**A Dissertation for the Degree of Master**

# Elucidation of characteristic flavor of dry-aged beef and its intensification by cooking conditions

건식숙성 우육의 풍미 구명 및 조리조건에 의한 풍미증진 효과

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# Abstract

# Elucidation of characteristic flavor of dry-aged beef and its intensification by cooking conditions

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Dry-aged beef is well known for its unique flavor compared to unaged or wet-aged beef. However, in spite of the importance, little research has been conducted to identify the origin of the characteristic flavor of dry-aged beef. In order to understand and to even enhance it, the analysis on the formation of aroma volatile compounds in dry-aged beef under various conditions is necessary. Therefore, the objectives of this study were i) to investigate the change of volatile compounds in dry-aged beef during aging process, and ii) to suggest the optimal cooking conditions for intensifying the flavor of dry-aged beef. For this, two consecutive experiments were conducted as follows:

**Experiment I.**

**Effect of different aging methods on the formation of aroma volatiles in beef strip loins**

The effects of different aging methods on the changes in the concentrations of aroma volatiles of beef were investigated. Each of fifteen strip loins were dry- and wet-aged for 28 days and their aroma volatiles were analyzed at a 7-day interval ($n = 3$ for each aging period). As the aging period increased, dry-aged beef showed higher concentrations of volatile compounds than those in wet-aged beef ($p < 0.05$). Most changes in the concentrations of aroma volatiles of dry-aged beef were associated with propanal, 2-methylbutanal, 2-methylpropanal, 1-butanamine, trimethylamine, 2-methyl-2-propanethiol, and ethyl propanoate, which were mainly produced by lipid oxidation and/or microbial activity (e.g., proteolysis and lipolysis) during the dry aging period. These compounds represent malty, nutty, fruity, ammoniacal and fermented flavors. Therefore, it is suggested that the differences in aroma between dry- and wet-aged beef could result from increased lipid oxidation and microbial activity in dry-aged beef possibly owing to its ambient exposure to oxygen.

**Experiment II.**

**Effects of cooking conditions on the physicochemical and sensory characteristics of dry- and wet-aged beef**

The effects of cooking conditions on the physicochemical and sensory characteristics of dry- and wet-aged beef strip loins were evaluated. Dry- and wet-

aged beef aged for 28 days were cooked at different combinations of cooking method (grilling or oven roasting) × cooking temperatures (150°C or 230°C), and their cooking time, pH, cooking loss, 2-thiobarbituric acid reactive substances (TBARS), volatile compounds, and color were measured. Cooking conditions did not affect pH, however, grilling resulted in lower TBARS but higher cooking doneness at the surface of dry-aged beef compared to oven roasting ($p < 0.05$). In descriptive sensory analysis, the roasted flavor of dry-aged beef was significantly stronger when grill-cooked compared to oven roasting. Dry-aged beef grill-cooked at 150°C presented a higher intensity of cheesy flavor, and that grilled at 230°C showed a greater intensity of roasted flavor compared to wet-aged beef at the same condition, respectively. Therefore, we suggest that grilling may be effective for enhancing the unique flavor in dry-aged beef.

**Keywords**: Dry aging, Flavor, Aroma volatiles, Aging periods, Cooking method, Cooking temperature

**Student Number**: 2019-29878

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## Chapter II.



## Chapter III.

# List of Abbreviations

| | | |
|---|---|---|
| $a^*$ | : | Redness |
| $b^*$ | : | Yellowness |
| MDA | : | Malondialdehyde |
| $L^*$ | : | Lightness |
| PCA | : | Principal component analysis |
| PLS-DA | : | Partial least squares-discriminant analysis |
| TBARS | : | 2-Thiobarbituric acid reactive substances |
| UFA | : | Unsaturated fatty acid |
| VIP | : | Variable importance in projection |

# Chapter I.

# General introduction

Dry aging refers to the aging method where the meat is stored for a period of time without packaging, in order to increase the eating quality of meat (Lee et al., 2019b; J. Oh et al., 2019). Among the various attributes, tenderness, juiciness, and flavor of meat are generally considered as the most important factors determining the eating quality of meat (Aaslyng et al., 2017; Yoo et al., 2020). Especially, the flavor of dry-aged beef is regarded as a unique sensory property which cannot be observed in unaged or wet-aged beef (Ha et al., 2019). As consumers begin to seek high-quality and novel flavor of meat, dry-aged beef has gaining popularity these days (Park et al., 2018).

Flavor is a complex attribute which consists of aroma and taste, and plays a critical role in determining the eating quality of meat and resultant consumer preference (Khan et al., 2016; Van Ba et al., 2012). Raw beef has metallic and bloody flavors, however, the formation of roasted and beefy flavor occurs in beef mainly by Maillard reaction and lipid oxidation during cooking (Kerth et al., 2015; Van Ba et al., 2012). Furthermore, the flavor of cooked meat can be influenced by cooking methods and cooking temperatures due to the difference in the degree and efficiency of heat transfer (Domínguez et al., 2014; Echegaray et al., 2020; Wall et al., 2019).

On the other hand, the increase of flavor precursors during dry aging was

reported in several studies, and their concentration were even higher compared to those in wet-aged beef (Kim et al., 2019; Lee et al., 2019a). Thus, in case of the formation of flavor in dry-aged beef, both aging condition and cooking condition should be taken account because of the generation of flavor precursors during dry aging period and the occurrence of Maillard reaction, lipid oxidation, and interaction between Maillard reaction products and lipid oxidation-derived products (Angel-Rendon et al., 2020; H. C. Kim et al., 2020; Lee et al., 2019b). Compared to taste compounds, aroma volatile compounds in dry-aged beef were rarely investigated despite their importance in understanding the flavor of dry-aged beef and optimizing the aging condition and cooking condition of dry-aged beef to intensify its flavor.

This study will provide the information about the formation of aroma volatile compounds in beef strip loins during 28 days of dry aging and suggest potential factors influencing the flavor of dry-aged beef in aging process. Furthermore, it will help to search optimize cooking conditions for enhancing dry-aged flavor of beef by comparing the physicochemical and sensory properties of dry-aged beef cooked at different cooking methods and cooking temperatures.

This manuscript consists of part of a paper which is published in *Foods* as partial fulfillment of the Master's program of Dongheon Lee.

# Chapter II.

# Effect of different aging methods on the formation of aroma volatiles in beef strip loins

## 2.1. Introduction

Aging is the process of storing meat in a controlled environment for a certain period to increase the palatability of meat (Khan et al., 2016). There are two forms of aging: dry and wet (J. H. Kim et al., 2017). Dry aging involves holding the meat unpacked in the open air where temperature, relative humidity, and air flow velocity are under control (Lee et al., 2019a). In wet aging, on the other hand, meat is vacuum-packaged and stored in a refrigerated condition (Khan et al., 2016). Because of the different aging conditions, the flavor of dry- and wet-aged beef is known to be discriminable. In general, dry-aged beef has more beefy, roasted, and nutty flavors, while wet-aged beef has more intense sour, metallic, and bloody flavors (M. Kim et

al., 2019).

Flavor is important for determining eating quality of meat, which affects consumer preferences (Van Ba et al., 2012). Flavor is defined as the combination of taste and aroma and is attributed to different flavor compounds (Aaslyng et al., 2017). Flavor compounds such as amino acids, sugars, organic acids, and inorganic salts can contribute to the five different taste sensations and participate in aroma development of beef under heating condition (Mottram, 1998). Therefore, in order to explain the different flavor of dry- and wet-aged beef, several studies compared the flavor compounds of dry- and wet-aged beef. J. H. Kim et al. (2017) reported that dry-aged beef had significantly higher amounts of free amino acids, including glutamic acid and aspartic acid, than those of wet-aged beef. In addition, Lee et al. (2019b) confirmed that dry aging of beef for 28 days showed significantly higher content of free amino acids and reducing sugars than those in wet aging of beef.

As for the aroma, Watanabe et al. (2015) and Yang et al. (2019) reported that wet aging had an effect on increasing the levels of volatile compounds such as aldehydes and furans as the aging period increased. However, there is little information about aroma volatiles in dry-aged beef, which could be important to understand the characteristic of dry-aged beef flavor and its contributors. From this point of view, the changes in the concentrations of volatile compounds of beef with different aging methods may provide valuable information to elucidate the effect of dry aging on its desirable flavor. Considering the description of flavor in dry- and wet-aged beef from literatures (Khan et al., 2016, J. H. Kim et al., 2019), we

hypothesized that each aging method (dry and wet aging) may have different effects on the formation of aroma volatiles in beef, leading to differences in the change of volatile patterns during the aging process. Therefore, we analyzed the volatile compounds in dry- and wet-aged beef during 28 days of aging period.

## 2.2. Materials and methods

### *2.2.1. Raw material and aging process*

In this study, 30 beef strip loins (longissimus lumborum) from both sides of 15 carcasses (21-month-old Holstein steers, quality grade 2) were purchased at 48 h post-mortem and transferred to a laboratory. The quality grade of the samples was based on the Korean beef grading system (KAPE, 2019). Approximately 500 g of lean meat was cut from each strip loin and its initial pH (5.52 ± 0.01) was measured before aging (SevenGo, Mettler-Toledo, Schwerzenbach, Switzerland). Then, the samples were allocated to dry or wet aging randomly (n = 15 for each aging method). Parameters for each aging method were set according to Lee et al. (2019b). For wet aging, the samples were vacuum packaged (HFV-600L, Hankook Fujee Machinery Co., Ltd., Hwaseong, Korea) in low density polyethylene/nylon bags ($O_2$ permeability of 2 mL/$m^2$/d at 0°C; 0.09 mm thickness; Sunkyung Co., Ltd., Seoul, Korea) and stored at 4°C, while dry aging was processed at 4°C, relative humidity of 75%, and air flow velocity of 2.5 m/s without any packaging. Both dry and wet aging processes continued for 28 days and the samples from each group were collected on day 0, 7, 14, 21, and 28 (n = 3 for each aging period). Before sampling, the crust of dry-aged beef (approximately 0.5 cm from surface) was trimmed off. The beef samples were vacuum packaged and frozen at −70°C until the volatile compound analysis.

### *2.2.2. Volatile compounds analysis*

Volatile compounds in dry- and wet-aged beef were analyzed by electronic nose (Heracles II, Alpha MOS, Toulouse, France) following the method of Lee et al. (2019b). The frozen samples were thawed for 12 h at 4°C and ground using a meat grinder (MG510, Kenwood, Hampshire, UK). Then, each sample (5 g) was weighed in a 20-mL vial and cooked for 10 min at 80°C to obtain the volatile compounds without possible loss in sampling process after cooking. Then, the volatiles were injected into an electronic nose equipped with dual columns of MXT-5 and MXT-1701 (10 m × 180 μm × 0.4 μm; length × diameter × thickness) (Restek, Bellefonte, PA, USA). The analytical conditions for volatile compounds were in Table 1. Each peak was integrated and identified using retention time and relevance index indicating the percentage of matching probability based on the comparison of Kovats retention index of the detected compound and the Kovats retention indices of known compounds from the AroChemBase library (Alpha MOS).

Table 1. Analytical conditions of electronic nose for volatile compounds in dry- or wet-aged beef strip loins during 28 days of aging

| Analytical conditions | | |
|---|---|---|
| Headspace generation | Incubation temperature | 80°C |
| | Incubation time | 10 min |
| Trap | Initial temperature | 40°C |
| | Split | 10 mL/min |
| | Trapping duration | 30 s |
| | Final temp. | 240°C |
| Injector | Carrier gas | Hydrogen |
| | Injected volume | 5 mL |
| | Injected speed | 250 μL/s |
| | Injector temperature | 200°C |
| Column | Column temperature | 40°C for 5 s<br>150°C (0.5°C/s, 5 s)<br>260°C (5°C/s, 30 s) |
| | Acquisition duration | 282 s |
| Detector | Type | Flame ionized detector (dual) |

### *2.2.3. Mold distribution*

Mold distribution at the surface of dry-aged beef was analyzed using photographic imaging software (Adobe Photoshop CC 2015, Adobe, California, USA) according to J. Oh et al. (2019). The photo of dry-aged beef illuminated with an LED light (MS-273, Myung Sung, Suwon, Korea) at 108 lx was taken (CMOS 16.0 MP, Samsung Co., Suwon, Korea). Mold distribution was measured by calculating the proportion of pixels over 128 levels in blue channel.

### *2.2.4. Free fatty acids*

Free fatty acids contents in dry- and wet-aged beef were assessed by the method of Lee et al. (2019a). Briefly, 1 g of lipid was put into the test tube with 1 mL of chloroform and an internal standard (1 mg of triundecanoate/mL isooctane). After removing triglycerides from the samples, free fatty acids were extracted using 2% acetic acid in diethyl ether. The extract was evaporated with nitrogen gas and heated at 85°C for 10 min. After that, 2 mL of 14% boron trifluoride-methanol was put into the test tube for methylation and heated at the same condition. Then, 2 mL of isooctane and 1 mL of saturated sodium chloride were added into the test tube and centrifuged at 1573 × g for 3 min (Continent 512R, Hanil Co. Ltd., Daejeon, Korea). The upper layer containing fatty acid methyl ester (FAME) was dehydrated with anhydrous sodium sulfate. FAME was analyzed using a gas chromatography (HP 7890, Agilent Technologies, Santa Clara, CA, USA) with DB-23 column (60 m × 250 μm × 0.25 μm; length × diameter × thickness) (Supelco, Bellefonte, PA, USA).

Each FAME was identified by comparing the retention time of external standards (Supelco® 37 Component FAME mix, Sigma-Aldrich, St. Louis, MO, USA).

### *2.2.5. Statistical analysis*

All samples for volatile analysis were triplicated and statistical analysis was performed using SAS 9.4 program (SAS Institute Inc., Cary, NC, USA). The effects of different aging methods on the aroma pattern in volatile changes of beef strip loins were evaluated by two-way analysis of variance. Mean values with standard error of the means were reported and their significant differences were determined by Student-Newman-Keuls multiple comparison test at a significance level of 0.05. PCA was performed to discriminate aroma patterns in dry- and wet-aged beef by their volatile compounds. Pearson correlation coefficients between volatile compounds, mold distribution and unsaturated fatty acids of aged beef were analyzed.

## 2.3. Results and discussion

### *2.3.1. Volatile profiling of aged beef*

A total of 37 volatile compounds in dry- and wet-aged beef was identified during 28 days of aging period (Table 2). They were assigned to the following chemical groups: aldehydes, furan and ketone (n = 6), N-compounds (n = 4), S-compounds (n = 3), alcohols (n = 4), hydrocarbons, esters and acids (n = 13), and others (n = 7).

Table 2. Identified volatile compounds in dry- or wet-aged beef strip loins during 28 days of aging

| No | Volatile compounds | RT[1] | RI[2] | Aroma description[3] | Odor threshold[4] (ppm) |
|---|---|---|---|---|---|
| | | | *Aldehyde, furan and ketone* | | |
| 1 | (E, E)-2, 4-Hexadienal | 116.86 | 81.10 | Citrus, floral, green, spicy, sweet | 94.8[P] |
| 2 | 2-Methylbutanal | 28.30 | 92.02 | Etheral, nutty[N], sweet[N] | 1[N] |
| 3 | 2-Methylpropanal | 16.39 | 51.36 | Camphor, green[H], malty[H], pungent[H] | 0.7[P] |
| 4 | Propanal | 14.13 | 65.50 | Almond, cherry, green[N], fruity[N] | 25.1[N] |
| 5 | Tetrahydrofuran | 21.72 | 93.82 | Aromatic, burnt, fruity, sulfurous, sweet | 92-61,000[A] |
| 6 | 3-Heptanone | 87.14 | 90.67 | Fatty, fruity, green, spicy, sweet | 140[G] |
| | | | *N-compound* | | |
| 1 | 1-Butanamine | 25.03 | 85.38 | Ammoniacal, fishy | 170[I] |
| 2 | Ethenyl-dimethylpyrazine | 147.23 | 93.75 | Earthy, musty | no reference |

| | | | | | |
|---|---|---|---|---|---|
| 3 | 2-Pentylpyridine | 108.94 | 84.18 | Fatty, green, mushroom, pepper, tallowy | 5[F] |
| 4 | Trimethylamine | 11.10 | 74.73 | Ammoniacal, fishy, fruity, oily, pungent | 2.4[N] |
| | | | *S-compound* | | |
| 1 | 2-Methyl-2-propanethiol | 17.99 | 88.64 | Sulfurous | 0.33[E] |
| 2 | Carbon disulfide | 17.30 | 71.60 | Burnt[H], cabbage[H], fruity[H], sulfurous[H] | 210[I] |
| 3 | Dimethyl trisulfide | 101.67 | 85.46 | Alliaceous, cabbage, fishy, meaty, onion | 0.1[N] |
| | | | *Alcohol* | | |
| 1 | 1-Methoxy-2-propanol | 34.76 | 76.18 | Mild | 839-33,000[A] |
| 2 | 2-Butanol | 28.35 | 56.68 | Pleasant, strong, sweet, wine | 220[I] |
| 3 | 4-Methyl-1-hexanol | 97.21 | 92.13 | Grassy, sweaty, nutty[D], oily[D], roasty[D] | 2,000[D] |
| 4 | 4-Nonanol | 139.31 | 76.28 | Sweet[L] | no reference |

*Hydrocarbon, ester and acid*

| | | | | | |
|---|---|---|---|---|---|
| 1 | 2, 2-Dichloropropane | 21.81 | 89.93 | no reference | no reference |
| 2 | 3-Methyldecane | 134.73 | 82.85 | Balsamic, mild, phenolic | no reference |
| 3 | 4-Methyldecane | 132.13 | 91.50 | Fatty[O], fresh[O], waxy[O] | no reference |
| 4 | Butane | 11.08 | 68.12 | Faint | 1,200,000[I] |
| 5 | Ethylcyclopentane | 38.58 | 76.25 | Alkane, fruity, gasoline, sweet | no reference |
| 6 | Heptane | 33.95 | 60.05 | Floral, fruity, sweet | 400,000[M] |
| 7 | Octane | 41.04 | 95.87 | Alkane, fruity, sweet, fatty[H], solvent[H] | 1,700[I] |
| 8 | Ethyl propanoate | 31.10 | 86.29 | Burnt, fermented, fruity, green, malty | 0.01[J] |
| 9 | Methyl 2-methylbutanoate | 44.63 | 90.84 | Fatty, fruity, green | 0.4[B] |
| 10 | Methyl 2-butenoate | 41.36 | 77.98 | Black currant, fruity | no reference |
| 11 | Propyl propanoate | 58.53 | 90.15 | Fruity, green, sweet | 0.88[J] |
| 12 | 2-Methylpropanoic acid | 49.12 | 77.12 | Dairy, fatty, pungent, rancid, sour, sweaty | 50[G] |

| | | | | | |
|---|---|---|---|---|---|
| 13 | Hexanoic acid | 110.28 | 91.42 | Cheesy, fatty, pungent, rancid, sour, sweaty | 3,000,000[C] |
| | | | *Others* | | |
| 1 | 1, 2, 4-Thiadiazole, 5-ethoxy-3-(trichloromethyl)- | 248.10 | 87.34 | Mild | no reference |
| 2 | Demeton-O | 264.10 | 59.09 | no reference | no reference |
| 3 | Diisopropyl ether | 16.83 | 89.64 | Etheral | no reference |
| 4 | Ethyl chloride | 13.93 | 92.29 | Etheral, pungent | 3800-379,000[A] |
| 5 | Limonene | 125.69 | 78.19 | Citrus, fruity, minty | 38[I] |
| 6 | P-cymene | 121.27 | 93.22 | Citrus, fruity, herbaceous, pleasant, solvent | 120[K] |
| 7 | Perfluorononane | 9.25 | 87.80 | no reference | no reference |

[1]RT, retention time.

