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Exploration of microwave dielectric and near infrared spectroscopy with multivariate data analysis for fat content determination in ground beef

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ABSTRACT

This study investigated using microwave dielectric and near infrared (NIR) spectroscopy for the determination of fat content in ground beef samples (n = 69) in a designed experiment. Multivariate data analysis (principal component analysis (PCA) and partial least squares (PLS) regression modelling) was used to explore the potential of these spectroscopic techniques over selected multiple frequency or wavelength ranges. Chemical reference data for fat and water content in ground beef were obtained using a nuclear magnetic resonance-based SMART Trac analyser. Best performance of PLS prediction models for fat content revealed a coefficient of determination in prediction (R^2P) of 0.87 and a root mean square error of prediction (RMSEP) of 2.71% w/w for microwave spectroscopy; in a similar manner, R^2P of 0.99 and RMSEP of 0.71% w/w were obtained for NIR spectroscopy. The influence of water content on fat content prediction by microwave spectroscopy was investigated by comparing the prediction performance of PLS regression models developed using a single Y-variable (PLS1; fat or water content) and using both Y-variables (PLS2; fat and water contents).

1. Introduction

In recent years, spectroscopy has become a popular technology for food quality assessment due to its rapid data acquisition and ability to simultaneously predict multiple quality parameters with only minimal sample preparation. This technique has shown potential in quality control applications and determination of proximate composition of meat and meat products (Damez & Clerjon, 2013). Spectroscopy uses electromagnetic radiation to induce and measure vibrational or rotational energy transitions at molecular and atomic levels respectively. Based on the respective motions of quantum particles in the applied electromagnetic fields, this technique has been categorised into vibrational and rotational spectroscopic methods (Hollas, 2004).

Microwave-based dielectric spectroscopy is a rotational spectroscopic method which has been used to analyse qualitative characteristics of meat and other food materials since 1983 (Metaxas & Meredith, 1993). Theoretically, in a microwave-excited electromagnetic field, microwave radiation can easily penetrate food materials to induce particle polarisation in different components, giving rise to the characteristic dielectric properties of materials (Metaxas & Meredith, 1993). The dielectric property of a food is described as the complex permittivity which arises when it is subjected to an electromagnetic field in a microwave heating application. Complex permittivity (ε) is defined as:

$$\varepsilon = \varepsilon' - j\varepsilon'',$$

where ε' is the real part, called the dielectric constant, which represents the capacity of the material to store energy (Nelson & Datta, 2001); ε'' is the imaginary part referred to as the loss factor which is relevant to high frequency heating generated from electrical energy and includes the effects of conductivity-it also can be described as the ability of the material to convert energy into heat; j is the imaginary unit ($j^2 = -1$). The ratio of $\varepsilon''/\varepsilon'$ is known as the loss tangent (Nelson & Trabelsi, 2012). As individual parameters, ε' or ε'' can be used to represent dielectric properties of a material while both of them are frequency- and temperature-dependent (Metaxas & Meredith, 1993).

The potential of dielectric spectroscopy to detect content, state

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and activity of water together with detection of added water in meat products has previously been reported (Castro-Giráldez, Botella, Toldrá, & Fito, 2010; Clerjon, Daudin, & Damez, 2003; Kent & Anderson, 1996). Microwave measurements in reflection or transmission modes were also used to predict beef aging and fish freshness (Clerjon & Damez, 2007), meat quality in poultry and pork (Castro-Giráldez et al., 2010; Samuel, Trabelsi, Karnuah, Anthony, & Aggrey, 2012) and a wide range of fat content (up to 50% w/w) in minced beef (Gunasekaran, Mallikarjunan, Eifert, & Sumner, 2005; Ng et al., 2008). However, most studies have focused on profiling the dielectric properties of different materials over a specific frequency range (e.g. 0.3–3 GHz, 0–25 GHz, 0.2–12 GHz, 0.5–20 GHz) (Castro-Giráldez et al., 2010; Gunasekaran et al., 2005; Kent & Anderson, 1996; Ng et al., 2008) under different temperature (Gunasekaran et al., 2005; Ng et al., 2008) or during the aging process (Castro-Giráldez et al., 2010; Kent & Anderson, 1996). Some previous studies have explored chemometric analysis such as principal component analysis (PCA) and partial least squares regression (PLSR) for the prediction of moisture in poultry meat, scallops and pork but only based on each single selected frequency (Kent & Anderson, 1996; Kent, Peymann, Gabriel, & Knight, 2002). There have been no studies which explored the use of different microwave frequency ranges for beef quality prediction using multivariate data analysis.

