Research Square

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Multi-Element Analysis of Commercial Cigarette Tobacco in Türkiye by ICP-MS: Manufacturer-Related Differences and Multivariate Compositional Patterns

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Research Article

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Abstract

Elemental concentrations were determined in 27 commercial cigarette tobacco samples from six manufacturers in Türkiye, with emphasis on manufacturer-related differences and multivariate compositional patterns. Thirteen elements (As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Ti, V, and Zn) were quantified by inductively coupled plasma mass spectrometry (ICP-MS). Given the non-normal distribution of most elemental data, group differences were assessed using the Kruskal–Wallis H test with Dunn–Bonferroni post-hoc comparisons; interelement associations were examined by Spearman correlation, and multivariate structure was explored through PCA and hierarchical cluster analysis (HCA). Mn exhibited the highest mean concentration (135.67 mg/kg), followed by Ba (66.56 mg/kg), Zn (30.30 mg/kg), and Ti (26.55 mg/kg), whereas Cd (0.36 mg/kg) and Pb (0.37 mg/kg) occurred at lower concentrations. Significant manufacturer-related differences were identified for 10 elements ($p < 0.05$). Strong positive correlations were observed between Ti–V ($\rho = 0.965$), Cu–Ti ($\rho = 0.883$), and Cu–V ($\rho = 0.864$). The first two principal components explained 64.61% of the total variance, and HCA identified two major clusters comprising 19 and eight samples. Complete manufacturer-specific separation was not achieved, suggesting that within-manufacturer variability in raw material sourcing and blending may be comparable in scale to between-manufacturer differences. The results confirm that ICP-MS combined with multivariate statistics offers a practical framework for exploratory characterization of commercial tobacco elemental profiles.

1. Introduction

Global tobacco use has been declining for over two decades, yet this trend has not been uniform across all regions. According to the World Health Organization (WHO) [1], the number of tobacco users worldwide decreased from approximately 1.38 billion in 2000 to 1.20 billion in 2024 [2]. Türkiye presents a notable exception: despite a population of approximately 86.1 million [3], the prevalence of tobacco use among individuals aged 15 and older rose from 29.5% in 2010 to 33.2% in 2025 [4]. This epidemiological context makes Türkiye a particularly relevant setting for investigating the chemical composition of locally available cigarette products, including their elemental profiles.

Cigarette tobacco and its combustion smoke contain a complex mixture of chemical constituents, including nicotine, tobacco-specific nitrosamines (TSNAs), polycyclic aromatic hydrocarbons (PAHs), volatile organic compounds (VOCs), and metals and metalloids. Some of these constituents are naturally present in tobacco leaves, while others arise during curing, processing, or combustion [5]. Among these, metals and metalloids warrant particular attention: they accumulate in tobacco leaves through soil uptake and environmental deposition, and several carry well-established toxicological risks, including carcinogenicity, nephrotoxicity, and neurotoxicity [6, 7, 8].

The elemental composition of tobacco reflects a combination of soil chemistry, agricultural inputs, and environmental deposition. Arsenic (As), cadmium (Cd), and lead (Pb) are of primary toxicological concern; chromium (Cr), nickel (Ni), cobalt (Co), and vanadium (V) are also considered potentially harmful constituents [9, 10]. Cadmium in particular is taken up efficiently by tobacco plants from phosphate-

fertilized soils, making it one of the most toxicologically relevant elements in cigarette tobacco [11, 12]. Copper (Cu), manganese (Mn), molybdenum (Mo), and zinc (Zn) are essential micronutrients whose concentrations in tobacco vary with cultivation conditions, while barium (Ba) and titanium (Ti) may serve as indicators of raw material origin or soil provenance [13]. Multi-elemental profiling thus captures both the toxicological and geochemical dimensions of tobacco composition [9].

Several studies have examined the elemental composition of commercial cigarette tobacco in different countries. Musharraf et al. [14] and Pourkhabbaz and Pourkhabbaz [15] reported differences in metal and metalloid concentrations among cigarette brands marketed in Pakistan and Iran, respectively. Hasan et al. [16] also measured metal concentrations in cigarette brands sold in Bangladesh, while Rodrigues et al. [17] investigated elemental, isotopic, and organic markers in Brazilian cigarette products. In China, O'Connor et al. [18] reported differences in elemental concentrations between domestic and imported cigarette brands. More recently, Liu et al. [19] examined potentially toxic elements in tobacco leaves collected from 20 Chinese provinces and identified soil characteristics and agricultural inputs as important factors associated with their accumulation. Regardless of market, both geographic origin and manufacturer-level factors appear to shape the elemental composition of commercial cigarette tobacco.

Elemental composition of cigarette tobacco has also been investigated within Türkiye. Duran et al. [20] determined the concentrations of Co, Ni, Cu, Mn, Pb, Cr, and Cd in 15 commercial cigarette brands by FAAS, and Söğüt & Uruş [21] subsequently analysed metal contents in different cigarette brands using ICP-OES. Ulutaş et al. [22] quantified 14 elements, including Fe, Zn, Mn, Cr, Cu, Ni, Cd, Mo, Co, Pb, As, Hg, and V, in roll-your-own tobacco samples from ten provinces, revealing regional differences attributed to geochemical variability across growing areas. More recently, Cankurt et al. [23] reported uranium and heavy metal concentrations in cigarette tobacco, further documenting the elemental heterogeneity of tobacco products available in Türkiye. In Türkiye, however, manufacturer-level comparisons of cigarette tobacco elemental profiles using ICP-MS combined with multivariate statistical approaches remain limited.

This study determined the concentrations of 13 elements (As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Ti, V, and Zn) in 27 commercial cigarette tobacco samples from six manufacturers in the Turkish retail market using ICP-MS. To our knowledge, it represents the first systematic comparison of elemental profiles across multiple cigarette manufacturers in Türkiye, integrating univariate and multivariate statistical approaches to characterize compositional differences and clustering patterns.

2. Materials and Methods

2.1. Sample Collection

Twenty-seven cigarette brands produced by six tobacco companies operating in the Turkish retail market were included in the study. Samples were purchased from supermarkets and fuel stations in Bingöl Province, Türkiye, and were selected to represent brands with nationwide retail availability. The six companies were designated A–F to maintain anonymity, with seven, four, six, five, three, and two brands from manufacturers A through F, respectively. Each sample was assigned a code comprising the

manufacturer letter, the letter T, and a sequential number (e.g., AT1, BT8); full sample identifications are provided in Table 4.

2.2. Sample Preparation

Five cigarettes were randomly selected from each pack. Tobacco was separated from paper and filters, ground with a mortar and pestle, and mixed to yield a homogeneous composite for moisture determination and elemental analysis.

2.3. Moisture Content Determination

Moisture content of the homogenized samples was determined in triplicate using an infrared moisture analyzer (Denver Instruments, Bohemia, NY, USA) and expressed as mean ± SD.

2.4. Elemental Analysis by ICP-MS

2.4.1. Microwave-Assisted Acid Digestion

Approximately 1.000 g (± 0.001 g) of each homogenized sample was transferred into a PTFE digestion vessel, followed by the addition of 10 mL of concentrated nitric acid (65%, Suprapur grade, Merck). A reagent blank containing the same volume of nitric acid without tobacco was processed under identical conditions. Closed-vessel microwave digestion (MARS 6 One Touch, CEM Corporation, USA) was performed by ramping the temperature to 210°C over 25 min and maintaining it for 15 min. After cooling, the digested samples were quantitatively transferred into 50 mL volumetric flasks and diluted to volume with ultrapure water.

