Changes in Chemical-Physical Index and Microstructure During Dry-cured Duck Processing

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Dry-cured duck is a high quality meat product processed by the traditional dry-curing procedure. The objective of this paper was to study the physical-chemical parameters and microstructure of the duck muscle during the manufacturing process. Thirty six ducks were used in this study and samples were taken after dry salting, marinating, piling, and drying for 6 days and 12 days. The increase in NaCl, TBA, protein, fat, and shear force were observed whereas water, cooking loss, L*, a*, b* and myofiber diameter decreased during the whole process. It showed the quality parameters such as NaCl, TBA, shear force and water were strongly correlated and associated with the microstructural changes of the muscles.

Key words: chemical-physical index, dry-cured duck, microstructure, processing, traditional


Introduction

Traditional Chinese dry-cured duck is a well known local delicacy in China and Southeast Asia due to its tasty flavor and texture, and has a history of over 300 years (Li, 1988). In Nanjing city alone, about five million dry-cured ducks are consumed annually (Li, 1988). Similar to dry-cured Jinhua ham, dry-cured duck is produced by dry curing, marinating, piling and drying naturally but the period of its production is shorter than that of hams (Xu et al., 2008).

The chemical-physical index and microstructure changes that occur during the processing of dry-cured duck associated with proteolysis, lipolysis and lipid oxidation processes could contribute to the final taste and texture of the dry-cured meat product. The control of complex biochemical reactions which lead to the development of the typical sensory traits and texture of dry cured products depends largely on the manufacturing process (Toldra and Flores, 1998). The manufacture and physical-chemical characteristics of dry-cured meat products such as hams and sausages have been studied in many previous studies (Toldra and Flores, 1998). Lorenzo et al. (2010, 2013) has reported the physic-chemical characteristics of dry-cured duck, however, there is a lack of information about the physical and chemical changes that occur throughout the dry-cured duck manufacturing process. The microstructure of the dry-cured duck and its relationship with the chemical-physical characteristics is also quite few. The biophysical methods such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) have been applied to study the structure of a wide variety of foods, and it’s known the quality of meat products is closely related to their microstructure (Monin et al., 1997; Perez-Alvarez et al., 1999). Therefore, the objective of this study was to track the chemical-physical parameters, microstructure, and their inter-relationships in the manufacture process of traditional Chinese dry-cured duck.

Materials and Methods

Sample Preparation

Thirty-six lean-type Cherry Valley ducks from a commercial feedlot were slaughtered humanely in a commercial meat processing company (Jiangsu Yurun Food Ltd.), each of which was about 2.0 kg. After chilling for 2 h, dry-cured ducks were processed as follows: duck carcasses were dry-salted for 24 h (salt content: 6.5% of carcass weight), marinated in brine for 24 h (saturated salt solution), piled for 48 h and then dried at 2°C–10°C in a well-ventilated room for 6 to 12 days (Xu et al., 2008). Sampling stages, processing time, average temperature and relative humidity (RH) is shown in Table 1. At the end of each processing stage (including raw), six carcasses were selected for chemical-physical index and
microstructure analyses. The breast muscles were removed from the carcasses for physical-chemical measurement or stored at \(-40°C\) for microstructure analysis.

**Determination of Microstructure**

The microstructure of meat samples was determined using a scanning electron microscope (SEM) according to Chang *et al.* (2010) with slight changes. The procedure for SEM analysis was conducted as follows: Pieces (\(2 \times 2 \times 0.5 \text{ mm}\)) were excised from muscle samples and fixed in 2.5% glutaraldehyde in 0.1 M phosphate buffer (pH 7.3) at room temperature. The specimens were then rinsed with 0.1 M phosphate buffer (pH 7.3) and dehydrated for 15 min in 50%, 70%, 80% and 90% ethanol, respectively and three times in absolute ethanol for 30 min each, then rinsed in isopentyl acetate and ultradried by critical point with CO2 (\(1100 \text{°C}\)) in a POLARON E3000 instrument (Watford, United Kingdom). Then they were gold-coated using POLARON E6100 Equipment and observed in SEM (S-Titan, United Kingdom). Then they were gold-coated using (1,1,3,3-tetraethoxypropane) in the range from 0.02 μg/ml to 0.3 μg/ml and expressed as mg of MDA per kg sample.

