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Research paper

Visible Diffuse Reflectance Spectroscopy Coupled with PCA–LDA for Non-Destructive Paper Age Prediction

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Abstract

Age prediction of paper samples belonging to the same commercial brand but manufactured in different years was carried out using non-destructive Visible Diffuse Reflectance Spectroscopy (DRS) combined with PCA–LDA-based chemometric analysis. Initially, the classification model was trained using the reflectance spectra of paper samples collected from different production years. The performance of the developed model was subsequently evaluated using unknown samples that were not included in the training stage. Blind test results demonstrated that the model was capable of correctly predicting the age of 50% of the unknown samples. Considering that the temporal difference between some samples was as small as one year, this level of prediction accuracy can be regarded as significant. The findings indicate that visible diffuse reflectance spectroscopy, coupled with chemometric techniques, has considerable potential as an accessible, cost-effective, and non-destructive approach for paper age prediction. Unlike previous studies that commonly employed temporal intervals of four to five years, the present work successfully investigated naturally aged paper samples with intervals as small as one year.

1. Introduction

Paper, as it is known today, originated in China and has played a fundamental role in human civilization since its invention [1]. Over time, paper has been extensively used in nearly all aspects of daily life, including books, historical manuscripts, banknotes, invoices, handwritten notes, and many other documentary materials [2]. Owing to its widespread and universal application, paper serves as a carrier of a vast amount of cultural [3], historical [4], and forensic information [5]. Consequently, paper has become an important subject of investigation in archaeological tracing, cultural dissemination studies, forensic identification, and related scientific fields. The present study was conducted on Pars paper, a commercial paper product manufactured from sugarcane bagasse fibers [6, 7]. All paper samples investigated in this research were collected from a personal library collection. The examined Pars paper samples belonged to different production years, with the minimum temporal interval between some samples being only one year. Various analytical methods have been reported for paper dating, document age estimation, and paper classification. The present research lies at the intersection of paper classification and paper age estimation; therefore, analytical approaches applied to both areas are considered relevant. Several destructive and non-destructive techniques have previously been employed for the discrimination and identification of paper materials, including Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR–FTIR) [8-12], Near-Infrared Spectroscopy (NIR) [13, 14], Hyperspectral Imaging (HSI) [15, 16], Raman Spectroscopy [17-19], Laser-Induced Breakdown Spectroscopy (LIBS) [20-24], X-ray Fluorescence Spectroscopy (XRF) [25, 26], Diffuse Reflectance Spectroscopy (DRS) [27, 28], Gas Chromatography–Mass Spectrometry (GC–MS) [29, 30], Ultra-High-Performance Liquid Chromatography (UHPLC) [31], Inductively Coupled Plasma–Mass Spectrometry (ICP–MS) [32], and Neutron Activation Analysis (NAA) [33]. Studies related to paper and document dating are generally divided into two categories: artificial aging studies and investigations based on naturally aged paper samples. Techniques such as Raman Spectroscopy [34, 35], GC–MS [36, 37], FTIR [8, 38], and DRS [39, 40] have been widely applied in these investigations. Deterioration of paper is caused by many factors such as acid hydrolysis, oxidative agents, light, air pollution, or the presence of microorganisms(biores). Paper degradation is caused by several factors such as acid hydrolysis, oxidizing agents, light, air pollution or the presence of microorganisms (biological agents/biores). Paper undergoes various physicochemical changes during natural aging [41]. These factors cause changes in the appearance and color of the paper, which affect the absorption and reflection properties of the paper.

Among the common techniques reported for both paper discrimination and age estimation, Diffuse Reflectance Spectroscopy was selected in the present work due to its low cost, accessibility, and widespread availability in forensic laboratories [27]. Due to the porous nature of the paper surface, the existing reflectance is called diffuse reflectance. The principal novelty of this research lies in the use of real paper samples with temporal differences as small as one year. In contrast, previous studies have generally investigated samples separated by intervals of four to five years. For example, in 2018, Carolina S. Silva and co-workers successfully performed paper dating on samples with a four-year temporal interval using infrared spectroscopy combined with chemometric analysis [8]. Subsequently, in 2020, Jing jing Xia and colleagues collected book paper samples produced between 1940 and 1980 with five-year intervals. Using Infrared Spectroscopy combined with a Sparse Partial Least Squares and Least Squares Support Vector Machine (SPLS–LS-SVM) model, they achieved prediction accuracies reaching 100% under optimal conditions after 100 repeated runs [38]. Despite the increasing number of studies on paper dating, most previous investigations have focused on samples separated by relatively large temporal intervals or on artificially aged papers. Moreover, advanced analytical techniques such as FTIR and NIR spectroscopy are often required. Therefore, the development of a low-cost, non-destructive, and accessible method capable of discriminating naturally aged paper samples with minimal temporal differences remains a significant challenge in forensic document analysis. In the present study, naturally aged paper samples belonging to the same commercial brand (Pars paper) and separated by temporal intervals ranging from one to seven years were investigated using Visible Diffuse Reflectance Spectroscopy combined with chemometric analysis based on PCA and LDA. The primary objective of this work was to evaluate the capability of a low-cost and non-destructive spectroscopic approach for the temporal discrimination and age prediction of paper samples with minimal age differences. Initially, the classification model was trained using reflectance spectra obtained from paper samples corresponding to different production years.

