium sulfate) allows the following: a) it slows the growth of microbes, which would otherwise rapidly putrefy the gelatin, b) it increases the paper's resistance to liquids, c) it reduces the concentration of gelatin required, and d) according to Barrow, it helps to stabilize the viscosity of the gelatin solution at various temperatures and concentrations [30, 31]. In addition to alum, white vitriol (zinc sulphate) was added to the gelatin solution [25]. Importantly, lime, i.e. calcium hydroxide, was added to the fibrous slurry before the beating process [27], probably to facilitate maceration of the rags by swelling the fiber [33, 32]. As an alkali, lime served to swell the cellulose, opening it up and making it more susceptible to chemical action by the enzymes secreted by the organisms present during the fermentation [30]. Lime reacts with CO_2 from air and forms calcium carbonate, which provides the alkaline reserve for the paper. As a result, old papers may show alkaline character even today [27]. Therefore, according to the type of paper and the watermark, it can be concluded that the paper is possibly made of rags (linen and/or cotton fibers). It was sized with a solution of gelatin with alum.

4.2. Fiber identification

Cotton and linen fibers have very special morphological characteristics that can be used for their identification under microscope [21]. Cotton fibers show ribbon-like twists and birefringence; while Salient features used to identify flax fibers are transverse dislocations, which are called cross-thatches or nodes [34]. By examining the paper fibers using both optical microscope (Fig.3) and scanning electron microscopy (Fig.4), it was found that the paper was made of both cotton and linen fibers.

Other images obtained by (SEM) of the surface view of the paper are shown in (Fig.5)

in which the surface micrographs show a layer coating the cellulosic fibers. This layer

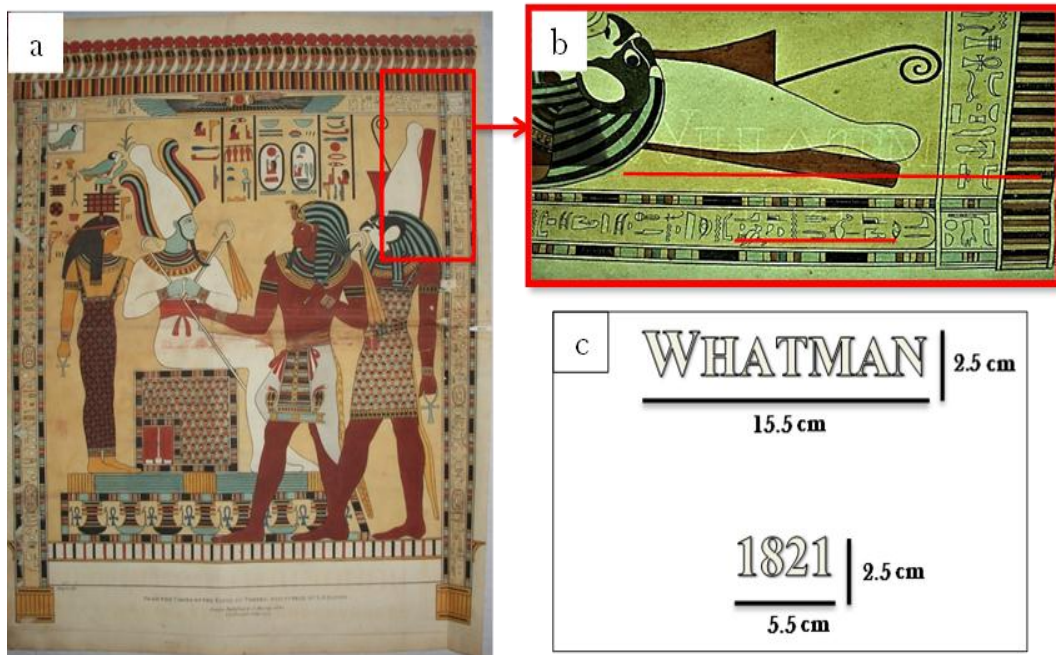


Fig. 2. Watermark in the lithographic Plate, (a) location of the watermark in the Plate, (b) A photo of the plate on top of the light box, (c) The shape and dimensions of the watermark.

