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Original Research Article

Gas chromatography–mass spectrometry approach to study fatty acid profiles in fried potato crisps

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ABSTRACT

The fatty acid profiles of crisp samples from *Hermes* and *Mustang* potato varieties were obtained using a validated GC–MS method. Among the four different solid–liquid extraction procedures tested, Soxhlet extraction with diethyl ether was chosen due to the higher recovery percentages obtained (~90%) and the shorter sample treatment time required. The proposed method was applied to analyze 28 crisps samples from both potato varieties, which were fried with two batches of similar vegetable oils, and the majority fatty acids in potato crisps (~33.3 g/100 g) were C18:0, C16:0, C18:2(n6) and C18:1(n9). The results of a two-way ANOVA test have demonstrated that the potato variety caused more significant differences (11) than the frying oil batch (4) in relation to the fatty acid content. A PCA analysis has made it possible to relate the potato variety with the fatty acid content; it was observed that the first four principal components represented 85.5% of the variability, with a consequent reduction in the dimensions of the data from 36 variables to 4 components. Finally, it was found that the *Hermes* variety contained lower amounts of total fatty acids (~34.4 g/100 g) and ω-6/ω-3 ratios (~17.3) than the *Mustang* variety (~35.6 g/100 g and ~18.6).

1. Introduction

Potato crisps are a widely extended snack food, and nowadays their consumption is having a greater influence on the dietary habits of populations around the world. Therefore, an evaluation of their composition is essential, as they are foods with a high calorie intake, mainly due to their considerable fatty acid (FA) composition (Fernández-San Juan, 2000). The presence of FAs in the final product could be influenced by several factors, such as the manufacturing process, but this also depends on the frying oil type used or the potato variety, as will be discussed throughout this work.

Regarding the oil, many food manufacture companies are currently increasing their commitment to consumers' health by carrying out research into the quality of the oils used for crisp frying. In this regard, it must be pointed out that vegetable oils are the most used. Many types of vegetable oils can be used, and subsequently a large amount of different FAs can be added to the final product. But, at the same time, the variety of potato should also be considered, because during frying, the water present in the raw material evaporates and is partially replaced by oil; this constitutes up to 40% of the finished product and consequently influences its properties. This affects not only the flavor and aroma of the product, but also the texture, in accordance with the quantity of oil absorbed during frying (Kita et al., 2007). Therefore, the selection of the potato variety according to its own physical–chemical characteristics is related with the oil content and the subsequent final fatty acid content in the potato crisps. Concerning this issue, a study has recently been published (Ooko and Kabira, 2011) in which five different potato varieties were evaluated as raw materials for producing French fries and potato crisps in Kenya, and one of the parameters which, according to the authors, showed some effect on the oil content was the dry matter inherent in each potato variety. In the existing literature, several studies have been published where the effect of the frying oil (type or temperature) on the FA content in potato crisps was evaluated (Aro et al., 1998; Fernández-San Juan, 2000; Kita et al., 2007; Ooko and Kabira, 2011; Sanches-Silva et al., 2004; Wagner et al., 2008), but to our knowledge no study has been published which has also made a detailed analysis of the relation between the potato variety alone, or in combination with the frying oil, and the fatty acid profile. For this reason, it was decided to perform the study summarized in this manuscript.

Abbreviations: PCA, principal component analysis; PC, principal component; FA, fatty acid; FAME, fatty acid methyl esther; FID, flame ionization detector; SFE, supercritical fluid extraction; ASE, accelerated solvent extraction; ICH, International cooperation on harmonization.

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The analytical technique most often employed to study FAs is gas chromatography (GC), with flame ionization (FID) usually selected for the identification of total fatty acids (Fernández-San Juan, 2000; Kita et al., 2007; Lo Scalzo et al., 2007; Wagner et al., 2008); however, mass spectrometry (MS) detectors have been preferred to determine minority FAs (Hauff and Vetter, 2009; Jiménez et al., 2009; Toribio et al., 2011; Zu et al., 2009), and nowadays the use of multidimensional gas chromatography is gaining in importance in this field (Hejazi et al., 2009; Herrero et al., 2009; Manzano et al., 2011). As a particular case, a reversed-phase high performance liquid chromatographic (RP-HPLC) method with UV detection has also been used for FAs determination (Sanches-Silva et al., 2004). It must be pointed out that the conversion of FAs into more volatile compounds than free acid components, usually in their methyl esters derivatives (FAMEs) (Li and Watkins, 2001; Morrison and Smith, 1964), is essential prior to GC analysis. Consequently, the FAs derivatization procedure and the reagents employed in this process played a significant role when identifying and determining FAs. Several derivatization reagents have been used, KOH in methanol catalyzed with BF_3 being widely used (Hauff and Vetter, 2009; Jiménez et al., 2009; Kita et al., 2007; Li and Watkins, 2001; Lo Scalzo et al., 2007; Morrison and Smith, 1964; Toribio et al., 2011), due to the fact that BF_3 provoked rapid methylation of FAs, and that when they were freshly made and stored properly, BF_3 solutions could last nearly two years (Li and Watkins, 2001).

