A REVIEW ON METHODS OF ANALYSIS OF THE PIGMENTS AND INKS IN ILLUMINATED MANUSCRIPT

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ABSTRACT

Ink and pigment significantly impact historical context because of their extensive colour palette. In modern and contemporary times, using coloured minerals, other natural materials, synthetic pigments, and inks contributed to creating an accessible colour palette. Therefore, this study aims to review the current methods for analyzing pigment and ink properties and characteristics to gain insights into the historical context of illuminated manuscripts. The study aims to achieve three objectives: firstly, identifying and evaluating the most effective and reliable methods for examining pigment and ink properties; secondly, determining suitable non-destructive and destructive approaches to obtain accurate information about the materials' composition and characteristics; and thirdly, enhancing the understanding of the historical and cultural significance of illuminated manuscripts by analyzing the materials used in their creation. Illuminated manuscripts are valuable artefacts that provide significant historical and cultural insights. The materials used in creating these manuscripts, such as pigments and inks, offer valuable clues about the artistic techniques, cultural influences, and historical context of the period in which they were produced. However, analyzing these materials requires specialized methods and techniques. The problem addressed in this study is the need to identify and evaluate the most effective and reliable methods for examining pigment and ink properties in illuminated manuscripts. This includes determining the appropriate non-destructive and destructive approaches that can provide accurate information about the materials used, their composition, and their characteristics. It is found that the instrumental analyses that have been used are spectroscopy and microscopic analysis through non-destructive and destructive approaches. The destructive analysis showed that a positive test that confirms the presence of an element is usually more reliable than a negative test that confirms its absence, while ink formulation and chemical composition are not evaluated using non-destructive ink analysis procedures. In conclusion, the study contributes to the field of art history and conservation by determining practical methods for analyzing pigment and ink qualities in illuminated manuscripts, which ultimately improves our understanding of their historical and cultural relevance. Identifying the suitable analytical approaches that can be implemented through various case studies from other researchers' work experience is crucial.

Keywords: Ink, Illuminated Manuscript, Pigment, Scientific Analysis

1.0 INTRODUCTION

As a valuable cultural heritage item, illuminated manuscripts have various colour palettes. Melo et al. (2016a) contributed to developing new methodologies that enable them to define and objectively quantify colour and colour loss in 12th and 13th-century manuscripts. The objective was to learn about the processes employed in colour production through material analysis. Analyses were conducted on inks and pigments, as well as binding medium compositions and additives. In addition to the analytical techniques, developing a database of references for ink and pigment was essential. Analytical methods include recreating the processes stated in the source substance, identifying the particle structures, and comparing the outcomes to the original colour. Melo et al. (2016b) stated that this leads to the development of essential compounds verified against the originals and used to evaluate and enhance the analytical procedures used to identify the originals. Pigment identification is employed to fully understand how the original artist worked, match colours for restoration, and determine compatibility with chemical treatment and authenticity. A sample of the pigment can be taken for analysis destructively, or it can be done in a non-destructive manner without sampling.

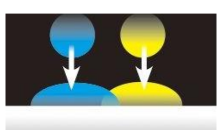
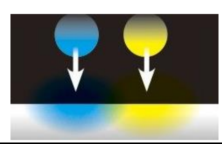
2.0 INK AND PIGMENTS USED FOR WRITING AND ILLUSTRATION

Ink is the most frequent medium for printed and handwritten documents discovered in archives, whereas pigment is described as the colouring agent. Due to the differences in techniques and materials used by early scribes, it was impossible to reproduce the same colour. Scribes began acquiring materials from stationers in the early 13th century, and certain common colourants began to appear. The pigments used in illumination were determined by what was available locally, through trade, and often by what was affordable (Phillips, 2005). Klockenkämper et al. (2000) stated that pigments were utilized for all of the pallet's colours due to their strong colouring power and resistance to temperature, weather, and light changes. The usage of pigments can prevent dyes from fading when exposed to light. The media's colour and light sensitivity are primarily determined by its colourants. Therefore, depending on whether a dye or pigment is used, the risk associated with the preservation varies (McGlinchey, 2012). However, pigments are preferable since they are more stable but costly and only come in a limited colour spectrum. Some natural, mineral, or organic colours have been known since antiquity, while others have only recently been discovered. Carbon black, a very stable pigment produced from elemental carbon, has long been the standard for black. Carbon black ink was often employed to write manuscripts. It was recurrently used for manuscript writing owing to its endurance and consistency (Johnson and Grau-Bové, 2018). Carbon black is a substance generated in various forms, including acetylene black, channel black, furnace black, lamp black, and thermal black. It is obtained from incomplete combustion of coal or petroleum products. Burning organic and inorganic components such as wood, oil, and debris provided the carbon for the black ink.