Subsequently, the predictive performance of the developed model was evaluated through blind testing using unknown samples that were not included in the training stage. The proposed methodology was designed to investigate the potential applicability of DRS as an accessible forensic tool for paper age estimation under naturally aged conditions.

2. Materials and methods

2.1. Experimental setup

Diffuse Reflectance Spectroscopy measurements were performed using a Y-branch fiber-optic configuration, as illustrated in Figure 1. The Y-branch optical fiber, with a core diameter of 100 μm, was employed to deliver light from a halogen light source to the paper surface and simultaneously collect the diffusely reflected light from the sample toward the spectrometer. Spectral acquisition was carried out using a Teksan TIDA spectrometer with a spectral resolution of 1 nm within the visible region (400–700 nm).

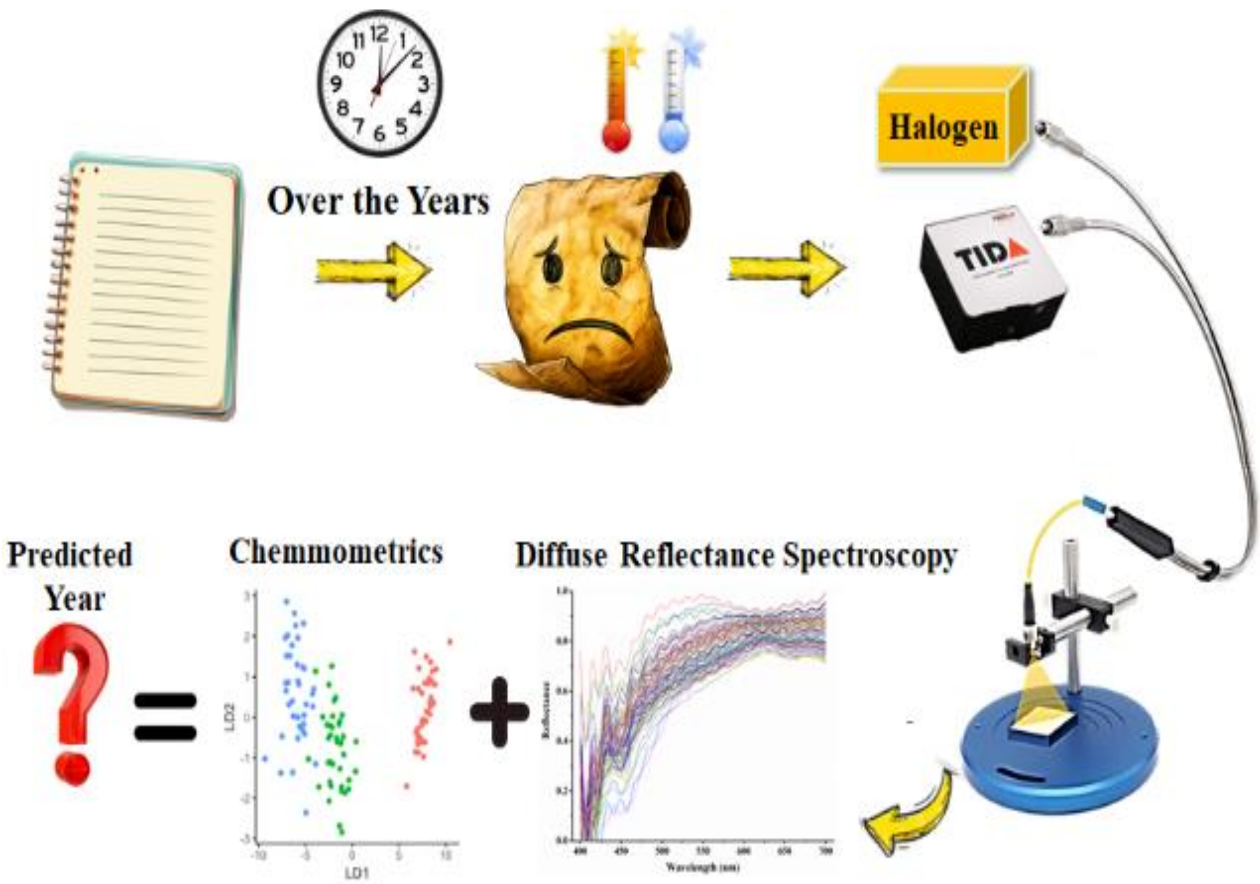

Figure 1. Schematic representation of the overall research workflow and the visible diffuse reflectance fiber-optic setup

2.2. Sample preparation and spectral acquisition

The paper samples investigated in this study covered a temporal range from 1356 to 1389 in the Solar Hijri calendar. The temporal interval between samples varied from one to seven years. All samples were maintained under identical storage conditions in a personal library collection. The identification of Pars paper was carried out based on its characteristic visual appearance, texture, and color, according to the experience and long-term familiarity of the archive owner with historical Iranian paper products. Accordingly, only books printed on Pars paper were included in this study. The approximate age of the paper samples was inferred from the publication year of each book. Based on field investigations regarding paper storage practices during the corresponding period, paper was generally stored for a relatively limited time before printing, typically less than one year. Therefore, the publication year was considered as an approximate indicator of paper age, although a certain degree of uncertainty associated with this estimation cannot be excluded. Diffuse reflectance spectra were recorded by positioning the samples at a fixed distance from the optical fiber probe. An integration time of 2 s was used for all measurements. Each recorded spectrum represented the average of three spectra acquired from three different locations on the paper surface for each production year in order to minimize local inhomogeneity effects. Polytetrafluoroethylene (PTFE) was used as the white reference standard for reflectance measurements.

2.3. Reflectance spectrum calculation

