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Pre-regulation of the water content impacts on the flavor and harmful substances of sesame paste

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ABSTRACT

In this study, the influence of pre-regulation of the water content (5–25 %) on the harmful substances and aroma compounds of sesame paste (SP) was investigated. The results indicated that pre-regulation of the water content reduced the generation of harmful substances in SP. Notably, the total heterocyclic amine content in SP-15 was significantly lower than in other samples. SP-10 had the lowest total polycyclic aromatic hydrocarbon content, while SP-5 exhibited the lowest PAH4 content. Using solvent-assisted aroma evaporation and GC-O-MS, 50 aroma compounds were identified in SP. Pre-regulation of water content in SP led to an elevated concentration of heterocyclic compounds thereby imparting a diverse aromatic profile. It enhanced the perceived intensity of roasted sesame and salty pastry aromas while reducing the perceived intensity of fermentation and burnt aromas. The findings suggested the pre-regulation of the water content played a crucial role in aroma modulation and harmful substances control in SP.

1. Introduction

Sesame (*Sesamum indicum* L.), an important oilseed crop, is widely cultivated in China, Ethiopia, Bolivia, Africa, Central and South America, and other parts of Asia (Liu et al., 2022). Sesame paste (SP), also known as “tahini”, is a highly nutritious semi-solid food obtained by grinding heat-treated sesame seeds. It has gained immense popularity worldwide, particularly in Eastern countries and Mediterranean coastal countries (Xu, Guan, Lin, Li, & Wang, 2021).

The production of a special flavor of SP is highly dependent on the heat treatment of sesame seeds, which induces a Maillard reaction that improved the sensory qualities of SP (Starowicz & Zieliński, 2019). On the other hand, heat treatment may produce harmful substances such as polycyclic aromatic hydrocarbons (PAHs) and heterocyclic amines (HCAs) (Liu, Yang, Shi, & Li, 2022; Zhang et al., 2022). How to balance the production of flavor and harmful substances in the Maillard reaction is an issue of concern. Existing articles demonstrate that the regulation of the water content of raw materials is closely related to heat treatment and is a key factor in producing pleasant aromas and tastes after heat treatment (Hou, Zhang, & Wang, 2019; Xu et al., 2019).

When water is added to sesame seeds, it migrates from the outside to the inside due to the existence of a water gradient. As the expansion after water absorption varies in magnitude and timing, the stress is generated by strain, causing a certain degree of displacement at the interface and forming tiny cracks in the cross-section. This reduces the resistance of sesame seeds to damage, making them easily crushed, and decreasing energy consumption. (Xu et al., 2019) found that the variety and content of volatile compounds in sesame oil were increased by regulating the water content of sesame seeds, favoring a strong nut-like aroma. (Baggenstoss, Perren, & Escher, 2008) showed that as the water content increased, the degradation of the aroma compounds accelerated and the aroma became less stable. However, the influence of water content on SP has not been reported. Therefore, exploring the impact of water content on the quality of SP is essential and critical for the development of SP.

Extracting flavor compounds is critical to analyzing the sensory characteristics of food products. Currently, there are three main methods used for flavor extraction: headspace solid-phase microextraction (HS-SPME), simultaneous distillation extraction (SDE), and solvent-assisted flavor evaporation (SAFE). Usually, HS-SPME is limited by its low separation efficiency and susceptibility to interference from impurities, while SDE is time-consuming and may lead to changes in flavor compounds due to heating. In contrast, SAFE is a highly efficient

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extraction method that has advantages such as high extraction efficiency and environmental friendliness, and increasingly being used for flavor analysis, providing the most representative flavor profile of food products (Sarhir, Amanpour, & Selli, 2019). In our study, SAFE is applied for the first time to extract flavor compounds from SP, contributing to a more comprehensive understanding of the flavor compounds of SP and the optimization of its flavor during processing.

While the empirical evidence from factories suggests that adjusting the water content is beneficial for the flavor formation and quality control in SP, scientific evidence is largely lacking. The aim of our study is to explore the contribution of pre-regulation of the water contents to the sensory and nutritional quality of SP, as well as its effect on the degree of the Maillard reaction and harmful substances in SP.

2. Material and methods

2.1. Materials and reagents

The Zhuzhi-22 sesame seeds used in this experiment were acquired from the Minxing Agricultural Technology Co., Ltd (Zhengzhou, P.R. China). They were harvested in 2022 and stored at 4 °C.

The *n*-alkanes solution (C5–C30) was sourced from Agilent Technology Co., Ltd (California, USA). An internal standard (4-nonanol) was supplied by Shanghai Macklin Biochemical Co., Ltd (P.R.China). The mixed standard of 16 PAHs, including Ant-d10, Acy-d8, Ace-d10, BaP-d12, BaA-d12, BbF-d12, BgP-d12, BkF-d12, Chr-d12, DhA-d14, Flu-d10, Flua-d10, IcP-d12, Nap-d8, Phe-d10, and Pyr-d10, was obtained from Organic Standards Solutions International LLC (South Carolina, USA). 1-methyl-9H-pyrido[3,4-*b*]indole (Harman) and 9H-pyrido[3,4-b] indole (Norharman) were purchased from Santa Cruz Biotechnology, Inc. (Texas, USA). A high purity level (≥95 %) was maintained for all other chemicals.

2.2. Preparation of SP

To attain various moisture levels, sesame seeds were sprayed with distilled water and then sealed in plastic bags. These bags were shaken vigorously for 10 min. Subsequently, the bags containing the seeds were placed in an incubator at 25 °C for 6 h to allow the water within the seeds to equilibrate. As a result of this process, the moisture content in the sesame samples was determined to be 4.99 %, 9.84 %, 14.83 %, 19.88 %, and 25.27 %, respectively. In the text, these samples are referred to as SP-5, SP-10, SP-15, SP-20, and SP-25.

To prepare the SP samples, sesame seeds at each moisture level were roasted in an electric roasting pan (ENG002, Weihai Hanjiang Food Co. Ltd, Shandong) at 175 °C for 40 min. After roasting, the sesame seeds were allowed to cool to room temperature. Following this, the sesame seeds were ground into a paste using a colloid mill (Dongchen JM-L80, Dongchen Fluid Equipment Co. Ltd, Zhengzhou, Henan). The resulting sesame paste samples were then stored at −20 °C for further analysis.

2.3. The proximate composition of SP

The water content and crude fat of the SP were determined in accordance with the method of (Jin et al., 2022). Briefly, SP was dried in an oven at a temperature of 103 ± 2 °C until the change in weight was within 0.1 mg. To determine the water content, the sample was reweighed. For the determination of crude fat, in brief, the sample (5 g) was defatted in a Soxhlet extractor with petroleum ether (150 mL) for 8 h. The flasks were then dried in an oven (103 °C, 60 min) until stable weights were reached.

