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1 **Assessing Quality Parameters in Dry-Cured Ham using Microwave Spectroscopy**

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10
11 **Abstract**

12 Microwave spectroscopy has been applied in numerous non-food industry applications, and
13 recently also in the food industry, for non-destructive measurements. In this study, a dry-cured
14 ham model was designed and chemical analyses were performed for determining water activity,
15 water content and salt content (sodium chloride) for all samples. These chemical parameters were
16 also measured using microwave spectroscopy, with a rectangular microwave cavity resonator.
17 Results indicate that microwave spectroscopy may be a promising technique for determination of
18 water activity, salt content and water content in dry-cured ham using either reflected or
19 transmitted signals.

20
21 Keywords: Dry-cured ham, microwave, water activity, salt content, water content

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26 **1. Introduction**

27 Microwave spectroscopy has become a well-known technique in the non-food industry, including
28 applications for determining particulate blend composition on-line, biomedical measurements,
29 and humidity detection (Austin, Gupta, McDonnell, Reklaitis, & Harris, 2014; Bernou, Rebière,
30 & Pistré, 2000; Kim et al., 2012; Mason, Korostynska, Ortoneda-Pedrola, Shaw, & Al-
31 Shamma'a, 2013). Recently, microwave spectroscopy has also been applied in the food industry,
32 and a previous study show that microwave spectroscopy is a promising technique for determining
33 the water holding capacity (WHC) of raw meat (Abdullah, Cullen, Korostynska, Mason, & Al-
34 Shamma'a, 2014).

35 Being able to measure different quality parameters during the dry-curing process of ham is also
36 of a great interest. Water activity and water content are important parameters for controlling the
37 production and quality of dry-cured meats. The definition of water activity is the current volume
38 and availability of "free" water in a sample, which is given in values ranging between 0 (absolute
39 dryness) and 1 (condensed humidity). Meat products have high moisture content, thus their water
40 activity lies in the upper range of the water activity scale for foods. While fresh meat has water
41 activity above 0.99, the water activity for dried meat products is lower, between 0.92 and 0.80. It
42 is the availability of water for microbial, enzymatic or chemical activity that determines the shelf
43 life of food; with reduced water activity the shelf life and safety of meat products improves
44 (Andrés, Barat, Grau, & Fito, 2008).

45 Another parameter that is important for controlling the production and quality of dry-cured meat
46 is salt content. While consumers demand lower salt content in dry-cured meat products, the salt
47 content is an important factor for preventing bacterial contamination. The salt content in a dry-
48 cured ham evens out during the process. However, some parts might have higher salt content than
49 other parts in the final product, depending on thickness for example. Being able to control the salt
50 content in different parts of the ham is of a great interest for the industry. The methods applied
51 for measuring water activity, water and salt content in dry-cured meat products today are usually
52 destructive, in addition to being time consuming.

53 Non-destructive and rapid on-line measurements would simplify and improve the production and
54 quality control of dry-cured meat products. The microwave sensor operates in a wide range of
55 microwave frequencies, providing selectivity in real time detection of water activity, salt and
56 water content. The principle of using microwaves in real-time monitoring is based on the

57 interaction of the matter under test and the electromagnetic (EM) waves. The velocity of the
58 signal is changed by the test object, which attenuates or reflects it. The main advantages of
59 microwave sensors is that it can be implemented cheaply, yet be used for a wide range of
60 applications in a non-destructive and robust manner. Furthermore, microwave sensors are capable
61 of measuring without contact from a short distance, using penetrating waves, without health
62 hazards to personnel.

63 The aim is to develop an on-line non-destructive instrument for measuring different quality
64 parameters for raw and dry-cured meat, including water activity and salt content. Therefore, the
65 aim of this study was to investigate whether water activity, salt and water content can be
66 predicted using microwave spectroscopy. In this purpose, a dry-cured ham model was designed
67 and analysed.

68

69 **2. Materials and Methods**

70 **2.1 Sample preparation**

71 Figure 1 shows the experimental design. Loins from 8 pigs were selected, 4 pigs for the high salt
72 group and 4 pigs for the low salt group. For both high and low salt groups there were five weight
73 loss groups; 20, 25, 30, 35 and 40 % loss of initial weight. Generally, dry-cured meat products
74 have 30-35 % weight loss in the final product (Fellows, 2000). Each loin was deboned and sliced
75 in a total of 5 meat samples of similar dimensions: approximately 7 cm thick, 5-6 cm high, and
76 10 cm long. Each loin was therefore represented in all 5 weight loss groups. In order to achieve a
77 final salt concentration of approximately 8.0 % in the 30 % weight loss group (high salt), all meat
78 samples had 5.5 % salt added prior to vacuum packing. For the low salt group all meat samples
79 had 3.85 % salt added to achieve a final salt concentration of approximately 5.5 % in the 30 %
80 weight loss group. All meat samples were stored vacuum packed for two weeks at 4°C during
81 salting and salt equalization. After salt equalization the meat samples were dried (without
82 vacuum) at 12-14°C and 72-74 % relative humidity (RH) to obtain the desired weight losses.
83 When each meat sample reached the desired weight loss it was vacuum packed and stored at 4°C
84 until microwave measurements were performed.