[2]RI, relevance index indicating the percentage of matching probability based on the comparison of Kovats retention index of the detected compound and the Kovats retention indices of known compounds from the AroChemBase library.

[3,4]Odor descriptions and odor threshold values from AroChemBase library and journals (A to P): A, American Industrial Hygiene Association (1989); B, Blank et al. (1992); C, Casaburi et al. (2015); D, Dregus et al. (2003); E, Giannoukos et al. (2019); F, Hou et al. (2019); G, Keatkrai et al. (2010); H, Madruga et al. (2010); I, Nagata (2003); J, Niu et al. (2019); K, Ozkara et al. (2019); L, Saini et al. (2012); M, Wojnowski et al. (2017); N, Wu et al. (2014); O, Yu et al. (2019); P, Zhu et al. (2017).

*2.3.1.1. Aldehydes, furan and ketone*

Aldehyde contents increased in dry-aged beef, while a decreasing trend during wet aging was observed, with some fluctuations as the aging duration increased (Table 3). From day 14, the dry-aged beef had significantly higher concentration of total aldehydes compared to those in wet-aged beef. The changes in the contents of aldehydes were affected mainly by propanal, which was predominant in both aging conditions but much higher in dry aging. Dry-aged beef also had significantly higher abundance of 2-methylbutanal than that in wet-aged beef during the whole aging period, and the concentration of 2-methylbutanal was the highest at day 28. 2-Methylpropanal level significantly increased at day 28 in dry-aged beef, whereas it could not be observed in wet-aged beef from day 14. (E, E)-2, 4-Hexadienal content decreased significantly after 28 days of both dry and wet aging, although the concentrations were relatively small compared to other aldehydes. Propanal is considered an indicator of lipid oxidation (Van Ba et al., 2012). Thus, the difference in propanal content between dry- and wet-aged beef, especially after 14 days, might result from the different susceptibility in the lipid oxidation process. Lipid oxidation is restrained during wet aging, because vacuum packaging prevents the exposure of meat to oxygen (Van Ba et al., 2012). Kahraman et al. (2019) reported that TBARS values of dry-aged beef were significantly higher than those of wet-aged beef from 14 days of aging, indicating that oxidation of lipid occurred more actively during dry aging. 2-Methylbutanal and 2-methylpropanal can be formed by Strecker degradation of isoleucine and valine, respectively (Muriel et al., 2004). Y. H. B. Kim et al. (2016) observed that the levels of tryptophan, phenylalanine, valine, tyrosine,

glutamate, isoleucine, and leucine were significantly higher in 3-week dry-aged beef compared to wet-age beef. Lee et al. (2019a; 2019b) also showed that the concentration of 18 free amino acids, including isoleucine and valine, was significantly higher in dry-aged beef than those in wet-aged beef mainly due to microbial proteolysis. Therefore, we suggest that higher concentrations of 2-methylbutanal and 2-methylpropanal in dry-aged beef than those in wet-aged beef could be attributed to the higher concentration of isoleucine and valine due to the increased microbial activity during dry aging (Lee et al., 2019b). It was reported that microbial metabolism favored the production of branched aldehydes in fermented meat products (Bruna et al., 2001). Aldehydes contribute largely to beef aroma with sweet, floral, salty, and cheesy notes, because they have low odor thresholds (Domínguez et al., 2019; Ozkara et al., 2019). Hence, differences in aldehyde content between differently aged beef could play an important role in creating a characteristic aroma.

Furans are odor-active volatiles formed by the oxidation of fatty acids (Ozkara et al., 2019). As shown in Table 3, both types of aging methods increased the concentration of tetrahydrofuran with the increase in aging time ($p < 0.05$). Especially, dry-aged beef showed a significantly higher concentration of tetrahydrofuran than that in wet-aged beef after 14 days of aging process.

3-Heptanone was the only ketone compound detected in the experiment. Generally, ketones are known as lipid-oxidation products with low odor thresholds (Muriel et al., 2004). The concentration of 3-heptanone increased after dry aging (p

< 0.05), and it was higher at day 14 and 21 than in any other periods. On the other hand, it significantly decreased in wet-aged beef at day 7 and disappeared at day 14 and 21. It was detected at day 28 and was significantly lower compared to that in unaged beef.

While moderate lipid oxidation could enhance the flavor of meat (DeGeer et al., 2009), excessive lipid oxidation products may be potentially harmful to human health as these compounds can react with cellular compartments strongly (Suleman et al., 2020; Vieira, Zhang, & Decker, 2017). One way to prevent the negative effect of lipid oxidation products is to intake the dietary antioxidants from foods. Furthermore, adopting adequate cooking methods may be effective to reduce excessive lipid oxidation (discussed in section 3.3.7.).

Table 3. Peak area of aldehydes, furan and ketone in beef during aging with different aging methods

| Compounds | Aging method | Aging period (day) | | | | | SEM[1] |
|---|---|---|---|---|---|---|---|
| | | 0 | 7 | 14 | 21 | 28 | |
| *Aldehyde* | | | | | | | |
| (E, E)-2, 4-Hexadienal | Dry | 376$^{a}$ | 137$^{bx}$ | 99$^{c}$ | 113$^{cy}$ | 153$^{bx}$ | 6.5 |
| | Wet | 376$^{a}$ | 121$^{by}$ | 94$^{c}$ | 137$^{bx}$ | 56$^{dy}$ | 6.7 |
| | SEM[2] | 10.5 | 2.9 | 8.8 | 4.1 | 2.5 | |
| 2-Methylbutanal | Dry | 845$^{c}$ | 742$^{cx}$ | 2495$^{bx}$ | 1667$^{cx}$ | 4503$^{ax}$ | 246.9 |
| | Wet | 845$^{b}$ | 529$^{cy}$ | 341$^{dy}$ | 639$^{cy}$ | 1827$^{ay}$ | 47.8 |
| | SEM[2] | 7.8 | 46.1 | 353.7 | 40.5 | 170.9 | |
| 2-Methylpropanal | Dry | 506$^{b}$ | 657$^{by}$ | 864$^{bx}$ | 1077$^{bx}$ | 2458$^{ax}$ | 309.1 |
| | Wet | 506$^{b}$ | 821$^{ax}$ | nd$^{cy}$ | nd$^{cy}$ | nd$^{cy}$ | 7.3 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | SEM[2] | 11.4 | 9.0 | 38.2 | 9.4 | 487.1 | |
| Propanal | Dry | 17635[c] | 9067[dy] | 43508[ax] | 20185[cx] | 38743[bx] | 1139.6 |
| | Wet | 17635[a] | 14111[bx] | 16915[ay] | 12344[cy] | 14394[by] | 275.4 |
| | SEM[2] | 300.0 | 663.7 | 676.5 | 838.4 | 1321.1 | |
| Total aldehydes | Dry | 19361[b] | 10603[cy] | 46966[ax] | 23042[bx] | 45857[ax] | 1174.5 |
| | Wet | 19361[a] | 15582[cx] | 17349[by] | 13119[dy] | 16277[cy] | 313.7 |
| | SEM[)] | 296.2 | 683.3 | 1027.0 | 841.9 | 1173.3 | |
| *Furan* | | | | | | | |
| Tetrahydrofuran | Dry | 292[e] | 900[d] | 2143[cx] | 3731[bx] | 6262[ax] | 60.6 |
| | Wet | 292[e] | 960[d] | 1128[cy] | 1399[by] | 3068[ay] | 21.4 |
| | SEM[2] | 9.4 | 31.2 | 28.6 | 35.8 | 84.6 | |
| *Ketone* | | | | | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 3-Heptanone | Dry | $104^{e}$ | $352^{dx}$ | $1237^{bx}$ | $1465^{ax}$ | $675^{cx}$ | 19.4 |
| | Wet | $104^{a}$ | $83^{by}$ | $nd^{dy}$ | $nd^{dy}$ | $60^{cy}$ | 3.6 |
| | SEM[2] | 6.6 | 17.4 | 7.3 | 22.6 | 8.0 | |

[1]Standard error of mean (n = 15), [2](n = 6).

[a–e]Different letters within same row differ significantly ($p < 0.05$).

[x,y]Different letters within same column differ significantly ($p < 0.05$).

nd, not detected.

*2.3.1.2. N-compounds*

Total N-compounds were mostly higher in wet-aged beef than those in dry-aged beef until day 21 (Table 4). However, at the end of the aging period, dry-aged beef showed higher concentration of N-compounds than those in wet-aged beef ($p < 0.05$). Especially, except ethenyl-dimethylpyrazine, the levels of these compounds dramatically increased at the late phase of dry aging (day 21 to 28). During that time, the concentration of 1-butanamine in dry-aged beef increased more than 23 fold. Similarly, trimethylamine level increased approximately 16 fold from day 21 to 28. As for 2-pentylpyridine, it was detected only at day 28 in dry-aged beef. The formation of amine compounds is usually attributed to the degradation of amino acids due to the decarboxylase activity of microorganisms (Iucci et al., 2007). Trimethylamine can be produced from the reduction of trimethylamine oxide by microorganisms and has been widely used for the assessment of microbial activity (Wu et al., 2014). 2-Pentylpyridine is believed to be formed by the interaction of 2, 4-decadienal with either ammonia or α-amino group of amino acids (Mottram, 1998). Finally, pyrazines result from Maillard reaction (Van Ba et al., 2012). Considering the possible origins of the N-compounds, it seemed that proteolysis and degradation of amino acids might be the main contributors to the increase in the concentration of these products. Proteolysis is influenced by the action of muscle endogenous enzymes and/or microorganism-origin enzymes (Lee et al., 2019a). Muscle endogenous proteolytic enzymes are responsible for meat tenderization at the early period of aging; however, their activities decrease as aging duration increases (Khan et al., 2016). The activity of aminopeptidases C and H, which could contribute to the

increase in the amount of free amino acids during aging, was the highest at day 4; however, it decreased afterwards and was maintained until day 50 (Iida et al., 2016). In this regard, protein degradation at the late period might be related more to microbial enzyme activity than muscle endogenous enzymes (Lee at al., 2019b). Lee et al. (2019b) found that the levels of free amino acids and trimethylamine were significantly higher in dry-aged beef at day 28 compared to those in wet-aged beef, indicating that growth of mold and/or yeast at the surface of dry-aged beef might result in further proteolysis. Altogether, change in the concentrations of N-compounds at the late phase of aging were highly noticeable, especially in dry-aged beef, and this observation was likely to be associated with different microbial enzyme activities. Differences in the concentration of N-compounds in dry- and wet-aged beef could discriminate their aroma characteristic as these compounds are the most important flavor precursors for meaty or beef flavor, with very low odor detection thresholds (Aaslyng et al., 2017; Yang et al., 2019).

Table 4. Peak area of N-compounds in beef during aging with different aging methods

| Compounds | Aging method | Aging period (day) | | | | | SEM[1] |
|---|---|---|---|---|---|---|---|
| | | 0 | 7 | 14 | 21 | 28 | |
| 1-Butanamine | Dry | 274$^{b}$ | 72$^{by}$ | 334$^{bx}$ | 133$^{by}$ | 3048$^{ax}$ | 62.9 |
| | Wet | 274$^{a}$ | 135$^{cx}$ | 145$^{cy}$ | 218$^{bx}$ | 87$^{dy}$ | 5.5 |
| | SEM[2] | 11.4 | 7.6 | 4.8 | 10.9 | 98.2 | |
| Ethenyl-dimethylpyrazine | Dry | 443$^{b}$ | 751$^{ax}$ | 441$^{bx}$ | 488$^{bx}$ | 507$^{b}$ | 41.9 |
| | Wet | 443$^{a}$ | 357$^{by}$ | 246$^{cy}$ | 218$^{cy}$ | 364$^{b}$ | 10.1 |
| | SEM[2] | 6.5 | 42.5 | 20.5 | 7.1 | 48.1 | |
| 2-Pentylpyridine | Dry | nd$^{b}$ | nd$^{b}$ | nd$^{b}$ | nd$^{by}$ | 425$^{ax}$ | 1.4 |
| | Wet | nd$^{c}$ | nd$^{c}$ | nd$^{c}$ | 361$^{ax}$ | 188$^{by}$ | 8.4 |
| | SEM[2] | - | - | - | 1.1 | 13.4 | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Trimethylamine | Dry | 11806$^{b}$ | 4728$^{c}$ | 2045$^{dy}$ | 1052$^{dy}$ | 17487$^{ax}$ | 546.6 |
| | Wet | 11806$^{a}$ | 4924$^{d}$ | 6727$^{cx}$ | 6949$^{cx}$ | 9019$^{by}$ | 212.8 |
| | SEM[2] | 110.4 | 611.2 | 145.3 | 232.9 | 631.7 | |
| Total N-compounds | Dry | 12523$^{b}$ | 5551$^{c}$ | 2820$^{dy}$ | 1673$^{dy}$ | 21466$^{ax}$ | 605.5 |
| | Wet | 12523$^{a}$ | 5415$^{d}$ | 7117$^{cx}$ | 7746$^{cx}$ | 9658$^{by}$ | 216.6 |
| | SEM[2] | 127.4 | 601.9 | 154.1 | 220.4 | 763.5 | |

[1]Standard error of mean (n = 15), [2](n = 6).

$^{a–d}$Different letters within same row differ significantly ($p < 0.05$).

$^{x,y}$Different letters within same column differ significantly ($p < 0.05$).

nd, not detected.

*2.3.1.3. S-compounds*

Overall, S-compound levels increased with time in both aging processes (Table 5). Those concentrations were mostly higher in dry-aged beef from day 14 compared to those in wet-aged beef. 2-Methyl-2-propanethiol level showed a tendency to increase during dry aging, especially from day 21 to 28. An exception was carbon disulfide at day 28, which was more abundant in wet-aged beef. With regards to dimethyl trisulfide, it generally decreased in both dry- and wet-aged beef, and, eventually, no difference was found between them at the end of the aging period ($p < 0.05$). S-compounds originate from the degradation of S-containing amino acids such as methionine, cysteine, and cystine (Casaburi et al., 2015). Carbon disulfide and 2-propanethiol can be produced via Strecker degradation of S-containing amino acids (Sekhon et al., 2010). Dimethyl trisulfide is particularly related to methionine degradation (Flores, 2018). Differences in S-compounds between dry- and wet-aged beef might also result from the different occurrence of proteolysis during aging period.

Table 5. Peak area of S-compounds in beef during aging with different aging methods

| Compounds | Aging method | Aging period (day) | | | | | SEM[1] |
|---|---|---|---|---|---|---|---|
| | | 0 | 7 | 14 | 21 | 28 | |
| 2-Methyl-2-propanethiol | Dry | 1025$^{d}$ | 875$^{dy}$ | 5342$^{bx}$ | 2729$^{cx}$ | 9680$^{ax}$ | 142.8 |
| | Wet | 1025$^{c}$ | 1644$^{ax}$ | 1015$^{cy}$ | 754$^{dy}$ | 1477$^{by}$ | 30.6 |
| | SEM[2] | 41.2 | 24.0 | 193.9 | 76.1 | 87.7 | |
| Carbon disulfide | Dry | 1174$^{d}$ | 758$^{dy}$ | 4401$^{cx}$ | 5267$^{bx}$ | 8403$^{ay}$ | 168.3 |
| | Wet | 1174$^{e}$ | 3628$^{bx}$ | 1523$^{dy}$ | 1989$^{cy}$ | 9787$^{ax}$ | 62.7 |
| | SEM[2] | 8.4 | 67.6 | 68.2 | 234.8 | 127.1 | |
| Dimethyl trisulfide | Dry | 122$^{a}$ | 125$^{ay}$ | 138$^{ax}$ | 101$^{bx}$ | 79$^{c}$ | 4.7 |
| | Wet | 122$^{b}$ | 133$^{ax}$ | 79$^{cy}$ | 59$^{ey}$ | 66$^{d}$ | 1.7 |
| | SEM[2] | 2.7 | 1.3 | 4.1 | 4.5 | 4.1 | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Total S-compounds | Dry | 2320$^{d}$ | 1758$^{dy}$ | 9882$^{bx}$ | 8097$^{cx}$ | 18162$^{ax}$ | 261.7 |
| | Wet | 2320$^{e}$ | 5405$^{bx}$ | 2616$^{dy}$ | 2802$^{cy}$ | 11330$^{ay}$ | 49.9 |
| | SEM[2] | 32.5 | 63.0 | 223.2 | 300.5 | 179.7 | |

[1]Standard error of mean (n = 15), [2](n = 6).

$^{a–e}$Different letters within same row differ significantly ($p < 0.05$).

$^{x,y}$Different letters within same column differ significantly ($p < 0.05$).

*2.3.1.4. Alcohols*

Total alcohol contents in dry-aged beef significantly increased with the increase in aging period, whereas those in wet-aged beef decreased from the beginning and then increased after day 14 (Table 6). Therefore, from the early phase of aging period, dry-aged beef showed significantly higher alcohol contents than those in wet-aged beef. The increase in alcohol levels in dry-aged beef was mostly attributed to the increase in 2-butanol concentration. It significantly increased during 28 days of dry aging, except for day 7. Wet-aged beef also showed an increase in 2-butanol concentration with the increase in aging period. However, its change was relatively lower than that in dry aging, resulting in significantly lower concentration than that in dry aging from day 21. During 28 days of aging period, the concentration of 1-methoxy-2-propanol in dry-aged beef peaked at day 14 and significantly decreased thereafter, but it was still higher than that in wet-aged beef, which gradually decreased and then increased at day 28. Besides, 4-methyl-1-hexanol was present only when the beef was dry-aged for 14 days or longer. 4-Nonanol appeared at an earlier time in dry-aged beef than that in wet-aged beef, although no significant differences were found from day 21. Unlike straight-chain alcohols, which generally result from the oxidation of UFAs, 2-butanol and branched-chain alcohols with low molecular weight are produced by microbial fermentation (Muriel et al., 2004). The significant differences in the levels of 2-butanol, 1-methoxy-2-propanol, and a branched-chain alcohol, 4-methyl-1-hexanol, in dry- and wet-aged beef might imply the effect of different microbial activities on the aroma volatiles of aged beef.

Table 6. Peak area of alcohol compounds in beef during aging with different aging methods

| Compounds | Aging method | Aging period (day) | | | | | SEM[1] |
|---|---|---|---|---|---|---|---|
| | | 0 | 7 | 14 | 21 | 28 | |
| 1-Methoxy-2-propanol | Dry | 749$^{d}$ | 723$^{dx}$ | 2532$^{ax}$ | 2212$^{bx}$ | 1515$^{cx}$ | 36.8 |
| | Wet | 749$^{a}$ | 291$^{by}$ | 106$^{cy}$ | 17$^{dy}$ | 316$^{by}$ | 20.7 |
| | SEM[2] | 33.7 | 37.3 | 24.9 | 17.8 | 31.4 | |
| 2-Butanol | Dry | 102$^{c}$ | nd$^{dy}$ | 891$^{c}$ | 2005$^{bx}$ | 5308$^{ax}$ | 231.3 |
| | Wet | 102$^{d}$ | 146$^{dx}$ | 236$^{c}$ | 402$^{by}$ | 890$^{ay}$ | 19.1 |
| | SEM[2] | 3.9 | 4.2 | 280.7 | 41.5 | 232.5 | |
| 4-Methyl-1-hexanol | Dry | nd$^{d}$ | nd$^{d}$ | 120$^{cx}$ | 168$^{ax}$ | 131$^{bx}$ | 3.2 |
| | Wet | nd | nd | nd$^{y}$ | nd$^{y}$ | nd$^{y}$ | - |
| | SEM[2] | - | - | 2.2 | 1.9 | 4.2 | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 4-Nonanol | Dry | nd$^{c}$ | nd$^{c}$ | 19$^{bx}$ | 60$^{b}$ | 135$^{a}$ | 14.1 |
| | Wet | nd$^{c}$ | nd$^{c}$ | nd$^{cy}$ | 60$^{b}$ | 177$^{a}$ | 11.0 |
| | SEM[2] | - | - | 13.2 | 3.3 | 24.9 | |
| Total alcohols | Dry | 850$^{d}$ | 723$^{dx}$ | 3562$^{cx}$ | 4444$^{bx}$ | 7089$^{ax}$ | 215.8 |
| | Wet | 850$^{b}$ | 437$^{cy}$ | 342$^{dy}$ | 479$^{cy}$ | 1382$^{ay}$ | 28.2 |
| | SEM[2] | 31.3 | 38.1 | 266.2 | 50.9 | 206.2 | |

[1]Standard error of mean (n = 15), [2](n = 6).

