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Research Article

Impact of Aging Time on Beef Quality: Evaluation of Dry and Wet Aging Techniques Through Tenderness and Aroma Profile Analysis

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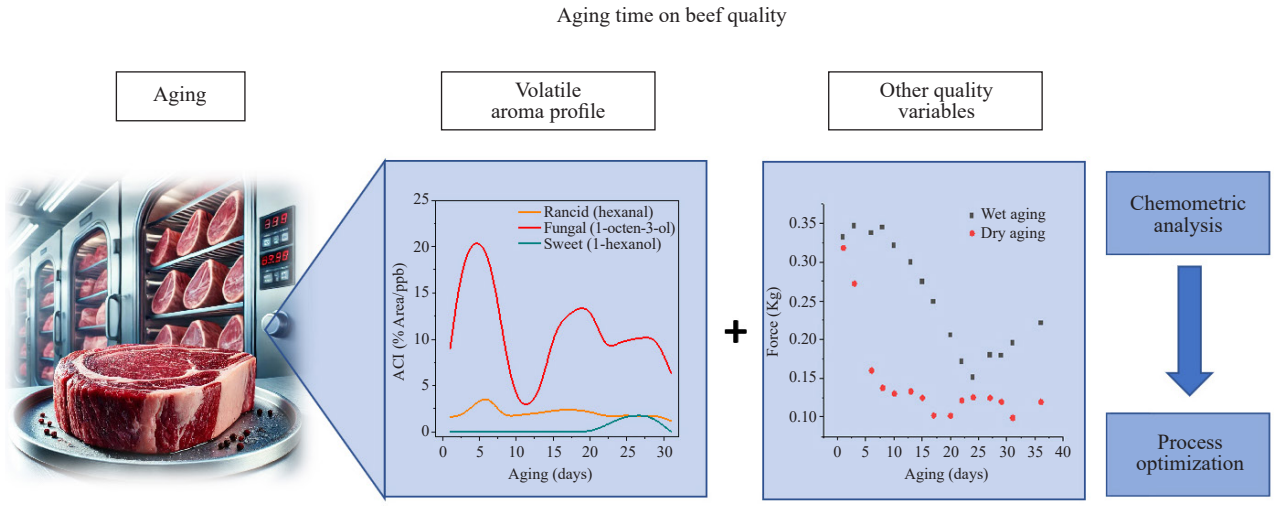
Graphical Abstract:



Abstract: This study aimed to assess the impact of aging time on quality factors that influence beef acceptability in meat subjected to both wet and dry aging. To achieve this, changes in water loss, pH levels, tenderness, and aroma volatile profiles were monitored at intervals of 2-3 days over a 31-day period for each aging method. The *longissimus dorsi* from yearling beef was used as the sample, and volatile analysis was conducted using solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS). A principal component analysis (PCA) was performed to identify the aging time that maximized tenderness and desirable aromas while minimizing undesirable ones for both techniques. Although pinpointing a definitive result was challenging due to the numerous variables influencing the process, dry-aged beef showed slightly higher pH and greater moisture loss than wet-aged beef. Tenderness peaked earlier (day 17)

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in dry-aged samples, while wet-aged beef improved more gradually until day 25. PCA proved to be an effective tool for approximating optimal conditions, revealing that, under consistent sample and method parameters, dry-aged samples reached optimal conditions earlier (days 10-13) than wet-aged counterparts (days 24-27).

***Keywords*:** SPME-GC-MS, shear force, flavor, water loss, pH, sensory, bovine meat

1. Introduction

Consumer opinions reveal that the tenderness and flavor of meat are considered the most important quality indicators. The aroma of fresh and raw meat is often described as blood-like with sweet connotations [1]. However, as the meat ages, its aroma evolves, ranging from sweet and fatty to fungal or rancid [2, 3].

Once the animal is slaughtered, the treatment of the meat becomes paramount. Some authors have studied the biochemical complexities involved in converting animal muscle into consumable meat and investigated texture changes [4, 5]. These studies have led to the development of new technologies aimed at improving meat's tenderness and flavor, with controlled aging in a chamber being the most common method. Aging enhances the meat's nutritional value, flavor, and tenderness through biochemical mechanisms such as lipid degradation, carbohydrate fermentation, and protein and lipid enzymatic hydrolysis, which generate aroma precursors for the Maillard reactions during cooking [6].

Currently, the most prevalent methods for beef maturation are dry aging and wet aging. Dry aging entails exposing the meat directly to a controlled aging environment for a variable duration, without the use of packaging or additives, within a decontaminated atmosphere inside a chamber where temperature and humidity are carefully regulated. Conversely, wet aging follows a similar process but involves vacuum-sealing the meat in a bag prior to its placement in the aging chamber. The choice between dry and wet aging markedly influences the characteristics of the aging product, mainly due to the differing microbiological fermentation processes, which are aerobic in dry aging and anaerobic in wet aging.

Over recent decades, extensive research has been conducted to compare the differences between dry and wet aging methods in beef, with the aim of determining which technique offers superior taste and tenderness while minimizing both time and product loss [7]. Some studies suggest minimal sensory differences between both methods [8], while others note that dry aging leads to more evaporation and product loss. However, dry-aging techniques such as using dry-aging bags have been shown to reduce these losses [9]. Wet-aged meat has shown greater product loss during cooking, suggesting that such losses in dry aging may not be a significant disadvantage [10], although in emerging cooking trends like sous-vide, wet-aged beef is preferred [11].

Modified atmosphere packaging (MAP) and edible packaging can significantly influence meat quality and yield during aging. Both systems involve direct contact between the packaging material and the meat surface, which may lead to changes in flavor, potentially affecting organoleptic acceptance and raising regulatory concerns [12]. MAP has been shown to improve color stability and extend shelf life by limiting microbial growth [13, 14], while edible packaging can similarly control microbial proliferation, enhancing safety and preservation [15]. Although these techniques offer notable advantages, their effects vary depending on factors such as meat type, packaging composition, and storage conditions, underscoring the need for further research to optimize their application in meat aging.