The protein content of the SP was determined according to a method described by (Jin et al., 2022). In short, a digestion tube was filled with each sample (0.2–1 g). Potassium sulfate (4 g) was added as a catalyst. The digestion was carried out at 420 °C for 40 min, and t the amount of nitrogen was calculated by titrating to gray with hydrochloric acid.

Total sugar content and γ-Tocopherol was determined using the method described by (Yin et al., 2022). Briefly, the defatted sesame meal was treated with hydrochloric acid (25 mL) and heated in a boiling water bath for 40 min. Afterwards, a mixture of phenol (1 mL) and sulfuric acid (5 mL) was added. The absorbance values at 490 nm were measured for both the sample solution and a blank. The total sugar content was determined using a glucose calibration curve. γ–Tocopherol: 0.1 g of sesame oil was accurately weighed and subjected to ultrasonication in hexane for 20 min. After cooling, the solution was filtered through a 0.22 μm membrane and subsequently injected into an HPLC system. The mobile phase consisted of isopropanol and hexane in a ratio of 1:99, and the flow rate was set at 1.0 mL/min. A 10 μL volume was injected, and the column temperature was maintained at 40 °C. Detection was performed using a fluorescence detector with excitation and emission wavelengths set at 298 nm and 325 nm, respectively.

2.4. Determination of color and browning strength

The color of SP was analyzed based on (Jin et al., 2022). The values were measured with a CR-400 colorimeter (Konica Minolta, Japan). The measured values were expressed as L^* (brightness), a^* (redness or greenness), and b^* (yellowness or blueness). The ΔE (total color difference) was determined as follows:

$$\Delta E = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{1/2} \tag{1}$$

where Δa^*, Δb^*, and ΔL^* represent the difference between SP samples (5, 10, 15, 20, and 25 %) and standard color palettes.

Browning strength: The determination method was slightly modified from that described by (Guo et al., 2023). The sample (1 g) was added to chloroform (5 mL), vortexed for 1 min, then centrifuged (4500 rpm, 15 min), filtered and the supernatant collected. The ultraviolet–visible spectrophotometer (UV-2401PC, Shimadzu Co., Ltd., Japan) was used to record the absorbances at 294 nm and 420 nm. Chloroform served as the control substance.

2.5. Determination of free amino acids

Following the method described by (Guo et al., 2022), an automatic amino acid analyzer (Hitachi L-8900, Tokyo, Japan) was employed to determine the free amino acid composition of the sample. Approximately 1 g of defatted SP was weighed and extracted with 50 mL of 0.01 M HCl in a container, followed by filtration with filter paper. The supernatant was mixed with 1:1 (v:v) 8 % sulfosalicylic acid and allowed to stand for 15 min before being centrifuged at 10,000 rpm for 10 min. Finally, the solution was passed through a filter membrane (0.22 μm). A volume of 20 μL of the solution was used for amino acid analysis.

2.6. Determination of HCAs

In accordance with the procedure outlined by (Zhang, Xi, Zhao, Ma, & Wang, 2020), solid phase extraction of HCAs from SP was conducted. The HCAs were subsequently analyzed using an E2695 HPLC system manufactured by Waters, USA, following the method described by (Wang et al., 2022). The HPLC system employed a Waters Sun Fire C18 column with dimensions of 250 mm x 4.6 mm x 5 μm. The mobile phase consisted of 85 % ammonium formate (10 mmol/L, pH = 3) and 15 % acetonitrile, with a flow rate of 0.8 mL/min. The column temperature was maintained at 30 °C, and the injection volume was 10 μL. Detection of HCAs was performed at an excitation wavelength of 300 nm and an emission wavelength of 440 nm. The concentration of HCAs was determined using an external standard method.

2.7. Determination of PAHs

Following the method outlined by (Ji, Liu, & Wang, 2020), the

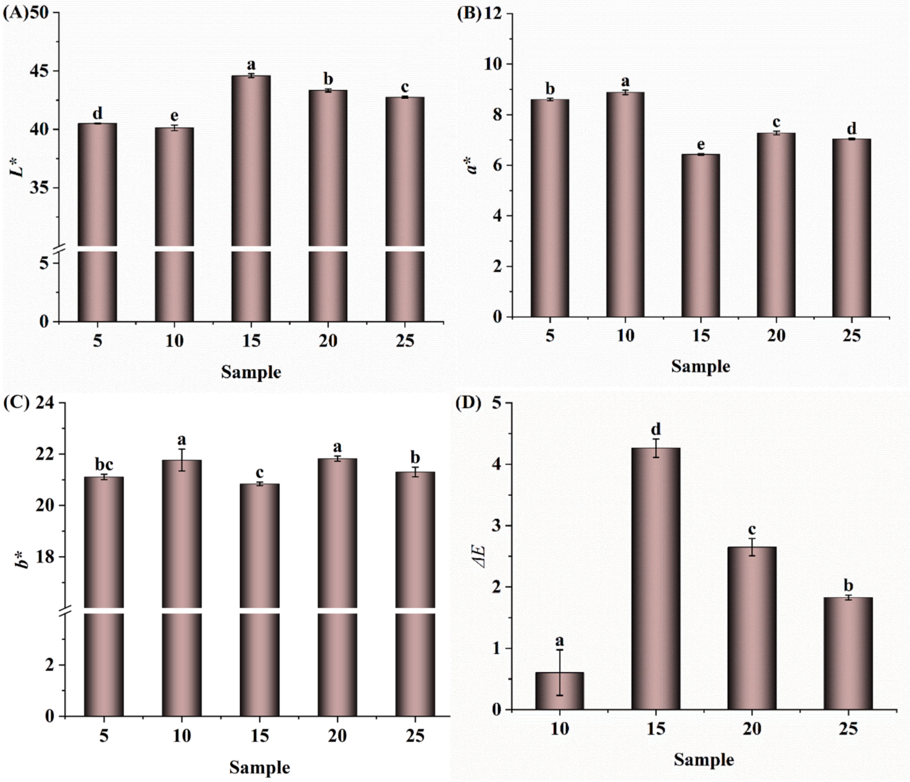

Fig. 1. Color difference of SPs prepared from the sesame seeds with different moisture contents. Note: (A) L* (lightness); (B) a* (redness/greenness); (C) b* (yellowness/blueness); (D) ΔE (total color difference). 5, 10, 15, 20, and 25, the moisture content of sesame seeds before roasting in SP, respectively. The different small letters represent the significant difference ($P < 0.05$).

analysis of PAHs (polycyclic aromatic hydrocarbons) was conducted. The PAHs present in the SP were extracted using a solid-phase extraction tube (Merck KGaA, Germany). The extracted samples were then analyzed using an E2695 HPLC system equipped with a fluorescence detector (FD) and an automatic sampler. A Waters PAH C18 column with dimensions of 250 mm × 4.6 mm × 5 μm was utilized in the analysis. The mobile phases consisted of acetonitrile and water, with a flow rate of 1 mL/min. The elution gradient conditions were as follows: 0–5 min, 50 % acetonitrile; 5–20 min, a linear increase from 50 % to 100 % acetonitrile; 20–28 min, 100 % acetonitrile; 28–32 min, a linear decrease from 100 % to 50 % acetonitrile. The column temperature was maintained at 30 °C, and the injection volume was set to 20 μL. The concentrations of PAHs were determined using the external standard method.

