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## Chemical Profiling and Chemometric Classification of Magic Pen (Disappearing) Ink Samples Using ATR-FTIR Spectroscopy

التنميط الكيميائي والتصنيف القياسي الكيميائي لعينات حبر الأقلام السحرية (المختفية) باستخدام

مطيافية الأشعة تحت الحمراء بتحويل فورييه (ATR-FTIR)



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### Abstract

Ink has always been an important aspect of document examination, which while may appear optically similar to the naked eye, but can vary significantly in terms of their chemical constituents. Majority of studies has been focussed on distinguishing these visibly similar inks. However, disappearing inks, also known as magic inks presents a unique set of challenge to document security given its ability to fade entirely within approximately seconds to days, thus enabling undetectable forgery. The current study attempted a non-destructive chemical profiling and classification of blue disappearing inks available in Indian markets using Attenuated Total Reflectance – Fourier Transform Infrared (ATR-FTIR) spectroscopy in conjunction with chemometric methods

In the present study, a total of ninety different blue magic pen (disappearing) inks, representing the 30 different brands were purchased from e-shopping websites and local markets of Jhansi district of Uttar Pradesh in India. The samples were prepared by drawing circles of 2 cm radius and filling it completely with ink in five subsequent strokes on 80 gsm white A4 copying paper for each pen which was then spectroscopically analyzed by ATR-FTIR within the range of 4000- 600 cm<sup>-1</sup>. The spectral data was further subjected to chemometric analysis viz. HCA and PC-DA.

FTIR spectroscopy of magic pen inks demonstrated significant similarities in spectra with characteristic peaks

**Keywords:** forensic sciences, magic pen, FTIR, PCA, DA, ink analysis, questioned document examination

### المستخلص

يُعد الحبر جانباً حيويًا في فحص المستندات؛ فبينما قد تظهر الأحبار متشابهة بصرياً للعين المجردة، إلا أنها قد تختلف اختلافاً جذرياً في مكوناتها الكيميائية. وركزت معظم الدراسات السابقة على التمييز بين هذه الأحبار المتشابهة ظاهرياً، ومع ذلك، فإن الأحبار المختفية (المعروفة بالأحبار السحرية) تمثل تحدياً فريداً لأمن المستندات؛ نظراً لقدرتها على التلاشي تماماً خلال ثوانٍ أو أيام، مما يتيح إجراء عمليات تزوير لا يمكن اكتشافها. حاولت الدراسة الحالية إجراء تنميط كيميائي وتصنيف (غير متلف للعينات) لأحبار سحرية زرقاء متوفرة في الأسواق الهندية باستخدام مطيافية الأشعة تحت الحمراء بتحويل فورييه وانعكاس كلي موهن (ATR-FTIR) بالاقتران مع طرق القياس الكيميائي (Chemometrics).

وفي هذه الدراسة، تم شراء تسعين حبراً مختلفاً من أقلام الحبر السحرية الزرقاء، تمثل 30 علامة تجارية مختلفة من مواقع التسوق الإلكتروني والأسواق المحلية في منطقة «جهانسي» بالهند. وتم إعداد العينات برسم دوائر بقطر 2 سم وتعبئتها بالكامل بالحبر على ورق تصوير أبيض (80 جراماً)، ثم جرى تحليلها طيفياً بواسطة جهاز (ATR-FTIR) ضمن نطاق 4000-600 سم<sup>-1</sup>. بعد ذلك، خضعت البيانات الطيفية للتحليل القياسي الكيميائي باستخدام التحليل

**الكلمات المفتاحية:** علوم الأدلة الجنائية، القلم السحري، الأشعة تحت الحمراء (FTIR)، تحليل المكونات الرئيسية (PCA)، تحليل التمايز (DA)، تحليل الحبر، فحص المستندات الجنائية

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for O-H (alcohol/water), C-H (aliphatic), C=C (aromatic) and C-O stretching peaks indicating the presence of thymolphthalein as fundamental constituent to most of the blue disappearing ink formulations available in the markets. Chemometric analysis using Hierarchical Cluster Analysis (HCA) revealed four major clusters within the samples. PC based Discriminant Analysis (DA) model was then used to assess the classification of samples in these respective groups which performed reasonably well achieving an accuracy of 100% in classification and cross-validation. Also, PC based DA model for classification of samples according to manufacturer successfully, linked samples to their respective manufacturer with an original and cross-validated accuracy of 97.8% and 82.2% respectively.

## 1. Introduction

The magic pens characterized by disappearing inks are gaining popularity these days although it is not as commonly used as a writing instrument with standard inks. Still, these are commonly available at stationary shops, making them an ideal gizmo for commoners as well as fraudsters to commit deception with malicious or non-malicious intention because of its magical writing affect. Magic pen ink is available in the market under trade names like disappearing ink, vanishing ink and invisible ink [1, 2]. Disappearing inks are not a recent addition, as they have been used for secret writing since ancient times. They have been weapons of choice for offenders in a variety of document and financial fraud as well as among spies and war criminals to convey messages [3]. Recent trends suggest that the forgers motivated by illicit financial gain often use these magic pens with disappearing inks to reach their fraudulent goal aimed at producing flawless forgeries. Banks remain a primary target for these fraudulent activities where majority of banking and verification services are still offline [4, 5]. While earlier fraudsters used different types of erasures, mechanical items, chemical solvents for tampering the cheques, wills, deeds and other documents, the widespread availability of these inks have made

العنقودي الهرمي (HCA) وتحليل التمايز القائم على المكونات الرئيسية (PC-DA).

أظهر التحليل الطيفي للأحبار تشابهاً كبيراً في الأطياف مع وجود قمم مميزة لمجموعات الهيدروكسيل (O-H)، والكربون الهيدروجيني الأليفاتي (C-H)، والكربون العطري (C=C)، وقيم تمدد الكربون والأكسجين (C-O)، مما يشير إلى وجود مادة «الثيمولفثالين» (Thymolphthalein) كمكون أساسي لمعظم تركيبات الأحبار الزرقاء المختفية. وكشف التحليل العنقودي الهرمي (HCA) عن أربع مجموعات رئيسية بين العينات. واستُخدم نموذج تحليل التمايز (DA) لتقييم تصنيف العينات في هذه المجموعات، حيث حقق أداءً ممتازاً بدقة بلغت 100% في التصنيف والتحقق المتبادل. كما نجح النموذج في ربط العينات بمصنعيها الأصليين بدقة تصنيف بلغت 97.8% ودقة تحقق متبادل بلغت 82.2%.

their task easy [6, 7, 8]. Eventually the complex composition of disappearing ink based on acid base chemistry make it perfect for such economical and white-collar crimes [9]. Disappeared writing are transparent in the visible spectrum and therefore invisible to unaided human eyes. Disappearing ink are composed of mixtures of chemicals that shows certain response to UV and IR lights [9, 11]. Although these are used as marking ink designed to be visible for a short time, with no intention of being revealed again, they can be visualized in some parts of electromagnetic spectrum [12]. Thymolphthalein and phenolphthalein are the major constituent of contemporary disappearing inks, which primarily functions as an indicator, transitioning from colourless to blue and pink colour in an alkaline solution respectively. This blue and pink coloration is transient because the base is progressively neutralized by atmospheric carbon dioxide, leading to a decrease in the solution's pH, at which point the indicator reverts to its colourless state [13]. The kinetics of this fading reaction are dependent on the initial concentrations of both the indicator and the base present in the ink composition. Consequently, the duration of the visible writing can be varied from a few seconds to several months [14]. Ink is a complex system composed of dyes, pigments,



resins, solvents, indicators, pH controllers the analysis of which require both destructive and non-destructive analysis. Destructive methods usually require a portion of the ink to be removed from the document using appropriate solvents. In contrast, non-destructive methods for forensic discrimination of pen inks preserve the integrity of a questioned document which can be further examined using other methods [15]. With increasing use of magic pen ink in committing forgeries, it has become important to chemically analyze, characterize, classify and discriminate these disappearing (magic) inks available in market of a developing country like India where majority of banking transactions are still offline [16]. While there are numerous studies integrating ink analysis with chemometrics for decipherment, discrimination and classification of inks, a vast majority of them are limited to normal writing inks rather than disappearing ones. The studies on disappearing inks have been largely limited to their visibility using physical, chemical examination and instrumental techniques such as video spectral comparators (VSC), none of them has focussed on their chemical classification and discrimination using other analytical techniques such as Fourier Transform Infrared (FTIR) spectroscopy [17, 18, 19, 20, 21]. FTIR spectroscopy is a non-invasive, low cost, easy to use analytical technique which can not only provide the preliminary information about the chemicals present in these inks but also aid in their classification and discrimination. The present study is first of its kind in assessing its practical utility in forensic chemical classification of disappearing inks.