85

86 **2.2 Chemical analysis**

87 Samples for chemical analyses, water activity, sodium chloride and water content, were taken
88 from each dry-cured meat sample at the same time as samples were analysed using microwave
89 spectroscopy. Water activity was measured by a water activity meter (Aqualab, USA), for all
90 replicates (3) from each of the meat samples. The water and sodium chloride content were
91 measured by an accredited lab (Eurofins, Norway). The sodium chloride content was calculated
92 using silver nitrate titration of chloride ions (Federation, 1997).

93

94 **2.3 Microwave spectroscopy**

95 Preparation of samples for microwave analysis was performed as shown in Fig. 2. A slice of
96 approximately 20 mm thickness was taken from the middle of each dry-cured meat sample. From
97 this slice, three replicates were taken with a 25 mm diameter borer (Fig. 3) utilized to cut samples
98 of meat. The meat sample was then placed into polypropylene tubes with a lid prior to
99 measurements. Each of the three replicates was measured 8 times with 1 hour interval between
100 measurements. The samples were kept at 4°C between measurements.

101 By using a Vector Network Analyzer (VNA) in a two port configuration, it is possible to measure
102 both the power reflected from the sample of matter as well as the power transmitted through the
103 sample. The reflected power is referred to as the S_{11} measurement, and the transmitted power as
104 the S_{21} measurement. Both the power reflected (S_{11}) from and transmitted (S_{21}) through the
105 sample were registered at the interval 2-6 GHz, which matched the operational range of the
106 cavity based upon its physical dimension (namely the aperture height and width). The cavity, and
107 the sample position, is illustrated in Fig.4. The same rectangular cavity that was designed and
108 used for measuring water holding capacity in raw meat (Abdullah et al., 2014) was used in this
109 study.

110

111 **2.4 Statistical analysis**

112 The data was statistically processed by one-way ANOVA and Tukey's multiple comparisons test
113 at P-value < 0.05 (R Foundation for statistical Computing, version 2.15.2). For statistical analyses
114 on water activity, the mean values for all three replicates from each dry-cured meat sample were
115 used.

116

117 **3. Results and Discussion**

118 **3.1 Chemical analysis**

119 Tables 1 and 2 show the mean and standard deviation for chemical analyses of dry-cured ham, for
120 high and low salt groups respectively, divided into different groups according to weight losses of
121 the dry-cured meat samples. The weight loss was calculated from the initial weight of the meat
122 samples. The water activity presented in the Tables is the mean value for all three replicates from
123 each sample of dry-cured ham. The results show that as the weight loss increases, the water
124 activity and the water content decrease. In addition, the salt content increases with higher weight
125 loss. These results are in agreement with previous knowledge (Andrés et al., 2008; Fellows,
126 2000). For the low salt group, the water content for the 40 % weight loss group is slightly higher
127 than for the 35 % weight loss group. This might be explained by low differences in final weight
128 loss between these groups, in addition to high variation within the 40 % weight loss group. For
129 all weight loss groups, both within the high and low salt group, the salt content is slightly higher
130 than expected. This might be explained by differences in the final water content for the dry-cured
131 meat samples, given that the initial water content in the meat samples was more or less equal.

132

133 **3.2 Microwave spectroscopy**

134 **3.2.1 Water activity**

135 The S_{11} spectra (reflection) and S_{21} spectra (transmission) were analysed, and a correlation
136 between water activity (from chemical analyses) and the spectra was observed at a number of
137 different frequencies. The results presented below show the spectra at 4.63-4.65 GHz for S_{11}
138 (Fig.5), and at 4.85-4.87 GHz for S_{21} (Fig.6). These frequencies were chosen since they gave high
139 correlation for both high and low salt groups. Fig.5 and Fig.6 shows that with decreasing
140 amplitude on the S_{11} and S_{21} spectra's, the water activity in the dry-cured meat samples also
141 decreases. This applies for both high and low (results not shown) salt groups. The average of all
142 five weight loss groups is shown. Given that the water activity decreases with higher weight loss,
143 the amplitude also decreases with increased weight loss of the dry-cured meat samples.