2.4.2. ICP-MS Analysis

Thirteen elements (As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Ti, V, and Zn) were quantified using an ICP-MS system (NexION® 2000 C, PerkinElmer, USA) equipped with a quartz nebulizer, cyclonic spray chamber, and autosampler. Samples were introduced via a peristaltic pump, and helium was employed as the collision gas in kinetic energy discrimination (KED) mode to minimize polyatomic interferences and improve analytical selectivity during elemental determination. External calibration was performed using multi-element standards (PerkinElmer) at concentrations of 0.5, 1, 5, 25, 50, and 100 µg/L, prepared in 1% (v/v) HNO_3 using ultrapure water (18.2 MΩ·cm, Arium Pro, Sartorius). Yttrium (^{89}Y, 25 µg/L) was added to all solutions as an internal standard to compensate for instrumental drift and matrix-induced signal suppression or enhancement, thereby improving the consistency of quantitative measurements. Data acquisition, instrumental calibration, and quantitative processing were performed using Syngistix for ICP-MS software (v2.2). The sample mass, final digestion volume, and corresponding dilution factor were entered into the software to enable automatic calculation of elemental concentrations in mg/kg relative to the original tobacco sample mass. All measurements were performed in triplicate, and the results were expressed as mean ± standard deviation (SD). The detailed ICP-MS operating conditions, including instrumental settings and measurement parameters used throughout the analytical procedure, are presented in Table 1.

Table 1
ICP-MS operating conditions

Parameter	Operating condition
Nebulizer	Meinhard Plus Glass, Type C
Spray chamber	Glass cyclonic (baffled), 4°C
Torch	One-piece quartz torch with 2.5 mm injector
RF power	1600 W
Cones	Nickel (Ni)
Nebulizer gas flow rate	0.93 L/min
Sample uptake rate	350 µL/min
Dwell time	50 ms
Replicates	3
Aerosol dilution	2.5×
Rinse time	45 s

2.4.3. Analytical Quality Control

Linearity was assessed over a six-point calibration range of 0.5–100 µg/L; correlation coefficients (r) ranged from 0.9976 to 0.9997 for all 13 elements (see Table 2).

Table 2
Calibration characteristics of the ICP-MS method for elemental analysis

Element	Calibration equation	Correlation coefficient (r)	Calibration range (µg/L)
As	y = 0.023x + 0.000	0.999560	0.5–100
Ba	y = 0.865x + 0.000	0.998856	0.5–100
Cd	y = 0.098x + 0.000	0.999352	0.5–100
Co	y = 0.480x + 0.000	0.999518	0.5–100
Cr	y = 0.276x + 0.000	0.999456	0.5–100
Cu	y = 0.272x − 0.928	0.997624	0.5–100
Mn	y = 0.070x − 0.053	0.999024	0.5–100
Mo	y = 0.355x + 0.000	0.999472	0.5–100
Ni	y = 0.130x + 0.000	0.999340	0.5–100
Pb	y = 1.869x + 0.000	0.999693	0.5–100
Ti	y = 0.074x + 0.000	0.999315	0.5–100
V	y = 0.220x + 0.000	0.999521	0.5–100
Zn	y = 0.022x − 0.005	0.998099	0.5–100

Post-digestion spike recovery and instrumental precision were assessed using sample AT3. For the recovery test, 6 mL of the digested sample solution was mixed with 6 mL of a multi-element standard solution containing 50 µg/L of each analyte, resulting in a final added concentration of 25 µg/L. Five replicate measurements were performed for each element. Spike recovery was calculated by subtracting the dilution-corrected native analyte concentration from the mean concentration of the spiked solution and dividing the difference by the final added concentration, following the Eurachem guidelines [24], as shown in Eq. (1). Instrumental precision was expressed as the relative standard deviation (RSD) of the five replicate measurements. Spike recoveries ranged from 105.08% to 117.72%, with all values falling within the 80–120% acceptance range specified in Health Canada's T-306 method for fortified tobacco samples [25]. RSD values ranged from 0.51% to 1.60%, indicating good instrumental precision. **These findings support acceptable recovery and instrumental repeatability.** The individual spike recovery and RSD values are presented in Table 3.

$$Recovery\ (\%) = \frac{\overline{C}_{\text{spiked}} - C_{\text{unspiked}}}{C_{\text{added}}} \times\ 100\ \#\ (1)$$

Table 3
Spike recovery and instrumental precision of ICP-MS measurements

Element	Spike recovery (%)	RSD (%)
As	108.30	1.46
Ba	111.52	0.94
Cd	108.13	1.60
Co	105.08	1.21
Cr	105.91	1.26
Cu	117.72	0.58
Mn	116.05	0.51
Mo	108.31	1.06
Ni	106.67	0.91
Pb	108.89	1.49
Ti	112.45	1.39
V	106.21	0.72
Zn	107.98	0.99

Yttrium (^{89}Y) was used as an internal standard to compensate for potential matrix effects and instrumental signal drift during ICP-MS analysis. According to Wilschefski and Baxter [26], variations exceeding 20% in internal standard recovery may indicate potential analytical issues. In the present study, internal standard recoveries ranged from approximately 102% to 118% across all 27 cigarette tobacco samples, remaining within ± 20% of the nominal response and indicating relatively stable instrumental performance (see Fig. 1).

2.5. Statistical Analysis

Statistical analyses were performed in IBM SPSS Statistics v22.0 (IBM Corp., Armonk, NY, USA) using mean values of triplicate measurements for each of the 27 samples. Descriptive statistics (mean, SD, median, min–max, IQR) were calculated for all variables. Normality was assessed with the Shapiro–Wilk test; because most elements showed non-normal distributions, manufacturer-related differences were evaluated using the Kruskal–Wallis H test with Dunn–Bonferroni post-hoc comparisons. Interelement associations were examined by Spearman's rank correlation. For multivariate analyses, PCA was applied to elemental concentration data with the first two components retained for interpretation, and HCA was conducted using Ward's linkage with squared Euclidean distance following Z-score standardization. Statistical significance was set at $p < 0.05$.

3. Results and Discussion

3.1. Moisture Content of Cigarette Tobacco Samples

Moisture content ranged from 7.84% to 10.95% across the 27 samples (mean: 9.57 ± 0.90%; median: 9.65%) (Tables 4 and 5), indicating relatively limited variability among the analyzed samples. Previous studies have reported wider ranges of moisture content. Ajab et al. [27] reported values of 4.90–17.15% for local cigarette brands and 4.48–13.08% for imported brands in Pakistan, while Osei-Owusu et al. [28] reported values of 13.37–15.62%. The moisture contents reported by Osei-Owusu et al. [28] were higher than those observed in the present study, whereas the ranges reported by Ajab et al. [27] were broader and overlapped with the present findings. These differences may be associated with variations in tobacco characteristics, curing methods, and storage conditions, particularly ambient relative humidity, which influences the moisture retention capacity of tobacco [29].

3.2. Elemental Concentrations in Cigarette Tobacco Samples

Elemental concentrations across the 27 samples varied substantially in both absolute levels and within-element dispersion (see Tables 4 and 5). Mn exhibited the highest mean concentration (135.67 ± 40.93 mg/kg; range: 59.55–227.51 mg/kg), followed by Ba (66.56 ± 16.81), Zn (30.30 ± 6.70), Ti (26.55 ± 8.25), and Cu (11.83 ± 2.66 mg/kg). Ni (2.09 ± 0.57), Cr (1.24 ± 0.62), and V (0.77 ± 0.33 mg/kg) occurred at intermediate levels. The lowest mean concentrations were recorded for As (0.48 ± 0.29), Co (0.38 ± 0.13), Mo (0.38 ± 0.23), Pb (0.37 ± 0.25), and Cd (0.36 ± 0.28 mg/kg). Cd showed the most pronounced relative variability (CV ≈ 78%), with a mean nearly 1.5-fold its median (0.25 mg/kg) and a notably elevated concentration in sample ET23 (1.40 mg/kg). Differences between mean and median concentrations were also observed for Ti and Zn, suggesting some degree of positive skewness in their distributions. Overall, mean concentrations followed the order: Mn > Ba > Zn > Ti > Cu > Ni > Cr > V > As > Co ≈ Mo > Pb > Cd.

