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ATR-FTIR SPECTROSCOPIC EVALUATION OF ADULTERATION IN MARKETED AYURVEDIC HERBAL POWDERS

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ABSTRACT

This study describes a rapid FTIR–based analytical method for detecting undeclared adulterants in marketed Ayurvedic Pure powders, addressing increasing concerns related to quality, safety, and authenticity in Ayurvedic formulations. Five commonly used herbs-Ashwagandha, Shatavari, Triphala, Neem and Tulsi were evaluated using Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy to detect the amount of Undeclared Adulterants. FTIR spectra were recorded in the mid-IR range of 4000–400 cm^{-1} using a Nicolet Summit X spectrometer, and processed through baseline correction, normalization, and fingerprint-region assessment. Five Authentic herbal standards (Ashwagandha, Shatavari, Triphala, Neem, Tulsi) and three frequent adulterants (calcium carbonate, starch, talc) were used to establish comparison profiles. Ashwagandha, Shatavari, and Triphala showed strong spectral correlation with authenticated standards, confirming purity and absence of adulterants. Tulsi and Neem samples exhibited significant deviations, including unexpected carbonyl peaks (1700–1735 cm^{-1}) and altered fingerprint patterns. Although these differences did not match adulterant-specific markers, they suggest chemotypic variations, use of different plant parts, or processing-related changes. Overall, ATR-FTIR proved to be a fast, non-destructive, and cost-effective preliminary screening tool for herbal quality evaluation. The method effectively highlights chemical inconsistencies

and supports strengthened quality assurance for Ayurvedic powders.

KEYWORDS: ATR-FTIR spectroscopy, Ayurvedic powders, Adulteration, Quality control, Authentication.

INTRODUCTION

Herbal medicines constitute an integral component of traditional medical systems such as Ayurveda and continue to be widely utilized owing to their complex phytochemical composition and broad spectrum of pharmacological activities, including antioxidant, anti-inflammatory, immunomodulatory, antimicrobial, and adaptogenic effects.[1] Medicinal plants such as Shatavari (*Asparagus racemosus*), Ashwagandha (*Withania somnifera*), Triphala, Neem (*Azadirachta indica*), and Tulsi (*Ocimum sanctum*) are extensively incorporated into Ayurvedic formulations for diverse therapeutic applications.[1]

The pharmacological significance of these herbs has been well documented. *Asparagus racemosus* exhibits rejuvenating, anti-ulcer, antioxidant, immunomodulatory, and galactagogue properties.[1] *Withania somnifera* has been extensively investigated for its adaptogenic and anti- stress potential, with clinical studies reporting significant reductions in stress and anxiety following administration of standardized extracts.[2,3] Polyherbal formulations such as Triphala demonstrate synergistic antioxidant, detoxifying, gastrointestinal, and immunological activities.[4,5] while *Ocimum sanctum* has been reported to possess antibacterial, anti-inflammatory, antistress, and anticancer properties.[6,7]

Despite their therapeutic relevance, the adulteration of herbal medicines represents a critical challenge in the global herbal industry. Factors such as economic incentives, high market demand, and insufficient regulatory oversight contribute to the substitution of authentic herbal materials with inferior plant components or the inclusion of undeclared synthetic drugs, thereby compromising product quality, safety, and therapeutic integrity. Although conventional analytical techniques including HPLC, GC-MS, and LC-MS are effective for adulterant detection, their routine application is often limited by high cost, complex sample preparation, and extended analysis time.

Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) spectroscopy has emerged as a rapid, non-destructive, and cost-effective analytical technique for herbal authentication. Previous investigations have demonstrated the successful detection of

synthetic adulterants such as sildenafil, sibutramine, and phenylbutazone using FTIR spectral fingerprinting approaches.[8]

In view of the widespread use and vulnerability of powdered Ayurvedic formulations to adulteration, the present study aims to develop and apply an ATR-FTIR-based analytical method for the detection of undeclared adulterants in selected Ayurvedic herbal powders. The proposed approach seeks to establish a reliable and efficient screening protocol to support quality assurance and authentication of herbal medicines.[1-8]

MATERIALS AND METHODS

Instrumentation

FTIR spectra were recorded using a Thermo Scientific Nicolet™ Summit X FTIR spectrometer (Thermo Fisher Scientific, USA) equipped with a DTGS detector and operated via OMNIC™ Paradigm software. Measurements were performed in attenuated total reflectance (ATR) mode using a single-reflection diamond ATR accessory, enabling direct analysis of powdered samples without KBr pellet preparation. Prior to analysis, the instrument was allowed to stabilize and performance parameters, including signal-to-noise ratio and wavenumber accuracy, were verified in accordance with the manufacturer's guidelines.

Materials

Commercial Ayurvedic formulations containing Shatavari (*Asparagus racemosus*), Ashwagandha (*Withania somnifera*), Triphala (combination of *Emblica officinalis*, *Terminalia chebula*, and *Terminalia bellirica*), Neem (*Azadirachta indica*), and Tulsi (*Ocimum sanctum / O. tenuiflorum*) were procured from the Indian market in the form of churnas, tablets, and capsules. Product details were recorded for traceability.

Authenticated raw herbal materials were obtained from certified suppliers and verified using macroscopic and microscopic characteristics. Reference adulterants, including selected pharmaceutical compounds reported in adulterated herbal products, were procured where available and stored as per manufacturer recommendations.

Sample Preparation

Authentic herbal materials were cleaned, dried at 40 ± 2°C to constant weight, pulverized, and passed through a #60 sieve to obtain uniform powder. Marketed tablets

were de-coated, powdered, and sieved, while capsule contents were collected and homogenized. Churnas were used as received after gentle de-agglomeration. All samples were stored in airtight containers under dry conditions until analysis.