2.8. Extraction of volatile components from SP by SAFE

Slightly modified from the method of (Yin et al., 2021). SP was weighed 15 g, added to dichloromethane (150 mL) and 150 μL (1 mg/mL) of 4-nonanol, shaken for 10 h in a constant temperature shaker (30 °C, 130 rpm/min), centrifuged three times (4000 rpm, 15 min) and the supernatant was retained every time. The supernatant was poured into the dropping funnel on the left side of the SAFE unit and the temperatures of both the water bath and the thermostatic circulating water bath were set at 50 °C. After the samples were extracted, the extracts were dried with anhydrous sodium sulfate and then placed in a refrigerator overnight (-24 °C, 12 h). The extract was then concentrated to 5 mL using a Wechsler column and then concentrated to 1.5 mL by nitrogen blowing, filtered through a 0.22 μm organic filter membrane, fed into a gas phase vial, and frozen (-24 °C) until further analysis.

2.9. GC-O-MS analysis of the aroma active ingredients of SP

Following the method described by (Yin et al., 2022), the identification of aroma-active compounds in SP was conducted. The analysis involved the use of a 7890B/5977 gas chromatograph (GC) coupled with a mass spectrometer (MS) from Agilent, as well as ODP-3 olfactometry (Gerstel Inc., Germany). For the separation of volatile components, an Agilent HP-5MS capillary column with dimensions of 30 m × 0.25 mm × 0.25 μm was employed. The GC oven temperature program was set as follows: initially held at 40 °C for 5 min, then increased to 130 °C at a rate of 3 °C/min (held for 5 min), and finally increased to 250 °C at a rate of 10 °C/min (held for 5 min). Helium with a purity of $\geq$ 99.999 % served as the carrier gas at a flow rate of 1.8 mL/min. The effluent from the GC column was split equally between the MS and ODP in a 1:1 (V:V) ratio. The temperatures of the GC inlet, quadrupole, ion source, and ODP were set at 250 °C, 150 °C, 230 °C, and 280 °C, respectively. The volatile compounds were analyzed using electron impact mode (70 eV) to generate mass spectra. The mass spectrometer scanned the mass-to-charge ratio (m/z) range from 33 to 400 at a rate of 2.0 scans per second. The aroma perception and description were conducted by three trained panel members (1 male and 2 females) using ODP. Aroma-active compounds were recorded when perceived by more than two panel members. To enhance their comfort, water was delivered to their nostrils through a separate capillary column.

The volatile compounds were identified by comparing their mass spectra with the NIST 17 library, and the retention indices (RIs) of the volatile compounds were obtained by running a series of n-alkanes (C5-C30). The semi-quantification of aromatic compound in the SAFE extracts was conducted by comparing the response area of the volatile compound and the internal standard area (Selli, Guclu, Sevindik, & Kelebek, 2021).

2.10. Determination of sensory quality

A team of 12 trained panelists (5 males and 7 females) from the Henan University of Technology evaluated the sensory characteristics of SP samples. The average age of the panelists was 24 ± 2 years, and all had at least one year of experience in sensory evaluation of sesame oil, SP, and other food products. Prior to this study, all participants acknowledged their informed consent and their rights and privacy were ensured. SP samples (15 g) were poured into brown glass bottles with covers and equilibrated at 35 °C in an oven for 0.5 h to allow the aroma to disperse. The samples were then labelled with random three-digit codes. Each panelist smelled and tasted each sample, and evaluated its sensory attributes using a 15-point scale. After tasting each sample, the panelists chewed an apple slice for 15 s and rinsed their mouths with water before resting for at least 3 min before evaluating the next sample. All sensory tests were conducted in individual booths in a testing room designed in accordance with the requirements of ISO 8589:2007.

2.11. Statistical analysis

Analyses were performed in triplicate and results were expressed as mean ± standard deviation (SD). One-way analysis of variance (ANOVA) and Waller Duncan's multiple comparison tests were performed using SPSS software, with significant differences at $p < 0.05$. IBM SPSS statistics 26.0 software (Chicago, USA).

3. Results and discussion

3.1. The proximate composition and γ-Tocopherol of SP

Supplementary S1 demonstrated that the water contents of all SPs met China's standards, which were less than 1 %. SP-15 had the lowest fat content (54.63 g/100 g), probably because insufficient friction during the grinding process, the sesame tissue did not break seriously, resulting in a decrease in the fat content of SP-15 (Martínez, Bordón, Lallana, Ribotta & Maestri, 2017). SP-25 had the lowest total sugar content (6.08 g/100 g). This observation might be attributable to the potential dilution of sugar content in sesame during the production process, leading to a decreased sugar content per unit volume in the final SP. There was no significant difference in crude protein content ($p <$ 0.05).

The γ-Tocopherol was a major antioxidant component in SP. It was found that it ranged from 169.58 mg/kg to 294.94 mg/kg ($p < 0.05$). The highest γ-Tocopherol content was observed in SP-15 (294.94 mg/kg), which suggested that moderate water content was a key factor for maintaining the γ-Tocopherol and thus the quality and nutritional value of SP.

3.2. Effect of the water content pre-regulation on the color of SP

Fig. 1 demonstrated the color variations between the samples. Fig. 1A showed that there were significant differences in the L* values of all samples ($p < 0.05$), with the highest L* value for SP-15 and the lowest L* value for SP-10, indicating that lower water content (10 %) of sesame seeds prior to roasting could benefit the Maillard reaction and thus yielded a darker color. Fig. 1B revealed significant differences in the a* values of all samples ($p < 0.05$). SP-5 and SP-10 had the highest a* values, indicating that they formed brown pigments through the Maillard reaction, which was consistent with the study of (Guo et al., 2022). Fig. 1C indicated that there were significant differences between the adjacent samples in b* values ($p < 0.05$). Fig. 1D has shown the significant differences in ΔE values for all samples ($p < 0.05$), indicating that the water content of the raw material had a very important effect on the color of the SP. Obviously, the largest ΔE value was found for SP-15, indicating when the water content of the sesame seeds reached 15 %, the intensity of the Maillard reaction was increased. It was worth noting that a certain amount of water was required for the occurrence of the Maillard reaction, and either too high (25 %) or too low water (5 %) was detrimental to the reaction (Eichner & Karel, 1972). Too low a water content resulted in poor mobility of the reactants and too high a water content resulted in too low a concentration of reactants, both of which reduced the effective frequency of intermolecular collisions and had an inhibitory effect on the reaction.