[a–d]Different letters within same row differ significantly ($p < 0.05$).

[x,y]Different letters within same column differ significantly ($p < 0.05$).

nd, not detected.

*2.3.1.5. Hydrocarbons, esters, and acids*

We observed a significant increase in total hydrocarbons in aged beef, regardless of the aging method (Table 7). Total hydrocarbon levels were significantly higher in dry-aged beef than those in wet-aged beef at day 14 and 21 of aging. When aging duration reached 28 days, however, total hydrocarbon levels were more abundant in wet-aged beef compared to those in dry-aged one. 2, 2-Dichloropropane and heptane levels significantly increased during wet aging period, and, at the last day of aging, the concentrations of the compounds in wet-aged beef were significantly higher than those in dry-aged beef. On the contrary, butane and octane contents were significantly higher in dry-aged beef than those in wet-aged beef during the aging process. 3- and 4-Methyldecane and ethylcyclopentane were detected in dry-aged beef only. When compared to the changes in hydrocarbon in dry-aged beef, its higher concentration in wet-aged beef were not expected as it was believed to be derived mainly from the autoxidation of lipids (Flores, 2018). As for wet-aged beef, Ma et al. (2012) stated that 21 days of postmortem storage affected little the changes in levels of volatile compounds and assumed that lipid oxidation progressed slowly up to 3 weeks under vacuum conditions. It is hard to explain the significantly higher concentration of 2, 2-dichloropropane in wet-aged beef than that in dry-aged beef. Nonetheless, this would not be the main factor for the distinctive aroma of dry- or wet-aged beef as hydrocarbons have limited effect on the flavor of meat because of their high odor detection thresholds (Maggiolino et al., 2019). Meanwhile, 3- and 4-methyldecane, which appeared only in dry-aged beef, could be generated by the secondary degradation of triglycerides by the activity of surface molds, possibly

indicating the differences in microflora in dry- and wet-aged beef (Domínguez et al., 2019).

After 28 days of aging period, the concentrations of esters increased in dry-aged beef and decreased in wet-aged beef ($p < 0.05$). During dry aging, a significant increase in ethyl propanoate level was detected from day 14. Moreover, an approximately 6-fold increase in its concentration at day 28 compared to day 0 was observed. In contrast, ethyl propanoate content was significantly lower in wet-aged beef from day 7 to 21 compared to that in unaged beef. Methyl 2-butenoate was present only in dry-aged beef from day 14. Concentration of propyl propanoate in both dry- and wet-aged beef showed lower values than those in unaged beef, and its content was higher in wet-aged beef at day 28 compared to that in dry-aged beef ($p < 0.05$). Finally, methyl 2-methylbutanoate levels showed significant changes in both dry- and wet-aged beef until day 21, but no significant difference was found in dry- and wet-aged beef at the last day of aging. Esters are produced by esterification reaction of alcohols and acids (Ozkara et al., 2019) and are related to the activity of microbial esterase (Casaburi et al., 2015). Bruna et al. (2001) reported that dry sausages inoculated with *Penicillium aurantiogriseum* had higher levels of esters, while sausages without inoculation had very few ester compounds. Corral et al. (2018) found that *Debaryomyces hansenii* inoculated into the sausages was responsible for the increase in the levels of ester compounds. Thus, differences in ester contents in dry- and wet-aged beef might indicate differences in metabolic activity of microorganisms affected by different aging methods.

As for acids, significantly higher concentrations of acids were observed in dry-aged beef after 14 days of aging compared to those in wet-aged beef. Generally, acids increased during dry aging, while they significantly decreased after wet aging. 2-Methylpropanoic acid was found only in dry-aged beef from day 14, and it decreased at day 28 ($p < 0.05$). Hexanoic acid was not detected in dry-aged beef at day 7, but its level was significantly higher in dry-aged beef after 14 days compared to that in wet-aged beef. 2-Methylpropanoic acid can result from the oxidation of 2-methylpropanal, and hexanoic acid can result from lipid oxidation (Muriel et al., 2004). In particular, Casaburi et al. (2015) stated that branched-chain fatty acids such as 2- and 3-methylbutanoic acids were observed in aerobically-stored meat, not in vacuum-packaged meat. These compounds accounted for only a small portion of volatile contents in both dry- and wet-aged beef. Nevertheless, acids are regarded as important compounds, which can be used as substrates for the production of esters, strongly affecting the aroma of beef products (Flores, 2018).

Table 7. Peak area of hydrocarbons, esters and acids in beef during aging with different aging methods

| Compounds | Aging method | Aging period (day) | | | | | SEM[1] |
|---|---|---|---|---|---|---|---|
| | | 0 | 7 | 14 | 21 | 28 | |
| *Hydrocarbon* | | | | | | | |
| 2, 2-Dichloropropane | Dry | 2604$^{e}$ | 4274$^{d}$ | 12837$^{bx}$ | 11361$^{c}$ | 15398$^{ay}$ | 319.2 |
| | Wet | 2604$^{c}$ | 4931$^{c}$ | 5041$^{cy}$ | 7597$^{b}$ | 20477$^{ax}$ | 659.1 |
| | SEM[2] | 35.8 | 231.0 | 405.9 | 992.9 | 368.0 | |
| 3-Methyldecane | Dry | nd$^{b}$ | nd$^{b}$ | nd$^{b}$ | nd$^{b}$ | 98$^{ax}$ | 1.2 |
| | Wet | nd | nd | nd | nd | nd$^{y}$ | - |
| | SEM[2] | - | - | - | - | 1.9 | |
| 4-Methyldecane | Dry | nd$^{b}$ | nd$^{b}$ | nd$^{b}$ | nd$^{b}$ | 149$^{ax}$ | 1.7 |
| | Wet | nd | nd | nd | nd | nd$^{y}$ | - |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | SEM[2] | - | - | - | - | 2.6 | |
| Butane | Dry | 251[d] | 316[bx] | 280[cx] | 297[bcx] | 715[ax] | 8.5 |
| | Wet | 251[b] | 203[dy] | 233[cy] | 251[by] | 261[ay] | 1.7 |
| | SEM[2] | 1.1 | 2.7 | 0.6 | 2.9 | 13.1 | |
| Ethylcyclopentane | Dry | nd[c] | nd[c] | 799[bx] | 1430[ax] | 1372[ax] | 48.9 |
| | Wet | nd | nd | nd[y] | nd[y] | nd[y] | - |
| | SEM[2] | - | - | 31.7 | 61.9 | 33.9 | |
| Heptane | Dry | nd[d] | nd[d] | 169[ax] | 127[cy] | 149[by] | 6.3 |
| | Wet | nd[d] | nd[d] | 28[cy] | 1370[bx] | 1537[ax] | 40.5 |
| | SEM[2] | - | - | 20.5 | 35.4 | 50.2 | |
| Octane | Dry | 803[d] | 937[cd] | 998[cx] | 1300[bx] | 1791[ax] | 44.0 |
| | Wet | 803[b] | 844[b] | 485[cy] | 967[ay] | 602[cy] | 36.2 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | SEM[2] | 60.5 | 44.3 | 18.0 | 14.9 | 44.0 | |
| Total hydrocarbons | Dry | 3658$^{d}$ | 5527$^{c}$ | 15083$^{bx}$ | 14515$^{bx}$ | 19671$^{ay}$ | 340.0 |
| | Wet | 3658$^{c}$ | 5978$^{c}$ | 5787$^{cy}$ | 10184$^{by}$ | 22877$^{ax}$ | 692.2 |
| | SEM[2] | 58.2 | 265.9 | 426.9 | 1043.7 | 375.7 | |
| *Ester* | | | | | | | |
| Ethyl propanoate | Dry | 1009$^{c}$ | 381$^{d}$ | 2854$^{bx}$ | 3087$^{bx}$ | 6761$^{ax}$ | 107.5 |
| | Wet | 1009$^{a}$ | 327$^{b}$ | 254$^{by}$ | 298$^{by}$ | 829$^{ay}$ | 73.3 |
| | SEM[2] | 154.2 | 74.8 | 25.8 | 35.1 | 105.0 | |
| Methyl 2-methylbutanoate | Dry | 326$^{b}$ | 205$^{dy}$ | 261$^{cx}$ | 367$^{ax}$ | 358$^{a}$ | 7.6 |
| | Wet | 326$^{b}$ | 234$^{dx}$ | 202$^{ey}$ | 280$^{cy}$ | 372$^{a}$ | 8.2 |
| | SEM[2] | 13.2 | 6.7 | 4.2 | 5.6 | 6.6 | |
| Methyl 2-butenoate | Dry | nd$^{b}$ | nd$^{b}$ | 123$^{ax}$ | 187$^{ax}$ | 158$^{ax}$ | 30.9 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Wet | nd | nd | nd$^{y}$ | nd$^{y}$ | nd$^{y}$ | - |
| | SEM[2] | - | - | 3.8 | 5.9 | 48.3 | |
| Propyl propanoate | Dry | 3015$^{a}$ | 80$^{ey}$ | 715$^{bx}$ | 322$^{dx}$ | 471$^{cy}$ | 15.1 |
| | Wet | 3015$^{a}$ | 380$^{cx}$ | 150$^{dy}$ | 176$^{dy}$ | 1183$^{bx}$ | 20.5 |
| | SEM[2] | 23.4 | 19.9 | 11.9 | 8.0 | 21.6 | |
| Total esters | Dry | 4349$^{b}$ | 666$^{d}$ | 3953$^{cx}$ | 3964$^{cx}$ | 7748$^{ax}$ | 88.5 |
| | Wet | 4349$^{a}$ | 941$^{c}$ | 606$^{dy}$ | 754$^{cdy}$ | 2383$^{by}$ | 65.8 |
| | SEM[2] | 136.2 | 77.5 | 29.6 | 44.5 | 54.7 | |
| *Acid* | | | | | | | |
| 2-Methylpropanoic acid | Dry | nd$^{c}$ | nd$^{c}$ | 87$^{ax}$ | 82$^{ax}$ | 64$^{bx}$ | 1.9 |
| | Wet | nd | nd | nd$^{y}$ | nd$^{y}$ | nd$^{y}$ | - |
| | SEM[2] | - | - | 2.6 | 1.0 | 1.2 | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Hexanoic acid | Dry | 167$^{b}$ | nd$^{cy}$ | 165$^{bx}$ | 140$^{bx}$ | 248$^{ax}$ | 9.5 |
| | Wet | 167$^{a}$ | 99$^{bx}$ | nd$^{cy}$ | 108$^{by}$ | 119$^{by}$ | 8.7 |
| | SEM[2] | 15.8 | 6.7 | 5.5 | 5.9 | 7.6 | |
| Total acids | Dry | 167$^{d}$ | nd$^{ey}$ | 252$^{bx}$ | 222$^{cx}$ | 311$^{ax}$ | 8.9 |
| | Wet | 167$^{a}$ | 99$^{bx}$ | nd$^{cy}$ | 108$^{by}$ | 119$^{by}$ | 8.7 |
| | SEM[2] | 15.8 | 6.7 | 4.1 | 5.6 | 6.6 | |

[1]Standard error of mean (n = 15), [2](n = 6).

$^{a–e}$Different letters within same row differ significantly ($p < 0.05$).

$^{x,y}$Different letters within same column differ significantly ($p < 0.05$).

nd, not detected.

### *2.3.2. Volatile patterns of aged beef*

The patterns of aroma volatiles in beef sirloin were changed with different trends in dry or wet aging (Fig. 1). For wet-aged beef, similar volatile patterns were observed until day 21, and only day 28 led to relatively distinct volatile patterns compared to those in unaged beef. The effect of wet aging on the development of volatile compounds is controversial. Watanabe et al. (2015) observed that wet aging for 30 days significantly increased the levels of oxygen, nitrogen, and sulfur heterocyclic compounds and concluded that wet aging could affect the flavor of beef. Similarly, Yang et al. (2019) found that the levels of volatile compounds (e.g., aldehydes, alkanes, pyrazines, and furans) increased in beef after 14 days of wet aging. In contrast, several studies documented that wet aging duration of 2–3 weeks was not enough for the production of additional volatile compounds (Ma et al., 2012; Maggiolino et al., 2019). In this study, the differentiation in volatile patterns of wet-aged beef was affected mainly by the increase in 2, 2-dichloropropane, carbon disulfide, and tetrahydrofuran levels at the late phase of wet aging (Tables 3, 5, and 7). However, when considering the high odor thresholds (Table 2) of 2, 2-dichloropropane, carbon disulfide, and tetrahydrofuran, the aroma of wet-aged beef may not be unique (Nagata et al., 2003), which is in accordance with Khan et al. (2016).

On the contrary, diverse volatile patterns were observed in beef during dry aging (Fig. 1). From day 14, the aroma pattern of dry-aged beef could be differentiated from those of wet- or unaged beef. Similarly, Lee et al. (2019b) mentioned that the dry-aged flavor began to be perceived from day 14 to 21 of aging period, and umami

flavors could be intensified by extended aging. We observed an overall increase in levels of volatile compounds in dry-aged beef, which were much higher than those in wet-aged beef. Lipid oxidation may be an important factor for the development of dry-aged flavor, because a large proportion of volatile compounds (e.g., propanal, hydrocarbons, furan and ketone) derive from lipid oxidation. Among them, aldehydes are known as low odor threshold products, and higher concentration of propanal in dry-aged beef may be responsible for dry-aged flavor. Also, PCA loading plot (Fig. 2) showed that 3-heptanone might be relevant to the characteristic volatile pattern in day 14 to 21 when its concentration was higher than other aging periods. Since 3-heptanone shows relatively low odor threshold value compared to other lipid oxidation-derived products like most hydrocarbons (Table 2), it might be regarded as an important volatile compound among the lipid oxidation-derived products in dry-aged beef. Moreover, the changes in the concentrations of N-compounds may be the key for the characteristic of aroma volatiles in dry-aged beef at day 28, indicating the importance of microbial activity in the formation of the unique dry-aged flavor. Given the low odor threshold values of trimethylamine (Table 2), it might be important for dry-aged flavor that the significant increase of trimethylamine was observed in dry-aged beef at day 28. This finding suggests the importance of microbial activity in the formation of the unique dry-aged flavor. Earlier studies also reported noticeable changes in levels of flavor compounds at the late phase of dry aging (from day 21 to 28). Lee et al. (2019b) observed significant increases in levels of flavor compounds of dry-aged beef between day 21 and 28 and explained that further proteolysis and lipolysis associated with microorganisms (especially mold

and/or yeast) could develop aroma compounds of dry-aged beef. H. Oh et al. (2019) observed that dry-aged beef inoculated with *Pilaira anomala* had higher oleic, palmitic, and stearic acid content at day 21, while inoculation with *D. hansenii* resulted in higher amounts of free amino acids at day 28. Finally, the contribution of 2-methylpropanal, 2-methylbutanal, 2-methyl-2-propanethiol, ethyl propanoate, and possibly 2-methylpropanoic acid to dry-aged flavor should be noted when their low odor threshold values (Table 2) and concentrations between in dry- and wet-aged beef are taken account (Tables 3, 5, and 7). Particularly, the result that at day 14 of aging or more, 2-methylpropanal and 2-methylpropanoic acid only existed in dry-aged beef indicates the potential key volatile compounds. The rapid increase in the levels of these amino acid-degradation products (e.g., 2-methylbutanal, 2-methylpropanal, and 2-methyl-2-propanthiol) may result from the increase in levels of flavor precursors by microbial metabolism at the late phase of dry aging. Although other compounds, e.g. 2-butanol and tetrahydrofuran, showed significantly higher concentrations in dry-aged beef compared to wet-aged beef, for their relatively high odor threshold values, they might act as minor contributors for dry-aged flavor.

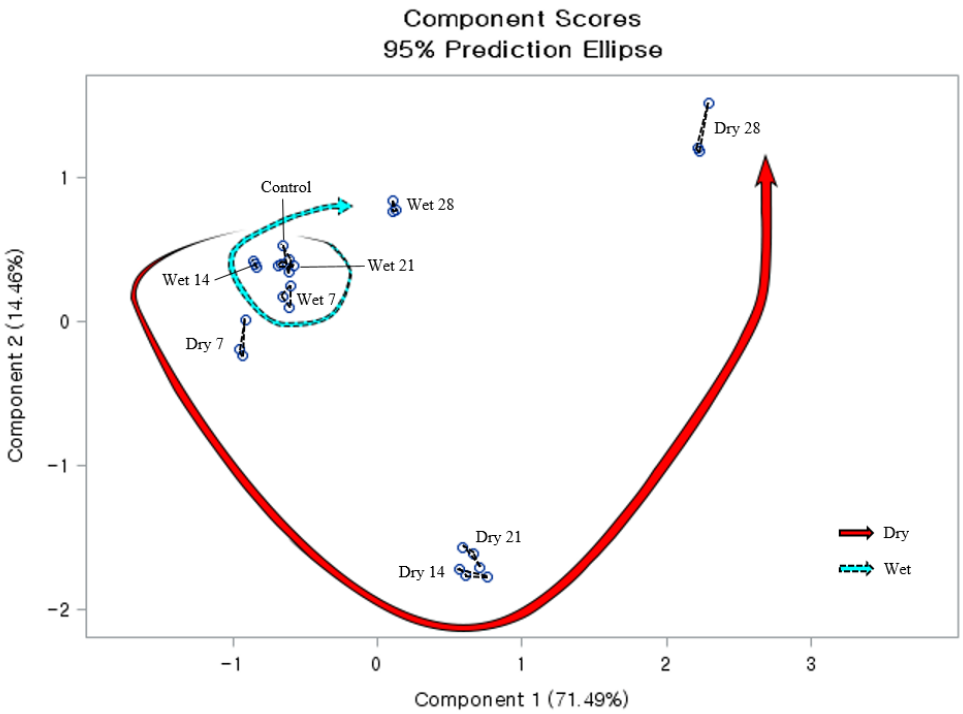


Figure 1. PCA score plot for the changes in patterns of aroma volatiles of beef during aging with different aging methods. Red solid-line arrow illustrates the change in aroma patterns in dry-aged beef, and blue dotted-line arrow indicates the change in aroma patterns in wet-aged beef during aging.