For method development, intentionally adulterated samples were prepared by spiking authentic herbal powders with known adulterants at concentrations of 1–20% w/w and homogenized thoroughly to ensure uniform distribution.

FTIR Measurement Conditions

ATR-FTIR spectra were acquired over the range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1}, with 64 co-added scans per spectrum. Background spectra were collected prior to each sample set.

Approximately 2–3 mg of sample was placed on the ATR crystal, and uniform pressure was applied to ensure optimal contact. The ATR crystal was cleaned with ethanol or methanol between measurements to prevent cross-contamination. Each sample was analyzed in triplicate.

Spectral Pre-processing and Analysis

Spectral data were subjected to quality assessment, baseline correction, and vector normalization to minimize variability arising from sample contact and quantity. Savitzky–Golay smoothing and first- derivative processing were applied where necessary. Data interpretation primarily focused on the fingerprint region (1800–600 cm^{-1}), while the higher wavenumber region (4000–2500 cm^{-1}) was evaluated for O–H and C–H stretching vibrations.

Identification of Adulteration Markers

Spectra of marketed formulations were compared with authenticated reference spectra using visual overlay and similarity matching. The presence of unexpected functional-group bands—such as sharp carbonyl absorptions (1700–1730 cm^{-1}), sulfonyl (S=O) bands (1350–1150 cm^{-1}), or nitrile (C≡N) stretching (~2250 cm^{-1})—was considered indicative of potential adulteration. Samples exhibiting only characteristic herbal spectral features were classified as free from detectable adulterants.

Prevention of Contamination

Strict precautions were followed during sample preparation to avoid biological, chemical,

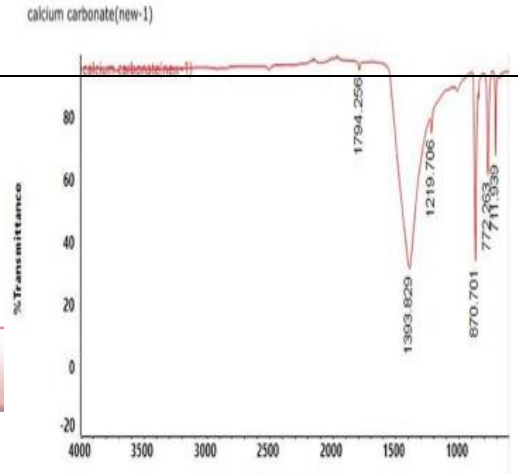
physical, and cross-contamination, as such contaminants may distort FTIR spectra, introduce baseline noise, or result in overlapping spectral features that compromise sample identification.

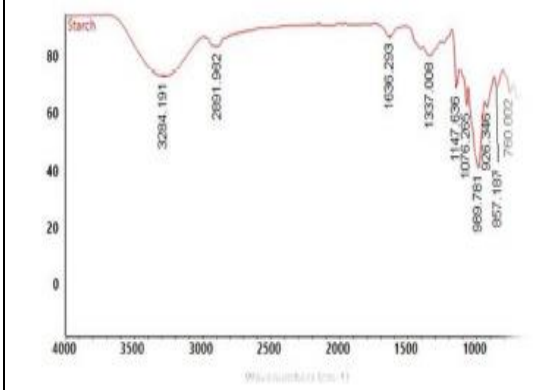
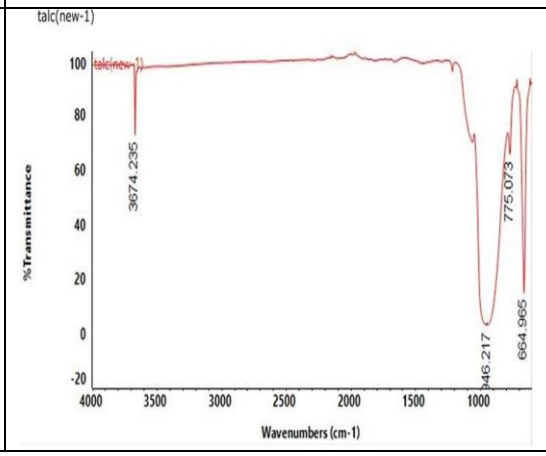
Herb Powder Sample	Contamination Source (Lab/Handling)	Adulteration Risk (Sample Quality)	Prevention Measures
Tulsi (Holy Basil)	Residue on ATR crystal - Cross-use of tools (e.g., spatulas)	Mixed with common basil - Added leaf powders or fillers	Clean ATR with ethanol - Use separate tools per sample - Compare with verified tulsi reference FTIR
Neem	Dust or ambient contamination - Moisture on glove or container	Mixed with other tree leaf powders - Stem powder added	Work in clean, dry environment - Dry powder before scanning - Verify fingerprint region vs. neem standard
Triphala	Improper tool cleaning between 3 ingredients - Residue from containers	Incorrect blend ratio - Use of substitutes like amla-only	Analyze each ingredient separately before mixing - Use reference spectra for all 3 - Record batch- wise FTIR
Shatavari	Shared grinding or weighing equipment - Glove residue	Mixed with starch or cheaper root powders	Clean tools with alcohol - Watch for starch peaks (e.g., broad OH stretch) - Use authentic dried root reference
Ashwagandha	Previous herb traces on equipment - Moisture in powder	Chalk powder or similar-looking root fillers	Clean ATR and spatulas - Ensure dry, homogenous powder - Compare full spectrum with known ashwagandha standard

