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Chemometric authentication of branded and crude spices using integrated GC–MS fingerprints and elemental profiling

E. Udensi*, R. B. Salau, S. O. Salihu, M. M. Ndamitso, M. T. Bisiriyu

Department of Chemistry, Federal University of Technology, Minna, Niger State, Nigeria

Abstract

Counterfeit and substandard spice products present significant risks to food safety, nutritional quality, and consumer confidence. This study applied an integrated chemometric approach, GC–MS chemical profiling, and elemental analysis to authenticate and discriminate among crude, genuine-branded, and potentially adulterated spice products. Twenty spice samples from curry, ginger, turmeric, nutmeg, and thyme were analysed using gas chromatography–mass spectrometry (GC–MS) for volatile compounds, flame photometry for macroelements, and atomic absorption spectrometry (AAS) for microelements, respectively. Principal component analysis (PCA) and hierarchical cluster analysis (HCA) revealed clear separation between crude and branded spices. An optimized model framework using two principal components explained 70.55% of the total calibration variance (70.29% cross-validated variance). The hierarchical cluster analysis (HCA) dendrograms confirmed distinct clustering, highlighting close substitutability among certain spices based on their nutrient profiles. Elemental analysis indicated significant variation across samples: branded curry 3 and branded ginger 3 were rich in zinc and selenium, while branded nutmeg 1 exhibited exceptionally high iron content. Conversely, some branded products, such as branded curry powder, consistently showed low or absent levels of essential micronutrients. GC–MS profiling identified dominant fatty acids, including Octadec-9-enoic acid and Octadecanoic acid. These compositional differences underpin the discriminatory power of chemometric models, enabling reliable authentication of the crude and branded spices and the detection of potential adulteration. The study demonstrated that integrated chemical fingerprinting with multivariate analysis provides a robust framework for quality assurance and regulatory monitoring of commercial spice products.

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Keywords: Spice authentication, Integrated chemical fingerprinting, GC–MS profiling, Elemental analysis, Chemometrics.

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1. Introduction

Spices constitute an indispensable component of global food systems due to their roles in flavor enhancement, preservation, nutritional enrichment, and therapeutic functionality. Beyond their culinary significance, spices such as turmeric (*Curcuma longa*), ginger (*Zingiber officinale*), nutmeg (*Myristica fragrans*), thyme (*Thymus vulgaris*), and curry blends contribute essential macro- and microelements as well as bioactive phytochemicals that confer antioxidant, antimicrobial, and anti-inflammatory benefits [1–5]. Consequently, spices are increasingly valued not only as food additives but also as functional ingredients with nutraceutical relevance [6, 7].

However, the high economic value and extensive demand for spices have rendered them particularly vulnerable to food fraud and counterfeiting, especially in powdered and blended forms where visual authentication is challenging [8, 9]. Docu-

*Corresponding author Tel. No.: +234 8166-144-613.
Email address: successe4j@gmail.com (E. Udensi)

mented fraudulent practices include dilution with inferior plant materials, substitution of botanical species, addition of unauthorized colorants, and compositional manipulation to mimic genuine products [10, 11]. Such practices compromise product quality, undermine consumer trust, and pose significant public health risks due to potential exposure to toxic metals, synthetic dyes, and undeclared additives [12–14].

In developing markets, where regulatory enforcement and routine surveillance may be limited, counterfeit spice brands circulate alongside genuine products, often bearing indistinguishable packaging and labeling [8, 15]. Previous studies have reported substantial variability in the mineral composition of spices arising from differences in soil geochemistry, agricultural practices, processing conditions, and geographical origin [16–18]. While these compositional variations are natural to some extent, abnormal deviations from established chemical fingerprints may indicate adulteration or formulation inconsistencies [19].

Modern food authentication research has therefore shifted toward chemical fingerprinting strategies combined with multivariate data analysis, enabling holistic characterization of complex food matrices [20, 21]. Gas chromatography–mass spectrometry (GC–MS) has emerged as a powerful tool for profiling volatile and semi-volatile constituents of spices, providing detailed compositional signatures reflective of botanical origin and processing history [22–24]. In parallel, elemental profiling using atomic absorption spectrometry (AAS) offers complementary information related to mineral content, environmental exposure, and potential contamination [25–28].

While individual analytical techniques have demonstrated utility in spice characterization, single-marker or univariate approaches are often insufficient for reliable authentication due to the inherent complexity of spice matrices [28]. As a result, chemometric techniques, including principal component analysis (PCA) and hierarchical cluster analysis (HCA), have gained prominence for pattern recognition, sample classification, and anomaly detection in food authenticity studies [20, 29, 30]. These multivariate tools enable the extraction of meaningful information from high-dimensional datasets and facilitate discrimination between genuine, adulterated, and counterfeit products without prior assumptions [31].

Recent studies have successfully applied spectroscopic and chromatographic data coupled with chemometrics to detect adulteration in cinnamon, cumin, ginger, and turmeric powders [31–34]. Nevertheless, most reported investigations rely on laboratory-prepared adulterants or single-spice systems, which may not adequately represent real-market conditions. Moreover, limited attention has been given to integrated chemical–elemental fingerprinting approaches for the authentication of commercially branded spices, particularly within African markets where product heterogeneity is pronounced [8, 35].

Against this backdrop, there remains a critical need for robust, reproducible, and market-relevant authentication frameworks capable of distinguishing crude reference spices from branded commercial products and identifying compositional anomalies indicative of counterfeiting. The integration of GC–MS chemical fingerprints with elemental profiles, interpreted through chemometric modelling, offers a promising solution to this challenge by capturing both organic and inorganic dimensions of spice authenticity.

Therefore, the present study aims to develop a chemometric-modelled authentication strategy for crude and branded spice products using combined GC–MS volatile profiling and FP and AAS-based elemental analysis.

Table 1: GC–MS operating and chromatographic conditions for chemical fingerprinting.

Parameter	Condition
Instrument	Shimadzu GCMS-QP2010 Plus
Ionization mode	Electron Impact (EI)
Ionization energy	70 eV
Column	HP-5 MS fused silica capillary column (30 m × 0.25 mm × 0.25 μm)
Carrier gas	Helium (99.999% purity)
Carrier gas flow rate	1.6–2.5 mL · min^{-1}
Injection volume	1.0 μL
Injection mode	Split (25:1)
Injector temperature	250 °C
Ion source temperature	200 °C
Interface temperature	250 °C
Mass scan range	45–700 m/z
Oven temperature program:	
Step 1	60 °C, hold 1 min
Step 2	Ramp to 220 °C at 18 °C·min^{-1}, hold 1 min
Step 3	Ramp to 280 °C at 15 °C·min^{-1}, hold 2 min

Table 2: HCA calibration model.

Principal Component	Eigenvalue of Cov(X)	% Variance This PC	% Variance Cumulative
1	3.75×10^{0}	23.45	23.45
2 (Selected)	3.44×10^{0}	21.52	44.98
3	2.28×10^{0}	14.25	59.23
4	1.81×10^{0}	11.33	70.55
5	1.36×10^{0}	8.47	79.03
6	1.13×10^{0}	7.06	86.09
7	8.20×10^{-1}	5.12	91.21
8	6.85×10^{-1}	4.28	95.50
9	4.72×10^{-1}	2.95	98.44

Table 3: PCA calibration model.

Principal Component	Eigenvalue of Cov(X)	% Variance This PC	% Variance Cumulative
1	3.75×10^{0}	23.45	23.45
2 (Suggested)	3.44×10^{0}	21.52	44.98
3	2.28×10^{0}	14.25	59.23
4	1.81×10^{0}	11.33	70.55
5	1.36×10^{0}	8.47	79.03
6	1.13×10^{0}	7.06	86.09
7	8.20×10^{-1}	5.12	91.21
8	6.85×10^{-1}	4.28	95.50
9	4.72×10^{-1}	2.95	98.44

2. Methodology

2.1. Chemicals and reagents

All reagents used in this study were of analytical grade. Nitric acid (HNO3, 65%), hydrogen peroxide (H2O2, 30%), and ethanol (≥99.5%) were purchased from Tahoise Chemicals

Table 4: Macroelement content of selected spice samples (mg/g).