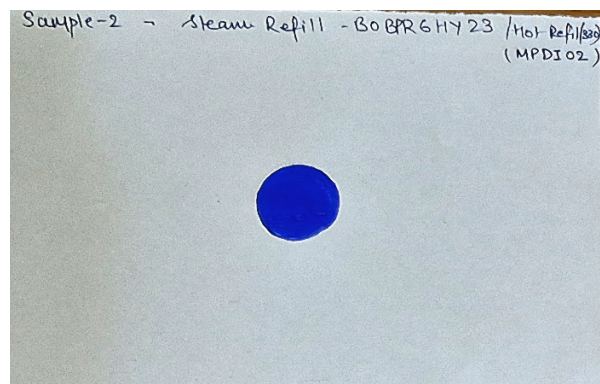
The current study aims to identify the chemical groups present in the disappearing (magic) ink samples using ATR-FTIR spectroscopic analysis. The study further attempts a chemometric classification of these samples based on IR spectra using Principal Component Analysis (PCA) and

Principal Components based Discriminant Analysis (PC-DA). Additionally, spectral information was also used to classify disappearing ink according to their manufacturer/brands using PC-DA.

## 2. Materials and Methods

### 2.1 Sample Collection and Analysis

Common brand available both at stationary shops in Jhansi district of Uttar Pradesh in India and e-commerce websites (Amazon, Flipkart, Snapdeal, Ebay) were exhaustively searched for magic pen inks. Magic pen inks of blue colour 30 different brands, available commercially were procured for the study, details of which are available in Table 1. The study was deliberately limited to blue ink because of its widespread utilization for signing documents of legal significance such as cheques, wills, affidavits etc. The samples were prepared by drawing circles of 2 cm radius and filling it completely with ink in five subsequent strokes on 80 gsm white A4 copying paper for each sample as depicted in Figure 1 [22]. The prepared samples were allowed to air dry at ambient temperature for approximately one hour, until the deposited ink had completely faded from visible observation, prior to FTIR spectral acquisition. Three replicates were prepared for each brand but with different ink cartridge making the total sample size of 90. ATR-FTIR spectra were



**Figure 1-** Prepared ink sample before drying.



**Table 1-** Description of ink samples used for chemical profiling

S.no.	Manufacturer of Magic Pen	Sample ID	Colour of Ink
1.	WBD Magic Disappearing ink	MPDI 1	Blue
2.	Steam Refill Magic Ink	MPDI 2	Blue
3.	Kiyaan Magic Ink Pen	MPDI 3	Blue
4.	Hunny Bunch Magic Ink Pen	MPDI 4	Blue
5.	Generic Invisible Ink	MPDI 5	Blue
6.	Invisible Refill Disappearing Ink	MPDI 6	Blue
7.	Jasol Heat Erasable Ink Pen	MPDI 7	Blue
8.	Jainsons Invisible Ink Pen	MPDI 8	Blue
9.	Zoqweid Magic Pen Ink	MPDI 9	Blue
10.	Zarquito Magic Pen Ink	MPDI 10	Blue
11.	Pakhi Magic Pen Ink	MPDI 11	Blue
12.	Paramza Magic Pen Ink	MPDI 12	Blue
13.	Crazy Buy Magic pen	MPDI 13	Blue
14.	Akshar New Brand Magic Pen	MPDI 14	Blue
15.	Alornor Magic Pen	MPDI 15	Blue
16.	Zodler Magic pen	MPDI 16	Blue
17.	Fashion Crazz Magic Ink Pen	MPDI 17	Blue
18.	Sank Magic Pen Ink	MPDI 18	Blue
19.	Siskey Magic Pen Ink	MPDI 19	Blue
20.	Sabee Magic Pen Ink	MPDI 20	Blue
21.	Marcrazy Magic Pen Ink	MPDI 21	Blue
22.	Mystify Magic Disappearing Ink	MPDI 22	Blue
23.	X-Force Plaza Magic Pen Ink	MPDI 23	Blue
24.	Pssopp Fabric Disappearing Ink	MPDI 24	Blue
25.	Rulex Magic Pen Ink	MPDI 25	Blue
26.	Aakriti Invisible Magic Pen Ink	MPDI 26	Blue
27.	Refulgix Magic Pen Ink	MPDI 27	Blue
28.	DD Enterprises Magic Pen	MPDI 28	Blue
29.	Almart Magic Pen Ink	MPDI 29	Blue
30.	Millar Magic Pen	MPDI 30	Blue



acquired directly from ink deposits on paper as ATR facilitates preferential sampling of surface-bound ink layer due to its inherent shallow penetration depth.

## 2.2 Equipment and Spectra Acquisition

The current study utilized ATR-FTIR spectrometer manufactured by Perkin Elmer, Llantrisant (UK) with a MIR (Mid-InfraRed) TGS (Triglycine Sulfate) detector to record the IR spectra of the samples. Thirty-two co-scans were collected at a resolution of  $4\text{ cm}^{-1}$  in the range of  $4000 - 600\text{ cm}^{-1}$  of the mid-IR region for each sample. All samples were mounted in the sample holder and no additional sample preparation was done. Prior to the recording of IR spectra of each sample, a background scan was run on A4 sheet used in sample preparation. Each sample was subjected to data pre-processing using Origin 2026 (Learning Edition).

## 2.3 Data Pre-processing

The entire spectra in the range of  $4000-600\text{ cm}^{-1}$  for all the samples were pre-processed with the same workflow, initiating with baseline offset and linear baseline correction, followed by spectra smoothing and range normalization. Savitzky Golay was used to smoothen the spectra with quadratic polynomial order and 3 smoothing points in symmetric kernel.