RESULTS FOR FT-IR ANALYSIS OF UNDECLARED ADULTERANTS IN PURE AYURVEDIC POWDERS

IR SPECTRA FOR STANDARD ADULTRANTS SELECTED

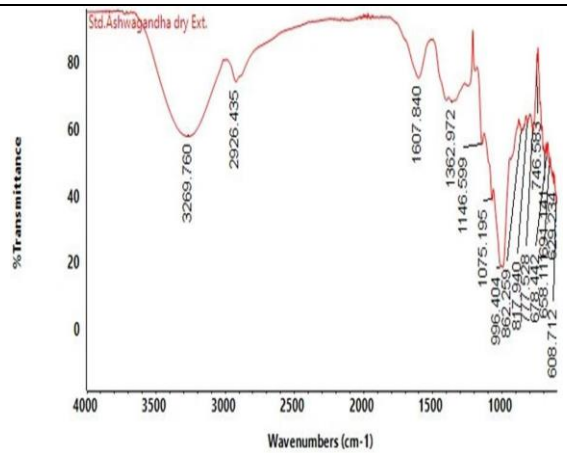
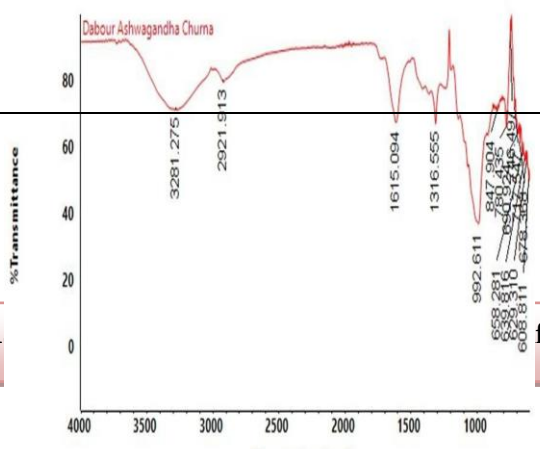
Table 01: FT-IR Spectral Data of Selected Undeclared Adulterants.

Adulterant	Spectra	Functional group	Peak Observed (Cm^{1})
Calcium Carbonate		CH_2	2877
		C=O	1793

		C-O	1394
Starch		OH	3284
		C-H	2941
		C-O	1337
Talc		OH	3674.24
		C=O	1820
		C-H	1062

THE IR SPECTRA FOR STANDARD & SAMPLES AYURVEDIC POWDERS

Table 02: FT-IR Spectral Data of Ashwagandha.

1.0	IR Spectra	Functional Group	Peak Observed (Cm^1)
ASHWAGANDHA STD		OH	3269.7
		C-H	2926.43
		C=C	1607
		C-O	1362
ASHWAGANDHA (SAMPLE 1)		OH	3281
		C-H	2921
		C=C	1615

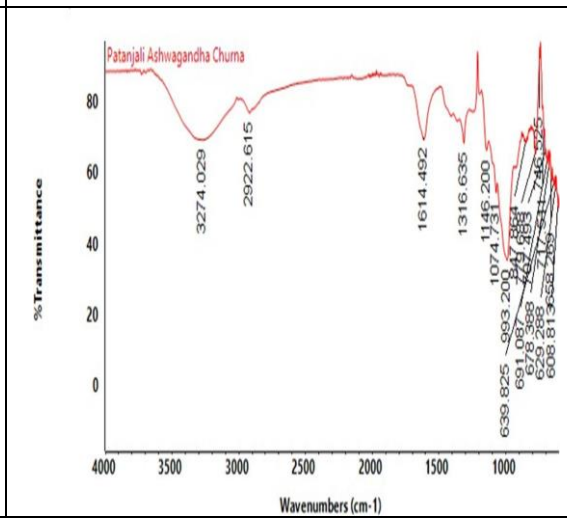
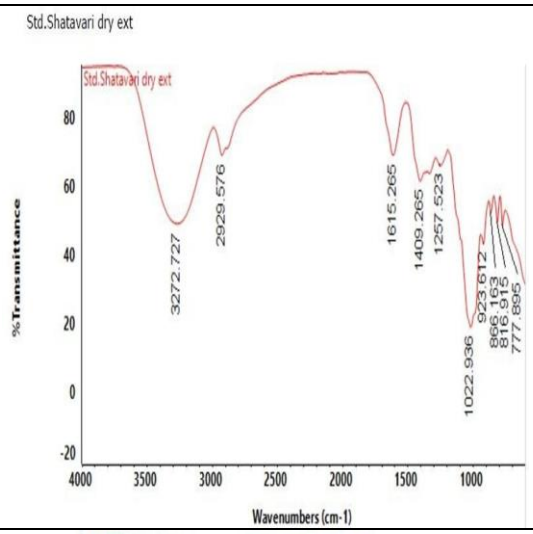
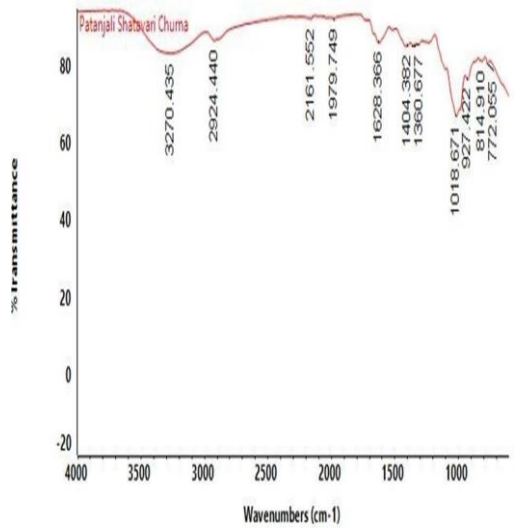
		C-O	1315
ASHWAGANDHA (SAMPLE 2)		OH	3274
		C-H	2922
		C=C	1614.4
		C-O	1316.6

Table 03: FT-IR Spectral Data of Shatavari.

