Decoding Source of Leather Odor: A Quantitative Analysis with Heracles NEO

by

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Abstract

Leather products are widely used in our daily life and in close contact with users, but the pleasant feeling during its usage is severely affected by the odor volatilized from the leather surface. In this study, a quantitative analysis method to investigate the differences in the odor profiles among four types of leather was proposed. The primary olfactory constituents of four leather types were examined by Heracles NEO ultra-fast gas-phase electronic nose, including Principal Component Analysis (PCA) and Discriminant Factor Analysis (DFA). In chrome-tanned cattle hide leather, the substance with the highest content was 2,4-Dinitrotoluene, while methyl dodecanoate was the predominant compound in chrome-free cattle hide leather. Notably, chrome-tanned sheepskin leather exhibited higher levels of dodecanal, clopyralid, n-octylbenzene, propyl cinnamate, and 3-methylhexadecane. Similarly, chrome-free sheepskin leather contained higher levels of dodecanal, clopyralid, n-octylbenzene, propyl cinnamate, 3-methylhexadecane, and tetradecanol. These findings indicate that each of the four types of leather possesses distinctive compounds, while also sharing common compounds. Furthermore, the results indicate that radar plots along with PCA and DFA analyses can effectively differentiate between the four types of leather.

1 Introduction

The smell and odor of leather products, such as furniture, handbags and footwear, has been paid a growing concern since consumers are increasingly demanding high-performance products.[1] However, leather has been made by a series of processes such as unhairing, liming, deliming, bating, pickling, chrome tanning, retanning, fatliquoring, dyeing, and finishing, in which a large number of chemicals are added, and impart leather products with unique smell.[2] The odor volatilized from footwear can cause dizziness, nausea and other adverse reactions and make it difficult to gain an advantage in sales.[3]

Throughout the leather production process, a multitude of chemicals are both generated and introduced. For instance, in the unhairing process, inorganic sulfides are employed as hair-burning agents, and may produce hydrogen sulfide during the process.[4] Moreover, this process also leads to the formation of ammonia gas due to the reaction between proteins and alkalis.[5]

During chrome-tanning process, acidic odor in leather primarily originates from acidic substances in the tanning agents.[6] Formaldehyde-containing chemicals are also introduced during retanning, neutralization, and dyeing processes.[7]

In processes like fatliquoring, dyeing and finishing, distinct agents are incorporated into the leather. Fatliquoring involves the application of animal and plant fats and oils, resulting in the production of fatty odors.[8,9] The dyeing process employs aldehyde and benzene compounds as pigments, giving rise to the creation of organic compounds like toluene, xylene, and formaldehyde.[10] These substances might linger on the leather surface, decomposing or oxidizing under high temperatures or light exposure, producing odorous compounds such as phenol and pyridine.[11] In the softening phase, waxes and oils are added, leading to the emission of a waxy odor from the leather.[12]

Current assessment predominantly hinges upon subjective appraisals conducted by evaluators' olfactory senses, which is aimed at discerning favorable from unfavorable aromas.[13] However, the reliability of those outcomes is poor; moreover, this method might be unhealthy to the assessors and only a limited array of odorant substances can be identified. As a result, the task of objectively quantifying the attributes of olfactory emanations arising from leather-based articles remains a formidable challenge. Therefore, it is necessary to propose an objective quantitative analysis to evaluate the odor of leather and leather products.[14]

Electronic nose technology transforms subjective odor analysis into quantitative and qualitative data.[15] It has been widely adopted in the fields of food and medicine, enabling rapid and objective assessment of

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product quality.[16-18] The Heracles NEO is an efficient tool that combines GC-MS and electronic nose capabilities, allowing for the swift separation and identification of components within volatile substances.[19] By collecting Kovats retention indices and qualitatively examining chromatographic peaks through the AroChemBase database, this method can derive corresponding substance compositions and odor information. Compared to traditional chromatographic methods, Heracles NEO offers advantages such as minimal sample usage, shorter detection times, simplified preprocessing, and the absence of additional reagent consumption. Therefore, Heracles NEO presents a feasible approach for identifying leather odors.