Sample	K	Na	Ca	Mg
C	5.82 ± 0.01^{k}	0.95 ± 0.01q	5.806 ± 0.03q	3.604 ± 0.03^{o}
BC1	6.32 ± 0.01^{m}	1.17 ± 0.21^{e}	5.681 ± 0.01^{p}	0.719 ± 0.01^{d}
BC2	4.88 ± 0.02^{h}	4.05 ± 0.02^{h}	4.690 ± 0.02^{l}	1.605 ± 0.02^{j}
BC3	4.65 ± 0.01^{g}	3.26 ± 0.01^{g}	2.554 ± 0.01^{b}	1.701 ± 0.01^{k}
G	6.31 ± 0.03^{l}	1.02 ± 0.01^{a}	3.920 ± 0.02^{e}	5.087 ± 0.01^{r}
BG1	5.82 ± 0.01k	0.86 ± 0.02^{o}	2.997 ± 0.01^{c}	0.919 ± 0.02^{e}
BG2	3.79 ± 0.01^{f}	0.87 ± 0.01^{p}	4.627 ± 0.02^{k}	1.306 ± 0.01^{h}
BG3	6.90 ± 0.02^{p}	1.22 ± 0.01	4.513 ± 0.03^{i}	1.768 ± 0.20^{m}
T	7.14 ± 0.01q	0.97 ± 0.02^{r}	5.574 ± 0.02^{o}	5.242 ± 0.03^{s}
BT1	6.84 ± 0.30^{o}	1.13 ± 0.01^{c}	5.918 ± 0.02^{r}	1.563 ± 0.01^{i}
BT2	5.15 ± 0.01^{i}	1.06 ± 0.11^{b}	5.325 ± 0.01^{n}	1.200 ± 0.01^{g}
BT3	6.41 ± 0.00^{n}	0.69 ± 0.02^{j}	1.446 ± 0.01^{a}	1.720 ± 0.02^{l}
N	3.10 ± 0.01^{e}	0.85 ± 0.03^{n}	3.365 ± 1.02^{d}	5.564 ± 0.02^{t}
BN1	2.18 ± 0.01^{a}	0.84 ± 0.01^{m}	4.359 ± 1.01^{h}	0.667 ± 0.01^{b}
BN2	3.05 ± 0.02^{c}	0.70 ± 0.01^{k}	4.552 ± 1.01^{j}	1.015 ± 0.01^{f}
BN3	7.14 ± 0.03q	1.14 ± 0.13^{d}	4.344 ± 0.02^{g}	0.389 ± 0.02^{a}
TH	6.90 ± 0.02^{p}	1.22 ± 0.01^{f}	4.955 ± 1.02^{m}	3.215 ± 0.02^{p}
BTH1	3.07 ± 0.00^{d}	0.82 ± 0.02^{l}	4.303 ± 0.01^{f}	0.708 ± 0.01^{c}
BTH2	2.95 ± 0.02^{b}	0.68 ± 0.02^{i}	6.463 ± 0.03^{s}	1.860 ± 0.01^{n}
BTH3	5.27 ± 0.03^{j}	1.02 ± 0.01^{a}	4.305 ± 0.01^{f}	4.403 ± 0.03q

Thus, the highest Zn and Se: BC3 (branded curry 3) and BG3 (branded ginger 3), also the highest Fe: BN1 (branded nutmeg 1, 1.164 mg/g), and the Lowest micronutrients: BC2 (branded curry 2) consistently low across Zn, Fe, Cu, Mn, and Mo. And finally, Toxic metals: Cd and Pb are negligible (< detectable limits). Collectively, the elemental profiles underscore the nutritional potential of fresh and branded spices, particularly as sources of potassium, magnesium, selenium, and iron. Variations highlight the impact of species, processing, and branded formulation on elemental content, emphasizing the need for monitoring and quality control to ensure both nutritional value and safety in commercial spice products.

Table 5: Microelement content of selected spice samples (mg/g).

Sample	Zn	Fe	Cu	Mn	Mo	Se
C	0.025 ± 0.01^{de}	0.249 ± 0.10^{m}	0.007 ± 0.00^{de}	0.197 ± 0.01^{s}	0.004 ± 0.01^{b}	1.707 ± 0.09^{m}
BC1	0.019 ± 0.04^{cd}	0.49 ± 0.02^{r}	0.006 ± 0.00^{cd}	0.067 ± 0.02^{s}	0.006 ± 0.01^{c}	0.577 ± 0.04^{c}
BC2	0.002 ± 0.00^{a}	0.01 ± 0.01^{a}	0 ± 0.00^{a}	0.001 ± 0.01^{a}	0 ± 0.00^{a}	1.619 ± 0.02^{l}
BC3	0.112 ± 0.00^{g}	0.11 ± 0.01^{c}	0.009 ± 0.01^{fg}	0.06 ± 0.01^{j}	0.004 ± 0.01^{b}	7.172 ± 0.03^{s}
G	0.013 ± 0.01^{bc}	0.195 ± 0.04^{j}	0.008 ± 0.02^{ef}	0.096 ± 0.01^{q}	0.004 ± 0.00^{b}	2.542 ± 0.02^{p}
BG1	0.044 ± 0.01^{f}	0.164 ± 0.02^{h}	0.018 ± 0.01^{i}	0.065 ± 0.01^{k}	0.004 ± 0.10^{b}	0.835 ± 0.02^{g}
BG2	0.01 ± 0.01^{b}	0.133 ± 0.03^{f}	0.007 ± 0.01^{de}	0.02 ± 0.01^{d}	0.004 ± 0.00^{b}	0.687 ± 0.06^{f}
BG3	0.015 ± 0.02^{bc}	0.235 ± 0.02^{k}	0.006 ± 0.01^{cd}	0.084 ± 0.01^{n}	0.003 ± 0.27^{b}	7.631 ± 0.20^{t}
T	0.011 ± 0.01^{b}	0.122 ± 0.01^{d}	0.005 ± 0.00^{bc}	0.088 ± 0.01^{o}	0.006 ± 0.19^{c}	4.064 ± 0.21^{r}
BT1	0.026 ± 0.01^{de}	0.175 ± 0.05^{i}	0.007 ± 0.01^{de}	0.092 ± 0.10^{p}	0.003 ± 0.00^{b}	2.01 ± 0.00^{o}
BT2	0.015 ± 0.01^{bc}	0.244 ± 0.01^{l}	0.004 ± 0.01^{b}	0.025 ± 0.30^{f}	0.003 ± 0.02^{b}	0.5898 ± 0.01^{d}
BT3	0.019 ± 0.01^{cd}	0.3 ± 0.18^{q}	0.011 ± 0.00^{h}	0.177 ± 0.02^{r}	0.004 ± 0.01^{b}	1.352 ± 0.24^{k}
N	0.014 ± 0.03^{bc}	0.126 ± 0.01^{e}	0.004 ± 0.00^{b}	0.016 ± 0.04^{b}	0.004 ± 0.01^{b}	2.619 ± 0.00^{q}
BN1	0.044 ± 0.12^{f}	1.164 ± 0.31^{s}	0.009 ± 0.01^{fg}	0.016 ± 0.12^{b}	0.006 ± 0.00^{c}	0.443 ± 0.00^{a}
BN2	0.009 ± 0.01^{ab}	0.27 ± 0.01^{o}	0.009 ± 0.21^{fg}	0.022 ± 0.01^{e}	0.004 ± 0.01^{b}	0.562 ± 0.06^{b}
BN3	0.025 ± 0.01^{de}	0.296 ± 0.01^{p}	0.01 ± 0.00^{gh}	0.037 ± 0.01^{h}	0.003 ± 0.02^{b}	1.026 ± 0.19^{j}
TH	0.013 ± 0.01^{bc}	0.157 ± 0.01^{g}	0.006 ± 0.02^{cd}	0.038 ± 0.01^{i}	0.003 ± 0.20^{b}	0.854 ± 0.25^{h}
BTH1	0.045 ± 0.01^{f}	0.091 ± 0.01^{b}	0.008 ± 0.03^{ef}	0.068 ± 0.02^{m}	0.003 ± 0.00^{b}	0.663 ± 0.03^{e}
BTH2	0.016 ± 0.05^{bc}	0.195 ± 0.00^{j}	0.007 ± 0.01^{de}	0.018 ± 0.05^{c}	0.003 ± 0.00^{b}	0.867 ± 0.04^{i}
BTH3	0.028 ± 0.01^{e}	0.261 ± 0.01^{n}	0.008 ± 0.01^{ef}	0.03 ± 0.01^{g}	0.003 ± 0.01^{b}	1.971 ± 0.01^{n}

& Allied Products Industry, Paiko Road, Minna, Niger State, Nigeria, a reputable chemical wholesaler supplying laboratory reagents. Where applicable, reagents were sourced through authorised distributors of internationally certified reagent brands. Ultrapure deionized water used throughout the analyses was produced in-house using a laboratory water system.

Table 6: Chemical composition of crude curry.

Peak	Name	R. Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H
1	Phenol, 2-methyl-5-(1-methylethyl)-	6.225	6.142	6.325	1085094	4.76	118851	1.44	9.13
2	Thymol	6.351	6.351	6.492	1754398	7.69	628335	7.63	2.79
3	Phenol, 3-(1,1-dimethylethyl)-4-met	7.935	7.883	7.992	453625	1.99	156996	1.91	2.89
4	beta-linalene	8.075	7.992	8.108	133308	0.58	60491	0.73	2.20
5	Benzene, 1,2,3-trimethoxy-5-(2-prop	8.383	8.358	8.442	291024	1.28	178730	2.17	1.62
6	1-Naphthalenol, 1,2,3,4a,7,8,8a-oc	9.084	9.050	9.117	116322	0.51	64070	0.78	1.82
7	Ledol	9.194	9.117	9.250	338461	1.48	139459	1.69	2.43
8	Bergamotol, z-alpha-trans-	9.404	9.367	9.442	219141	0.96	121832	1.48	1.80
9	Isoaromadendrene epoxide	9.651	9.617	9.700	149351	0.66	81219	0.99	1.84
10	Tetradecanoic acid	9.732	9.700	9.767	143470	0.63	96811	1.18	1.48
11	1-(+)-Ascorbic acid 2,6-dihexadecan	11.176	11.133	11.258	2126379	9.33	1020254	12.39	2.08
12	Nerolidol	11.367	11.308	11.400	132326	0.58	58510	0.71	2.26
13	6,10,10-Dodecatrien-3-ol, 3,7,11-trim	11.600	11.567	11.642	126484	0.55	75994	0.92	1.66
14	Naphthalene, decahydro-1,1,4a-trime	11.849	11.817	11.883	597510	2.62	350902	4.26	1.70
15	2-Dodecen-1-yl-succinic anhydride	12.108	12.083	12.133	115926	0.51	53421	0.65	2.17
16	7-Isopropyl-1,1,4-trimethyl-1,2,3,4,	12.157	12.133	12.192	756740	3.32	458103	5.56	1.65
17	1-Heptatriacotanol	12.225	12.192	12.325	176746	0.78	63456	0.77	2.79
18	Thunbergol	12.373	12.325	12.408	715887	3.14	239519	2.91	2.99
19	Ethyl iso-allocholate	12.436	12.408	12.467	270384	1.19	111233	1.35	2.43
20	Octadec-9-enoic acid	12.538	12.467	12.617	6590513	28.91	2220105	26.96	2.97
21	Octadecanoic acid	12.681	12.617	12.758	3083617	13.52	777980	9.45	3.96
22	cis,cis-Linoleic acid	12.775	12.758	12.792	204552	0.90	113974	1.38	1.79
23	Tricyclo[20.8.0.0(7,16)]triacontane,	12.867	12.792	13.117	1108066	4.86	122735	1.49	9.03
24	2-Dodecen-1-yl-succinic anhydride	13.143	13.117	13.208	339696	1.49	82279	1.00	4.13
25	2,10-Dodecadien-1-ol, 3,7,11-trimetl	13.333	13.208	13.367	231424	1.02	55004	0.67	4.21
26	Pregnan-3,17,20-triol-(3.alpha.,5.b	14.017	13.983	14.100	610057	2.68	305367	3.71	2.00
27	Stigmasta-4,7,22-trien, 3.α.-ol	14.306	14.275	14.383	391559	1.72	192500	2.34	2.03
28	E,E,Z-1,3,11-Nonadecatriene-5,14-d	14.790	14.758	14.833	155476	0.68	112942	1.37	1.76
29	Hexadecanoic acid, 2-hydroxy-1-(hy	15.005	14.975	15.050	229407	1.01	88519	1.07	2.03
30	9-(Z)-Dimethylpropanohydrazyl	15.264	15.233	15.300	152545	0.67	86577	1.05	1.76
				Total	22799488	100.00	8236168	100.00	

