Research

Development of a Fiber Database for Timurid-era Papers to Facilitate Manuscript Classification and Dating

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Abstract

The aim of this study is to investigate a comprehensive dataset related to fiber identification in historical manuscript papers. Previous research in this field was limited to small collections and did not focus on specific historical periods. To address this gap, this study examines the fiber characteristics of 50 historical manuscripts from the Timurid period, preserved in the manuscript repository of the Astan Quds Razavi Library. The fibers were identified using microscopic methods based on morphological and biometric features, along with Fourier-transform infrared spectroscopy (FTIR) analysis. According to historical sources, non-wood fibers such as flax and hemp were traditionally used in early Iranian papermaking. The results of this study confirmed that the fibers used in the production of the 50 Timurid paper samples were primarily hemp and flax. The pulp also contained calcium carbonate and alum with very low lignin content, and the sizing agents were starch and gelatin. Furthermore, it was determined that if a sample contains cotton fibers, it does not belong to the Timurid period. Additionally, based on historical sources, it can be inferred that by recognizing the structural similarities and differences among manuscript papers, it is possible to approximately date undated samples through comparative analysis. However, further research and more extensive studies are needed to solidify these findings.

Keywords: fibers, historical papers, manuscripts, Timurid, microscopic analysis, FTIR spectroscopy.

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1. Introduction

Manuscripts from the Timurid period (14th-15th centuries CE / 8th-9th centuries AH) represent some of the most significant cultural artifacts of Iran and Central Asia, reflecting a golden age of artistic and intellectual achievement. These manuscripts, preserved in repositories such as the Astan Quds Razavi Library in Mashhad, Iran, offer invaluable insights not only through their textual and artistic content but also through their physical composition. The paper used in these manuscripts, often crafted with meticulous care, serves as a historical record of papermaking techniques, material availability, and cultural practices of the era.

Traditionally, scholars have relied on colophons, which are scribal notes detailing the date, place, and scribe of a manuscript, for authentication and dating. However, many Timurid manuscripts suffer from incomplete, illegible, or missing colophons due to physical deterioration, insect damage, or intentional removal. This limitation necessitates alternative methods, such as material analysis, to determine a manuscript's age, provenance, and authenticity. By examining the fibers and additives used in papermaking, researchers can uncover details about historical craft practices and regional material preferences.

Despite the richness of collections like that of the Astan Quds Razavi Library, few studies have systematically analyzed the material composition of Timurid manuscript papers using modern scientific techniques. Previous research in this field was limited to small collections and did not focus on specific historical periods, leaving a significant gap in our understanding of Timurid papermaking traditions. According to historical sources, non-wood fibers such as flax and hemp were traditionally used in early Iranian papermaking. However, comprehensive scientific validation of these historical accounts through systematic analysis of dated manuscripts has been lacking.

This study addresses this gap by investigating 50 dated manuscripts from the Timurid period, employing light microscopy and Fourier-transform infrared spectroscopy (FTIR). The objectives are to characterize the fiber types, identify chemical additives, and establish a material profile for Timurid papermaking. The Timurid era, centered in cities like Herat and Samarkand, was marked by standardized artisanal practices, suggesting a potential uniformity in papermaking materials and techniques. These findings aim to enhance manuscript authentication, support conservation efforts, and contribute to the broader understanding of Islamic papermaking traditions. By combining morphological and chemical analyses, this study provides a scientific foundation for distinguishing Timurid papers from those of other periods and offers a model for non-destructive heritage studies.

2. Research Methodology

The study analyzed 50 dated manuscripts from the Astan Quds Razavi Library, selected based on their confirmed attribution to the Timurid period (14th-15th centuries CE). The manuscripts, written in Arabic, Persian, and Samarqandi (Uzbekistani), had precise dates of transcription provided by scribes at the end of each manuscript. To ensure preservation, sampling was minimally invasive, targeting small fragments (approximately 1 mm^2) from ink-free, unillustrated, and undamaged margins. This approach avoided compromising the manuscripts' textual or artistic integrity. Four reference fiber samples with known compositions of flax, hemp, jute, and cotton, which were native to Iran and historically used in papermaking, were prepared for comparative analysis.

Light microscopy was used to examine fiber morphology. Samples were stained with Herzberg, Graff, and Selg reagents, which produce distinct color reactions to differentiate fiber types and assess lignin content. The staining procedures followed TAPPI standard T 401 om-93. For samples that did not disintegrate in distilled water, a 1% sodium hydroxide solution was used, followed by washing with 2% hydrochloric acid and repeated rinsing with distilled water. Microscopic slides were prepared and examined to determine fiber composition.

FTIR spectroscopy was conducted using a Nicolet Nexus 470 spectrometer equipped with a PIKE MIRacle attenuated total reflectance (ATR) accessory, featuring a ZnSe/Diamond composite crystal. Spectra were recorded in the range of 4000-600 cm^{-1} with a resolution of 4 cm^{-1}, and each spectrum represented the average of 32 individual scans collected at room temperature. The Omnic software was used for spectrum control and processing. This technique identified molecular bonds in the paper matrix, focusing on cellulose, lignin, and additives such as calcium carbonate, starch, and gelatin. The combination of microscopy and FTIR provided a comprehensive dataset, integrating morphological and chemical evidence to characterize Timurid papermaking materials.

3. Discussion

3.1. Fiber Identification and Morphological Characteristics

Microscopic analysis identified flax and hemp as the exclusive fiber types in all 50 samples, confirming the historical accounts regarding the use of non-wood fibers in early Iranian papermaking. Flax fibers were characterized by long, slender shapes with pointed ends and distinct cross-markings. These fibers showed dislocations and transverse nodes, particularly visible with staining, and often exhibited V-shaped markings. The fibers frequently appeared in bundles, with associated cells that were generally narrower than those found in jute or hemp. Epidermal cells with smoother walls, unlike the toothed type often found in grasses, were also present.