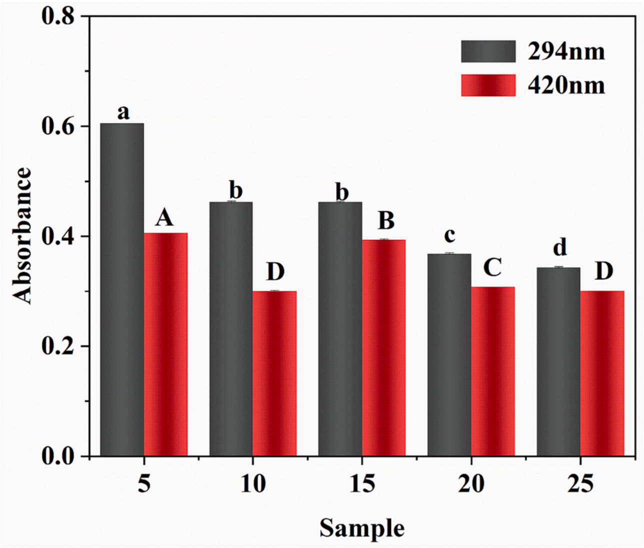

Fig. 2. Absorbance of SPs prepared from the sesame seeds with different moisture contents. Note: 5, 10, 15, 20, and 25, the moisture content of sesame seeds before roasting in SP, respectively. The different small letters represent the significant difference at the 294 nm ($P < 0.05$). The different big letters represent the significant difference at the 420 nm ($P < 0.05$).

3.3. Browning intensity

At 294 nm, the formation of non-enzymatic brown intermediate compounds in the early stage of the Maillard reaction was represented, while at 420 nm, the polymerization and degradation of intermediate compounds in the later stage culminated in the generation of brown pigments, namely advanced glycation end-products(AGEs) (Guo et al., 2022). From Fig. 2, it could be observed that the absorbance values of SP significantly decreased ($p < 0.05$) at 294 nm and 420 nm with the water content increased. The absorbance at 420 nm was weaker than that at 294 nm, which was consistent with the findings of (Yin et al., 2022). These results indicated that when SP-10 was roasted at 175 °C for 40 min, it was more susceptible to the early stages of the Maillard reaction and slowly progresses to the later stages. SP-5 showed a significantly higher absorbance value at 294 nm than other SPs ($p < 0.05$), indicating that reducing the water contents of sesame seeds easily led to the formation of early Maillard reaction products. However, SP-5 and SP-15 exhibited higher absorbance values at 420 nm. When the appropriate moisture contents were maintained for sesame seeds prior to roasting, water was fully absorbed by the protein, and molecular mobility of the protein was increased, which resulted in the expansion of inter- and intra-molecular molecular rearrangements and caused increased browning. In addition, all SPs had much lower absorbance values at 420 nm than at 294 nm, indicating the presence of more non-enzymatic browning intermediate compounds than melanoidins.

3.4. Effect of water content regulation on harmful substances in SP

3.4.1. HCAs

Heterocyclic amines (HCAs) are carcinogenic and mutagenic compounds that are typically generated by the pyrolysis of proteins or amino

Table 1
Effect of moisture content regulation on harmful substances of SPs;

	Concentration of PAHs and HCAs (ng/g)				
	5[1]	10[1]	15[1]	20[1]	25[1]
HCAs					
Norharman[2]	59.92 ± 0.07a	61.09 ± 0.24a	0.95 ± 0.08d	3.40 ± 0.29c	14.42 ± 1.77b
Harman[2]	23.55 ± 0.07a	23.28 ± 0.55a	0.78 ± 0.00d	2.75 ± 0.09c	20.74 ± 0.86b
Total content[2]	83.47 ± 0.11a	84.37 ± 0.32a	1.72 ± 0.08d	6.15 ± 0.37c	35.16 ± 2.63b
PAHs					
Naphthalene[2]	ND	ND	ND	ND	0.67 ± 0.03
Acenaphthene[2]	0.09 ± 0.01b	1.03 ± 0.06a	ND	ND	ND
Fluorene[2]	11.58 ± 0.59a	9.70 ± 1.05a	9.95 ± 1.42a	10.42 ± 1.53a	6.50 ± 0.69b
Phenanthrene[2]	23.48 ± 1.06b	ND	56.54 ± 5.3a	20.30 ± 2.14b	2.97 ± 0.16c
Anthracene[2]	1.15 ± 0.17c	ND	1.89 ± 0.11b	11.38 ± 0.49a	0.95 ± 0.04c
Fluoranthene[2]	57.79 ± 1.22a	7.08 ± 0.38c	5.30 ± 0.22d	2.54 ± 0.31e	10.55 ± 0.61b
Pyrene[2]	0.40 ± 0.04b	0.20 ± 0.03c	0.22 ± 0.01c	0.08 ± 0.00d	0.78 ± 0.05a
Benzo[a] anthracene[2]	2.27 ± 0.34c	6.52 ± 0.59a	2.06 ± 0.07c	2.08 ± 0.12c	4.77 ± 0.27b
Chrysene[2]	1.11 ± 0.12d	ND	6.93 ± 0.68a	3.20 ± 0.22c	4.07 ± 0.22b
Benzo[k] fluoranthene[2]	0.30 ± 0.00	ND	ND	ND	ND
Dibenzo [a, h] anthracene[2]	ND	ND	ND	ND	1.06 ± 0.02
Total content[2]	98.17 ± 2.27a	24.54 ± 0.75e	82.89 ± 0.41b	50.11 ± 4.01c	32.32 ± 0.15d
PAH4[2]	3.37 ± 0.46d	6.52 ± 0.59b	8.99 ± 1.05a	5.27 ± 0.34c	8.83 ± 0.05a

[1] SP made from sesame seeds with a moisture content of 5, 10, 15, 20 and 25%, respectively.

[2] Significant differences within each group are indicated by different superscripts ($P < 0.05$).

Table 2
The content of free amino acids in SPs (mg/100 g).