In addition to the election of the aging method, the duration of aging significantly affects meat quality. The optimal aging time for achieving desired tenderness and flavor is not yet precisely predictable, as factors like animal breed, diet, genetics, sex, age at slaughter, castration status, and rearing conditions play a key role [16-19]. Furthermore, the choice of muscle examined plays a crucial role in the variability of the results [20, 21]. Understanding aroma-active compounds, tenderness, pH, and moisture content could help approximate the ideal aging period for similar meat cuts under consistent conditions, such as meat mass and dimensions, muscle selected, aging chamber conditions, muscle type, and animal rearing practices.

In this study, multiple quality parameters of beef were assessed over a 31-day aging period using both dry and wet aging methods. Key variables, including moisture content, pH, tenderness, and volatile profiles, were monitored at intervals of 2-3 days throughout the aging process. The outcomes were then compared between the two aging techniques. A principal component analysis (PCA) was subsequently conducted based on the collected data to identify

the most suitable aging day, aiming to maximize tenderness and desirable aromas while minimizing undesirable aromas in raw meat.

2. Materials and methods

2.1 *Sample preparation*

The *longissimus dorsi* muscle was selected for its consumer-perceived tenderness, juiciness, and flavor, making it a reliable proxy for overall beef quality [20]. Its marbling plays a key role in meat grading systems, linking it to studies on economic valuation and market classification [9, 21]. More affordable than tenderloin yet highly appreciated, it remains one of the most preferred dietary cuts.

The slaughtered animal weighed 624 kg, was male, 19 months old, and a crossbreed of limousin and retinto. It was fed on a fattening diet with the following composition: corn (36.0%), distillers dry grains (DDG) from imported corn (20.0%), barley (15.9%), triticale (15.0%), soy 47 (3.3%), soy husk (3.2%), calcium soap fat (2.5%), glycerol 80% (2.0%), Setnamix calves' buffer (1.0%), calcium carbonate (0.9%), and salt (0.3%).

Two days after slaughter, the samples were packaged and transported under refrigeration at 4 °C to the analysis laboratory. The samples were then divided into two parts for different aging processes (wet and dry).

The samples were stored in an aging chamber (Brunetti MDR40, Capacity: 75 kg, 118 l) equipped with a ultraviolet (UV) lamp for 31 days at a temperature of 2 ± 2 °C, a relative humidity of 75 ± 1%, and an airflow of 1.5 ± 0.5 m/s. Temperature and humidity were controlled in real time through a control device made by engineers from Siemens, in collaboration with University of Malaga (UMA), with data acquired via a mobile application. The airflow velocity was monitored daily using a portable anemometer (410i, Testo SE & Co. KGaA) with a tolerance of ± 0.44 m/s.

Two sample formats were prepared: minced meat samples and cubic pieces samples of 3 × 3 × 3 cm size. The former ones were used for the analysis of volatiles by solid-phase microextraction and gas chromatography/mass spectrometry (SPME-GC/MS) and the determination of moisture content, while the latter ones were used for the determination of pH and tenderness.

A grinder (IKA A11, IKA Werke, Staifen, Germany) was used to mince the meat and finally, the mixture was homogenized manually.

The analyses were performed on days 1, 3, 6, 8, 10, 13, 15, 17, 20, 22, 24, 27, 29, and 31 of the aging processes. Each test was carried out in triplicate for both wet and dry aging samples.

2.1.1 *Dry aging process*

For the dry aging process, a 5.9 kg sample with approximate dimensions of 58 × 25 × 16 cm was prepared. During each scheduled analysis, a fragment of the sample from the dry aging process was excised from the bulk of the meat.

2.1.2 *Wet aging process*

For wet aging, a 2.8 kg sample was sectioned into 3 × 3 × 3 cm pieces and vacuum packed in groups of 8 pieces. Specific polyamide/polyethylene (PA/PE) vacuum bags were used, with a thickness of 90 µm, an O_2 permeability of 1.3 cc/m^2/day at 4 °C and 75% relative humidity, and a water transmission rate of 4.2 g/m^2/day at 38 °C and 75% relative humidity. During each scheduled analysis, a wet aging blister of samples was opened for testing.

2.2 *pH analysis*

For the pH analysis, the samples were measured in bulk format using a penetration probe. However, the device was calibrated with liquid standards prepared from buffer solutions (Hach Lange Spain, SLU), with pH values of 4.01, 7.00, and 9.21. Prior to measurement, the samples were tempered to 25 °C in a thermostatic water bath (Precisterm, JP Selecta SA, Barcelona, Spain) to minimize pH differences caused by temperature variations among samples. The measurements were performed using a portable pH meter (Sension+, Hach Lange Spain, SLU) equipped with a penetration electrode (LZW 5053, Hach Lange Spain, SLU) and a temperature probe (Peaktech 5110, Peaktech Prüf, Ahrensburg, Germany).

2.3 *Moisture content and weight loss*

The determination of moisture content was carried out in accordance with the ISO 1442:1997 standard, which outlines the procedure for measuring moisture in meat and meat products. Each aging condition was analyzed in triplicate to ensure the reliability and reproducibility of the results. The moisture content percentage was calculated using the formula (1).

$$\text{Moisture (\%)} = \frac{(M_1 - M_2)}{(M_1 - M_0)} \times 100 \quad (1)$$

where M_0 is the mass of the empty, pre-dried crucible with sand and stirring rod, M_1 is the mass after adding the homogenized sample, and M_2 is the final mass after drying.

Six porcelain crucibles, each containing a glass stirring rod and between 15 and 20 grams of sea sand, were initially dried in a laboratory oven (Selecta, JP Selecta, Barcelona, Spain) at 102 ± 2 °C (Panreac, Panreac Química SLU, Barcelona, Spain) for a duration of one hour. The sand used in this process had a particle size ranging from 1 to 2 mm. Once dried, the crucibles were cooled in a glass desiccator containing silica gel with a humidity indicator (Panreac, Panreac Química SLU, Barcelona, Spain).

After reaching room temperature, the crucibles were weighed using an analytical balance (Mettler Toledo EL104, Mettler Toledo, Columbus, Ohio, USA), and the initial mass (M_0) was recorded in four decimal places. For the analysis, sample pieces measuring 3 × 3 × 3 cm were prepared, ensuring that the outer crust was included in the case of dry-aged samples. These pieces were then ground using a mill (IKA A11, IKA Werke, Staufen, Germany) and thoroughly homogenized by hand. A 5 g portion of the processed sample was placed in each of the pre-dried crucibles, and the combined mass of the capsule, glass rod, sand, and sample was noted (M_1).