2.0	IR Spectra	Functional Group	Peak Observed (Cm^1)
SHATAVARI STD		OH	3272
		C-H	2929.5
		C=C	1615
SHATAVARI SAMPLE 1		OH	3272
		C-H	2924
		C=C	1628
		C-O	1360
SHATAVARI		OH	3280

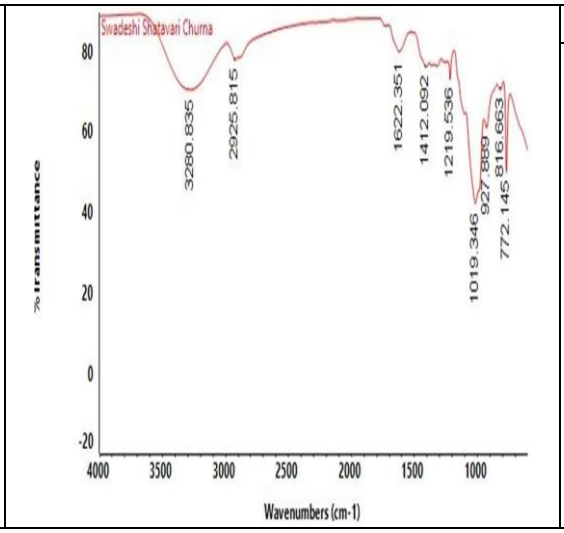
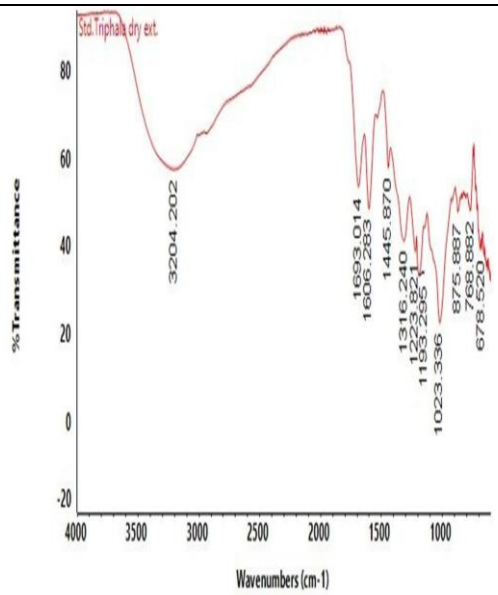
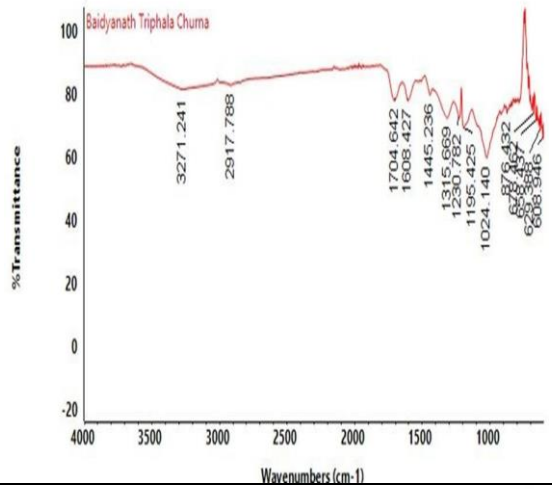
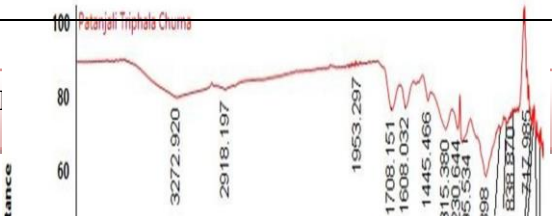
SAMPLE 2		C-H	2925
		C=C	1622

Table 04: FT-IR Spectral Data of Triphala.

3.0	**IR Spectra**	**Functional Group**	**Peak Observed (Cm^1)**
TRIPHALA STD		OH	3204.2
		C=O	1693
		C-O	1316
TRIPHALA SAMPLE 1		OH	3271
		C-H	2917
		C=O	1704
		C-O	1315
TRIPHALA CHURNA		OH	3272

SAMPLE 2		C-H	2918
		C=O	1708
		C-O	1315

Table 05: FT-IR Spectral Data of Neem.