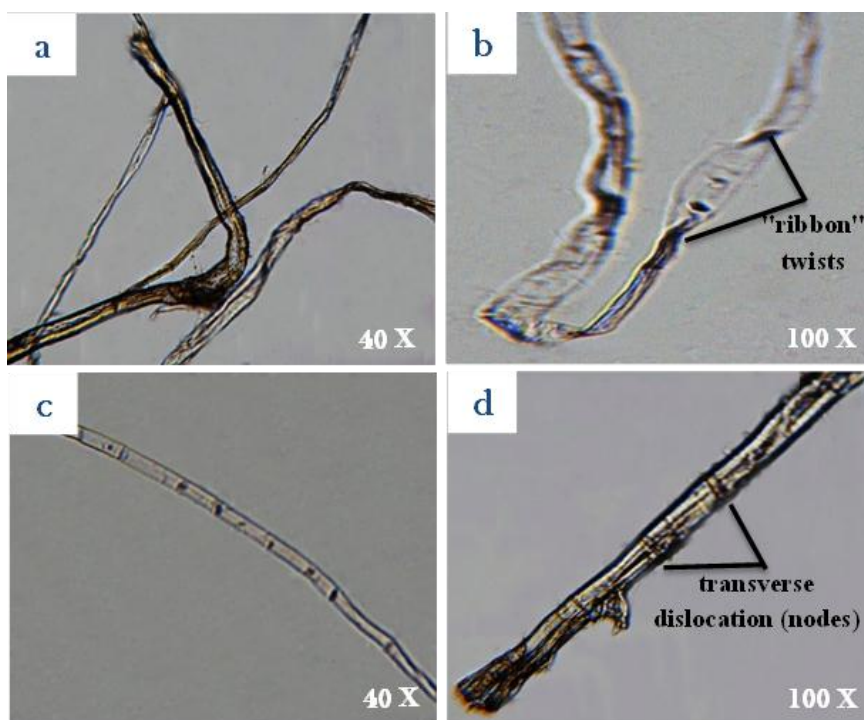


Fig. 3. Optical microscopy (OM) shows the presence of both cotton (a, b) and linen fibers (c, d) in paper support of the lithographic plate.

could be the result of surface sizing, which was intended to reinforce this sort of paper

so that it could be used for drawing and printing.