One of the main problems when determining FAs composition is the fat extraction step, as it depends to a great deal on the nature of the sample and the diversity of the FAs in terms of their chain length, branching, degree of unsaturation and the position and geometry of double bonds (Aro et al., 1998; Shahidi, 2001). So far, within the huge variety of extraction methods employed, the most commonly used were: solid–liquid extraction (Fernández-San Juan, 2000; Folch et al., 1957; Jiménez et al., 2009; Kita et al., 2007; Li and Watkins, 2001; Shahidi, 2001), acid digestion (Shahidi, 2001), supercritical fluid extraction (SFE) (Catchpole et al., 2009; Hauff and Vetter, 2009; Sahena et al., 2009; Toribio et al., 2011), accelerated solvent extraction (ASE) (Hauff and Vetter, 2009; Wagner et al., 2008) and microwave procedures (Zu et al., 2009). One of the most commonly used extraction methods was described by Folch et al. (1957). This has been modified several times (Bligh and Dyer, 1959; Fernández-San Juan, 2000; Li and Watkins, 2001; Shahidi, 2001), the best known version being performed by Bligh and Dyer (1959), but always based on the use of a solvent mixture (chloroform and methanol), to obtain the simultaneous extraction of neutral and polar lipids. Furthermore, Soxhlet extraction has also usually been employed in order to extract fats and oils in food matrices (using different ethers (Jiménez et al., 2009; Kita et al., 2007; Shahidi, 2001), together with other solvents like hexane (Pedneault et al., 2008).

To summarize, the main aim of this study was to analyze in detail the influence of the potato variety alone or combined with the effect of frying oil on the fatty acid content of commercial potato crisps, as this had not been done before. To achieve this aim and taking into account the existing scientific literature concerning the determination of FAs in food matrices and, more specifically, in potato derived products, it was decided to use GC–MS as an analytical separation tool. In order to extract as many FAs as possible from the potato crisp samples, four different solid–liquid extraction procedures were tested. Once the analytical method was optimized, it was validated and applied to analyze 28 potato crisps samples. Finally, and to evaluate the influential factors mentioned above, namely potato variety and frying oil, statistical tools such as two-way analysis of variance (ANOVA) and principal component analysis (PCA) were used.

2. Materials and methods

2.1. Chemicals and standard solutions

A standard mixture of FAMEs in dichloromethane (reference 47885-u), and reference standards of methyl palmitate (C16:0), octadecanoate (C18:0), *cis*-9 oleate (C18:1(n9)) and linoleate (C18:2(n6)), were purchased from Supelco (Bellefonte, PA, USA). Fatty acids were named using the formula C*x*:*y*(nz;catb), where "*x*" is the number of carbon atoms, "*y*" the number of double bonds and "z" is the position of the first double bond beginning at the methyl terminal group; "a" and "b" were the conventional positions of the double bonds with *cis*, "c", or *trans*, "t", stereoisomerism, which were omitted in the formula when all the double bonds of FAs were *cis*-type.

Standard stock solutions were prepared in dichloromethane (Labscan, Dublin, Ireland) at a concentration of 1000 mg/L. These standard stock solutions were diluted daily with dichloromethane to produce a set of working standards. All standards and stock solutions were kept in the dark at +4 °C and were stable for at least 1 month.

Deionized water was obtained in a Milli-RO plus system together with a Milli-Q system from Millipore (Bedford, MA, USA). Chloroform and hexane were obtained from Labscan (Dublin, Ireland). Diethyl ether was supplied by Panreac (Barcelona, Spain), while 1-propanol, hydrochloric acid (HCl) 32%, potassium hydroxide (KOH) 1 M in methanol and boron triflouride (BF_3) 14% (w/w) in methanol were purchased from Sigma–Aldrich (St. Louis, MO, USA). All the reagents used were of analytical grade.

2.2. Samples

A total of 28 different potato crisp samples directly obtained after the frying process were analyzed. All samples were provided by Facundo S.A. (Villada, Palencia, Spain) and belonged to two different potato varieties, *Hermes* and *Mustang*, both of which are largely used for industrial food processing. Some of the more important physico-chemical characteristics of both potato varieties are summarized in the website of Agrico UK Ltd. (Agrico, 2009). Two batches of a similar mixture of vegetable oils for frying the potatoes were employed: vegetable oil 1, which was composed of olive oil:sunflower oil (80:20), and vegetable oil 2, composed of olive oil:sunflower oil (82:18).

In the factory the potatoes were processed in an automatic continuous snack fryer, where they were washed, trimmed and cut into slices of 1.48 ± 0.18 mm in thickness. After removal of the starch in hot water at 70–90 °C for 3–4 min and superficial drying, the potatoes were placed in a fryer (3000 L capacity) with oil heated to 170–180 °C for 3–4 min, until the moisture content was below 2%. Finally, sampling was carried out as will be below mentioned, and crisps samples were taken to the laboratory, where they were finely ground and stored at 4 °C until analysis.

A batch sampling was carried out. Once the industrial process of making the potato crisps was completed, sample portions (15 g) from 10 different points of the same industrial batch were taken. It must be pointed out that seven batches of every possible combination of potato variety and frying oil (4 combinations) were used to perform the sampling. Following a homogenization process, a large sample portion (150 g) was stored at 4 °C in a dark atmosphere. All samples were treated in the same conditions regarding washing, trimming, cutting, starch removal, temperature and other frying conditions, the differential factors represented by the potato variety and the oil frying. A sample from each of the batches of every possible combination of potato variety and frying oil was analyzed (28 samples in total), and every sample was analyzed in triplicate not later than 48 h after their sampling.