Original Article
Food Science and Technology

ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)

DOI: https://doi.org/10.1590/fst.58922

Characteristic the volatiles compounds of fractionation beef tallow by gas chromatography-ion mobility spectrometry

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Abstract

In this study, drying fractionation beef tallow was obtained, and then the beef tallow was analyzed by headspace gas chromatography-ion mobility spectrometry (GC-IMS). The experimental results showed that with the decrease in fractionation temperature, the content of saturated fatty acids displayed a decreasing trend, while the content of unsaturated fatty acids displayed an increasing trend. The results of determination of peroxide value, acid value and iodine value showed that the acid value of the components increased with the decrease of the fractionation temperature. A total number of 63 volatile compounds were detected through (GC-IMS), and qualitative and quantitative analyses were performed in order to determine the contents of volatile compounds in samples. The results also indicated that esters, aldehydes, ketones, and alcohols of macromolecular volatile components mainly appear in the MP_{30}, MP_{40}, and MP_{40+} components, while the volatile components of MP_{24} are mainly small molecules of esters, alcohols, aldehydes, and ketones. Indeed, in this study, ion migration chromatography fingerprint library of the characteristic components of beef tallow was also constructed, and the generated (GC-IMS) two-dimensional spectrum was used to analyze the difference characteristics of beef tallow, which provides theoretical guidance for the physical and chemical properties of beef tallow.

Keywords: beef tallow; dry fractionation; volatiles compounds; GC-IMS.

Practical Application: For beef tallow, its melting point is higher, and it is solid at room temperature, which is not easy to extract. High melting point beef tallow is suitable for frying or shortening, medium melting point beef tallow for blending, and low melting point beef tallow for flavoring.

1 Introduction

Beef tallow, the fat derived from the fatty tissue of cows, is a mixture of triglycerides (Machiels et al., 2004). The variety of vegetable oils has been constantly enriched, and the quality has been gradually improved, but there is limited development in the field of animal fats, especially beef tallow (Cao et al., 2013). In the early 20th century, oil fractionation and crystallization technology was developed (Wang et al., 2017), and the dry fractionation of palm oil emerged in the 1950s. In the 1970s, the fractionation and crystallization technology developed rapidly. Dry fractionation is a physical process which uses different melting points for separation (Danthine et al., 2017; Chaleepa et al., 2010; Sweers et al., 2022). It has many advantages in industry, such as no waste water, simple and safe operation, high flexibility, no use of solvents and low production costs (Jonkman et al., 2020; Schutyser et al., 2015; Barakat et al., 2014; Licari et al., 2016).

Referring to the method of palm oil extraction, the dry fractionation was appropriate to carry out the beef tallow (Bashiri & Fatemi, 2004). Some studies have shown that the adverse effects of animal fats on human body are far less than that of vegetable oil. Animal fats contain certain unsaturated fatty acids. Proper intake of certain animal fats can prevent cardiovascular and cerebro-vascular diseases, atherosclerosis and other diseases (Yang & Li, 2012). For beef tallow, its melting point is higher, and it is solid at room temperature, which is not easy to extract.Beef tallow products with different melting points can be produced by using the fractionation technology to meet the different needs of beef tallow products in the market (Meng et al., 2010). For example, high melting point beef tallow is suitable for frying or shortening, medium melting point beef tallow for blending, and low melting point beef tallow for flavoring (Meng et al., 2010). With the development of beef tallow separation technology, the new generation of beef tallow products have bright color, mellow taste, rich fat flavor, stable processing performance and rich nutrients. At present, many scholars have focused on studying the physicochemical properties of fractionated tallow, but less attention has been given to the volatiles compounds. This study has a guiding role in the application of fractionated beef tallow.

Gas chromatography ion mobility spectrometry coupled with automatic headspace sampling (GC-IMS) is a advanced synergistic technique that characterizes chemical substances based on differences in the migration rates of gas-phase ions in an electric field (Huang et al., 2019; Garrido-Delgado et al., 2015). GC-IMS combines the high efficiency of GC separation technology with the high sensitivity of IMS detection technology to give superior analytical results. GC-IMS has the advantages

Received 29 Apr., 2022
Accepted 21 June, 2022
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of short detection cycle (Arroyo-Manzanares et al., 2019; Wang et al., 2020), simple operation, rapid sensitivity and so on It has been applied for the detection of fruit and vegetable residues (Chen et al., 2020a), detection of additives in food, detection of adulteration of edible oil (Jiang et al., 2020) and the construction of fingerprint map (Contreras et al., 2019), but there are few reports on the construction of fingerprint map of characteristic flavor of beef tallow.

Principal component analysis (PCA) is a classic mathematical technique for the reduction of dimensionality of data in the form of n observations (or cases) of a vector with p variables (Johnstone & Lu, 2009). The PCA is a way of identifying patterns in data and expressing the data in such a way as to emphasize their similarities and differences. These principal components are orthogonal and have been shown to retain a significant amount of the original data set variation (Bu & Brown, 2000; Knorr & Futrell, 1979). It has been applied quite extensively and successfully in the area of applied chemistry for a wide variety of functions, ranging from Fourier transform infrared spectroscopy (Nieuwoudt et al., 2004), liquid chromatography (Vandeginste et al., 1985), or surface enhanced Raman scattering (Etchegoin et al., 2007). In early work, Brown et al. (1975) segregated Spanish peanuts from other peanut varieties via the (PCA) successfully and grouped cultivars from different locations based on their fatty acid composition.

Based on the previous work of the research group, liquid beef tallow was prepared through dry fractionation (Liu, 2020), which was used as raw material. In our experiment, four components of extracted beef tallow, MP_{40+}, MP_{40}, MP_{30} and MP_{24}, were used as samples and systematically analyzed. The differences in physicochemical properties, fatty acid composition, and volatile matter of different beef tallow components were compared. GC-IMS combined with principal component analysis (PCA) method was used to determine and analyze the characteristics of beef tallow. Ion migration chromatography fingerprint library of beef tallow was constructed, and the generated (GC-IMS) two-dimensional spectrum was used to analyze the different characteristics of beef tallow. This study provides a potential method and further data support for the development of beef tallow discrimination techniques.