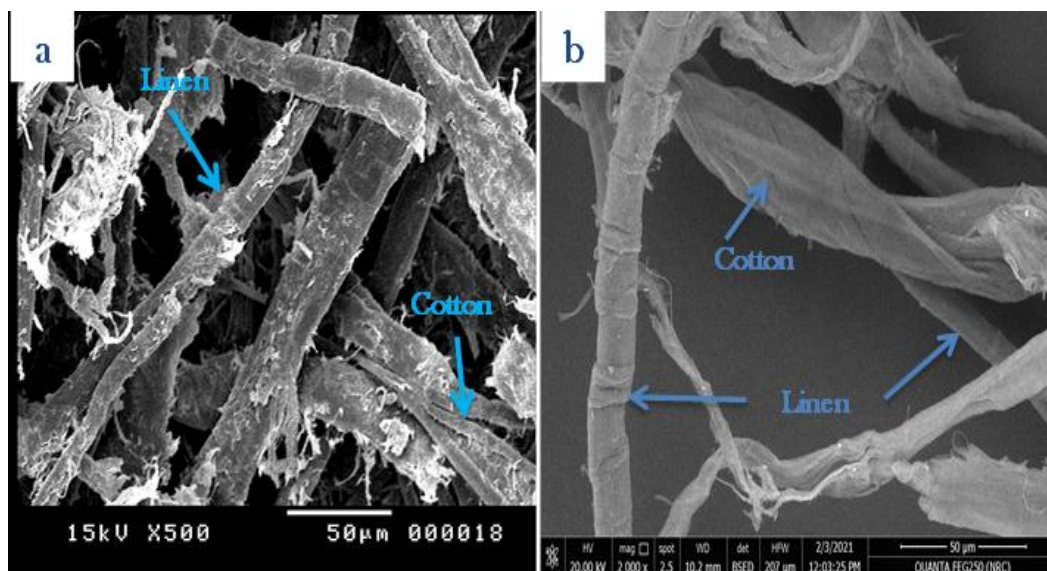


Fig. 4. SEM showing the Cotton and linen fibers, with pictures a and b from different locations of paper support.

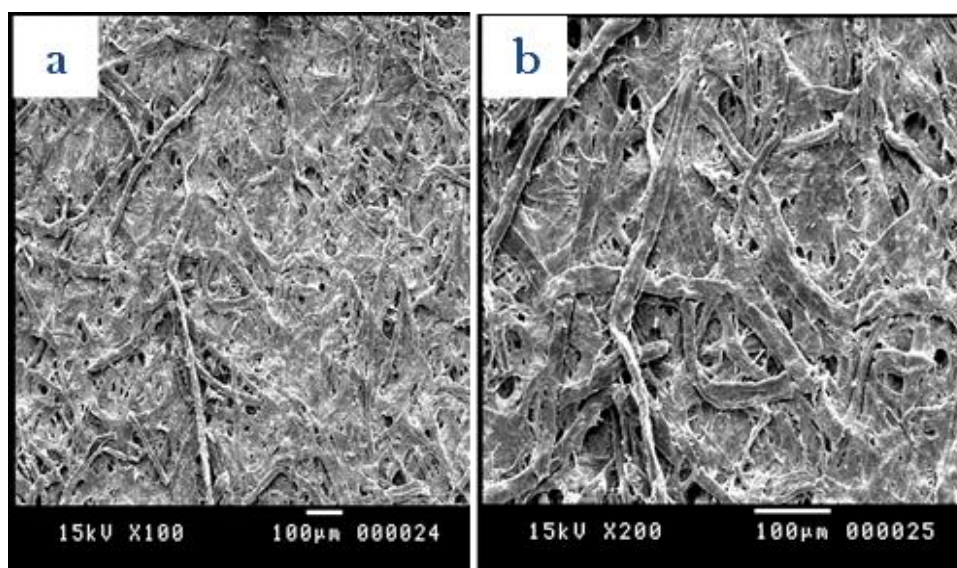


Fig. 5. SEM images of paper support of the lithographic plate show surface morphology of paper, Pictures a, b and c at magnifications of 100, 200× show the presence of *layer covering the fibers*.

4.3. EDX and X-Ray Diffraction Analysis

An elemental analysis was undertaken to further investigate the fillers or other materials used in the paper support of lithographic plate. The EDX results (Table. 1) showed the presence of silicon in the sample, as well as sulphur, aluminum, iron, copper, zinc, potassium and calcium (Fig. 6a). The results also showed a strong variation of the inorganic elements present in the sample, having the highest content in sulphur and calcium. The presence of these elements can be explained either by the use of specific fillers and sizing materials or as originating from the cellulose sources, impurities in the fillers, contaminant either from the manufacturing process, or from surrounding environmental conditions. The presence of calcium in a high percentage in the paper may confirm presence of calcium carbonate in the paper support, resulting from the reaction of calcium hydroxide residues (lime) with atmospheric carbon dioxide, which was added by European paper makers to the fibrous slurry before the beating process. The presence of aluminum, potassium and sulfur may be due to the alum ($KAl(SO_4)_2 \cdot 12H_2O$), which was added to the gelatin during the sizing process [35, 36]. The presence of silicon in the sample in a small percentage may be due to the accumulation of dust particles on the surface of the paper. As for iron, copper and zinc, their presences are due to the pigments in the Plate (Fig. 6b) (Table. 1). Since these elements are transition metal ions, they migrate through the paper from colored areas to surrounding areas [37].