Table 7: Chemical composition of branded curry 1.

Pk	Name	R. Time	I.Time	F.Time	Area	Area %	Height	Height%	A/H
1	1-Bromo-7-octanol	6.175	6.142	6.325	208456	1.20	32829	0.51	6.35
2	Thymol	6.350	6.325	6.425	5558284	3.22	276571	4.28	2.02
3	Phenol, 3-(1,1-dimethylethyl)-4-metl	7.900	7.900	8.000	151874	0.88	71309	1.10	2.13
4	Pentanedioic acid, (2,4-di-t-butylphe	8.075	8.042	8.125	98527	0.57	62472	0.97	1.58
5	Ledol	9.198	9.158	9.250	183047	1.06	77729	1.20	2.35
6	Bergamotol, z-alpha-trans-	9.405	9.367	9.467	138363	0.80	67990	1.05	2.04
7	Undecanoic acid, 10-bromo-	9.733	9.708	9.925	68040	0.39	30404	0.47	2.24
8	1-(+)-Ascorbic acid 2,6-dihexadecan	11.177	11.133	11.258	1915243	11.05	912038	14.11	2.10
9	Phthalic acid, dodecyl 2-(2-methoxy)	11.283	11.258	11.317	89262	0.51	44793	0.69	1.99
10	6,10,10-Dodecatrien-3-ol, 3,7,11-trim	11.367	11.317	11.400	88062	0.50	35222	0.54	2.44
11	6,10,10-Dodecatrien-3-ol, 3,7,11-trim	11.603	11.575	11.650	72799	0.42	39533	0.61	1.84
12	Naphthalene, decahydro-1,1,4a-trime	11.850	11.825	11.883	325630	1.88	198657	3.07	1.64
13	7-Isopropyl-1,1,4-trimethyl-1,2,3,4,	12.159	12.133	12.192	396742	2.29	256068	3.96	1.55
14	2-Dodecen-1-yl-succinic anhydride	12.362	12.317	12.408	440566	2.54	151453	2.34	2.91
15	Ethyl iso-allocholate	12.439	12.408	12.467	168486	0.97	71292	1.10	2.36
16	Octadec-9-enoic acid	12.540	12.467	12.625	6453810	37.22	2176098	33.67	2.97
17	Octadecanoic acid	12.682	12.625	12.758	2607280	15.04	728722	11.27	3.58
18	2-Methyl-Z,Z,3,13-octadecadienol	12.775	12.758	12.800	213636	1.23	99002	1.53	2.16
19	Z,Z-8,10-Hexadecadien-1-ol	12.872	12.800	12.992	697623	4.02	102998	1.59	6.77
20	2-Dodecen-1-yl-(y)-succinic anhydride	13.061	12.992	13.108	225187	1.30	59542	0.92	3.78
21	Octadecanoic acid	13.145	13.108	13.225	318643	1.84	72544	1.12	4.39
22	2-Dodecen-1-yl-(y)-succinic anhydride	13.331	13.225	13.367	200017	1.15	45664	0.71	4.38
23	Silane, tetra-2-propenyl-	13.444	13.367	13.483	102683	0.59	64547	1.00	1.59
24	Ethyl iso-allocholate	13.639	13.608	13.675	73784	0.43	41895	0.65	1.76
25	Pregnane-3,17,20-triol, (3.alpha.,5.b	14.019	13.983	14.108	488980	2.82	246221	3.81	1.99
26	Stigmasta-4,7,22-trien-3.alpha.-ol	14.308	14.267	14.400	378793	2.18	165680	2.56	2.29
27	Octadecanol, 2-bromo-	14.792	14.758	14.833	165726	0.96	98757	1.53	1.68
28	Ethyl iso-allocholate	14.886	14.833	14.967	119624	0.69	34718	0.54	3.45
29	Hexadecanoic acid, 2-hydroxy-1-(hy	15.008	14.967	15.050	268749	1.55	123145	1.91	2.18
30	9-(Z)-Dimethylpropanohydrazyl	15.267	15.233	15.300	123983	0.72	75834	1.17	1.63
				Total	17339899	100.0	6463727	100.00	

Table 8: Chemical composition of branded curry 2.

Peak#	Name	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H
1	Pentane, 1-bromo-5-methoxy-	6.167	6.142	6.317	66690	0.37	17294	0.27	3.85
2	Thymol	6.349	6.317	6.417	295721	1.65	150327	2.36	1.97
3	Pentanedioic acid, (2,4-di-t-butylphe	8.075	8.042	8.117	71164	0.40	48848	0.77	1.46
4	Tumerone	9.234	9.167	9.283	852102	4.75	299660	4.70	2.84
5	1,3,6,10-Dodecatetraene, 3,7,11-trim	9.404	9.283	9.433	77444	0.43	36762	0.58	2.11
6	Curlone	9.464	9.433	9.508	260775	1.46	172142	2.70	1.51
7	l-(+)-Ascorbic acid 2,6-dihexadecanx	11.178	11.133	11.258	1805635	10.07	844237	13.24	2.14
8	Phthalic acid, dodecyl 2-(2-methoxy-	11.283	11.258	11.325	88734	0.50	38596	0.61	2.30
9	1,6,10-Dodecatrien-3-ol, 3,7,11-trim	11.367	11.325	11.400	65095	0.36	29033	0.46	2.24
10	Naphthalene, decahydro-1,1,4a-trim	11.850	11.825	11.883	191584	1.07	122207	1.92	1.57
11	9-Undecenal, 2,6,10-trimethyl-	12.117	12.083	12.133	57256	0.32	33936	0.53	1.69
12	7-Isopropyl-1,1,4a-trimethyl-1,2,3,4	12.160	12.133	12.192	253817	1.42	166508	2.61	1.52
13	Phytol	12.360	12.317	12.408	348285	1.94	130439	2.05	2.67
14	Ethyl iso-allocholate	12.439	12.408	12.467	122782	0.69	50771	0.80	2.42
15	Octadec-9-enoic acid	12.540	12.467	12.625	6234543	34.79	2155191	33.80	2.89
16	Octadecanoic acid	12.682	12.625	12.767	2567671	14.33	712589	11.17	3.60
17	cis,cis-Linoleic acid	12.775	12.767	12.800	167118	0.93	103340	1.62	1.62
18	Ethyl iso-allocholate	12.871	12.800	12.983	758659	4.23	101769	1.60	7.45
19	2-Dodecen-l-y1(-succinic anhydride	13.061	12.983	13.108	349071	1.95	76520	1.20	4.56
20	2-Dodecen-l-y1(-succinic anhydride	13.145	13.108	13.225	434607	2.42	87755	1.38	4.95
21	2-Dodecen-l-y1(-succinic anhydride	13.331	13.225	13.400	298043	1.66	52408	0.82	5.69
22	Silane, terta-2-propenyl-	13.444	13.400	13.483	153981	0.86	75512	1.18	2.04
23	2-Dodecen-l-y1(-succinic anhydride	13.640	13.483	13.675	110529	0.62	53115	0.83	2.08
24	Pregnane-3,17,20-triol, (3.alpha.,5.b	14.019	13.983	14.100	459267	2.56	232428	3.64	1.98
25	Stigmsta-4,7,22-trien-3.alpha.-ol	14.309	14.267	14.400	369986	2.06	169327	2.66	2.19
26	E,E,Z-1,3,12-Nonadecatriene-5,14-d	14.793	14.750	14.833	172016	0.96	94382	1.48	1.82
27	Ethyl iso-allocholate	14.887	14.833	14.908	68741	0.38	37085	0.58	1.85
28	Hexadecanoic acid, 2-hydroxy-1-(h	15.009	14.908	15.050	191321	1.07	103498	1.62	1.85
29	9-(2,2'-Dimethylpropanoylhydrazonc	15.268	15.233	15.300	144667	0.81	78112	1.22	1.85
30	Ergos-25-ene-3,5,6,12-tetrol, (3.bet	15.501	15.300	15.625	884862	4.94	103149	1.62	8.58

Table 9: Chemical composition of branded curry 3.