2 Materials and methods

2.1 Materials

Refined beef tallow was supplied by Tianjin Yixing Halal Food Company (Tianjin, China). Cyclohexane, glacial acetic acid, and 95% ethanol were purchased from Tianjin Tianli Chemical Reagent Co., Ltd. (Tianjin, China). Phenolphthalein indicator and potassium iodide were purchased from Tianjin Damao Chemical Reagent Factory. Trichloromethane was obtained from Tianjin Chemical Reagent Supply and Marketing Company (Tianjin, China). Soluble starch, 0.1010 mol/L sodium thiosulfate standard titrant, 0.1020 mol/L potassium hydroxide standard titrant, and 0.01009 mol/L sodium thiosulfate standard titrant were obtained from Tianjin No. 3 Chemical Reagent Factory (Tianjin, China).

The following instruments were used for analysis: Precision analytical balance (XPE105, METTLER TOLEDO); Crucible press (NETZSCH); Gas chromatography ion mobility spectrometer (1H1-00053, GAS Co., Ltd., Germany).

2.2 Preparation of beef tallow dry fractionation components

The technological process of beef tallow dry fractionation was established. Slow crystallization rather than fast crystallization was used to extract beef tallow,as shown in Figure 1. First, 500 g beef tallow was weighed and heated to 85 °C. It was melted completely, and kept for 30 min. After rapidly cooling to 70 °C, it was slowly cooled to 40 °C at a cooling rate of 0.5 °C/min and crystallized at this temperature (40 °C) for 21 h. Then, the liquid oil was filtered through gauze, and the solid part was labeled MP_{40+}. The liquid beef tallow was crystallized at 30 °C for 21 h and filtered through gauze. The filtered solid beef tallow was collectively called MP_{40}. The liquid oil was crystallized at 24 °C for 21 h. After filtering, the solid beef tallow was collectively referred to as MP_{30}, and the liquid one was labeled MP_{24}. The liquid oil and solid fat were bagged and weighed.

2.3 Determination of melting point

According to the recommended Chinese national standard GB12766-2008 determination of melting point of animal fat.

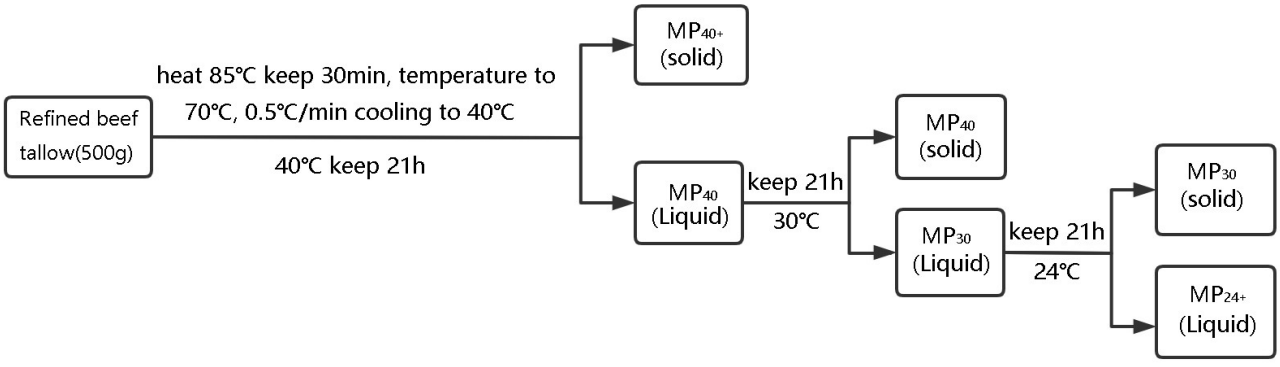

Figure 1. Dry fractionation process of refined beef tallow step by step.

2.4 Determination of acid value

According to the national standard 5009.229-2016 determination of acid price in food according to the national standard for food safety, hot ethanol indicator titration method was used.

2.5 Determination of peroxide value

According to the national standard 5009.227-2016 determination of peroxide value in Food according to national food safety standard, the titration method was implemented.

2.6 Determination of iodine value

According to the recommended national standard 5532-2008 determination of iodine value of animal and plant oils and fats.

2.7 Determination of cholesterol

According to the national standard 5009.128-2016 determination of cholesterol according to national food safety standard, high-performance liquid chromatography method was used.

Chromatographic conditions: chromatographic column: C18 reverse phase chromatographic column, column length 4.6 mm, inner diameter 150 mm, particle size 5 μm, or chromatographic column with equivalent performance; column temperature: 38 °C; mobile phase: methanol; flow rate: 1.0 mL/min; detection wavelength: 205 nm; injection volume: 10 μL.

2.8 Determination of fatty acid constituents

According to the recommended Chinese national standard GB5009.168-2016 for determination of fatty acids in foods according to the national standard for food safety, the normalization method was used to detect the fatty acids in beef tallow.

Chromatographic conditions: chromatographic column: DB-5; injection volume: 0.5 μL; flow rate: 1.0 mL/min; split ratio: 60 : 1; carrier gas: helium; injection port temperature: 270 °C; initial temperature: 150 °C; heating rate: 15 °C/min, 2 °C/min, 10 °C/min; heating temperature: 200 °C, 224 °C, 270 °C. Retention time at 150 °C and 270 °C: 2 min, 10 min; Sample outlet temperature: 25 °C.

2.9 Determination of volatile substances by GC-IMS

Headspace conditions: Headspace incubation temperature: 100 °C; Incubation time: 15 min; Headspace probe temperature: 85 °C; Sample injection volume: 500 μL; Drift gas/Carrier gas: nitrogen; Cleaning time: 15 min. Incubation speed: 500 rpm.

GC-IMS conditions: IMS detector temperature: 45 °C; Chromatographic column: FS-SE-54-CB-1 15 m, ID: 0.53 mm; Analysis time: 30 min; Column temperature: 60 °C; Flow rate: 1.0 mL/min; Drift gas/Carrier gas: nitrogen (Chen et al., 2019; Chang et al., 2020).