Duran et al. [20] reported mean concentrations of 66.0, 14.0, 4.1, and 3.7 mg/kg for Mn, Cu, Ni, and Pb, respectively. The mean Mn concentration in the present study (135.67 mg/kg) was approximately twice that reported by Duran et al. [20], whereas the mean concentrations of Cu, Ni, and Pb were lower. Mean concentrations for all four elements fell within the ranges reported by Özcan et al. [30] (Mn: 81.24–254.01; Cu: 10.36–30.47; Ni: 1.00–3.17; Pb: 0.16–7.37 mg/kg). Cankurt et al. [23] reported concentration ranges for As, Cd, Cr, Cu, Ni, Pb, and Zn in Turkish cigarettes whose lower limits exceeded the corresponding mean concentrations in the present study. These comparisons should be interpreted in light of differences in sample selection, preparation procedures, and analytical methods.

Similarities in the concentrations of certain elements were also observed when the present findings were compared with international studies. The mean Mn concentration (135.67 mg/kg) was relatively close to those reported by Musharraf et al. [14] in Pakistan (124.04 mg/kg) and Rubio Armendáriz et al. [31] in Spain (112.03 mg/kg), while Zazouli et al. [32] reported a lower mean Mn concentration (108.64 mg/kg) in Iranian cigarette tobacco. Likewise, the mean Zn concentration in the present study (30.30 mg/kg) was

comparable to those reported by Musharraf et al. [14] (34.27 mg/kg) and Verma et al. [33] (27.00 mg/kg). The mean Cu concentration (11.83 mg/kg) was also relatively close to the values reported by Pourkhabbaz and Pourkhabbaz [15] (9.70 mg/kg) and Zazouli et al. [32] (12.65 mg/kg). These similarities may partly reflect the essential physiological roles of Mn, Zn, and Cu in tobacco plants, although their accumulation is also influenced by soil characteristics, environmental conditions, and agricultural practices.

In contrast, considerable differences were observed in the concentrations of potentially toxic elements, particularly Cd and Pb. The mean Cd concentration in the present study (0.36 mg/kg) was lower than the range of brand-specific mean concentrations reported by Fresquez et al. [34] for cigarette tobacco in the United States (1.0–1.7 mg/kg) and the mean concentrations reported by Janaydeh et al. [35] in Malaysia (0.80 mg/kg) and Pourkhabbaz and Pourkhabbaz [15] in Iran (2.71 mg/kg), but was comparable to the value reported by Chebli et al. [36] in Algeria (0.353 mg/kg). Similarly, the mean Pb concentration (0.37 mg/kg) was substantially lower than those reported by Janaydeh et al. [35] (3.05 mg/kg) and Chebli et al. [36] (10.54 mg/kg), while being relatively close to the value reported by Viana et al. [37] in Brazil (0.27 mg/kg). Furthermore, the mean Cr concentration (1.24 mg/kg) was comparable to those reported by Viana et al. [37] (1.43 mg/kg) and Rubio Armendáriz et al. [31] (1.44 mg/kg), whereas the mean Ni concentration (2.09 mg/kg) was close to the values reported by Chebli et al. [36] (2.35 mg/kg) and Rubio Armendáriz et al. [31] (2.24 mg/kg).

Inter-study comparisons should be interpreted with caution due to differences in brand selection, sample preparation, analytical techniques, and the basis used to express elemental concentrations (dry weight versus as-received weight). Manufacturer-related differences and interelement relationships are examined in the following sections.

3.3. Differences in Elemental Concentrations Among Manufacturers

Significant manufacturer-related differences were identified for 10 of the 13 elements (Ba, Cd, Co, Cr, Cu, Mn, Mo, Ti, V, and Zn; unadjusted $p < 0.05$), whereas As ($H = 5.950$, $p = 0.311$), Ni ($H = 9.053$, $p = 0.107$), and Pb ($H = 2.837$, $p = 0.725$) did not differ significantly across manufacturers (see Table 7).

Mn showed the largest test statistic ($H = 21.498$, $p = 0.001$), with post-hoc comparisons identifying lower concentrations in manufacturer A relative to C (adjusted $p = 0.020$), D (adjusted $p = 0.022$), and E (adjusted $p = 0.002$). Ba concentrations were also lower in manufacturer A than in C (adjusted $p = 0.002$) and D (adjusted $p = 0.013$). For Cr, manufacturers A and D (adjusted $p = 0.037$) and C and D (adjusted $p = 0.017$) differed significantly.

Significant manufacturer-related differences were also observed for Co ($H = 13.779$, $p = 0.017$), Cu ($H = 13.357$, $p = 0.020$), Mo ($H = 13.558$, $p = 0.019$), Ti ($H = 14.789$, $p = 0.011$), and V ($H = 14.562$, $p = 0.012$). Post-hoc comparisons revealed significant differences between manufacturers A and C for Co (adjusted $p = 0.005$), B and C for Cu (adjusted $p = 0.014$) and Ti (adjusted $p = 0.006$), and E and F for Mo (adjusted $p = 0.021$). For V, significant differences were identified between manufacturers B and C (adjusted $p = 0.012$) and C and D (adjusted $p = 0.045$). Zn concentrations also differed significantly among manufacturers ($H =$

11.182, p = 0.048), with a significant pairwise difference between manufacturers B and C (adjusted p = 0.044).

Although the Kruskal–Wallis test indicated overall differences in Cd concentrations (H = 15.309, p = 0.009), none of the pairwise comparisons remained statistically significant after Bonferroni adjustment.

The element-specific nature of these differences — significant for 10 elements but not for As, Ni, or Pb — suggests that manufacturer identity captures some, but not all, of the compositional variability in the dataset. Differences in leaf origin, blending ratios, and curing practices are plausible contributing factors, though these could not be assessed directly.

Manufacturer-related differences in cigarette tobacco elemental composition have also been reported in previous investigations. Rubio Armendáriz et al. [31] identified significant differences among manufacturers only for Mn (p = 0.027) in cigarette tobacco products marketed in Spain, whereas the present study revealed significant differences for ten of the thirteen investigated elements, including Mn. Similarly, Viana et al. [37] reported manufacturer-related differences in the concentrations of Pb, Cd, As, Ni, and Cr in Brazilian cigarette tobacco. Furthermore, Chebli et al. [36] observed higher Pb concentrations in locally manufactured cigarettes than in imported brands in Algeria (12.61 vs. 7.12 mg/kg; p = 0.02). Pourkhabbaz and Pourkhabbaz [15] also reported higher concentrations of Cd, Cu, Ni, and Pb in locally manufactured cigarettes than in imported brands in Iran. These findings indicate that elemental concentrations may vary among cigarette manufacturers and product groups across different markets, although the magnitude and element-specific patterns of variation differ among studies.

The unequal and in some cases small group sizes (A = 7, B = 4, C = 6, D = 5, E = 3, F = 2) limit the statistical power of pairwise comparisons, particularly for manufacturers E and F, and should be taken into account when interpreting the post-hoc results.