## 2.4 Chemometric Analysis

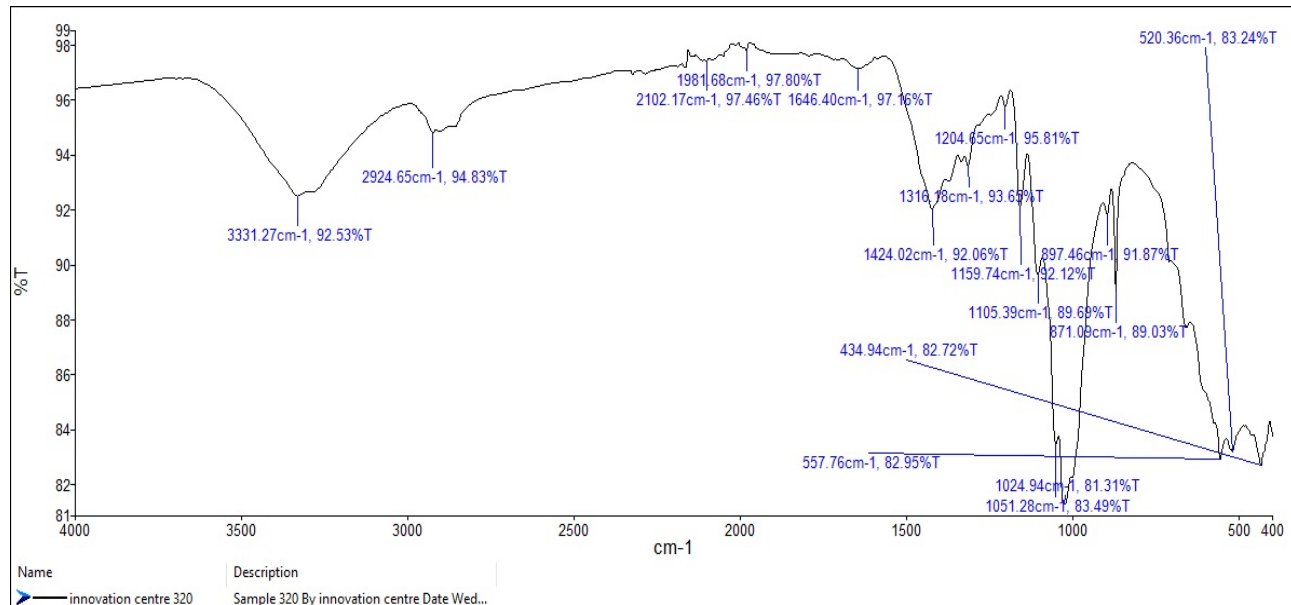
The use of chemometric methodologies facilitates an impartial interpretation of multivariate datasets when manual observation is not sufficient to identify intricate patterns within the spectral data. The present study employed Principal Components Analysis (PCA) for dimensionality reduction. For initial classification of samples in the current study, PCs were subjected to cluster analysis by hierarchical complete cluster linkage method with squared

euclidian distance. These Principal Components (PCs) were subsequently employed for supervised classification based on clusters identified, using Discriminant Analysis. Further, brand classification was also performed to link spectra of specific sample to their manufacturer. All the statistical analyses were performed using IBM SPSS Statistics 27.0.1.

### 2.4.1 Principal Component Analysis (PCA)

PCA is an unsupervised multivariate statistical technique for dimensionality reduction and pattern recognition, initially has been proposed by Karl Pearson in 1901 [23] and was developed in its current form by Harold Hotelling in 1933 [24]. This method projects high-dimensional data points that are characterised by  $n$  variables, represent a point in an  $n$ -dimensional space, in two or three lower dimensions, while retaining much of the information about their variance without changing the data structure. The core objective of this process is that the variables describing the samples are transformed into new variables, called Principal Components (PCs) that are linear combinations of the original variables. The principal components are at right angles to each other, a property known as orthogonality, the variables are measured be 'similar' or at least 'not very dissimilar'. These new PCs are derived from the eigenvectors and eigenvalues of the data's covariance matrix. Covariance is a measure of the joint variance of two variables. Eigenvectors represent the direction of largest variation whereas, eigenvalues are measure of variance by each eigenvector in the dataset. In the present study, pre-processed spectra were mean-centered and subjected to PCA using Singular Value Decomposition (SVD) algorithm with full cross-validation [25, 26, 27, 28, 29, 30, 31].





**Figure 2-** Representative spectra of disappearing (magic pen) Ink (sample MPDI 1).

#### 2.4.2 Discriminant Analysis (DA)

DA functions as a powerful supervised technique for both dimensionality reduction and classification tasks. Mathematically it is the ratio of the between-classes variance to the within-classes variance. Although LDA appears to multivariate analysis of all the groups simultaneously, the method is actually equivalent to analysing the groups pairwise [32, 33, 34, 35]. The discriminant efficacy of an DA model can be evaluated using metrics such as the F-ratio and Wilk's Lambda. Theoretically, higher F-ratio and lower Wilk's Lambda indicate greater discrimination [36]. The current study used PC-based discriminant analysis with leave one out cross-validation (LOOCV) for assessing classification of disappearing inks of magic pens. All the statistical parameters were computed at 95% confidence interval.

### 3. Results

The key peaks and corresponding functional groups associated with the 30 samples of magic pen (disappearing) inks (MPDI 1 – MPDI 30) is enlisted in Table 2. All the samples showed

significant similarities in spectra with characteristic peaks for O-H (alcohol/water), C-H (aliphatic), C=C (aromatic) and C-O stretching peaks indicating the presence of thymolphthalein as fundamental to most disappearing ink formulations available in Indian markets [37, 38]. A representative spectrum of sample (MPDI 1) is shown in Figure 2.

All thirty samples showed a broad peak in 3400-3200  $\text{cm}^{-1}$ , indicating the presence of hydroxyl group possibly originating from water and ethanol which may have been used as primary solvent and co-solvent respectively given the nature of the ink. A consistent sharp peak in 2950-2850  $\text{cm}^{-1}$  region for all the samples correspond to C-H stretching vibrations typical of aliphatic groups which may be attributed to methyl and methylene groups in ethanol and thymolphthalein. Samples MPDI 4, MPDI 9, MPDI 11-12, MPDI 14, MPDI 17-30 showed the presence of carbonyl peak (C=O stretching) in the range 1799-1700  $\text{cm}^{-1}$  likely arising due to ester or ketone groups in thymolphthalein or other additives like plasticizers. All the samples showed peaks in the range 1600-1500  $\text{cm}^{-1}$ , indicating C=C



**Table 2-** Peaks corresponding to functional groups in magic pen inks sample

Sample ID	O-H Stretch (cm <sup>-1</sup> )	C-H Aliphatic Stretch (cm <sup>-1</sup> )	CO <sub>2</sub> Absorption (cm <sup>-1</sup> )	C=O Stretch (cm <sup>-1</sup> )	C=C Aromatic Stretch (cm <sup>-1</sup> )	C-O Stretch (cm <sup>-1</sup> )	C-H Bending (cm <sup>-1</sup> )	Aromatic C-H OOP (cm <sup>-1</sup> )
MPDI 1	3330 - 3300	2924.65, 2894.03	2324.81	-	1646.40	1204.45, 1051.28, 1024.94	1424.02, 1371.40	897.46, 871.08, 659.64
MPDI 2	3330 - 3300	2924.81, 2894.03	2324.81	-	1646.40	1204.78, 1049.41, 1026.13	1424.21, 1371.40	898.74, 871.08, 659.64
MPDI 3	3330 - 3300	2888.67	2324.83	-	1646.76	1204.33, 1049.00, 1025.80	1424.27, 1370.74	898.43, 871.10, 659.41
MPDI 4	3330 - 3300	2934.05, 2889.42	-	1793.76	1664.06	1204.52, 1049.93, 1025.34	1423.70, 1371.17	898.16, 871.13
MPDI 5	3330 - 3300	2889.10	-	-	1664.06	1208.83, 1025.00	1423.22	897.96, 871.18
MPDI 6	3330 - 3300	2937.49, 2881.23	-	-	1590.50	1204.62, 1051.81, 1032.31	1423.85	896.97, 871.31
MPDI 7	3330 - 3300	2893.06	-	-	1637.93	1204.54, 1025.75	1424.02	897.55, 871.25
MPDI 8	3330 - 3300	2891.29	-	-	1646.31	1204.42, 1051.00, 1028.39	1425.14	898.22, 871.17
MPDI 9	3330 - 3300	2886.59	-	-	1646.31	1204.43, 1026.65	1423.96	897.59, 871.05
MPDI 10	3330 - 3300	2884.94	-	-	1591.84	1204.55, 1049.20, 1028.51	1424.82	898.67, 871.14
MPDI 11	3330 - 3300	2882.09, 2832.96	-	-	1654.87	1204.13, 1024.22	1424.41	898.67, 871.09
MPDI 12	3330 - 3300	2882.12, 2836.18	-	-	1647.23	1203.99, 1051.67, 1027.90	1424.29	897.78, 871.24
MPDI 13	3330 - 3300	2891.38	-	-	-	1204.01, 1026.76	1424.59	897.00, 871.31
MPDI 14	3330 - 3300	2887.79	-	1736.27	1647.01	1204.38, 1027.27	1425.47, 1370.90	898.67, 871.48
MPDI 15	3330 - 3300	2889.12	-	-	1590.93	1204.33, 1027.30	1421.00	897.22, 871.28
MPDI 16	3330 - 3300	2916.84	-	-	1645.78	1204.71, 1026.90	1423.93	897.71, 871.32