Given the limited existing research on leather odors, the purpose of this study was to investigate the variations in odor profiles among four types of leather with chrome-tanned or chrome-free leathers, and then to identify the primary odor volatile components.

2 Experimental

2.1 Materials and sampling process

Chrome-tanned cattle hide leather (CTCL), chrome-tanned sheep leather (CTSL), chrome-free cattle hide leather (CFCL), and chrome-free sheep leather (CFSL) were purchased from the local market. In addition, 20 mL headspace vials and screw caps equipped with silicone/Teflon septa (Palo Alto, CA, USA), n-alkane mixture (C6-C16) (A10142930, Restek, USA), equipped with a sealed bag were prepared.

Leather samples were cut into small pieces of approximately 0.5 × 0.5 cm in size. Then, 0.6 g of leather samples were put into a 20 mL headspace vial for the electronic nose, approximately one third of the total volume of the feed vial, sealed via a PTFE spacer, and the prepared sample was placed on the autosampler unit to be analyzed.

2.2 Heracles NEO instrumentation and settings

In this study, the odor analysis was conducted using a Heracles NEO electronic nose (Alpha M.O.S., France). The instrument was equipped with an automated sampling unit, an ultrafast GC system, as well as two flame ionization detectors (FIDs) and two distinct polar columns (MXT-5 and MXT-1701). The integration of classical GC functions and advanced data processing software within the instrument enabled a comprehensive analysis of the collected data.

The experiments were conducted based on pre-established parameters (Table I) that were optimized for the assessment of leather odor.

Table I

The set parameters of Heracles NEO

Parameters	Numerical values
Headspace generation	
Injection bottle volume	20 mL
Sample size	0.6 g
Heating and oscillation temperature	80°C
Heating and oscillation time	20 min
Feeding samples	
Injection volume	5000 μL
Injection speed	500 μL/s
Inlet temperature	200°C
Feed duration	15 s
Trap	
Initial temperature	40°C
Diversion	10 mL/min
Catch duration	20 s
Final temperature	240°C
Column temperature	
Initial temperature	40°C (5 s)
Procedure heating up	1.0°C/s -80°C (0 s)
	2.0°C/s -250°C (30 s)
Collection time	160 s
Detectors	
Detector temperature	280°C
FID gain	12

2.3 Data Analysis and Comparison

The Heracles Neo ultra-fast GC e-nose compensates for the inability of sensor-based e-noses to qualify by quickly obtaining GC information on the target, calibrating it with n-alkane standard solutions (nC_6 to nC_{16}), converting retention times to Kovats retention indices, and generating qualitative results from the AroChemBase database.[19] The AroChemBase database is a Kovats RI qualitative library of millions of constituent odor profiles of compounds assessed by professional odor tasters. The AroChemBase database is a qualitative database of Kovats RI. For data analysis, a method of data radar plots, PCA and DFA, was applied.

Chemical pattern recognition uses data radar plots to visualize the odor composition based on available data. Combining the efficient separation capabilities of gas chromatography with the biomimicry of odors, the Heracles NEO electronic nose provides a comprehensive odor profile of volatile compounds. PCA is a commonly used data dimensionality reduction method and data pre-processing technique that converts high-dimensional data into low-dimensional data while retaining the primary information of the original data. DFA is a method of multivariate statistical analysis, which is based on multivariate analysis, where a set of discriminant factors are identified to discriminate between different groups of samples.[20]

3 Results

3.1 Qualitative analysis of volatile compounds by Heracles NEO e-nose

A comparison of the retention parameter profiles of MXT-5-FID1 and MXT-1701-FID2 in Figure 1 and Figure 2 showed that there was a large difference in odor between cattle hide leathers and sheep leathers, while there were similar profiles between CTCL and CFCL, and between CTSL and CFSL. The content of the odor components