Peak	Name	R. Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H
1	Pentanedioic acid, (pt-butylphenyl) e	6.351	6.325	6.417	162732	0.94	96804	1.63	1.68
2	Pentanedioic acid, (2,4-d-t-butylphe	8.076	8.008	8.117	103096	0.60	63302	1.06	1.63
3	1-(+)-Ascorbic acid 2,6-hexadecanx	11.177	11.125	11.258	171888	0.95	830202	13.94	2.07
4	Phthalic acid, dodecyl 2-(2-methoxy	11.283	11.258	11.325	91620	0.53	33820	0.57	2.71
5	Naphthalene, decahydro-1,1,4a-trim	11.850	11.825	11.883	133342	0.77	77901	1.27	1.71
6	7-Isopropyl-1,1,4a-trimethyl-1,2,3,4	12.159	12.133	12.192	215519	1.25	132601	1.63	2.03
7	Cholestane, 4,5-epoxy-, (4.alpha.,5.a	12.292	12.267	12.317	107691	0.62	22637	0.38	4.76
8	Phytol	12.360	12.292	12.408	371955	2.15	136401	2.29	2.73
9	Ethyl iso-allocholate	12.440	12.408	12.467	151961	0.88	55246	0.89	2.75
10	9-Octadecenoic acid, (E)-	12.593	12.567	12.625	6113746	35.39	2075940	34.87	2.94
11	Octadecanoic acid	12.682	12.625	12.808	2700761	15.03	705270	11.85	3.83
12	9-Octadecenoic acid, 1,2,3-propanetri	12.871	12.808	12.892	431357	1.96	102150	1.72	4.22
13	Tridecanedial	12.908	12.892	12.992	333383	1.92	84385	1.39	3.95
14	2-Dodecen-1-yl(-)succinic anhydride	13.060	12.992	13.108	332539	1.92	66905	1.12	4.97
15	2-Dodecen-1-yl(-)succinic anhydride	13.145	13.108	13.167	231433	1.34	79905	1.34	2.90
16	2-Dodecen-1-yl(-)succinic anhydride	13.183	13.167	13.225	165141	0.96	64685	1.09	2.55
17	2-Dodecen-1-yl(-)succinic anhydride	13.258	13.225	13.283	135912	0.79	51044	0.86	2.66
18	2-Dodecen-1-yl(-)succinic anhydride	13.319	13.283	13.392	223625	1.29	54958	0.92	4.07
19	Silane, tetra-2-propenyl-	13.444	13.392	13.483	191888	1.10	79429	1.34	2.42
20	10,11-Epoxy-undecan-1-ol	13.640	13.483	13.675	265957	1.54	68095	1.14	3.91
21	2-Dodecen-1-yl(-)succinic anhydride	13.716	13.675	13.758	188813	1.09	68119	1.14	2.77
22	2-Dodecen-1-yl(-)succinic anhydride	13.792	13.758	13.858	153664	0.89	33042	0.55	4.65
23	2-Dodecen-1-yl(-)succinic anhydride	13.915	13.858	13.942	190640	1.10	50240	0.84	3.79
24	Pregnane-3,17,20-triol, (3.alpha.,5.b	14.018	13.942	14.108	704885	4.08	244331	4.10	2.88
25	Ethyl iso-allocholate	14.142	14.108	14.275	245195	1.42	43808	0.51	5.60
26	Ergosterol	14.309	14.275	14.408	479539	2.78	181927	3.06	2.64
27	2-Dodecen-1-yl(-)succinic anhydride	14.502	14.408	14.583	116546	0.67	21222	0.36	5.49
28	9-Octadecenoic acid, 1,2,3-propanetri	14.793	14.750	14.842	219738	1.27	102381	1.72	2.15
29	Ethyl iso-allocholate	14.886	14.842	14.908	135669	0.79	52994	0.89	2.56
30	9-Octadecenoic acid, 1,2,3-propanetri	14.925	14.908	14.958	88505	0.51	40813	0.67	2.17
31	Hexadecanoic acid, 2-hydroxy-1-(h	15.008	14.958	15.050	338108	1.96	135265	2.27	2.50
32	Hexadecanoic acid, 3-(3-bromoprop-2-	15.075	15.050	15.117	92956	0.54	35836	0.60	2.58
33	3',8,8'-Trimethoxy-3-piperidyl-2,2'-bi	15.267	15.117	15.308	1409977	0.82	75734	1.27	1.86
					17277437	100.00	5954072	100.00	

Table 10: Gas chromatographic chemical composition of crude ginger.

Pk	Name	R.Time	I.Time	F.Time	Area	Area %	Height	Height %	A/H
1	Pentanedioic acid, (p-t-butylphenyl) e	6.351	6.317	6.425	186363	14.24	102041	1.91	1.83
2	n-Hexadecanoic acid	11.174	11.125	11.250	1644030	10.94	801663	14.98	2.05
3	Naphthalene, decahydro-1,4,4a-trimeth	11.847	11.817	11.883	79150	0.53	51333	0.96	1.54
4	7-Isopropyl-1,1,4a-trimethy-1,2,3,4,4a,5,6,7	12.156	12.133	12.183	124549	0.83	84207	1.57	1.48
5	Phytol	12.356	12.317	12.408	266038	1.77	106429	1.99	2.50
6	Urs-12-en-28-ol	12.440	12.408	12.485	101117	0.67	40779	0.76	2.48
7	Octadec-9-enoic acid	12.535	12.458	12.625	5937553	39.93	2022168	37.79	2.94
8	Octadecanoic acid	12.679	12.625	12.758	2225324	14.81	631049	11.79	3.53
9	2-Methylethyl-2,3.13-octadecadienol	12.775	12.758	12.800	199495	1.33	90523	1.69	2.20
10	1,2-Is,16-Diepoxyoctadecanoic acid	12.869	12.800	12.983	707600	4.71	89733	1.68	7.89
11	2-Dodecen-1-(y)-succinic anhydride	13.057	12.983	13.100	268462	1.79	60720	1.13	4.42
12	Tridecanediol	13.140	13.100	13.167	216473	1.44	67813	1.27	3.19
13	2-Dodecen-1-(y)-succinic anhydride	13.183	13.167	13.225	130885	0.87	53756	10.7	2.28
14	2-Dodecen-1-(y)-succinic anhydride	13.258	13.225	13.275	84990	0.57	36058	0.67	2.36
15	2-Dodecen-1-(y)-succinic anhydride	13.324	13.275	13.367	161924	1.08	44617	0.83	3.63
16	Silane, tetra-2-propenyl-	13.441	13.367	13.483	168784	1.12	67924	1.27	2.48
17	10,11-Epoxy-n-undecan-1-ol	13.637	13.483	13.675	219221	1.46	64542	1.21	3.40
18	2-Dodecen-1-y)-succinic anhydride	13.800	13.675	13.825	135775	0.90	24313	0.45	5.58
19	2-Dodecen-1-y)-succinic anhydride	13.913	13.892	13.933	87019	0.58	48055	0.76	2.14
20	9-Octadecenoc acid, 1,2,3-propanet	13.967	13.933	13.983	82951	0.55	29974	0.56	2.77
21	Pregnane-3,17,20-triol, (3.alpha., 5.b	14.016	13.983	14.117	499163	3.32	223334	4.17	2.24
22	Ergost	14.305	14.267	14.383	361741	2.41	153651	2.87	2.35
23	Ergostan-6-one, 3,25-bis(acetyloxy)-	14.510	14.383	14.600	122945	0.82	25590	0.48	4.80
24	9-Octadecenic acid, 1,2,3-propanet	14.790	14.742	14.842	214284	1.43	99166	1.88	2.16
25	Ethyl iso-allocholate	14.884	14.842	14.908	150568	0.99	56674	1.06	2.65
26	Ethyl iso-allocholate	14.925	14.908	14.958	85135	0.57	38532	0.70	2.21
27	Hexadecanoic acid, 2-hydroxy-1-(hy	15.006	14.958	15.050	335950	2.22	131798	2.25	2.25
28	Ethyl iso-allocholate	15.067	15.050	15.108	79172	0.53	35715	0.67	2.22
29	3',8',8'-Trimethoxy-3-piperidyl-2,2'-bi	15.262	15.108	15.300	145171	0.97	73035	1.36	1.99
					15021832	100.00	53351332	100.00	

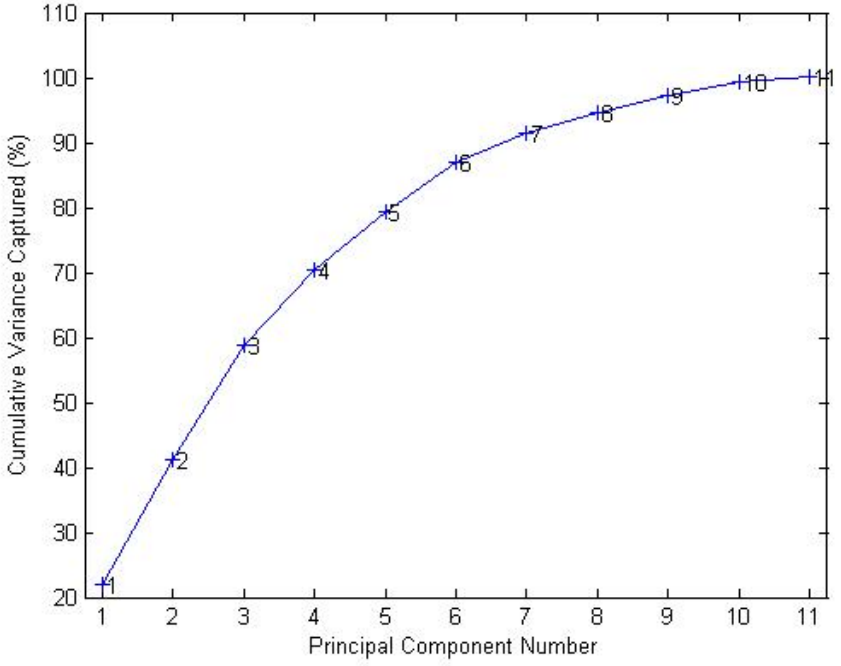

Figure 1: PCA eigenvalue plot of the spice elemental dataset.