Table 4
Elemental concentrations in 27 cigarette tobacco samples (mg/kg; mean ± SD, n = 3).

Manufacturer	Brand	Sample ID	Moisture (%)	As	Ba	Cd	Co	Cr	Cu
A	T1	AT1	8.86 ± 0.18	0.43 ± 0.013	57.69 ± 1.212	0.53 ± 0.015	0.31 ± 0.008	1.33 ± 0.035	10.36 ± 0.198
	T2	AT2	7.84 ± 0.37	0.28 ± 0.017	46.69 ± 1.474	0.13 ± 0.006	0.21 ± 0.010	2.61 ± 0.111	10.07 ± 0.317
	T3	AT3	10.89 ± 0.26	0.22 ± 0.005	45.50 ± 1.148	0.14 ± 0.004	0.21 ± 0.009	1.94 ± 0.074	10.52 ± 0.293
	T4	AT4	10.49 ± 0.23	0.95 ± 0.042	55.54 ± 2.023	0.17 ± 0.005	0.32 ± 0.013	1.20 ± 0.044	13.21 ± 0.320
	T5	AT5	10.15 ± 0.27	0.38 ± 0.012	46.95 ± 1.392	0.16 ± 0.005	0.29 ± 0.012	2.38 ± 0.092	11.54 ± 0.277
	T6	AT6	9.81 ± 0.31	0.33 ± 0.018	50.03 ± 1.183	0.25 ± 0.011	0.25 ± 0.010	1.25 ± 0.047	10.24 ± 0.337
	T7	AT7	10.18 ± 0.13	0.17 ± 0.003	58.05 ± 1.992	0.16 ± 0.007	0.28 ± 0.011	1.24 ± 0.045	9.81 ± 0.289
B	T8	BT8	8.74 ± 0.25	0.32 ± 0.017	58.14 ± 1.622	0.12 ± 0.004	0.41 ± 0.012	0.83 ± 0.023	10.02 ± 0.262
	T9	BT9	8.48 ± 0.14	0.59 ± 0.014	57.27 ± 1.364	0.13 ± 0.006	0.38 ± 0.016	0.82 ± 0.033	9.07 ± 0.220
	T10	BT10	9.65 ± 0.30	0.97 ± 0.036	54.15 ± 1.751	0.16 ± 0.005	0.35 ± 0.016	0.71 ± 0.029	9.38 ± 0.190
	T11	BT11	9.59 ± 0.29	0.46 ± 0.012	58.16 ± 1.528	0.31 ± 0.009	0.38 ± 0.014	0.92 ± 0.031	10.58 ± 0.333
C	T12	CT12	10.94 ± 0.17	0.86 ± 0.052	66.95 ± 1.470	0.37 ± 0.010	0.37 ± 0.011	1.35 ± 0.038	11.50 ± 0.332
	T13	CT13	10.95 ± 0.28	0.12 ± 0.006	93.51 ± 2.857	0.47 ± 0.014	0.61 ± 0.024	2.47 ± 0.090	14.25 ± 0.356

Manufacturer	Brand	Sample ID	Moisture (%)	As	Ba	Cd	Co	Cr	Cu
	T14	CT14	10.20 ± 0.23	0.95 ± 0.037	75.03 ± 1.997	0.31 ± 0.014	0.45 ± 0.021	1.93 ± 0.080	16.04 ± 0.340
	T15	CT15	10.76 ± 0.10	0.98 ± 0.055	82.26 ± 2.883	0.76 ± 0.032	0.65 ± 0.015	1.75 ± 0.041	16.98 ± 0.571
	T16	CT16	9.69 ± 0.16	0.54 ± 0.016	88.82 ± 2.567	0.45 ± 0.017	0.57 ± 0.024	1.45 ± 0.055	17.56 ± 0.524
	T17	CT17	10.62 ± 0.16	0.14 ± 0.003	84.25 ± 2.081	0.55 ± 0.019	0.53 ± 0.013	1.68 ± 0.040	16.60 ± 0.434
D	T18	DT18	8.82 ± 0.25	0.39 ± 0.007	107.80 ± 3.580	0.43 ± 0.019	0.63 ± 0.016	0.86 ± 0.021	14.52 ± 0.327
	T19	DT19	9.10 ± 0.29	0.14 ± 0.008	103.57 ± 3.047	0.45 ± 0.012	0.57 ± 0.023	1.18 ± 0.044	15.34 ± 0.354
	T20	DT20	9.91 ± 0.21	0.38 ± 0.011	60.21 ± 1.379	0.19 ± 0.008	0.20 ± 0.007	0.41 ± 0.013	9.33 ± 0.292
	T21	DT21	9.02 ± 0.25	0.82 ± 0.020	68.97 ± 2.174	0.22 ± 0.008	0.30 ± 0.010	0.58 ± 0.018	9.96 ± 0.276
	T22	DT22	9.84 ± 0.26	0.45 ± 0.021	66.17 ± 1.816	0.20 ± 0.008	0.23 ± 0.007	0.50 ± 0.015	8.99 ± 0.219
E	T23	ET23	8.01 ± 0.18	0.91 ± 0.016	52.75 ± 1.121	1.40 ± 0.041	0.41 ± 0.015	1.17 ± 0.041	10.96 ± 0.253
	T24	ET24	9.19 ± 0.22	0.46 ± 0.016	65.22 ± 2.101	0.74 ± 0.019	0.34 ± 0.008	0.52 ± 0.012	10.76 ± 0.354
	T25	ET25	8.52 ± 0.18	0.42 ± 0.008	63.02 ± 1.614	0.41 ± 0.012	0.31 ± 0.011	0.65 ± 0.022	10.28 ± 0.305
F	T26	FT26	9.41 ± 0.25	0.14 ± 0.008	62.55 ± 2.136	0.23 ± 0.010	0.40 ± 0.013	0.87 ± 0.028	10.83 ± 0.298
	T27	FT27	8.86 ± 0.23	0.19 ± 0.008	67.96 ± 2.052	0.20 ± 0.008	0.40 ± 0.017	0.78 ± 0.031	10.71 ± 0.252

Table 4
(continued)

Manufacturer	Brand	Sample ID	Mn	Mo	Ni	Pb	Ti	V	Zn
A	T1	AT1	105.02 ± 2.170	0.23 ± 0.006	1.64 ± 0.036	0.21 ± 0.006	24.26 ± 0.734	0.69 ± 0.015	27.16 ± 0.424
	T2	AT2	93.11 ± 2.684	0.54 ± 0.014	1.95 ± 0.048	0.34 ± 0.010	18.88 ± 0.383	0.47 ± 0.011	22.93 ± 0.417
	T3	AT3	59.55 ± 1.294	0.31 ± 0.008	3.03 ± 0.083	0.18 ± 0.005	30.12 ± 0.531	1.04 ± 0.028	45.76 ± 0.951
	T4	AT4	100.60 ± 3.013	0.15 ± 0.006	2.02 ± 0.060	0.97 ± 0.027	22.81 ± 0.577	0.72 ± 0.022	27.61 ± 0.646
	T5	AT5	72.45 ± 1.654	0.23 ± 0.007	2.48 ± 0.080	0.47 ± 0.013	37.47 ± 0.689	1.51 ± 0.050	25.93 ± 0.698
	T6	AT6	90.74 ± 2.822	0.19 ± 0.004	1.75 ± 0.039	0.23 ± 0.006	20.83 ± 0.507	0.68 ± 0.015	25.11 ± 0.392
	T7	AT7	97.41 ± 2.332	0.29 ± 0.011	1.66 ± 0.041	0.92 ± 0.026	22.26 ± 0.402	0.64 ± 0.016	29.07 ± 0.530
B	T8	BT8	122.20 ± 3.941	0.43 ± 0.013	2.15 ± 0.060	0.42 ± 0.012	19.61 ± 0.378	0.56 ± 0.015	24.67 ± 0.515
	T9	BT9	113.32 ± 2.841	0.27 ± 0.006	2.05 ± 0.060	0.33 ± 0.009	17.20 ± 0.421	0.45 ± 0.014	23.22 ± 0.558
	T10	BT10	114.98 ± 4.114	0.52 ± 0.011	1.80 ± 0.058	0.25 ± 0.007	17.37 ± 0.326	0.41 ± 0.014	23.88 ± 0.631
	T11	BT11	121.71 ± 3.190	0.10 ± 0.004	1.79 ± 0.041	0.57 ± 0.016	21.65 ± 0.388	0.52 ± 0.011	29.12 ± 0.462
C	T12	CT12	129.03 ± 4.744	0.32 ± 0.009	1.97 ± 0.050	0.10 ± 0.003	30.80 ± 0.786	0.84 ± 0.020	29.23 ± 0.541
	T13	CT13	172.71 ± 4.727	0.52 ± 0.014	3.17 ± 0.085	0.18 ± 0.005	45.66 ± 0.882	1.36 ± 0.038	35.38 ± 0.749