2.10 Analysis software: instrumentation supporting analysis software includes

1. VOCal: It is used for the qualitative and quantitative analyses of spectrum and data. The NIST database and IMS database built into the application software can conduct qualitative analysis on substances. Users can expand the data by using standard products according to their needs. Each point in the figure represents a volatile organic compound, which can be quantitatively analyzed after a standard curve has been established. The application classifier is included in the VOCal software, which is the function of the built-in plugin in VOCal. It is completed automatically.
2. LAV: It is used to view the analytical spectrum, where each point represents a volatile organic compound. It can be quantitatively analyzed after the standard curve is established.
3. Reporter plug-in: Direct comparison of spectrogram differences between samples (2D top view and 3D spectrogram).
4. Gallery Plot plug-in: Fingerprint comparison, intuitively and quantitatively compares the differences of volatile organic compounds between different samples.
5. Dynamic PCA plug-in: Dynamic principal component analysis, used for sample cluster analysis and to quickly determine the types of unknown samples.
6. GC × IMS Library Search: Application software built-in NIST, IMS database for material qualitative analysis.

The volatile compounds were tentatively identified based on comparison of RI and drift time with the NIST library and IMS database retrieval software obtained from G.A.S (Dortmund, Germany).

3 Results and discussion

3.1 Comparion of melting point

Beef tallow was an animal oil with high saturated fatty acid, whose melting point generally ranged from 43 °C to 49 °C (Wang et al., 2017). It could be seen from Table 1 that with the decrease of the crystallization temperature, the melting point of the extracted beef tallow also decreased gradually, indicating that the crystallization temperature was an important condition for obtaining the beef tallow with desired-melting point. Tirtiaux used natural fractionation in a large, multi-purpose vacuum filter to produce beef tallow at different melting points (20 °C-56 °C) (Tirtiaux, 1983). In this study, the crystallization temperature was controled, and the crystallization time with melting points was 22.10, 34.17, 46.83 and 50.77 °C. Compared with Tirtiaux, our method was relatively simple and easy to operate, and could be operated at any time with low cost. Besides, large-scale vacuum filter and other equipment in China were relatively rare and expensive, so the method was practical and feasible (Tirtiaux, 1983).

Table 1. Comprehensive table of various indexes of beef tallow fractionation.

Indexes	The raw material	MP_{40+}	MP_{40}	MP_{30}	MP_{24}
Acid value (mg/g)	1.28 ± 0.0236	0.91 ± 0.0479	0.97 ± 0.0619	1.35 ± 0.0171	1.4 ± 0.0331
Peroxide value (g/100 g)	0.0341 ± 0.0036	0.0372 ± 0.0046	0.0522 ± 0.0032	0.0453 ± 0.0047	0.0601 ± 0.0203
Iodine value (g/100 g)	41.97 ± 1.95	31.7 ± 1.72	38.06 ± 0.67	46.25 ± 0.65	51.97 ± 0.67
melting point	44.67 ± 0.76	50.77 ± 0.25	46.83 ± 0.64	34.17 ± 0.21	22.1 ± 0.12
Oil production rate (%)	100	6.36 ± 1.70	55.68 ± 7.54	10.18 ± 4.76	27.85 ± 4.05
Cholesterol content (mg/100 g)	56.71 ± 10.61	27.05 ± 12.53	45.66 ± 6.18	78.94 ± 27.76	70.03 ± 3.14

MP = melting point.

3.2 Comparion of acid value

Acid value was an indicator of the hydrolysis degree of oil. The lower the acid value, the higher quality of oil and the higher the refining degree (Zhou & Chen, 2014). In Table 1, the acid value of beef tallow component increased step by step with the decrease of stripping temperature. MP_{40+} had the lowest acid value and relatively stable properties. The acid value of beef tallow in MP_{40} and MP_{30} was 0.97 mg/g and 1.35 mg/g respectively. MP_{24} had the highest acid value and the most free fatty acid content. MP_{40+} had the least free fatty acid content and could be used for premium shortening. Maybe it's because MP_{24} had been processed many times that it had the highest acid value. In addition, the molecule of MP_{24} components was smaller than that of other beef tallow components, which may also be due to the higher content of free fatty acids resulted from the fractionation. But the content was still lower than the limit value (2.5 mg/g) stipulated in the national standard.

3.3 Comparion of peroxide value

During the processing of beef tallow, it is easy to be oxidized by light, heat, enzymes and metal ions. The peroxide value represents the peroxide content, which is also the product of the first stage of oil autoxidation. Fats and oils with high content of saturated fatty acid generally had better stability (Zheng et al., 2014). In Table 1, the trend of peroxide value was as follows: raw material < MP_{40+} < MP_{30} < MP_{40} < MP_{24}. The peroxide value of MP_{30} was slightly lower than MP_{40}, indicating that the induction period of fatty acids in this component was longer than that of fatty acids in MP_{40}, that was, less peroxides were produced. On the whole, the peroxide value showed an increasing trend with the fractionation, indicating that light and heat had a certain influence on the quality of beef tallow with the fractionation. Because MP_{24} had been processed for the longest time, its peroxide content was the highest but far less than the limit value (0.20 g/100 g), indicating that this processing technology was feasible and the product still had edible value of food.

3.4 Comparion of iodine value

Iodine value reflects the content of unsaturated fatty acids in fats and oils, which enables people to have a more intuitive understanding of the nutritional status of fats & oils (Li, 2016). The iodine value of beef tallow is generally between 40 g and 50 g per 100 g. As shown in Table 1, the iodine value of the raw materials used in this study was 41.83 g/100 g. The iodine value of extracted beef tallow: MP_{40+} < MP_{30} < MP_{40} < MP_{24}, indicating that the content of unsaturated fatty acid in extracted beef tallow increased with the fractionation. Due to the difference in fatty acid composition, the lower the melting point, the higher the iodine value of beef tallow, indicating that the melting point, unsaturated fatty acid content and iodine value were correlated. After fractionation, the iodine value of MP_{40+} was the smallest (31.7 g/100 g), and the iodine value of MP_{24} beef tallow was the largest (51.97 g/100 g). The iodine value of beef tallow ranged from 30 to 52 g/100 g, which indicated that the species distribution of beef tallow was more widely after the fractionation and could meet people's more needs.

