IMPACT OF LIPID AND SODIUM CHLORIDE CONTENTS ON WATER TRANSFERS IN DRY-FERMENTED SAUSAGES

Hassan Safa*¹, Jean-Dominique Daudin¹, and Pierre-Sylvain Mirade¹

¹ INRA, UR370 Qualité des Produits Animaux, F-63122 Saint-Genès-Champanelle, France.

* Corresponding author: Tel. +33 4 73 62 41 79; E-mail address: hassan.safa@clermont.inra.fr

Abstract - Drying and fermentation are oldest methods allowing meat preservation. Dry-fermented sausage (DFS) manufacture consists in decreasing water activity 'aw' by adding high quantity of sodium chloride and proceeding to drying. Reducing salt and fat contents in these kinds of products could be interesting from a human health point of view but it could affect water transfer phenomena. Therefore, the purpose of this study was to evaluate the effect of modifying salt and fat contents on DFS weight loss and water activity and to build a sorption isotherm model that relates DFS water activity to water, salt and fat contents. Results showed that water transfer phenomena (weight losses and a_w) depended strongly on fat and salt contents and that combined salt and fat reductions led to faster weight losses but to higher aw values. On the other hand, we highlighted that DFS a_w could be evaluated from a Ross model corresponding to a salted and fatty gelatin gel, however with a new γ coefficient.

Key Words – dry-fermented sausages, sorption isotherm, weight losses, Ross model.

I. INTRODUCTION

Meat is essentially constituted of water, proteins, lipids and mineral elements. In food products, water exists under various forms. A part of water content is associated with other hydrophilic molecules such as proteins and mineral elements, while another quantity of water is present in free form (water availability). This water availability can be evaluated by measuring water activity (a_w). Actually, free water content and thus a_w are critical factors in the determination of the organoleptic qualities and of the microbiological safety of food products. Preserving meat such as during drycured ham and dry-fermented sausage manufacture is essentially based on adding a large amount of sodium chloride and on drying which means lower water content and lower aw values. For example, dry-fermented sausages is a

preparation of minced meat (70% lean and 25% fat) mixed with additives and curing agents (5%), pushed in casing and dried for many days. However, drying provokes a concentration in salt and fat contents in food products which can have harmful consequences on human health. Therefore, it was recommended to reduce salt and fat contents in these kinds of products. Nevertheless, reducing salt and fat contents during DFS manufacture could modify water transfers. On the other hand, a_w can be determined by sorption isotherm curves which relate a_w value to water content and contents of other components such as fat and salt. Thus, the objective of the present study is twofold: first, to evaluate from an experimental design the effect of modifying salt and fat levels on the water losses and a_w in the case of different DFS formulations and second, to build a sorption isotherm curve for DFS.

II. MATERIALS AND METHODS

DFS manufacture

A Doehlert design was established to investigate the effects of fat and salt contents on water transfers during DFS processing. This design was built based on two factors: fat content in the range [8.4%-21%] which was studied at five levels and salt content in the range [2.0%-2.8%] which was investigated at three levels. Therefore, seven formulations of dry-fermented sausages with different amounts of salt and fat were obtained applying this design. Additionally, as is shown in Table 1, a control formulation (S8) which corresponds to classic dry-fermented sausages was added (Table 1) and used as baseline reference for the other seven formulations.

For each formulation in Table 1, about 30 dryfermented sausages were manufactured as per the following procedure. Raw pork meat was purchased from a local distributor (DISTRIPORC, Clermont-Ferrand, France). On receipt of the pork meat, we verified the pH, moisture and water activity (aw) of the pork lean. Pork shoulders were defatted and cut into small parallelepipeds. For each formulation, the corresponding amount of defatted pork shoulder and backfat was weighed, ground to 6 mm diameter, and mixed with the corresponding amount of salt, a set of additives and a starter culture corresponding to a midacidification kinetic starter (MF55, Biovitec, Lissieu, France) prepared at 100 kg/L and added at 10 g/kg. In each meat batter, we added dextrose (5 g/kg), potassium nitrate (0.3 g/kg), potassium erythorbate (0.5 g/kg), black pepper (2 g/kg), garlic powder (0.5 g/kg) and finally a solution of starters (10 g/kg). The meat batter was then stuffed into 50 mm-diameter collagen casings. The raw sausages (450 g and about 20 cm in length) were then plunged in a Penicillium nalgiovensis solution to cover their surface. All products were steamed for 4 days at 24°C and 70% relative humidity (RH) then dried for 25 days at 13°C and 70% RH in the same ripening room.