Reflectance data were calculated according to the following equation:

$$\mathrm{R} = I/I_0$$

where I represents the intensity of light reflected from the sample and I_0 denotes the intensity of light reflected from the reference standard [42]. Prior to spectral acquisition, dark current correction was applied to all measurements in order to eliminate noise contributions originating from the optical fiber, spectrometer, and ambient light, thereby ensuring reliable reflectance data.

2.4. Data preprocessing

Appropriate preprocessing of raw spectral data is essential for obtaining reliable statistical analysis, as it can significantly improve the predictive performance of chemometric models. Reflectance spectra were acquired within the visible spectral range of 400–750 nm. Subsequently, the spectral data were smoothed using a Fast Fourier Transform (FFT) filter to improve spectral visualization and reduce high-frequency noise. In the next step, spectral normalization was performed using the Z-score method in order to minimize intensity variations among samples. In this approach, each variable was transformed to have a mean value of zero and a variance of one [43, 44].

2.4.1. Principal component analysis

Principal Component Analysis (PCA) is an unsupervised multivariate analysis technique that provides data interpretation without prior assumptions regarding sample classification. The primary objective of PCA is dimensionality reduction of the original data space through the construction of a smaller and more efficient abstract space composed of latent variables, while preserving most of the information contained in the original dataset [45].

2.4.2. Linear discriminant analysis

Linear Discriminant Analysis is a supervised pattern recognition technique widely used for sample discrimination and class prediction. This method generates a linear mathematical function

capable of separating a set of samples into distinct groups.
Furthermore, the developed classification model can be employed to predict the class membership of unknown samples.
The analysis was implemented in Python using the Scikit-learn library [46, 47].
To evaluate the reproducibility and predictive performance of the model, Leave-One-Out Cross-Validation (LOOCV) was employed [48].
The performance of the LDA model was assessed using validation parameters including precision, recall, and F-score, calculated according to the following equations:
Recall, and F-score, calculated as follows:
Precision = (TP / (TP + FP)) × 100,
Recall = (TP / (TP + FN)) × 100,
F-score = 2 × (Precision × Recall) / (Precision + Recall), where TP denotes true positives, FP false positives, and FN false negatives [49].