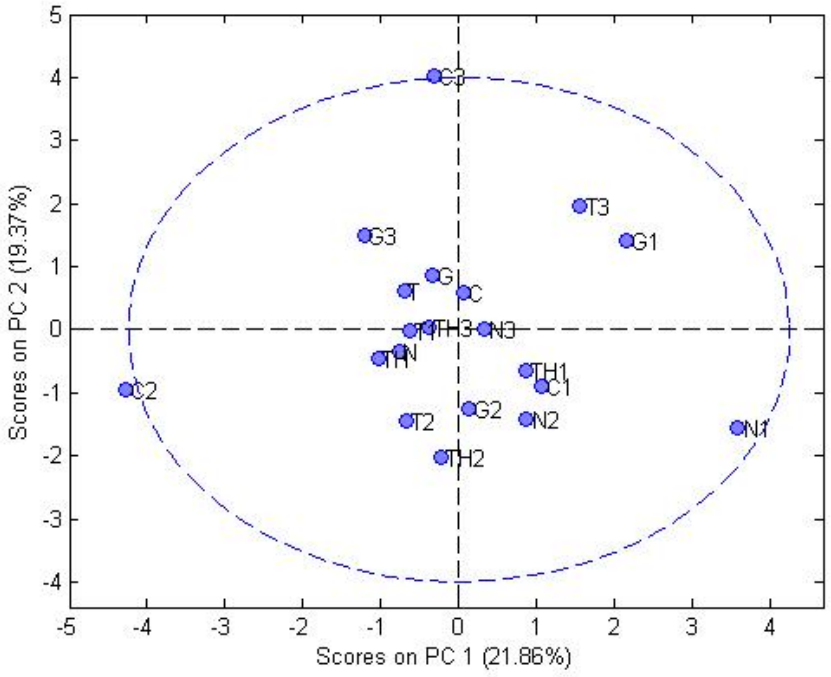

Figure 2: PCA score plot for crude and branded spice elemental dataset.

Key: C = curry crude; BC1 = branded curry 1; BC2 = curry powder; BC3 = Tiger Curry Masala; G = ginger crude; BG1 = branded ginger 1; BG2 = branded ginger 2; BG3 = branded ginger 3; T = turmeric crude; BT1 = branded turmeric 1; BT2 = branded turmeric 2; BT3 = branded turmeric 3; N = nutmeg crude; BN1 = branded nutmeg 1; BN2 = branded nutmeg 2; BN3 = branded nutmeg 3; TH = thyme crude; BTH1 = branded thyme 1; BTH2 = branded thyme 2; BTH3 = branded thyme 3.

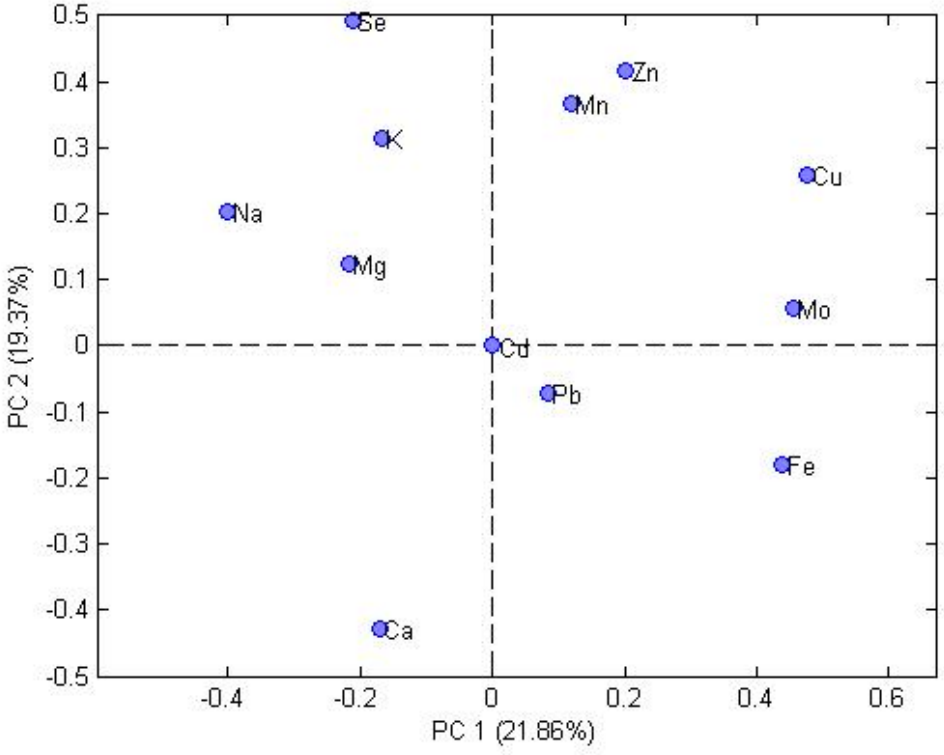

Figure 3: PCA loading plot for crude and branded spice elemental dataset.

Key: Na = sodium; K = potassium; Ca = calcium; Mg = magnesium; Zn = zinc; Mn = manganese; Fe = iron; Cd = cadmium; Se = selenium; Mo = molybdenum; Cu = copper; Pb = lead.

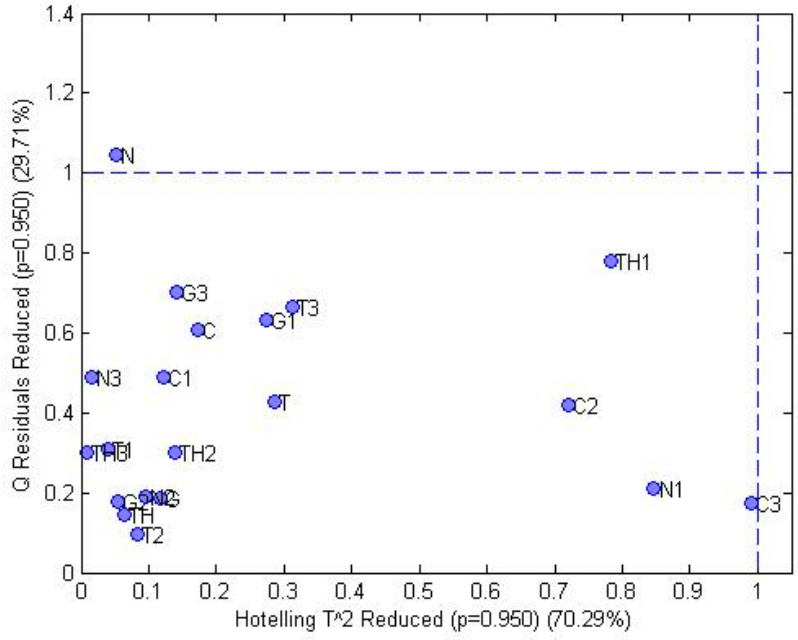

Figure 4: PCA Hotelling's T^2 residual plot for crude and branded spice elemental dataset.

Key: C = curry crude; BC1 = branded curry 1; BC2 = curry powder; BC3 = branded curry; G = ginger crude; BG1 = branded ginger 1; BG2 = branded ginger 2; BG3 = branded ginger 3; T = turmeric crude; BT1 = branded turmeric 1; BT2 = branded turmeric 2; BT3 = branded turmeric 3; N = nutmeg crude; BN1 = branded nutmeg 1; BN2 = branded nutmeg 2; BN3 = branded nutmeg 3; TH = thyme crude; BTH1 = branded thyme 1; BTH2 = branded thyme 2; BTH3 = branded thyme 3.

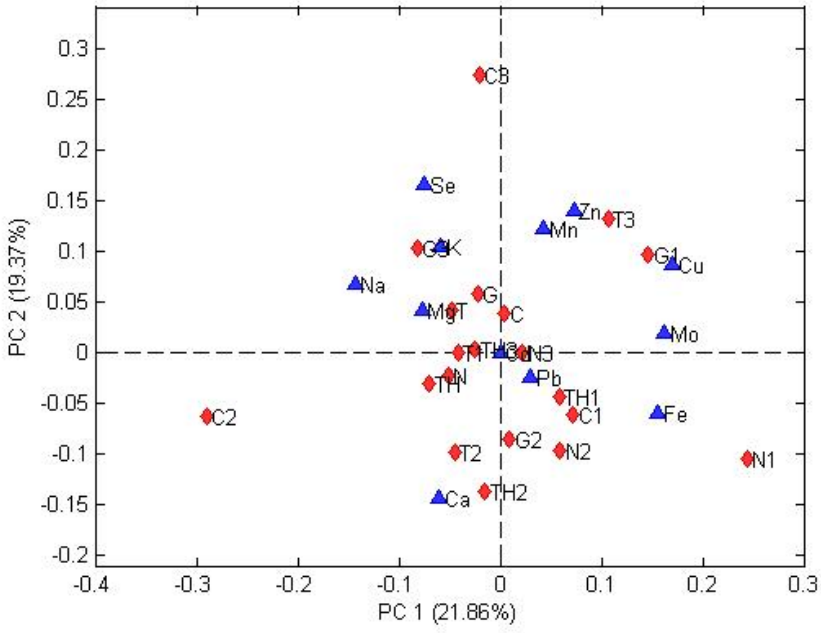

Figure 5: PCA biplot (loading score) for crude and branded spice elemental dataset.