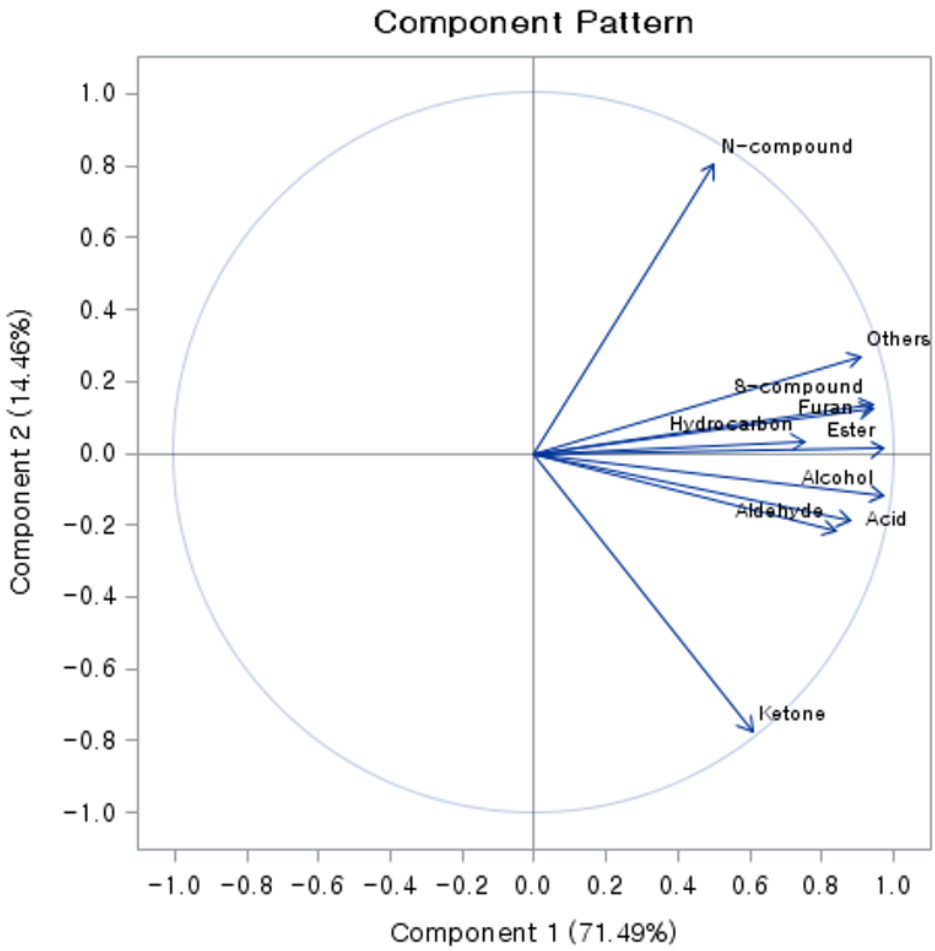


Figure 2. PCA loading plot for the changes in patterns of aroma volatiles of beef during aging with different aging methods.

*2.3.3. Correlation analysis with mold distribution and UFA*

The degree of mold distribution and the amounts of free fatty acids of dry- and wet-aged beef were measured to evaluate the potential effects of microbial activity and lipid oxidation on the change of aroma volatile compound profile of dry- and wet-aged beef. The percentage of mold distribution on the surface of dry-aged beef was significantly increased during 28 days of aging period (Table 9). Similarly, J. Oh et al. (2019) observed the significant increase of mold distribution value on the surface of dry-aged beef after 28 and 35 days of dry aging. They also suggested that the quantification of mold on the surface of dry-aged beef could be effective for evaluating the degree of dry aging. H. C. Kim et al. (2020) reported the accelerated proteolysis at the crust of dry-aged beef based on the result of free amino acid and biogenic amine contents. On the other hand, the existence of mold on the surface of wet-aged beef could not be identified after 7 days of aging period due to the anaerobic condition.

Different trends in the amounts of free fatty acids were observed during 28 days of dry and wet aging, respectively. In dry-aged beef, most free fatty acids were more abundant at day 14 than other periods, while their contents in wet-aged beef were the highest at day 21 ($p < 0.05$). The amount of total saturated fatty acids was significantly higher in wet-aged beef compared to dry-aged beef from 21 days of aging period. However, at the end of aging period (28 days), dry-aged beef had significantly higher UFAs compared to wet-aged beef. Especially, the increase of linoleic acid at the late phase of dry aging (from day 21 to day 28) was noticeable in contrast to wet aging period. Propanal, which was the most abundant compound in

dry-aged beef, and other linear aldehydes (hexanal, nonanal, etc.) can be derived from UFAs such as linoleic acid (Pérez-Santaescolástica et al., 2018; Wu et al., 2014). This might explain the higher concentration of propanal in dry-aged beef compared to wet-aged beef (Table 3). Corral et al. (2018) reported that the inoculation of *D. hansenii* into the dry fermented sausages containing boar backfat increased the amounts of free UFAs and total free fatty acids after 63 days. H. Oh et al. (2019) also found out the overall increase of free fatty acids in dry-aged beef inoculated with *P. anomala* and *D. hansenii*, respectively, indicating the high lipolytic activity of *P. anomala* and *D. hansenii* during dry aging.

Table 8. Mold distribution (%) and free fatty acids contents (mg/g) of beef during aging with different aging methods

| Items | Aging method | Aging period (day) | | | | | SEM[1] |
|---|---|---|---|---|---|---|---|
| | | 0 | 7 | 14 | 21 | 28 | |
| Mold distribution (%) | Dry | 0.16$^{b}$ | 0.02$^{bx}$ | 0.58$^{bx}$ | 0.87$^{bx}$ | 6.18$^{ax}$ | 0.321 |
| | Wet | 0.16$^{a}$ | nd$^{by}$ | nd$^{by}$ | nd$^{by}$ | nd$^{by}$ | 0.041 |
| | SEM[2] | 0.041 | 0.002 | 0.013 | 0.084 | 0.500 | |
| C14:0 (mg/g) | Dry | 0.22$^{b}$ | 0.21$^{b}$ | 0.29$^{ax}$ | 0.22$^{by}$ | 0.22$^{bx}$ | 0.012 |
| | Wet | 0.22$^{b}$ | 0.20$^{b}$ | 0.19$^{by}$ | 0.63$^{ax}$ | 0.16$^{by}$ | 0.017 |
| | SEM[2] | 0.015 | 0.019 | 0.020 | 0.009 | 0.007 | |
| C15:0 (mg/g) | Dry | 0.14$^{c}$ | 0.27$^{bx}$ | 0.32$^{ax}$ | 0.12$^{cy}$ | 0.13$^{cx}$ | 0.010 |
| | Wet | 0.14$^{b}$ | 0.12$^{bcy}$ | 0.11$^{cy}$ | 0.26$^{ax}$ | 0.10$^{cy}$ | 0.008 |
| | SEM[2] | 0.003 | 0.018 | 0.008 | 0.002 | 0.005 | |
| C16:0 (mg/g) | Dry | 1.90$^{ab}$ | 0.80$^{by}$ | 3.38$^{a}$ | 1.49$^{aby}$ | 1.55$^{abx}$ | 0.535 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Wet | 1.90$^{b}$ | 1.75$^{bx}$ | 1.76$^{b}$ | 3.10$^{ax}$ | 1.01$^{cy}$ | 0.061 |
| | SEM[2] | 0.064 | 0.062 | 0.844 | 0.051 | 0.044 | |
| C17:0 (mg/g) | Dry | 0.15$^{a}$ | 0.10$^{b}$ | 0.12$^{ab}$ | 0.13$^{aby}$ | 0.14$^{ax}$ | 0.009 |
| | Wet | 0.15$^{b}$ | 0.12$^{bc}$ | 0.11$^{c}$ | 0.30$^{ax}$ | 0.10$^{cy}$ | 0.009 |
| | SEM[2] | 0.006 | 0.014 | 0.013 | 0.003 | 0.005 | |
| C18:0 (mg/g) | Dry | 2.28$^{c}$ | 3.24$^{bx}$ | 4.29$^{ax}$ | 1.57$^{dy}$ | 1.56$^{dy}$ | 0.098 |
| | Wet | 2.28$^{c}$ | 2.01$^{cy}$ | 2.07$^{cy}$ | 2.90$^{bx}$ | 3.47$^{ax}$ | 0.110 |
| | SEM[2] | 0.059 | 0.041 | 0.153 | 0.040 | 0.155 | |
| SFA (mg/g) | Dry | 4.70$^{b}$ | 4.63$^{b}$ | 8.39$^{ax}$ | 3.53$^{by}$ | 3.62$^{by}$ | 0.610 |
| | Wet | 4.70$^{b}$ | 4.21$^{b}$ | 4.24$^{by}$ | 7.19$^{ax}$ | 4.85$^{bx}$ | 0.152 |
| | SEM[2] | 0.142 | 0.135 | 0.961 | 0.081 | 0.142 | |
| C14:1 (mg/g) | Dry | 0.04$^{bc}$ | 0.05$^{ab}$ | 0.05$^{ab}$ | 0.06$^{ay}$ | 0.06$^{ax}$ | 0.005 |
| | Wet | 0.04$^{bc}$ | 0.05$^{b}$ | 0.04$^{bc}$ | 0.18$^{ax}$ | 0.03$^{cy}$ | 0.004 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | SEM[2] | 0.003 | 0.005 | 0.007 | 0.003 | 0.000 | |
| C16:1 (mg/g) | Dry | 0.23[b] | 0.25[b] | 0.37[ax] | 0.34[ay] | 0.35[ax] | 0.015 |
| | Wet | 0.23[b] | 0.24[b] | 0.22[by] | 0.83[ax] | 0.18[by] | 0.016 |
| | SEM[2] | 0.015 | 0.020 | 0.021 | 0.008 | 0.011 | |
| C18:1n9c (mg/g) | Dry | 3.02[bc] | 2.33[c] | 4.21[ax] | 2.85[bcy] | 3.28[bx] | 0.178 |
| | Wet | 3.02[b] | 2.35[b] | 2.22[by] | 7.78[ax] | 2.38[by] | 0.192 |
| | SEM[2] | 0.195 | 0.202 | 0.261 | 0.073 | 0.137 | |
| MUFA (mg/g) | Dry | 3.29[bc] | 2.64[c] | 4.63[ax] | 3.26[bcy] | 3.68[bx] | 0.190 |
| | Wet | 3.29[b] | 2.64[b] | 2.48[by] | 8.80[ax] | 2.60[by] | 0.208 |
| | SEM[2] | 0.208 | 0.222 | 0.280 | 0.079 | 0.146 | |
| C18:2n6c (mg/g) | Dry | 0.74[b] | 0.36[dy] | 0.54[cy] | 0.93[ay] | 1.08[ax] | 0.053 |
| | Wet | 0.74[b] | 0.83[bx] | 0.84[bx] | 1.62[ax] | 0.51[cy] | 0.044 |
| | SEM[2] | 0.047 | 0.016 | 0.030 | 0.058 | 0.073 | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| C20:3n6 (mg/g) | Dry | 0.24[a] | 0.07[cy] | 0.11[by] | 0.10[by] | 0.10[by] | 0.007 |
| | Wet | 0.24[b] | 0.27[bx] | 0.29[bx] | 0.36[ax] | 0.15[cx] | 0.015 |
| | SEM[2] | 0.010 | 0.007 | 0.010 | 0.020 | 0.007 | |
| PUFA (mg/g) | Dry | 0.98[a] | 0.43[cy] | 0.65[by] | 1.03[ay] | 1.18[ax] | 0.057 |
| | Wet | 0.98[b] | 1.10[bx] | 1.12[bx] | 1.98[ax] | 0.67[cy] | 0.055 |
| | SEM[2] | 0.042 | 0.009 | 0.035 | 0.080 | 0.080 | |
| Total (mg/g) | Dry | 8.97[b] | 7.70[b] | 13.67[ax] | 7.82[by] | 8.49[b] | 0.739 |
| | Wet | 8.97[b] | 7.95[b] | 7.84[by] | 17.96[ax] | 8.10[b] | 0.327 |
| | SEM[2] | 0.376 | 0.363 | 1.132 | 0.181 | 0.210 | |

[1]Standard error of mean (n = 15), [2](n = 6).

[a–d]Different letters within same row differ significantly ($p < 0.05$).

[x,y]Different letters within same column differ significantly ($p < 0.05$).

nd, not detected.

As seen in Table 9, it was observed that most lipid oxidation-derived volatile compounds were positively correlated with UFAs in dry-aged beef. Especially, the correlation coefficients between aldehydes and UFAs had strong correlation when the beef was dry-aged, mainly influenced by the increase of propanal which was the most abundant compound in dry-aged beef (Table 3). However, furan and ketone in dry-aged beef were not significantly correlated with UFA contents, which might be explained by the fluctuation of 3-heptanone during dry aging process.

The increase of mold at the surface of dry-aged beef also had positive correlation with all volatile compound groups, indicating the importance of microbial activity for the formation/increase of aroma volatile compounds at the late phase of dry aging period. From the results, the difference of dry- and wet-aged beef flavor could result from the difference of aroma volatile profile which might be originated from lipid oxidation and microbial enzyme activity.

Table 9. Pearson correlation coefficients for volatile compounds in dry- and wet-aged beef with their mold distribution and UFAs

| | Mold distribution | | UFA | |
|---|---|---|---|---|
| | Dry | Wet | Dry | Wet |
| Alcohol | 0.84*** | - | 0.61* | ns |
| Aldehyde | 0.59* | - | 0.84*** | -0.71** |
| Furan and ketone | 0.78** | - | ns | ns |
| Hydrocarbon | 0.71** | - | 0.63* | ns |
| Acid | 0.63* | - | 0.83** | ns |
| Ester | 0.81** | - | 0.67** | ns |
| N-compound | 0.80** | - | ns | ns |
| S-compound | 0.87*** | - | 0.62* | ns |

Mold distribution was conducted for dry-aged beef only as no mold growth was shown in wet-aged beef.
*, $p < 0.05$; **, $p < 0.01$; ***, $p < 0.0001$.
ns, not significant.

## 2.4. Conclusion

Dry-aged beef showed significantly higher concentrations of volatile compounds, with more distinctive changes than those in wet-aged beef. This was mainly attributed to i) propanal known to be generated from the oxidation of lipids and ii) 2-methylbutanal, 2-methylpropanal, 1-butanamine, trimethylamine, 2-methylpropanthiol, and ethyl propanoate, possibly derived from the metabolism of microorganisms. Based on the results, the formation of aroma volatiles responsible for the unique flavor in dry-aged beef, clearly separated from wet-aged counterpart, may be induced by lipid oxidation and microbial activity.

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# Chapter III.

# Effects of cooking conditions on the physicochemical and sensory characteristics of dry- and wet-aged beef

## 3.1. Introduction

Dry aging is a method that exposes meat to controlled temperature, humidity, and air flow in the absence of packaging, in contrast to wet aging, which stores the meat vacuum-packaged (H. C. Kim et al., 2020). In recent days, the consumer demand for dry-aged beef has been increasing, mainly due to its unique flavor, which is characterized as intensely roasted, beefy, and nutty (Park et al., 2018). It has been reported that dry-aged beef has higher quantities of flavor precursors, such as free amino acids and nucleotide-related compounds (M. Kim et al., 2019; Lee et al., 2019b), and aromatic volatile compounds, such as aldehydes, compared to wet-aged beef (Ha et al., 2019).

On the other hand, meat flavor can be developed through the cooking process (Kerth et al., 2015), and cooked meat sensory characteristics can vary depending on the cooking method (e.g., grilling, roasting, boiling, etc.) and conditions, including heating temperature and rate (Angel-Rendon et al., 2020; Pathare et al., 2016). The influence of cooking methods on meat flavor has been observed in several studies to be mainly due to differences in the type of heat transfer (categorized as conduction, convection, and radiation) and the efficiency of heat treatment on meat (Domínguez et al., 2014; Echegaray et al., 2020; Peñaranda et al., 2017; Yoo et al., 2020). Cooking can also be classified by cooking temperatures: low-temperature cooking below 100°C, high-temperature cooking above 100°C, and very high temperature cooking above 200°C (Angel-Rendon et al., 2020). Cooking at high temperatures increases the heating rate and the degree of Maillard reaction, which enhances the roasted aroma of meat. On the other hand, extensive cooking can cause high oxidative reactions and the generation of undesirable polycyclic aromatic hydrocarbons (Mottier et al.2000; Pathare et al., 2016). However, little is known about the effect of cooking method and temperature on dry-aged beef. King et al. (1995) applied oven roasting and microwave cooking to dry-aged beef and observed higher hydrocarbons and lower terpenoids in oven-roasted beef than in microwave-cooked beef. Nonetheless, as the authors stated, the lack of sensory evaluation restricts the estimation of the effect of cooking method on dry-aged beef. For this reason, the effective cooking method and temperature for dry-aged beef remain unclear.

Among the cooking methods for steaks in households and restaurants, grilling at temperatures above 200°C and oven roasting in the range of 150-250°C are widely

used conduction and convection cooking methods, respectively (Mottier et al., 2000; Pathare et al., 2016; Yancey et al., 2011). Therefore, the objective of this study was to investigate the effects of different cooking methods (grilling and oven roasting) and temperatures (150°C and 230°C) on the physicochemical and sensory characteristics of dry-beef.

## 3.2. Materials and methods

### *3.2.1. Sample preparation*

#### *3.2.1.1. Raw material and aging process*

Beef strip loins (longissimus lumborum) from Holstein steers (21 months old, quality grade 3) (KAPE, 2019) were obtained at 48 h post-mortem. The visual fat and connective tissues were removed from the surface of beef strip loins, and each muscle was cut into an average weight of 500 g. Then, beef samples were randomly divided into two groups. One group was placed in a dry aging chamber (4°C, 75% relative humidity, and 2.5 m/s air flow velocity) and dry-aged for 28 days. The other group was vacuum packaged (HFV-600L, Hankook Fujee Machinery Co., Ltd., Hwaseong, Korea) in low-density polyethylene/nylon bags ($O_2$ permeability of 2 $mL/m^2/d$ at 0°C; 0.09 mm thickness; Sunkyung Co., Ltd., Seoul, Korea) for wet aging at 4°C, with the same aging duration. After the aging process, the dark and thickened crust of the dry-aged beef was trimmed off. Both dry- and wet-aged beef were stored in a vacuum packaged bag and frozen to −70°C for further analyses.

#### *3.2.1.2. Cooking process*

The beef strip loins were thawed at 4°C for 18 h and sliced into 3.5 cm thick samples (average weight of 100 g). Next, four different cooking conditions (cooking method × cooking temperature) were applied to the dry- (n = 3 for each cooking treatment) and wet-aged beef steaks (n = 3 for each cooking treatment): grilling at 150°C or 230°C, and oven roasting at 150°C or 230°C. Each cooking condition was

replicated three times. During cooking, the surface temperature of the electric grill (EG-GW1700, Kitchenart, Incheon, Korea) was measured using an infrared thermometer (ST-101, Sincon, Bucheon, Korea), and the internal temperature of the sample was monitored using a digital thermometer (TM-747DU, Tenmars Electronics Co., Ltd., Taipei, Taiwan). Steaks cooked by grilling were turned every two min, and those cooked in an electric oven (MA324DBN, LG Electronics, Seoul, Korea) were turned at 40°C internal temperature. All cooking processes continued until the core temperature of the steak reached 72°C. In every cooking condition, dry-aged beef was compared with wet-aged beef, to which the same cooking treatment was applied, to determine whether the changes in meat sensory and physicochemical properties derived from the aging methods or cooking conditions. Cooking time was recorded for each treatment.

### *3.2.2. pH*

One gram of meat sample with 9 mL of distilled water was homogenized at 9,600 rpm for 30 s using a homogenizer (T25 basic, IKA Works, Staufen, Germany). Then, the homogenates were centrifuged at 2265 × g for 10 min (Continent 512R, Hanil Co. Ltd., Daejeon, Korea), followed by filtration with filter paper (No. 4, Whatman International Ltd., Kent, UK). The pH values of dry- and wet-aged beef before and after cooking were measured using a pH meter (Seven2Go, Mettler-Toledo International Inc., Schwerzenbach, Switzerland). The pH meter was pre-calibrated with pH 4.01, pH 7.00, and pH 9.21 standardized buffer solutions at room temperature.