Table 2- continues

Sample ID	O-H Stretch (cm <sup>-1</sup> )	C-H Aliphatic Stretch (cm <sup>-1</sup> )	CO <sub>2</sub> Absorption (cm <sup>-1</sup> )	C=O Stretch (cm <sup>-1</sup> )	C=C Aromatic Stretch (cm <sup>-1</sup> )	C-O Stretch (cm <sup>-1</sup> )	C-H Bending (cm <sup>-1</sup> )	Aromatic C-H OOP (cm <sup>-1</sup> )
MPDI 17	3330 - 3300	3026.66, 2916.63, 2850.50	-	1729.64	1642.98	1213.5, 1042.62, 1028.59	1493.00, 1452.99, 1389.31	814.50, 755.67, 697.36
MPDI 18	3330 - 3300	2889.53	-	1736.29	1638.70	1204.85, 1050.62, 1028.77	1426.79, 1370.78	896.25, 871.50, 658.45
MPDI 19	3330 - 3300	2889.37	-	1735.64	1641.44	1204.65, 1050.91, 1028.56	1426.79, 1370.40	897.86, 871.59, 658.52
MPDI 20	3330 - 3300	2925.49, 2855.0	-	1747.69	1646.63	1029.19	1425.23	871.25
MPDI 21	3330 - 3300	2925.49, 2855.0	-	1747.69	1646.63	1028.97	1425.23	871.25
MPDI 22	3330 - 3300	2924.17	-	1747.58	1637.64	1204.14, 1051.54, 1028.53	1425.66, 1370.46	897.85, 871.24, 658.90
MPDI 23	3330 - 3300	2922.99	-	1751.15	1637.37	1204.75, 1050.08, 1026.23	1421.19	897.59, 871.15, 658.61
MPDI 24	3330 - 3300	2923.84	-	1751.17	1638.04	1205.81, 1027.00	1421.03, 1371.00	899.10, 870.67, 656.03
MPDI 25	3330 - 3300	2920.88	2325.91	1747.41	1642.23	1204.90, 1050.03, 1028.05	1425.05, 1370.84	898.17, 871.16, 658.27
MPDI 26	3330 - 3300	2892.83	2317.60	1752.05	1645.53	1205.17, 1027.21	1423.85, 1371.22	899.06, 870.92, 659.20
MPDI 27	3330 - 3300	2891.58	2317.46	1734.65	1643.97	1205.11, 1027.11	1424.27, 1370.77	896.82, 870.86, 658.96
MPDI 28	3330 - 3300	2924.19	2317.42	1736.03	1643.64	1205.61, 1029.99	1426.11, 1370.40	899.40, 871.16, 656.09
MPDI 29	3330 - 3300	2924.01	2317.53	1738.31	1641.73	1206.32, 1027.75	1419.89	898.22, 871.24, 658.81
MPDI 30	3330 - 3300	2890.79	2317.62	1735.06	1644.18	1205.44, 1047.95, 1029.98	1426.74, 1370.64	898.64, 871.13, 656.18



aromatic stretching consistent with aromatic rings in the thymolphthalein. The spectra of samples further showed presence of peaks in 1250-1200  $\text{cm}^{-1}$  and 1100-1000  $\text{cm}^{-1}$  due to C-O stretching from ester linkages (1250-1200  $\text{cm}^{-1}$ ) and alcohol groups (1100-1000  $\text{cm}^{-1}$ ). The absence of this peak in sample MPDI 20-21, possibly indicates reduction of ester additives. The presence of peaks in 900-600  $\text{cm}^{-1}$  in all the samples indicates out-of-plane aromatic C-H bond bending, further indicating the presence of phthalein derivative constituents in all the inks. However, IR spectral interpretation about ink formulation in disappearing ink is indicative only and require further confirmation using techniques such as GC-MS and HPLC.

The comparative analysis of ink formulations showed a great degree of spectral homogeneity with common characteristic bands and comparable intensity indicating a common underlying chemical formulation making it difficult to classify samples manually. To accomplish this spectral information was subjected to dimensionality reduction followed by unsupervised classification and supervised classification. Dimensionality reduction was performed through principal components analysis (PCA). Principal component analysis is a dimension reduction technique, which reduces the invariably large number of variables in a dataset while retaining their possible variability. The eigenvalues and variance explained by the principal components for the spectra are shown in Table 3. A higher eigenvalue indicates greater amount of variance

explained. The aggregate of variance explained by ten PCs was found to be approximately 99% (as depicted in Figure 3) with first PC explaining 79% variance and eigenvalue greater than 1.

The score plot of PC 1 v. PC 2 revealed some samples forming distinct cluster in the graph as shown in figure 4.

Analysis of Principal Components (PCs) further revealed the influential wavenumbers contributing to each PC. PC1 exhibited high loadings at 1171, 1254, and 1213  $\text{cm}^{-1}$ , corresponding to C–O stretching vibrations, along with a band at 1730  $\text{cm}^{-1}$  assigned to C=O stretching. A prominent contribution at 1515  $\text{cm}^{-1}$  was attributed to aromatic C=C stretching. Additional features from the fingerprint region (738, 831, and 814  $\text{cm}^{-1}$ ), together with a broad O–H stretching band at 3275  $\text{cm}^{-1}$ , indicate that this component predominantly reflects variations in solvent composition (alcohol/ether content), dye structure, and overall formulation complexity. PC2 showed strong contributions from 1554  $\text{cm}^{-1}$  associated with aromatic (C=C stretching) variations, suggesting variability in the chromophoric structure of the dye, while PC3 showed strong loadings at 1002  $\text{cm}^{-1}$  and 1057  $\text{cm}^{-1}$ , indicative of differences in alcohol/ether components and formulation additives. PC4 to PC7 captured more subtle chemical variations, particularly within the carbonyl and fingerprint regions. PC4 exhibited a complex profile with strong loading at 1143  $\text{cm}^{-1}$  and 1754  $\text{cm}^{-1}$ , contrasted by negative loadings at 1730  $\text{cm}^{-1}$  and 733  $\text{cm}^{-1}$ . PC5 reflected notable variability in both the aliphatic

**Table 3-** Total variance explained for the magic pen inks

Components	PC 1	PC 2	PC 3	PC 4	PC 5	PC 6	PC 7	PC 8	PC 9	PC 10
Eigenvalue	6.65	0.61	0.52	0.16	0.11	0.10	0.07	0.04	0.04	0.02
Proportion	0.79	0.07	0.06	0.02	0.01	0.01	0.01	0.01	0.005	0.005
Cumulative	0.79	0.86	0.92	0.94	0.95	0.96	0.97	0.98	0.985	0.99