Next, 5 ml of 96% v/v ethanol (Panreac, Panreac Química SLU, Barcelona, Spain) was added to each crucible, and the mixture was stirred using the glass rod. The crucibles were then placed in a thermostatic water bath (Precisterm Selecta, JP Selecta, Barcelona, Spain) at 80 ± 1 °C for 15 minutes. Following this step, they were transferred to a drying oven (Selecta, JP Selecta, Barcelona, Spain) set at 102 ± 2 °C for four hours.

Once the drying phase was complete, the samples were again cooled in a desiccator and subsequently reweighed with the analytical balance to obtain the final mass after drying (M_2).

2.4 *Tenderness determination*

Tenderness, which is defined as the opposite of toughness, is associated with a reduction in shear force value (SFV) [22]. To evaluate SFV, a digital force gauge (Mark-10 EG20, Mark-10 Corporation, NY, USA) was employed. This device, accurate to ± 0.009 kg, was fitted with a blade measuring 4.2 cm in length and 2.5 cm in width, specifically designed to slice through meat samples. The depth of blade penetration was monitored using a depth gauge (Physical Test Solutions, Culver City, CA, USA), offering a measurement range of up to 5 cm with a precision of ± 0.01 cm.

Prior to testing, all meat samples were trimmed and standardized to uniform dimensions of 3 × 3 × 3 cm. Each piece was consistently positioned so that the blade would cut perpendicular to the direction of the muscle fibers. In the case of dry-aged samples, which develop a dehydrated outer layer or crust, this crusted surface was preserved and always placed downward in the analyzer setup, ensuring it served as the base during testing.

During the test, the force gauge continuously recorded the resistance encountered as the blade advanced through the sample, while the depth gauge simultaneously tracked how deeply the blade penetrated from the surface. As the tenderness is considered the inverse of toughness, or, in other words, the inverse of SFV, results were expressed as the inverse of the force required to achieve a 1 cm penetration into the meat.

2.5 *Determination of the volatile profile*

A precise 2.00 ± 0.02 g sample of the minced meat was weighed using an analytical balance and transferred into a glass vial. To this, 2 ml of osmotized water, obtained through a water purification system, was added (Millipore Elix 3,

Millipore Corporation, Massachusetts, USA). Additionally, 0.3 ml of a 10% NaCl solution, prepared in the laboratory using solid NaCl (Panreac, Panreac Química SLU, Barcelona, Spain) and osmotized water, was added. The mixture was then subjected to homogenization in an ultrasound bath (Selecta 300514, JP Selecta, Barcelona, Spain) for 2 minutes before being analyzed. The analysis was carried out using solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) composed by a Trace GC Ultra chromatograph (Thermo Fisher Scientific, Massachusetts, USA) coupled to an ITQ 900 mass spectrometer (Thermo Fisher Scientific, Massachusetts, USA), which features an ion trap detector and an Autosampler Triplus RSH (Thermo Fisher Scientific, Massachusetts, USA).

The chromatographic conditions were set as follows [23, 24]: The sample was incubated for 10 minutes at 50 °C, then extracted for 30 minutes at the same temperature while being agitated. A polydimethylsiloxane/divinylbenzene (PDMS/DVB) fiber of 65 µm and 23 Ga was used for sample absorption. After that, desorption occurred in the injector at 250 °C for 2 minutes. A ZB-5 column (60 m length, 0.25 mm inner diameter, 0.25 µm film thickness, Zebron) was used, with a heating ramp starting at 40 °C for 3 minutes, followed by an increase of 8 °C per minute up to 250 °C, where it was held for an additional 5 minutes. The source temperature was maintained at 230 °C, and the transfer line was kept at 250 °C. The mass spectrometer recorded data in positive mode, scanning the mass-to-charge ratio (m/z) from 30 to 200.

The retention times for peak identification were compared with those of internal standards, which were analyzed under identical conditions as the samples (Sigma-Aldrich, Steinheim, Germany). The abundance of aromatic compounds was determined by normalizing the area of each compound relative to the total peak area of the chromatogram. Odor descriptions and threshold values of the volatile compounds were sourced from the material safety data sheets of the products, relevant literature references, and databases such as http://www.chemicalbook.com and https://pubchem.ncbi.nlm.nih.gov/.

2.6 *Statistical analysis*

A Spearman's 2-tailed correlation test was conducted using Origin software (version 2018) to evaluate the statistical significance of the correlations between various parameters of meat samples from wet and dry aging processes, with a confidence level of 0.05. Additionally, a principal component analysis (PCA) was performed using the multivariate analysis module of the same software to investigate and quantify the relationships between flavor precursor volatiles and the aging duration across both processes.

3. Results and discussion

3.1 *Impact of dry and wet aging on pH and moisture content*

The pH evolution and moisture content were assessed during wet and dry aging and represented in Figure 1. As illustrated in Figure 1a, meat samples subjected to dry aging consistently exhibit slightly higher pH levels compared to those undergoing wet aging. This observation aligns with the findings of other researchers who declared that the pH did not differ greatly between dry and wet-aged beef [9, 25, 26], although a slight variation is apparent in these results. This variation has been investigated by other authors [27-29], who stated that the pH of wet-aged samples is lower than that of dry-aged samples. This difference is attributed to the anaerobic conditions of wet aging, which promote the growth of lactic acid bacteria over other species, leading to the generation of acidic metabolites that lower the pH of the medium [29].

Regarding dry-aged meat, microbiological growth occurs primarily on the surface, where strict and facultative aerobic microorganisms proliferate, while anaerobic and facultative bacteria colonize the internal portions. Under aerobic conditions, bacteria of the genus *Pseudomonas* predominate, followed by *Acinetobacter*, *Moraxella*, and *Flavobacterium*. Conversely, in anaerobic conditions, Gram-positive flora, particularly lactic acid bacteria and *Brochothrix thermosphacta*, become dominant [30]. Lactic acid bacteria produce lactic acid by metabolizing glucose, leading to a decrease in pH and the development of a sour, fermented taste and aroma. These sensory attributes become perceptible to consumers as signs of meat spoilage when metabolite concentrations reach sufficiently high levels [31].