Free amino acid[2]	5[1]	10[1]	15[1]	20[1]	25[1]
Asp	23.30 ± 0.06a	19.67 ± 0.49c	21.43 ± 0.59b	19.63 ± 0.84c	17.99 ± 0.03d
Thr	ND	ND	ND	6.18 ± 0.05a	4.53 ± 0.04b
Ser	21.06 ± 0.98c	17.55 ± 0.27d	46.51 ± 1.46a	29.90 ± 2.19b	28.21 ± 0.84b
Glu	21.18 ± 0.69b	8.34 ± 0.65d	22.62 ± 0.41a	13.91 ± 0.57c	8.95 ± 0.14d
Gly	8.09 ± 0.58c	7.21 ± 0.12d	11.29 ± 0.23b	10.66 ± 0.86b	15.05 ± 0.18a
Ala	10.41 ± 0.15d	10.55 ± 0.25d	23.07 ± 0.05c	26.78 ± 1.63b	31.19 ± 0.73a
Cys	16.36 ± 0.05d	21.34 ± 0.16c	22.45 ± 0.05b	21.09 ± 0.49c	24.70 ± 0.13a
Val	25.08 ± 0.42a	3.13 ± 0.24d	10.51 ± 0.18c	10.23 ± 0.08c	12.42 ± 0.35b
Ile	5.27 ± 0.14c	2.04 ± 0.03d	7.31 ± 0.46b	10.18 ± 0.90a	10.83 ± 0.62a
Leu	5.18 ± 0.12c	2.03 ± 0.49d	7.31 ± 0.64a	6.67 ± 0.09b	7.76 ± 0.02a
Tyr	5.52 ± 0.11c	5.01 ± 0.07d	11.06 ± 0.27a	9.58 ± 0.57b	10.92 ± 0.01a
Phe	13.75 ± 0.04c	9.34 ± 0.43d	25.91 ± 2.24a	16.08 ± 0.64b	15.92 ± 0.10b
His	11.65 ± 0.47 cd	11.16 ± 0.88d	15.90 ± 1.28a	13.78 ± 0.53b	12.81 ± 0.92bc
Lys	12.81 ± 0.06b	10.90 ± 0.94c	20.65 ± 1.37a	5.94 ± 0.47d	6.45 ± 0.24d
Arg	24.08 ± 0.53d	20.24 ± 0.45e	43.18 ± 1.06a	35.67 ± 1.75c	40.80 ± 0.30b
Pro	4.50 ± 0.05d	0.39 ± 0.03e	12.06 ± 0.55a	5.98 ± 0.08c	10.40 ± 0.10b
Met	ND	ND	6.64 ± 0.05	ND	ND
TFAA[3]	208.25 ± 2.17d	148.89 ± 0.83e	307.89 ± 0.40a	242.27 ± 2.02c	258.92 ± 0.8b
EAA[3]	53.38 ± 0.05c	45.64 ± 1.68d	71.69 ± 2.15a	66.15 ± 2.31b	70.20 ± 0.20a
NEAA[3]	154.87 ± 2.22d	103.25 ± 2.48e	236.2 ± 1.76a	176.13 ± 0.29c	188.72 ± 1.00b
EAA/ TFAA (%)	25.63 ± 0.29c	30.66 ± 1.29a	23.28 ± 0.67d	27.30 ± 0.73b	27.11 ± 0.16b

[1] SP made from sesame seeds with a moisture content of 5, 10, 15, 20 and 25%, respectively.

[2] Significant differences within each group were indicated by the different superscripts ($P < 0.05$). ND: the free amino acid was not detected.

[3] EAA: Essential amino acids; NEAA: Non-essential amino acids; TFAA: Total free amino acids.

acids at high temperatures, especially in foods with high protein content (Liu, Yang, Shi, Cui, & Li, 2021). As shown in Table 1, the total amount of HCAs in SP-10 was the highest (84.37 ng/g), while SP-15 had the lowest amount (1.73 ng/g), indicating that the proper water control of sesame seeds could reduce the concentration of HCAs in SP. (Liu et al., 2021) found that the reduction of Harman and Norharman in oil blends during heating was mainly due to the oxidation degradation reaction between β-carbolines (Harman and Norharman) and lipid oxidation products. Therefore, the reason for the sharp decrease in the content of β-carbolines in SP-15 might be the rapid oxidative degradation of β-carbolines under aerobic conditions during heating. Both Norharman and Harman contents were high in SP-5 and SP-10, which might be attributed to the low water content in sesame seeds and the viscous texture during grinding, making it difficult for the heterocyclic amine to mix thoroughly with water molecules. On the other hand, with the increase of water content in sesame seeds, the texture of SP during grinding was found to be less viscous, and the heterocyclic amine molecules had the ability to form hydrogen bonds with the oxygen atoms in water molecules, making them soluble in water and leading to a decrease in their contents.

3.4.2. PAHs

PAHs are a group of abundant, complex, and widely distributed carcinogens that pose a significant threat to human health (Ji, Jiang, Zhang, Hou, & Sun, 2022). Due to their lipophilic nature, the fatty foods are easily contaminated by PAHs.

The study revealed that the total content of PAHs in the sample ranged from 24.54 ng/g to 98.17 ng/g, with the lowest PAH content found in SP-10 (24.54 ng/g). However, it was worth noting that although SP-5 had the highest total content of PAHs, its PAH4 (including benzo[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, and benzo[*a*] pyrene) content was the lowest (3.37 ng/g). The fluoranthene was significantly higher in the SP-5 sample compared to others ($p < 0.05$). This difference could be attributed to the changes in roasting temperature or other conditions resulting from increased moisture in sesame seeds, which might influence the stability of fluoranthene. Under unfavorable conditions, fluoranthene could undergo decomposition, degradation, or reactions, leading to a decrease in its concentration. Further investigations indicated that during thermal processing if the temperature was excessively high, the pyrolysis of organic components such as oil, proteins, and carbohydrates inevitably generated PAHs (Ji, Jiang, Zhang, Hou, & Sun, 2022). Therefore, we hypothesized that sesame seeds with lower moisture content might experience higher temperatures during the roasting process, increasing the concentration of PAHs (Liu, et al., 2022; Xu et al., 2019).

It was worth noting that benzo[*a*]pyrene (BaP), a “Group (1)″

Table 3
Effect of moisture content regulation on aroma active compounds in SP.