2.1 Comparing Pigment-Based and Dye-Based Inks

Ink can be categorized into pigment-based and dye-based inks. Pigmented-based inks are powders that are either black, white, or coloured and do not dissolve in binding media or solvents. Pigments are ground to a fine powder to produce the highest colour intensity and brilliance. Pigment sources influence the ink's qualities, such as hues, saturation, brightness, and lightness. Dye-based ink is soluble in water or other liquids. Whereas pigmented inks and dye-based inks may generate more colour. It happens because the dye-based inks are dispersed in liquid form and tend to soak into the paper, making them less effective. Table 1 demonstrates that it exhibits greater brightness under UV light compared to pigment-based ink.

Table 1 A comparison of advantages and disadvantages of Pigment and Dye Inks

	Pigment Ink 	**Dye Ink** 
Advantages	Water resistant and more clarity. Have excellent stability and longevity.	Excellent colour saturation.
Disadvantages	Produce uneven appearance due to reflections. Weak against rubbing and peeling.	It is weak against water and easily gets blurred. Less stability and longevity compared to pigment ink.

Source: http://asia.canon/en/campaign/fineart/related-information-links

2.2 Sources of Pigment

Pigments have been used to embellish different ornamental objects and illuminated manuscripts, as well as in paintings and drawings. Pigments may be categorized into four groups: earth pigment, natural minerals, organic pigments, and synthetic pigments. Earth pigments, such as red ochre, yellow ochre, and terre verte, have been used in various historical manuscripts. Coloured minerals are a term used to describe mineral colours that come from natural sources. Ultramarine, Orpiment, Azurite, and Malachite are just a few examples of natural colours in historical contexts. Due to their resistance to temperature, climate fluctuations, light and other environmental variables, mineral pigments have been used for centuries in art history and archaeology. Mineral pigments are available in a wide variety of colours. Eastaugh et al. (2007) described that pigments might be distinguished by their colour, grain, size, structure, geographical origin, location of usage, and kind of application, as well as by their natural or synthetic origin. The organic pigment consists of plant and animal sources used for making illustrations in manuscript writing. Organic pigments were produced primarily by living organisms that occurred in the environment, such as plants, animals, fungi and microorganisms, and were not chemically changed (Melo et al., 2016b).

Siddall (2018) said that natural pigments come from plants such as madder and indigo, as well as from animals like molluscs (for purple pigments), beetles (for cochineal), and bone and ivory (for black and white pigments). Synthetic pigments are produced from natural compounds but have been chemically changed to form a new material. In the laboratory, synthetic inorganic pigments are created. These pigments comprise metallic compounds such as manganese violet and cobalt blue. Because these pigments are created in a laboratory, they are pure and have tiny particles. Several standard synthetic pigments, such as vermillion, red lead and zinc white, can be used to write ink and illuminate the manuscript. The pigments used are indicative of human cognition and technological choices. Reiche (2019) observed that the availability and cost of raw materials strongly influence the selection of materials used to decorate artwork.

2.3 Ink Varieties Utilized in Manuscript Writing

Black and coloured ink are commonly used for writing and illuminated manuscripts. Black ink can be derived from two types, which are either carbon-based or iron gall. Rabin (2021) discovered that one of the oldest writing and drawing pigments combines soot or charcoal with a binder dissolved in a water-soluble solution. Binders such as gum Arabic (ancient Egypt) or animal glue (China) are the main components of soot inks, together with soot. Lamp black was used to make the highest grade carbon ink, and colours ranging from dark brown to deep blue-black. The first preparation records of iron gall inks date back to the fourth century AD, and they were widely used until the beginning of the twentieth century in official documents once they were hard to erase from paper (Corregidor et al., 2019).