Moisture loss during dry aging is a critical parameter, as it contributes to flavor concentration while simultaneously

impacting yield and texture. To regulate moisture loss effectively without compromising sensory quality, a range of environmental and technological variables can be optimized. These include the precise regulation of temperature, UV light, airflow velocity and relative humidity, along with the incorporation of salt blocks and a water reservoir within the aging chamber [32, 33]. Figure 1b illustrates the variation in moisture content for both aging processes. A significant difference in water loss is evident between dry-aged and wet-aged products. This water loss translates to a reduction in both the weight and size of the product, which can impact the producer's interests. However, this moisture loss also contributes to the concentration of aroma and flavor in the meat [8, 9] and results in a reduced volume of the product after aging. Nevertheless, the water loss in dry-aged samples alone does not fully account for the enhanced flavor and aroma, as the variation in these qualities is not directly proportional to the water loss [32]. Other researchers [34] have proposed that the heightened concentration of flavor and aroma in dry-aged samples is partly due to proteolysis, which increases the free amino acid content in the meat [7, 35].

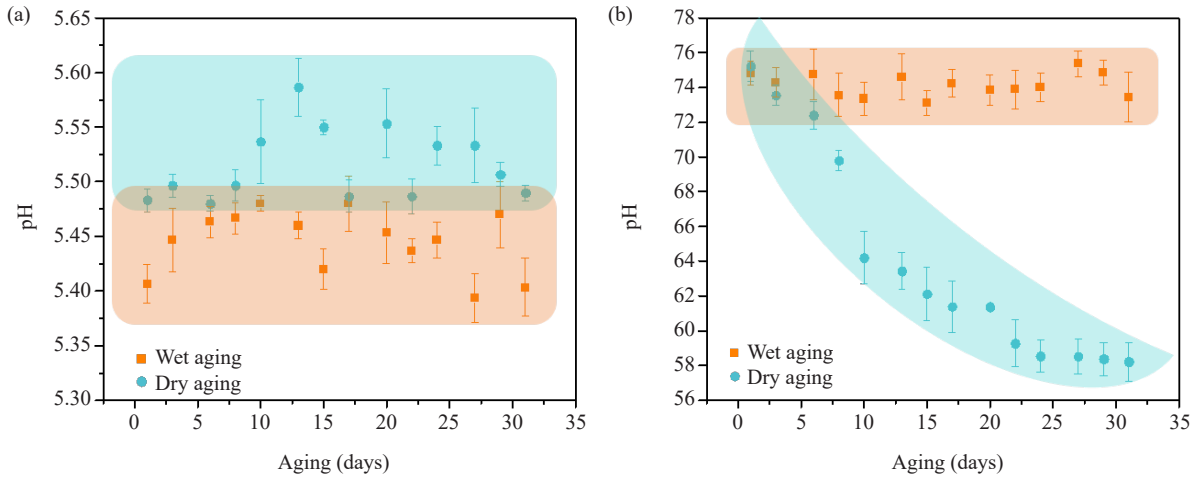

Figure 1. pH (a) and water content (b) variation in samples subjected to dry and wet aging processes. Error bars represent the standard deviation of each data point ($n = 3$)

In this study, three dry-aged and three wet-aged samples were analyzed each test day. As shown in Figure 1b, the wet-aged samples exhibit moisture content variability regardless of the aging day ($P > 0.05$). This variability is attributed to the encapsulation in a polymer throughout the aging process, where humidity variations are primarily influenced by biological and biochemical factors. Conversely, the figure demonstrates a correlation between the number of dry aging days and the moisture content in samples, described by a second-degree equation ($y = 77.42 - 1.32x + 0.023x^2$, $R^2 = 0.9632$, $P < 0.001$). This correlation leads to a relative humidity loss of more than 22% over the 31 days of dry aging.

From a financial standpoint based on current prices in the Spanish market, dry aging yields a higher profit margin (approx. 34.5%) despite a 22% weight loss, as the elevated selling price compensates for the reduced yield. However, it requires a higher initial investment in infrastructure and technology compared to wet aging. In contrast, wet aging involves no weight loss and lower operational complexity but results in a lower profit margin of approx. 23.3% due to its comparatively lower market value.

3.2 *Study of tenderness*

Tenderness increases during the aging process due to enzymatic activity, specifically the proteolysis of muscle fiber proteins, which leads to the degradation of myofibrils and the disruption of myofibril and muscle fiber junctions [36]. The enzyme primarily responsible for improving tenderness in aged meat is calpain protease, which reduces post-rigor toughness [7, 37]. This enzymatic action results in an enhancement of meat tenderness.

Previous research has indicated that the optimal aging duration differs between wet-aged and dry-aged samples [7]. Throughout both aging processes, the hardness of the samples was assessed using a force gauge equipped with a blade and a depth gauge (Figure 2b), with tenderness considered inversely related to hardness.

As illustrated in Figure 2a, wet-aged samples exhibited a progressive increase in tenderness from the 5th to the 25th day of aging. After the 25th day, a slight increase in hardness was observed, likely attributable to ongoing biochemical and microbiological reactions within the samples. In contrast, dry-aged samples displayed a more pronounced increase in tenderness during the initial stages of aging, peaking around the 17th day. Following this peak, a gradual decline in tenderness was observed until the 20th day. Beyond this point, tenderness decreased slightly but remained relatively stable until the 36th day.