3.5 Comparion of cholesterol

Cholesterol is a constituent of tissue cells and the raw material for liver synthesis. Food less than 100 mg/100 g is low-cholesterol food, food with moderate cholesterol in 100-200 mg/100 g and food with high cholesterol in 200-300 mg/100 g (Zhang, 2013). In Table 1, the cholesterol content in MP_{30} was the highest, and that in MP_{40+} was the lowest (27.05 mg/100 g). MP_{40+} could be used to develop high quality shortening, which provided favorable conditions for further development of beef tallow products. The cholesterol content in all components was low, so the products could be safely consumed in the proper amounts.

3.6 Correlation analysis

The correlation coefficient of each index was shown in Table 2. The melting point of beef tallow was correlated with acid value, peroxide value, iodine value and cholesterol content. SPSS17.0 software was used for correlation analysis, and the results showed that the correlation coefficients between melting point, acid value, peroxide value, iodine value and cholesterol content were -0.839, -0.719, -0.951 and -0.807 respectively. The melting point and iodine value were significantly negatively correlated at P = 0.05 level. The correlation coefficient between iodine value and acid value was 0.933, both presented significant positive correlation, especially at P = 0.05 level. The correlation coefficient

between acid value and cholesterol was 0.924, both presented significant positive correlation, especially at P = 0.05 level.

3.7 Fatty acid composition analysis of each component in the refined beef tallow

According to the method described, fatty acid composition was determined and the results of five samples (the raw material, MP_{24}, MP_{30}, MP_{40} and MP_{40+}) are shown in Table 3. Chromatogram of the determination of fatty acids is shown in Figure 2. The results in Table 3 and Figure 2 display the fatty acid components and their proportion in the total fatty acid content of extracted beef tallow. The contents of unsaturated fatty acids, such as oleic acid ($C_{18:1}$), linoleic acid ($C_{18:2}$), palmitoleic acid ($C_{16:1}$, $C_{14:1}$) and heptaenoic acid ($C_{17:1}$) decreased as the melting point increased. The contents of saturated fatty acids, such as lauric acid ($C_{12:0}$), myristic acid ($C_{14:0}$, $C_{15:0}$), palmitic acid ($C_{16:0}$), heptadecanoic acid ($C_{17:0}$) and stearic acid ($C_{18:0}$) increased with the melting point. Oleic acid ($C_{18:1}$) was the most abundant in MP_{24}, which is one of the reasons why MP_{24} can reach liquid state at a normal temperature. The composition analysis of fat showed that at 40 °C ~ 24 °C, with the decrease in fractionation temperature, the content of saturated fatty acids of fractionation components showed a decreasing trend, while that of unsaturated fatty acids showed an increasing trend (Du, 2019; O'Shea et al., 2000). MP_{24}, MP_{30} and MP_{40} contained more oleic acid (41.93%, 40.26%, and 32.83%, respectively), while MP_{40} contained more palmitic acid (34.65%). The results of physical and chemical analyses showed that the fatty acid composition of beef tallow is different from that of palm oil. Although beef tallow is animal fat, its oleic acid content is higher than that of palm oil. Beef tallow also contains slightly more saturated fat than palm oil.

After extraction, the contents of saturated fatty acids in MP_{24} and MP_{40+} were 50.14% and 68.16%, respectively. The content of saturated fatty acid in MP_{24} was reduced by 5.48% compared

Table 2. Pearson correlation coefficient of each indicator.

index	Acid value	Peroxide value	Iodine value	melting point	cholesterol
Acid value	1	0.297	0.933*	-0.839	0.924*
Peroxide value	0.297	1	0.618	-0.719	0.417
Iodine value	0.933*	0.618	1	-0.951*	0.913
melting point	-0.839	-0.719	-0.951*	1	-0.807
cholesterol	0.924*	0.417	0.913	-0.807	1

*Was significantly correlated at 0.05 level (bilateral).