As a mature vibrational spectroscopic technique, near infrared (NIR) spectroscopy combined with multivariate data analysis has been widely used in meat analysis and implemented to provide timely measurements in on-, in- and at-line applications (Weeranantanaphan, Downey, Allen, & Sun, 2011). NIR and visible light in transmission mode were used to evaluate beef quality through detection and quantification of marbling (Ziadi, Maldague, Saucier, Duchesne, & Gosselin, 2012). NIR transmittance spectroscopy was also used to measure the content of fat and other compositional parameters in fish (Nortvedt, Torrissen, & Tuene, 1998; Solberg & Frendriksen, 2001; Wold & Isaksson, 1997; Xiccato, Trocino, Tulli, & Tibaldi, 2004). NIR reflectance spectroscopy has been used for identification of meat species in homogenised meat muscle (Cozzolino & Murray, 2004); quality control on meat trimmings (Wold, Sjöström, & Eriksson, 2001) and to detect adulterations in minced beef and beefburgers (Morsy & Sun, 2013; Zhao, Downey & O'Donnell, 2013).

Commercial ground beef is derived from skeletal muscle, including adherent fatty tissues and added beef fat trimmings. The European Community has issued a regulation for the quality grades of typical ground beef (EU Regulation 853/2004) which are referred to as Class-I 'lean mince beef' and Class-II 'minced pure beef'. Based on the compositional criteria of fat content, lean mince beef should contain< 7% w/w fat while minced pure beef should contain <20% w/w fat (FSAI, 2013). The fat content of ground beef has always been a critical issue with respect to accurate nutritional labelling, fraudulent industrial practice (e.g. addition of extra fat) and consumer protection. Commercial ground beef contains both intramuscular and additional fat. When beef is comminuted, all gross physical characteristics of the meat are lost; therefore, timely processing analytical technology is required to assist quality control and inspection. Comparing the mechanics of microwave and NIR spectroscopy, microwaves can obtain useful data from bulky and non-homogenised samples by significant penetration into the material while NIR radiation may only provide information about the surface of the material because of its shallow penetration depth. NIR is thus problematic for measurements of non-homogenised opaque solids. Among disadvantages of the microwave technique is the fact that the dielectric properties of moisture in samples may dominate or influence the dielectric spectral signals in certain frequency ranges (Clerjon & Damez, 2009; Kent & Anderson, 1996), as water has significantly higher permittivity than other organic compounds (Gunasekaran et al., 2005).

The current study investigated the determination of fat content in ground beef using a laboratory-based dielectric measurement system and compared the results to those obtained on the same samples using a NIR spectroscopic method. Multivariate data analysis was utilised to develop and validate prediction models for ground beef fat and moisture contents. Performance of microwave and NIR prediction models was compared to evaluate the relative merits of each technique for use in ground beef quality control. This study is innovative and novel for the following reasons: 1) this study involved a greater number of ground beef samples with more variables than previous research; 2) the determination of fat content in ground beef samples has been explored using multivariate data analysis using different dielectric frequency ranges, and 3) a chemometric method using PLS-1 and PLS-2 modelling has been introduced to microwave spectroscopy to examine the potential influence of water content on the determination of fat content in ground beef.

2. Methods and materials

2.1. Sample preparation

Ground beef samples (n = 69) were produced in the abattoir of Teagasc Food Research Centre (Ashtown, Dublin 15, Ireland). These samples were produced to include both a higher and a lower quality level in beef. In the higher quality group, round steak beef cuts were trimmed to remove visible fat; beef fat trimmings were added to aliquots of lean beef to produce samples (0.5 kg each) with 0%, 3%, 5% and 7% w/w of added fat content. In the lower quality group, plate or flank cuts were trimmed of visible fat but connective tissue and tendons were left. Lower quality samples (0.5 kg each) were produced using these trimmed cuts with added beef fat trimmings at 5%–30% w/w in 2.5% w/w increments. Samples at each added fat level were produced on three separate occasions using meat sourced from three different butcher shops. In all, 36 higher quality samples and 33 lower quality samples (primary samples) were produced using raw materials from 9 local butchers. During preparation, each sample was ground twice using a Mainca meat mincer (PM70/12; Cheshire, UK) followed by homogenisation for 1 min using the Robot coupe R301 ultra (Vincenes, France). Homogenised samples were transferred into lidded plastic containers and stored at +3 °C. Before measurement, samples were allowed to equilibrate to ambient laboratory temperature (20 ± 2 °C).

2.2. Sample measurements

2.2.1. Measurements dielectric

A time-domain reflectometry dielectric system was used in this study. Dielectric spectra were derived from the reflected signals in an electro-magnetic field. Dielectric properties (i.e. dielectric constant (ε'), dielectric loss factor (ε'') and loss tangent ($\varepsilon''/\varepsilon'$)) were recorded over the 200 MHz to 20 GHz frequency range using an Agilent 85070E high temperature coaxial probe kit connected to an impedance analyser (E4991A; Agilent Technologies, Santa Clara, USA). This probe featured a hermetic glass-to-stainless steel 19 mm diameter seal and a 3.5 mm aperture; this configuration requires homogeneous samples with a flat, gas-free surface to ensure contact at the probe face during measurement and a certain minimum sample thickness, i.e., $> \sqrt{|\varepsilon|}$mm (Keysight Technologies, 2014). A probe stand was used to fix the probe and the connected coaxial cable in a constant position as even subtle changes in instrument configuration may affect calibration; the sample, contained in a