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Determination of C22:5 and C22:6 marine fatty acids in pork fat with Fourier transform mid-infrared spectroscopy

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Abstract

Fatty acids in samples (n = 74) of pork adipose tissue were measured with a Fourier transform mid-infrared (FT-MIR) spectrometer and by gas chromatography. The measured absorption spectra provided information to estimate partial least squares regression models for fatty acid groups, the iodine value and several fatty acids. The iodine values were predicted with correlation coefficient R = 0.996 and root mean square error of cross-validation RMSECV = 0.658. The sum of the two marine fatty acids of main interest, C22:5n3 + C22:6n3, were predicted with R = 0.982 and RMSECV = 0.062. The K nearest neighbours procedure successfully classified the samples in three classes, depending on their proportions of marine fatty acids. Application of fat and absorption measurements were rapid, requiring less than 5 min of labour per sample. The results reported in this paper demonstrate that FT-MIR measurements can serve as a rapid method to determine marine fatty acids in pork fat.

Keywords: Fourier transform infrared spectroscopy; FTIR; MIR; Mid-infrared; Pork fat; Adipose tissue; Subcutaneous fat; Fatty acid composition; SFA; MUFA; PUFA; Marine fatty acids; Docosapentaenoic acid; Docosahexaenoic acid; Iodine value; PLS; Partial least squares; *K*NN; *K* nearest neighbours

1. Introduction

The quality of meat is influenced by its lipid content and fatty acid composition. Pork meat is more or less susceptible to oxidative deterioration, depending on the degree of lipid saturation. Polyunsaturated fatty acids (PUFA) benefit human health, but also increase susceptibility to lipid oxidation. Oxidation leads to negative effects on quality parameters such as flavour, colour, texture and nutritive value (Buckley, Morrissey, & Gray, 1995). The fatty acid composition of pork adipose tissue reflects the fatty acid content of the diet. Although saturated fatty acids (SFA) and monounsaturated fatty acids (MUFA) are synthesised in vivo, some polyunsaturated fatty acids in pigs, such as linoleic acid (C18:2, $n-6$) and α-linolenic acid (C18:3, $n-3$), mainly reflect diet composition (Enser, Richardson, Wood, Gill, & Sheard, 2000). Docosapentaenoic acid (DPA, C22:5n3) and docosahexaenoic acid (DHA, C22:6n3) originate from marine species. Several studies show that changes in fatty acid composition of pork adipose tissue, as well as sensory characteristics of the meat, reflect dietary alterations of these fatty acids (Bryhni, Kjos, Ofstad, & Hunt, 2002; Lauridsen et al., 1999; Overland, Taugbol, Haug, & Sundstol, 1996). Some countries with a substantial catch of marine species, produce fishmeal

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at competitive prices compared to other feeds, accompanied with an increased risk for fishmeal to affect the quality of pork meat.

Information about the fatty acid composition is useful to evaluate carcass quality and could determine further processing of carcass parts as well as the carcass value in payment systems. Shelf life prediction may also be more accurate with information about the fatty acid composition. A method for rapid characterisation and determination of fatty acid composition of pork fat is therefore of great interest.

Fourier transform infrared spectroscopy (FTIR) is a promising technique for measuring quality parameters in lipids. It has the advantage of easy sample preparation, rapid measurements and no use of chemicals in contrast to traditional solvent methods accompanied by chromatographic techniques. Lipid analyses of fatty acid composition usually involve organic solvents for fat extraction (AOAC, 1990). Organic solvents have undesirable effects due to their carcinogenic properties and harmful effect on the environment.

Mid-infrared (MIR) spectroscopy, using wavelengths from 4000 to 400 cm^{-1}, is frequently used for spectroscopic studies of organic compounds. Although edible fats and oils consist mainly of triglycerides, and their MIR spectra seem to be visually similar, differences in absorption intensities and exact frequency associated with maximum absorption are present (Guillèn & Cabo, 1997b). Relationships between lipid composition and position of the absorption bands in this region of the electromagnetic spectrum have been found, as well as their assignment to functional groups, vibration modes and intensities (Guillèn & Cabo, 1997a; Guillèn & Cabo, 1998).

FT-MIR transmission spectroscopy has been widely used for the study of lipids. Supported by multivariate statistical methods, it is an important method for the development of rapid lipid analysis. The content of *trans*-double bonds has been determined by this method (Sedman, van de Voort, & Ismail, 1997; Sedman, van de Voort, Ismail, & Maes, 1998), as well as free fatty acid content (Che Man, Moh, & van de Voort, 1999) and degree of unsaturation in terms of iodine value (IV) (Che Man, Setiowaty, & van de Voort, 1999; Hendl, Howell, Lowery, & Jones, 2001; Sedman et al., 1998). Several studies show the usefulness of FT-MIR transmission measurements to monitor the oxidation processes in fats and lipids. The spectroscopic technique has shown good correlations to classical chemical methods, such as peroxide value (PV) (Moh, Tang, Che Man, & Lai, 1999), anisidine value (AV) (Che Man & Setiowaty, 1999) and content of thiobarbituric acid reactive substances (TBARS) (Mirghani, Che Man, Jinap, Baharin, & Bakar, 2001). Guillèn and Cabo (1999, 2000) have found changes in FT-MIR spectra that reflected chemical changes in the different stages of lipid oxidation, and have proposed a method that directly utilizes FT-MIR spectra instead of the classical parameters such as PV and AV (Guillèn & Cabo, 2002).

Ripoche and Guillard (2001) have investigated the usefulness of FTIR spectroscopy to measure the fatty acid composition of pork fat, in terms of proportions of SFA, MUFA and PUFA, as well as certain fatty acids. However, C22:5*n*3 and C22:6*n*3 fatty acids were not included in the study. Measurements directly on fat slices by diffuse reflectance near infrared (NIR) spectroscopy gave low predictive ability. NIR transmission measurements on fat, extracted with organic solvents, gave better correlation coefficients. However, attenuated total reflectance (ATR) of extracted fat in the MIR region, gave the most promising prediction results (Ripoche & Guillard, 2001).

The purpose of this study was to investigate the possibility of determining the fatty acid composition in pork fat, using absorption spectra of FT-MIR transmission measurements in a multivariate statistical approach. Our aim was to determine the proportion of marine fatty acids (C22:5*n*3 + C22:6*n*3), as they may reduce shelf life and meat quality, and to classify the samples into different classes of marine fatty acid content. To our knowledge, no previous work has been reported on the feasibility of rapid determination of marine fatty acids in pork fat. We determined the degree of unsaturation (IV), and the general fatty acid composition, as proportions of SFA, MUFA and PUFA. In addition, we investigated whether fat extraction by melting and without use of organic solvents, could replace traditional solvent extraction.

2. Materials and methods

2.1. Sample selection

For this experiment, 74 samples were selected from a population of 725 samples already analysed by GC. Selection was done to achieve an evenly distributed and wide range of values in the parameters of interest.

2.2. Fat samples and preparation

Back fat without meat and rind was taken from necks of pig carcasses at the slaughterhouse. The samples were frozen and stored aerobically at −20 °C. The frozen samples were thawed, minced, placed in a tun-dish with filter paper, and melted at 70 °C for 15–30 min. Liquid fat was collected in a test tube below the tun-dish. About 20 mg was taken for gas chromatography (GC) analysis. Fat for spectroscopic analyses was stored aerobically at −20 °C. Before the FT-MIR spectroscopic analyses, the fat was thawed and stored at 4 °C for one to three days. Immediately before spectroscopic analyses the samples were melted at 50 °C.