3. Results and discussion

To provide a direct visualization of the acquired spectral data, representative diffuse reflectance spectra of paper samples from different production years are presented in Figure 2. As illustrated in Figure 2, aging influences the diffuse reflectance characteristics of the investigated paper samples. Variations in reflectance intensity can be observed among papers from different production years. However, the resulting spectral changes do not exhibit a simple linear relationship with paper age, suggesting that the aging process is governed by multiple physicochemical factors that evolve nonlinearly over time. Following the calculation of the diffuse reflectance spectra and the application of PCA to the spectral dataset (Range to 400-700nm), PCA alone was insufficient for reliable temporal discrimination of the production years of the paper samples.

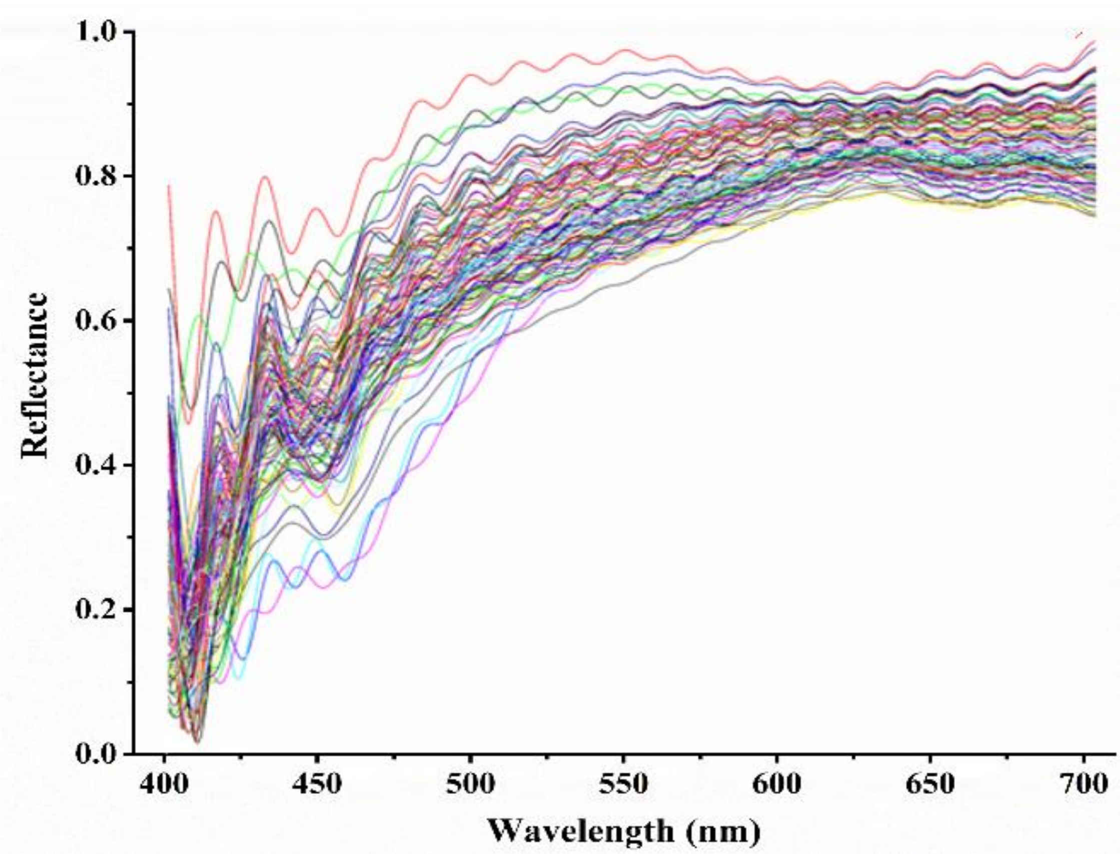

Figure 2. Diffuse reflectance spectra from samples of Different ages

In this model, the first principal component (PC1) accounted for 82.67% of the total data variance, while the PC2 and PC3 explained 11.96% and 2.72% of the variance, respectively. The cumulative explained variance reached 97.32% of the total dataset variance. Consequently, LDA was subsequently applied to achieve more accurate temporal discrimination of the samples. In the LDA model, the first discriminant component explained 78.42% of the variance, whereas the second and third components accounted for 14.36% and 3.09%, respectively, resulting in a cumulative variance of 95.87%.