The presence of both calcium carbonate and alum in the paper is confirmed by X-ray diffraction analysis. The main peak identified in the X-ray diffractograms (Fig.7) is cellulose I at 14.53, 16.85, 22.74 2 θ . Calcium carbonate is identified in the sample by the peaks appearing at 29.40 degrees [33]. Among the other characteristic peaks of calcium carbonate which appeared in the sample the 2 θ peaks appeared at 36.08, 39.48, 43.28, 47.58, 48.58, 56.68 and 57.48 [24]. As for the potash alum, it was identified in the sample by the peaks that appeared at 16.26, 17.82,

20.61, 21.874, 27.38, 29.31 and 32.00. These peaks of diffraction match very well the reported values of the peaks for potash alum crystal [38, 39].

Table 1. EDX Analysis of paper sample and paper sample with pigment

<i>Elements (wt.%)</i>	<i>Paper without pigment</i>	<i>Paper sample with reddish brown pigment</i>
<i>Al</i>	<i>10.8</i>	<i>3.0</i>
<i>Si</i>	<i>3.4</i>	<i>0.9</i>
<i>S</i>	<i>31.7</i>	<i>30.9</i>
<i>K</i>	<i>5.7</i>	<i>-</i>
<i>Ca</i>	<i>20.8</i>	<i>31.3</i>
<i>Fe</i>	<i>3.4</i>	<i>30.3</i>
<i>Cu</i>	<i>16.1</i>	<i>2.3</i>
<i>Zn</i>	<i>8.1</i>	<i>1.3</i>

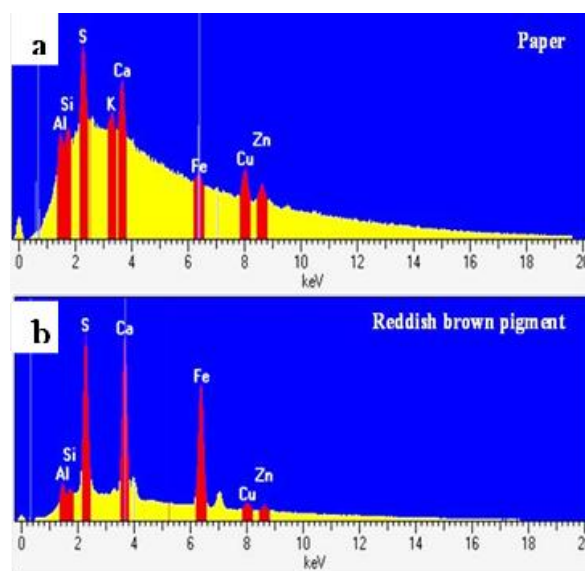


Fig. 6. EDX analysis of paper (a) and pigment on paper (b)