Key: C = curry crude; BC1 = branded curry 1; BC2 = curry powder; BC3 = branded curry; G = ginger crude; BG1 = branded ginger 1; BG2 = branded ginger 2; BG3 = branded ginger 3; T = turmeric crude; BT1 = branded turmeric 1; BT2 = branded turmeric 2; BT3 = branded turmeric 3; N = nutmeg crude; BN1 = branded nutmeg 1; BN2 = branded nutmeg 2; BN3 = branded nutmeg 3; TH = thyme crude; BTH1 = branded thyme 1; BTH2 = branded thyme 2; BTH3 = branded thyme 3.

2.2. Sample collection and experimental design

Five widely consumed spices, turmeric (*Curcuma longa*), nutmeg (*Myristica fragrans*), curry leaves (*Murraya koenigii*),

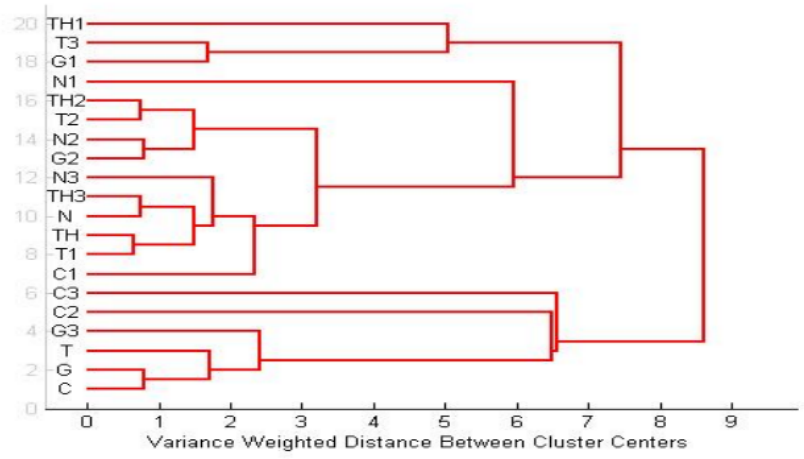

Figure 6: HCA dendrogram for crude and branded spice elemental dataset.

Key: C = curry crude; BC1 = branded curry 1; BC2 = curry powder; BC3 = branded curry; G = ginger crude; BG1 = branded ginger 1; BG2 = branded ginger 2; BG3 = branded ginger 3; T = turmeric crude; BT1 = branded turmeric 1; BT2 = branded turmeric 2; BT3 = branded turmeric 3; N = nutmeg crude; BN1 = branded nutmeg 1; BN2 = branded nutmeg 2; BN3 = branded nutmeg 3; TH = thyme crude; BTH1 = branded thyme 1; BTH2 = branded thyme 2; BTH3 = branded thyme 3.

thyme (*Thymus vulgaris L.*), and ginger (*Zingiber officinale*) were selected based on culinary relevance and reported susceptibility to adulteration and compositional variability. A total of twenty samples per spice were obtained, comprising crude and commercially branded products. Samples were purchased from multiple retail outlets within Kure Market, Minna, Nigeria, to capture market-level variability. Each sample was collected in triplicate, pooled to form a composite, sealed in polyethylene bags, and stored in the laboratory for analysis.

2.3. Sample preparation

Each spice sample was air-dried (where applicable) and ground using a clean ceramic mortar and pestle. The powdered samples were sieved through a stainless-steel laboratory sieve with a mesh size of 250 μm to obtain a uniform particle size distribution. The processed samples were transferred into airtight containers, properly labelled, and stored at room temperature before analysis to minimize moisture absorption and contamination.

2.4. GC–MS chemical fingerprinting

2.4.1. Extraction procedure

Fifty grams (50 g) of each powdered spice sample was extracted with 250 cm^3 of absolute ethanol using the maceration extraction method. The extraction mixture was continuously agitated using an orbital shaker at 150 rpm and maintained at room temperature (25 ± 2 °C) for 24 h to ensure efficient solvent penetration and extraction of volatile constituents.

After extraction, the mixture was filtered through Whatman No. 1 filter paper to separate the solid residues. The filtrate was concentrated under reduced pressure using a rotary evaporator operated at 40 °C until near dryness. The concentrated extract was subsequently reconstituted in 2 mL of analytical-grade ethanol and filtered through a 0.45 μm polytetrafluoroethylene (PTFE) membrane filter into labelled GC–MS vials before instrumental analysis.

2.4.2. GC–MS instrumental conditions

Chemical fingerprinting analysis was performed using a Shimadzu GCMS-QP2010 Plus gas chromatography–mass spectrometry system equipped with an electron impact (EI) ionization source operated at 70 eV. Separation was achieved using an HP-5 MS fused silica capillary column (30 m × 0.25 mm internal diameter × 0.25 μm film thickness).

High-purity helium (99.999%) was used as the carrier gas at a constant flow rate of 1.6–2.5 mL min^{-1}. A 1.0 μL aliquot of each extract was injected in split mode with a split ratio of 25:1. The injector temperature was maintained at 250 °C, while the ion source and interface temperatures were set at 200 °C and 250 °C, respectively. Mass spectra were acquired within the scan range of 45–700 m/z.

The oven temperature program is presented in Table 1. Volatile compounds were tentatively identified by comparison of their mass spectra with entries in the National Institute of Standards and Technology (NIST) Mass Spectral Library. Relative abundances of identified compounds were calculated using percentage peak area normalization and subsequently used for chemometric analysis.

Volatile compounds were identified by matching mass spectra against the NIST Mass Spectral Library. Relative abundances were calculated as percentage peak area normalization, forming the GC–MS fingerprint dataset used for chemometric analysis.

2.5. Acid digestion and AAS analysis

2.5.1. Acid digestion

The acid digestion procedure was carried out following previously reported methods [25, 36] with slight modifications. Briefly, 1.00 g of each powdered spice sample was accurately weighed into a 100 mL borosilicate digestion flask. Thereafter, 10 mL of concentrated nitric acid (HNO3, 65%) was added, and the mixture was allowed to predigest at room temperature for 30 min to minimize excessive frothing during heating.

The digestion flask was subsequently heated on a temperature-controlled hot plate inside a fume hood at 120 °C until the evolution of brown nitrogen oxide fumes significantly subsided. After cooling, 2 mL of hydrogen peroxide (H2O2, 30%) was added dropwise to complete oxidation of residual organic matter. Heating was continued at 120 °C until a clear or pale-yellow digest was obtained.

The digest was cooled to room temperature, filtered through Whatman No. 42 filter paper into a 50 mL volumetric flask, and diluted to volume using deionized water. The resulting solutions were transferred into acid-washed polyethylene bottles and stored at 4 °C prior to elemental analysis.

2.5.2. Atomic absorption spectrometry

Elemental concentrations of calcium (Ca), magnesium (Mg), iron (Fe), manganese (Mn), and zinc (Zn) were determined using a Buck Scientific Atomic Absorption Spectrophotometer (Model 210 VGP or equivalent) equipped with element-specific hollow cathode lamps under manufacturer-recommended operating conditions.

Sodium (Na) and potassium (K) were analyzed separately using a flame photometer because of their high sensitivity in flame emission mode. Instrument calibration was performed using certified single-element standard stock solutions (1000 mg L^{-1}, Merck or equivalent). Appropriate working standards were prepared by serial dilution with deionized water.

Calibration curves were generated by plotting absorbance values (for AAS) or emission intensities (for flame photometry) against standard concentrations. Blank solutions were analyzed alongside samples to correct for background interference. All analyses were carried out in triplicate, and results were expressed as mean ± standard deviation.

2.6. Data preprocessing and chemometric analysis

2.6.1. Data matrix construction

GC–MS peak area percentages and elemental concentration values were combined into a single multivariate data matrix. Before chemometric analysis, variables were autoscaled (mean-centered and scaled to unit variance) to eliminate differences in magnitude and ensure equal weighting of volatile and elemental variables.

2.6.2. Hierarchical cluster analysis (HCA)

Hierarchical Cluster Analysis (HCA) was performed using Ward's linkage method with Euclidean distance as the similarity metric. The analysis was executed using SOLO 8.9 (Eigenvector Research Inc., USA), powered by PLS-Toolbox in MATLAB. Dendrograms were generated to visualize clustering patterns among spice samples and to assess discrimination between crude and branded products. The model shown in Table 2 illustrates the HCA calibration window.

2.6.3. Principal component analysis (PCA)

Principal Component Analysis (PCA) was applied to the autoscaled dataset to reduce dimensionality and identify the principal sources of variance in the integrated chemical fingerprint. Score plots were used to visualize sample distribution, while loading plots identified the chemical and elemental variables contributing to class separation. The model shown in Table 3 illustrates the PCA calibration window.