Iron gall ink was popular from the 17th to the 19th century because it was available and used for documentation purposes. Iron gall inks are at the boundary between pigment-based ink and dye-based ink. Due to its low efficiency and high production, iron gall or iron gallotannate ink dominated for centuries. Křížová and Wiener (2015) explained that the oak apples-galls, which are created by the growth factors of gall wasp larvae and whose formation the oak resists against the presence of this insect, are the bases of ink preparation. Galls are high in gallotannins (tannic acid with its monomer gallic acid), and when these tannins react with ferrous salt, they produce a dark violet or black complex, which has been employed for writing. Along with the passage of time, this ink would progressively deepen to a purple-black.

Ink gradually darkened due to atmospheric oxygen oxidation of iron ions from ferrous to the ferric state. Iron gall ink was best used on vellum and parchment because the iron-tannin pigment did not form chemical connections with the paper's cellulose fibres. Mineral pigments were used to produce coloured ink. Different coloured inks were employed to identify the manuscripts' beginning and end,. There are a variety of coloured inks, such as red, blue, green and more. Red ink was mainly utilized to make margins on the left and right edges of the text. Red ink was sometimes used to mark the conclusion of chapters and phrases. There is no smudging with red ink, unlike carbon ink. Red ink that has been freshly written is washable with water, but the ink becomes more permanent as time passes. Red ink was also used to highlight selected words in the manuscript.

3.0 A COMPREHENSIVE REVIEW OF INK AND PIGMENT ANALYSIS METHODS

Fifty articles underwent thorough review, with thirteen meeting specific criteria, including considerations related to destructive and non-destructive methods, as well as the instrumentation utilized for scientific analysis. A systematic search strategy was employed to retrieve relevant ink and pigment analysis papers for illuminated manuscripts. The process involved both keyword-based searches and a snowballing approach. The research began by searching various academic databases like Google Scholar, PubMed, JSTOR, and specialized resources. Keyword combinations such as "ink analysis" and "illuminated manuscripts" were employed during the search. Subsequently, following an initial assessment of search outcomes, the search queries were further optimized. Pertinent scholarly articles were identified, and a comprehensive examination of their respective reference sections was conducted. Furthermore, the research design incorporated the snowball methodology, encompassing an investigation into recent publications that referenced our chosen articles and an exploration of seminal research cited within the selected articles. In the context of this research, "ink analysis" pertains to the chemical examinations conducted on minute ink samples extracted from documents or manuscripts. These analyses serve to unveil valuable insights into the authenticity of these manuscripts by unveiling their chemical and physical ink properties.

Comparative assessments of the chemical and physical attributes of two or more inks can unveil critical information, such as whether they originate from a common manufacturer or derive from the same production batch and the potential dating of the ink formulation. Precise dating tags analyzing the ink's creation year(s) can be derived from this analysis. This study employed two distinct approaches to analyze the colourant and writing ink employed in these manuscripts: destructive and non-destructive analysis techniques. Conducting chemical analyses on the materials used in the manuscript production process is paramount. As Arias (2007) articulated, the data generated through these analyses equips researchers to understand the respective time periods' economic, social, and cultural elements comprehensively. Furthermore, it offers objective insights into the ageing processes affecting the materials in question. Adhering to such analytical methods minimizes the utilization of products or procedures that might otherwise modify the materials' chemical properties, thus avoiding potential harm or irreversible alterations.

According to Arias (2007), it was noted that limited research had been dedicated to the identification and assessment of paper quality. Existing studies tend to be confined to visual examinations for composition determination, an approach with inherent limitations. An important consequence of excessive moisture exposure on paper is the dissolution of stiffeners and adhesives, resulting in paper delamination a consequence of this flawed method. To address these shortcomings, spectroscopic techniques have been employed to discern various fibre types, including cellulose, protein, and synthetic fibres. These techniques are instrumental in identifying multiple absorption bands corresponding to distinct components within the paper. Table 2 presents a selection of scholarly journal articles, each intricately relevant to exploring ink and pigment analysis in illuminated manuscripts.