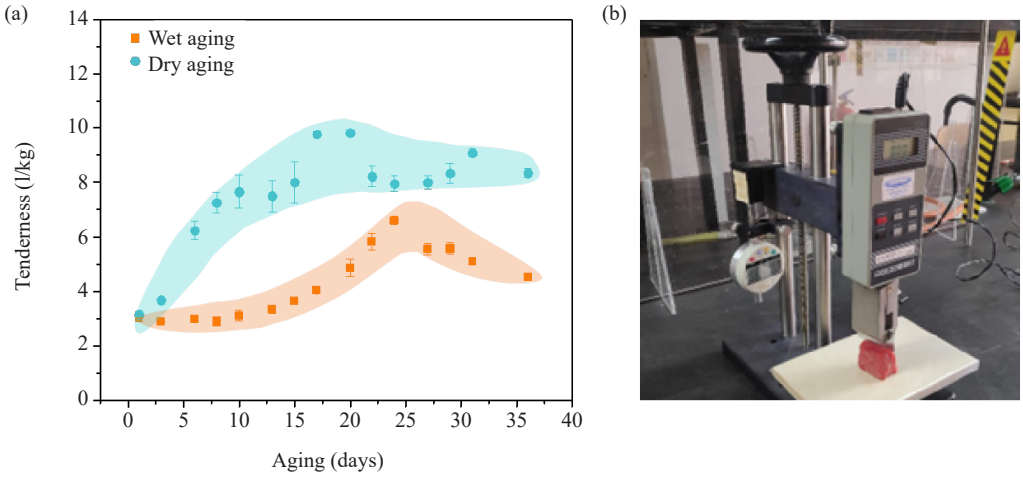

Figure 2. Tenderness evolution (a) under wet and dry aging processes and tenderness tester illustration (b). Error bars represent the standard deviation of each data point (n = 3)

The dry aging process demonstrated an earlier and more substantial increase in tenderness, with higher levels of tenderness achieved from the 5th day onward. The maximum reduction in force required to penetrate the samples in both aging processes indicates that dry aging results in a 14.3% greater tenderness value compared to wet aging. These results are consistent with other research, which has reported greater improvements in tenderness with dry aging compared to wet aging [21, 27, 32, 38]. The most pronounced effect of dry aging on tenderness occurs during the first two weeks of the aging process [7].

Additionally, the correlation between moisture content and tenderness for dry-aging samples was assessed, showing a decrease in tenderness with increasing moisture content, with a Spearman correlation coefficient of -0.7978 and a P-value < 0.001. No significant correlation was found for wet-aged samples.

3.3 *Study of volatile compounds related to sensory attributes in meat*

Many studies have explored the relationship between aging processes and sensory evaluation of meat samples [6, 39-41]. Key aroma compounds in meat, such as aldehydes, ketones, alcohols, and sulfur-containing compounds, are primarily formed during cooking through Maillard reactions. In raw meat, alcohols and aldehydes act as important precursors to these aromas. For this work, compounds identified in the literature as key aroma-active contributors to the aroma and flavor of raw meat, which subsequently undergoes thermal processing, were selected [42-46]. The aldehydes and alcohols selected, which significantly impact the product's aroma [47], can also serve as precursors to other volatile compounds generated during thermal processing. Table 1 presents the relationship between the analyzed volatile compounds and the aroma characteristics they impart to the sample, including odor thresholds and the connotations (positive or negative) consumers associate with each flavor type.

The contribution to aroma perception can be approximated by its odor activity value (OAV), which is defined as the concentration of a specific volatile compound divided by its odor threshold (OT) [48, 49]. This method has been widely regarded as effective for evaluating the contribution of individual aromas to both raw and cooked meat [44, 50]. Nevertheless, a modification of this approach has been proposed, emphasizing that the relative concentration of aroma compounds is more significant than the individual concentration of aroma compounds, as the ratio of aroma primarily describes the global flavor of the mixture [51, 52]. Accordingly, the aroma character impact (ACI), a relative measure of OAV, is considered more useful for comparing the aroma contributions of individual components within a mixture. The ACI is determined by dividing the percentage area of a volatile compound by its odor threshold.

The samples were analyzed using the solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) technique, with prior optimization of the instrumental setup using standards for the relevant compounds. Figure 3 illustrates the progression of ACI throughout the aging process for both dry-aged (a-b) and wet-aged (c-d) samples. Graphs a and c, located at the top of the figure, depict the evolution of volatiles with ACI values exceeding 2% area/ppb, while graphs b and d, positioned at the bottom of the figure, show the aromas with ACI values below 2% area/ppb throughout the process. The scales for dry-aged and wet-aged samples have been kept uniform to facilitate easier comparison of the levels. Samples were analyzed in triplicate, resulting in a relative standard deviation (RSD) range of 0.95% to 11.63%. Error bars were omitted from the graph to enhance the clarity of the observed trends.

Overall, the data presented in Figure 3 indicate that dry-aged samples exhibit higher levels of volatile compounds compared to wet-aged samples. This observation is consistent with findings from other authors who reported that compared to wet-aging, all the dry-aged samples presented significantly higher concentrations of all the compounds on all days examined [21, 53]. As discussed previously, the dry aging process involves partial dehydration of the meat, which contributes to flavor concentration and the enzymatic production of new volatile compounds [7, 34]. This leads to a higher level of aroma and flavor in dry-aged meats compared to those subjected to wet aging.

Starting from day 13, there is a marked increase in the formation of a cardboard-like aroma, attributed to E-2-nonenal, in both aging processes. This development marks the onset of undesirable flavors in the meat samples from that point onward. Additionally, 1-octen-3-ol, a volatile compound associated with a negative fungal aroma, remains significantly higher in the later stages of dry aging compared to wet aging. Hexanal and heptanal, which impart rancid and fishy odors to the meat, respectively, exhibit an earlier decline during the wet aging process compared to dry aging.

Table 1. Relationship between the volatile key aroma-active selected and the type of odor and flavor they provide to the sample, indicating the connotation that consumers attribute to each type of flavor

Volatile	Odor description[1]	OT[2] (ppb)	Consumer connotation
1-pentanol	balsamic	5.5	+
hexanal	rancid, grass	4.5	-
heptanal	fishy, oily	2.8	-
1-octen-3-ol	fungal	1.0	-
2-pentylfuran	beam, fruity, green	5.8	+
octanal	orange, fatty	0.6	+
E-2-nonenal	cardboard	1.0	-
1-hexanol	sweet, fruity, grassy	5.6	+

[1]Odor description found in the literature [37, 54-56] and online database [www.flavornet.org; www.odour.org.uk]
[2]Odor Threshold. All odor thresholds were obtained from literature [16, 17, 37, 54-56] and online database [www.chemicalbook.com, https://pubchem.ncbi.nlm.nih.gov/] with water applied as matrix and expressed in µg/kg