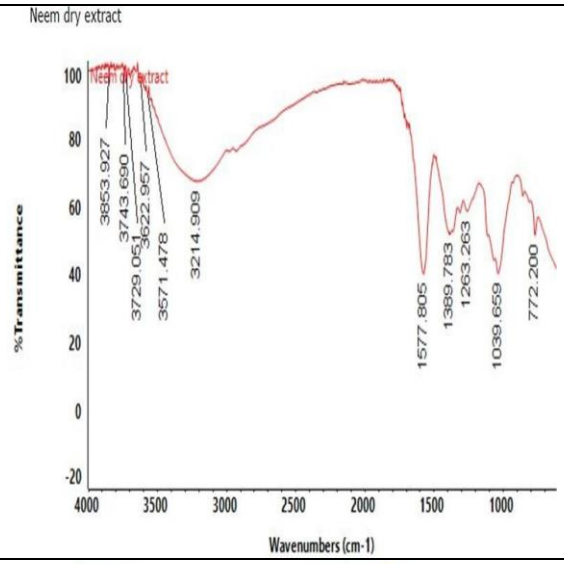
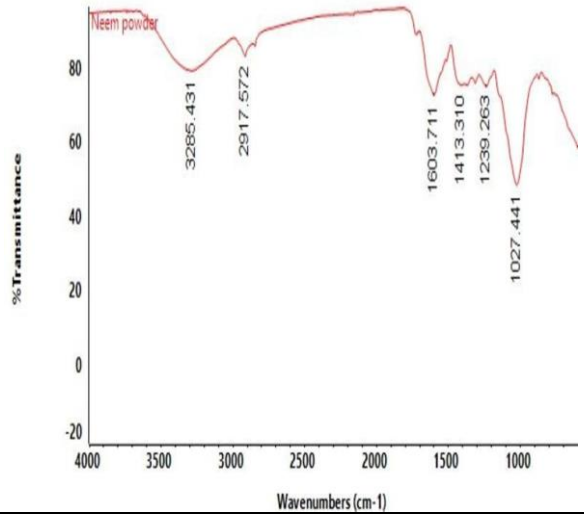
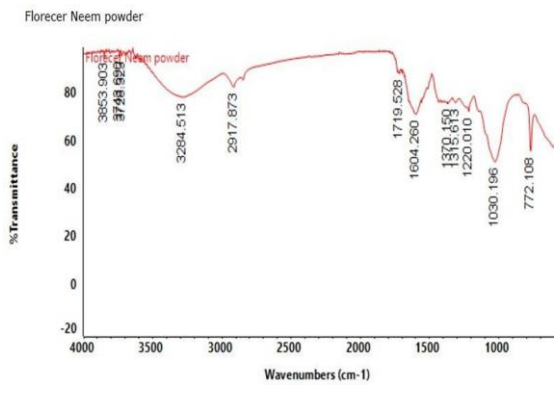
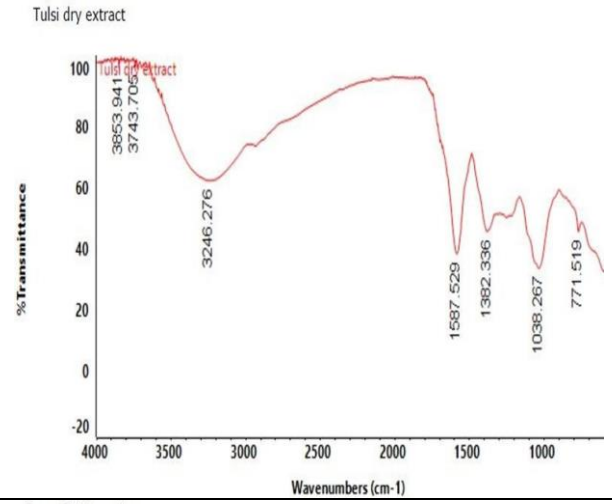
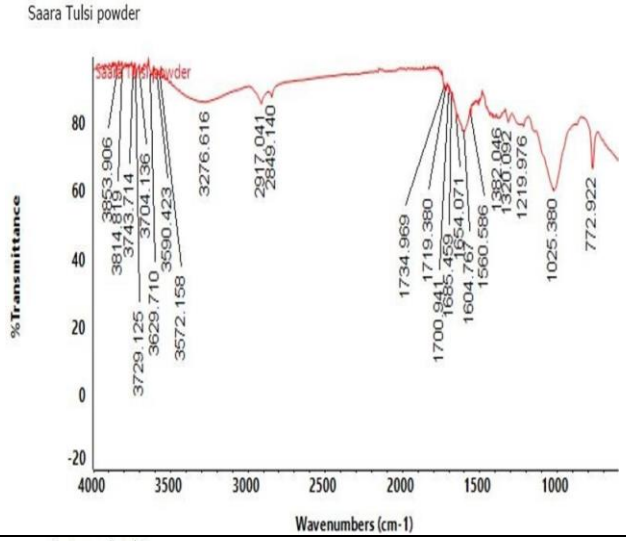
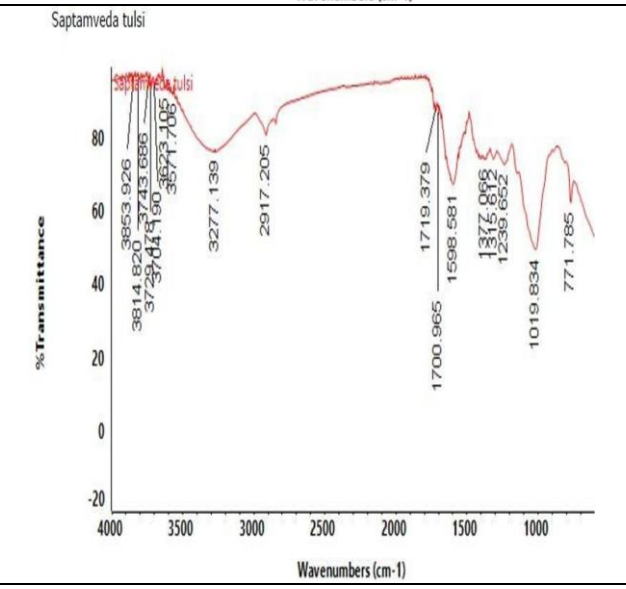
4.0	IR Spectra	Functional Group	Peak Observed (Cm^{1})
NEEM STD		OH	3214
		C-O	1389
		N=O	1577
NEEM SAMPLE 1		OH	3285
		C-H	2917
		C=O	1603
NEEM SAMPLE 2		OH	3284
		C-H	2917
		C=O	1719,1604
		C-O	1370

Table 06: FT-IR Spectral Data of Tulsi.