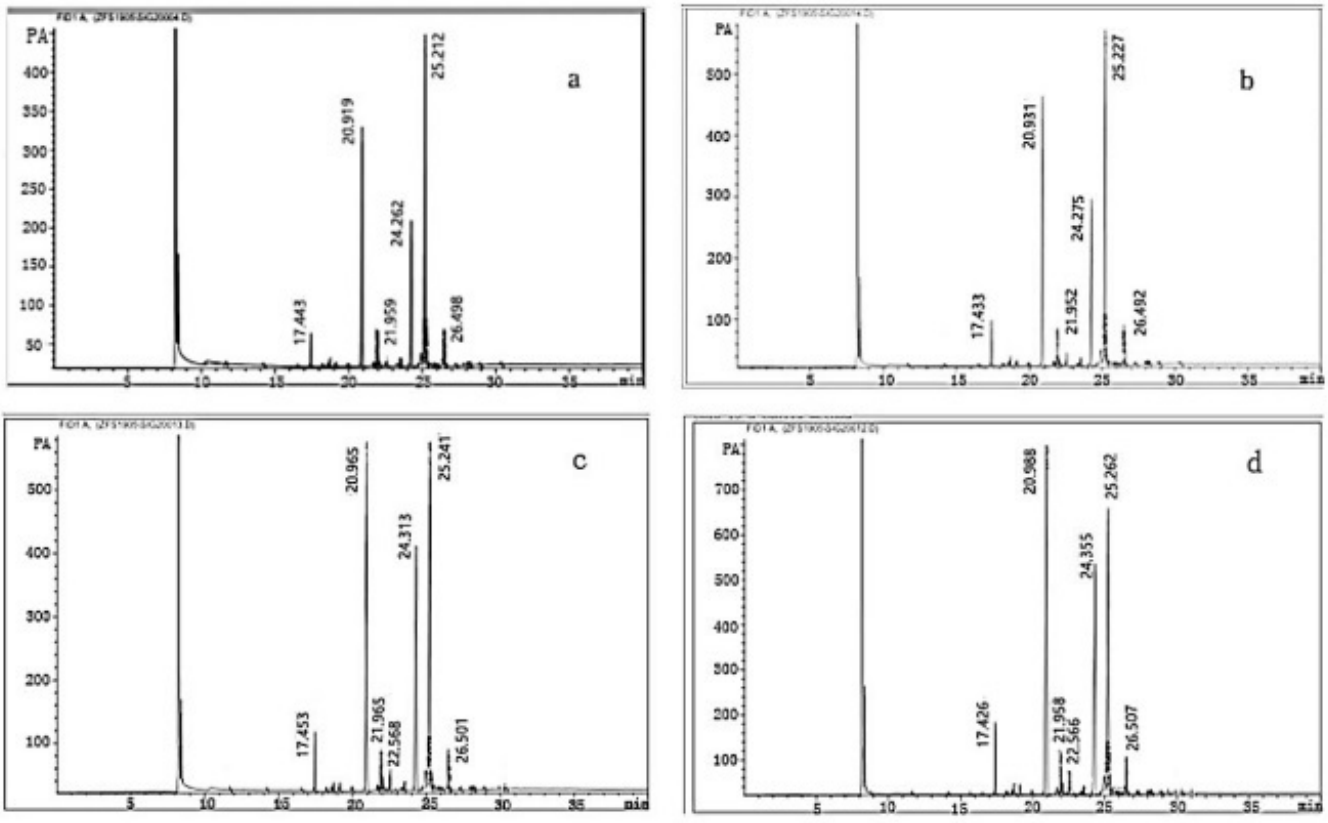

Figure 2. Gas chromatograph of fatty acid composition of sample (a, b, c, d represents $MP_{24,}$ $MP_{30,}$ $MP_{40,}$ MP_{40+} respectively).

Table 3. Category and total fatty acid content of extracted beef tallow fatty acid.

The kind of fatty acid	Relative fatty acid content/%				
	MP_{24}	MP_{30}	MP_{40}	MP_{40+}	Raw Materials
$C_{8:0}$	3.23 ± 0.07	0.00	0.00	0.00	0.00
$C_{10:0}$	0.05 ± 0.01	0.06 ± 0.02	0.04 ± 0.01	0.06 ± 0.02	0.05 ± 0.01
$C_{12:0}$	0.08 ± 0.02	0.09 ± 0.03	0.07 ± 0.02	0.09 ± 0.03	0.08 ± 0.03
$C_{14:0}$	2.76 ± 0.08	3.29 ± 0.1	3.35 ± 0.07	3.82 ± 0.09	3.16 ± 0.11
$C_{14:1}$	0.51 ± 0.01	0.49 ± 0.01	0.37 ± 0.01	0.37 ± 0.01	0.41 ± 0.01
$C_{15:0}$	0.40 ± 0.02	0.46 ± 0.02	0.51 ± 0.03	0.59 ± 0.02	0.46 ± 0.02
$C_{16:0}$	25.47 ± 0.87	28.02 ± 0.94	31.04 ± 1.11	34.65 ± 0.97	28.78 ± 0.74
$C_{16:1}$	2.88 ± 0.12	2.74 ± 0.08	2.16 ± 0.09	2.01 ± 0.10	2.37 ± 0.13
$C_{17:0}$	0.95 ± 0.03	1.06 ± 0.04	1.30 ± 0.04	1.47 ± 0.06	1.16 ± 0.02
$C_{17:1}$	0.61 ± 0.01	0.59 ± 0.01	0.47 ± 0.01	0.39 ± 0.01	0.52 ± 0.01
$C_{18:0}$	16.88 ± 0.73	18.91 ± 0.81	24.45 ± 0.94	27.17 ± 1.04	21.61 ± 0.86
$C_{18:1t}$	0.00	0.00	0.00	0.00	2.27
$C_{18:1}$	41.93 ± 1.56	40.26 ± 1.37	32.83 ± 0.89	26.65 ± 0.67	35.52 ± 1.89
$C_{18:2t}$	0.37 ± 0.01	0.27 ± 0.01	0.28 ± 0.01	0.13 ± 0.01	0.31 ± 0.01
$C_{18:2}$	3.26 ± 0.14	3.06 ± 0.15	2.44 ± 0.09	2.02 ± 0.13	2.68 ± 0.06
$C_{20:0}$	0.07 ± 0.01	0.17 ± 0.01	0.14 ± 0.01	0.12 ± 0.01	0.15 ± 0.01
$C_{18:3}$	0.17 ± 0.01	0.19 ± 0.01	0.22 ± 0.01	0.21 ± 0.01	0.20 ± 0.01
$C_{20:1}$	0.11 ± 0.01	0.11 ± 0.01	0.09 ± 0.01	0.07 ± 0.01	0.10 ± 0.01
$C_{21:0}$	0.17 ± 0.01	0.18 ± 0.01	0.17 ± 0.01	0.14 ± 0.01	0.17 ± 0.01
$C_{22:0}$	0.08 ± 0.01	0.08 ± 0.01	0.06 ± 0.01	0.05 ± 0.01	0.00
Saturated fatty acids	50.14 ± 1.56	52.32 ± 2.14	61.13 ± 2.03	68.16 ± 2.47	55.62 ± 1.79
Monounsaturated fatty acids	46.04 ± 2.06	44.19 ± 1.57	35.92 ± 1.68	29.49 ± 0.79	41.19 ± 1.54
Polyunsaturated fatty acids	3.8 ± 0.13	3.52 ± 0.09	2.94 ± 0.07	2.36 ± 0.14	3.19 ± 0.12

with the raw material (55.62%), and the fractionation effect was obvious (Qiu et al., 2018). reported that the contents of saturated fatty acids in Yanbian Yellow Cattle fat before and after fractionation were 49.80% and 41.51%, respectively. The content of unsaturated fatty acids decreased by 8.29%, which showed a significant fractionation effect. However, in their study, urea-packing method was used, which is not suitable for food applications.