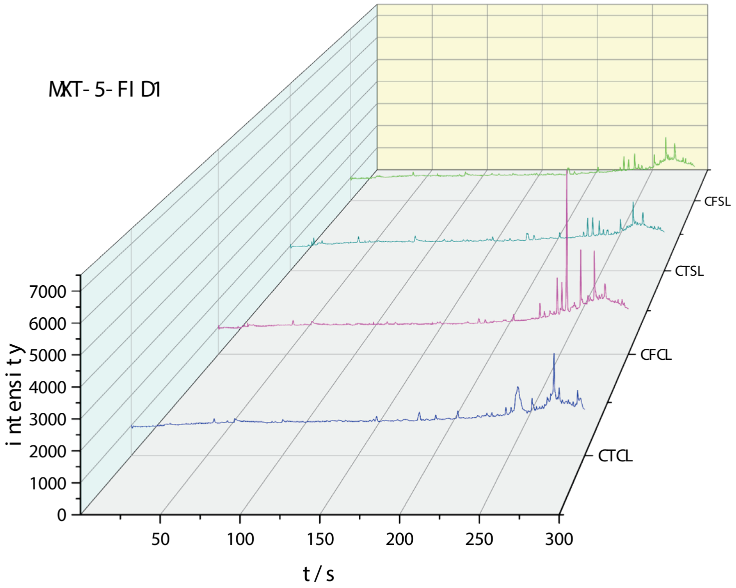

Figure 1. The odor fingerprint of CTCL, CFCL, CTSL and CFSL obtained by the MXT-5

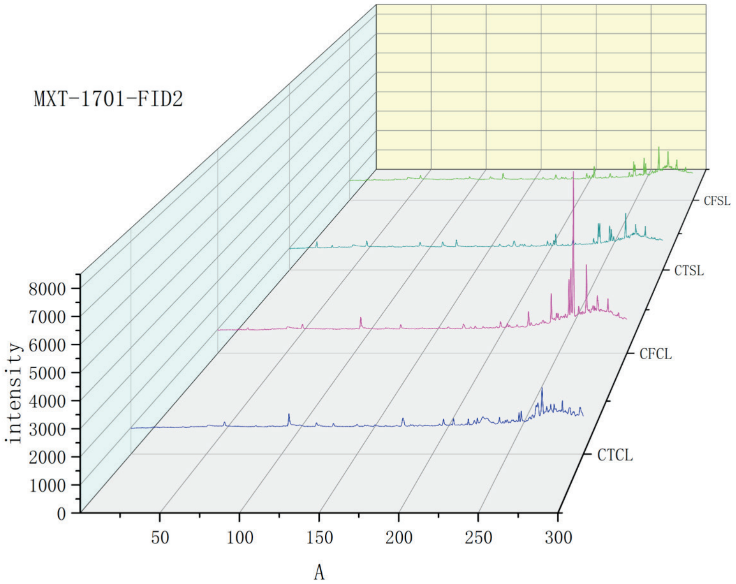

Figure 2. The odor fingerprint of CTCL, CFCL, CTSL and CFSL obtained by the MXT-1701

could be inferred from the peak values of the peaks, the higher the content of the odor components the greater the chromatographic stimulus and the higher the peak.

Table II and Table III described the peak location of CTCL and CFCL and the corresponding possible compounds and their relative contents. In this context, it is notable that both CTCL and CFCL shared the same peak values in the MXT-5 analysis, specifically at 767, 1198, 1298, 1475, and 1497. Similarly, in the MXT-1701 analysis, the shared peak values were 822, 1298, and 1677. These data indicated the presence of identical odoriferous components in the two types of leather. The preliminary analysis suggested that the shared substances included 2-Decanol, Pentadecane-3-Tetradecanol and Toluene.