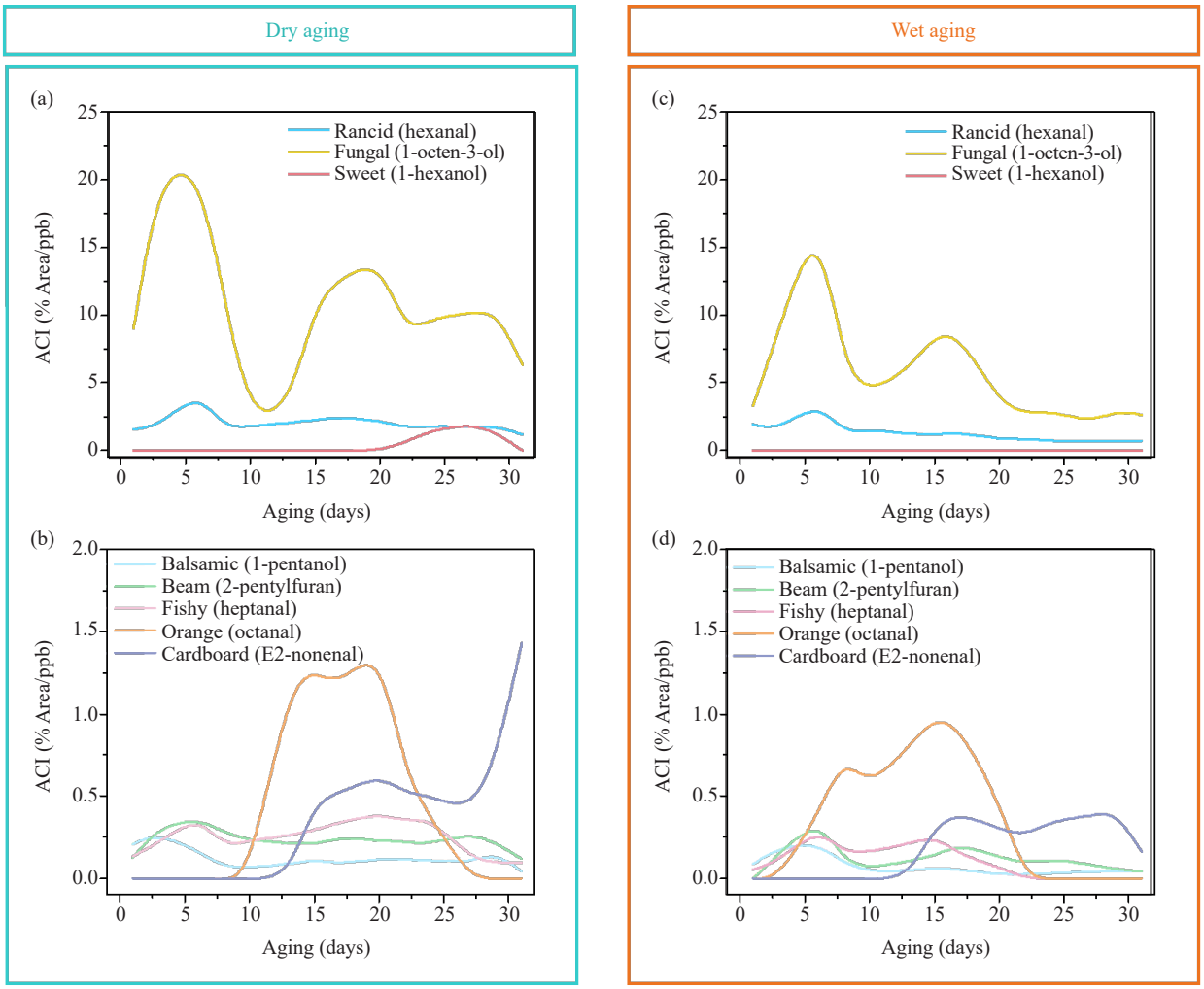

Figure 3. Evolution of the ACI values of the main volatile compounds responsible for aroma in meat samples subjected to the dry aging (a-b) process and wet aging (c-d)

In relation to desirable odors, the sweet odor associated with 1-hexanol becomes notably apparent after day 20 and before 30, exclusively in dry-aged samples, likely due to the aerobic conditions inherent in the dry aging process [3, 54]. In contrast, balsamic and beany odors, linked to 1-pentanol and 2-pentylfuran respectively, persist at slightly higher levels during dry aging. Additionally, the orange odor, attributed to octanal, emerges as one of the most significantly enhanced aromas in the dry aging process when compared to wet aging, increasing from 8 to 27 aging days in the former and from 3 to 22 aging days in the latter.

Researchers have identified aldehydes as crucial lipid-derived flavor compounds that significantly contribute to meat flavor due to their low odor detection threshold [55]. Among the aldehydes, hexanal is the most abundant, predominantly formed from the oxidation of unsaturated fatty acids (C18:2n-6) and unsaturated aldehydes [56]. Additionally, heptanal, octanal, and nonenal are as well products of unsaturated fatty acid oxidation [57, 58], but can also arise from microbial metabolism [59].

The main cause of the formation of aldehyde and alcohol volatiles in meat during maturation is lipid oxidation [60]. As maturation progresses, the exposure to atmospheric oxygen and the increase in free radicals accelerate this process, resulting in the progressive increase of volatiles concentration. Lipid oxidation can be catalyzed by enzymes like lipoxygenases and peroxidases present in muscle tissue or released through autolysis after slaughter. Additionally, enzymes such as cathepsins and calpains can expose more phospholipids to oxidation by breaking down structural proteins [6, 34]. These processes are influenced by maturation conditions (e.g., temperature, humidity, airflow), which

can either slow down or speed up the reactions [7, 10].

The decline of some of these volatiles during the maturation process can be attributed to several factors, including volatilization, with more volatile products like octanal, for example, evaporating earlier than E-2-nonenal. These volatiles may also participate in Maillard reactions or undergo oxidation into acids and other lower aldehydes and alcohols [61]. A key factor is the interaction with the meat matrix: as the protein matrix becomes more compact during maturation, volatile compounds are trapped in the protein and fat network, reducing their release into the headspace and effectively sequestering them in the meat.

The increased degradation of unsaturated fatty acids due to lipolysis during aging occurs at a higher rate in dry-aged muscles compared to wet-aged ones [21]. Additionally, the environmental conditions in dry aging may further enhance microbial metabolism compared to wet aging, which may partially explain the elevated ACI levels observed for aldehydes in dry-aged samples.