**Determination of Chemical Index**


Lipid oxidation of all samples was assessed by the 2-thiobarbituric (TBA) method according to Sorensen and Jorgensen (1996). Ten grams sample was homogenized with 30 ml of a 7.5% trichloroacetic acid (TCA) solution containing 0.1% propylgallate (PG) and 0.1% ethylenediamine-tetraacetic acid, disodium salt (EDTA) for 30 s in an Ultra Turrax blender (9500 rpm) and filtered through a Whatman filter No. 42. Equal 5 ml volumes of filtrate and 0.02 M TBA solution were mixed with glassed stopped tubes and incubated in a water bath at 100°C for 40 min before cooling to room temperature under running cold tap water. The absorbance was measured at 532 nm using spectrophotometer. TBARS were calculated from a standard curve of malondialdehyde (MDA), freshly prepared by acidification of TEP (1,1,3,3-tetraethoxypropane) in the range from 0.02 μg/ml to 0.3 μg/ml and expressed as mg of MDA per kg sample.

**Determination of Physical Index**

The meat color (L*, a*, b*) was measured using a Colorimeter (CR 400, Minolta, Japan). The colorimeter was calibrated using a standard white ceramic tile before measuring each sample.

To measure the cooking loss of the samples, each breast fillet was weighted accurately prior to cooking. After cooking, the breast fillets were cooled to the internal temperature of room temperature and wiped with blotting paper to remove excess water and weighted immediately. Cooking loss was calculated as

\[
\text{Cooking loss (％)} = \frac{\text{[raw weight} - \text{cooked weight]}}{\text{raw weight}} \times 100
\]

After measurements of cooking loss, the same muscles were then used for the determination of shear force. Shear force was determined through the application of the Meullenet-Owens razor shear (MORS) test (Meullenet *et al.*, 2004), using a texture analyzer (TVT-300XP, TexVol Instruments, Viken, Sweden) equipped with a razor blade with a height of 24 mm and a width of 8.9 mm. Muscle strips were cut across the fiber axis. The crosshead speed was set at 2 mm/s, and the test was triggered by a 10 g contact force. The shear was perpendicular to the axis of muscle fibers. In each treatment, the MORS test value was determined in triplicates at predetermined locations on each of the fillets.

**Statistical Analyses**

The changes of chemical-physical index were evaluated by one-way analysis of variance techniques where these measurements were as dependent variables and the processing stage as independent variables. And means of the measurements at different processing stages were compared using the Duncan’s multiple-range test at the significance level of 0.05. Correlation coefficients among all the variables were evaluated by descriptive analysis of correlations. All statistical analyses were performed by SPSS 18.0 (Argyrous, 2011).

**Results and Discussion**

As can be observed from the SEM photograph (Fig. 1 & Fig. 2), after 6 days of drying, the muscle showed higher extent of hollows in both transverse and longitudinal directions in comparison with the raw muscle because of the salt diffusion and water loss. The hollows weakened the water retaining capacity of the muscle, and caused the increase in TBA as the interior surface of the muscle was exposed to the

<table>
<thead>
<tr>
<th>Sampling stage</th>
<th>Raw</th>
<th>Dry-salted</th>
<th>Marinated</th>
<th>Piled</th>
<th>Dried for 6 days</th>
<th>Dried for 12 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>Processing time / d</td>
<td>1.0</td>
<td>2.0</td>
<td>3.0</td>
<td>5.0</td>
<td>11.0</td>
<td>17.0</td>
</tr>
<tr>
<td>Mean temp. / °C</td>
<td>6.04</td>
<td>9.08</td>
<td>6.95</td>
<td>3.60</td>
<td>5.76</td>
<td>3.47</td>
</tr>
<tr>
<td>Mean RH / %</td>
<td>63.8</td>
<td>92.83</td>
<td>95.60</td>
<td>72.38</td>
<td>84.24</td>
<td>60.42</td>
</tr>
</tbody>
</table>

Table 1. Sampling stages, processing time, average temperature and RH during the processing of dry-cured duck
The raw muscle had adhesive surface, while the dry-cured muscle appeared to be dry. In the raw muscle, the fibers were firmly attached to one another by the endomysial connective tissue and the myofibrils inside the cells are strongly attached to each other and sarcolemma. While in the dry-cured muscle, the endomysial connective tissue was not intact, and the myofibril bundles and plasmatic membrane was apparently separated as a result of the degradation or denaturation of membrane and proteins that join the membrane to the myofibrils. The proteolysis of myosin, actin and other myofibrillar proteins throughout the drying stage has been reported in many studies (Toldra et al., 1993; Tabilo et al., 1999). Besides this reason, the high salt concentration might have caused partial solubilization of myofibrillar proteins, resulting in the weakness of the muscle structure (Sultana et al., 2008).