The supervised nature of LDA significantly improved the discrimination of paper samples from different years despite the substantial overlap observed among their diffuse reflectance spectra. The transformed data distribution within the three-dimensional LDA space is illustrated in Figure 2. The developed model achieved a testing accuracy of 83.33% and a cross-validation accuracy of 79.96%. As shown in Figure 3, several classes exhibit overlap within the three-dimensional LDA space; therefore, additional viewing angles are required for a more comprehensive visualization of class separation. Since such visualization is limited in the static figure, a more detailed evaluation is provided in Table 1. In this table, the letter "C" at the beginning of the Class ID represents the abbreviation of the term "Class," while "Support" indicates the number of samples belonging to each group. Macro average computes the average for each class independently, without considering the number of samples in each class. % Bias Weighted average takes into account for the number of samples per class.

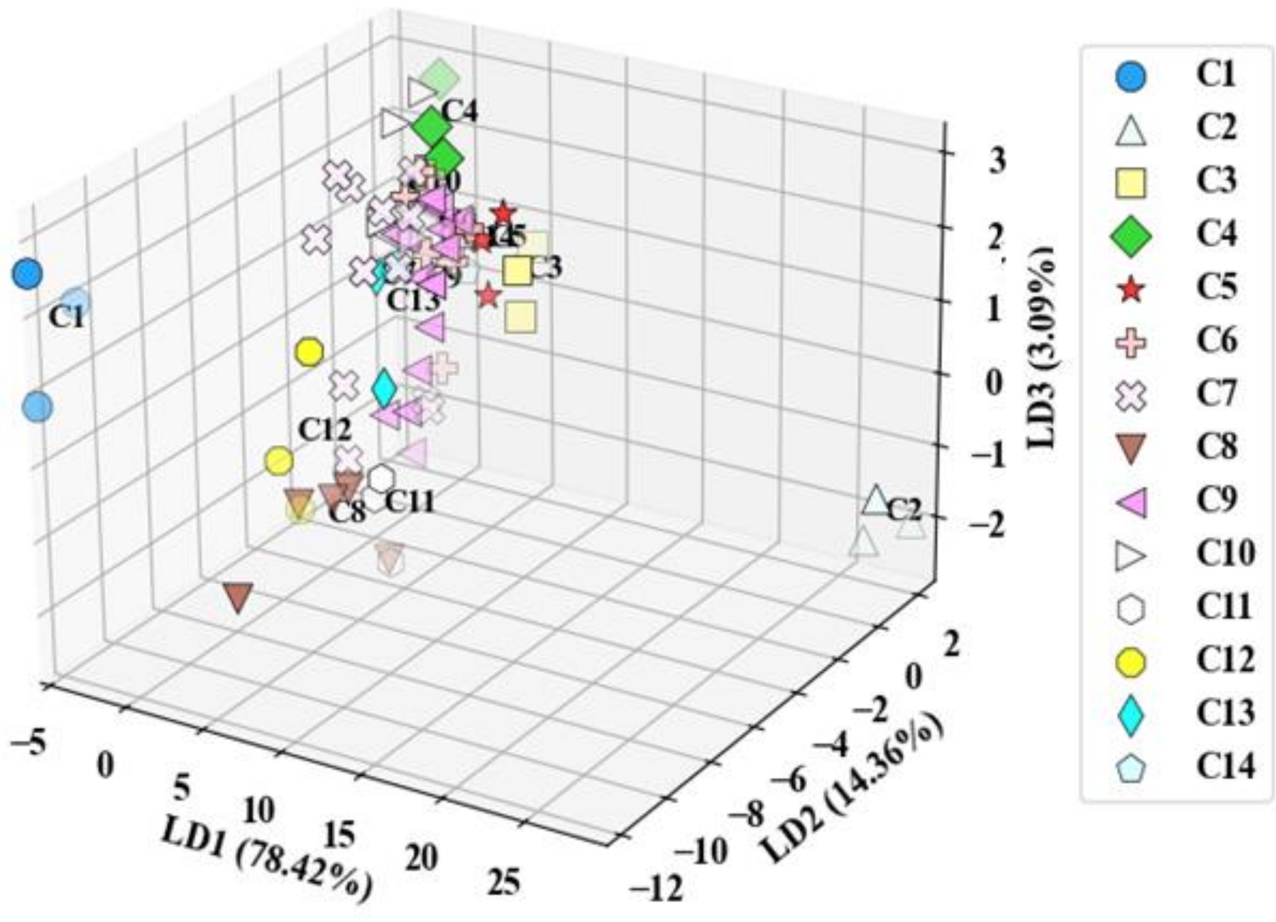

Figure 3. Discrimination and classification of the training dataset using LDA

Table 1. Performance metrics of the LDA classifier for various samples identification