Based on the data from Tables II and III, it was evident that the potential primary constituents of the odor emitted from CFCL included (E,E)-2,4-Hexadienal, Fenugreek lactone, L-Carvone, (E,Z)-2,4-decadien-1-al, Methyl laurate, 2,4-Dinitrotoluene, Heptyl octanoate, Butyl cinnamate, Methyl tetradecanoate, n-octadecane, and 3-Methyltetradecane. Significantly, 2,4-Dinitrotoluene exhibited a higher concentration, accounting for 47.3% in MXT-5 and 27.59% in MXT-1701. This substance was characterized by an unpleasant and pungent odor.

In contrast, the main odor constituents in CFCL included (*E,E*)-2,4-Nonadienal, delta-octalactone, Skatole, Propyl nonanoate, Propyl decanoate, beta-Himachalene, Methyl dodecanoate, Hexadecane, tetrachloro-m-xylene, and 6-methyl hexadecane. Among these, Pentadecane, Methyl dodecanoate, Hexadecane, and 3-Methylhexadecane were present in relatively higher proportions. Specifically, Methyl dodecanoate had the highest

Table II

Possible volatile compounds identified in CTCL and relative contents

No.	MXT-5		MXT-1701		Possible compounds	Sensory descriptions	Relative content (%)	
	RT	RI	RT	RI			MXT-5	MXT-1701
1	54.84	767	62.07	822	Toluene	Paint; Rubber; Solvent; Sweet	2.51	4.42
2	100.3	915	134.23	1049	(*E,E*)-2,4-Hexadienal	Orange; flowering, vegetative; spicy; sweet	2.32	2.97
3	162.38	1105	213.93	1329	Fenugreek lactone	burnt; caramel; caramel; coffee; marshmallow; maple	3.26	5.69
4	190.68	1198	207.23	1298	2-Decanol	Alkanes; Miscellaneous alcohols	7.21	6.59
5	201.36	1240	223.77	1377	L-Carvone	Herbaceous; Liquorice; minty	2.95	4.40
6	216.12	1298	233.39	1429	(*E,Z*)-2,4-decadien-1-al	Greasy; metal; candle	5.20	7.00
7	247.88	1475	244.37	1495	Pentadecane	Alkanes; Miscellaneous alcohols	3.91	5.05
8	257.54	1541	258.77	1595	Methyl laurate	Soap; sweet; candle scented.	3.16	7.83
9	255.76	1528	272.61	1692	2,4-Dinitrotoluene	Unpleasant smell	47.30	27.59
10	275.18	1670	281.89	1758	Heptyl octanoate	Fresh; freshly cut green grass; greasy	2.29	3.13
11	275.76	1674	293.75	1841	Butyl cinnamate	Fruit; Spicy	2.79	3.76
12	277.58	1688	286.09	1787	Methyl tetradecanoate	Brandy wine; floral, botanical	4.64	6.22
13	283.16	1729	290.67	1819	*n*-octadecane	Alkanes; Fruits; Fuel oil flavor; Miscellaneous alcohols	6.30	5.83
14	251.41	1497	244.52	1496	3-Methyltetradecane	Alkanes; Miscellaneous alcohols	3.16	5.05
15	265.18	1597	275.71	1714	3-Tetradecanol	Fresh; Sweet	3.01	4.48

relative content, constituting 38.47% in MXT-5 and 38.55% in MXT-1701. Interestingly, this compound imparted a relatively pleasant fragrance characterized by notes of wine and floral scents.

Table IV and Table V delineated the peak values and the corresponding potential substances along with their relative abundances in CTSL and CFSL. Specifically, within the MXT-5 analysis, the shared peak values were 768, 915, 1216, 1299, 1417, 1443, 1475, 1596, and 1669. In the context of the MXT-1701 analysis, the corresponding shared peak values were 822, 958, 1529, 1585, 1676, and 1732. Preliminary analysis indicated the presence of common substances, including Toluene, 3,3-Diethylpentane, Methyl nonanoate, Cinnamaldehyde, Dodecanal, Clopyralid, *n*-Octylbenzene, Propyl cinnamate, and 3-Methylhexadecane.