The solvent extraction method is used to distribute triglycerides with low melting point into the oil, so that the melting point of the oil is reduced (Zhang et al., 2014). The crystallization temperature of solvent extraction is low, the molecular weight of most fatty acid molecules in the extracted oil is small, and the carbon chain of the generated fatty acid molecules is short. Consequently, the saponification value of the extracted oil is increased (Chen et al., 2020b). Therefore, dry fractionation of beef tallow was adopted in this experiment.

3.8 Analysis of volatile substances

Chemical analysis and sensory analysis are two effective methods for flavor research. Chemical analysis can accurately detect the content of various components in samples and judge their contribution to the flavor of sample according to their characteristics. Sensory analysis can directly describe the sensory properties of samples, present the overall flavor characteristics, and make up for the deficiency of chemical detection. The flavor analysis of this experiment focuses on chemical analysis.

Figure 3 shows the two-dimensional spectrum of beef tallow. The ordinate is the retention time of GC separation, and the abscissa is relative drift time. Each point on the right side of the reaction ion peak represents a kind of product (Du et al., 2019), with blue as the background. The darker the color, the higher is the content of the product. It can be seen from Figure 4 that volatile organic compounds in samples with different processing groups can be separated well by (GC-IMS), and the differences in beef tallow extracted under different processing technologies can be intuitively seen. Beef tallow is qualitatively analyzed, according to its gas chromatographic retention time and ion drift time. Table 4 shows a total of 63 volatile compounds which were detected in the different beef tallow samples. The Gallery Plot plug-in of LAV software was used to automatically generate fingerprint (Figure 3) (Arroyo-Manzanares et al., 2018; Wang et al., 2014).

Most of the substances in Figure 4, such as methyl benzoate, ethyl isovalerate, valeraldehyde, e-2-amyl-aldehyde, hexanal, heptanal, e-2-heptanal, e-2-octanal, e-2-nonanal, benzaldehyde, 2-butanone, 2-heptanone, 2-heptanone, 2-octanone, 6-methyl-5-heptene-2- one, 1-pentanol, 2-hexenol, 2,4,5-trimethylthiazole, etc., were present in relatively high content in MP_{30}, MP_{40} and MP_{40+} (compared with MP_{24}). Among them, the contents of ethyl isovalerate, e-2-octanal, e-2-valeraldehyde, e-2-heptanal,

e-2-nonanal, 6-methyl-5 -heptene-2-one, 2-hexenol, and 2,4,5-trimethyl thiazole were the highest in MP_{30}. The substances in Figure 3 such as propyl acetate, ethyl acetate, n-nonanal, hydroxyacetone, 1-propanol, 1-octanol, benzyl alcohol, 2-5-dimethyl furan, and ethylene sulfide, had the highest contents in MP_{24}. In Figure 5, MP_{24} tended to cluster to the left, whereas MP_{30}, MP_{40} and MP_{40+} tended to move to the right, and the peaks of MP_{24}, MP_{30}, MP_{40}, and MP_{40+} were well separated. The results of principal component analysis showed the proximity of MP_{40} and MP_{40+}, which indicated that the volatile compounds had certain similarity (Ferreira et al., 2019).

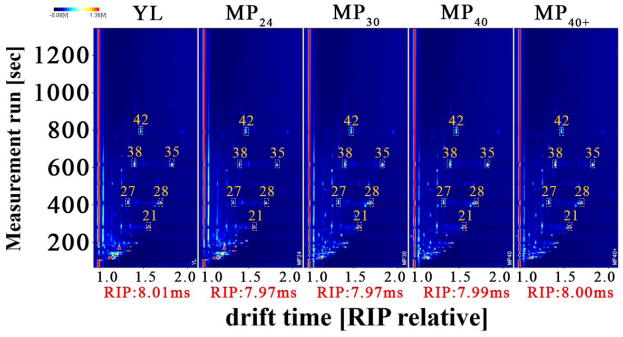

Figure 3. Two-dimensional plots of four components of beef tallow by GC-IMS (YL, MP_{24}, MP_{30}, MP_{40}, MP_{40+}).

In addition to chemical analysis, sensory analysis also plays an important role in food flavor analysis. TDS and TCATA successfully determined the temporal sensory profiling of the reduced salt and fat Bolognas sausages with emulsion gels, with good discrimination and similarity among them (Paglarini et al., 2020). Vidal et al. (2020) applied Q method to meat products processing that found it can be an interesting tool for meat processors, together with the traditional sensory test with consumers, to obtain more consistent and complementary information about meat products (Vidal et al., 2020). The flavor analysis of this study mainly focuses on chemical analysis, and

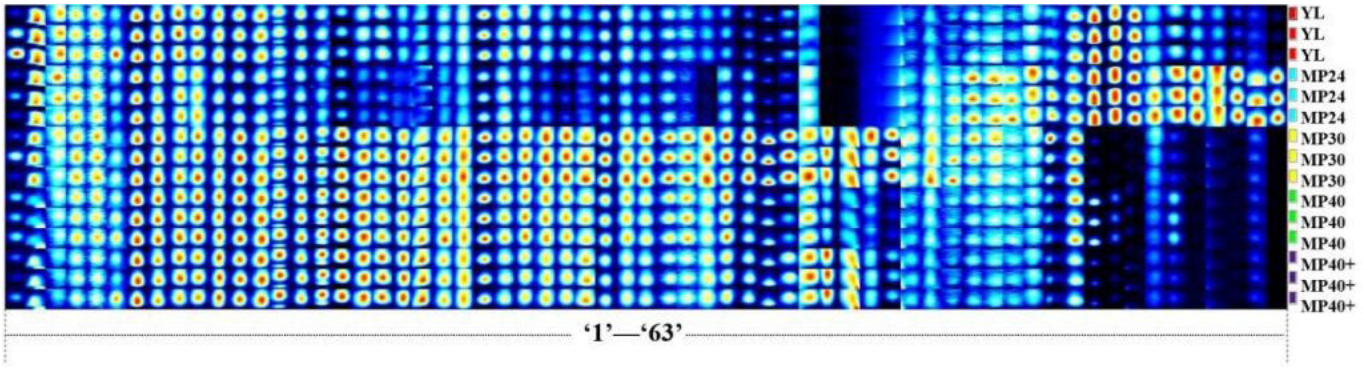

Figure 4. Fingerprint of volatile substances from four components of beef tallow (YL, MP_{24}, MP_{30}, MP_{40}, MP_{40+}).