Table 2 List of selected journals relevant to ink and pigment analysis in illuminated manuscript

No.	Paper Title	Author/Year	Objective
1	The use of digital technology (2.5D) in the authenticity of a manuscript from the Islamic Era	Noshy et al., 2020	Identification of the authenticity of a Quranic manuscript using 2.5D scanning technique
2	Investigation of pigments in Medieval Manuscripts by Micro Raman Spectroscopy and Total Reflection X-Ray Fluorescence Spectrometry	Wehling et al., 1999	Identification of the pigments in old manuscripts or paintings
3	The use of a multi-method approach to identify the pigments in the 12th century manuscripts Liber Floridous	Deneckere et al., 2011	Determine the pigments used in order to search for anachronisms
4	Non-destructive analysis for the investigation of decomposition phenomena of historical manuscripts and prints	Faubel et al., 2007	Developing the consistent set of analytical techniques for the non-destructive measurement of the condition of paper
5	Identification of Colouring Materials and Inks in Manuscripts Using XRF Spectroscopy	Karapanagiotis and Palvatsios, 2014	Identify the illumination materials.
6	Characterization of colourants on illuminated manuscripts by portable fibre optic UV-visible-NIR reflectance spectrophotometry	Aceto et al., 2014	Identification of several colourants used by the ancient illuminators
7	Scientific investigations on paper and writing materials of Mali: A pilot study	Eva et al., 2019	Analyzed physical properties and chemical composition
8	Pigment and ink analysis of the University of Portland Library's Illuminated Manuscripts using Spectroscopic Techniques	Hunter et al., 2021	Analyzed pigments and inks of illuminated manuscripts
9	Pigment analysis by Raman Microscopy of the non-figurative illumination in 16th to 18th century Islamic manuscript	Burgio et al., 2008	Analyzed the non-figurative illumination in the 16th to 18th century Islamic religious manuscripts
10	Raman spectroscopy analysis of pigments on 16-17th century Persian manuscripts	Muralha et al., 2012	Analyzed of pigments on Persian manuscripts
11	Identification of pigment in different layers of illuminated manuscripts by X-ray Fluorescence mapping and Raman Spectroscopy	Mosca et al., 2016	Identification of the pigments employed in the original and overpainting layers
12	Identification of pigments by multispectral imaging; a flowchart method	Cosentino, 2014	Identification of historical pigments applied with gum Arabic
13	Laser-induced breakdown spectroscopy and hyper-spectral imaging analysis of pigments on an illuminated manuscript	Melessanaki et al., 2001	Identification of pigments and inks for dating illuminated manuscripts and systematic characterization

Table 3 provides data from the selected scholarly articles comprising of sampling procedures, instrumentation used, types of analysis conducted, and the findings related to pigments and inks in illuminated manuscripts. It covers a range of analytical methods, both destructive and non-destructive used to study the materials and characteristics of manuscripts, including ink composition and pigment identification. The articles address issues of authenticity, dating, and preservation, offering valuable insights into the scientific analysis of these historical documents.

Table 3 Overview of analysis methods, sampling procedures, type of instruments and findings for ink and pigments in Illuminated Manuscripts

