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

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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.

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

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## 1. Introduction

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

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

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

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

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

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

## 2. Materials and Methods

### 2.1 Sample preparation

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

### 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).

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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.

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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.

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117   **3. Results and Discussion**

### 3.1 Chemical analysis

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

### 3.2 Microwave spectroscopy

### 3.2.1 Water activity

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

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

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**3.2.2 Salt content**

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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.

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**3.2.3 Water content**

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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.

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## 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

212 **Acknowledgement**

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