Hemp fibers exhibited coarser textures, thicker cell walls, and frequent transverse nodes. They showed numerous dislocations and longitudinal lines, terminating in blunt ends, although irregular ends commonly resulted from processing. The distinction between hemp and flax could be challenging, but hemp fibers were generally wider and had different associated cells. The fibers from textile sources typically lacked associated cells, whereas fibers from raw materials might contain vessel elements, parenchyma, and short fiber cells.

Notably, no cotton fibers were detected in any of the 50 samples, challenging assumptions about cotton's widespread use in Islamic papermaking during this period. Cotton fibers, which are smooth, ribbon-like and easily identifiable by their twisted nature, were completely absent. The staining with Herzberg and Graff reagents produced reddish-purple hues, indicating very low lignin content, consistent with the use of retted, recycled textile fibers rather than raw plant stalks. This finding aligns with historical records indicating that during the early centuries of Islam, paper raw materials were exclusively recycled fabric textiles.

3.2. Chemical Composition and FTIR Spectroscopic Analysis

FTIR spectroscopy confirmed the morphological findings and provided detailed information about the chemical composition of the papers. Cellulose was identified as the primary structural component, with prominent peaks at 1030 cm^{-1} (C-O stretching), 1105 cm^{-1} (C-H stretching), and 1200 cm^{-1} (C-O-C stretching). The spectra showed very weak lignin signals at 1506 cm^{-1} (aromatic skeletal vibration) and 1740 cm^{-1} (C=O stretching), corroborating the low lignin content observed in staining. The absence of a peak at approximately 1729 cm^{-1}, which is present in untreated fibers and corresponds to C=O stretching of acetyl groups in hemicellulose, confirmed that the fibers had undergone extensive processing, including alkalization during textile production and subsequent papermaking.

The comparison between the 50 historical samples and reference fibers revealed subtle but significant spectral differences. Flax fibers showed distinct absorption ranges at 2915-2923 cm^{-1} and 2841-2848 cm^{-1}, while hemp fibers exhibited peaks at 2895-2897 cm^{-1} and 2939 cm^{-1}. These differences, though minor, allowed for the differentiation of fiber types. The intensity of the OH stretching peak around 3200 cm^{-1} was reduced in historical samples due to the removal of hemicellulose and lignin through retting and bleaching processes.

Quantitative analysis based on the absorption ratios at 1595 cm^{-1}, 1105 cm^{-1}, and 2900 cm^{-1}, representing organic content, lignin content, and cellulose content respectively, further supported the identification. The base fiber in the majority of samples was identified as hemp with some flax content. The absence of cotton was confirmed by the lack of characteristic absorption bands associated with cotton fibers, particularly the absence of lignin-related peaks at 1510 cm^{-1} and 1505 cm^{-1}.

3.3. Additives and Sizing Agents

The FTIR analysis consistently detected the presence of additives across all samples. Calcium carbonate was identified through a sharp peak at 1415 cm^{-1}, corresponding to the asymmetric stretching of carbonate ions. The presence of this filler in all samples, even though the selected manuscripts had never undergone conservation treatment, suggests it was an integral part of the original papermaking process. Starch was detected through broad O-H and C-H peaks between 3000-3500 cm^{-1}, although the quantity appeared to be very low, suggesting it may have been applied as a surface coating rather than incorporated into the pulp. Gelatin was identified through Amide I and II peaks at 1650 cm^{-1} and 1540 cm^{-1}, consistent with historical descriptions of Islamic papermaking where gelatin was used as a sizing agent. The presence of these additives enhanced paper opacity, smoothness, and ink absorption, contributing to the high quality of Timurid manuscripts suitable for calligraphy and illumination.

3.4. Standardization and Regional Practices

The uniformity in fiber types and chemical profiles across all 50 samples, despite their diverse origins (including manuscripts from Mashhad, Samarkand, and Mecca), suggests a highly standardized papermaking process during the Timurid period. This standardization likely reflects regulated workshops in cultural hubs such as Herat and Samarkand, where artisanal practices were formalized under Timurid patronage. The exclusive use of flax and hemp, with no cotton despite its availability, points to deliberate choices by Timurid papermakers. The superior durability of flax and hemp fibers, combined with established regional traditions, may have influenced this material selection. The low lignin content, confirmed by both staining and FTIR, indicates advanced fiber processing techniques such as retting and bleaching, which produced high-quality paper suitable for calligraphy and illumination.

4. Conclusion

This study confirms that Timurid-period manuscript papers were primarily composed of flax and hemp fibers, sourced from recycled textiles, with no evidence of cotton usage. The presence of additives like calcium carbonate, starch, and gelatin, combined with low lignin content, reflects sophisticated papermaking techniques tailored for high-quality manuscripts. These findings align with historical accounts of Islamic papermaking and provide a scientific basis for authenticating and dating Timurid manuscripts, particularly those lacking colophons. The combination of light microscopy and FTIR spectroscopy proved highly effective, offering non-destructive or minimally invasive methods ideal for fragile manuscript studies. These techniques provided complementary data, with microscopy revealing fiber morphology and FTIR identifying chemical composition. The results contribute to the material history of Iranian papermaking and enhance conservation practices by establishing a clear material profile for Timurid papers. This study underscores the value of interdisciplinary approaches in cultural heritage research, bridging art history, material science, and conservation to preserve and interpret these invaluable artifacts. Future research could expand this approach by analyzing manuscripts from other Islamic dynasties, creating a comparative database of fiber compositions and additives to refine our understanding of papermaking evolution across Islamic history.