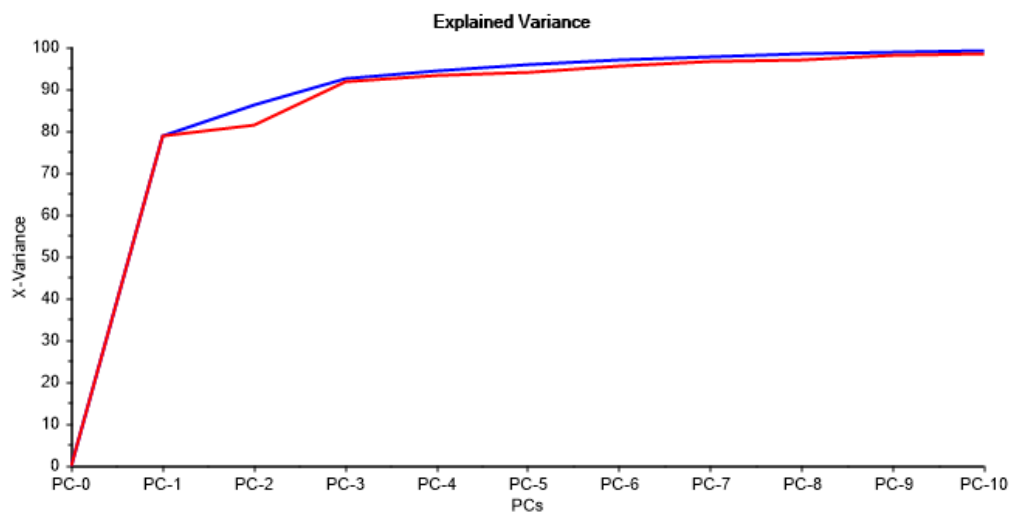


Figure 3- Scree plot

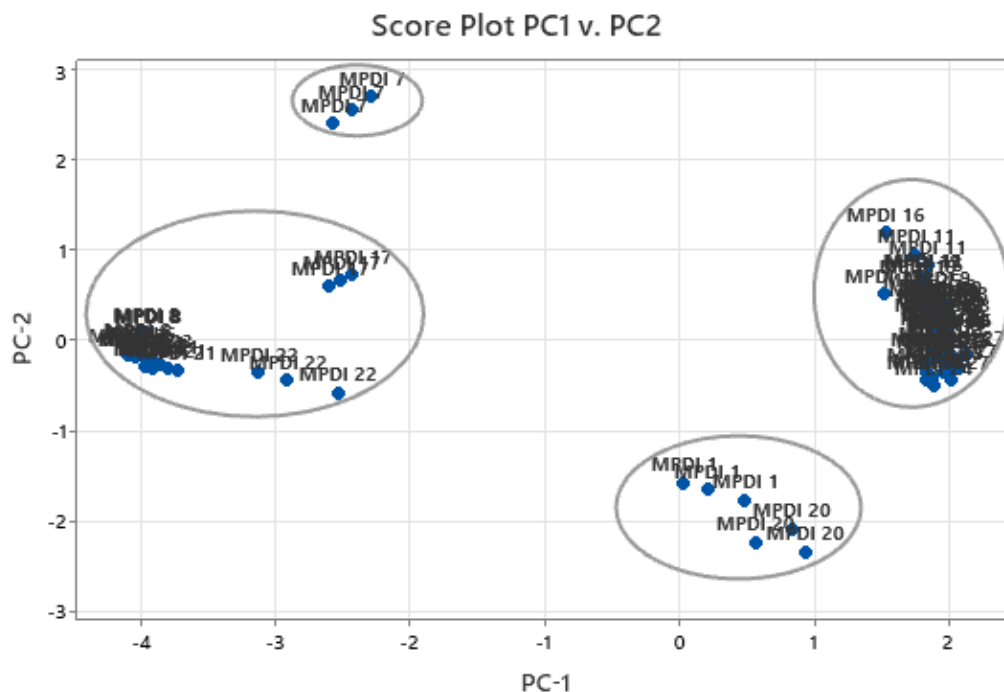


Figure 4- Score plot between PC1 and PC2 showing sample clusters.

C–H stretching and fingerprint regions, indicating differences in hydrocarbon solvent composition and formulation complexity. PC6 demonstrated a pronounced negative loading at  $1731\text{ cm}^{-1}$  (C=O stretching) and a prominent positive contribution at  $1142\text{ cm}^{-1}$  (C–O stretching), suggesting opposing contributions from carbonyl-containing compounds

and solvent-related components. PC7 was characterised by strong negative loadings at  $1418\text{ cm}^{-1}$  (C–H bending/aromatic ring vibrations) and  $697\text{ cm}^{-1}$  (fingerprint region), representing subtle structural and compositional variations. PC8 to PC10 accounted for comparatively minor spectral variations, primarily associated with aliphatic regions,



**Table 4-** Groups based on HCA analysis

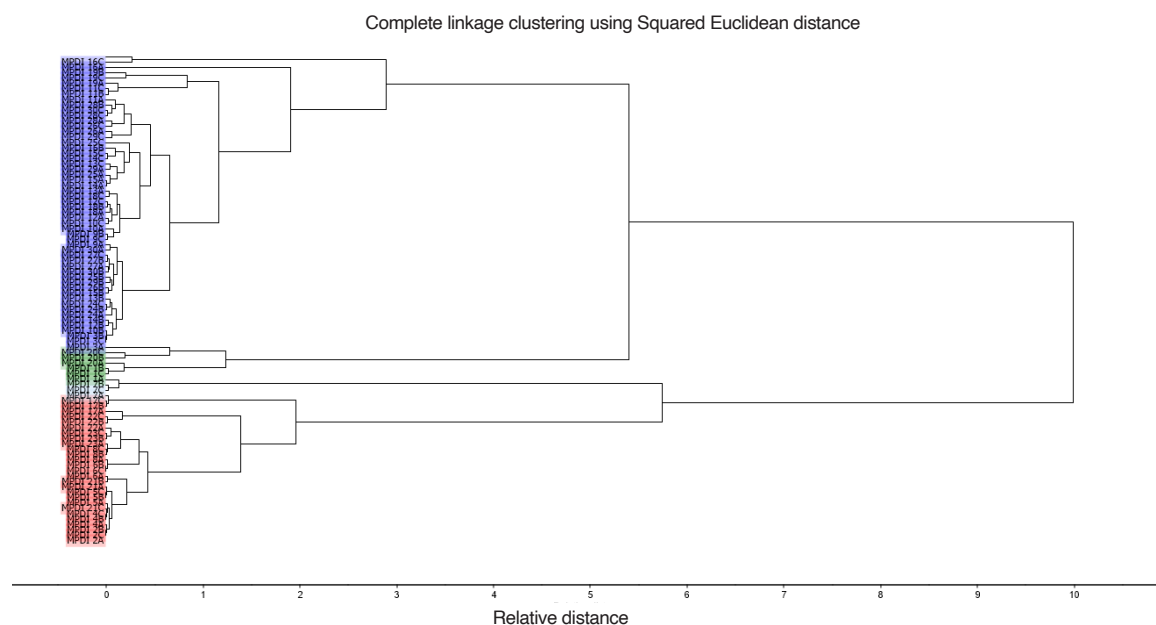
S. No.	Group	Samples	Total
	Group A (Figure 6)	MPDI 3, MPDI 9, MPDI 10, MPDI 11, MPDI 12, MPDI 13, MPDI 14, MPDI 15, MPDI 16, MPDI 18, MPDI 19, MPDI 24, MPDI 25, MPDI 26, MPDI 27, MPDI 28, MPDI 29, MPDI 30	18
	Group B (Figure 7)	MPDI 2, MPDI 4, MPDI 5, MPDI 6, MPDI 8, MPDI 17, MPDI 21, MPDI 22, MPDI 23	9
	Group C (Figure 8)	MPDI 1, MPDI 20	2
	Group D (Figure 9)	MPDI 7	1

reflecting fine-scale differences in hydrocarbon solvent composition and overall ink formulation.

To substantiate the inherent similarity between samples and identify the group, PC scores were subjected to cluster analysis to accomplish unsupervised classification. Hierarchical complete linkage clustering technique with squared Euclidean distance was used for this purpose. Based on the clustering seen in the scatterplot between PC1 and PC2, the number of clusters for HCA complete linkage was set to 4. The dendrogram in Figure 5 and Table 4 shows the 4 identified groups of disappearing ink samples based on their spectra. Group A comprises of 18 samples followed by Group

B with 9 samples. Both these groups derived from HCA can also be seen as separate clusters in PC score plot in Figure 4. Group C only comprises of only two samples MPDI 1 and MPDI 20 while Group D only contains sample MPDI 7 which is visually distinguishable in the score plot also. The spectra of group wise sample are shown in Figure 6-9.