Distinctly, only CTSL exhibited the odoriferous substances Acetone, 2-Dodecanone, and 2-Tridecanone. The odoriferous constituents with the highest content in CTSL encompassed Dodecanal, Clopyralid, n-Octylbenzene, Propyl cinnamate, and 3-Methylhexadecane. Their aromas primarily constituted a blend of aromatic hydrocarbons reminiscent of paints and spices. Conversely, solely CFSL contained the odoriferous substances Skatole, Propyl cinnamate, delta-Undecalactone, and Tetradecanol. Among these, Dodecanal, Clopyralid, n-Octylbenzene, Propyl cinnamate, 3-Methylhexadecane, and Tetradecanol exhibited elevated concentrations. The aromas of these substances ranged from petroleum- and pigment-like scents to hydrocarbon-like notes, fatty odors, and some mildly stimulating fragrances.

Table III

Possible volatile compounds identified in CFCL and relative contents

No.	MXT-5		MXT-1701		Possible compounds	Sensory descriptions	Relative content (%)	
	RT	RI	RT	RI			MXT-5	MXT-1701
1	54.84	767	62.07	822	Toluene	Paint; Pungent; Rubber; Solvent; Sweet	1.84	1.96
2	190.68	1198	207.23	1298	2-Decanol	Flower; fruit; alcohols	2.13	2.27
3	195.33	1216	212.38	1321	(*E, E*)-2,4-Nonadienal	Cucumber;greasy; deep-fried; freshly cut grass	1.45	1.33
4	216.13	1298	248.44	1522	delta-octalactone	Dairy products; Greasy; Peach; Sweet	1.96	2.05
5	235.41	1398	264.74	1637	Skatole	Animals; Medicinal; Mothballs; Intense; warm	3.97	3.62
6	238.69	1418	238.64	1460	Propyl nonanoate	Fermented or brewed; muskmelon	1.58	1.42
7	247.88	1475	244.37	1495	Pentadecane	Alkanes; Miscellaneous alcohols	9.47	6.97
8	250.35	1490	254.94	1568	Propyl decanoate	Fruit, sweet	1.39	1.19
9	251.41	1497	257.52	1586	beta-Himachalene	Pine; trees	7.67	9.59
10	255.05	1523	260.8	1609	Methyl dodecanoate	Coconut;Creamy; Greasy; Floral or botanical; Fruits	38.47	38.55
11	265.23	1598	257.52	1596	Hexadecane	None	12.42	12.44
12	266.11	1604	275.2	1711	3-Tetradecanol	Alkane; Fruits	2.19	2.76
13	270.51	1636	278.84	1736	tetrachloro-m-xylene	Aromatics; pungent	2.68	2.22
14	272.07	1648	266.54	1650	6-methyl hexadecane	None	1.87	1.22
15	275.19	1670	270.38	1677	3-Methylhexadecane	None	10.91	12.41

Table IV

Possible volatile compounds identified in CTSL and relative contents

No.	MXT-5		MXT-1701		Possible compounds	Sensory descriptions	Relative content (%)	
	RT	RI	RT	RI			MXT-5	MXT-1701
1	18.95	505	22.05	602	Acetone	Acetone; Ethanol	4.43	5.35
2	54.89	768	62.03	822	Toluene	Paint; Pungent; Rubber; Solvent	5.67	4.62
3	100.29	915	104.99	958	3,3-Diethylpentane	None	4.27	3.36
4	195.53	1216	207.33	1298	Methyl nonanoate	Sweet; tropical; candle-scented	5.84	4.05
5	216.21	1299	244.45	1495	Cinnamaldehyde	Cinnamon; Clove; Spicy	5.09	4.78
6	235.29	1397	244.45	1495	2-Dodecanone	Oranges; Floral or botanical; Fruits	3.95	4.08
7	238.57	1417	248.29	1521	Dodecanal	Aldehyde group; Octanoic acid; Orange; Oily	14.84	16.81
8	242.65	1443	257.33	1585	Clopyralid	Aromatic;aromatic	14.39	13.36
9	247.91	1475	249.44	1529	*n*-Octylbenzene	Amber; Orange; Pepper	10.66	9.95
10	251.25	1496	260.57	1608	2-Tridecanone	Coconut; Dairy; Earthy; Oily	3.43	4.08
11	264.97	1596	278.27	1732	Propyl cinnamate	Peach	9.64	8.81
12	275.05	1669	270.23	1676	3-Methylhexadecane	None	17.78	20.75