Paper Number	Analysis Methods	Sampling Procedures	Type of Instruments	Findings of Pigment/ Ink
1	Destructive	samples were prepared by hand mill to soften the ingredients and remove the undesirable materials (90% cotton and 10% additives). Moreover, oxides were used to prepare the ink for writing on the experimental samples.	• Scanning Electron Microscopy (SEM)-Energy Dispersive X-Ray (EDX)	• Blue ink • Red ink • Black ink
2	Destructive and non-destructive	Samples can be taken with cotton wool swabs (Q-tips), which causes no visible alteration to the artefact.	• Micro Raman Spectroscopy • Total Reflection X-Ray Fluorescence Spectrometry	• White pigment • Coloured pigment
3		Samples were taken at the same points measured during the in situ analysis. The samples were taken with a cotton swab (Q-tip) by gently rubbing over the manuscript's surface. In the laboratory, the samples were fixed with n-hexane on ultralene, to be appropriate for Raman spectroscopy and X-ray fluorescence spectroscopy.	• In situ Raman Spectroscopy • Confocal Micro-Raman Spectroscopy • Energy Dispersive X-Ray Fluorescence Spectroscopy (EDXRF) • UV-Fluorescence Photography • Infrared • Reflectography (IRR)	• White pigment • Coloured pigments
4		Samples are positioned and scanned by three translational stages (Micos) driven by stepper motors with sub-µm resolution.	• Synchrotron Radiation Induced Micro X-Ray Fluorescence (SR-µXRF)	• Iron Gall ink • Coloured pigments
		Small samples of 10 µm or less allow the acquisition of spectra from single fibres.	• Fourier Transform Infrared (FTIR)	
5	Non-Destructive	No sample pretreatment is required	• X-Ray Fluorescence (XRF) Spectroscopy	• Black ink • Coloured pigments • Iron Gall ink
6		No sample pretreatment is required.	• Fibre Optic UV-Visible-NIR Reflectance Spectrophotometry (FORS)	• Coloured pigments • Metallic pigments • White, grey and black pigment
7		No sample pretreatment is required.	• Light Microscopy • Infrared (IR) Reflectography • Visible Reflectance Spectroscopy • Vibration Spectroscopy (ATR-FTIR and Raman Spectroscopy) • X-Ray Fluorescence (XRF) Spectroscopy	• Carbon ink • Iron Gall ink • Organic pigments • Red pigment
8		No sample pretreatment is required.	• Raman Spectroscopy • X-Ray Fluorescence (XRF) Spectroscopy	• Coloured pigments • Black ink
9		No sample pretreatment is required.	• Raman Microscopy • X-Ray Fluorescence (XRF) Spectroscopy	• Gold pigment • White pigment • Black pigment • Coloured pigments • Carbon ink

10		No sample pretreatment is required.	• Raman Microscopy	• Carbon ink • Coloured pigments
11		No sample pretreatment is required.	• Near-Infrared (NIR) Reflectance Imaging • X-Ray Fluorescence (XRF) Mapping • Raman Spectroscopy	• Coloured pigments
12		No sample pretreatment is required.	• Multispectral Imaging	• Carbon ink • White pigment • Coloured pigments
13		No sample pretreatment is required.	• Laser Induced Breakdown Spectroscopy (LIBS)	• White pigment • Red pigment • Gold pigment • Coloured pigment

4.0 RESEARCH FINDINGS AND DISCUSSION

This section highlights the importance of destructive and non-destructive analysis methods in evaluating textual artefacts, emphasizing the advantages and limitations of each approach. Effective sampling principles ensure the integrity and representativeness of samples, which is crucial for meaningful scientific research and preservation efforts. The following sections delved into various facets of challenges in pigment analysis and explored the hurdles and strategies researchers employ to overcome them.

4.1 Destructive analysis for textual artefacts

The destruction analysis of a document is essential for a more thorough and scientifically valid methodology of document evaluation. The integrity and identical value of the sample are retained as long as the volume of the sample ingested is minimal. The additional knowledge offered through destructive techniques can contribute to solving questions. Microchemical experiments are used to detect a specific component of a functional group. The sample ink chip is examined with various solvents and other components or by heating. A positive test indicating the existence of an element is typically more reliable than a negative test that establishes the element's absence. Positive testing can also indicate a variety of things. According to Noshy et al. (2020), destructive analysis employs several methods, including Laser-Induced Breakdown Spectroscopy (LIBS), Scanning Electron Microscopy (SEM)-Energy Dispersive X-Ray (EDX), and Micro Raman Spectroscopy. Scanning Electron Microscopy (SEM)-Energy Dispersive X-Ray (EDX) is one of the definitive procedures for determining authenticity and identifying forgeries, and it is tough to circumvent. Melessanaki et al. (2001) reported that Laser-induced breakdown spectroscopy (LIBS) was utilized for the first time to identify pigments in illuminated manuscripts from the 12th to 13th centuries AD. Micro Raman Spectroscopy allows the identification of the different materials of the pigment grains (Wehling et al., 1999).

