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The analysis of *trans* fatty acid profiles in deep frying palm oil and chicken fillets with an improved gas chromatography method

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ABSTRACT

An improved gas chromatography method for the simultaneous separation of 52 fatty acids (FAs) has been developed. For both oleic acid and linoleic acid, a good resolution was achieved for their positional and geometrical (*cis*/*trans*) isomers. This method was validated to be precise, accurate and sensitive. With this method, the FAs profiles in palm oil and chicken fillets were analyzed. In general, small changes were observed in the composition of FAs and formation of *trans* isomers after 8 h frying at the temperature lower than 200 °C. However, with extreme deep-frying process, the thermal degradation and TFAs formation in frying oil increased in direct proportion to frying temperature and time. Moreover, the FAs composition of fried chicken fillets was found to be mostly in correspondence with that of the frying oil, which might be due to the oil absorption or interaction between the frying oils and frying materials.

1. Introduction

trans Fatty acids (TFA) refer to a group of unsaturated fatty acids that contain one or more isolated, non-conjugated, double bonds in a *trans* geometric configuration. It has been reported that a higher intake of TFA may increase the risk of cerebrovascular disease (Mozaffarian, Katan, Ascherio, Stampfer, & Willett, 2006), coronary heart disease (Oomen et al., 2001), diabetes (Kim, Chunawala, & Linde, Reaven, 2006) and even breast cancer (Kohlmeier, 1997). In light of this, Food and Drug Administration (FDA) and Canadian Food Inspection Agency (CFIA) have issued the rule requiring manufacturers to include TFA contents in food products label (CFIA, 2008; FDA, 2003). This rule prompts the urgent need to develop a simple and accurate method for the quantification of TFAs in food products.

TFAs in foods are mainly derived from the partially chemical hydrogenation of vegetable and fish oils. Deep frying has been considered a source for the production of TFAs. As one of the most common methods of food cooking, frying is a process of immersing food in hot oil with a contact among oil, air and food at high temperatures from 150 °C to 200 °C. The simultaneous heat and mass transfer of oil, food and air during deep-fat frying produces a food product with desired sensory characteristics, including fried food flavor, golden brown color and a crisp texture, which is very popular to consumers. However, this process will also initiate the thermal oxidative deterioration of unsaturated FAs, which is considered to be linked with TFAs accumulation in edible oils by heating or frying. The formation of TFAs during frying has been investigated in several hydrogenated vegetable oils. Aladedunye and Przybylski (2009) reported that the final concentrations of total TFAs after 7 days frying of French fries in canola oil increased from its initial value of 2.4–3.3% at 185 ± 5 °C, to 5.9% at 215 ± 5 °C. Similar results have been reported by Yang, Yang, Nie, Xie, and Chen (2012) for corn oil. However, Romero, Cuesta, and Sánchez-Muniz (1998) reported a very minimal production of elaidic acid (less than 0.5 g/100 g) after 20 frying cycles of extra virgin olive oil, high oleic sunflower oil and regular sunflower oil. Tsuzuki, Nagata, Yunoki, Nakajima, and Nagata (2008) also reported that statistically significant differences were observed for the amounts of *trans* 18:1 FAs in rice bran oil, safflower oil and sesame oil heated in a glass tube at 180 °C for 4 h. Thus these previous studies indicated that several factors such as the frying conditions, the FA composition and coexisted antioxidants in frying materials and even the methods of TFAs measurements would contribute to the variation in the thermal TFAs

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accumulation profiles. Furthermore, when the frying materials containing TFAs were introduced to frying process, the *trans* level in frying materials and the frying oils can affect each other due to fast exchange of fats during frying (Zhou, Tan, Neo, & Lo, 2009). Thus respective monitoring of TFAs accumulation in various frying materials and frying oil will be necessary in order to investigate TFAs formation in edible oils during frying.