These clusters were further assessed for supervised classification utilizing Discriminant Analysis (DA) using ten PCs. The mean values for ten PCs varied significantly across groups suggesting possible disparities in their spectral characteristics with group A showing a high positive mean for PC1 (~1.92) while group B has a strong negative mean

**Figure 5-** Dendrogram from HCA showing sample classification.

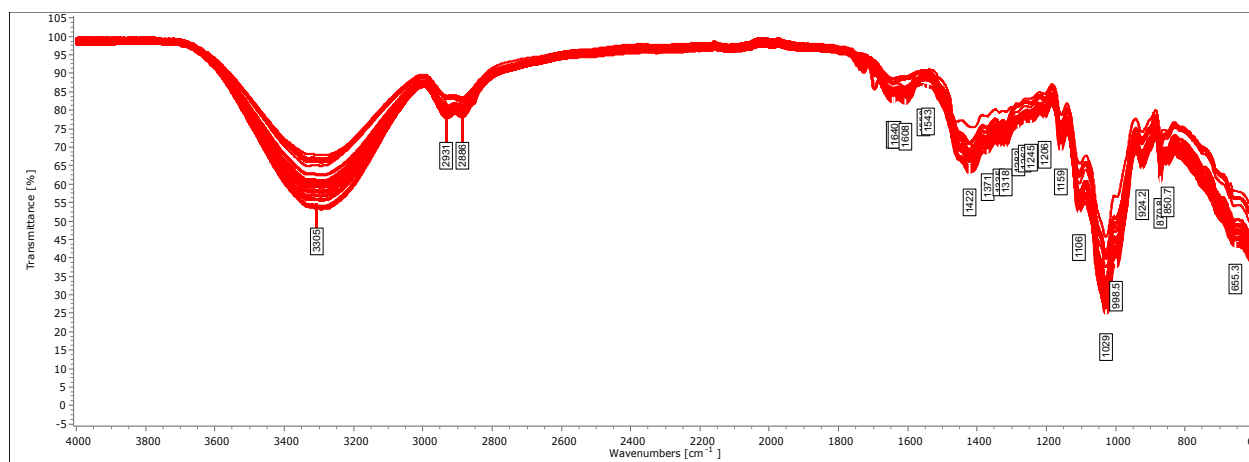


Figure 6- Group A (MPDI 3, 9, 10-16, 18-19, 24-30)

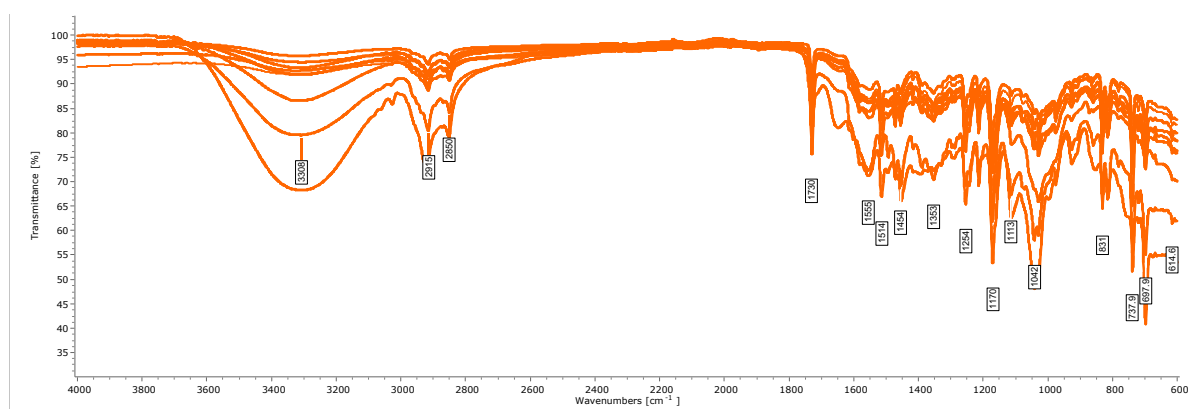


Figure 7- Group B (MPDI 2-6, 8, 17, 21-23)

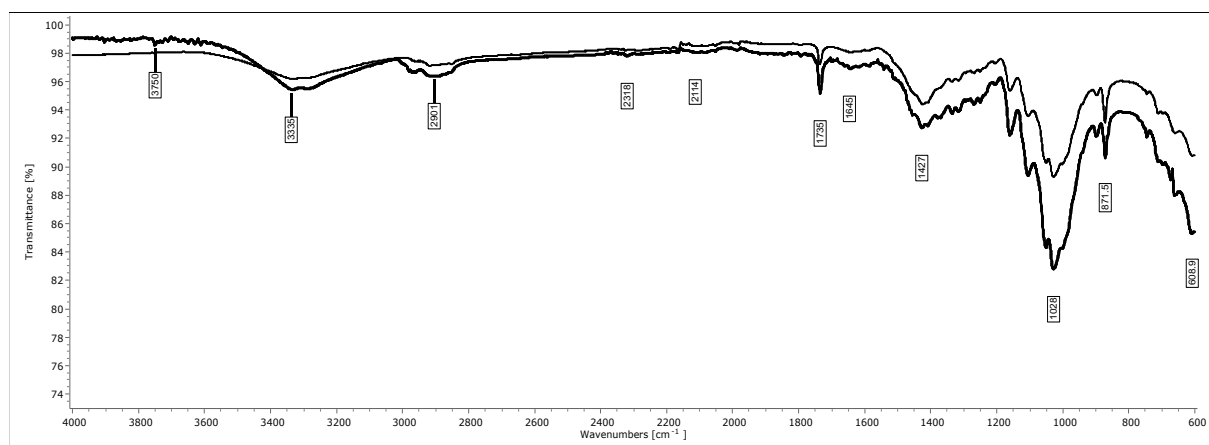


Figure 8- Group C (MPDI 1 and MPDI 20)

for PC1 (~3.68). Group C is distinguished by a high PC-3 mean (1.54) and a very low PC-2 mean (-1.94). Group D stands out with significantly high positive scores in PC-2 (2.57) and PC-3 (2.86). Table 5

shows the result of univariate F- test for each of 10 PCs considered for discriminant analysis to assess whether group means are significantly different or not. The results of the test clearly depict PC 1 and



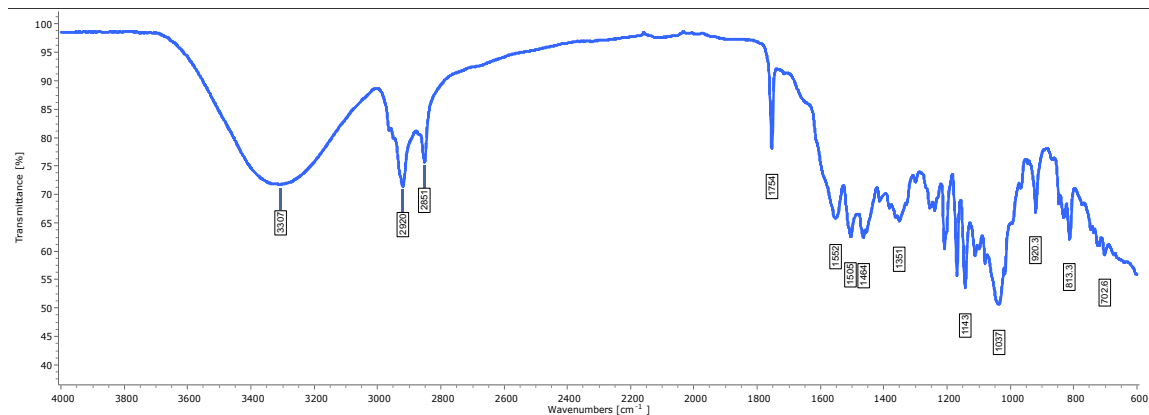


Figure 9- Group D (MPDI 7)

Table 5- Results of test of equality of means

	Wilk's Lambda	F	df1	df2	Sig.
PC 1	.016	1783.818	3	86	.000
PC 2	.199	115.174	3	86	.000
PC 3	.070	382.823	3	86	.000
PC 4	.862	4.588	3	86	.005
PC 5	.990	.284	3	86	.837
PC 6	.993	.191	3	86	.902
PC 7	.981	.546	3	86	.652
PC 8	.987	.375	3	86	.771
PC 9	.995	.152	3	86	.928
PC 10	.984	.465	3	86	.707

PC 3 as the strongest discriminator with higher F value, wilk's lambda close to 0 and statistically significant p value followed by PC 2 and PC 4. PC 9 was found to be least discriminating.