Table V

Possible volatile compounds identified in CFSL and relative contents

No.	MXT-5		MXT-1701		Possible compounds	Sensory descriptions	Relative content (%)	
	RT	RI	RT	RI			MXT-5	MXT-1701
1	54.6	766	62.03	822	Toluene	Paint; Pungent; Rubber; Solvent	4.14	4.00
2	100.3	915	104.98	958	3,3-Diethylpentane	butter	3.08	2.55
3	195.48	1216	207.24	1297	Methyl nonanoate	Sweet; tropical; candle-scented	3.71	5.57
4	216.04	1298	244.52	1496	Cinnamaldehyde	Cinnamon; Clove; Spicy	5.36	3.88
5	235.3	1398	264.54	1636	Skatole	Animals; Fecal; Medicinal; Mothballs	2.99	2.71
6	238.52	1417	248.36	1522	Dodecanal	Aldehyde group; Octanoic acid; Orange; Oily	12.34	13.34
7	242.62	1442	257.37	1585	Clopyralid	Aromatic, aromatic	9.88	13.30
8	247.91	1475	249.44	1529	*n*-Octylbenzene	Amber; Orange; Pepper	13.72	10.85
9	265.08	1596	278.32	1732	Propyl cinnamate	Peach	10.64	13.34
10	275.05	1669	270.23	1676	3-Methylhexadecane	None	23.14	20.24
11	265.82	1602	293.68	1841	delta-Undecalactone	Coconut; Creamy; Greasy; Fruits	4.31	3.78
12	276.98	1684	286	1786	Tetradecanol	Coconut; oily fat	6.69	6.46

3.2 Chemical pattern recognition analysis

Figure 3 illustrates the comprehensive radar plots generated from MXT-5 and MXT-1701 analyses of the four leather types, enabling a visual comparison of the distinctions among them. Notably, the odor intensity of CFCL is the highest, with a peak reaching up to 4000 units. In contrast, the odor intensity of chrome-tanned sheep leather and chrome-free sheep leather is comparatively lower. Furthermore, it is evident that discernible differences existed in the volatile components among the four leather types.

3.3 PCA principal component analysis

Figure 4 presents the PCA analysis of the four leather types, yielding a recognition pattern index of 90. The combined contribution of principal component 1 and principal component 3 is 84.444%, indicating a high level of information. The positions of the four leather types are predominantly located in different intervals, signifying that each type of leather possesses unique components allowing for the distinction among them.