### *3.2.3. Cooking loss*

Cooking loss was expressed as the percentage of weight difference between raw and cooked beef relative to the weight of the raw sample.

$$\text{Cooking loss (\%)} = \frac{\text{Weight before cooking - Weight after cooking}}{\text{Weight before cooking}} \times 100$$

### *3.2.4. Lipid oxidation*

Lipid oxidation was determined by measuring TBARS values. Each sample (5 g) was homogenized at 9,600 rpm for 30 s using a homogenizer (T25 basic, IKA works), with the addition of 15 mL of distilled water and 50 μL of 7.2% butylated hydroxy toluene solution. After centrifugation at 2265 × g for 15 min (Continent 512R, Hanil Co. Ltd.), the supernatants were filtered using filter paper (No. 4, Whatman International Ltd.). Then, 2 mL of the filtrates was transferred to a test tube and mixed with 4 mL of 20 mM 2-thiobarbituric acid in 15% trichloroacetic acid. The mixture was heated in a water bath at 90°C for 30 min, cooled, and centrifuged at 2265 × g for 15 min (Continent 512R, Hanil Co. Ltd.). The supernatant absorbances were measured at 532 nm using a spectrophotometer (X-ma 3100, Human Co. Ltd., Seoul, Korea). TBARS values were expressed as mg malondialdehyde per kg of meat sample.

### *3.2.5. Volatile compounds analysis*

The analysis of volatile compounds in cooked beef was performed by solid-phase microextraction and gas chromatography-mass spectrometry (SPME-GC-MS). Five grams of cooked meat samples were placed into a 20-mL headspace vial and sealed with a PTFE-faced silicone septum. The samples were incubated at 40°C for 5 min, and then, a 65 μm thick polydimethylsiloxane/divinylbenzene fiber (Supelco Inc., Bellefonte, USA) was exposed to the headspace of the vial for 60 min. The volatile compounds were desorbed in the injection port of the GC (Trace 1310, Thermo Fisher Scientific, Waltham, USA) at 270°C in splitless mode. Helium was used as the carrier gas at a flow rate of 2 mL/min, and volatile compounds were separated using a fused silica capillary column (DB-Wax, 60 m × 0.25 mm i.d., and 0.50 μm film thickness; Agilent Technologies Inc., Santa Clara, U.S.A.). The GC oven was programmed as follows: initial temperature of 40°C, subsequently increased to 180°C at a rate of 5°C/min, then increased to 200°C at a rate of 2°C/min and held for 5 min, and then increased to a final temperature of 240°C at a speed of 10°C/min and held for 10 min. The column was directly coupled to a triple quadrupole mass spectrometer (TSQ 8000, Thermo Fisher Scientific) operating in the electron ionization mode at 70 eV and 250°C. Mass spectra were obtained with a scan ranging from 35 to 550 m/z at intervals of 0.2 s. The identification of volatile compounds was performed by comparing their mass spectra with those of the National Institute of Standards and Technology (NIST) mass spectral library.

### *3.2.6. Meat color*

The cooked meat was cut horizontally to measure its surface and internal color. Meat color was measured using a colorimeter (CM-5, Konica Minolta Censing Inc., Osaka, Japan), which was calibrated using a standard plate before measurements. The CIE $L^*$- (lightness), $a^*$- (redness), and $b^*$- (yellowness) values were determined in the condition of illuminant D65 and 10° standard observer with a 30 mm aperture size plate. A reflectance ratio of 630:580 nm was calculated to estimate the degree of doneness after different cooking treatments (Sawyer et al., 2008).

### *3.2.7. Descriptive sensory analysis*

The design of the descriptive sensory analysis for dry- and wet-aged beef was reviewed and approved by the Institutional Review Board (IRB) of Seoul National University (SNU) (IRB No. 1810/003-001). Immediately after cooking, the samples were cut to 1 cm in thickness, wrapped in aluminum foil and plastic wrap to preserve the aroma and prevent moisture evaporation, and kept in a drying oven (BF-80N, BioFree, Seoul, Korea) at 60°C. The holding time of the cooked samples in the drying oven was less than 20 min. Ten trained panelists (6 males and 4 females aged 26–33 years) were recruited from SNU researcher and faculty populations, and the panelists participated in the descriptive sensory analysis. Before the analysis, panelists were trained over several sessions for the descriptive sensory analysis of dry- and wet-aged beef and practiced rating the score of each sensory attribute. All training sessions and descriptive sensory analyses were conducted at SNU. The sensory properties were evaluated using a nine-point hedonic scale, in which the flavor scores ranged from one to nine (extremely weak to extremely strong), the

score of surface color ranged from one to nine (extremely bright to extremely dark), the internal color score ranged from one to nine (extremely white to extremely red), the tenderness score ranged from one to nine (extremely tender to extremely tough) after 15 bites, and the score of juiciness ranged from one to nine (extremely dry to extremely juicy) after 15 bites. Drinking water was provided to the panelists to cleanse their palates between sample evaluations.

Table 10. Sensory attributes and descriptions of dry- and wet-aged beef strip loin for descriptive sensory analysis

| Sensory attribute | | Description |
|---|---|---|
| Color | Surface | The darkness of surface color. |
| | Internal | The *a** of internal color. |
| Flavor | Roasted[a] | A flavor of roasted nuts and coffee beans. |
| | Dry-aged | A flavor of dairy products found in dry-aged beef. |
| | Cheesy[b] | A flavor of cheese. |
| | Fatty[a] | A flavor of cooked animal fat. |
| | Savory[b] | A flavor of glutamate, salts of amino acids and nucleotides. |
| Texture | Tenderness[c] | How easy the meat is divided during chewing. |
| | Juiciness[c] | The amount of juice released after 15 chews. |

[a]Sensory descriptors taken from Myrdal Miller et al. (2014), [b]Adhikari et al. (2011), and [c] Christensen et al. (2012).

### *3.2.8. Statistical analysis*

All experiments were conducted in triplicate, except the descriptive sensory analysis, where a randomized block design was applied using the trial and panelist as the block (n = 10 per trial, 20 per 2 trials). Statistical analysis was performed using the general linear model (SAS 9.4, SAS Institute Inc., Cary, NC, USA), which included the aging method, cooking condition, and their interactions as fixed effects and carcass and carcass side as random effects. For the evaluation of descriptive sensory analysis data, the trial and panelist were also included as random factors. The results were reported as mean values with standard error of the mean, and significant differences among the mean values were determined by the Tukey's multiple comparison test at a significance level of 0.05. In order to identify the difference in the composition of volatile compounds between treatments and classify them, PCA, PLS-DA, and VIP scores for the PLS-DA model were performed with the contents of volatile compounds using MetaboAnalyst 4.0 (www.metaboanalyst.ca) according to H. C. Kim et al. (2020), and the samples were log-transformed and auto-scaled before conducting multivariate analyses. Pearson correlation coefficients and linear mixed model (Starkey et al., 2017) between sensory properties and overall acceptability of dry- and wet-aged beef strip loins were analyzed using SAS 9.4 (SAS Institute Inc.). In the mixed model, random terms included the trial and panelist. The model is as follows:

Overall acceptability = Surface color + Internal color + Roasted flavor + Dry-aged flavor + Cheesy flavor + Fatty flavor + Savory flavor + Tenderness + Juiciness + *trial* + *panelist*

## 3.3. Results and discussion

### *3.3.1. Cooking time*

There were significant differences between the cooking times of dry- and wet-aged beef when they were cooked at 230°C regardless of cooking methods (Table 11). One possible reason might be the difference in the moisture content of dry- and wet-aged beef before cooking, although moisture was not measured in this study. Many studies reported that dry-aged beef possessed significantly lower moisture compared to wet-aged beef after 21 or 28 days of aging owing to the phenomenon of moisture evaporation during dry aging process (M. Kim et al, 2019; Lee et al., 2019b). It was observed that higher level of initial moisture content was correlated with prolonged cooking time, which might be explained as increased heat loss for evaporating moisture (Zeidler et al., 1989). In case of cooking method, grilling required lesser cooking time than oven roasting at the same cooking temperature condition except the result of dry-aged beef at 230°C ($p < 0.05$). Cooking time is greatly influenced by the efficiency of heat transfer (Yancey et al., 2011). Grilling and oven roasting have different mode of heat transfer (conduction and convection, respectively), and conduction cooking can decrease the cooking time through direct heat transfer from the heating surface to the sample (Fabre et al., 2018). The result indicates that grilling was more effective in increasing the core temperature of meat compared to oven roasting. Similarly, Kerth et al. (2003) and Yancey et al. (2011) reported that cooking rate of steaks was faster in conduction method compared to convection cooking method. Finally, cooking was generally done significantly faster

when the beef was cooked at 230°C, since greater temperature gradient would be created when cooking temperature increased. Cooking time is considered an important factor in cooking meat products as it can influence the physical and sensory properties of cooked meat profoundly (Pathare et al., 2016).

Table 11. Time (sec) consumed for cooking dry- and wet-aged beef at different cooking conditions

| Aging method | Grilled | | Oven-roasted | | SEM[1] |
|---|---|---|---|---|---|
| | 150°C | 230°C | 150°C | 230°C | |
| Dry | 853.00$^{b}$ | 654.67$^{by}$ | 1489.33$^{a}$ | 777.33$^{by}$ | 67.311 |
| Wet | 911.00$^{b}$ | 759.00$^{cx}$ | 1811.33$^{a}$ | 988.33$^{bx}$ | 42.928 |
| SEM[2] | 34.759 | 25.869 | 96.371 | 39.780 | |

[1]Standard error of mean (n = 12), [2](n = 6).

$^{a-c}$Different letters within the same row differ significantly ($p < 0.05$).

$^{x,y}$Different letters within the same column differ significantly ($p < 0.05$).

*3.3.2. pH*

The pH values of dry- and wet-aged beef differed before cooking (5.57 and 5.27, respectively; $p < 0.05$). Similarly, it was reported that dry-aged beef showed significantly higher pH compared to wet-aged beef after 21 or 40 days of aging (Dikeman et al., 2014; J. H. Kim et al., 2019). The pH difference between dry- and wet-aged beef could be due to their different microbiological compositions (Terjung, Witte, & Heinz, 2021). Various environmental factors such as temperature, relative humidity, air flow velocity, and the presence of oxygen affect the growth of microorganisms (Lee et al., 2019b). Higher degrees of total aerobic bacteria and mold and yeast counts were found in dry-aged beef, while lactic acid bacteria was more dominant in wet-aged beef after 14 and 21 days of aging (Gudjónsdóttir et al., 2015). Notably, the proteolytic and lipolytic effect of mold and yeast on dry-aged beef was suggested in previous studies (Choe et al., 2020; Lee et al., 2019b). As a result, the formation of ammonia, amines, and basic amino acids by proteolysis might lead to the increase of the pH of dry-aged beef. On the other hand, the accumulation of lactic acid by the increase of lactic acid bacteria in wet-aged beef could decrease its pH (J. H. Kim et al., 2019). Dry-aged beef also had a higher pH than wet-aged beef after cooking ($p < 0.05$; Table 12). According to Kerth et al. (2015), an increase in pH can lead to an increase in the water holding capacity, which affects heat transfer efficiency and ultimately results in the flavor of meat. They stated that the increase in beef surface temperature with low water holding capacity during cooking is disturbed by the evaporation of free water on the surface, which leads to the formation of fewer Maillard reaction products that are associated with

meaty and roasted aroma. Furthermore, it was reported that the formation of specific Maillard reaction products such as pyrazines was favored as the pH of meat increased between 4.5 and 6.5 (Jayasena et al., 2013). Madruga et al. (1995) measured volatile compounds in cooked meat at pH values ranging from 4.0 to 5.6, and observed a decrease in 2-methyl-3-furanyl group compounds and sulfur compounds and the increase of pyrazines as the pH of the meat increased. In the present study, we also detected a significant increase in pyrazine compounds in dry-aged beef compared to wet-aged beef and discussed the results in section 3.3.5. However, the effects of cooking method and cooking temperature on pH were not observed in this study.

Table 12. Effect of different cooking conditions on pH of dry- and wet-aged beef strip loins

| Aging method | Grilled | | Oven-roasted | | SEM[1] |
|---|---|---|---|---|---|
| | 150°C | 230°C | 150°C | 230°C | |
| Dry | 5.83[x] | 5.84[x] | 5.82[x] | 5.82[x] | 0.011 |
| Wet | 5.42[y] | 5.43[y] | 5.45[y] | 5.43[y] | 0.012 |
| SEM[2] | 0.008 | 0.010 | 0.016 | 0.008 | |

[1]Standard error of mean (n = 12), [2](n = 6).
[x,y]Different letters within the same column differ significantly ($p < 0.05$).

### *3.3.3. Cooking loss*

Cooking loss involves the loss of liquid and soluble matter during cooking procedure, thus it relies on the ability of meat to retain water and fat (Domínguez et al., 2014; Turp, 2016). High cooking loss may decrease the tenderness or juiciness of meat product (Angel-Rendon et al., 2020). The loss of moisture during cooking could be explained as three reasons: moisture evaporation by increased temperature, heat-induced denaturation of myofibrillar proteins which reduces the water holding capacity of meat, and contraction of perimysial connective tissue that makes the muscle fiber compressed (del Pulgar et al., 2012). Dry-aged beef grilled at 230°C had significantly lower cooking loss compared to wet-aged beef cooked at the same condition (Table 13). The effect of aging method on cooking loss was found to be significant ($p = 0.02$). It might be derived from the difference in initial moisture level between in dry- and wet-aged beef or the difference in pH before and after cooking, since water holding capacity decreases as pH decreases to the isoelectric point of myofibrillar protein (Angel-Rendon et al., 2020). In wet-aged beef, the cooking loss of meat cooked by oven roasting at 230°C was significantly lower than that cooked by grilling at the same cooking temperature. Turp (2016) stated that beef meatball cooked by oven roasting until the core temperature reached 75°C showed higher cooking yield, moisture and fat retention that grilled meatball. However, Echegaray et al. (2020) found that grilled pork steak at 130-150°C for 10 min had lower cooking loss compared to oven-roasted pork at 200°C for the same time. The difference in cooking loss between different cooking method may be linked with the rate of increase of meat surface temperature or the formation of crust which could help to

retain the water during cooking (Kerth et al., 2003). The increase of cooking temperature did not show the difference of cooking loss. It was in accordance with the result of study conducted by Wall et al. (2019) which showed that cooking yield was not significantly different by grilling cooking temperature.

Table 13. Effect of different cooking conditions on cooking loss (%) of dry- and wet-aged beef

| Aging method | Grilled | | Oven-roasted | | SEM[1] |
|---|---|---|---|---|---|
| | 150°C | 230°C | 150°C | 230°C | |
| Dry | 19.68 | 19.58$^{y}$ | 17.90 | 18.12 | 1.060 |
| Wet | 20.14$^{ab}$ | 22.97$^{ax}$ | 20.21$^{ab}$ | 18.94$^{b}$ | 0.850 |
| SEM[2] | 0.863 | 0.744 | 0.821 | 1.311 | |

[1]Standard error of mean (n = 12), [2](n = 6).
$^{a,b}$Different letters within the same row differ significantly ($p < 0.05$).
$^{x,y}$Different letters within the same column differ significantly ($p < 0.05$).

### *3.3.4. Lipid oxidation*

During the cooking process, lipid oxidation has a huge role in the generation of desirable and characteristic flavor compounds in meat, although excessive lipid oxidation leads to the deterioration of meat quality, such as undesirable off-flavor and texture changes (Broncano et al., 2009; Echegaray et al., 2020).

The TBARS value of dry-aged beef was significantly lower than that of wet-aged beef, regardless of cooking conditions (Table 14). This finding was inconsistent with the results from the study conducted by Kahraman et al. (2019), who found that dry-aged beef possessed higher TBARS than wet-aged beef after 14 days of aging. Similarly, Ribeiro et al. (2021) reported that the TBARS value of beef loin was significantly higher after dry aging for 42 days compared to that after wet aging for the same duration. The increase of lipid oxidation in dry-aged beef may be related with air exposure during aging process, while lipid oxidation is prohibited in vacuum-packed wet-aged beef (Ribeiro et al., 2021). In the present study, the lower TBARS value of cooked dry-aged beef might be related to an increase in antioxidant compounds in dry-aged meat, as reported in previous studies (Choe et al., 2020; H. C. Kim et al., 2020; Park et al., 2018). H. C. Kim et al. (2020) found that after 28 days of aging, the concentrations of anserine and carnosine compounds with strong antioxidant activities, were significantly higher in dry-aged beef than in wet-aged beef. Lee et al. (2019b) also reported higher amounts of amino acids, including phenylalanine, tryptophan, and tyrosine, in dry-aged beef compared to wet-aged beef, which could result from concentration effects due to moisture evaporation and

microbial proteolysis during the dry aging process. Moreover, Park et al. (2018) observed that in dry-aged beef patties made with 5% crust, the surface of dry-aged beef, which is usually trimmed off, showed lower TBARS values compared to those made without crust, and suggested potential antioxidant activity in the crust. The antioxidant activities of dry- and wet-aged beef and crust from dry-aged beef were compared by Choe et al. (2020) through radical scavenging activities, ferric ion reducing capacity, and metal chelating activity tests. In this study, the investigators found that dry-aged beef possessed higher antioxidant activity than wet-aged beef, and crust showed the highest antioxidant activity. This antioxidant activity in dry-aged beef, especially in the crust, might be attributed to the increase of small peptides (< 3 kDa) through the action of microbial enzymes in the crust (Choe et al., 2020).

Meanwhile, the TBARS value of oven-roasted beef strip loin was significantly higher than that of grilled beef at both cooking temperatures ($p < 0.05$). Moreover, steaks cooked at lower temperatures showed higher TBARS values than those cooked at higher temperatures, even though there was no significant difference between grilled dry-aged beef cooked at different temperatures. It was reported that the TBARS value could be affected by the cooking temperature and cooking time (Broncano et al., 2009; Domínguez et al., 2014). Broncano et al. (2009) found that the TBARS value of Iberian pork roasted at 150°C for 20 min was significantly higher than that of pork grilled at 190°C for 4 min. Domínguez et al. (2014) also observed that roasting of foal meat at 200°C for 12 min produced more oxidation compared to grilling at 130-150°C for 5 min on each surface. In this study, we observed that grilling required less cooking time compared to oven roasting (13 min

14 s and 21 min 7 s, respectively; $p < 0.05$). The correlation between the TBARS value and cooking time was found to be significant ($r^2 = 0.64$; $p < 0.01$).

Accordingly, the lower TBARS values in dry-aged beef compared to those in wet-aged beef in the present study might result from the increase of bioactive peptides and antioxidants by the action of mold and yeast as described above. Furthermore, prolonged cooking process would accelerate the lipid oxidation, which might negatively affect the sensory quality of beef. Nonetheless, as the TBARS value of raw dry- and wet-aged beef were not measured in this study, the definite interpretation of the present result is not easy. Further studies are required for the estimation of antioxidant activity in cooked dry-aged beef at various cooking conditions.

Table 14. Effect of different cooking conditions on lipid oxidation (mg MDA/kg meat) of dry- and wet-aged beef strip loins

| Aging method | Grilled | | Oven-roasted | | SEM[1] |
|---|---|---|---|---|---|
| | 150°C | 230°C | 150°C | 230°C | |
| Dry | 1.44$^{cy}$ | 1.55$^{cy}$ | 2.36$^{ay}$ | 1.91$^{by}$ | 0.076 |
| Wet | 2.98$^{cx}$ | 2.43$^{dx}$ | 4.59$^{ax}$ | 4.06$^{bx}$ | 0.149 |
| SEM[2] | 0.157 | 0.137 | 0.075 | 0.083 | |

[1]Standard error of mean (n = 12), [2](n = 6).

