

2018

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Yash Dixit

Technological University Dublin, yash.dixit@mydit.ie

Patrick Cullen

Technological University Dublin

Carl Sullivan

Technological University Dublin, carl.sullivan@tudublin.ie

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Recommended Citation

Dixit, Y., Cullen, P.J. & Sullivan, C. (2018). Introduction to laser induced breakdown spectroscopy imaging in food: salt diffusion in meat. *Journal of Food Engineering*, vol. 216, pp. 120-124. doi: 10.1016?j.jfoodeng.2017.08.010.

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1 **Introduction to laser induced breakdown spectroscopy imaging in food: salt diffusion in meat**

2 **Y. Dixit^{a, *}, Maria P. Casado-Gavalda ^a, R. Cama-Moncunill^a, X. Cama-Moncunill^a, Maria**
3 **Markiewicz-Keszycka ^a, Franklyn Jacoby ^b, P. J. Cullen ^{a,c}, Carl Sullivan^a**

4 ^aSchool of Food Science and Environmental Health, Dublin Institute of Technology, Dublin 1, Ireland.

5 ^bSchool of Culinary Arts and Food Technology, Dublin Institute of Technology, Dublin 1, Ireland.

6 ^cDepartment of Chemical and Environmental Engineering, University of Nottingham, UK

7 * Corresponding author:

8 Y. Dixit

9 School of Food Science and Environmental Health,

10

11 Dublin Institute of Technology, Cathal Brugha St, Dublin 1, Ireland

12

13

14 Tel: +353 1 402 4543

15 E-mail: yash.dixit@mydit.ie

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19 **Keywords:** LIBS, beef, brine, imaging, spatial.

20

21 **Abstract.**

22 This study illustrates the ability of laser induced breakdown spectroscopy (LIBS) to detect and map
23 minerals in food. A LIBS system was used to spatially collect spectra of beef samples. Samples were
24 brined in a 6% salt solution for 2h and 24h along with a control sample. Samples were measured by
25 scanning the cross-section of each sample in a 90 X 90 square grid. Sodium (Na) distribution images
26 with respect to emission peak at 589.05 nm were generated after pre-processing the spectral data which
27 directly corresponds to salt levels. As expected, the control sample showed the lowest Na distribution
28 whereas 2h brined sample showed distribution along the sample's edges decreasing towards the centre.
29 The 24 h brined sample showed increased diffusion. Overall, results show the ability of LIBS to map
30 salt diffusion in meat via Na LIBS imaging, which could be used to optimize brining conditions.

31

32

33 1. Introduction

34 Salt curing is an ancient technique which has been used over centuries for preserving perishable food
35 products such as meat. Salt curing can be of the following types: dry, wet and a combination of both.
36 Sodium chloride (NaCl) is the main ingredient used in meat curing. It offers various functionalities such
37 as preservation, improved technological yields, and influences meat tissue properties such as water-
38 holding capacity and protein solubilisation, which in turn defines meat texture (Sharedeh et al., 2015).
39 NaCl diffusion in meat during wet (immersion) curing, also known as brining, is defined by Fick's
40 diffusion theory. **The theory of diffusion in isotropic substances is based on the hypothesis that the rate**
41 **of transfer of diffusing substance through unit area of a section is proportional to the concentration**
42 **gradient measured normal to the section** (Crank, 1979). NaCl diffusion into the muscles is usually rapid
43 and an equilibrium is reached in about 48 h. However, salt diffusion is slower in meats with a close
44 tissue micro-structure when immersed in a weak brine solution (Lawrie, 2006). It is important to study
45 salt diffusion in meat in order to optimize brining time, brine concentration as well brine temperature,
46 which can have a direct effect on the microbiological, physico-chemical and sensorial characteristics of
47 meat.

48 Laser-induced breakdown spectroscopy (LIBS) is an emerging elemental technique for mineral
49 analysis of food (Andersen et al., 2016; Bilge et al., 2016b; Cama-Moncunill et al., 2017; Casado-
50 Gavalda et al., 2017; Dixit et al., 2017b). LIBS provides numerous advantages such as minimal or no
51 sample preparation, chemical free, rapid detection, portability and spatial information (Abdel-Salam et
52 al., 2017; Bilge et al., 2016a; Er et al., 2016; Moncayo et al., 2016; Singh et al., 2017; Wang et al.,
53 2016).