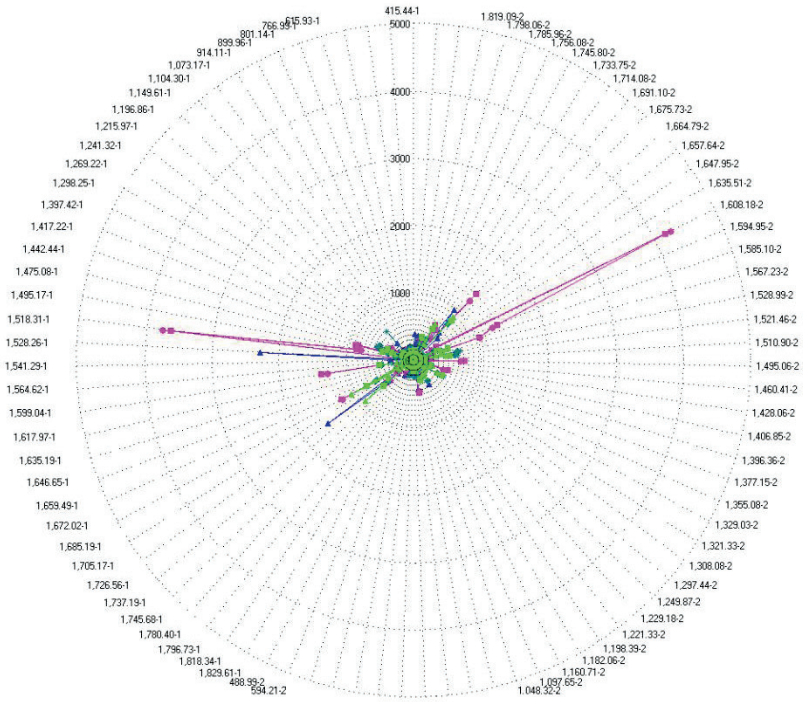

Figure 3. Radar fingerprint map of leather material

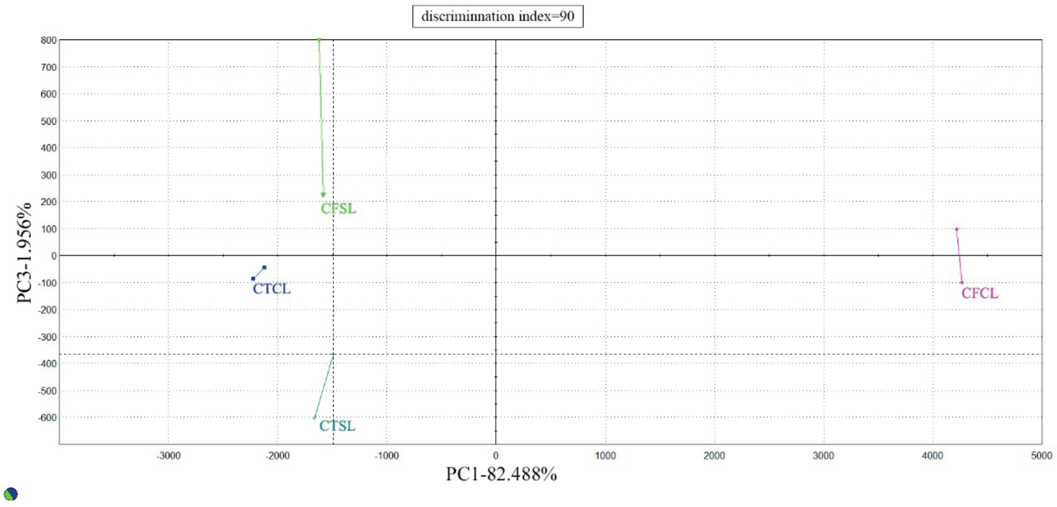

Figure 4. Principal Component Analysis (PCA)