3. Results

3.1. Elemental composition of crude and branded spices

The quantitative analysis of both macro- and microelements in the selected spice samples revealed significant variability across crude and branded products (Tables 4 and 5), reflecting differences in species, processing, and branded formulation. In Table 4, potassium was the most abundant macroelement, with concentrations ranging from 2.18 mg/g in branded nutmeg powder (BN1) to 7.14 mg/g in crude turmeric (T) and branded spice nutmeg powder (BN3). Crude turmeric (T) and turmeric powders (BT1–BT3) consistently showed high K levels (5.15–7.14 mg/g), whereas branded nutmeg and thyme powders exhibited lower concentrations (2.18–3.07 mg/g). Among curry and ginger samples, K ranged from 4.65 mg/g (BC3, branded curry powder) to 6.90 mg/g (BG3, branded spices Ginger). Sodium concentrations were generally low, between 0.68 mg/g (BTH2, branded thyme leaves) and 1.22 mg/g (BG3, branded spices Ginger; TH, crude thyme). Elevated Na in turmeric crude (T, 0.97 mg/g), curry crude (C, 0.95 mg/g), and branded spices ginger (BG2, 0.87 mg/g) suggest possible contributions from processing or formulation, while branded dried thyme (BTH3, 1.02 mg/g) and crude ginger (G, 1.02 mg/g) had moderate levels. Calcium concentrations varied markedly, from 1.446 mg/g in branded spice turmeric powder (BT3) to 6.463 mg/g in branded thyme leaves (BTH2).

Magnesium was highest in nutmeg crude (N, 5.564 mg/g), crude turmeric (T, 5.242 mg/g), and crude ginger (G, 5.087 mg/g), while the lowest concentrations were noted in branded spice nutmeg powder (BN3, 0.389 mg/g) and branded thyme leaves (BTH1, 0.708 mg/g).

Similarly, in Table 5, Zn levels ranged from 0.002 mg/g in branded curry powder (BC2) to 0.112 mg/g in branded curry powder (BC3). Branded samples such as branded thyme leaves (BTH1, 0.045 mg/g) and branded curry powder (BC1, 0.019 mg/g) also contained elevated Zn, whereas crude ginger (G, 0.013 mg/g) and turmeric (T, 0.011 mg/g) had moderate levels. Fe varied widely, from 0.01 mg/g (BC2, branded curry powder) to 1.164 mg/g (BN1, branded nutmeg powder), which had the highest iron content. Other high-Fe samples included branded spice ginger (BG3, 0.235 mg/g) and branded spice nutmeg powder (BN3, 0.296 mg/g), while crude turmeric (T, 0.122 mg/g) and crude nutmeg (N, 0.126 mg/g) had moderate values. Cu concentrations were generally low, ranging from 0 mg/g (BC2, branded curry powder) to 0.018 mg/g (BG1, branded ginger). Crude turmeric (T, 0.005 mg/g) and crude ginger (G, 0.008 mg/g) contained similarly low Cu levels.

Mn ranged from 0.001 mg/g (BC2, branded curry 2) to 0.197 mg/g (C, crude curry). Other notable Mn concentrations were observed in branded spice turmeric powder (BT3, 0.177 mg/g), branded curry powder (BC3, 0.06 mg/g), and branded

spice nutmeg powder (BN3, 0.037 mg/g). Mo was consistently low across all samples, between 0.003 mg/g (BTH3, BN3) and 0.006 mg/g (BC1, branded curry powder). Se showed the greatest variability. Highest levels were found in branded curry powder (BC3, 7.172 mg/g), branded spice ginger (BG3, 7.631 mg/g), and fresh turmeric (T, 4.064 mg/g). Moderate Se levels were observed in crude ginger (G, 2.542 mg/g) and crude nutmeg (N, 2.619 mg/g), while many branded powders had lower Se content (0.443 mg/g in BN1, branded nutmeg powder; 2.01 mg/g in BT1, branded turmeric powder). Cd and Pb were below detectable limits (0 mg/g) in all samples, indicating negligible toxicological risk. Minor variability may reflect environmental exposure during cultivation, but all values confirm the safety of these spices.

Thus, the highest Zn and Se: BC3 (branded curry 3) and BG3 (branded ginger 3), also the highest Fe: BN1 (branded nutmeg 1, 1.164 mg/g), and the Lowest micronutrients: BC2 (branded curry 2) consistently low across Zn, Fe, Cu, Mn, and Mo. And finally, Toxic metals: Cd and Pb are negligible (< detectable limits). Collectively, the elemental profiles underscore the nutritional potential of fresh and branded spices, particularly as sources of potassium, magnesium, selenium, and iron. Variations highlight the impact of species, processing, and branded formulation on elemental content, emphasizing the need for monitoring and quality control to ensure both nutritional value and safety in commercial spice products.

3.2. Principal component analysis (PCA)

3.2.1. PCA eigenvalue distribution

The eigenvalue plot (Figure 1) summarizes the variance explained by the principal components (PCs) derived from the combined elemental dataset. Two principal components were retained, together accounting for 70.29% of the total variance. This indicates that the majority of the variability in elemental composition among crude and branded spice samples can be effectively captured by these two components. The cumulative variance explained by PC1 and PC2 highlights their dominant contribution to the differentiation among samples, providing a robust basis for subsequent score and loading analyses. Retaining only the most informative PCs avoids overfitting while ensuring that the key patterns underlying sample variability are preserved.

3.2.2. PCA score plot

The PCA score plot (Figure 2) reveals a clear and consistent separation between crude and branded spice samples. Crude samples clustered predominantly on one side of PC1, while branded samples occupied a distinct region, reflecting systematic compositional differences.

The analysis revealed five distinct clusters:

Cluster 1: BC3 (branded curry 3) and BTH3 (branded thyme 3)

These two branded spices cluster closely due to their relatively higher selenium (7.172 mg/g in BC3, 1.971 mg/g in BTH3) and zinc contents (0.112 mg/g in BC3, 0.028 mg/g in BTH3), as well as comparable magnesium levels. This indicates similar mineral enrichment likely from processing practices or sourcing of ingredients.

Cluster 2: BC1 (branded curry 1), BG2 (branded ginger 2), BN2 (branded nutmeg 2), and BTH1 (branded thyme 1)

These branded products cluster together because they have moderate potassium (3.07–6.32 mg/g) and low selenium (0.443–2.01 mg/g), reflecting similar elemental profiles despite belonging to different spice types.

Cluster 3: BT1 (branded turmeric 1), N (Nutmeg crude), BN3 (branded nutmeg), TH (Thyme crude), and BT3 (branded turmeric 3)

This mixture of crude and branded spices shows moderate selenium and iron levels, as well as comparable calcium and magnesium content, explaining their proximity in the PCA space.

Cluster 4: C (Curry crude), G (Ginger crude), BG3 (brand Ginger 3), and T (Turmeric crude)

These samples are predominantly crude spices or minimally processed, showing higher magnesium and potassium content relative to many branded powders, which contributes to their distinct clustering.

Cluster 5: Remaining branded spices (BC2, BT2, BN1, etc.)

These samples exhibit unique elemental fingerprints, such as low zinc or elevated iron, separating them from other clusters.

The PCA score plot demonstrates that samples with similar elemental compositions naturally cluster together, regardless of spice type or brand. Clusters 1 and 2 exemplify branded spices with similar mineral enrichment, whereas clusters 3 and 4 reflect mixtures of crude and branded spices with moderate mineral content. This grouping highlights the potential of elemental composition as a marker for differentiating crude versus processed spices and identifying variations in mineral profiles due to processing, sourcing, or formulation.

3.2.3. PCA loading plot

The PCA loading plot (Figure 3) identifies the variables contributing most strongly to sample discrimination.

The PCA loading plot (Figure 3) reveals the contribution of individual elemental variables to the two principal components (PCs). Among these, PC1 contributes the most information, accounting for 21.86% of the cumulative 70.29% variance captured by the first two principal components. This indicates that PC1.

is the primary driver of differences among the crude and branded spice samples, while PC2 contributes an additional 19.37% of the variance.

3.2.4. Model diagnostics

The Hotelling's T^2 and residual plots (Figure 4) confirm the robustness of the PCA model.

Certain samples (C, BC1, BC2, BC3, G, BG1, BG3, T, BT1, N, BN1, BN3, BTH1, and BTH2; Figure 4) are positioned farther from the model centre and the main cluster of samples in

the PCA space, indicating outlier behavior relative to the majority of samples, which consist primarily of moderately mineralized branded powders such as BT2, BT3, BN2, TH, and BTH3. Their distinct placement reflects elemental compositions that differ markedly from the bulk of samples, likely due to variations in botanical origin, processing methods, or brand-specific formulation practices. The PCA biplot (Figure 5) further integrates score and loading information, visually linking the specific elemental variables (e.g., Se, K, Mg) to the sample groupings, providing insight into which elements drive the observed separation and highlighting potential markers for differentiating crude versus branded spice products.

The PCA biplot (Figure 5) integrates score and loading information, simultaneously linking specific elemental variables to sample groupings. This allows visualization of which elements drive the clustering observed in the score plot. The biplot confirms that the first two PCs together account for 40.86% of total variance, effectively capturing the key variations that differentiate crude and branded spice samples. By representing both samples and elemental variables in the same plot, the biplot facilitates interpretation of which mineral components are most influential in distinguishing sample clusters.

3.3. Hierarchical cluster analysis (HCA)

The hierarchical cluster dendrogram presented in Figure 6 shows distinct clustering patterns that mirror the PCA results. Samples were segregated into two major clusters corresponding to crude and branded spices.