Manufacturer	Brand	Sample ID	Mn	Mo	Ni	Pb	Ti	V	Zn
	T14	CT14	126.88 ± 2.562	0.40 ± 0.015	2.83 ± 0.084	0.74 ± 0.021	37.02 ± 0.708	1.25 ± 0.038	39.07 ± 0.924
	T15	CT15	168.65 ± 4.812	0.44 ± 0.010	2.86 ± 0.092	0.28 ± 0.008	38.49 ± 0.893	1.20 ± 0.040	41.52 ± 1.086
	T16	CT16	160.22 ± 3.417	0.45 ± 0.014	2.61 ± 0.062	0.25 ± 0.007	35.80 ± 0.646	1.16 ± 0.026	42.75 ± 0.700
	T17	CT17	159.84 ± 4.748	0.30 ± 0.009	2.89 ± 0.070	0.12 ± 0.004	38.51 ± 1.265	1.18 ± 0.029	36.87 ± 0.677
D	T18	DT18	214.67 ± 4.826	0.28 ± 0.006	2.01 ± 0.054	0.43 ± 0.012	30.70 ± 0.874	0.73 ± 0.020	36.81 ± 0.768
	T19	DT19	182.11 ± 6.011	0.29 ± 0.011	2.63 ± 0.078	0.12 ± 0.003	35.43 ± 0.613	0.91 ± 0.028	38.28 ± 0.897
	T20	DT20	135.06 ± 3.194	0.19 ± 0.008	1.21 ± 0.039	0.27 ± 0.007	15.84 ± 0.283	0.29 ± 0.010	23.81 ± 0.619
	T21	DT21	120.60 ± 4.102	0.65 ± 0.028	1.52 ± 0.033	0.48 ± 0.013	21.51 ± 0.557	0.47 ± 0.010	26.06 ± 0.407
	T22	DT22	141.32 ± 3.509	0.39 ± 0.013	1.23 ± 0.030	0.40 ± 0.011	18.45 ± 0.431	0.33 ± 0.008	24.27 ± 0.442
E	T23	ET23	227.51 ± 7.969	0.98 ± 0.024	2.37 ± 0.065	0.66 ± 0.019	28.30 ± 0.739	0.79 ± 0.022	32.09 ± 0.667
	T24	ET24	179.78 ± 4.676	0.99 ± 0.023	1.07 ± 0.032	0.11 ± 0.003	19.30 ± 0.654	0.50 ± 0.015	27.52 ± 0.644
	T25	ET25	182.46 ± 6.580	0.62 ± 0.023	2.15 ± 0.069	0.17 ± 0.005	21.57 ± 0.651	0.61 ± 0.021	28.06 ± 0.755
F	T26	FT26	134.56 ± 3.661	0.15 ± 0.005	1.97 ± 0.044	0.10 ± 0.003	23.53 ± 0.673	0.74 ± 0.016	25.81 ± 0.404
	T27	FT27	136.61 ± 2.725	0.10 ± 0.003	1.73 ± 0.043	0.72 ± 0.020	23.44 ± 0.596	0.68 ± 0.017	26.01 ± 0.474

Table 5
Descriptive statistics of elemental concentrations and moisture content in cigarette tobacco samples

Parameter	Mean ± SD	Median	Min–Max	IQR
As	0.48 ± 0.29	0.42	0.12–0.98	0.60
Ba	66.56 ± 16.81	62.55	45.50–107.80	19.49
Cd	0.36 ± 0.28	0.25	0.12–1.40	0.29
Co	0.38 ± 0.13	0.37	0.20–0.65	0.16
Cr	1.24 ± 0.62	1.18	0.41–2.61	0.90
Cu	11.83 ± 2.66	10.71	8.99–17.56	4.23
Mn	135.67 ± 40.93	129.03	59.55–227.51	63.63
Mo	0.38 ± 0.23	0.31	0.10–0.99	0.29
Ni	2.09 ± 0.57	2.01	1.07–3.17	0.88
Pb	0.37 ± 0.25	0.28	0.10–0.97	0.30
Ti	26.55 ± 8.25	23.44	15.84–45.66	15.82
V	0.77 ± 0.33	0.69	0.29–1.51	0.54
Zn	30.30 ± 6.70	27.61	22.93–45.76	11.70
Moisture (%)	9.57 ± 0.90	9.65	7.84–10.95	1.34

Note: SD, standard deviation; IQR, interquartile range. Elemental concentrations are expressed in mg/kg and moisture in %. Descriptive statistics are based on 27 samples (n = 27).

The Shapiro–Wilk test indicated significant deviations from normality for nine elements (As, Ba, Cd, Cr, Cu, Mo, Pb, Ti, and Zn; $p < 0.05$). In contrast, Co, Mn, Ni, V, and moisture content did not show statistically significant deviations from normality ($p > 0.05$). Given the non-normal distributions observed for most elements, the Kruskal–Wallis H test was selected to compare elemental concentrations among manufacturers.

Table 6
Shapiro–Wilk normality test results for elemental concentrations and moisture content in cigarette tobacco samples (n = 27)

Parameter	Shapiro–Wilk	df	p-value
As	0.878	27	0.004
Ba	0.895	27	0.010
Cd	0.759	27	< 0.001
Co	0.926	27	0.056
Cr	0.922	27	0.045
Cu	0.828	27	< 0.001
Mn	0.976	27	0.774
Mo	0.882	27	0.005
Ni	0.965	27	0.488
Pb	0.888	27	0.007
Ti	0.905	27	0.018
V	0.935	27	0.092
Zn	0.869	27	0.003
Moisture	0.965	27	0.474
Note: Statistical significance was set at $p < 0.05$ (n = 27).			