Destructive analysis frequently produces more accurate and dependable results than non-destructive procedures because it allows direct interaction with the sample, allowing for a more thorough investigation. Balamuralikrishnan et al. (2017) discovered that destructive testing allows for a thorough grasp of the material's qualities. Using destructive analysis techniques, researchers can gain precise information on materials' chemical composition and structural properties. Many materials characterization studies focus on employing destructive techniques to characterize the structure and properties of materials more thoroughly (Sharma, 2018). Destructive analysis is an effective method for discovering flaws and defects in various materials. Juan et al. (2021) explained that its effectiveness relies on its capacity to show in-situ information regarding material constituents, such as inclusions, and their prospective correlations with processes and material attributes. Material property validation is critical for ensuring that materials fulfil certain quality criteria. This method ensures that materials will function as intended in various applications.

One of the major disadvantages of destructive analysis is that it permanently damages the sample or material under examination. Destructive analysis can be costly due to the need for replacements, additional testing, or repairs after the analysis procedure. Moreover, if the testing process is intricate or requires significant time, there will be associated costs (Pfattheicher and Schindler, 2015). The costs of destructive testing may outweigh the benefits of the data gathered. Elfil and Negida (2017) described that the destructive analysis method frequently requires examining a small product sample, which is not usually representative of the complete product. This limitation has been extensively studied in the literature. To ensure the representativeness and validity of the results, researchers must carefully choose and examine samples.

4.2 Non-Destructive Analysis for Textual Artefacts

Based on the article review that has been classified in Table 3, mainly all the researchers employed these non-destructive techniques in their analysis. Non-destructive techniques are recommended since they keep the manuscript intact and retain the document's value as evidence. However, one disadvantage of non-destructive approaches is the limited quantity of information and discrimination that some of these methods give. Using the non-destructive method, most of that may be learned about a pigment by inspecting it using a low-powered binocular microscope, which does not need the sample to be degraded. Non-destructive ink analysis techniques do not evaluate ink formulation or chemical composition. Deneckere et al. (2011) emphasized combining analytical spectroscopic techniques, such as Raman spectroscopy and XRF, with imaging techniques, such as UV fluorescence photography and infrared reflectography (IRR), a significant amount of interesting information could be gathered about the pigments used in the manuscripts. X-ray fluorescence (XRF) spectroscopy is used to identify illumination materials, including inks, used on paper in the past (Karapanagiotis and Palvatsios, 2014). According to Aceto et al. (2014), FORS (Fibre Optic Reflectance Spectrophometry) possesses properties that make it well adapted for investigating ancient illuminated manuscripts, and it is more user-friendly than some of the other techniques. For the studies of the original manuscripts, the systematic non-destructive investigation protocol was developed by using Light Microscopy, Infrared (IR) Reflectography, Visible Reflectance Spectroscopy, Vibration Spectroscopy (ATR-FTIR and Raman Spectroscopy) and X-Ray Fluorescence (XRF) Spectroscopy (Eva et al., 2019; Hunter et al., 2021). Raman microscopy and X-ray fluorescence (XRF) also determine the pigments used in each case and establish whether the range of pigments evolved with time (Burgio et al., 2008; Muralha et al., 2012). According to Mosca et al. (2016), near-infrared reflectance imaging and XRF mapping assist in detecting the pigment dispersion in an illuminated manuscript's surface and hidden layers. Cosentino (2014) stated that the application of Multispectral Imaging for assessing broad regions has the benefit of being a rapid and reasonably inexpensive method. Utilizing X-Ray Fluorescence (SR-XRF) and Fourier transform infrared, the elemental composition of pigments and the breakdown processes of writing substrates could be determined.

Non-destructive testing is excellent for evaluating materials and objects without inflicting permanent damage. Understanding the advantages and disadvantages can assist industries in making educated decisions and employing appropriate approaches for efficient and dependable analysis. One of the most notable advantages of non-destructive analysis is that it protects the integrity of the original manuscript. The ability to integrate non-destructive procedures with a variety of analytical and imaging techniques is a significant advantage. When combined with spectroscopic techniques like Raman spectroscopy and XRF, imaging techniques such as UV fluorescence photography and infrared reflectography can yield significant information about the pigments used in the manuscripts. According to Faubel et al. (2007), assessing the status of paper-based cultural artefacts using non-destructive studies is critical for creating a balanced conservation policy.