The HCA dendrogram (Figure 6) provides a complementary view of sample similarity based on their nutritional (elemental) profiles, clustering the crude and branded spices into two main groups. The dendrogram illustrates the relative proximity of samples, offering insights into potential substitutability among spices based on nutrient content. Pairs of samples with short linkage distances (0.0–0.7 cm) indicate the highest likelihood of substitution, suggesting that one spice could provide a similar nutritional contribution if the other is unavailable. Notable substitutable pairs include:

C (Curry crude) and G (Ginger crude): Both samples are crude spices with relatively high potassium, magnesium, and selenium content, accounting for their close clustering.

BT1 (branded turmeric 1) and TH (Thyme crude): These samples have moderate mineral content, including calcium and zinc, resulting in similar elemental profiles.

N (Nutmeg crude) and BTH3 (brandedThyme): Both exhibit comparable magnesium and selenium levels, supporting their functional similarity.

BG2 (branded ginger) and BN2 (branded nutmeg 2): Branded products with moderate potassium and sodium content cluster closely.

BT2 (branded turmeric) and BTH2 (branded Thyme): These branded powders share similar calcium and iron concentrations.

BG1 (branded Ginger) and BT3 (branded Turmeric Powder): Both have comparable selenium and manganese levels, explaining their proximity in the dendrogram.

These pairings reinforce the chemometric analysis and demonstrate the practical applications of HCA in nutritional planning, dietary substitution, and quality control. Overall, the dendrogram corroborates the clustering patterns observed in PCA, highlighting functionally similar spices based on their elemental composition.

3.4. Representative GC–MS chemical fingerprints

Representative GC–MS chromatograms of selected spice samples are presented in Table 6–10. These profiles were chosen to illustrate key compositional contrasts between crude and branded products across different spice categories. The selected chromatograms include:

- One crude spice serving as a natural chemical baseline,
- One branded counterpart highlighting formulation-induced alterations,
- Additional representative samples demonstrating variability across spice types.

While all samples were analyzed and incorporated into the chemometric models, only representative GC–MS profiles are shown here to emphasize diagnostically relevant differences. Minor constituents (<3% area) include phenolics such as Thymol (3.22%), Bergamotol, Ledol, and fatty acid derivatives like Ethyl iso-allocholate and 2-dodecen-1-yl-succinic anhydride. The chromatogram demonstrates a chemically complex mixture dominated by saturated and unsaturated fatty acids, terpenoids, phenols, and esters. Variations in peak area-to-height (A/H) ratios provide additional insight into compound concentrations and peak sharpness, supporting detailed chemical characterization.

(0.95%) and Phytol (2.15%), which contribute to the chemical diversity of the sample.

Minor constituents (<2% area) included sterols (e.g., Cholestane, 4,5-epoxy-), esters (e.g., Ethyl iso-allocholate), and various succinic anhydride derivatives, which enhance the overall complexity and may influence the bioactivity or chemical reactivity of the sample. The observed area-to-height (A/H) ratios indicate differences in peak shape and compound concentration, reflecting variability in compound volatility, chromatographic behaviour, or detector response. Overall, branded 3displays a chemically complex profile dominated by fatty acids, supplemented with minor bioactive compounds, typical of natural lipid-rich extracts.

4. Discussion

The elemental analysis of the selected spices revealed significant variations in both macro- and micronutrient contents. Turmeric crude, branded nutmeg 3, and branded thyme 1 exhibited notably higher potassium levels, consistent with previous reports indicating that potassium accumulation in spices is influenced by botanical origin and soil composition [1, 37]. In contrast, branded nutmeg, branded thyme leaves, and nutmeg

crude contained relatively low potassium, highlighting the heterogeneity of mineral content even among closely related spice types. Similarly, sodium concentrations varied widely, with turmeric crude and curry crude samples showing elevated levels, while branded dried thyme and ginger crude contained low sodium. This variation aligns with earlier findings that sodium content in spices is highly affected by both soil salinity and post-harvest processing [18].

Calcium and magnesium levels also displayed marked differences across samples. Tiger thyme leaves and curry crude were enriched in calcium, whereas branded turmeric 3 and branded curry 3 were relatively low. These results corroborate reports that calcium uptake is influenced by soil pH and environmental growing conditions [16, 38]. Magnesium content was highest in nutmeg crude and turmeric crude, suggesting that the presence of magnesium-rich soils or particular agronomic practices significantly affects the elemental profile [15, 25]. The wide range of macronutrient content reflects both natural variability and potential processing effects, consistent with previous analyses of Nigerian spices [13, 19].

Micronutrient profiling revealed substantial heterogeneity. Zinc was highest in branded curry 3, while branded curry 2 and branded ginger 2 had low levels, supporting the notion that zinc concentration is strongly genotype- and soil-dependent [2, 5]. Iron content was particularly elevated in branded nutmeg 1 and branded curry 1, suggesting their potential as dietary sources to address iron deficiency anemia [4, 19]. Copper, manganese, molybdenum, and selenium also showed wide variability. The exceptionally high selenium in branded ginger3 highlights the need for cautious consumption due to potential toxicity, corroborating WHO recommendations on heavy metal intake [12]. Low or non-detectable levels of copper, molybdenum, and selenium in certain samples emphasize the uneven micronutrient contribution of commonly consumed spices [13, 14]. Importantly, toxic elements such as cadmium and lead were either absent or present in minute quantities, aligning with findings that environmental contamination may sporadically affect spice safety [10, 12, 27].

Chemometric analysis further reinforced the nutritional differentiation of crude versus branded spices. Principal component analysis (PCA) showed that two principal components captured 70.29% of cumulative variance, with PC1 alone explaining 21.86%. The score plots revealed five distinct groupings, reflecting compositional similarities and differences among samples. Loading plots indicated that specific elemental variables drove these separations, illustrating the influence of both major and minor minerals in discriminating samples [20, 30]. Outlier samples with unusual leverage and residual distances suggest potential quality variations or adulteration, which is consistent with reports on spice fraud and the need for stringent authentication [8, 9, 37]. Hierarchical cluster analysis (HCA) supported the PCA findings, with two major clusters revealing substitutability patterns among crude and branded spices, offering practical insights for nutritional equivalency in diet formulation [19, 20].

The GC–MS profiling of representative spices (Samples 1–5) demonstrated complex chemical compositions, dominated by fatty acids such as Octadec-9-enoic acid and Octadecanoic acid, while minor constituents, including sterols, phenols, esters, and terpenoids contributed to chemical diversity. For instance, brand Curry 3 (Sample 4) showed 9-Octadecenoic acid as the major component (35.39% area), whereas Ginger Crude (Sample 5) contained a mixture of fatty acids accounting for ~65% of total composition. These findings are consistent with prior GC–MS analyses of spices, which highlight the predominance of lipophilic compounds and the presence of minor bioactive constituents influencing antioxidant and antimicrobial properties [22–24]. The combination of major and minor compounds supports the spices' functional roles in culinary and medicinal applications [3, 6, 7]. Overall, the integration of elemental profiling, chemometric analysis, and GC–MS characterization provides a comprehensive understanding of the nutritional and chemical quality of crude and branded spices. The observed heterogeneity emphasizes the importance of quality control, authentication, and nutritional evaluation to inform safe and effective dietary use, particularly in regions where spices are major contributors to micronutrient intake. These findings corroborate earlier reports on spice mineral variability and chemical complexity [1, 2, 4], while also highlighting potential gaps in standardization and the need for routine monitoring to prevent adulteration or contamination [8–10].

5. Conclusion

The comprehensive analysis of crude and branded spices demonstrates significant variability in both elemental composition and chemical profiles. Macronutrients such as potassium, sodium, calcium, and magnesium showed wide inter-sample differences, reflecting the influence of botanical origin, soil composition, and processing methods. Micronutrients, including zinc, iron, copper, manganese, molybdenum, and selenium, also exhibited marked heterogeneity, with certain samples like branded curry 3 and branded ginger 3 standing out as rich sources of zinc and selenium, respectively. Conversely, some branded products, notably branded curry 2, consistently showed low or non-detectable levels of essential microelements, highlighting the potential nutritional limitations of certain commercially available spices.

Chemometric analyses using PCA and HCA effectively differentiated crude and branded samples based on their elemental composition, revealing distinct clustering patterns and potential substitution relationships among spices. This approach provides a practical framework for evaluating spice quality, authenticity, and nutritional equivalency. GC–MS characterization further identified dominant fatty acids (Octadec-9-enoic acid, Octadecanoic acid) alongside diverse minor bioactive compounds, reinforcing the chemical complexity and functional potential of these spices. Overall, the integrated findings confirm that spice selection can significantly influence dietary mineral intake, bioactive compound exposure, and potentially health outcomes.

5.1. Recommendations

1. Consumers should prioritize spices with higher essential mineral contents, such as branded curry 3 and branded ginger 3, to maximize nutritional benefits, especially in regions prone to micronutrient deficiencies.
2. Routine monitoring of both crude and branded spices is recommended to ensure consistent elemental and chemical profiles, minimizing risks associated with low nutrient content or potential adulteration.
3. Adoption of chemometric tools (PCA, HCA) combined with GC–MS profiling should be incorporated into quality assurance protocols to detect fraudulent or adulterated products.
4. Regulatory agencies should establish stricter standards for allowable ranges of essential and toxic elements in spices, aligning with WHO guidelines on heavy metal intake.

Data availability

The data supporting the findings of this study are available from the corresponding author upon request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this manuscript.

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