Table 7
Kruskal–Wallis H test and Dunn–Bonferroni post-hoc results for elemental concentrations across six cigarette manufacturers

Element	H	df	p-value	Significant pairwise comparisons (adjusted p-value)
As	5.950	5	0.311	–
Ba	20.347	5	**0.001**	A–C (0.002); A–D (0.013)

Table 7
(continued)

Element	H	df	p-value	Significant pairwise comparisons (adjusted p-value)
Cd	15.309	5	**0.009**	None
Co	13.779	5	**0.017**	A–C (0.005)
Cr	19.939	5	**0.001**	A–D (0.037); C–D (0.017)
Cu	13.357	5	**0.020**	B–C (0.014)
Mn	21.498	5	**0.001**	A–C (0.020); A–D (0.022); A–E (0.002)
Mo	13.558	5	**0.019**	E–F (0.021)
Ni	9.053	5	0.107	–
Pb	2.837	5	0.725	–
Ti	14.789	5	**0.011**	B–C (0.006)
V	14.562	5	**0.012**	B–C (0.012); C–D (0.045)
Zn	11.182	5	**0.048**	B–C (0.044)

Note: H, Kruskal–Wallis test statistic; df, degrees of freedom. Bold p-values indicate significant omnibus tests ($p < 0.05$), unadjusted across 13 elements. Only significant Dunn–Bonferroni pairwise comparisons are reported. "–", not performed; "None", no significant adjusted pairwise comparisons ($n = 27$).

3.4. Spearman Correlation Analysis

Spearman correlation analysis revealed several significant positive associations among the 13 elements (see Fig. 2). The strongest correlation was Ti–V ($\rho = 0.965$, $p < 0.001$), followed by Cu–Ti ($\rho = 0.883$), Cu–V ($\rho = 0.864$), Ti–Zn ($\rho = 0.805$), Ni–V ($\rho = 0.785$), and Cu–Zn ($\rho = 0.780$; all $p < 0.001$). The tight co-variation among Ti, V, Cu, Ni, and Zn across samples from multiple manufacturers points to a shared accumulation pathway — possibly lithogenic soil inputs or a common mineral fertilizer source rather than manufacturer-specific practices.

A second correlation cluster emerged among Ba, Cd, Co, and Mn (Ba–Mn: $\rho = 0.719$; Cd–Mn: $\rho = 0.717$; Ba–Co: $\rho = 0.689$), suggesting a partially shared accumulation pattern distinct from the Ti–V–Cu group. The co-variation of Cd with Mn is particularly notable given that both elements are known to accumulate under acidic soil conditions.

In contrast, As, Mo, and Pb did not show statistically significant correlations with any other element, suggesting that their concentrations were governed by independent sources or uptake mechanisms not captured by the remaining elemental profile.

Previous studies have also reported associations among elemental concentrations in cigarette tobacco. Viana et al. [37] identified strong positive correlations between Ni and Cr (r = 0.829) and between Ni and As (r = 0.799) in Brazilian cigarette tobacco. Similarly, O'Connor et al. [18] reported a strong positive association between As and Pb (Spearman's ρ = 0.801) in cigarette tobacco samples collected in China. In contrast, the strongest correlations in the present study were observed for Ti–V (ρ = 0.965), Cu–Ti (ρ = 0.883), and Cu–V (ρ = 0.864), whereas As and Pb did not exhibit statistically significant correlations with the other investigated elements. These findings suggest that interelement correlation patterns may differ among cigarette tobacco products investigated in different geographical markets. Nevertheless, differences in sample characteristics, analytical methods, and statistical approaches should be considered when interpreting these comparisons.

These correlation clusters suggest that elemental concentrations in cigarette tobacco are not independent, but reflect at least two partially distinct accumulation patterns. Disentangling their origins, whether pedogenic, agronomic, or process-related, would require isotopic tracing or provenance data beyond the scope of the present study.

3.5. Principal Component Analysis (PCA)

PCA of the 13-element dataset yielded two interpretable components explaining 64.61% of total variance (PC1: 45.14%, eigenvalue = 5.869; PC2: 19.46%, eigenvalue = 2.530) (see Table 8).

PC1 was dominated by Ti (0.944), Cu (0.916), Zn (0.847), Co (0.844), V (0.841), Ni (0.835), and Ba (0.718), consistent with the Ti–V–Cu–Ni–Zn correlation cluster identified in the Spearman analysis. PC2 was characterized by positive loadings for Mn (0.816), Cd (0.655), and Mo (0.651), and a notable negative loading for Cr (− 0.619), indicating that samples with high Mn and Cd tended to have lower Cr concentrations along this axis (see Table 9). The PCA loading structure thus mirrored the two correlation clusters identified earlier: the Ti–V–Cu–Ni–Zn group on PC1, and the Mn–Cd axis on PC2.

In the biplot (Fig. 3), manufacturer C samples clustered predominantly on the positive PC1 side, consistent with their higher Cu, Ti, and Zn concentrations, while manufacturer A, B, and D samples were more dispersed toward the negative PC1 region.

Manufacturer E samples, particularly ET23 and ET24, showed elevated PC2 scores associated with high Mn and Cd concentrations. Sample ET23 had the highest Cd (1.40 mg/kg) and Mn (227.51 mg/kg) concentrations in the dataset and appeared as a clear outlier along the PC2 axis.

Despite these tendencies, samples from different manufacturers overlapped considerably in the PC1–PC2 space, confirming that elemental profiles alone are insufficient for definitive manufacturer classification in this dataset. The partial separation observed, particularly for manufacturer C and the ET23 outlier, nonetheless demonstrates that ICP-MS-based elemental profiling can reveal meaningful compositional structure when combined with multivariate analysis.

Table 8
Eigenvalues and explained variance of the first two principal components obtained from PCA of elemental concentrations in cigarette tobacco samples (n = 27).

Principal Component	Eigenvalue	Explained Variance (%)	Cumulative Variance (%)
PC1	5.869	45.144	45.144
PC2	2.530	19.462	64.606

Table 9
Component loadings for the first two principal components obtained from PCA of elemental concentrations in cigarette tobacco samples (n = 27).

Element	PC1 (45.144%)	PC2 (19.462%)
As	0.007	0.327
Ba	0.718	0.312
Cd	0.475	0.655
Co	0.844	0.265
Cr	0.538	-0.619
Cu	0.916	-0.009
Mn	0.480	0.816
Mo	0.107	0.651
Ni	0.835	-0.353
Pb	-0.180	-0.118
Ti	0.944	-0.217
V	0.841	-0.405
Zn	0.847	-0.073
Note: PCA extraction; two unrotated components explaining 64.606% of total variance.		

3.6. Hierarchical Cluster Analysis (HCA)

HCA yielded two major clusters comprising 19 and eight samples, respectively (Fig. 4).

Cluster 2 comprised CT13–CT17 (manufacturer C), DT18 and DT19 (manufacturer D), and ET23 (manufacturer E), while the remaining 19 samples formed Cluster 1.

The assignment of CT12 to Cluster 1, despite its origin from manufacturer C, illustrates the within-manufacturer heterogeneity noted earlier and suggests that CT12 may differ from the other C-brand products in raw material composition or blend ratio. The presence of samples from multiple manufacturers within each cluster indicates that elemental composition does not correspond directly to

manufacturer identity. The clustering of high-Mn, high-Ba samples from manufacturers C and D into Cluster 2 suggests a shared raw material or growing region, though this cannot be confirmed without provenance data. Taken together, PCA and HCA converge on a consistent picture: commercial cigarette tobacco in Türkiye shows structured elemental variability that is partly but not fully associated with manufacturer identity.

4. Conclusion

ICP-MS analysis of 27 commercial cigarette tobacco samples from six manufacturers in Türkiye revealed a wide range of elemental concentrations, with Mn consistently dominating the elemental profile (mean: 135.67 mg/kg) and Cd, Pb, Co, and Mo occurring at comparatively low but variable levels.