5.0	IR Spectra	Functional Group	Peak Observed (Cm^{1})
TULSI STD		OH	3246
		N=O	1587
		C-O	1382
TULSI SAMPLE1		OH	3276
		C-H	2917, 2849
		C=O	1604
		C-C	1320
TULSI SAMPLE 2		OH	3277
		C-H	2917
		C≡O	1719
		N=O	1598
		C-O	1315

COMPARISION OF IR SPECTRA OF STANDARD & SAMPLE WITH ADULTERANTS

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS

Table 7: Consolidated data for standard adulterants.

Adulterants	Functional Group	Characteristics peak (cm^{-1})
Calcium Carbonate	• Asymmetric C-O Stretch (Carbonate) • Out of Plane Bend (Carbonate) • Other Key Peak	1394.55 870.85 2511.13, 711.14
Starch	• O-H Stretch (Hydroxyl Groups) • C-H Stretch • Fingerprint Region	3284.19 2891.98 1636.29, 1147.63
Talc	• O-H Stretch • Si-O Stretch	3674.24 1062.39, 665.11

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS WITH STANDARD AND SAMPLE AYURVEDIC POWDER

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS WITH STANDARD AND SAMPLES OF POWDERED ASHWAGANDHA

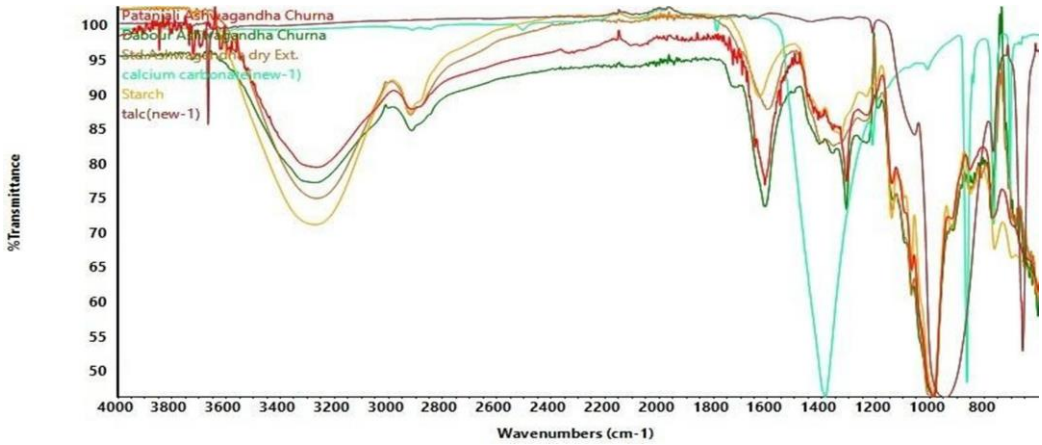


Fig. 1: Overlaid IR spectrum of Ashwagandha.

Table 8: IR Spectra Comparision for Ashwagandha.

Functional Group/Vibration	Std. Ashwagandha (cm^{-1})	Sample 1 Ashwagandha (cm^{-1})	Sample 2 Ashwagandha
O-H Stretch	2926.43	2921.91	2922.61
C-H Stretch	1607.84	1615.09	1614.49
C=O Stretch	1362.97, 1075.19	1316.55, 1074.73	1316.63, 1074.73
Fingerprint Region	2926.43	2921.91	2922.61

Interpretation and Conclusion

The spectra of both Ashwagandha sample show an excellent correlation with the standard Ashwagandha extract. The major peaks for O-H, C-H, and C=O stretching are highly comparable. Crucially, the fingerprint region (< 1500 cm^{-1}) of both commercial samples aligns well with the standard, indicating a similar chemical composition. There are no sharp, anomalous peaks that would indicate the presence of talc (e.g., 3674 cm^{-1}) or the characteristic peaks of calcium carbonate (e.g., 870 cm^{-1})

Authentication: The high degree of spectral similarity strongly suggests that both commercial samples are authentic.

Adulteration: There is no evidence of adulteration with talc, starch, or calcium carbonate.

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS WITH STANDARD AND SAMPLES OF POWDERED TULSI

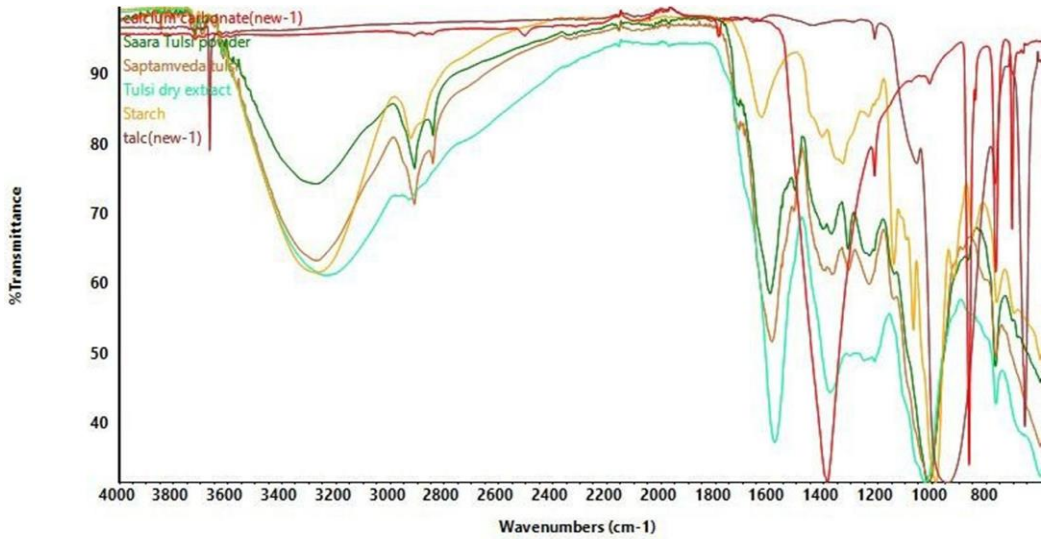


Fig. 2: Overlaid IR Spectrum of Tulsi Table 9: IR Spectra Comparision for Tulsi.