No.	Aroma-active compounds	RI[1]	Identification Method[2]	Aroma descriptor[3]	Concentration (μg/kg)[4]				
					5	10	15	20	25
1	2-Methylpyrazine	828	MS/O/RI/STD	roasted	710.63 ± 19.88b	972.74 ± 5.02a	216.22 ± 15.25d	317.29 ± 6.42c	688.64 ± 53.14b
2	3,5-Dimethyl-2-(3-methylbutyl)-pyrazine	1319	MS/O/RI	salty pastry	48.27 ± 0.00c	51.19 ± 0.57bc	20.38 ± 0.78d	57.3 ± 4.17b	162.65 ± 7.71a
3	2,5-dimethylpyrazine	916	MS/O/RI/STD	roasted sesame	1400.75 ± 48.19c	1841.66 ± 5.20a	530.93 ± 10.80e	954.95 ± 101.60d	1670.40 ± 143.95b
4	2,6-Diethyl-pyrazine	1095	MS/O/RI	roasted	42.55 ± 6.34c	67.76 ± 4.08b	216.9 ± 0.45a	225.07 ± 10.64a	ND
5	2-Ethyl-5-methylpyrazine	1007	MS/O/RI	nutty	317.96 ± 4.47b	479.17 ± 1.99a	218.24 ± 1.22d	260.87 ± 15.79c	272.09 ± 24.7c
6	3-Ethyl-2,5-dimethyl-pyrazine	1086	MS/O/RI	Roasted,salty pastry	521.67 ± 28.19b	737.59 ± 5.44a	203.51 ± 5.06d	372.32 ± 9.07c	494.13 ± 29.88b
7	2-Ethyl-pyrazine	920	MS/O/RI/STD	roasted sesame	281.10 ± 29.58b	319.60 ± 0.00a	ND	ND	ND
8	2-Ethyl-6-methylpyrazine	1006	MS/O/RI	nutty	254.07 ± 16.48a	170.26 ± 3.16c	194.86 ± 6.69b	170.55 ± 6.99c	243.79 ± 17.07a
9	2,3,5-trimethylpyrazine	1008	MS/O/RI/STD	roasted peanut	502.73 ± 13.80c	667.89 ± 5.18a	215.42 ± 32.29e	440.34 ± 45.72d	599.84 ± 20.83b
10	2,3-diethyl-5-methylpyrazine	1164	MS/O/RI	roasted sesame	14.92 ± 0.64c	25.24 ± 0.08b	ND	14.6 ± 1.46c	30.27 ± 0.00a
11	2-Methyl-3,5-diethyl-pyrazine	1165	MS/O/RI	roasted	64.73 ± 7.00c	108.6 ± 1.30b	28.99 ± 2.11d	ND	170.95 ± 13.15a
12	Pyrazine	1043	MS/O/RI	salty pastry	28.27 ± 1.06a	24.18 ± 1.75b	ND	ND	22.87 ± 0.84b
13	2,3-dimethyl-pyrazine	925	MS/O/RI	salty pastry	65.93 ± 0.13b	77.44 ± 6.67a	22.01 ± 0.57e	44.11 ± 3.47d	55.49 ± 1.84c
14	2,3-dimethyl-5-ethylpyrazine	1096	MS/O/RI/STD	roasted	44.96 ± 0.00b	ND	ND	ND	378.71 ± 30.22a
15	5-methyl-6,7-dihydro-5H-cyclopentapyrazine	1148	MS/O/RI	Roasted,nutty	ND	31.74 ± 1.37a	ND	ND	32.69 ± 7.73a
16	2-Ethylpyrrole	807	MS/O/RI	roasted	ND	16.33 ± 1.54a	3.68 ± 0.17b	ND	ND
17	1,5-Dimethyl-2-pyrrolecarbonitrile	1115	MS/O/RI	caramel	19.38 ± 1.11b	25.99 ± 0.64a	3.91 ± 0.00d	12.83 ± 0.00c	24.92 ± 2.03a
18	3-Methylpyrrole	860	MS/O/RI	salty pastry	ND	56.79 ± 6.13a	21.19 ± 0.01c	51.29 ± 2.88b	ND
19	2-Methylpyrrole	859	MS/O/RI	salty pastry	60.55 ± 2.19a	14.77 ± 0.00d	18.59 ± 2.25c	41.02 ± 1.74b	ND
20	2-Pyrrole formaldehyde	1034	MS/O/RI	Burnt,roasted	158.32 ± 5.48	ND	ND	ND	ND
21	3-Acetyl pyrrole	1080	MS/O/RI	medicinal	ND	61.16 ± 7.70	ND	ND	ND
22	Pyrrole	763	MS/O/RI	popcorn	262.55 ± 16.88a	206.77 ± 2.85b	ND	ND	ND
23	*N*-methyl-2-pyrrolecarboxaldehyde	1158	MS/O/RI	salty pastry	49.92 ± 1.55a	27.50 ± 2.64b	ND	ND	ND
24	Furanone	1093	MS/O/RI	roasted peanut	881.97 ± 48.58b	ND	ND	1080.89 ± 117.62a	ND
25	2-pentylfuran	997	MS/O/RI	mushroom	ND	73.08 ± 0.08a	53.37 ± 1.02b	ND	ND
26	5-Methyl-2-acetylfuran	1050	MS/O/RI	salty pastry	142.38 ± 5.38a	ND	ND	40.57 ± 0.00b	ND
27	2-Ethyl-5-methylfuran	812	MS/O/RI	burnt	39.59 ± 3.99	ND	ND	ND	ND
28	2(5H)-furanone	924	MS/O/RI	caramel	84.79 ± 12.76	ND	ND	ND	ND
29	5-Methylfuranal	971	MS/O/RI	roasted	142.38 ± 5.38a	39.22 ± 1.60b	ND	ND	ND
30	Furfural	841	MS/O/RI	almond	635.91 ± 11.97	ND	ND	ND	ND
31	Dimethyl trisulfide	973	MS/O/RI/STD	sulphur-like,	ND	ND	ND	36.16 ± 0.00b	138.82 ± 1.21a
32	Benzeneacetaldehyde	1051	MS/O/RI/STD	flower	433.26 ± 6.27b	147.33 ± 4.06c	532.60 ± 19.53a	ND	ND
33	2,4-Dimethylbenzaldehyde	1226	MS/O/RI	almond	318.3 ± 13.67a	ND	22.89 ± 0.00c	ND	103.12 ± 9.29b
34	2-Methylbutyraldehyde	<700	MS/O/STD	almond	432.50 ± 34.28d	506.20 ± 15.37c	630.91 ± 2.49b	718.57 ± 110.30a	627.03 ± 42.10b
35	Benzaldehyde	967	MS/O/RI/STD	sweet	108.53 ± 12.47a	ND	82.88 ± 7.68b	ND	ND
36	2-vinyl-4-methoxyphenol	1325	MS/O/RI	burnt	ND	ND	ND	5498.32 ± 199.01a	4533.86 ± 140.33b
37	Guaiacol	1101	MS/O/RI	smoked	89.56 ± 0.61b	111.12 ± 2.01a	ND	40.84 ± 1.99c	ND

(*continued on next page*)

Table 3 (*continued*)