Table 1 Dry-fermented sausage formulations based on an experimental Doehlert design

Formulations	Animal fat content (%)	Sodium Chloride content (%)
S1	14.7	2.4
S2	21	2.4
S 3	17.9	2.8
S 4	8.4	2.4
S5	11.6	2.0
S 6	17.9	2.0
S 7	11.6	2.8
S8	21	2.8

DFS chemical composition

Water content was determined by drying about 1.5 g of sample at 80 ± 2 °C in a controlled-temperature chamber (Model FT127U, Firlabo, France) to constant weight, i.e. at least 48 h. Moisture content was expressed on a total matter basis (kg H₂O/kg TM).

Salt content was deduced from measurement of concentration in chloride ions by ion chromatography (850 professional IC, Metrohm, France). To achieve this, 2 g of DFS was diluted in 10 mL of pure water and centrifuged at room temperature (11,300 rpm, 10 min). 0.2 mL of the

supernatant was collected and diluted in 10 mL of pure water to measure chloride content.

Total fat content was determined according to the method described in [1], using dichloromethane: ethanol (2:1) instead of chloroform: methanol (2:1) as solvent.

Physicochemical measurements

During the drying period, the percentage of weight loss was determined by weighing together nine DFS from each formulation almost every day.

DFS water activity was determined using a LabMaster apparatus (Novasina –Aw SPRINT TH-500 Swiss).

DFS sorption isotherm model

The specific Ross model established for salted and fatty gelatin gels [2] was adapted to DFS measurements (a_w , salt content, fat content, water content) to allow prediction of DFS a_w as a function of water, salt and fat contents.

Statistical analysis

To make the results easier to interpret and the figures easier to read, a specific statistical treatment called hierarchical cluster analysis (HCA, Ward's method) was applied to all the measured raw values using STATISTICA 10-V2014 software.

Analysis of variance (ANOVA) was also performed using STATISTICA 10-V2014 software with the objective of assessing the effect of each factor (time, fat content, salt content and the interaction between salt and fat) on each variable measured in this study. Post-hoc procedures were then used when a significant effect was found (p < 0.05). Multiple comparisons among means were examined by the Tukey test to determine the level of significance between groups.

III. RESULTS AND DISCUSSION

Physicochemical properties

Results for weight losses are shown in Fig 1.

Analyzing this figure shows that, during the ripening period, all DFS lost weight constantly as a consequence of water transfers from products to air flux in the room. As shown in Fig 1, HCA led to the formation of three classes, with the following order: S1-S2-S3-S6-S8 (control) < S7 <

S4-S5. The first class corresponded to the fattiest formulations (21% for S1 and S2; 17.9% for S3 and S6 and 14.7% for S1). This class showed the lowest weight losses which reached about 42.9% at day 29.

Fig 1 Hierarchical cluster analysis performed on raw values of weight losses of the eight formulations of dry-

fermented sausages. Values not bearing common superscripts differed significantly (p < 0.05)



The two other classes pooled the leanest formulations (11.6% for S5 and S7 and 8.4% for S4). During the first two weeks (14 days), these two classes were very close in terms of percentage of weight losses. Beyond day 14, a differentiation in the percentage of weight losses could be observed. The class formed by formulation S7 which corresponded to the highest-salt formulation led to lower weight losses (45.4% at day 29) compared to the two other formulations (S4 and S5) which were less salty (2.4% and 2.0%, respectively), and for which water loss reached 49% at the end of drying. Furthermore, ANOVA showed that the water loss process was significantly affected by ripening time, salt content and by fat content. In contrast, there was no significant effect of the interaction between salt and fat content. These findings are in good accordance with results of [3, 4].

Fig 2 that shows the time-course of a_w indicates that a_w values of all formulations decreased constantly during ripening as a result of water losses and salt concentration. All formulations could be considered as safe from a microbiological point of view because all the final a_w values were below 0.92, i.e. the 'safe' threshold value. The highest a_w values were found in the formulations elaborated with the lowest salt content (2.0%, i.e. formulations S5 and S6) and in the leanest and salt-reduced formulation (S4). The lowest a_w values were found in the highest salted formulations (S3, S7 and control S8). As observed for DFS weight losses, ANOVA showed very significant effects of ripening time, salt content and fat content on the time-course of a_w . On the other hand, ANOVA showed that a_w was not affected by the interaction between salt content and fat content.