Functional Group/Vibration	Std. Tulsi (cm^{-1})	Sample 1 Tulsi (cm^{-1})	Sample 2 Tulsi (cm^{-1})
O-H Stretch	3246.27	3276.61	3277.13
C-H Stretch	3246.27	3276.61	3277.13
C=O Stretch	Absent	1734.96, 1719.38	1719.37, 1700.96
C=O Stretch	1587.52	1604.76, 1560.58	1598.58
Fingerprint Region	1382.33, 1038.26	1382.04, 1025.38	1378.06, 1019.83

InterpretationandConclusion

Tulsi Sample powders show significant deviation from the standard Tulsi extract, notably strong C=O peaks at 1700–1735 cm^{-1} that are absent in the standard, indicating major chemical differences. Poor correlation in the fingerprint region further confirms this mismatch.

Authentication

The commercial samples are not chemically consistent with the standard, possibly due to a different Tulsi chemotype, degradation, or altered processing.

Adulteration

Although no specific tested adulterants were detected, the pronounced chemical inconsistencies raise concerns about purity and equivalence to the standard.

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS WITH STANDARD AND SAMPLES OF POWDERED SHATAVARI

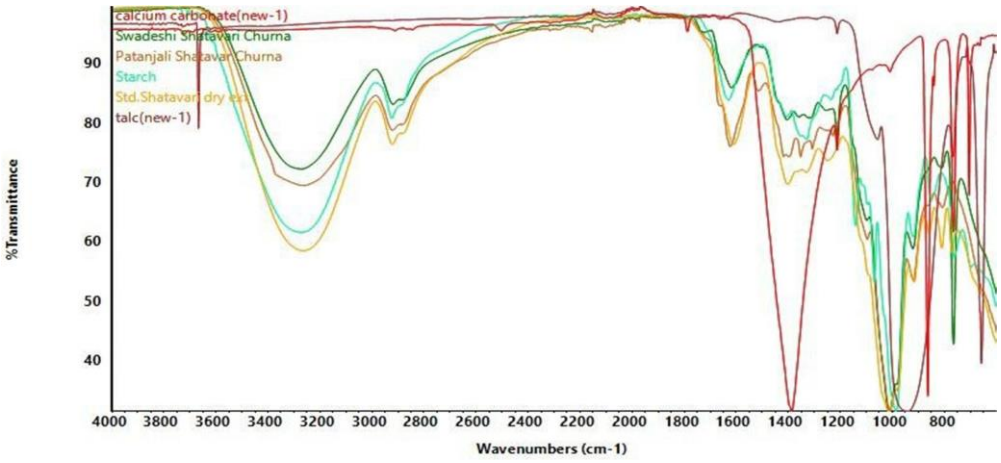


Fig. 3: Overlaid IR spectrum of Shatavari Table10: IR Spectra Comparision for Shatavari

Functional Group/Vibration	Std. Shatavari (cm^{-1})	Sample1 Shatavari (cm^{-1})	Sample 2 Shatavari (cm^{-1})
O-H Stretch	3272.72	3270.43	3280.83
C-H Stretch	2929.57	2924.44 32	2925.81
Amide I / C=O	1615.26	1626.36	1622.35
Fingerprint Region	1409.26, 1022.93	1404.38, 1018.67	1412.09, 1019.34

Interpretation and Conclusion

Shatavari samples show strong spectral similarity with the standard extract, including

matching functional group peaks and closely aligned fingerprint regions. Minor peak shifts fall within acceptable limits for natural products.

Authentication

Both commercial samples are authentic.

Adulteration

No evidence of adulteration with the tested substances was observed

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS WITH STANDARD AND SAMPLES OF POWDERED TRIPHALA

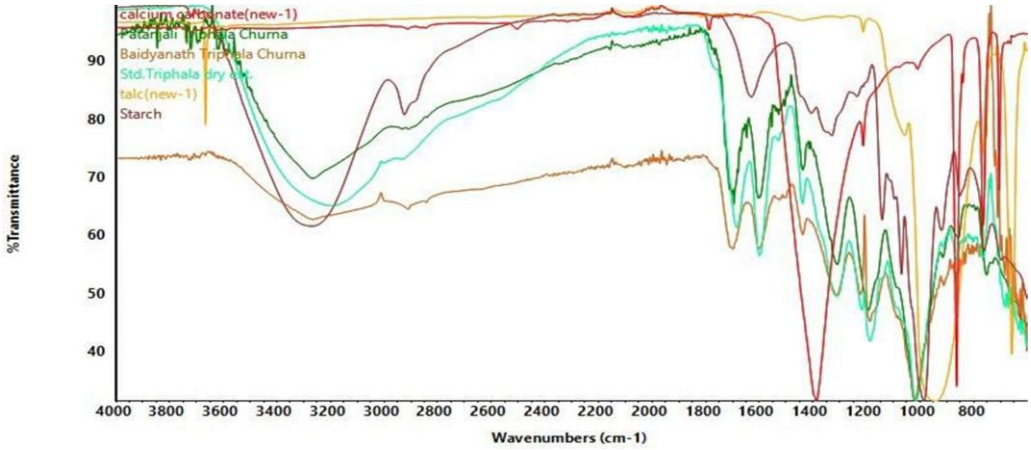


Fig. 4: Overlaid IR spectrum of Triphala Table11: IR Spectra Comparision for Triphala.