Table 2 shows chemical-physico properties, color parameters and instrumental texture measurements during the manufacture of dry-cured duck breast. There is no significant change in pH value in dry-cured duck meat during the whole process ($P > 0.05$). The final pH might be due to the integrated consequence of ammonia and amine generation (Hughes et al., 2002), and proteolytic activity of endogenous cathepsins (Verplaetse, 1994).

As we expected, NaCl content increased with the processing days from 0.29% to 8.92% (Table 2). This is similar to Lorenzo et al. (2010) who obtained the sodium chloride content of around 7.5 ± 1.22% in 21 days dry-cured duck.
breast and lower than Lorenzo et al. (2013) who obtained 12.73±0.99% in dry-cured ducks after 28 days of ripening. In the present study, the ducks were subjected to dry salting and saturated brine, which has been reported to improve the sensory properties such as greater firmness and retaining the color (Birkeland et al., 2003; Gallart-Jornet et al., 2007). NaCl has significant pro-oxidant effect in meat products (Rhee and Ziprin, 2001). Salt accelerates lipid oxidation but the mechanism of action is not fully elucidated. There is evidence that chloride ions may displace iron ions from binding macromolecules and make them available as initiators of lipid peroxidation (Rhee and Ziprin, 2001). Our results showed that TBARS values of dry-cured duck increased markedly and it’s relevant with the salt content. The TBARS value at the end of the process was much higher than those by others. Fernandez and Rodriguez (1991) and Sarrage et al. (2002) observed the value of 2.21 and 2 mg of malonaldehyde/kg in chorizo and dry cured loins respectively. Lipid oxidation plays a key role in the overall aroma of dry-cured products because of the generated volatiles, however, the high content of volatile compounds derived from lipid oxidation also lead to the rancid flavor.

The water content in dry-cured duck muscle declined progressively from the marinating period and was highly correlated with NaCl diffusion (Table 2). The water loss caused the hollows and the precipitation of salts on the surface of the muscles in the SEM photograph. The result was higher than those found by others in dry cured duck muscles that were
dried for longer time (Lorenzo et al., 2010; Lorenzo et al., 2013). The water content may also be influenced by the temperature and relative humidity of air (Armou et al., 2003). Cooking loss decreased significantly ($P<0.05$) during all process but increased during marinating. In dry salting, the intercellular water is extracted to the surface of the flesh, while in brine salting, the meat is soaked in a solution which reduces the outward diffusion of water (Rora et al., 2004). Birkeland et al. (2004) showed fillets subjected to dry salting had significantly higher liquid loss than that of marinating. However, Barat et al. (2002) reported that brine salting had great water losses when saturated brine was used.

Water content was negatively correlated with shear force (Table 3), which was in accordance with other studies (Monin et al., 1997; Virgili et al., 1995). This is due in part to the fact that during the drying of meat products there is product shrinkage proportional to the water loss, increasing the dry matter content of the sample used in the texture analysis (Potter, 1986). This may also explain the increase in protein and fat content at the end of the drying process. The shrinkage of the muscle also resulted in decreased myofiber diameter as shown in the SEM photograph (Fig. 1 & Fig. 2). The fiber diameter is highly correlated with shear force, water, and TBA values (Table 3). The fiber diameter of the final product was more uniform and better ordered than those at other stages, indicating the drying time is enough to guarantee the salt permeate into the inner duck muscle.

$L^*$ values decreased rapidly during the whole process and it was significantly correlated with NaCl, protein, fat, and water content (Table 3). The decrease in $L^*$ values might be due to the browning reaction and loss of water during the ripening (Ventanas et al., 2007). $a^*$ and $b^*$ values was constant during the initial processing stages, but decreased significantly from 6 days drying to 12 days drying. It was inferred that the reason for decreased $a^*$ and $b^*$ values was the formation of metmyoglobin when the muscle was exposed to air (Millar et al., 1994). From the SEM photograph
(Fig. 1 & Fig. 2), with increasing extent of hollows, myoglobin in the interior surface of the muscle had more contact with air, making it more easily to be oxidized to metmyoglobin. During the processing of dry-cured duck, NaCl, water, tenderness, color, lipids and protein parameters showed dramatically changes with the exception of pH value. Water content and cooking loss decreased significantly while NaCl, shear force and TBA increased. The chemical-physical process assisted with the microstructural study could contribute to our knowledge of exactly what changes take place during processing, and might be useful to define and optimize the process.

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