$^{a-d}$Different letters within the same row differ significantly ($p < 0.05$).

$^{x,y}$Different letters within the same column differ significantly ($p < 0.05$).

### *3.3.5. Volatile compounds analysis*

A total of 60 volatile compounds including 15 alcohols, 10 aldehydes, 15 aliphatic hydrocarbons, 12 aromatic hydrocarbons, 6 ketones, and 2 unclassified compounds were identified in the headspace of cooked dry- and wet-aged beef. PCA showed that the volatile profiles of dry- and wet-aged beef differed, except for those of oven-roasted samples at 150°C (Fig. 3).

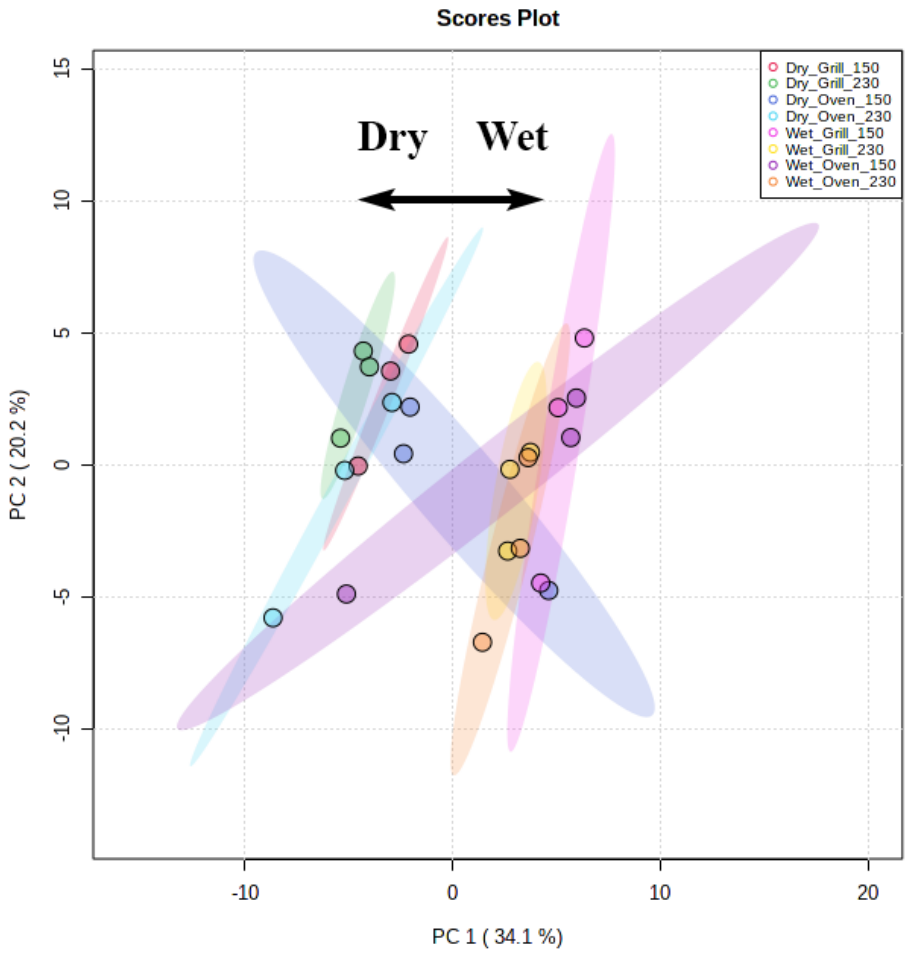


Figure 3. PCA of dry- and wet-aged beef strip loins cooked at different cooking conditions.

Similarly, PLS-DA plot distinguished dry- and wet-aged beef, and we found that dry-aged beef had higher 2-heptanol, isoamyl alcohol, 3-octanone, 2-heptanone, and benzaldehyde concentrations, whereas wet-aged beef had more abundant benzyl alcohol, 1,2-dimethylbenzene, 2,2,6-trimethyloctane, 2,5-octanedione, and 2,3-butanediol species (Fig. 4). The aforementioned compounds were regarded as the most characteristic variables for the separation of the two groups, and the alcohol, aldehyde, aliphatic hydrocarbon, and ketone variables represent lipid oxidation-derived products (Domínguez et al., 2014). In particular, aldehydes and ketones contribute highly to cooked meat flavor because they have low odor threshold values (Echgaray et al., 2020; Ha et al., 2019). The difference in the concentration of lipid oxidation-derived products might result from the different susceptibilities of dry- and wet-aged beef to lipid oxidation (Table 14). J. H. Kim et al. (2019) reported differences in the composition of free fatty acids and free amino acids between dry- and wet-aged beef, and Lee et al. (2019b) observed that dry aging for 28 days was more effective in increasing free amino acids and reducing sugars compared to wet aging for the same duration. It seems that different flavor precursors might influence the volatile formation of cooked dry- and wet-aged beef.

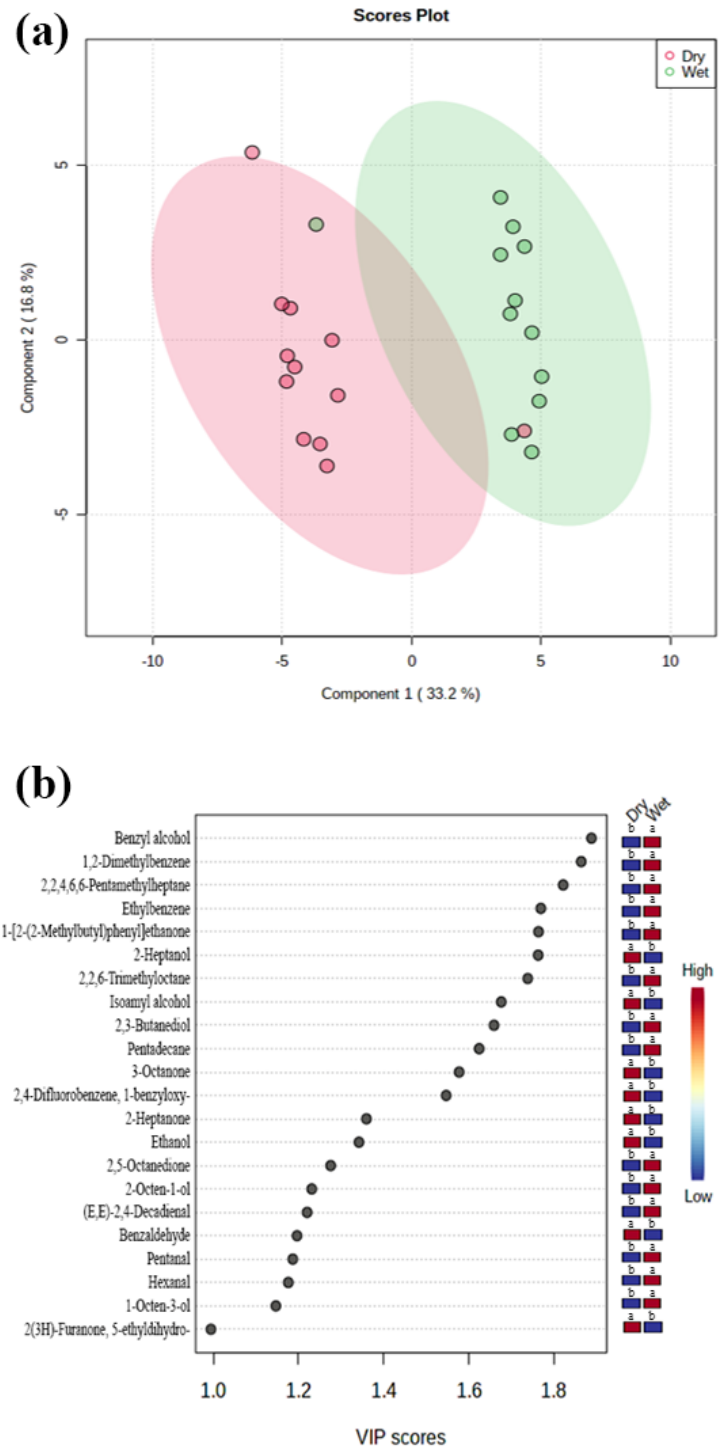


Figure 4. PLS-DA (a) and its VIP scores (b) from dry- and wet-aged beef.

The effect of cooking method on the formation of volatile compounds was detected using the PLS-DA model (Fig. 5). Most volatile compounds with VIP scores higher than one were more abundant in grilled beef than in oven-roasted samples. This observation could be related to the surface temperature of the samples and the efficacy of heat transfer depending on the cooking method. Peñaranda et al. (2017) reported that the intensity of meat odor was higher in grill-cooked pork compared to oven-roasted pork, possibly because of the higher surface temperature. Silva et al. (2016) found significantly higher amounts of Maillard reaction products in grilled and fried jerky chicken than in oven-roasted and sous-vide cooked chicken, and stated that conduction cooking was more effective in heat transfer than convection cooking.

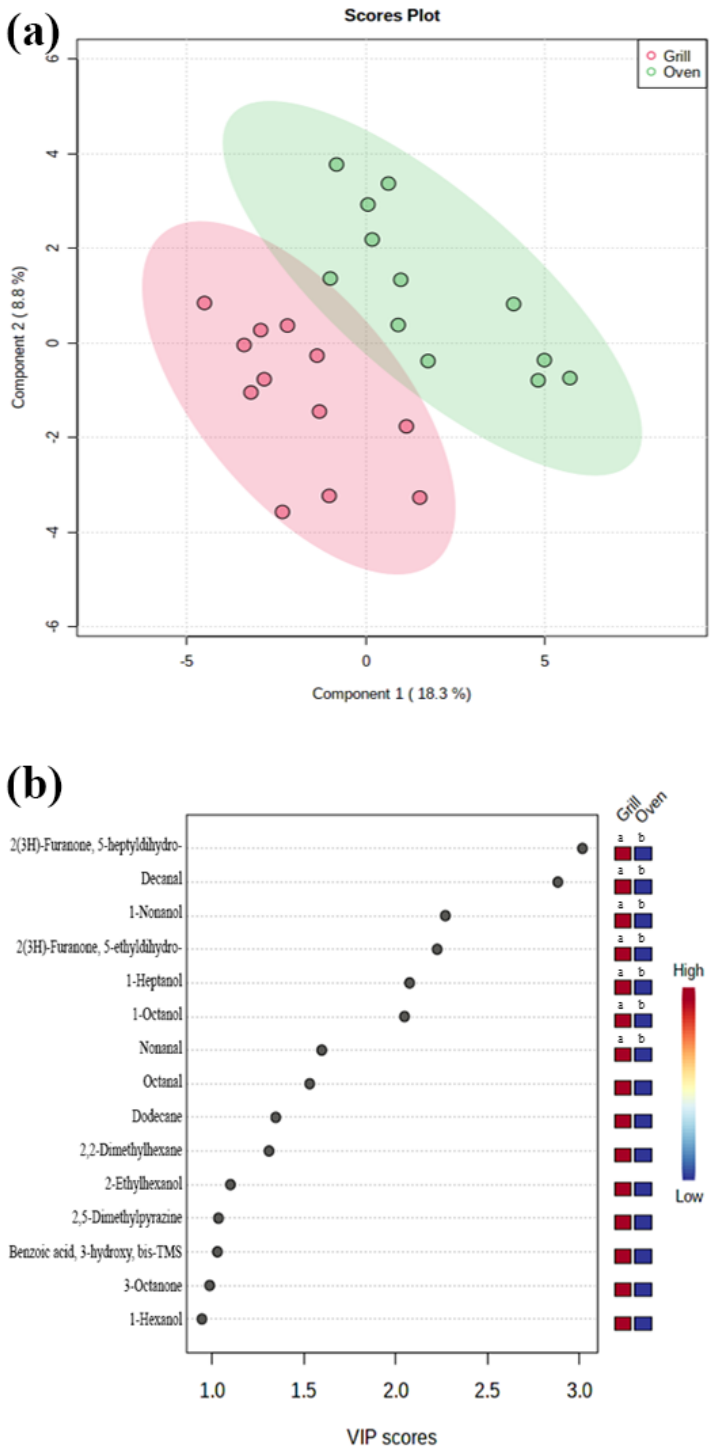


Figure 5. PLS-DA (a) and its VIP scores (b) from grill- and oven-cooked beef.

Finally, noticeable changes were found in five pyrazines (2-ethyl-3,5-dimethylpyrazine, 2,3-, 2,5-, and 2,6-dimethylpyrazine, and methylpyrazine) as cooking temperature increased (Fig. 6). This observation was in accordance with the study conducted by Wall et al. (2019), where the production of pyrazines in beef steak increased with increasing grill surface temperature from 177°C to 232°C. Pyrazines are mainly derived from the Maillard reaction, which requires high temperatures above 110°C in meat (Watanabe et al., 2015; Whitfield et al., 1992), and the formation of these compounds increases at elevated surface temperatures (Silva et al., 2016). Yoo et al. (2020) reported that the searing of beef steaks at 250°C increased meaty and roasted aromas compared to oven-cooking 180°C, due to the increased occurrence of the Maillard reaction. Furthermore, 2,3- and 2,5-dimethylpyrazine and methylpyrazine were present in significantly higher quantities in dry-aged beef when cooked by grilling compared to wet-aged beef (data not shown). Ha et al. (2019) also observed higher abundances of pyrazine, methylpyrazine, 2,5-dimethylpyrazine, 2-ethylpyrazine, and 2,5-dimethyl-3-ethylpyrazine in dry-aged beef compared to wet-aged beef. Pyrazines have meaty, nutty, and roasted aroma flavors. The higher concentration of pyrazine compounds in dry-aged beef may contribute to the development of characteristic dry-aged flavor (Echegaray et al., 2020; Watanabe et al., 2015).

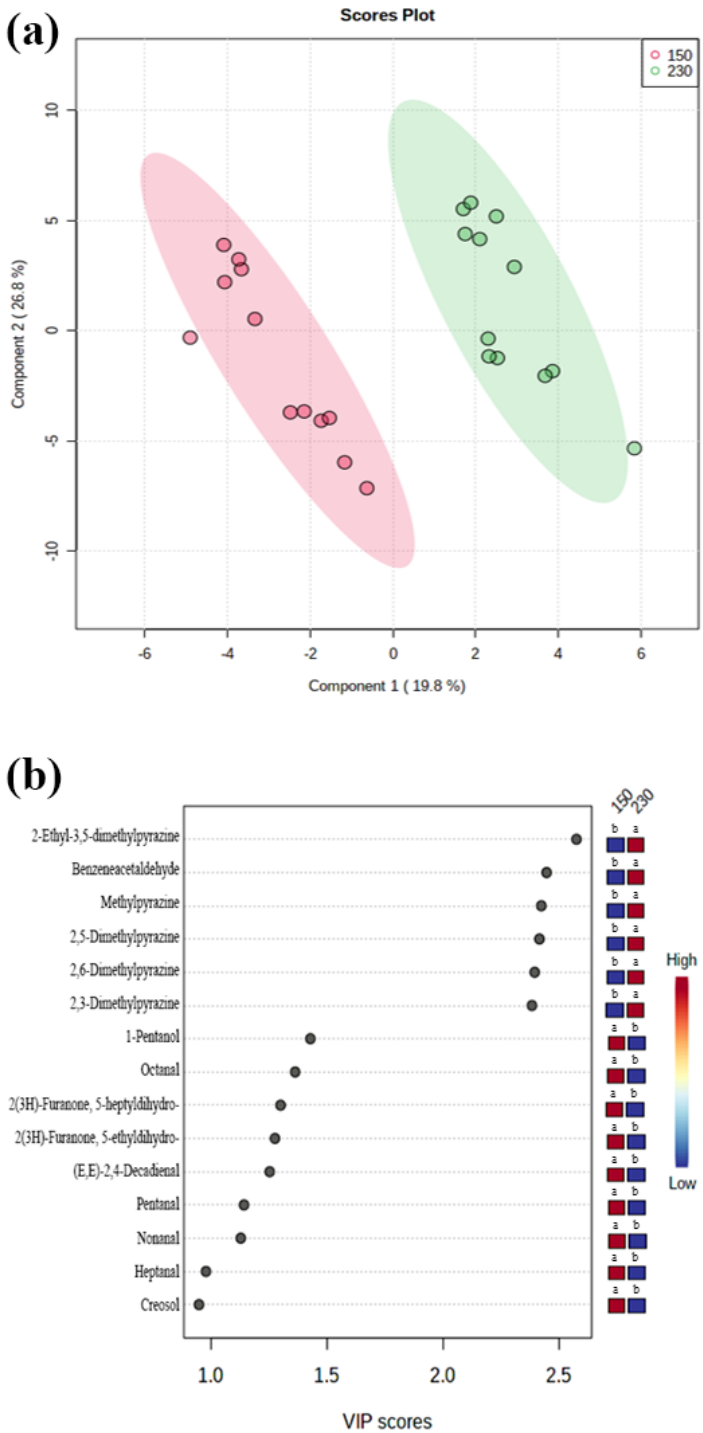


Figure 6. PLS-DA (a) and its VIP scores (b) from beef cooked at 150°C and 230°C.

### *3.3.6. Meat color*

The color of cooked meat is attributed to the heat-induced denaturation of myoglobin, which results in a brown appearance (Suman et al., 2016). Meat color can be influenced by various factors, such as pH, cooking conditions, the chemical state of myoglobin, and other variables (Sawyer et al., 2008). In order to analyze the effect of cooking conditions on cooked meat color in depth, both the surface and internal meat colors were measured independently. As a result, the beef surface color was generally affected by cooking conditions rather than by the aging method (Table 15). Noticeably, we found that $L^*$-, $a^*$-, and $b^*$- values were significantly lower at the surface of grill-cooked steaks at 230°C compared to other treatment combinations, regardless of the aging method. In both dry- and wet-aged beef, oven roasting led to a brighter surface color compared to grill cooking at the same cooking temperature ($p < 0.05$). Moreover, the $b^*$-value was significantly higher at the surface of oven-roasted beef compared to that of grilled beef when cooked at 230°C. In the case of cooking temperature, lower temperature cooking generally led to higher $L^*$-, $a^*$-, and $b^*$-values of the beef surface compared to higher temperature cooking. Lower $L^*$-values due to grilling or higher-temperature cooking might be related to moisture loss and surface drying due to a higher meat surface temperature (Ribeiro et al., 2021). The decrease in redness that was observed as cooking temperature increased from 150°C to 230°C indicates that myoglobin denaturation occurred to a higher degree at higher cooking temperatures (del Pulgar et al., 2012). Regarding $b^*$, Mitacek et al. (2017) stated that the increase in metmyoglobin might be related to a decrease in $b^*$. Consequently, grill-cooked dry- and wet-aged beef showed a higher

degree of doneness than oven-cooked dry-aged beef at 230°C and oven-cooked wet-aged beef at 150°C, respectively.

On the other hand, all internal beef steak color parameters showed significant differences depending on the aging method. In general, $L^*$-, $a^*$-, and $b^*$-values were higher and the degree of doneness was lower in wet-aged beef than in dry-aged beef. It has been reported that the surface of dry-aged beef had lower $L^*$ and $a^*$-values than that of wet-aged beef (Dikeman et al., 2013; Ribeiro et al., 2021); however, the internal color of cooked dry- and wet-aged beef has been rarely compared. The differences in beef color stability might be attributed to lipid oxidation, microbial growth, reducing ability, oxygen consumption rate, or the composition of three forms of myoglobin (Colle et al., 2016; Ribeiro et al., 2021; Suman et al., 2016). In the case of cooking conditions, oven roasting at 230°C showed significantly higher internal meat color $L^*$- and $b^*$-values compared to grilling at the same cooking temperature. While the internal color of grill-cooked dry-aged beef at 230°C showed overall low $a^*$- and $b^*$-values and a high degree of doneness, grilled dry-aged beef at 150°C had the highest $a^*$- and $b^*$-values and the lowest degree of doneness among the four cooking conditions. Yancey et al. (2011) observed the effect of cooking method on the internal cooked color of meat and reported that the conduction cooking method denatured myoglobin to a greater extent, resulting in a less red appearance compared to oven cooking. In accordance with the surface color results, the high abundance of pyrazine compounds in grilled dry-aged beef at 230°C could be explained by the high degree of doneness estimated by the surface and internal color measurements.