Years	Class ID	Precision	Recall	F1-score	Support
1356	C1	1	1	1	3
1362	C2	1	1	1	3
1363	C3	1	1	1	3
1367	C4	0.75	1	0.86	3
1370	C5	0.83	0.83	0.83	6
1371	C6	0.78	0.78	0.78	9
1372	C7	0.78	0.58	0.67	12
1373	C8	1	0.83	0.91	6
1374	C9	0.86	0.80	0.83	15
1375	C10	1	0.83	0.91	6
1376	C11	0.60	1	0.75	3
1377	C12	0.75	1	0.86	3
1384	C13	0.75	1	0.86	3
1389	C14	0.75	1	0.86	3
Macro avg		0.85	0.90	0.86	78
Weighted avg		0.85	0.83	0.83	78

According to Table 1, classes with temporal intervals of one year or relatively close production dates exhibited lower F1-scores compared to classes separated by larger time intervals. This behavior is likely associated with the uncertainty considered for the publication year of each book included in this study.

In contrast, the comparatively improved discrimination observed for classes with greater temporal differences further supports the rationale behind previous studies, in which samples with minimum temporal intervals of approximately four years were selected.

3.1. Blind test

The samples used in the blind test consisted of papers that were not included in the training stage of the model.

These samples comprised four different books belonging to four distinct temporal intervals. Three samples were collected from each book, resulting in a total of 12 samples used for blind testing. The results obtained from the blind test are presented in Table 2. Unknown samples belonging to classes C10 and C6, corresponding to the years 1371 and 1375, respectively, achieved a correct prediction rate of 66.7%. In contrast, unknown samples associated with classes C7 and C9, representing the years 1372 and 1374, showed a lower prediction accuracy of 33.3%. Overall, the total correct prediction rate obtained for all unknown samples in the blind test was 50%. These findings indicate that, despite the presence of samples with minimal temporal intervals, the developed model was able to correctly predict the age of half of the unknown samples.

Table 2. Results of blind-test prediction for unknown paper samples

Class ID	Unknown Sample	Predicted ID	%Correct
C6	1	C9	66.7
	2	C6	
	3	C6	
C7	1	C11	33.3
	2	C10	
	3	C7	
C9	1	C10	33.3
	2	C10	
	3	C9	
C10	1	C7	66.7
	2	C10	
	3	C10	

Although the overall blind-test accuracy was limited to 50%, the prediction task involved naturally aged paper samples with temporal differences as small as one year, making the classification problem substantially more challenging compared to previously reported studies employing larger temporal intervals.

The relatively lower prediction accuracy observed in the blind test can be attributed to the minimal temporal differences between samples, the natural aging process of the papers, and uncertainties associated with the exact production dates. Nevertheless, the obtained results demonstrate that DRS combined with chemometric analysis possesses promising potential for forensic paper dating applications involving naturally aged samples.

3.2. Study limitations

Despite the promising results obtained in the previous sections, several limitations should be considered.

The first limitation concerns the restricted availability of naturally aged paper samples belonging to a single commercial brand. As a consequence, the number of samples included in this study remained relatively limited for chemometric analysis.

Another important limitation was the lack of samples with precisely known aging histories.

This uncertainty likely contributed to the overlap observed among some of the investigated classes.

Furthermore, it should be noted that the developed model is only capable of identifying samples similar to those previously encountered during the training stage, since the proposed approach is based on supervised learning.

Nevertheless, despite the limited sample size, the developed model demonstrated promising predictive capability under challenging conditions involving minimal temporal intervals, but still requires complementary methods.

The results obtained are only valid for the type of paper used in this study and cannot be directly generalized to other types of paper.

4. Conclusion

paper age estimation and the discrimination of production years using chemometric approaches involve several analytical challenges. In the present study, paper samples separated by one-year and multi-year temporal intervals were investigated using Diffuse Reflectance Spectroscopy combined with PCA–LDA. The developed model achieved a cross-validation accuracy of 79.96% for the classification of the training samples.

Furthermore, during blind testing, the model successfully predicted the age of 50% of the unknown samples. Considering the existence of samples separated by only one year, this result can be regarded as significant. Studies employing accessible and cost-effective analytical techniques for paper age estimation may represent an important step toward the development of practical and non-destructive approaches for document dating applications. Future studies should focus on increasing the number of naturally aged samples, incorporating papers from different manufacturers, and applying advanced machine learning algorithms to improve prediction accuracy.

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