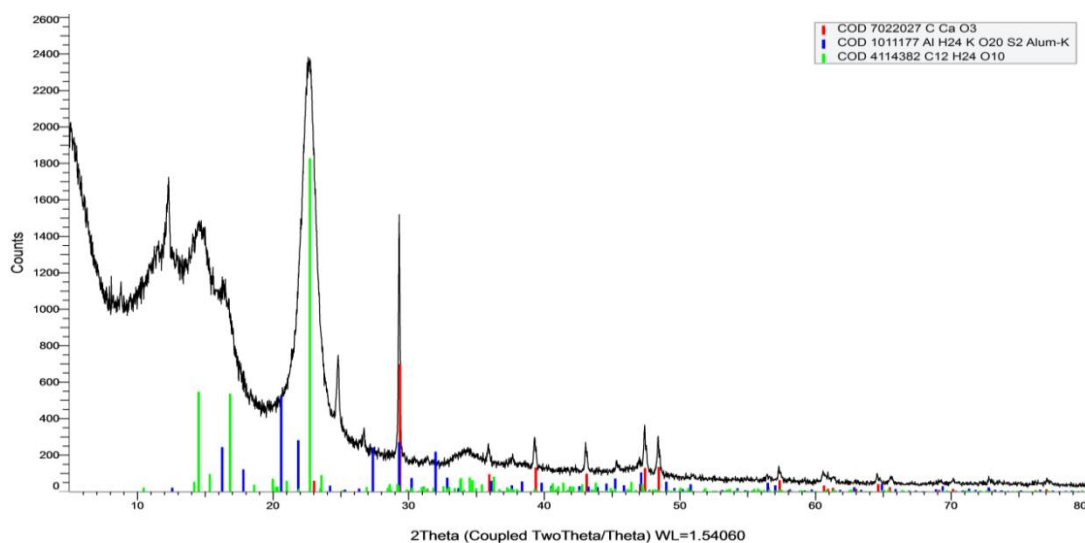


Fig. 7. X-ray diffraction pattern of paper sample from lithographic plate, which confirms the presence of both alum and calcium carbonate

4.4. ATR/FTIR Spectroscopy Analysis

As mentioned earlier, ATR-FTIR spectra were recorded on paper samples, to confirm the presence of gelatin, alum and calcium carbonate. These components can be identified by their characteristic absorption bands (table.2). The recorded spectra of paper sample is shown in (Fig. 8). Gelatin was identified in the examined sample by the amide I (Typical peptide C=O) and amide II (Typical peptide N-H) absorption bands at about 1643 cm^{-1} and 1560 cm^{-1} respectively [12, 40-45] and amide III (Typical peptide N-H) absorption band at 1315 cm^{-1} [13, 40-43]. As for calcium carbonate in the sample, its presence was confirmed by absorption band in the fingerprint region at 1424 cm^{-1} and absorption band at 875 cm^{-1} [13, 21, 24, 27, 33, 42, 43, 45-49]. A broad peak at 1424 cm^{-1} includes the vibration of C = O bonds in the carbonate ion (CO_3^{2-}) [50, 51], and the band at 875 cm^{-1} belongs also to the C=O bonds in carbonate anion [50]. Alum was detected in the sample by the presence of a band around 1106 cm^{-1} [46]. Although the most intense peak for alum is around 1106 cm^{-1} it overlaps with cellulose bands in the fingerprint region [13], but the presence of alum can be confirmed by FTIR analysis by the presence of sulfates

(SO_4^{2-}). The presence of sulfates was detected in the sample at 1158 cm^{-1} and 665 cm^{-1} [13, 43, 47].

5- Conclusions

The historical paper samples belonging to the lithographic plate published in 1820 were analyzed to identify the type of paper support, and the materials included in its composition. The historical plate has been investigated using different analytical techniques such as visual assessment, optical microscopy, SEM-EDX, ATR/FTIR spectroscopy and X-ray diffraction. The results of these investigations (supported by the historical background) confirmed that the paper support of the lithographic plate is woven paper, which was made from rags (both of cotton and linen fibers). Gelatin and alum were used in manufacturing of the paper of lithographic plate, as a sizing agent to strengthen this type of paper. In addition, the presence of calcium carbonate in the paper is attributed to the lime that was added to the pulp before or during the beating process. The main conclusion of the study is that a correlation between the age of the papers, the manufacturing process, paper origin and the analytical results could lead to the development of a

database for the identification of historical paper.