144 The correlation between the amplitude at 4.63-4.65 GHz and water activity was 0.95 and 0.98 for
145 high and low salt groups, respectively. The correlation plot for the high salt group is shown in
146 Fig.5 (b). For the low salt group, there was a partial overlap between the spectra's for the 20 %
147 and 25 % weight loss groups. This might be explained by very small differences in the average
148 values for water activity for those groups (Table 2).

149 The correlation between the amplitude at 4.85-4.87 GHz and water activity was 0.96 and 0.97 for
150 high and low salt groups, respectively. The correlation for the high salt group is shown in Fig.6
151 (b). As for the S_{11} spectra, a partial overlap for the low salt group was also found between the 20
152 % and 25 % weight loss groups on the S_{21} spectra.

153 The results above indicate that it is possible to separate samples with differences in water activity
154 levels by using microwave spectroscopy. This applies even though there are only minor
155 differences in water activity level between some of the weight loss groups, and for both S_{11} and
156 S_{21} spectra.

157

158 **3.2.2 Salt content**

159 As for the water activity, the S_{11} spectra and S_{21} spectra were analysed in order to find possible
160 correlations with salt content (from chemical analyses). However, high correlations between salt
161 content and spectra were only observed for S_{21} . The results presented below show the spectra at
162 4.24-4.28 GHz for S_{21} (Fig.7). This frequency was chosen since it resulted in the highest
163 correlation for both high and low salt groups. Only the results for the high salt group, average of
164 all five weight loss groups, is shown. Fig.7 a) shows that with decreasing amplitude the salt
165 content in the dry-cured meat samples from the high salt group increases. The same trend was
166 observed for the low salt group; however, the 20 % weight loss group seemed to be an outlier
167 which did not follow the trend. Given that the salt content increases with higher weight loss, the
168 amplitude also decreases with increased weight loss of the dry-cured meat samples.

169 The correlation between the amplitude at 4.24-4.28 GHz and salt content was 0.97 and 0.76 for
170 high and low salt groups, respectively. Figure 7 (b) shows the correlation for the high salt group.
171 As mentioned above, the 20 % weight loss group might be an outlier and therefore the correlation
172 for the low salt group is reduced.

173 In general, the results presented above indicate that it is possible to separate samples with
174 different salt content levels by using microwave spectroscopy. Here, the S_{21} spectrum seems to be
175 more promising for differentiating between samples with different salt content than the S_{11}
176 spectra.

177

178 **3.2.3 Water content**

179 As for the water activity and salt content, both S_{11} spectra (reflection) and S_{21} spectra
180 (transmission) were analysed, and a correlation between water content (from chemical analyses)
181 and the spectra's were observed at a number of different frequencies. The results presented below
182 show the spectra at 4.89-4.93 GHz for S_{11} (Fig.8), and at 5.08-5.09 GHz for S_{21} (Fig.9).
183 Frequencies giving high correlation for both low and high salt groups are presented. However,
184 only results for the high salt group are shown. For both the S_{11} and S_{21} spectra, the results shows
185 that with decreasing amplitude, the water content in the dry-cured meat samples also decreases.
186 This applies for both high and low salt groups, where the average of all five weight loss groups
187 was used.

188 The correlation between the amplitude at 4.89-4.93 GHz and water content was 0.94 and 0.97 for
189 high and low salt groups, respectively. The correlation for the high salt group is shown in Fig. 8
190 (b).

191 The correlation between the amplitude at 5.08-5.09 GHz and water content was 0.94 and 0.98 for
192 high and low salt groups, respectively. In fig. 9 (b) the correlation plot for the high salt group is
193 shown. A partial overlap for the low salt group was found between the 35 % and 40 % weight
194 loss groups on the S_{21} spectra. This might be explained by very small differences in measured
195 water content for those groups, in addition to lower water content than expected for the 35 %
196 weight loss group (Table 2).

197 As for the water activity and salt content, the results indicate that it is possible to separate
198 samples with different water content by using microwave spectroscopy. This applies even though
199 the differences in water content are small between some of the weight loss groups, and for both
200 S_{11} and S_{21} spectra.

201

202 **4. Conclusion**

203 A shift in amplitude for the S_{11} (reflection) and the S_{21} (transmission) signal was observed
204 between different weight loss groups at given frequencies. This shift was related to results from
205 chemical analyses of water activity, salt and water content. The correlation with the microwave
206 measurements was ranging from 0.76 to 0.98, depending on type of parameters measured and
207 spectra analysed. The results presented here indicate that microwave measurements might be a
208 promising technique for determination of different quality parameters for the process control of

209 dry-cured hams. Further studies should include investigation of microwave measurements for
210 other types of dry-cured meat.

211

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217

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