Manufacturer identity was associated with significant differences in 10 of the 13 elements. However, post-hoc comparisons and multivariate analyses revealed considerable within-manufacturer variability, suggesting that raw material sourcing and blending practices may be as important as manufacturer identity in shaping elemental profiles. PCA and HCA revealed broadly consistent compositional patterns, with most samples from manufacturer C tending to cluster together. Sample ET23 was also distinguished by its exceptionally high Mn and Cd concentrations. Nevertheless, complete manufacturer-specific separation was not achieved, indicating that manufacturer identity alone cannot fully explain the observed variability in elemental composition.

Cd and Pb showed the greatest inter-study variability, likely reflecting differences in tobacco-growing region and soil contamination history; concentrations of most other elements fell within previously reported international ranges.

The unequal group sizes, particularly the inclusion of only two brands from manufacturer F, limit the statistical power of pairwise comparisons. Therefore, manufacturer-level differences should be interpreted with caution. Critically, the concentrations reported here reflect unburned tobacco; transfer efficiencies into mainstream smoke vary considerably by element and cigarette design, and cannot be inferred from the present data. Future work should prioritize isotopic or multi-marker tracing to link elemental profiles to specific growing regions, and should extend analysis to mainstream smoke to quantify element-specific transfer rates particularly for Cd and Mn, which showed the greatest variability in the present dataset.

The present dataset demonstrates that commercially available cigarette tobacco in Türkiye is elementally heterogeneous at both the manufacturer and brand level. This finding has direct implications for exposure monitoring programmes that rely on brand-level sampling strategies.

Declarations

Authorship Statement

The author declares that this manuscript is original, has not been published previously, and is not under consideration for publication elsewhere.

Declaration of Competing Interest

The author declares no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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Author Contribution

The sole author was responsible for the study conception and design, sample collection, data analysis and interpretation, statistical analysis, and manuscript preparation. The author reviewed and approved the final version of the manuscript.

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Data Availability

Data will be made available upon request.

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Figures

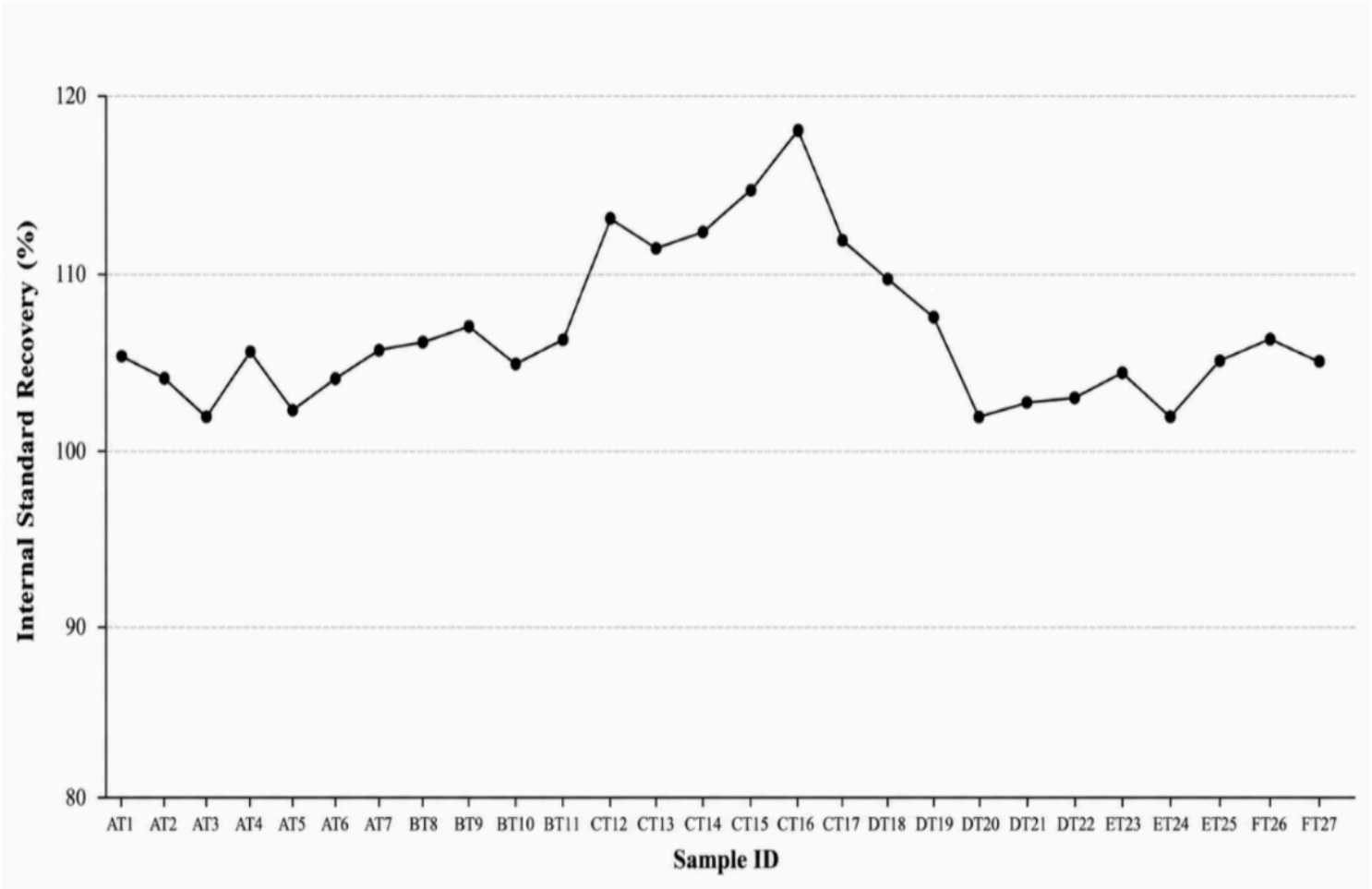


Figure 1

Internal standard (^{89}Y) recovery across 27 cigarette tobacco samples analyzed by ICP-MS

ELEMENT	As	Ba	Cd	Co	Cr	Cu	Mn	Mo	Ni	Pb	Ti	V	Zn
As	1.000												
Ba	-0.078	1.000											
Cd	0.126	**0.570****	1.000										
Co	0.063	**0.689****	**0.558****	1.000									
Cr	-0.174	-0.087	0.101	0.182	1.000								
Cu	0.019	**0.532****	**0.632****	**0.720****	**0.559****	1.000							
Mn	0.022	**0.719****	**0.717****	**0.635****	-0.299	**0.419***	1.000						
Mo	0.275	0.128	0.261	0.105	0.007	0.019	0.359	1.000					
Ni	-0.107	0.198	0.196	**0.568****	**0.652****	**0.656****	0.176	0.167	1.000				
Pb	0.300	-0.187	-0.340	-0.069	-0.046	-0.132	-0.243	-0.169	-0.136	1.000			
Ti	-0.145	**0.463***	**0.544****	**0.612****	**0.698****	**0.883****	0.265	-0.003	**0.729****	0.120	1.000		
V	-0.142	0.303	**0.443***	**0.542****	**0.756****	**0.864****	0.154	-0.023	**0.785****	-0.131	**0.965****	1.000	
Zn	0.038	**0.498****	**0.600****	**0.557****	**0.465***	**0.780****	**0.383***	0.158	**0.634****	-0.100	**0.805****	**0.750****	1.000

Figure 2

Spearman’s rank correlation coefficients (ρ) among elemental concentrations in cigarette tobacco samples (n = 27). Note: Spearman’s ρ; *p < 0.05, **p < 0.01 (two-tailed, unadjusted). Only the lower triangular matrix is shown.

