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

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Abstract.

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

1. Introduction

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

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

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

2. Materials and methods

2.1 Sample Preparation

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

2.2 LIBS spectra acquisition

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

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

2.3 Data analysis

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

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

3. Results and Discussion

3.1 Image analysis

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

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

4. Conclusions

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

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

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