54 Various studies have been conducted regarding NaCl diffusion in meat during immersion-brining
55 with or without other additives (Graiver et al., 2006, 2009; Hansen et al., 2008). However, the literature
56 reveals no study has been reported using LIBS for analysing salt diffusion in meat. In the current study,
57 an experiment was conducted for imaging salt diffusion in beef using a LIBS system combined with an
58 automatic sample chamber. Chlorine emission peaks are not easily resolvable under normal atmospheric
59 conditions due to interference with nitrogen (N) and oxygen (O) peaks, thus emission peaks related to
60 sodium (Na) were utilized to create salt diffusion images (Weritz et al., 2007). The aim of this study was
61 to illustrate the ability of LIBS as a novel chemical free technique for imaging NaCl diffusion in beef.

62

63 2. Materials and methods

64 2.1 Sample Preparation

65 Fresh round beef steak weighing approximately 1.5 Kg was purchased from a local butchers shop
66 in Dublin city and transferred to the School of Food Science and Environmental Health in the Dublin
67 Institute of Technology, Dublin, Ireland. On the same day, the steak was carefully cut in order to
68 separate the lean from the fat beef trimmings. The lean beef was cut into three cubes of approximately 4
69 cm diameter. A 6 % salt brine solution was prepared using laboratory grade NaCl (cas no: 7647-14-5,
70 >99.5 %, Sigma Aldrich, Inc.). Three beakers of volume 250 ml were used. One beaker was filled with
71 distilled water while the remaining were filled with a 6 % brine solution. The beef cubes were separately
72 immersed in beakers and were placed on a clean plastic stand to ensure a uniform diffusion from all
73 sides. Beakers were covered with aluminium foil to minimize evaporation. The sample immersed in
74 distilled water was used as a control (0 hrs). The samples in the brine solution were allowed to stand for
75 2 hrs and 24 hrs respectively. Firstly, the control sample was removed from the distilled water solution,
76 dried from all sides, using ashless paper, in order to remove any excess of liquid solution and then
77 horizontally cut to reveal the cross-section. The obtained cross-section was then subjected to LIBS
78 analysis. A similar procedure was conducted for cross-sections of the 2 hr and 24 hr brined samples.

79 2.2 LIBS spectra acquisition

80 LIBS spectra were recorded with a LIBSCAN 150 system (Applied Photonics Limited, Skipton
81 North Yorkshire, United Kingdom) used in the study by Dixit et al. (2017a). The System consists of a
82 Q-switched Nd:YAG laser (ultra, Quantel laser, 601 Haggerty Lane Bozeman, MT, USA) and a series
83 of six spectrophotometers covering the wavelength range of 185-904 nm. The head incorporates a
84 miniature CCD camera and 6 lens holders which collect plasma light of different wavelength regions.
85 The laser used for sample ablation had a pulse energy of 150 mJ and a pulse duration of 5 ns operating
86 at 1064 nm. A repetition rate of 1 Hz was employed along with a 1.27 μ s gate delay and 1.1 ms
87 integration time in Q-switched mode. The spectrograph was externally triggered from the laser at every
88 pulse with a delay generator. The sample was placed at a LTSD (lens to sample distance) of
89 approximately 80 mm to ensure that the laser was focussed onto the sample. The control sample (0 hr)
90 and 2 hr brined sample were analysed on the same day of sample preparation while the 24 hr brined
91 sample was analysed on the subsequent day. Samples were measured by applying shots in a whole
92 cross-section in a 90 X 90 square grid pattern while moving the sample after each shot by an automated
93 sample chamber (XYZ-750, Applied Photonics Limited, Skipton North Yorkshire, United Kingdom)

94 with a step size of 0.50 mm. The number of shots were selected to ensure complete coverage of the
95 sample cross-section.

96 2.3 Data analysis

97 Data analysis was performed using R (R Core Team, 2014). The packages *baseline* and *EBImage* were
98 utilized to perform spectral pre-processing and image processing respectively.