Table 2. ATR/FTIR analysis of paper support of the lithographic plate

Component	Wavenumber cm ⁻¹	Assignment	In historical plate no.19	Reference
Gelatin	~ 1644	Typical peptide C=O (Amide I band)	1643	12, 13, 42 , 44, 45
	1500-1565	Typical peptide N-H (Amide II band); C-N stretching vibration	1560	12, 13, 41, 44, 45, 48, 52
	1315-1320 (Cellulose, gelatin)	CH wagging; Typical peptide N-H (Amide III band)	1315	13, 43
Alum KAl(SO₄)₂·1 2H₂O	~ 1105 (Cellulose, Alum)	Asym. in-plane ring stretch in β-glycoside (C1-O-C4) bond	1106	13, 43
	~ 670	Sulfates (SO ₄ ²⁻) bending vibration	665	13, 33, 43
	~ 1155 (Cellulose, Sulfates)	Asym. ring breathing in β-glycoside (C1-O-C4) bond; Sulfates (SO ₄ ²⁻) asym. valence vibration	1158	13
Calcium carbonate	~ 1425 (Cellulose; CaCO ₃)	CH ₂ bending; asym. C-O stretch in CaCO ₃ (H-C-H and O-C-H in plane bending vibrations)	1424	13, 24, 41, 47, 51
	~ 875	Sym. C-O stretch of CaCO ₃ (C-O out-of-plane bend in the CO ₃ ²⁻)	875	13, 21, 27, 33, 42, 43, 44, 45

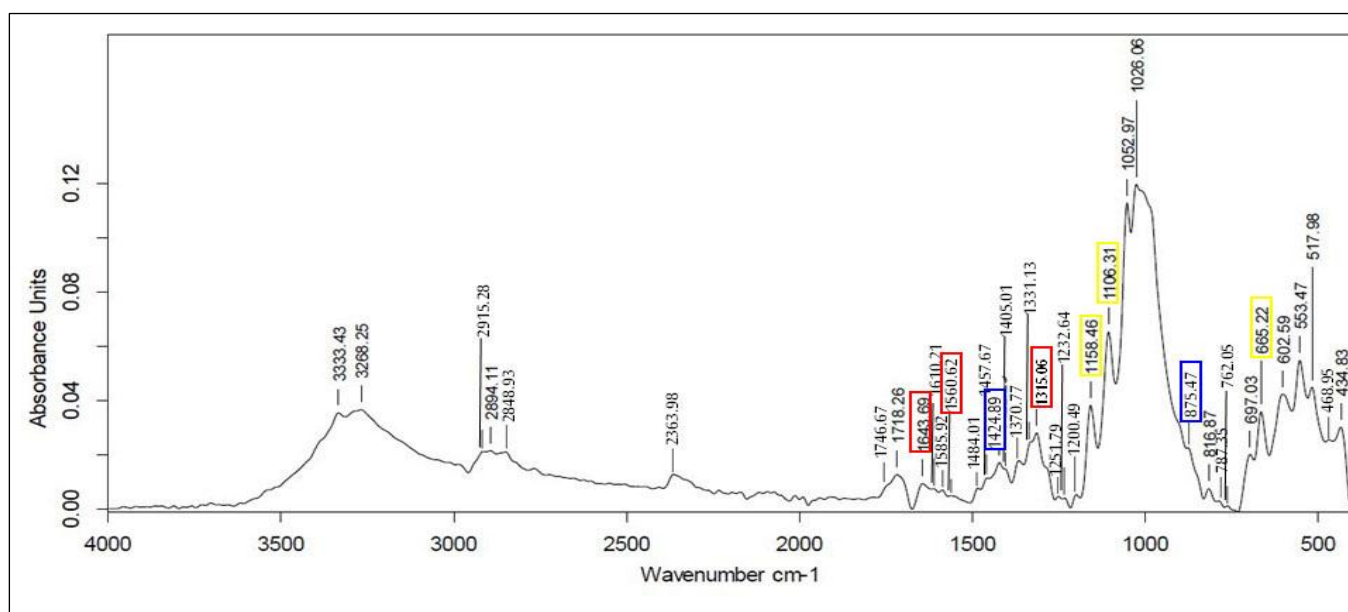


Fig. 8. ATR-FTIR Spectrum of paper sample from historical lithographic plate, it shows the characteristic bands of calcium carbonate (blue), gelatin (red) and alum (yellow) in the spectrum of the paper sample.

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