Alcohols such as 1-hexanol, 1-pentanol, and 1-octen-3-ol are also by-products of lipid oxidation (C18:2n-6 fatty acids) [62], and are promoted by the high concentration of oxygen in meat. The detection of 1-octene-3-ol is also associated with meat spoilage. Studies have shown a strong correlation between aldehydes, ketones, and alcohols, emphasizing their interrelationship in beef samples [3].

Additionally, 2-pentylfuran, as well derived from the oxidation of n-6 polyunsaturated fatty acids, has been proposed as a potential odor shelf-life marker for raw beef [63, 64], due to its association with bacteria responsible for meat spoilage.

As corroborated by other researchers, volatile compound detection is higher in the dry aging process compared to wet aging, attributed to two factors: the loss of water during dry aging, which results in greater flavor concentration, and the delayed deterioration observed in wet-aged samples [8, 9, 53, 65]. Consequently, volatile concentrations, whether contributing positively or negatively to flavor, were found elevated in dry-aged samples, with some originating from both fatty acid oxidation and microbial metabolism. Therefore, optimizing aging conditions (e.g., temperature, humidity, UV sterilization) could impact microbial development and regulate the levels of selected metabolites.

3.4 *Statistical analysis*

The analysis of variable correlations was performed for both dry and wet aging throughout the maturation process. A Spearman correlation method was employed, focusing on odor and tenderness variables. As illustrated in Figure 4, the correlation observed in dry aging involves fewer variables compared to wet aging. In dry aging (Figure 4a), a strong correlation was identified between cardboard-like odor and tenderness with the number of aging days. Additionally, fungal and beam aromas were found to be correlated, as were fishy odors with rancid and orange odors. To a lesser extent, sweet aroma also correlated with aging days. In the case of wet aging (Figure 4b), the strongest correlation was similarly found between cardboard-like odor and tenderness with aging days. A significant relationship was observed between fungal and fishy aromas, a pair that also correlated with other odors such as balsamic, rancid, and orange, and to a lesser degree, with tenderness and aging days. Rancid and cardboard aromas showed a slight correlation. These findings were also noticed in the principal components analysis (PCA) conducted in this study, which incorporated both tenderness and volatile compounds responsible for aromas with positive and negative connotations.

The objective of this multivariate analysis was to identify the aging day that yields the most favorable conditions for aged meat, characterized by the highest levels of desirable aromas, maximum tenderness, and minimal levels of undesirable aromas. A balance between positive and negative odors must be maintained, as negative odors become more prominent at a certain aging stage. Statistical tools were used in this study to optimize the balance, considering both tenderness and volatile compounds. Figure 5 presents the PCA results for samples undergoing dry (a) and wet (b) aging.

In the PCA analysis, parameters are organized into two principal components. The first two components account for 40.08% and 23.19% of the variance, respectively, totalling 63.27% for the dry aging process, and 56.23% and 15.26% of the variance, respectively, totalling 71.49% for the wet aging process. Notably, aging days, tenderness, and cardboard-like odor are clustered in the first quadrant across both aging processes. Additionally, beam, fungal rancid, and fishy odors are grouped in the second quadrant, while balsamic odor is positioned in the third quadrant for both processes. As can be noticed in Figure 5, the distribution of odor-tenderness components differs between dry and wet aging, with higher number of aging days associated with the tenderness variable in the wet aging process.