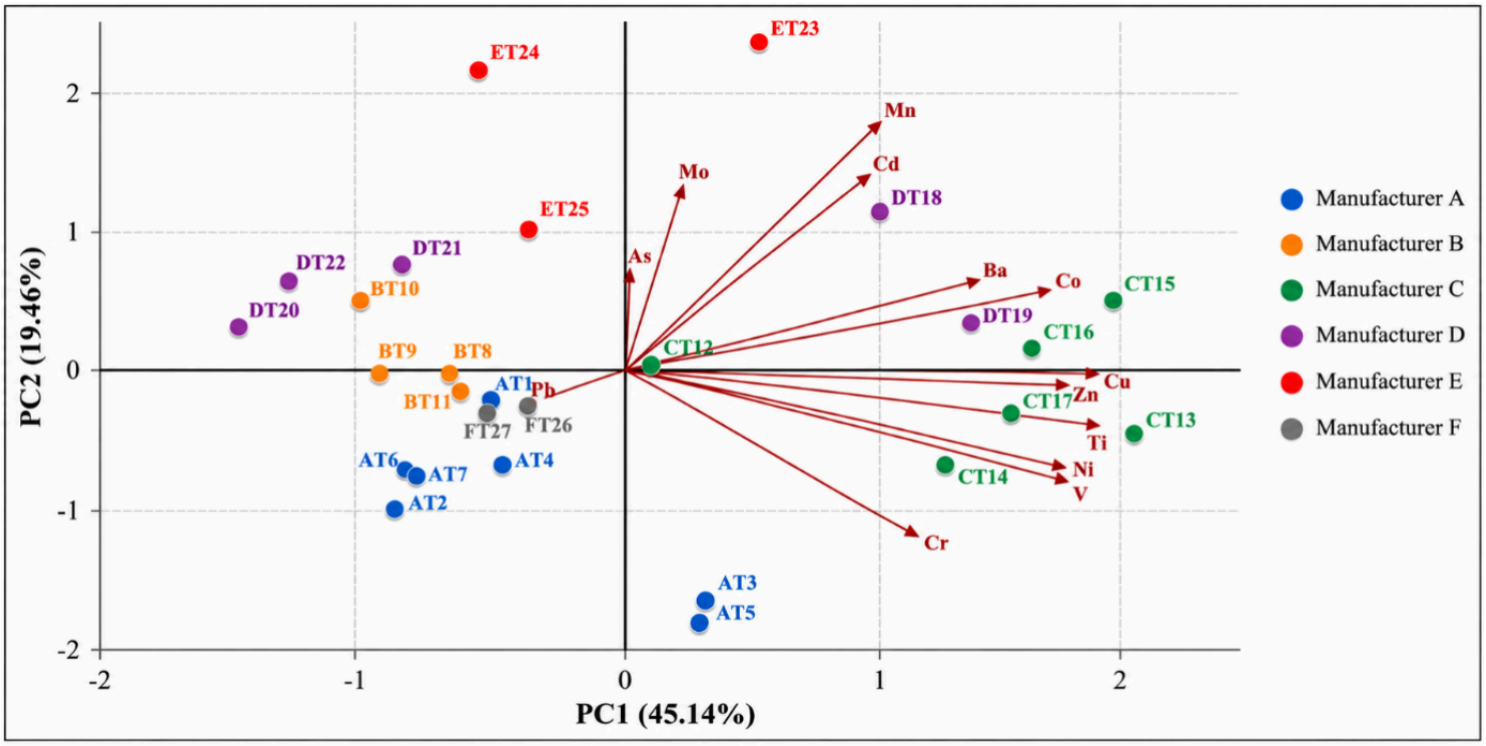


Figure 3

Principal Component Analysis (PCA) Biplot of Elemental Concentrations in Cigarette Tobacco Samples from Six Manufacturers (A–F).

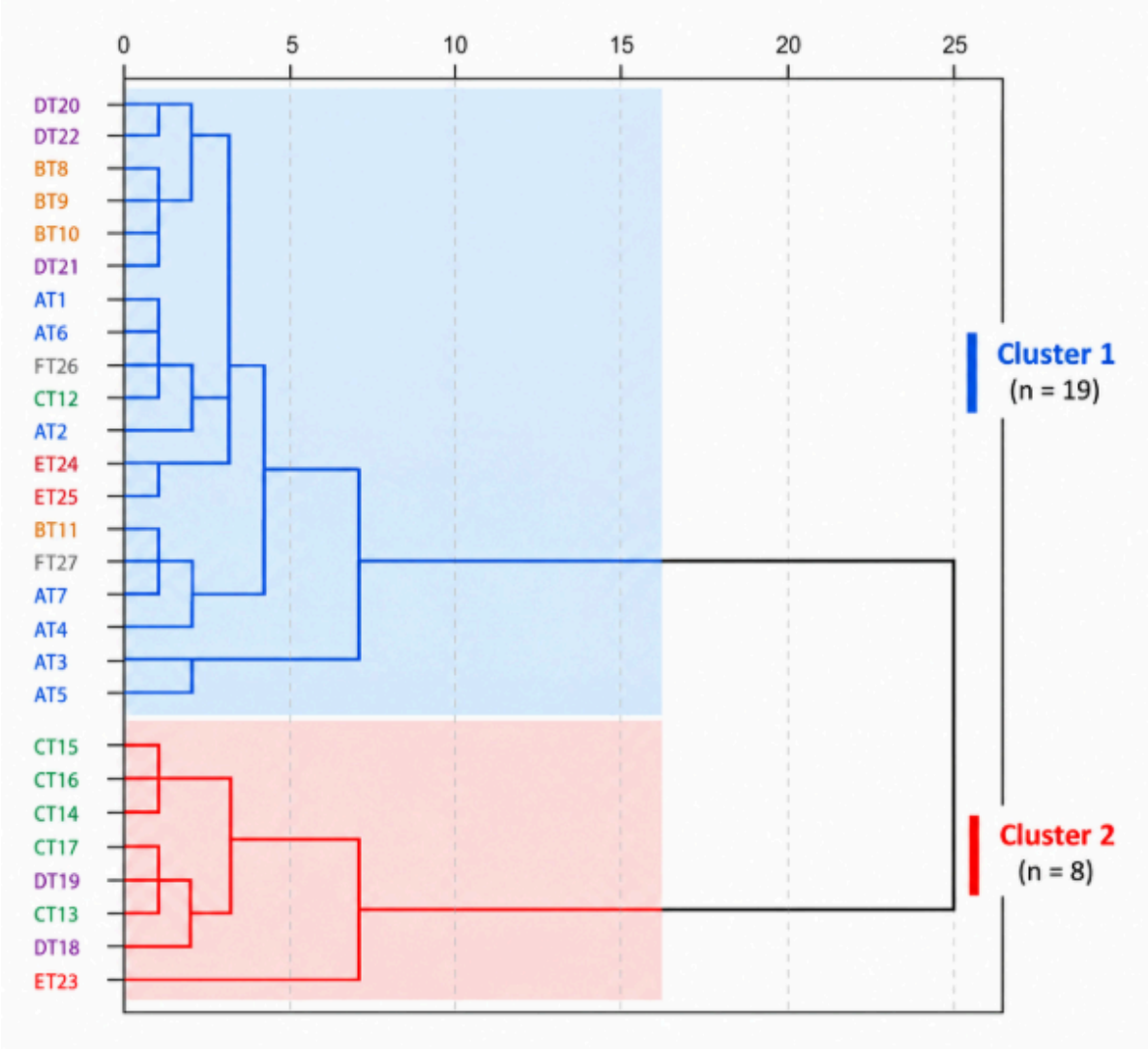


Figure 4

Hierarchical clustering dendrogram of 27 cigarette tobacco samples based on 13 standardized elemental concentrations using Ward's linkage method and squared Euclidean distance.