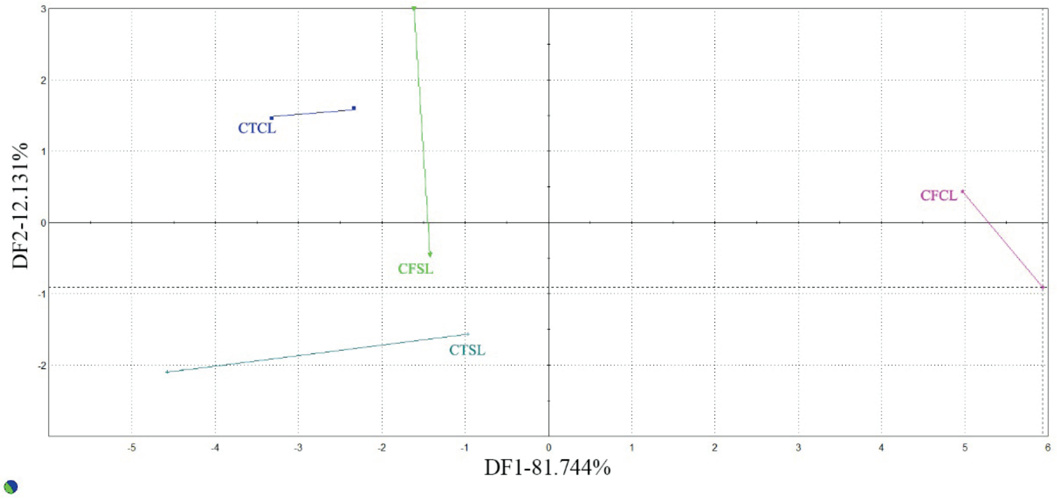

Figure 5. Discriminant analysis (DFA)

3.4 DFA discriminant factor analysis

In the DFA analysis shown in Figure 5, the contributions of discriminant factor 1 and discriminant factor 2 were 93.875%, demonstrating high discriminatory power and indicating clear differentiation among the four leather types.

4 Discussion

Leather is characterized by its numerous volatile components and unique odor. However, current odor evaluation studies in the realm of leather remain largely subjective, relying on experiential assessments, lacking objective quantitative methods. As an emerging technology, Heracles NEO offers advantages such as simple sample preprocessing, convenient instrument operation, rapid detection, and minimal environmental impact. It provides comprehensive odor information about the tested samples and has been applied in fields such as Traditional Chinese Medicine odor analysis, rapid identification, and quality control.

It has been ascertained that benzene-based dyes are employed during the dyeing stage of leather production.[21] In the present study, Benzene-derived volatiles were detected in all leather samples, leading to the preliminary inference that the presence of benzene constituents in leather emissions can be attributed to the dyeing process. Among these, CTCL exhibited the highest concentration of 2,4-Dinitrotoluene at 47.3% for MXT-5 and 27.59% for MXT-1701. Aliphatic hydrocarbons and alcohol volatiles, commonly utilized in leather dyeing and fatliquoring, were also identified in all four leather types.[22] All tested leather samples emitted ester compounds, with a diverse range of types and concentrations. Literature investigation revealed that plasticizers and emulsifiers are commonly employed in leather processing, primarily comprising various organic esters.[23] Among these, Methyl dodecanoate exhibited the highest concentration in chromed bovine leather, at 38.47% for MXT-5 and 38.55% for MXT-1701. CFTL emitted fewer aldehyde compounds, whereas CTCL exhibited a slightly higher presence of aldehyde compounds. Aldehydes are widely utilized as auxiliary tanning agents in leather tanning processes.[7] And CFCL did not use aldehyde tanning agents and related heavy metal salts.

Based on the detected organic compounds, it is hypothesized that these chemicals are a result of the substances used in the tanning and fatliquoring dyeing processes. Due to the similarity in chemical constituents used during processing, different leathers may contain similar volatile components. These substances are highly likely to have formed after the completion of the leather tanning process.

PCA reduces the original data, i.e. the linear combinations that explain the maximum variance of the high-dimensional data to a lower dimension by finding the principal components of the original data, i.e. the linear combinations that explain the maximum variance of the original data, and data down to lower dimensions, thereby reducing redundant information in the data and improving the operational efficiency of the model.[24] DFA is often used for classification or discriminant analysis, for example to classify patients into disease and non-disease groups, or to classify products into different quality levels, etc. DFA can also be used for downscaling and variable selection to help researchers identify the most important variables for discriminating between groups.[25]

It was evident that these odoriferous substances were mostly composed of benzene-derived compounds, alkanes, alcohols, and esters, contributing to the overall leather odor through their mixture. The distinct prominence of different odor constituents among the four leather types indicated substantial differences in odor profiles among different leather types.

5 Conclusion

The quantitative analysis method by Heracles NEO was capable of distinguishing different types of leather produced through different tanning methods. Heracles NEO's electronic nose testing differed from traditional leather odor detection methods; it employed a mechanized and standardized approach, providing objective and visualized results.

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