The analysis of *trans* fatty acid is extremely challenging and complex due to the various positional isomers of unsaturated fatty acids. The reported analytical approaches mainly include silver-ion thin-layer chromatography (Ag^+-TLC), infrared spectroscopy (IR), reversed phase high performance liquid chromatography (RP-HPLC), gas chromatography (GC), GC–MS, and comprehensive two-dimensional gas chromatography. The IR method is considered to be inaccurate and unreliable for quantifying isolated *trans* fats due to the interference of absorption from other functional groups other than the *trans* bond, especially in samples containing below 5% *trans* fats (Albuquerque, Costa, Castilho, & Sanches, 2011). Ag^+-TLC is able to separate the *trans* isomer from the *cis*, but for quantitative analysis of *trans* fat, the isolated *trans* fraction still needs to be analyzed with subsequent capillary GC method (Golay, Dionisi, Hug, Giuffrida, & Destaillats, 2006). RP-HPLC also has several disadvantages, such as complexity of derivatization method for fatty acids, limited availability of derivatives standards and lower separation efficiency compared with capillary GC. The drawbacks outlined above can be partly avoided by GC with mass spectrometry (MS) detectors (Manzano, Diego, Nozal, Bernal, & Bernal, 2012; Schröder & Vetter, 2013) and the use of multidimensional gas chromatography (Manzano, Arnáiz, et al., 2012; Manzano, Diego, et al., 2012; Tranchida, Franchina, Dugo, & Mondello, 2012), which can be a good alternative to determine minority fatty acids and quantify the different isomers. However, these particular instruments are expensive and bulky and not typically used in most small enterprises or laboratories. In comparison, the most acceptable and conventional method is GC coupled with a flame ionization detector (GC-FID) with appropriate accuracy, convenience and cost (Antolín, Delange, & Canavaciolo, 2008). However, due to the complexity of the fatty acid composition in different food, GC-FID methods for the analysis of fatty acids described so far suffer from analysis times longer than 80 min, no separation or bad resolution of geometric and positional isomers. For instance, Berdeaux, Dutta, Dobarganes, and Sébédio (2009) reported in their review that numerous peaks were poorly resolved, or eluted together in one peak, or co-eluted with cis-18:1 isomers even using a 100-m BPX70 capillary column when mono-trans-18:2 or di-trans-18:2 positional isomers are present. Similarly, Sébédio and Ratnayake (2008) developed several improved GC methods on highly polar columns such as 100 m CPSil 88 or equivalent, however, a complete separation of the seven geometrical isomers from linolenic acid still cannot be achieved. Actually, individual separation of each isomer is essential for an optimal quantitation of *trans* fatty acids in various foods, preventing any risk of under- or over-estimation due to the method. Therefore, future developments to simplify and improve the current GC procedure will be in great need to meet the diversity of food products.

The aim of this study was to optimize a simple and reliable GC method based on the AOCS Official Method (AOCS, 2005) for the determination of FAMEs, and to prove its applicability for determining the fatty acid profile especially the *trans* fat in frying food samples. Palm oil is commonly used by consumers as a medium to fry food. However, the effects of deep-frying on the TFA formation of unhydrogenated palm oils during the frying process, have received little attention. Therefore, TFAs formation during frying process was investigated using chicken fillets and the commercially available palm oil, as the frying materials and the frying oil.

2. Materials and methods

2.1. Chemicals and reagents

Reference standards GLC-463, internal standards monoheneicosanoin (C21:0, TAG) and methyl heneicosanoate (C21:0, FAME) were purchased from Nu-Chek Prep, Inc. (Elysian, MN, USA). FAMEs of C18:2 isomer mixture (No. 47791) and C18:3 isomer mixture (No. 47792) were bought from Supelco Inc. (Bellefonte, PA, USA). Standard FAMEs (C12:0, C14:0, C16:0, *9c* C16:1, C17:0, C18:0, *9t* C18:1, *9c 12t* C18:2, *9t 12c* C18:2, *9c 12c* C18:2, C20:0, *9c 12c 15c* C18:3) were purchased from Sigma (St. Louis, MO, USA). HPLC grade n-heptane was obtained from Fisher Scientific (Fair Lawn, NJ, USA). Palm oil was bought from South Sea Oils & Fats Industrial Ltd. (Shenzhen, China) and chicken fillet was bought from local supermarket (Shengyuan Food Co., Ltd., China). All solvents and chemicals were of analytical grade and used without further purification unless otherwise specified. Stock standard solutions were prepared in n-hexane and stored at 4 °C until use. Potassium hydroxide, ethyl alcohol, ether, phenolphthalein, hydrochloric acid, peroxide, chloroform, methanol, ferrous chloride, potassium thiocyanate and reduced iron powder were of analytical grade.

2.2. Frying procedure

Palm oil (3 L) was poured into a commercial electrical fryer (Laierjia Co., Shanghai, China), which could control the oil temperature within ±5 °C from the set temperature. The oil was heated to 150, 200 and 250 °C, and then a batch of 40 ± 5 g chicken fillets was added and fried for 10 min as one frying cycle at each temperature. In each day, the oil was continuously heated for 8 h with 48 frying cycles performed. Frying oil (10 mL) was collected every 2 h with 12 frying cycles. The first, 24th and the last batches of fried chicken fillets were also collected. All the samples were stored at −20 °C for further chemical analysis.

2.3. Lipid extraction from chicken fillets

The total lipids contained in fried chicken fillets were extracted according to the method by Folch, Lees, and Sloane-Stanley (1957) with minor modification. Briefly, 15 g of crushed chicken fillets sample was poured into a round bottom flask, followed by addition of 60 mL chloroform-methanol (1:1, v/v) and homogenization for 1.5–2.0 min. Successively, the mixture was reflux-heated in a water bath at 60 °C for 1 h and then cooled to room temperature. After being filtered through a fluted filter paper, the resultant filtrate was collected and dried under reduced pressure using a vacuum evaporator. Then the fat obtained was resolved in 25 mL petroleum ether, and mixed with 5 g sodium sulfate anhydrous to further remove the residual solvent. Subsequently, the mixture was separated by centrifugation at 3000 *g* for 5 min and the supernatant was collected, evaporated and dried at 105 °C in succession. The residue was then weighed and stored at −20 °C.

2.4. Preparation of fatty acid methyl esters (FAMEs)

Corresponding FAMEs of the oil samples and the lipids extracted from fat samples were prepared by esterification with potassium hydroxide following the AOCS Official Method Ce 2-66 (AOCS, 1997) with minor modification. Approximately 10 mg of sample was