The canonical discriminant functions are summarized in Table 6. The number of functions is determined as  $n-1 = 3$  where "n" is the number of groups. The first function is found to be most discriminating with eigenvalue of 1049.343 explaining 86.4% of the variance and exhibiting an exceptionally strong canonical correlation of 1.000.

The sequential significance test, represented in Table 6 further corroborate that all the three

functions hold statistical significance implying each function contributing uniquely and meaningfully in overall separation of the groups of disappearing inks. The test for "1 through 3" functions yield a Wilks' Lambda of almost 0.000 with a chi square value of 1200.658 and significance  $<.001$  suggesting the full model is capable of discriminating between the groups. The test for "2 through 3" functions and function 3 alone also show high significance indicating all these functions contribute significantly to the group separation requiring a three-dimensional discriminant space for interpretation or visualization.



**Table 6-** Eigenvalues and wilk's lambda for canonical discriminant functions

Function	Eigenvalues			Wilks' Lambda			
	1	2	3	Test of Function(s)	1 through 3	2 through 3	3
Eigenvalues	1049.343	151.323	13.286	Wilks' Lambda	.000	.000	.070
% of Variance	86.4	12.5	1.1	Chi-square	1200.658	630.195	218.062
% cumulative	86.4	98.9	100.0	df	30	18	8
Canonical Correlation	1.000	.997	.964	Sig.	.000	.000	.000

**Table 7-** Confusion matrix for discriminant analysis model

		Predicted Group Membership				Total	
		Group A	Group B	Group C	Group D		
Original	Count	Group A	54	0	0	0	54
		Group B	0	27	0	0	27
		Group C	0	0	6	0	6
		Group D	0	0	0	3	3
	%	Group A	100.0	0.0	0.0	0.0	100.0
		Group B	0.0	100.0	0.0	0.0	100.0
		Group C	0.0	0.0	100.0	0.0	100.0
		Group D	0.0	0.0	0.0	100.0	100.0
Cross-validated	Count	Group A	54	0	0	0	54
		Group B	0	27	0	0	27
		Group C	0	0	6	0	6
		Group D	0	0	0	3	3
	%	Group A	100.0	0.0	0.0	0.0	100.0
		Group B	0.0	100.0	0.0	0.0	100.0
		Group C	0.0	0.0	100.0	0.0	100.0
		Group D	0.0	0.0	0.0	100.0	100.0

a. 100.0% of original grouped cases correctly classified.

b. Cross validation is done only for those cases in the analysis. In cross validation, each case is classified by the functions derived from all cases other than that case.

c. 100.0% of cross-validated grouped cases correctly classified.

The discriminant function analysis model based on the clusters determined from the HCA performed considerably well attaining an original and cross-validated accuracy of 100.00% (Table 7). The DFA

plot in Figure 10 represents the four groups and their centroid, plotted between function 1 and function 2 clearly reveals the four clusters separable from each other, further proving the robustness of the model.



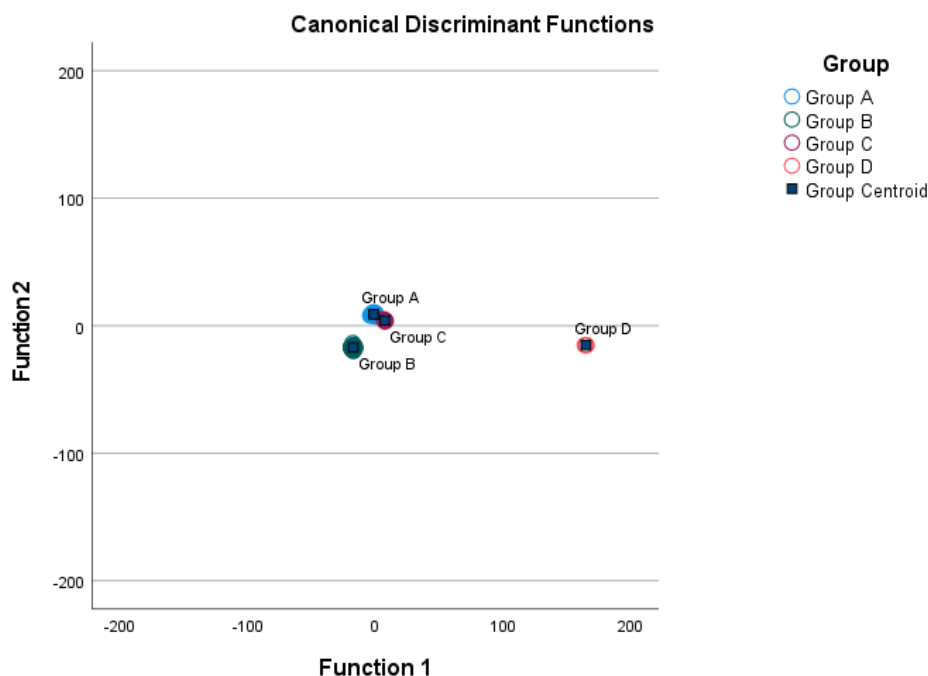


Figure 10- Combined group-wise plot of canonical discriminant functions.

Table 8- Summary of canonical discriminant functions for brand/maker-wise classification of sample

Function	Eigenvalues			Canonical Correlation	Test of Function(s)	Wilks' Lambda			
	Eigenvalues	% of Variance	% cumulative			Wilks' Lambda	Chi-square	df	Sig.
1	9838.251	75.8	75.8	1.000	1 through 10	0.000	2774.947	290	0.000
2	2613.574	20.1	95.9	1.000	2 through 10	0.000	2140.552	252	0.000
3	386.374	3.0	98.9	0.999	3 through 10	0.000	1597.601	216	0.000
4	81.567	0.6	99.5	0.994	4 through 10	0.000	1186.403	182	0.000
5	24.681	0.2	99.7	0.980	5 through 10	0.000	881.863	150	0.000
6	16.844	0.1	99.8	0.972	6 through 10	0.000	657.907	120	0.000
7	11.45	0.1	99.9	0.959	7 through 10	0.001	459.072	92	0.000
8	6.326	0.0	100.0	0.929	8 through 10	0.016	285.074	66	0.000
9	4.33	0.0	100.0	0.901	9 through 10	0.118	147.662	42	0.000
10	0.595	0.0	100.0	0.611	10	0.627	32.196	20	0.041

### 3. 1. Brand-wise (Manufacturer) Classification

The study also attempted to link each sample to their respective manufacturer which can be useful in forensic casework using DA. Although, the major composition for the disappearing ink is largely same across different manufacturer, there

are minute variations which can be utilized in their discrimination and identification in real caseworks and can be relevant exclusionary evidence. For manufacturer/brand wise DA classification of the 90 samples from 30 different manufacturers, computed 10 PCs were used as input.