99 Initially, the spectra recorded for each sample at 90 X 90 different locations were pre-processed
100 in order to remove non-linearities introduced by light scattering, which can have a considerable effect
101 on the spectra (Rinnan et al., 2009). Pre-processing was performed using the *baseline* function in order
102 to remove background effects from the acquired spectra. The processed data obtained for the three
103 samples were transformed into individual hypercubes (Dixit et al., 2014; Gowen, 2014), where x-axis
104 and y-axis represent the coordinates (location) of the laser shots and z-axis represents the wavelength
105 range used. Hypercubes were further processed using the package *EBImage* for performing
106 morphological operations by utilizing functions “*makeBrush*” and “*filter2*” in order to obtain
107 smoothed images. Images were generated with respect to main elemental peaks using a false colour
108 scheme indicating emission intensity. Images of the emission intensity distribution for potassium (K) at
109 766.458 nm showed the maximum contrast between the background and the meat samples (Fig. 1) and
110 hence, the K emission peak at 766.458 nm was used to obtain a threshold for distinguishing the meat
111 samples from the background. It can be seen from Figure 2, this threshold performed well. It is evident
112 from Figure 1 that a correlation exists between Na (589.05 nm) and K (766.458 nm) distribution which
113 could be related to Na-K pump (Skou, 1988) and hence was utilized to normalize the spectral data. In a
114 next step, masking was performed followed by normalization using the potassium (K) emission peak at
115 766.458 nm, where the baseline corrected data of the Na emission peak (589.05 nm) intensities at each
116 shot/location were divided by the corresponding emission intensities of the K (766.458 nm) peak at the
117 same shot/location. Normalization was used in order to compensate for signal variations and sample
118 matrix differences (Castro and Pereira-Filho, 2016). Finally, salt (with regard to Na) distribution images
119 were generated. In order to compare the samples, the same intensity scale was implemented for the three
120 samples. Maximum and minimum intensities of Na emission peak (589.05 nm) for 24h brined sample
121 were used for rescaling.

122 3. Results and Discussion

123 3.1 Image analysis

124 Fig. 3 shows the Na distribution for the cross-section of the control, 2h brined and 24h brined samples
125 with respect to the Na peak at 589.05 nm which directly represents the salt distribution. The colour scale
126 represents the normalized intensity of Na at various locations of the meat cross section. Fig. 3 (a)
127 illustrates the cross-section image of the control sample which clearly shows the emission intensities of
128 salt (Na) equally distributed throughout the sample at the lowest level of the intensity colour scale. Fig.
129 3 (b) illustrates the cross-sectional image of the 2h brined sample, where higher intensities of Na were
130 evident along the edges, which decreases towards the centre illustrating salt diffusion from all sides.
131 Lower Na intensities were observed at the right edge of the sample as compared to other sides which
132 could be related to non-uniformity of the sample, subsequently affecting the diffusion process. As
133 expected, salt diffusion increased with brining time of 24 h which is evident from Fig. 3 (c). The
134 variability in Na intensities along the edges could be caused by the same reason as for 2h brined sample.
135 Ideally, the distribution of salt should be uniform from all sides; however factors such as sample
136 geometry, sample size, brine concentration and brine temperature plays a significant role in defining the
137 diffusion process (Chabbouh et al., 2012). Also factors affecting the LIBS spectra such as LTSD,
138 sample uniformity and matrix cannot be neglected (Markiewicz-Keszycka et al., 2017; Radziemski and
139 Cremers, 2006). Overall, it can be concluded that LIBS can be a rapid and chemical-free technique to
140 image salt diffusion as well different minerals in various meats.

141 For future studies, factors affecting the LIBS spectra as well as salt diffusion for various meats
142 should be studied. Reference values for salt or Na should be obtained in order to correlate the actual
143 concentration values with spectral intensities.

144 4. Conclusions

145 This study illustrates the ability of LIBS as a novel spectral technique for imaging NaCl diffusion in
146 beef by utilizing the Na emission peak at 589.05 nm in LIBS spectra. Spectral images of the cross-
147 section of brined beef samples (2h and 24 h) along with a control sample were generated with respect to
148 the Na emission peak at 589.05 nm. As expected, the control sample showed the lowest salt (Na)
149 distribution whereas 2h brined sample showed Na distribution along the sample edges which decreased
150 towards the centre illustrating salt diffusion. Salt diffusion increased for the 24 h brined sample,
151 indicated by an increased gradient of Na intensity towards the centre. Variability in the Na intensities
152 along the edges was observed which could be related to non-uniformity of the sample, subsequently

153 affecting the diffusion process. Overall, results illustrate the ability of LIBS combined with an
154 automated chamber to map salt diffusion in meat via Na LIBS imaging. The findings presented here
155 show the potential for LIBS in optimizing brining time, brine concentration as well as brine temperature.
156

157 **5. Acknowledgements**

158 The authors would like to acknowledge funding from the Food Institutional Research Measure
159 administered by the Department of Agriculture, Food and the Marine, Ireland (Grant agreement:
160 13/F/508).

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