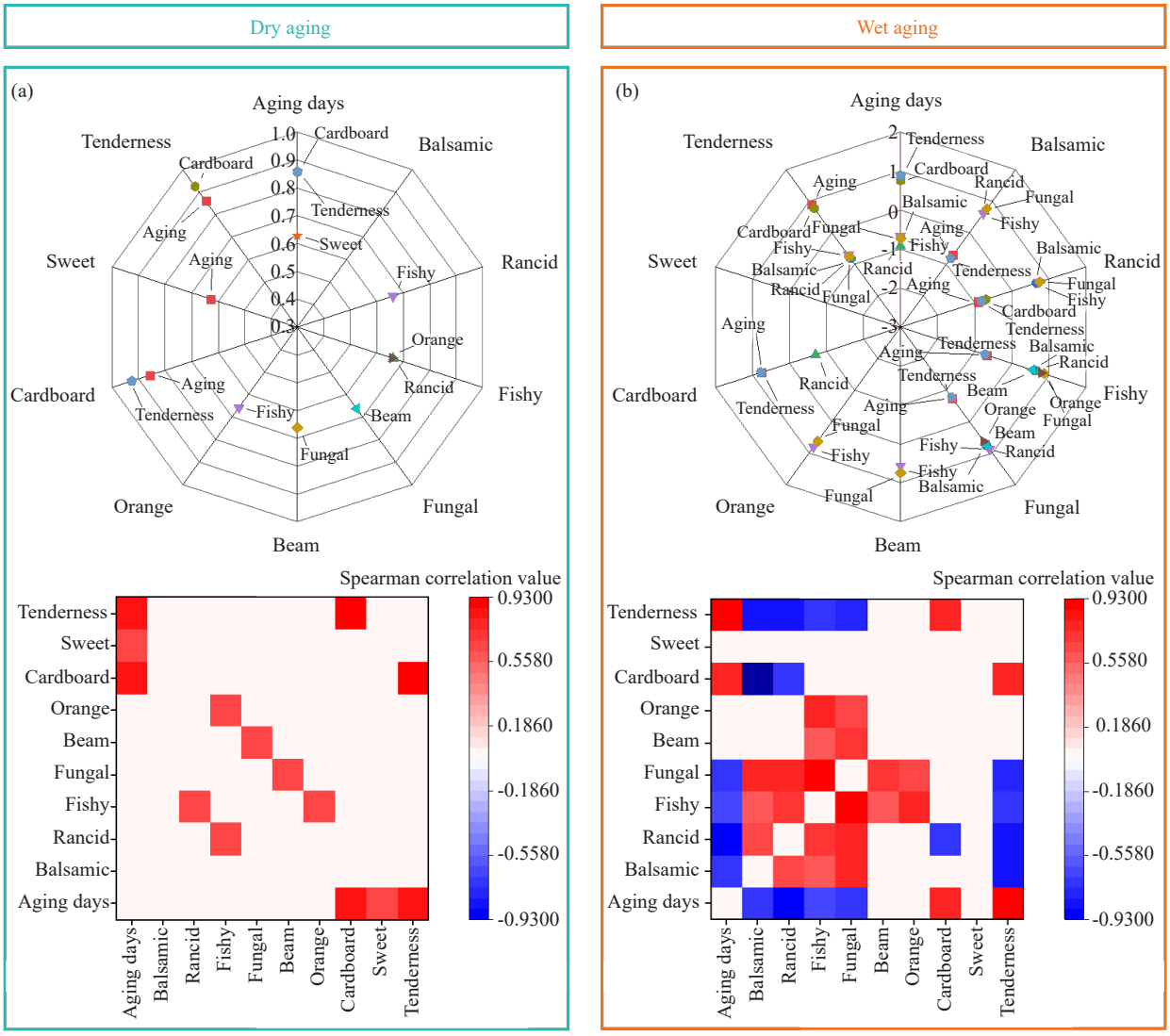

Figure 4. Spearman correlation analysis between aromas, tenderness and aging days for the dry aging (a) and wet aging (b) processes, illustrated as a radar chart in the upper side, where the radial axes represent the different odors, and as a heatmap on the lower side, each cell in the chart representing a specific value in a matrix of data. On the right side of the chart, you see a color scale that indicates which value corresponds to each color, with the colors ranging from blue (representing lower or negative values) to red (representing higher or positive values), with white representing values near zero

Sensory perception of flavor and tenderness is the most important yardstick for evaluating the period of aging. When a sensory analysis and/or olfactometry is conducted, there will be a combination of flavors and sensations that can lead to different perceptions. PCA results provide objective values to the subjectivity of olfactometric methods, achieving more robust results through the combination of both methods. To determine the desired aging period that best combines the most favorable characteristics of the meat, a weighted sum model was applied, incorporating variables such as ACI of the 8 selected key aroma-active, with a + or - sign assigned based on whether the aroma is pleasant or unpleasant, respectively, as well as tenderness. The analysis indicated that, during the dry aging process, the 10th day yielded the most favorable objective results, followed by the 13th and 31st days. In contrast, for the wet aging process, the 24th day proved to be the most favorable, with the 27th and 22nd days also showing significant potential. These optimal aging days are illustrated in Figure 5.

This method enables the adjustment of desirable characteristics in aged meat by modifying the weight assigned to each attribute, such as aroma or tenderness, to align with consumer preferences.

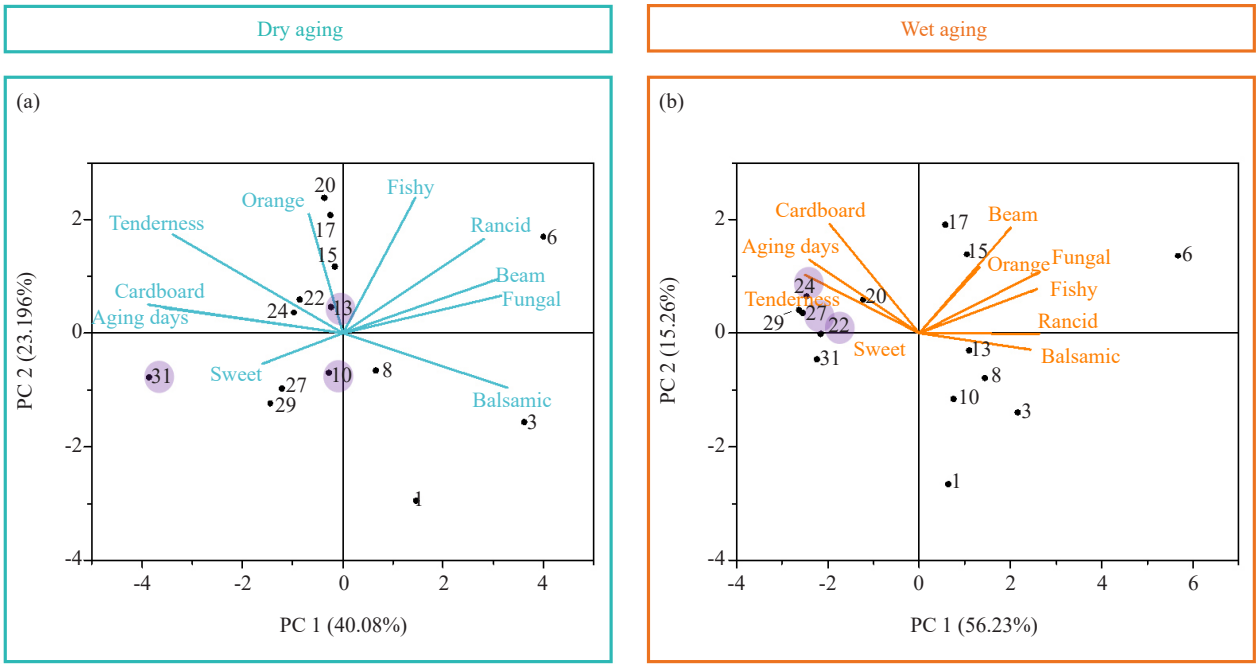

Figure 5. PCA for the (a) dry aging and (b) wet aging processes. The numbers near the data points indicate the day of aging. The highlighted numbers denote the desired aging period, as determined by the weighted sum of variables

4. Conclusions

This article investigates the effects of dry and wet aging on various quality parameters of beef over a 31-day period. Key variables such as water content, pH, tenderness, and volatile profiles were assessed at regular intervals. The results indicated that dry-aged beef exhibited slightly higher pH levels and greater water loss compared to wet-aged beef. Tenderness increased more rapidly in dry-aged beef, peaking around day 17, while wet-aged beef showed a slower progressive increase until day 25. Volatile compounds responsible for aroma were also more abundant in dry-aged beef, contributing to a higher concentration of flavor. Principal component analysis (PCA) identified the optimal aging days to maximize tenderness and desirable aromas while minimizing undesirable ones. The results indicated that, under the studied specific conditions and for the type of meat analyzed, dry-aged samples reached optimal conditions at an earlier stage (10-13 days) than those subjected to wet aging (24-27 days). In conclusion, dry aging appears to offer significant advantages in terms of flavor concentration and tenderness improvement, although it involves greater weight and volume loss.

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Conflict of interest

The authors declare no conflict of interest.

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