Meat color can provide information about the eating quality of meat to consumers (Pathare et al., 2016). For example, browned surface color can be utilized as an indicator of the Maillard reaction and caramelization, and the internal cooked color can indicate the doneness of meat (Suman et al., 2016; Yoo et al., 2020). The degree of doneness was further evaluated through a descriptive sensory analysis, as discussed in section 3.3.7.

Table 15. Effect of different cooking conditions on the color of dry- and wet-aged beef strip loins

| Trait | Aging method | Grilled | | Oven-roasted | | SEM[1] |
|---|---|---|---|---|---|---|
| | | 150°C | 230°C | 150°C | 230°C | |
| | | | *Surface* | | | |
| $L^*$ | Dry | 37.19$^{bx}$ | 29.94$^{c}$ | 39.97$^{ax}$ | 38.42$^{by}$ | 0.314 |
| | Wet | 34.03$^{cy}$ | 30.63$^{d}$ | 38.97$^{by}$ | 42.02$^{ax}$ | 0.447 |
| | SEM[2] | 0.370 | 0.616 | 0.239 | 0.149 | |
| $a^*$ | Dry | 6.28$^{a}$ | 5.18$^{b}$ | 6.31$^{ay}$ | 6.76$^{a}$ | 0.157 |
| | Wet | 6.55$^{b}$ | 6.03$^{b}$ | 7.42$^{ax}$ | 6.50$^{b}$ | 0.170 |
| | SEM[2] | 0.129 | 0.276 | 0.086 | 0.084 | |
| $b^*$ | Dry | 10.22$^{a}$ | 3.93$^{by}$ | 10.40$^{ax}$ | 11.00$^{ay}$ | 0.391 |
| | Wet | 8.69$^{b}$ | 5.97$^{cx}$ | 9.34$^{by}$ | 12.64$^{ax}$ | 0.165 |
| | SEM[2] | 0.493 | 0.290 | 0.125 | 0.133 | |
| 630:580[3)] | Dry | 1.28$^{b}$ | 1.27$^{b}$ | 1.31$^{aby}$ | 1.34$^{a}$ | 0.011 |
| | Wet | 1.29$^{b}$ | 1.29$^{b}$ | 1.45$^{ax}$ | 1.29$^{b}$ | 0.011 |
| | SEM[2] | 0.006 | 0.014 | 0.007 | 0.014 | |
| | | | *Internal* | | | |
| $L^*$ | Dry | 49.05$^{by}$ | 51.64$^{a}$ | 52.07$^{ay}$ | 50.28$^{aby}$ | 0.423 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | Wet | 51.94$^{cx}$ | 52.88$^{b}$ | 55.06$^{ax}$ | 54.71$^{ax}$ | 0.186 |
| | SEM[2] | 0.150 | 0.425 | 0.207 | 0.425 | |
| $a^*$ | Dry | 12.35$^{a}$ | 6.06$^{by}$ | 6.83$^{by}$ | 6.97$^{by}$ | 0.476 |
| | Wet | 13.39$^{a}$ | 9.21$^{bx}$ | 12.78$^{ax}$ | 13.24$^{ax}$ | 0.482 |
| | SEM[2] | 0.653 | 0.553 | 0.320 | 0.290 | |
| $b^*$ | Dry | 16.16$^{ay}$ | 13.28$^{cy}$ | 12.81$^{dy}$ | 14.00$^{by}$ | 0.091 |
| | Wet | 17.09$^{ax}$ | 15.91$^{bx}$ | 17.38$^{ax}$ | 17.60$^{ax}$ | 0.191 |
| | SEM[2] | 0.129 | 0.173 | 0.148 | 0.144 | |
| 630:580 | Dry | 2.57$^{a}$ | 1.43$^{by}$ | 1.53$^{by}$ | 1.50$^{by}$ | 0.101 |
| | Wet | 2.74$^{a}$ | 1.90$^{bx}$ | 2.59$^{ax}$ | 2.66$^{ax}$ | 0.087 |
| | SEM[2] | 0.141 | 0.096 | 0.051 | 0.062 | |

[1]Standard error of mean (n = 12), [2](n = 6).

[3]the ratio of reflectance of light at 630 nm and 580 nm which indicates the degree of doneness.

[a-d]Different letters within the same row differ significantly ($p < 0.05$).

[x,y]Different letters within the same column differ significantly ($p < 0.05$).

### *3.3.7. Descriptive sensory analysis*

Grill-cooked steak had a darker surface color compared to oven roasted steak, except for wet-aged beef cooked at 150°C (Table 16). However, there was no difference in internal redness between grilled and oven-roasted beef steaks cooked at the same temperature. Considering the results from the instrumental color measurements (Table 15) and descriptive sensory analysis, the aged beef color was more likely to be affected by cooking conditions than aging method, and grilling at higher temperatures was more desirable for the cooked meat color.

In the dry-aged beef, grill-cooked steaks showed no difference in color, flavor, tenderness, or juiciness scores (Table 16). Grilling had a significantly higher score for roasted flavor in dry- and wet-aged beef at both cooking temperatures compared to oven roasting. This result supports the results of the volatile compound analysis and meat color, where grilling was more effective for producing desirable flavor compounds and increasing cooking doneness (efficiency of heat transfer). In case of tenderness, no effect of cooking condition was found within dry-aged beef, while in wet-aged beef grilling at 150°C led to significantly lower shear force. Wall et al. (2019) observed no difference in the shear force of grill-cooked beef by surface temperature of grill (177°C, 205°C, and 232°C), whereas Yancey et al. (2011) found that conduction cooking resulted in a higher shear force than convection cooking. The juiciness score was higher in grill-cooked dry-aged beef at the cooking temperature of 230°C compared to oven-roasted dry-aged beef. The juiciness score was higher in grill-cooked dry-aged beef at the cooking temperature of 230°C compared to oven-roasted dry-aged beef. In general, the juiciness is influenced by

the amounts of moisture in meat after cooking (Pathare & Roskilly, 2016; Peñaranda et al., 2017). In the result of present study, the surface of grilled dry-aged beef showed higher degree of doneness compared to that of oven-roasted one cooked at 230°C. During grilling or roasting, crust can be formed at the dried surface of meat (Yoo et al., 2020) which might reduce the moisture permeability (Rao, Wang, Meng, Suleman, & Zhang, 2020). If higher heat transfer efficiency of grilling is considered than oven roasting in this study, crust of beef may potentially help increase the juiciness.

Table 16. Descriptive sensory analysis on dry- and wet-aged beef strip loins cooked at different cooking conditions

| Trait | Aging method | Grilled | | Oven-roasted | | SEM[1] |
|---|---|---|---|---|---|---|
| | | 150°C | 230°C | 150°C | 230°C | |
| | | *Color* | | | | |
| Surface | Dry | 6.85$^{ax}$ | 7.25$^{a}$ | 5.75$^{b}$ | 5.20$^{b}$ | 0.215 |
| | Wet | 5.75$^{by}$ | 6.85$^{a}$ | 5.55$^{b}$ | 5.40$^{b}$ | 0.217 |
| | SEM[2] | 0.286 | 0.192 | 0.213 | 0.151 | |
| Internal | Dry | 4.85$^{ab}$ | 4.85$^{ab}$ | 5.05$^{a}$ | 4.30$^{b}$ | 0.319 |
| | Wet | 5.30 | 4.90 | 4.40 | 4.65 | 0.407 |
| | SEM[2] | 0.502 | 0.348 | 0.312 | 0.253 | |
| | | *Flavor* | | | | |
| Roasted | Dry | 6.40$^{a}$ | 7.20$^{ax}$ | 5.25$^{b}$ | 5.10$^{b}$ | 0.281 |
| | Wet | 5.85$^{a}$ | 6.10$^{ay}$ | 4.75$^{b}$ | 4.95$^{b}$ | 0.294 |
| | SEM[2] | 0.345 | 0.216 | 0.279 | 0.296 | |
| Dry-aged | Dry | 4.75 | 4.95 | 5.95 | 5.10 | 0.374 |
| | Wet[3] | - | - | - | - | - |
| Cheesy | Dry | 5.00$^{x}$ | 4.25 | 5.45$^{x}$ | 4.15 | 0.384 |
| | Wet | 2.65$^{y}$ | 3.65 | 2.85$^{y}$ | 2.95 | 0.366 |
| | SEM[2] | 0.321 | 0.417 | 0.321 | 0.427 | |
| Fatty | Dry | 4.20 | 4.40 | 4.70 | 3.95 | 0.392 |
| | Wet | 4.30 | 4.20 | 3.65 | 4.50 | 0.360 |
| | SEM[2] | 0.371 | 0.363 | 0.404 | 0.366 | |
| Savory | Dry | 5.30 | 5.55 | 5.75 | 5.45 | 0.345 |
| | Wet | 5.60 | 5.45 | 4.50 | 4.65 | 0.402 |
| | SEM[2] | 0.342 | 0.290 | 0.468 | 0.376 | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *Texture* | | | | |
| Tenderness | Dry | 5.55$^{x}$ | 5.85 | 6.45$^{x}$ | 5.80 | 0.326 |
| | Wet | 3.65$^{by}$ | 5.35$^{a}$ | 5.15$^{ay}$ | 4.85$^{a}$ | 0.367 |
| | SEM[2] | 0.294 | 0.317 | 0.349 | 0.417 | |
| Juiciness | Dry | 3.90$^{aby}$ | 4.35$^{a}$ | 3.70$^{ab}$ | 2.95$^{by}$ | 0.312 |
| | Wet | 5.30$^{x}$ | 4.05 | 4.10 | 4.20$^{x}$ | 0.403 |
| | SEM[2] | 0.373 | 0.390 | 0.340 | 0.334 | |

The descriptive sensory analysis was performed twice and the result was analyzed by randomized block design with 10 panelists per trial.

[1]Standard error of mean (n = 8), [2](n = 4).

[3]Not tested as dry-aged flavor cannot exist in wet-aged beef.

[a-c]Different letters within the same row differ significantly ($p < 0.05$).

[x,y]Different letters within the same column differ significantly ($p < 0.05$).

Based on the Pearson correlation analysis, the meat color, roasted and savory flavor, and juiciness were strongly related to the overall acceptability of both aged beef (Table 17). The relationships between internal meat color and roasted flavor with overall acceptability were also significant, respectively, when using linear mixed model. As the brown surface color indicates the degree of caramelization and the point of consumption (Suman et al., 2016; Yoo et al., 2020), the positive correlation between the degree of doneness and overall acceptability is natural. The sensory scores of meat color and roasted flavor suggest that grilling instead of oven roasting would be effective for dry-aged beef (Table 16). Moreover, dry-aged beef that was grill-cooked at 150°C had significantly higher scores for surface color, dry-aged and cheesy flavor compared to wet-aged beef. We found that cheesy and savory flavors are positively correlated ($r^2 = 0.53$; $p < 0.05$). This indicates that the unique flavor of dry-aged beef could be perceived strongly even at lower cooking temperature and might be more attractive for consumers who prefer dry-aged and cheesy flavors than that of wet-aged beef after grilling. The flavor difference between dry- and wet-aged beef was not observed (except the dry-aged flavor) only when the dry-aged beef was grill-cooked at 150°C while the wet-aged beef was grilled at much higher cooking temperature ($p > 0.05$). This range of cooking temperature for dry-aged beef (150°C) has benefits for reducing overcooking and increases of heterocyclic aromatic amines, polycyclic aromatic hydrocarbons, and trans fatty acids compared to the higher cooking temperature for wet-aged beef (230°C) (Suleman et al., 2020). Meanwhile, an obvious contrast between the roasted flavor of dry- and wet-aged beef was observed when both samples were grill-cooked

at 230°C. As discussed in section 3.3.5, higher pyrazine compound concentrations in dry-aged beef compared to wet-aged beef could intensify the roasted flavor of dry-aged beef. Although no difference in cheesy flavor was found between them ($p > 0.05$), the characteristic flavor of dry-aged beef compared to wet-aged beef could be derived from the significantly higher intensities of roasted and dry-aged flavor. As roasted flavor was positively correlated with overall acceptability of beef (Table 17), grilling at higher cooking temperature could also be effective for the purpose of dry aging to develop the desirable beef flavor.

From the results, grilling of dry-aged beef at both lower temperature (150°C) and higher temperature (230°C) had their own advantages. The former led to higher intensities of surface color and cheesy flavor of dry-aged beef compared to those of wet-aged beef cooked at the same temperature. The latter presented a higher roasted flavor than wet-aged beef without any perceivable sensory defects. Consequently, grill cooking at both 150°C and 230°C might be promising cooking conditions for dry-aged beef to obtain the characteristic flavors and acceptance by a wide variety of consumers.

Table 17. Pearson correlation coefficients and regression coefficients by linear mixed model between sensory properties and acceptability of dry- and wet-aged beef strip loins

| Coefficients | Surface color | Internal color | Roasted flavor | Dry-aged flavor | Cheesy flavor | Fatty flavor | Savory flavor | Tenderness | Juiciness |
|---|---|---|---|---|---|---|---|---|---|
| Correlation | 0.55* | 0.58* | 0.60* | ns | ns | ns | 0.61* | ns | 0.54* |
| Regression | ns | 0.17* | 0.26** | ns | ns | ns | ns | ns | ns |

*, $p < 0.05$; **, $p < 0.01$.
ns, not significant.

## 3.4. Conclusion

The present results suggest that the advantages of dry aging can be enhanced by grill cooking instead of oven roasting, as grilling improves desirable flavor and color. In addition, the grill-cooked dry-aged beef might be appealing to consumers due to its intense roasted flavor, compared to grill-cooked wet-aged beef at the same cooking condition, and it is greater when cooking temperature is higher. Within the treatments in this study, grill cooking of dry-aged beef at a higher temperature (230°C) would be recommended.

# References

Aaslyng, M. D., & Meinert, L. (2017). Meat flavor in pork and beef – From animal to meal. *Meat Science*, *132(1)*, 112-117.

Adhikari, K., Chambers IV, E., Miller, R., Vázquez-Araújo, L., Bhumiratana, N., & Philip, C. (2011). Development of a lexicon for beef flavor in intact muscle. *Journal of Sensory Studies*, *26(6)*, 413-420.

American Industrial Hygiene Association. (1989). Odor thresholds for chemicals with established occupational health standards. Fairfax, USA: American Industrial Hygiene Association.

Ángel-Rendón, S. V., Filomena-Ambrosio, A., Hernández-Carrión, M., Llorca, E., Hernando, I., Quiles, A., & Sotelo-Díaz, I. (2020). Pork meat prepared by different cooking methods. A microstructural, sensorial and physicochemical approach. *Meat Science*, *163*, 1-9.

Blank, I., Sen, A., & Grosch, W. (1992). Sensory study on the character-impact flavour compounds of dill herb (*Anethum graveolens* L.). *Food Chemistry*, *43(5)*, 337-343.

Broncano, J. M., Petrón, M. J., Parra, V., & Timón, M. L. (2009). Effect of different cooking methods on lipid oxidation and formation of free cholesterol oxidation products (COPs) in *Latissimus dorsi* muscle of Iberian pigs. *Meat Science*, *83(3)*, 431-437.

Bruna, J. M., Hierro, E. M., de la Hoz, L., Mottram, D. S., Fernández, M., & Ordóñez, J. A. (2001). The Contribution of *Penicillium aurantiogriseum* to the volatile composition and sensory quality of dry fermented sausages. *Meat Science*, *59(1)*, 97-107.

Casaburi, A., Piombino, P., Nychas, G. J., Villani, F., & Ercolini, D. (2015). Bacterial populations and the volatilome associated to meat spoilage. *Food Microbiology*, *45(1)*, 83-102.

Choe, J., Park, B., Lee, H. J., & Jo, C. (2020). Potential antioxidant and angiotensin I-converting enzyme inhibitory activity in crust of dry-aged beef. *Scientific Reports*, *10*(1), 1-8.

Christensen, L., Gunvig, A., Tørngren, M. A., Aaslyng, M. D., Knøchel, S., & Christensen, M. (2012). Sensory characteristics of meat cooked for prolonged times at low temperature. *Meat Science*, *90(2)*, 485-489.

Colle, M. J., Richard, R. P., Killinger, K. M., Bohlscheid, J. C., Gray, A. R., Loucks, W. I., Day, R. N., Cochran, A. S., Nasados, J. A., & Doumit, M. E. (2016). Influence of extended aging on beef quality characteristics and sensory perception of steaks from the *biceps femoris* and *semimembranosus*. *Meat Science*, *119*, 110-117.

Corral, S., Belloch, C., López-Díez, J. J., & Flores, M. (2018). Lipolysis and aroma generation as mechanisms involved in masking boar taint in sodium reduced fermented sausages inoculated with *Debaryomyces hansenii* yeast. *Journal of the Science of Food and Agriculture*, *98(6)*, 2121-2130.

DeGeer, S. L., Hunt, M. C., Bratcher, C. L., Crozier-Dodson, B. A., Johnson, D. E., & Stika, J. F. (2009). Effects of dry aging of bone-in and boneless strip loins using two aging processes for two aging times. *Meat Science*, *83(4)*, 768-774.

del Pulgar, J. S., Gázquez, A., & Ruiz-Carrascal, J. (2012). Physico-chemical, textural and structural characteristics of sous-vide cooked pork cheeks as affected by vacuum, cooking temperature, and cooking time. *Meat Science*, *90(3)*, 828-835.

Dikeman, M. E., Obuz, E., Gök, V., Akkaya, L., & Stroda, S. (2013). Effects of dry, vacuum, and special bag aging; USDA quality grade; and end-point temperature on yields and eating quality of beef *Longissimus lumborum* steaks. *Meat Science*, *94(2)*, 228-233.

Domínguez, R., Gómez, M., Fonseca, S., & Lorenzo, J. M. (2014). Effect of different cooking methods on lipid oxidation and formation of volatile compounds in foal meat. *Meat Science*, *97(2)*, 223-230.

Domínguez, R., Purriños, L., Pérez-Santaescolástica, C., Pateiro, M., Barba, F. J., Tomasevic, I., Campagnol, P. C. B., & Lorenzo, J. M. (2019). Characterization of volatile compounds of dry-cured meat products using HS-SPME-GC/MS technique. *Food Analytical Methods*, *12(6)*, 1263-1284.

Dregus, M., & Engel, K. H. (2003). Volatile constituents of uncooked rhubarb (*Rheum rhabarbarum* L.) stalks. *Journal of Agricultural and Food Chemistry*, *51(22)*, 6530-6536.

Echegaray, N., Paterio, M., Domínguez, R., Purriños, L., Bermúdez, R., Carballo, J., & Lorenzo, J. M. (2020). Effects of different cooking methods and of the inclusion of chestnut (*Castanea sativa* Miller) in the finishing diet of Celta pig breed on the physicochemical parameters and volatile profile of *Longissimus thoracis et lumborum* muscle. *Food Research International*, *137*, 109407.

Fabre, R., Dalzotto, G., Perlo, F., Bonato, P., Teira, G., & Tisocco, O. (2018). Cooking method effect on Warner-Bratzler shear force of different beef muscles. *Meat Science*, *138*, 10-14.