No.	Aroma-active compounds	RI[1]	Identification Method[2]	Aroma descriptor[3]	Concentration (μg/kg)[4]				
					5	10	15	20	25
38	Sesamol	1349	MS/O/RI	burnt	ND	523.41 ± 72.12	ND	ND	ND
39	3-Methyl-2-butanone	<700	MS/O	mushroom	ND	480.04 ± 0.00a	255.23 ± 17.46b	ND	ND
40	Isophorone	1128	MS/O/RI	woody	171.84 ± 8.33a	177.19 ± 0.57a	ND	ND	45.46 ± 3.75b
41	Methyl ethyl ketone	<700	MS/O	fermented	ND	ND	ND	21.29 ± 0.80b	83.23 ± 0.00a
42	Hydroxyacetone	<700	MS/O	burnt	652.37 ± 48.23a	73.77 ± 0.00b	ND	ND	ND
43	Acetophenone	1074	MS/O/RI	medicinal	ND	15.70 ± 0.67b	ND	ND	25.55 ± 4.07a
44	3,7,7-rimethylbicyclo[4.1.0]hept-3-ene	937	MS/O/RI	sweet	13.74 ± 0.48b	ND	ND	41.54 ± 0.00a	ND
45	α-pinene	1380	MS/O/RI	woody	ND	17.89 ± 0.23b	20.9 ± 0.16a	ND	ND
46	3-Methyl-3-ethylheptane	1090	MS/O/RI	almond	ND	ND	66.32 ± 2.33b	ND	91.82 ± 0.50a
47	1,1,3-trimethylcyclohexane	837	MS/O/RI	almond	348.13 ± 24.17a	306.43 ± 0.02b	ND	172.55 ± 0.96c	ND
48	5-Ethyl-2-methyloctane	1105	MS/O/RI	green	ND	ND	ND	230.11 ± 9.16a	158.32 ± 4.36b
49	Methanesulfonylmethane	934	MS/O/RI	burnt	338.64 ± 16.93a	ND	ND	32.91 ± 0.00b	ND
50	M−Xylene	898	MS/O/RI/STD	salty pastry	187.69 ± 9.01d	143.47 ± 15.13d	1311.95 ± 151.64a	329.43 ± 30.02c	477.71 ± 1.12b

[1] RI: Retention index on HP-5MS capillaries.

[2] Identification methods: MS, RI, O, and STD representative compounds were identified by mass spectrometry, retention indices, olfactometry, and standard chemical, respectively.

[3] Aroma descriptions were obtained by GC-O analysis and by panelists (2 females and 1 male) at the sniffer port.

[4] Concentrations of the aroma compounds in SP samples were obtained by the internal standard method. The absence of any identical letter in the same row indicated a significant difference between samples ($P < 0.05$). ND indicated that the compound was not detected.

carcinogen formed by incomplete combustion of organic compounds, was not detected in the study. Additionally, the PAH4 content in all samples did not exceed the recommended limit (<10 ng/g) specified in the European Commission Regulation No. 835/2011(European Commission, 2011).

3.5. *Analysis of free amino acids*

Table 2 presented the free amino acids composition of SP, indicating the presence of 17 free fatty acids, including 6 essential amino acids. However, methionine (Met) and tryptophan (Trp) were not detected in any of the samples. The content of each amino acid exhibited significant variation ($p < 0.05$) among SPs prepared from sesame seeds with different water contents. Notably, SP-10 had the lowest total amount of free amino acids (148.89 mg/100 g).

Certain amino acids such as glycine (Gly), valine (Val), phenylalanine (Phe), and alanine (Ala) are crucial precursors for the formation of aroma compounds. They undergo reactions with reducing sugars in processes like the Maillard reaction, caramelization, and Strecker degradation, leading to the production of volatile compounds containing pyrazine, pyrrole, or furan structures (Guo, Ho, Schwab, & Wan, 2021). The lower levels or absence of specific amino acids in SP can be attributed to their involvement in these reactions, ultimately resulting in the generation of volatile compounds.

3.6. *Effect of water content regulation on aroma active compounds in SP*

A total of 676 volatile compounds were detected in the five SPs, and the number of each type of volatile compounds was shown in Supplementary S2. By GC-O-MS analysis, a total of 50 aroma compounds were detected for the first time in the five SPs, including 15 pyrazines, 8 pyrroles, 7 furans, 5 ketones, 4 aldehydes, 4 alkanes, 3 phenols, 2 olefins, 1 sulfur-containing compound, and 1 benzene. The variation of aroma compounds with water content was shown in Table 3.

3.6.1. *Heterocyclic compounds*

Heterocyclic compounds are one class of the products of the Maillard reaction, Strecker degradation, or caramelization, they contain heteroatoms such as nitrogen, oxygen, sulfur, and aromatic rings, which give them some reactivity in the reactions. As shown in Table 3, the total concentration of heterocyclic compounds in SP-10 was the highest, combined with its highest non-enzymatic browning degree, which was enough to indicate that SP-10 has the highest degree of Maillard reaction. Pyrazine compounds were the most abundant aromatic active compounds in SP, with aromas of roasting, nuts, roasted sesame, and salty pastry. Pyrrole had characteristic caramelized, salty pastry, and roasted aromas. Furan derivatives were characterized by salty pastry, almond, and caramel aromas. In caramelization reactions, they could undergo condensation reactions with small molecule compounds produced by sugar decomposition (Turan Ayseli, Kelebek, & Selli, 2021). It could be seen that the concentration of heterocyclic compounds in SP was closely related to the water content of the sesame seeds. This might be because sesame seeds with higher water content contained more water, which could lead to oxidative reactions forming an oxidative layer. This oxidative layer made it difficult for oxygen molecules inside sesame seeds to escape and also caused water molecules and amino acid molecules inside sesame seeds to be relatively densely distributed, creating an environment more conducive to the Maillard reaction.

3.6.2. *Aliphatic compounds*

SP contains both alkenes and alkanes, which were mainly generated through the thermal treatment of unsaturated fatty acids during the sesame roasting process. These alkenes and alkanes were the important flavor compounds in SP, endowing it with a unique aroma and taste. High levels of alkane compounds might give SP a relatively dull aroma (almond, raw green, and burnt), while alkene compounds might impart