Fig 2 Hierarchical cluster analysis performed on raw values of a_w of the eight formulations of dry-fermented sausages. Values not bearing common superscripts differed significantly (p < 0.05)

differed significantly (p < 0.05)



These results are in quite accordance with the results of [3]. Therefore, they showed clearly that water transfer phenomena (weight losses and a_w) depended strongly on fat and salt contents.

DFS sorption isotherm model

The Ross model established for salty and fatty gelatin gels [2] and used in this study corresponds to the following equation:

$$a_{w,mix} = [-0.4553(X^{water}_{NaCl})^2 - 0.5242(X^{water}_{NaCl}) + 0.999] x [\gamma exp(-\alpha (X^{A}_{water})^{-Rf})]$$
(1)

In fact, Equation (1) corresponds to the product of two water activities: first, the water activity due to the presence of a certain amount of NaCl in the water contained in the gelatin gel, given by this relation:

$$[-0.4553(X^{water}_{NaCl})^2 - 0.5242(X^{water}_{NaCl}) + 0.999] (2)$$

Second, the water activity due to the presence of proteins in the unsalted gelatin gel, which is given by:

 $[\gamma \exp(-\alpha (X^{A}_{water})^{-Rf})]$ (3)

Therefore, total fat and salt contents as well as moisture were measured for all the DFS formulations of Table 1, at different ripening times. From these measurements, the NaCl content as a function of water content (i.e. the term $X^{\text{water}}_{\text{NaCl}}$) and the water content as a function of protein content (i.e. the term $X^{\text{water}}_{\text{water}}$) were calculated and then used for estimating the DFS a_{w} values.

By directly applying the 'salty and fatty gelatin gel' Ross model, it was found that the calculated a_w values do not match perfectly with the measured aw values. This may be due to the presence in DFS of other solutes such as sugars and nitrite, which would also exert a depressant effect on aw. Therefore, an adjustment of the coefficients corresponding to the 'salted and fatty gelatin gel' Ross model was performed using the solver procedure of Excel software. Only an adjustment of the γ coefficient was done, leading to a slight reduction of this coefficient from 0.993 to 0.991. Performing this adjustment then resulted in a quite good alignment between the measured aw values and the estimated values, as shown in Fig 3.

Fig 3 Dry-fermented sausage sorption isotherm built from analysis of DFS chemical composition and a_w measurement and from the 'salty and fatty gelatin gel' Ross model



In order to assess the quality of the new sorption isotherm model built for dry-fermented sausages, we calculated the mean discrepancy between estimated a_w values and measured values. A 0.012 mean discrepancy was obtained, revealing a quite small difference between water activity calculation and measurement.

Therefore, the following model:

 $\begin{array}{l} a_{w,mix} = \left[-0.4553 ~ (X^{water}_{NaCl})^2 ~ 0.5242 ~ (X^{water}_{NaCl}) + 0.999\right] * \left[\gamma ~ exp ~ (-\alpha (X^A_{water})^{-Rf})\right] & (4) \\ \text{with } \alpha = -0.02, ~ \gamma = 0.991 \text{ and } Rf = -1.96 \text{ can be} \\ \text{used to predict the } a_w \text{ value in a dry-fermented} \\ \text{sausage, knowing its fat content, moisture content} \\ \text{and salt content.} \end{array}$

IV CONCLUSION

The two main conclusions of this study are that: (1) performing a combined reduction in salt and fat contents during DFS manufacture leads to an increase in product water loss, but also to higher a_w values, and (2) a specific sorption isotherm curve based on a modified Ross model has been built allowing DFS water activity to be calculated as a function of water, fat and salt contents.

ACKNOWLEDGEMENTS

This work was funded by the European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement n°289397 (TeRiFiQ project).

REFERENCES

- Folch, J., Lees, M., & SloaneStanley, G.H. (1957). A simple method for isolation and purification of total lipids from animal tissues. Journal of Biological Chemistry 226: 497–508.
- Rougier, T., Bonazzi, C., & Daudin, J.D. (2007). Modeling the incidence of lipid and sodium chloride contents on sorption curves of gelatin in the high humidity range. LWT -Food Science and Technology 40(10): 1798-1807.
- Corral, S., Salvador, A., Belloch, C., & Flores, M. (2014). Effect of fat and salt reduction on the sensory quality of slow fermented sausages inoculated with Debaryomyces hansenii yeast. Food Control 45: 1-7.
- 4. Liaros, N.G., Katsanidis, E., & Bloukas, J.G. (2009). Effect of the ripening time under

vacuum and packaging film permeability on processing and quality characteristics of low-fat fermented sausages. Meat Science 83(4): 589-598.