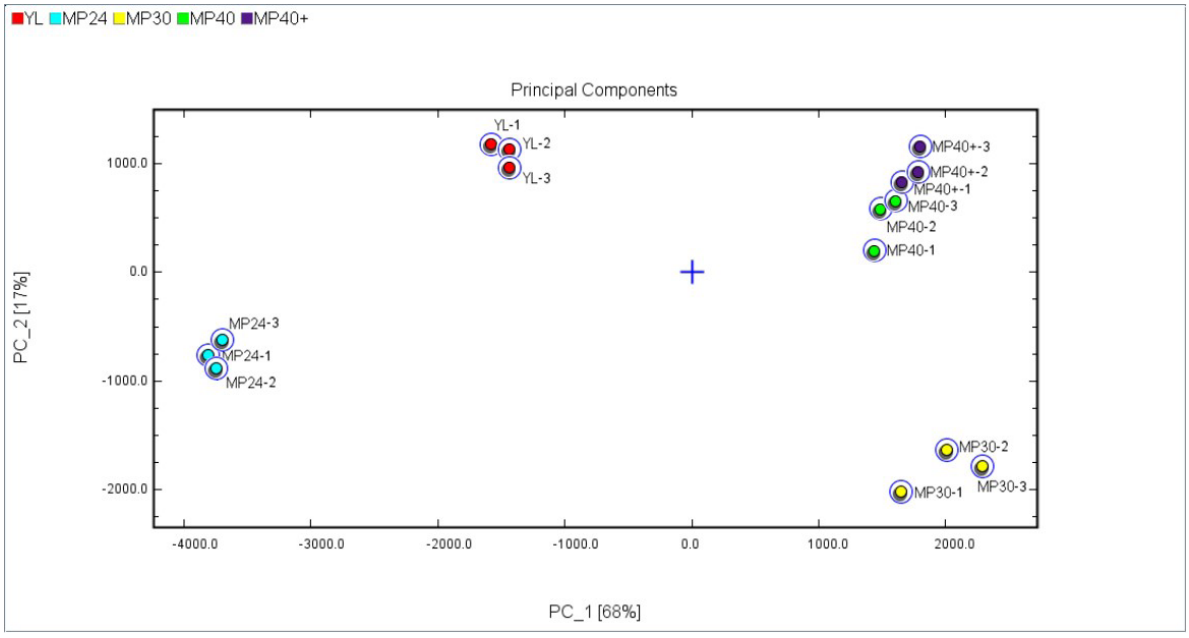

Figure 5. Principal Component Analysis (PCA) diagram of beef tallow (YL, MP_{24}, MP_{30}, MP_{40}, MP_{40+}).

Table 4. Qualitative and quantitative determination of corresponding volatile components in fingerprint.

Serial Number	Compounds/composition number	Retention time/s	relative drift time	Corresponding peak average relative strength/a.u.				
				YL	MP_{24}	MP_{30}	MP_{40}	MP_{40+}
1	Benzyl alcohol	233.4	1.4	186.1	76.4	95.1	128.4	80.7
2	2-Propanone	121.3	1.1	4299.9	3775.9	3295.3	2380.0	2544.2
3	Ethyl isovalerate	396.8	1.3	137.9	118.4	107.9	109.8	87.4
4	ethyl octanoate	1177.4	1.4	336.5	344.5	365.3	341.4	298.4
5	Decanal	1010.7	1.5	182.9	178.2	199.7	189.6	169.7
6	2 - butanone	628.1	1.6	246.2	221.2	244.9	269.8	246.6
7	hexanal D	282.4	1.5	5404.7	5077.5	5755.5	6056.1	6200.7
8	n-Nonanal M	798.3	1.4	2399.1	2289.4	2603.8	2568.5	2358.5
9	Methyl benzoate	768.5	1.2	378.577	323.7	330.0	358.5	367.9
10	benzaldehyde	513.2	1.1	556.4	559.1	612.8	620.4	581.
11	Heptanal M	415.3	1.3	1903.8	1534.2	2097.5	2271.5	2352.3
12	Heptanal D	412.2	1.7	2722.3	2155.5	3335.3	3245.8	3134.8
13	2 - hexylene glycol	620.9	1.4	2420.5	1755.6	2490.2	2643.6	2687.2
14	hexanal M	284.9	1.2	1351.7	1026.3	1526.1	1702.0	1760.0
15	E 2-octenal M	712.1	1.3	566.6	488.8	704.8	669.1	625.6
16	pentanal M	198.1	1.1	596.1	548.8	779.6	937.3	956.2
17	pentanal D	195.9	1.4	1022.4	922.8	2652.9	2424.5	2511.9
18	2-heptanone M	397.8	1.2	542.0	279.5	739.8	764.5	780.3
19	1-pentanol D	250.9	1.5	168.4	86.2	204.1	193.7	224.6
20	2-Hexanone M	270.8	1.1	96.4	66.1	148.1	147.5	151.3
21	1-pentanol M	253.8	1.2	360.7	247.7	567.3	580.4	630.2
22	2-Octanone M	595.5	1.3	304.7	196.9	404.1	406.6	378.0
23	E-2-Heptenal M	519.8	1.2	813.8	577.8	1118.4	1100.9	1088.0
24	Hexanal	617.3	1.8	2533.1	1886.9	3082.0	2987.5	2578.3
25	E 2-nonenal	901.1	1.4	404.8	357.9	465.4	432.5	361.8
26	E-2-octena	661.0	1.2	300.3	207.3	376.9	370.1	330.6
27	ethyl isovalerate	409.9	1.7	786.1	421.8	1156.5	1103.5	1011.7
28	2-heptanone D	394.9	1.6	496.2	202.2	953.8	849.1	771.8
29	2-hexenol M	346.3	1.2	330.6	217.5	688.5	596.2	657.3
30	1 - propanol	588.3	1.3	827.7	575.5	1159.2	992.2	864.5
31	2_4_5-Trimethylthiazole	513.2	1.6	226.5	182.7	300.2	270.1	244.1
32	E-2-Heptenal D	512.9	1.7	324.5	280.5	619.2	489.4	444.2
33	E 2-octenal D	711.8	1.8	88.8	78.1	124.6	110.8	96.1
34	2-Octanone D	590.5	1.8	64.7	54.4	113.9	106.1	78.2
35	2-Hexanone D	269.9	1.5	76.2	23.8	176.5	127.7	136.5
36	6-methyl-5-heptene-2-ketone	483.2	1.2	140.7	194.1	342.8	328.2	288.1
37	E-2-Pentenal	239.8	1.4	277.1	520.7	1035.8	604.1	645.7
38	2-hexenol D	347.1	1.5	222.2	162.3	703.5	442.7	472.3
39	2-butanone	148.4	1.2	612.8	407.0	1581.4	689.8	1174.5
40	Propionic acid	230.295	1.1	60.1	79.0	154.8	131.9	171.0
41	3-methylbutanal	172.9	1.4	22.9	17.2	473.2	255.1	496.0
42	2-methylbutanal	178.2	1.4	40.8	22.9	408.4	206.4	383.6
43	1-Butanol	165.2	1.2	29.8	34.5	197.9	92.8	142.6
44	2-methylbutyraldehyde	171.2	1.2	88.8	103.3	267.7	52.5	130.1
45	hexanal M	787.0	1.2	82.9	92.0	119.2	90.7	88.9
46	2-hexanone D	638.6	1.2	172.9	201.1	279.1	193.2	181.1
47	E-E 2-4-heptadienal M	605.9	1.2	81.3	108.8	110.1	89.8	85.9
48	2 - heptyl ketone D	714.5	1.5	97.9	140.5	128.5	99.3	93.3
49	Heptyl aldehyde M	525.5	1.4	360.9	439.7	402.7	374.9	301.5
50	Ethyl acetate	584.2	1.2	75.2	92.2	76.6	82.4	88.2
51	1-Octanol	613.9	1.5	328.3	412.5	332.9	293.1	275.1
52	decanal	142.5	1.2	978.7	1804.5	1323.5	958.9	677.2