Flores, M. (2018). Understanding the implications of current health trends on the aroma of wet and dry cured meat products. *Meat Science*, *144(1)*, 53-61.

Giannoukos, S., Brkić, B., & Taylor, S. (2019). Direct analysis and monitoring of organosulphur compounds in the gaseous phase using portable mass spectrometry. *Analytical Methods*, *11(38)*, 4882-4889.

Gudjónsdóttir, M., Gacutan Jr, M. D., Mendes, A. C., Chronakis, I. S., Jespersen, L., & Karlsson, A. H. (2015). Effects of electrospun chitosan wrapping for dry-ageing of beef, as studied by microbiological, physicochemical and low-field nuclear magnetic resonance analysis. *Food Chemistry*, *184*, 167-175.

Ha, Y., Hwang, I., Van Ba, H., Ryu, S., Kim, Y., Kang, S. M., Kim, J., Kim, Y., & Cho, S. (2019). Effects of dry-and wet-ageing on flavor compounds and eating quality of low fat Hanwoo beef muscles. *Food Science of Animal Resources*, *39(4)*, 655-667.

Hou, L., Zhang, Y., & Wang, X. (2019). Characterization of the volatile compounds and taste attributes of sesame pastes processed at different temperatures. *Journal of Oleo Science*, *68(6)*, 551-558.

Iida, F., Miyazaki, Y., Tsuyuki, R., Kato, K., Egusa, A., Ogoshi, H., & Nishimura, T. (2016). Changes in taste compounds, breaking properties, and sensory attributes during dry aging of beef from Japanese black cattle. *Meat Science*, *112(1)*, 46-51.

Iucci, L., Patrignani, F., Belletti, N., Ndagijimana, M., Guerzoni, M. E., Gardini, F., & Lanciotti, R. (2007). Role of surface-inoculated *Debaryomyces hansenii* and *Yarrowia lipolytica* strains in dried fermented sausage manufacture. Part 2: Evaluation of their effects on sensory quality and biogenic amine content. *Meat Science*, *75(4)*, 669-675.

Jayasena, D. D., Ahn, D. U., Nam, K. C., & Jo, C. (2013). Factors affecting cooked chicken meat flavour: a review. *World's Poultry Science Journal*, *69(3)*, 515-526.

Kahraman, H. A., & Gurbuz, U. (2019). Effects of three aging methods on the Longissimus lumborum muscle from Holstein-Friesian steers. *Medycyna Weterynaryjna*, 75(03), 179-184.

KAPE (Korea Institute for animal products quality evaluation). (2019). *Animal products grading statistical yearbook*. Kunpo: KAPE.

Keatkrai, J., & Jirapakkul, W. (2010). Volatile profile of *khanom jeen*, Thai fermented rice noodles, and the changes during the fermentation process. *ScienceAsia*, *36*, 46-51.

Kerth, C. R., Blair-Kerth, L. K., & Jones, W. R. (2003). Warner-Bratzler shear force repeatability in beef longissimus steaks cooked with a convection oven, broiler, or clam-shell grill. *Journal of Food Science*, *68(2)*, 668-669.

Kerth, C. R., & Miller, R. K. (2015). Beef flavor: a review from chemistry to consumer. *Journal of the Science of Food and Agriculture*, *95(14)*, 2783-2798.

Khan, M. I., Jung, S., Nam, K. C., & Jo, C. (2016). Postmortem aging of beef with a special reference to the dry aging. *Korean Journal for Food Science of Animal Resources*, *36(2)*, 159-169.

Kim, H. C., Baek, K. H., Ko, Y. J., Lee, H. J., Yim, D. G., & Jo, C. (2020). Characteristic metabolic changes of the crust from dry-aged beef using 2D NMR spectroscopy. *Molecules*, *25(13)*, 3087.

Kim, J. H., Kim, D. H., Ji, D. S., Lee, H. J., Yoon, D. K., & Lee, C. H. (2017). Effect of aging process and time on physicochemical and sensory evaluation of raw beef top round and shank muscles using an electronic tongue. *Korean Journal for Food Science of Animal Resources*, *37(6)*, 823-832.

Kim, J. H., Jeon, M. Y., & Lee, C. H. (2019). Physicochemical and sensory characteristics of commercial, frozen, dry, and wet-aged Hanwoo sirloins. *Asian-Australasian Journal of Animal Sciences*, *32(10)*, 1621-1629.

Kim, M., Choe, J., Lee, H. J., Yoon, Y., Yoon, S., & Jo, C. (2019). Effects of aging and aging method on physicochemical and sensory traits of different beef cuts. *Food Science of Animal Resources*, *39(1),* 54-64.

Kim, Y. H. B., Kemp, R., & Samuelsson, L. M. (2016). Effects of dry-aging on meat quality attributes and metabolite profiles of beef loins. *Meat Science*, *111(1)*, 168-176.

King, M. F., Matthews, M. A., Rule, D. C., & Field, R. A. (1995). Effect of beef packaging method on volatile compounds developed by oven roasting or microwave cooking. *Journal of Agricultural and Food Chemistry*, *43(3)*, 773-778.

Lee, H. J., Yoon, J. W., Kim, M., Oh, H., Yoon, Y., & Jo, C. (2019a). Changes in microbial composition on the crust by different air flow velocities and their effect on sensory properties of dry-aged beef. *Meat Science*, *153(1)*, 152-158.

Lee, H. J., Choe, J., Kim, M., Kim, H. C., Yoon, J. W., Oh, S. W., & Jo, C. (2019b). Role of moisture evaporation in the taste attributes of dry- and wet-aged beef determined by chemical and electronic tongue analyses. *Meat Science*, *151(1)*, 82-88.

Ma, Q. L., Hamid, N., Bekhit, A. E. D., Robertson, J., & Law, T. F. (2012). Evaluation of pre-rigor injection of beef with proteases on cooked meat volatile profile after 1 day and 21 days post-mortem storage. *Meat Science*, *92(4)*, 430-439.

Madruga, M. S., Elmore, J. S., Oruna-Concha, M. J., Balagiannis, D., & Mottram, D. S. (2010). Determination of some water-soluble aroma precursors in goat meat and their enrolment on flavour profile of goat meat. *Food Chemistry*, *123(2)*, 513-520.

Madruga, M. S., & Mottram, D. S. (1995). The effect of pH on the formation of Maillard-derived aroma volatiles using a cooked meat system. *Journal of the Science of Food and Agriculture*, *68(3)*, 305-310.

Maggiolino, A., Lorenzo, J. M., Marino, R., Della Malva, A., Centoducati, P., & De Palo, P. (2019). Foal meat volatile compounds: effect of vacuum ageing on semimembranosus muscle. *Journal of the Science of Food and Agriculture*, 99(4), 1660-1667.

Mitacek, R. M., Ke, Y., Prenni, J. E., Jadeja, R., VanOverbeke, D. L., Mafi, G. G., & Ramanathan, R. (2019). Mitochondrial degeneration, depletion of NADH, and oxidative stress decrease color stability of wet-aged beef longissimus steaks. *Journal of Food Science*, *84(1)*, 38-50.

Mottier, P., Parisod, V., & Turesky, R. J. (2000). Quantitative determination of polycyclic aromatic hydrocarbons in barbecued meat sausages by gas chromatography coupled to mass spectrometry. *Journal of Agricultural and Food Chemistry*, *48(4)*, 1160-1166.

Mottram, D. S. (1998). Flavour formation in meat and meat products: a review. *Food Chemistry*, *62(4)*, 415-424.

Muriel, E., Antequera, T., Petrón, M. J., Andrés, A. I., & Ruiz, J. (2004). Volatile compounds in Iberian dry-cured loin. *Meat Science*, *68(3)*, 391-400.

Myrdal Miller, A., Mills, K., Wong, T., Drescher, G., Lee, S. M., Sirimuangmoon, C., Schaefer, S., Minor, B., & Guinard, J. X. (2014). Flavor-enhancing properties of mushrooms in meat-based dishes in which sodium has been reduced and meat has been partially substituted with mushrooms. *Journal of Food Science*, 79(9), S1795-S1804.

Nagata, Y., & Takeuchi, N. (2003). Measurement of odor threshold by triangle odor bag method. *Odor Measurement Review*, 118, 118-127.

Niu, Y., Wang, R., Xiao, Z., Zhu, J., Sun, X., & Wang, P. (2019). Characterization of ester odorants of apple juice by gas chromatography-olfactometry, quantitative measurements, odour threshold, aroma intensity and electronic nose. *Food Research International*, *120*, 92-101.

Oh, H., Lee, H. J., Lee, J., Jo, C., & Yoon, Y. (2019). Identification of microorganisms associated with the quality improvement of dry-aged beef through microbiome analysis and DNA sequencing, and evaluation of their effects on beef quality. *Journal of Food Science*, *84(10)*, 2944-2954.

Oh, J., Lee, H. J., Yoon, J. W., Choe, J., & Jo, C. (2019). Electrical resistance and mold distribution on beef surface as indicators of dry aging. *Journal of Food Process Engineering*, *42(5)*, e13122.

Ozkara, K. T., Amanpour, A., Guclu, G., Kelebek, H., & Selli, S. (2019). GC-MS-Olfactometric differentiation of aroma-active compounds in turkish heat-treated sausages by application of aroma extract dilution analysis. *Food Analytical Methods*, *12(3)*, 729-741.

Park, B., Yong, H. I., Choe, J., & Jo, C. (2018). Utilization of the crust from dry-aged beef to enhance flavor of beef patties. *Korean Journal for Food Science of Animal Resources*, *38(5)*, 1019-1028.

Pathare, P. B., & Roskilly, A. P. (2016). Quality and energy evaluation in meat cooking. *Food Engineering Reviews*, *8(4)*, 435-447.

Peñaranda, I., Garrido, M. D., Egea, M., Díaz, P., Álvarez, D., Oliver, M. A., & Linares, M. B. (2017). Sensory perception of meat from entire male pigs processed by different heating methods. *Meat Science*, *134*, 98-102.

Pérez-Santaescolástica, C., Carballo, J., Fulladosa, E., Garcia-Perez, J. V., Benedito, J., & Lorenzo, J. M. (2018). Effect of proteolysis index level on instrumental adhesiveness, free amino acids content and volatile compounds profile of dry-cured ham. *Food Research International*, *107*, 559-566.

Rao, W., Wang, Z., Li, G., Meng, T., Suleman, R., & Zhang, D. (2020). Formation of crust of dried meat and its relationship to moisture migration during air drying. *Journal of Food Processing and Preservation*, *44*, e14255.

Ribeiro, F. A., Lau, S. K., Pflanzer, S. B., Subbiah, J., & Calkins, C. R. (2021). Color and lipid stability of dry aged beef during retail display. *Meat Science*, *171*, 108274.

Saini, R., Jaitak, V., Guleria, S., Kaul, V. K., Kiran Babu, G. D., Singh, B., Lal. B., & Singh, R. D. (2012). Comparison of headspace analysis of volatile constituents with GCMS analysis of hydrodistilled and supercritical fluid extracted oil of *Capillipedium parviflorum*. *Journal of Essential Oil Research*, *24(3)*, 315-320.

Sawyer, J. T., Apple, J. K., & Johnson, Z. B. (2008). The impact of lactic acid concentration and sodium chloride on pH, water-holding capacity, and cooked color of injection-enhanced dark-cutting beef. *Meat Science*, *79(2)*, 317-325.

Sekhon, R. K., Schilling, M. W., Phillips, T. W., Aikins, M. J., Hasan, M. M., Corzo, A., & Mikel, W. B. (2010). Effects of phosphine and methyl bromide fumigation on the volatile flavor profile and sensory quality of dry cured ham. *Meat Science*, *86(2)*, 411-417.

Silva, F. A., Ferreira, V. C., Madruga, M. S., & Estévez, M. (2016). Effect of the cooking method (grilling, roasting, frying and sous-vide) on the oxidation of thiols, tryptophan, alkaline amino acids and protein cross-linking in jerky chicken. *Journal of Food Science and Technology*, *53(8)*, 3137-3146.

Starkey, C. P., Geesink, G. H., van de Ven, R., & Hopkins, D. L. (2017). The relationship between shear force, compression, collagen characteristics, desmin degradation and sarcomere length in lamb *biceps femoris*. *Meat Science*, *126*, 18-21.

Suleman, R., Wang, Z., Aadil, R. M., Hui, T., Hopkins, D. L., & Zhang, D. (2020). Effect of cooking on the nutritive quality, sensory properties and safety of lamb meat: Current challenges and future prospects. *Meat Science*, *167*, 108172.

Suman, S. P., Nair, M. N., Joseph, P., & Hunt, M. C. (2016). Factors influencing internal color of cooked meats. *Meat Science*, *120*, 133-144.

Terjung, N., Witte, F., & Heinz, V. (2020). The dry aged beef paradox: Why dry aging is sometimes not better than wet aging. *Meat Science*, *172*, 108355.

Turp, G. Y. (2016). Effects of four different cooking methods on some quality characteristics of low fat Inegol meatball enriched with flaxseed flour. *Meat Science*, *121*, 40-46.

Van Ba, H., Hwang, I., Jeong, D., & Touseef, A. (2012). Principle of meat aroma flavors and future prospect. *Latest Research into Quality Control*, *2*, 145-176.

Vieira, S. A., Zhang, G., & Decker, E. A. (2017). Biological implications of lipid oxidation products. *Journal of the American Oil Chemists' Society*, *94(3)*, 339-351.

Wall, K. R., Kerth, C. R., Miller, R. K., & Alvarado, C. (2019). Grilling temperature effects on tenderness, juiciness, flavor and volatile aroma compounds of aged ribeye, strip loin, and top sirloin steaks. *Meat Science*, *150*, 141-148.

Watanabe, A., Kamada, G., Imanari, M., Shiba, N., Yonai, M., & Muramoto, T. (2015). Effect of aging on volatile compounds in cooked beef. *Meat Science*, *107(1)*, 12-19.

Whitfield, F. B., & Mottram, D. S. (1992). Volatiles from interactions of Maillard reactions and lipids. *Critical Reviews in Food Science & Nutrition*, *31(1-2)*, 1-58.

Wojnowski, W., Majchrzak, T., Dymerski, T., Gębicki, J., & Namieśnik, J. (2017). Poultry meat freshness evaluation using electronic nose technology and ultra-fast gas chromatography. *Monatshefte für Chemie-Chemical Monthly*, *148(9)*, 1631-1637.

Wu, W., Tao, N. P., & Gu, S. (2014). Characterization of the key odor-active compounds in steamed meat of *Coilia ectenes* from Yangtze River by GC-MS-O. *European Food Research and Technology*, *238(2)*, 237-245.

Yancey, J. W. S., Wharton, M. D., & Apple, J. K. (2011). Cookery method and end-point temperature can affect the Warner–Bratzler shear force, cooking loss, and internal cooked color of beef longissimus steaks. *Meat Science*, *88(1)*, 1-7.

Yang, J., Dashdorj, D., & Hwang, I. (2019). Volatile flavor components as a function of electrical stimulation and chiller aging for *m. longissimus* and *biceps femoris* of Hanwoo beef. *Food Science of Animal Resources*, *39(3)*, 474-493.

Yoo, J. H., Kim, J. W., Yong, H. I., Baek, K. H., Lee, H. J., & Jo, C. (2020). Effects of searing cooking on sensory and physicochemical properties of beef steak. *Food Science of Animal Resources*, *40(1)*, 44-54.

Yu, H., Seow, Y. X., Ong, P. K., & Zhou, W. (2019). Effects of ultrasonic processing and oil type on Maillard reaction of D-Glucose and L-Alanine in oil-in-water systems. *Food and Bioprocess Technology*, *12(2)*, 325-337.

Zeidler, G., Pasin, G., Luh, B. S., Thompson, J. F., & Rice, R. D. (1989). Reducing cooking time, yield losses and energy utilization of Salisbury steaks as affected by various meat extenders and meat composition. *Foodservice Research International*, *5(3)*, 215-236.

Zhu, J., Chen, F., Wang, L., Niu, Y., & Xiao, Z. (2017). Evaluation of the synergism among volatile compounds in Oolong tea infusion by odour threshold with sensory analysis and E-nose. *Food Chemistry*, *221*, 1484-1490.

# Summary in Korean

## 건식숙성 우육의 풍미 구명 및 조리조건에 의한 풍미증진 효과

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건식숙성 우육은 숙성되지 않은 우육 및 습식숙성 우육과 뚜렷하게 구별되는 독특한 풍미를 가지는 것으로 알려져 있다. 하지만 건식숙성 우육의 풍미가 가지는 중요성에 비해 풍미를 구성하는 중요한 요소인 휘발성 향 화합물에 대한 연구는 미흡한 실정이다. 건식숙성 우육 특유의 풍미가 형성되는 기작을 이해하고 풍미를 증진시켜 건식숙성 우육의 관능적 품질 및 경쟁력을 높이기 위해서는 숙성조건, 조리조건 등 풍미에 영향을 미칠 수 있는 다양한 조건에서의 건식숙성 우육 내 휘발성 향 화합물의 형성에 대한 연구가 진행되어야 한다. 따라서 본 연구는 i) 건식숙성 과정에서 숙성기간에 따른 휘발성 향 화합물의 변화를 분석하고 ii) 건식숙성 우육의 풍미를 돋보일 수 있는 최적의 조리조건을 찾고자 수행되었다.

실험 Ⅰ에서는 숙성방법에 따른 우육 내 휘발성 향 화합물의 변화를 분석하였다. 우육 채끝을 각각 건식 또는 습식숙성하여 28일 기간 동안 7일

간격으로 휘발성 향 화합물을 분석비교하였다. 숙성기간이 길어질수록 건식숙성 우육 내 휘발성 향 화합물 농도가 습식숙성 우육에 비해 높은 값을 기록하였다($p < 0.05$). 건식숙성 과정 중 발생한 휘발성 향 화합물의 변화는 지방 산화 또는 미생물 효소 작용에 의해 생성될 수 있는 propanal, 2-methylbutanal, 2-methylpropanal, 1-butanamine, trimethylamine, 2-methyl-2-propanethiol, ethyl propanoate에서 집중적으로 관찰되었다. 이들 화합물은 malty, nutty, fruity, ammoniacal, fermented한 향을 낼 수 있다. 연구 결과를 종합해볼 때 건식숙성 우육의 휘발성 향 화합물이 습식숙성 우육과 차이를 보이는 이유는 외부에의 노출에 따른 지방 산화 및 미생물 활성의 증가에 의한 것으로 사료된다.

실험 Ⅱ에서는 조리조건이 건식 및 습식숙성 우육 채끝의 물리화학적 품질과 관능적 품질에 미치는 영향을 평가하였다. 각각 28일간 숙성시킨 건식 및 습식숙성 우육을 4종류의 조리조건[2종류의 조리방법(그릴 조리, 오븐 조리) × 2종류의 조리온도(150℃, 230℃)]으로 조리시간, 조리 후 pH, 조리감량, 지질산패도, 휘발성 향 화합물, 육색을 측정하였다. 조리조건은 pH에 영향을 미치지 않았으나, 그릴 조리 시 오븐 조리에 비하여 낮은 지질 산패도와 높은 수준의 겉면 익힘 정도를 보였다($p < 0.05$). 묘사분석 결과 그릴조리육이 오븐조리육에 비해 roasted flavor가 강하게 나타났다. 건식숙성 우육을 150℃에서 그릴 조리하였을 경우 같은 조건에서 조리한 습식숙성 우육에 비해 cheesy flavor가 더 강했으며, 건식숙성 우육을

230℃에서 그릴 조리하였을 경우 roasted flavor 강도가 더 높게 나타났다.

따라서 건식숙성 우육을 그릴 조리하는 것이 건식숙성 우육 특유의 풍미를 증진시키는 데 효과적일 것으로 사료된다.