The analysis yielded 10 canonical discriminant functions with first function accounting for 75.8% of the total variances and eigenvalue of 9838.251. First three functions cumulatively attributed for 98.5% of the total variance. The results of the eigen analysis of canonical discriminant functions used for classification is shown in Table 8. Further, test of function revealed that combination of all ten functions had a high chi square value, wilk's lambda value closer to zero and high significance. The results show that function 10 alone couldn't discriminate between different brands.

The model attained an overall original classification accuracy of 97.8% with two misclassifications where MPDI 13C and MPDI 26B were wrongly classified as belonging to MPDI 14 and MPDI 10 class respectively. The model was further validated using leave one out classification (LOOCV) cross-validation method. However, it achieved an accuracy of only 82.2% in cross-validation revealing some degree of overfitting which can be curbed by increasing the number of samples. This however, shows FTIR spectra integrated with chemometrics can be utilized for linking disappearing ink samples available in the market to their particular manufacturer with a fair degree of accuracy.

#### 4. Discussion

Not much literature exists on the analytical examination of magic pen inks. Existing works are mostly focussed on the visual examinations, chemical tests and Video Spectral Comparator (VSC) analysis of disappearing works. However, a lot of studies have been carried out on the IR spectral analysis of standard inks used in ballpoint, roller, gel pen inks. Kamil et al. [39] analysed eleven unbranded black ballpoint ink pens collected from an educational fair. The samples were non-

destructively analysed using FTIR within the range 1800-1200  $\text{cm}^{-1}$  and subjected to chemometric analysis, utilizing both Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA). The application of PCA data revealed the presence of seven distinct clusters (A, B, C, D, E, F, and G) in the score plot between PC 1 and PC 2 (accounting for 85.6% variance), from which five clusters (A, C, E, F and G) exhibited homogeneity, while two (B and D) were characterized as heterogeneous. HCA performed using the Euclidean distance, identified six clusters (A, B, C, D, E and F) in which two clusters (A and E) were found to be homogeneous, with the remaining four demonstrating heterogeneity at the similarity index of 86%. The study concluded that both PCA and HCA chemometric techniques failed to establish homogenous or neat clusters corresponding to the number of pens used in the study because certain unbranded black ballpoint pens were likely containing inks with similar chemical compositions. Silva et al. [22] employed Linear Discriminant Analysis (LDA) for classification of blue pens of 3 types: ballpoint (5 brands), roller ball (2 brands) and gel (3 brands). The study was carried on ten different pens of each brand and the samples were prepared on three different types of paper. The study achieved 100% accuracy in classifying pen ink samples according to their pen type on paper 1 using Genetic Algorithm (GA) and Successive Projections Algorithm (SPA) while 99.5% accuracy with Stepwise Formulation (SW). The study reported classification accuracy of 97.3%, 100% and 93.8% using GA, SPA and SW respectively on paper 2 while 100% for both SPA and GA, and 94.9% for SW on paper 3. The study further attained 100% accuracy in classifying blue pen ink according to their brand on paper 1 and 2 with all the three models. The study found LDA



models for classifications of pens according to their brand were 100% correct for the test set of blue pen ink spectra obtained from papers 1 and 2. The study found LDA-SPA and LDA-GA performed slightly better than LDA-SW models. Lee [40] subjected the results from ATR-FTIR of 37 black ballpoint pen samples representative of 9 different models from Papermate brand collected from Malaysia, to chemometric analysis. The study used PCA, PCA-DA and ZCA-PCA-DA for dimension reduction of dataset and achieved a discrimination of up to 85.70%. Halim *et al.* [41] aimed to discriminate and categorize fifteen blue gel-pen inks from five different brands (Pilot, Faster, M&G, G-Soft, and Faber-Castell) by Fourier Transform Infrared Spectroscopy (FTIR) collected from Malaysian market. The study demonstrated that PCA alone offered 74.67% statistical discrimination among the fifteen blue gel-pen ink. Both FTIR-PCA and FTIR-AHC successfully discriminated Pilot and Faber-Castell inks, while the remaining brands Faster, M&G, and G-Soft inks were not perfectly discriminated. Zhao and Li [42] employed FTIR spectroscopy to analyse the ink from 30 different erasable pens. Chemometric technique such as Principal Component Analysis (PCA) and Heatmap analysis were applied to the spectra within the 650-1500  $\text{cm}^{-1}$  range to classify the erasable pen types related to varying fading mechanisms. Loading plot used first two principal components to form scatter plots and to establish the relation between all the infrared spectrum over more than 79% of the cumulative contribution rate.

The PC-DA method employed in the current study achieved an original and cross-validated accuracy of 100% for the classification of 90 samples from 30 manufacturers into 4 groups based on the spectral similarities and differences.

However, group C and D comprised of only 6 samples (3 each from MPDI 1 & 20) and 3 samples (MPDI 7) respectively. This minor cluster reveals distinct chemical formulation rather than random variance; however limited size necessitates cautious interpretation of the model's performance. The use of leave-one-out cross-validation partially mitigates this issue by maximizing data utilization, but it does not fully compensate for class imbalance. The PCs based DA for brand classification also performed reasonably well attaining an original and cross-validated accuracy of 97.8% and 82.2% respectively. The study shows FTIR with chemometrics has potential to classify and discriminate the ink if residues of such are collected from the crime scene.

The proposed ATR-FTIR-chemometric framework is preliminary and performed under controlled conditions but offers an objective, reproducible and statistically validated evidence for discrimination and classification of disappearing ink formulations. Since the FTIR, techniques in ink examination also rely on binders and other polymeric components rather than just transient components, it will potentially be useful even in cases where documents have aged or even with partial or overwritten strokes. The present findings suggest that this approach can be useful in assisting the crime scene investigators and questioned document examiners in forgery detection. These classifications can be important circumstantial evidences, strengthening expert opinions on ink similarity or exclusion.

However, the study needs to be further expanded on with greater sample size, originating from different countries, analyzed over longer period of time and with other sophisticated analytical techniques to truly compare the potential of different techniques.



## 5. Conclusion

Magic ink that seemingly vanishes or changes over time attracts criminal minds to indulge in crime. It became a powerful tool to forge signature and other bank and will-related documents that disappear without leaving any traces. FTIR is an ideal tool for analysing and classifying magic pen inks. It can be used as a fast, accurate and cost-effective technique with no sample preparation. The IR spectra of magic pen inks showed the presence of thymolphthalein as the chief constituent in all the samples. The chemometric framework, successfully distinguished and grouped the 90 samples from 30 different manufacturers into four clusters, revealing underlying compositional differences and showed acceptable statistical discrimination with original and cross-validated accuracy of 100% respectively. Further, brand classification was also performed using PC-DA achieving an original and cross-validated accuracy of 97.8% and 82.2% respectively. Results have shown that ATR-FTIR spectroscopy coupled with chemometrics can be used as an effective tool for examining forged documents involving disappearing (magic pen) inks.

## 6. Limitations

- Controlled experimental conditions: The study was conducted under controlled laboratory conditions and does not explicitly simulate all real work variables such as, environmental exposure, substrate heterogeneity, or overwritten strokes, which may influence spectral characteristics in real scenarios.
- Sample type: All the ink samples used in this study was blue in colour and sourced from Indian markets which may limit the generalizability of the results.
- Preliminary nature of the study: The study

is preliminary in nature on a small sample size of 90 from 30 different manufacturers. ATR FTIR spectral interpretation is largely indicative rather than confirmatory and the study needs further expansion with higher sample size and employing sophisticated analytical techniques along with FTIR.

- Unequal group sizes: Unequal group size may also affect the robustness and stability of the model.

## Conflict of Interest

The authors affirm that they have no known financial conflicts or personal affiliations that could have influenced the research presented in this article.

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