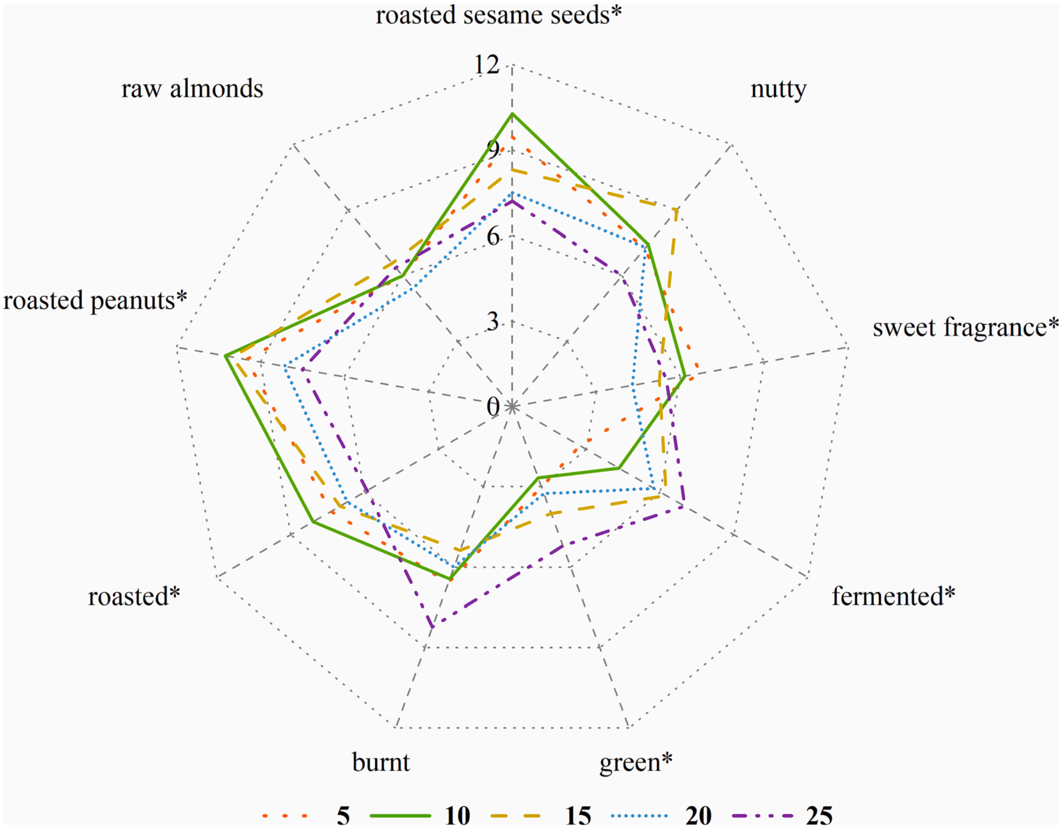

Fig. 3. Sensory evaluation of SPs. Note: "*" indicate significance at $p < 0.05$. Aroma attributes: roasted sesame seeds, nutty, sweet fragrance, fermented, green, burnt, roasted; roasted peanuts, raw almonds. 5, 10, 15, 20, and 25, the moisture content of sesame seeds before roasting in SP, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

a fresher and more aromatic aroma (sweet and woody).

3.6.3. *Sulfur-containing compounds and benzenes*

Sulfur-containing volatile compounds contributed to the sulfur aroma of SP. Additionally, sulfur compounds possessed certain antioxidant properties that could extend the shelf life of SP (Hill et al., 2022). However, high concentrations of sulfur-containing aroma compounds could cause discomfort to humans (Sun et al., 2023). Fortunately, the concentration of sulfur-containing compounds in all samples was found to be very low, and some samples did not contain any detectable levels of these compounds. SP-15 had the highest concentration of m-xylene and it was likely that the production of m-xylene was promoted by the moderate moisture content.

3.6.4. *Oxygen-containing compounds*

Positive impacts on the aromas of SP were exerted by oxygenated compounds. Aldehyde compounds bestowed the SP with sweet or floral aromatic characteristics, while ketone compounds contributed to savory, roasted peanut, and caramel aromas. Furthermore, phenolic compounds served as the primary source of smoky notes in SP.

Although the concentrations of aldehyde and ketone compounds in SP were relatively low, their low perception thresholds suggested their potential to significantly influence the aromatic profile of the SP (Yin et al., 2022). In contrast, phenolic compounds exhibited higher perception thresholds, indicating the need for relatively higher concentrations to be perceptible and contributed to their characteristic aroma.

Notably, the presence of sesamol was exclusively detected in the SP-10, while it remained undetected in other samples. Sesamol was gradually formed through the decomposition of compounds such as sesamolin during the thermal processing of SP (Wei, Zhao, Wang, Wang, Chai, Hou, & Meng, 2022). This observation suggested that appropriate regulation of moisture content might favor the formation of sesamol during thermal processing.

3.7. *Sensory evaluation*

The sensory characteristics of the five samples showed significant differences ($p < 0.05$), as shown in Fig. 3. SP-15 exhibited an intense, pronounced nutty aroma. SP-25 had a strong burnt aroma. Compared with the other samples, SP-10 had more positive aroma attributes, it exhibited extremely high roasted sesame, roasted, and roasted peanut aroma. When the water content further increased, the fermented and burnt aromas of the SP samples increased, indicating the hydrolysis rancidity degree increased, and the perceptible positive aroma was suppressed and ultimately resulted in the lowest sensory quality at SP-25. In a word, the water content of 10 % could provide rich positive aromas, and avoid the negative aromas, which might be due to its high degree of heat reaction of SP during roasting.

4. Conclusion

In this study, we investigated the crucial role of pre-regulation of water content in sesame seeds on the formation of Maillard reaction products, color intensity, and sensory attributes of sesame paste (SP). Our findings shed light on the intricate relationship between moisture content and the various aspects of SP quality.

First and foremost, it became evident that pre-regulating water content significantly impacts the Maillard reaction, which is pivotal in determining color and flavor attributes. SP-10, with an intermediate moisture content, exhibited a more pronounced Maillard reaction, leading to deeper coloration and intensified non-enzymatic browning.

This heightened reaction, however, came at the cost of potentially harmful compounds such as heterocyclic amines (HCAs) and polycyclic aromatic hydrocarbons (PAHs). Our study highlights the essential role of moisture content in temperature management during the roasting process, as lower moisture content led to elevated temperatures, further accelerating Maillard reaction and thermal degradation, thus enhancing the potential for PAH formation.

Furthermore, our research made a significant contribution by identifying a total of 50 aroma-active compounds in SP using GC-O-MS. Notably, SP-10 stood out with a higher concentration of heterocyclic compounds, indicating a more advanced Maillard reaction. These compounds imparted distinct aroma notes, including roasting, nuttiness, roasted sesame, and salty pastry. The profound influence of water content in sesame seeds on the concentration of these heterocyclic compounds underscores the need for precise control in SP production.

Importantly, we linked these findings to sensory attributes. SP-10 exhibited a richer roasted sesame, burnt, and roasted peanut aroma, along with other positive sensory qualities, while SP-25, with the highest moisture content, displayed the lowest sensory quality, marked by an increase in fermented and burnt flavors, signifying elevated hydrolysis rancidity.

CRediT authorship contribution statement

Ming Yang: Conceptualization, Data curation, Methodology, Writing – original draft. **Lixia Hou:** Supervision, Methodology, Funding acquisition, Writing – review & editing. **Bingkai Wang:** Methodology, Investigation, Data curation. **Xiaomei Sun:** Methodology, Formal analysis. **Lei Jin:** Methodology, Formal analysis. **Yifan Dong:** Data curation, Methodology. **Huamin Liu:** Supervision, Resources. **Xuede Wang:** Supervision, Resources.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Lixia Hou reports financial support was provided by Ministry of Agriculture of the PRC.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2023.101100.

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