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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### 26 1. Introduction

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

Being able to measure different quality parameters during the dry-curing process of ham is also 35 36 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 37 and availability of "free" water in a sample, which is given in values ranging between 0 (absolute 38 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 activity above 0.99, the water activity for dried meat products is lower, between 0.92 and 0.80. It 41 is the availability of water for microbial, enzymatic or chemical activity that determines the shelf 42 life of food; with reduced water activity the shelf life and safety of meat products improves 43 (Andrés, Barat, Grau, & Fito, 2008). 44

Another parameter that is important for controlling the production and quality of dry-cured meat 45 is salt content. While consumers demand lower salt content in dry-cured meat products, the salt 46 47 content is an important factor for preventing bacterial contamination. The salt content in a drycured ham evens out during the process. However, some parts might have higher salt content than 48 49 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 50 for measuring water activity, water and salt content in dry-cured meat products today are usually 51 destructive, in addition to being time consuming. 52

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 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.

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.

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

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## 86 2.2 Chemical analysis

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

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# 94 **2.3 Microwave spectroscopy**

Preparation of samples for microwave analysis was performed as shown in Fig. 2. A slice of approximately 20 mm thickness was taken from the middle of each dry-cured meat sample. From this slice, three replicates were taken with a 25 mm diameter borer (Fig. 3) utilized to cut samples of meat. The meat sample was then placed into polypropylene tubes with a lid prior to measurements. Each of the three replicates was measured 8 times with 1 hour interval between 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 both the power reflected from the sample of matter as well as the power transmitted through the 102 sample. The reflected power is referred to as the  $S_{11}$  measurement, and the transmitted power as 103 the  $S_{21}$  measurement. Both the power reflected ( $S_{11}$ ) from and transmitted ( $S_{21}$ ) through the 104 sample were registered at the interval 2-6 GHz, which matched the operational range of the 105 cavity based upon its physical dimension (namely the aperture height and width). The cavity, and 106 the sample position, is illustrated in Fig.4. The same rectangular cavity that was designed and 107 108 used for measuring water holding capacity in raw meat (Abdullah et al., 2014) was used in this study. 109

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#### 111 **2.4 Statistical analysis**

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

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#### 117 **3. Results and Discussion**

#### 118 **3.1 Chemical analysis**

Tables 1 and 2 show the mean and standard deviation for chemical analyses of dry-cured ham, for 119 120 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 121 samples. The water activity presented in the Tables is the mean value for all three replicates from 122 each sample of dry-cured ham. The results show that as the weight loss increases, the water 123 124 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, 125 126 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 127 128 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 129 than expected. This might be explained by differences in the final water content for the dry-cured 130 meat samples, given that the initial water content in the meat samples was more or less equal. 131

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#### 133 **3.2 Microwave spectroscopy**

### 134 **3.2.1 Water activity**

The  $S_{11}$  spectra (reflection) and  $S_{21}$  spectra (transmission) were analysed, and a correlation 135 between water activity (from chemical analyses) and the spectra was observed at a number of 136 different frequencies. The results presented below show the spectra at 4.63-4.65 GHz for  $S_{11}$ 137 (Fig.5), and at 4.85-4.87 GHz for  $S_{21}$  (Fig.6). These frequencies were chosen since they gave high 138 correlation for both high and low salt groups. Fig.5 and Fig.6 shows that with decreasing 139 amplitude on the  $S_{11}$  and  $S_{21}$  spectra's, the water activity in the dry-cured meat samples also 140 141 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, 142 the amplitude also decreases with increased weight loss of the dry-cured meat samples. 143

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

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

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## 158 **3.2.2 Salt content**

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

The correlation between the amplitude at 4.24-4.28 GHz and salt content was 0.97 and 0.76 for high and low salt groups, respectively. Figure 7 (b) shows the correlation for the high salt group.

As mentioned above, the 20 % weight loss group might be an outlier and therefore the correlation

172 for the low salt group is reduced.

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

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## 178 **3.2.3 Water content**

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

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

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

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

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#### 202 **4. Conclusion**

A shift in amplitude for the  $S_{11}$  (reflection) and the  $S_{21}$  (transmission) signal was observed between different weight loss groups at given frequencies. This shift was related to results from chemical analyses of water activity, salt and water content. The correlation with the microwave measurements was ranging from 0.76 to 0.98, depending on type of parameters measured and spectra analysed. The results presented here indicate that microwave measurements might be a promising technique for determination of different quality parameters for the process control of dry-cured hams. Further studies should include investigation of microwave measurements forother types of dry-cured meat.

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