Despite its many advantages, non-destructive testing can occasionally give limited data and struggle with material discrimination compared to more intrusive approaches. Segovia et al. (2023) also emphasized that these methods may sometimes provide limited detail in their results. The limitation in non-destructive techniques' ability to comprehensively analyze ink formation and its chemical composition underscores the need to develop improved methods, optimizing the balance between preserving the physical integrity of materials and acquiring essential compositional information (Rehrl and Griesser, 2018). In summary, Figure 1 discusses the importance of 2D information and point analysis in the analysis of ink and pigments in illuminated manuscripts. It highlights the significance of microscopic and spectral techniques for understanding the composition and characteristics of these materials.

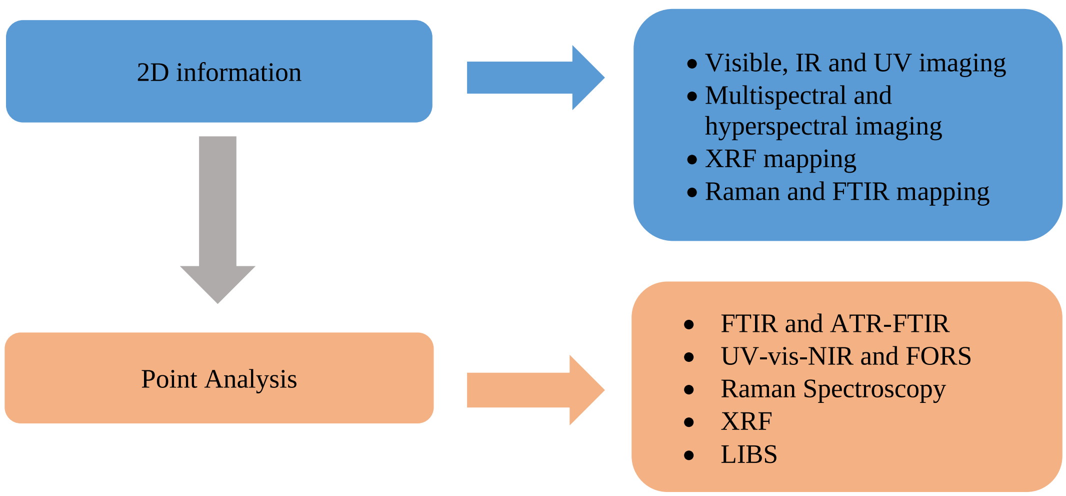


Fig 1: The procedures outline for the scientific analysis of illuminated manuscripts

4.3 Principles of Effective Sampling in Scientific Research and Preservation of Textual Artifacts

Sampling is fundamental to various fields, including scientific research, quality control, and environmental monitoring. It plays a pivotal role in gathering representative data and ensuring the reliability of analyses. This section delves into the principles and procedures of effective sampling, drawing insights from the reviewed literature and research articles. Sampling is crucial in various domains, such as scientific research, quality control, and environmental monitoring. The sample-taking procedures follow two fundamental principles, as outlined by Arias (2007):

1. **Preserving Physical Integrity:** The first principle emphasizes the importance of maintaining the original condition of the text or sample during the sampling process. It is particularly vital when dealing with textual data, such as written texts, manuscripts, or historical records.

Careful handling is essential to prevent damage or tampering that could compromise their integrity and readability. In these cases, non-destructive sampling technologies like photocopying, digital scanning, or high-resolution photography are commonly employed to safeguard physical integrity while collecting the necessary information.

2. **Ensuring Representativeness**: The second principle aims to obtain a sample that accurately reflects the qualities and attributes of the entire population. To achieve this, researchers must ensure that the sample is diverse and covers the full range of variations. This approach enables them to draw meaningful conclusions based on the observed features within the sample. To maintain sample purity, it is essential to minimize contamination from external sources, which can result from various factors such as the inclusion of irrelevant components, cross-contamination with other samples, or external interference. Adhering to proper sampling procedures, rigorous protocol adherence and suitable sample storage conditions are all critical in preventing contamination and preserving the sample's representativeness.