Functional Group/Vibration	Std. Triphala (cm^{-1})	Sample 1 Triphala (cm^{-1})	Sample 2 Triphala (cm^{-1})
O-H Stretch	3204.20	3271.24	3272.92
C-H Stretch	Not clearly defined	2917.78	2918.19
C=O Stretch	1693.01,	1704.64,	1708.15,
(Acid/Ketone)	1606.28	1608.42	1608.03
Fingerprint	1445.87,	1445.23,	1445.46,
Region	1023.33	1024.14	1023.89

Interpretation and Conclusion

The spectra of Triphala samples align very well with the standard. The characteristic double carbonyl (C=O) peaks, indicative of the rich presence of tannins and organic acids in Triphala, are present in both commercial samples. The high correlation in the fingerprint region further confirms that the complex mixture of compounds is consistent with the standard.

Authentication: Both commercial samples appear to be authentic.

Adulteration: There is no evidence of adulteration with the specified impurities.

COMPARISION OF IR SPECTRA OF STANDARD ADULTERANTS WITH STANDARD AND SAMPLES OF POWDERED NEEM

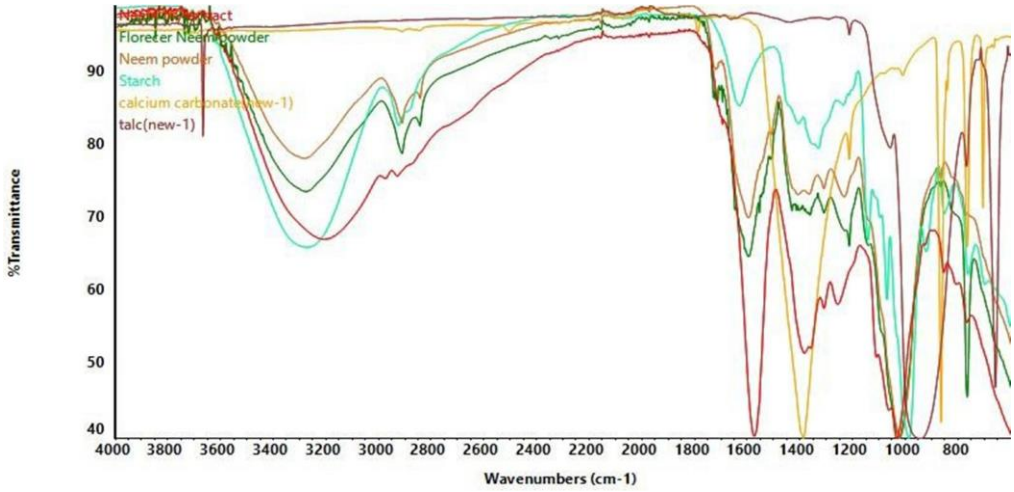


Fig. 5: Overlaid IR spectrum of Neem.

Table 12: IR Spectra Comparision for Neem.

Group/Vibration	Std. Neem (cm^{-1})	Sample 1 (cm^{-1})	Sample 2 (cm^{-1})
O-H Stretch	3214.90	3285.43	3284.51
C-H Stretch	Not clearly defined	2917.57	2917.87
C=O / C=C Stretch	1577.80	1603.71	1719.52, 1604.26
Fingerprint Region	1389.78, 1039.65	1413.31, 1027.44	1378.15, 1030.19

Interpretation and Conclusion

The commercial Neem powders show noticeable spectral differences compared to the standard Neem extract. Although the broad O–H stretching band is common to all samples, the sample 2 Neem exhibits a distinct sharp C=O peak at 1719.52 cm^{-1}, which is absent in the standard, indicating the presence of different or higher concentrations of carbonyl-containing compounds. Variations in the fingerprint region further confirm compositional differences.

Authentication: The commercial samples do not closely match the standard spectrum, suggesting they are not chemically identical. These differences may be due to variation in plant part used, geographical origin, or processing methods rather than adulteration.

Adulteration: There is no clear evidence of adulteration with talc, starch, or calcium carbonate. The anomalies are related to the organic composition itself.

CONCLUSION

The present study successfully demonstrated the applicability of ATR-FTIR spectroscopy as a rapid, non-destructive screening tool for evaluating the authenticity and purity of marketed Ayurvedic herbal powders. Comparative spectral analysis between authenticated standards, commercial samples, and common adulterants enabled effective assessment of chemical consistency.

Commercial samples of Ashwagandha, Shatavari, and Triphala exhibited high spectral concordance with their respective standards, particularly in characteristic functional-group regions and the fingerprint region, with no detectable peaks corresponding to calcium carbonate, starch, or talc. These findings confirm the authenticity and acceptable quality of these formulations.

Conversely, Tulsi and Neem samples showed notable deviations from the standard spectra, including unexpected carbonyl absorptions and altered fingerprint-region patterns. Although none of these deviations matched the spectral markers of the tested adulterants, the observed variations suggest influences of natural chemotypic diversity, plant part selection, processing conditions, or degradation rather than intentional adulteration.

Overall, the results highlight the effectiveness of ATR-FTIR spectroscopy for preliminary quality control of herbal products and underscore the necessity of complementary advanced techniques such as HPLC or LC–MS for confirmatory analysis, particularly when significant spectral deviations are observed.

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