YL = (yuan liao) Raw Materials; a.u. = represent relative intensity of corresponding peak.

Table 4. Continued...

Serial Number	Compounds/composition number	Retention time/s	relative drift time	Corresponding peak average relative strength/a.u.				
				YL	MP_{24}	MP_{30}	MP_{40}	MP_{40+}
53	n-Nonanal D	795.9	1.9	584.7	570.3	630.9	605.9	474.7
54	Hydroxyacetone	169.8	1.2	9528.8	10299.7	737.5	2897.1	918.8
55	Ethyl Acetate	155.2	1.3	4804.3	5541.8	155.4	974.9	285.2
56	2 - heptyl ketone M	130.7	1.2	3996.4	3687.1	401.7	419.4	453.1
57	Benzyl alcohol	617.9	1.5	622.4	955.3	515.3	424.8	373.5
58	1-propanol	136.3	1.3	502.6	825.8	166.7	305.9	168.1
59	2-5-dimethyl-furan	177.6	1.4	411.5	858.6	45.9	43.5	44.9
60	Ethylsulfide	208.1	1.4	100.8	267.5	42.9	37.8	37.5
61	2 - hexylene glycol alcohol	177.7	1.5	54.9	207.4	17.5	15.2	14.9
62	amyl alcohol	403.7	1.2	73.1	212.2	69.9	65.4	69.9
63	propyl acetate	209.2	1.5	126.1	1539.4	26.7	23.7	23.2

YL = (yuan liao) Raw Materials; a.u. = represent relative intensity of corresponding peak.

the next step will focus on the sensory analysis and chemical analysis combined to analyze the flavor components of beef tallow.

4 Conclusion

In this study, beef tallow components were obtained by step-by-step cooling and drying fractionation technology. The beef tallow with melting points of 22.10, 34.17, 46.83 and 50.77 °C was extracted from the beef tallow with melting points of 46.5 °C by dry fractionation. The acid value and iodine value of each component increased with the melting point, and the cholesterol content was negatively correlated with the melting point. The content of unsaturated fatty acid in MP_{24} increased by 5.48% compared with the raw material and was the highest. MP_{24} had the lowest melting point, the second most oil yield after MP_{40} and the crystal with minimum stability. The oil yield of MP_{30} was lower than that of MP_{24}, the content of unsaturated fatty acid was slightly lower than that of MP_{24}, the melting point was higher than that of MP_{24}, and the crystal stability was slightly higher than that of MP_{24}. However, MP_{30} has more comprehensive characteristic aroma of beef tallow.

The volatile substances of the beef tallow was analyzed by headspace gas chromatography-ion mobility spectrometry (GC-IMS). The experimental results showed that with the decrease in fractionation temperature, the content of saturated fatty acids displayed a decreasing trend, while unsaturated fatty acids displayed an increasing trend. MP_{24} exhibited the highest content of unsaturated fatty acids,which volatile components were mainly small molecules of esters, aldehydes, ketones and alcohols. The unsaturated fatty acid content of MP_{30} was slightly lower than that of MP_{24}, and MP_{30} showed a more comprehensive composition with the characteristic aroma of beef tallow. The volatile components in MP_{40+} and MP_{40} were mainly small molecules including esters, aldehydes, ketones and alcohols, which were suitable for the products with high stability requirements. In this study, Ion migration chromatography fingerprint library of the characteristic components of beef tallow was also constructed, and the generated GC-IMS two-dimensional spectrum was used to analyze the difference characteristics of beef tallow. This study provides theoretical guidance for the physical and chemical properties of beef tallow and a reliable basis for its applications.

Acknowledgements

The Project Supported by Tianjin Science and Technology Major Project (No. 18ZXYENC00120), the Tianjin Science and Technology planning project grant number (No. 21YDTPJC00840), and Tianjin Municipal Education Commission Research Project (No. 2020 KJ090).

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