4.4 Challenges in Pigment Analysis

Despite these inherent challenges, it is important to approach pigment analysis with optimism and purpose. Chiari and Scott (2004) emphasize that these challenges should not deter researchers. There are several reasons for this optimism. Firstly, the number of common pigment types, historically limited to a range of 50 to 100, simplifies the landscape of pigments under investigation. Secondly, the compositions of the majority of these pigments are well-documented and widely published, providing valuable reference material. Lastly, these pigments can often be categorized into a concise array of basic colors, which helps streamline identification.

Identifying pure pigments, which are fundamental to this analysis, is generally straightforward, given their availability. Visual inspection, whether with the naked eye or an optical microscope, often proves effective in resolving the identification puzzle. Additionally, applying X-ray fluorescence (XRF) analysis is pivotal in corroborating or refuting hypotheses, particularly in uncertain situations. XRF analysis, when penetrating beyond the surface of materials, yields integrated data from multiple layers, enriching the understanding of pigment composition. However, it is worth acknowledging that there may be complex scenarios where alternative analytical approaches become necessary. This is especially true when pigments are present in trace amounts that conventional methods may struggle to detect.

5.0 CONCLUSION AND RECOMMENDATION

The scientific analysis of inks and pigments in manuscripts has undergone extensive scrutiny, enabling the classification of various ink types, which is fundamental for analytical research on a wide spectrum of inks. Destructive and non-destructive techniques have been employed to examine these manuscripts, each offering unique advantages. Destructive analyses, including Scanning Electron Microscopy (SEM)-Energy Dispersive X-Ray (EDX), Synchrotron Radiation Induced Micro X-Ray Fluorescence (SR-XRF), In situ Raman Spectroscopy, and Laser Induced Breakdown Spectroscopy (LIBS), provide highly detailed information, particularly in terms of elemental composition. However, they come with the cost of potentially damaging valuable samples. Non-destructive techniques, such as X-Ray Fluorescence (XRF) Spectroscopy, Fibre Optic UV-Visible-NIR Reflectance Spectrophotometry (FORS), Raman Spectroscopy, and Multispectral Imaging, have been crucial for preserving valuable manuscripts by allowing elemental composition determination without harming the object. The selection of the appropriate scientific test, whether destructive or non-destructive, should consider factors such as the manuscript's nature, desired information, and available resources. SEM-EDX is ideal for destructive analysis, while XRF Spectroscopy excels in non-destructive analysis due to its adaptability and non-invasiveness.

In light of the considerations outlined, it is recommended that researchers and conservators working on the analysis of inks and pigments in historical manuscripts, when conducting analyses on historical manuscripts or valuable materials, prioritize non-destructive techniques, such as XRF Spectroscopy. This approach minimizes the risk of damaging the samples and helps preserve the integrity of these precious artefacts. Non-destructive methods should be the first choice, especially when dealing with irreplaceable cultural heritage. In cases where a deeper understanding of inks and pigments is required, consider combining multiple analytical methods. When used alongside FTIR, Raman Spectroscopy can provide a more comprehensive analysis, enhancing the depth of insights gained. This multidisciplinary approach can reveal nuanced information about the materials, aiding their identification and characterization. Understand the pivotal role of ink and pigment identification in shedding light on historical usage and dating of manuscripts. Moreover, acknowledge that this analysis informs conservation efforts and can lead to recommendations for enhancing pigment longevity and durability. The insights gained from these analyses contribute to scientific understanding and the preservation of cultural heritage. By following these recommendations, researchers and conservators can conduct more effective and meaningful analyses of inks and pigments in historical manuscripts, contributing to preserving and interpreting our cultural heritage. In conclusion, the detailed analysis and scientific testing of inks and pigments within manuscripts offer valuable insights that contribute to a deeper understanding of historical artefacts and aid in their preservation and future enhancement. Researchers must carefully select the most suitable methodologies, considering each manuscript's unique characteristics and value.

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