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Actas do 5º Encontro de Química de Alimentos

Universidade Católica Portuguesa
Escola Superior de Biotecnologia

Sociedade Portuguesa de Química

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Influence of the polarity of the derivatization medium on fatty acid assays in cod liver oil

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The rate of transmethylation of fatty acid residues of a given lipid depends not only on the type of catalyst and processing conditions used, but also on the solubility of said lipid in the reaction medium (which is in turn a function of the polarities of both the lipid and the medium). In this work, the influence of the reaction medium polarity, employed to bring about acidic transmethylation, was studied using cod liver oil as model system; acetyl chloride solubilized with various amounts of methanol, diethyl ether and chloroform has produced different yields of transmethylated fatty acids. The accuracies of these methods were tested via comparison with the AOCS standard method, whereas their reproducibilities were assessed via analysis of variance of replicated data.

Experimental Procedure

Sample and reagents. Cod liver oil was purchased in gelatine capsules from Bioarga (Portugal).

Acidic methylation. 4 mL of a freshly prepared 5 % (v/v) solution mixture of acetyl chloride/solvent and tricosanoic acid (Sigma, USA) (used as internal standard) were added to the sample in a light-protected teflon-capped tube, under nitrogen, and then heated at 80°C for 1 h; after cooling, 2 mL of water and 2 mL of hexane were added; finally, the two phases were allowed to separate, the upper phase was recovered, dried over anhydrous Na2SO4 (Merck) and subjected to gas chromatography.

Seven different solvent mixtures were tested: A1 (3 ml of methanol and 1 ml of diethyl ether), A2 (2.5 ml of methanol and 1.5 ml of diethyl ether), A3 (2 ml of methanol and 2 ml of diethyl ether), A4 (2 ml of methanol and 2 ml of chloroform), A5 (2.5 ml of methanol, 0.75 ml of diethyl ether and 0.75 ml of chloroform), A6 ((2 ml of methanol, 1 ml of diethyl ether and 1 ml of chloroform) and A7 (4 ml of methanol).

Standard method. The AOCS Official Method Ce1b-89 using BF3 (Supelco) (1) was followed thoroughly. This method will hereafter be denoted as BF3.

Experimental analysis. Assay of FAME was carried out with a 5890 gas chromatograph from Hewlett Packard (USA), equipped with a flame ionization detector and a polar, 50-m capillary column of fused silica (CP-Sil 88, Chrompack). Helium was used as carrier gas at a split ratio of 1:40, and the remaining conditions were as specified elsewhere (1). Pure standards (Sigma) were used for fatty acid identification. Peak areas were quantified with an HP-3395 integrator, and calculations were performed according to the AOCS method.

Statistical design and analysis. The methods described above were tested following a randomized experimental design replicated at least five times. Analyses of variance (ANOVA) of the overall dataset, and Fisher's protected least significant difference (PLSD) test for pairwise comparison, were performed using the software StatViewTM (Abacus Concepts, USA).

Results and Discussion

Results of the assays regarding some of the fatty acids obtained during transmethylation of cod liver oil samples using the solvent mixtures described and the standard method are presented in the Figure 1 below.

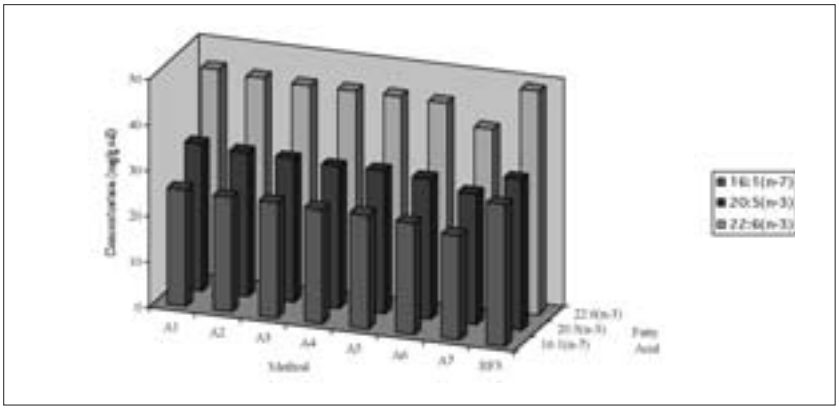

Figure 1

Combination of all pieces of experimental and statistical information generated indicates that the reference analytical method (BF3) should be selected for assays of total fatty acids, since it provides the highest yields. However, if one is interested only in the concentrations of EPA, the method A1 is also a suitable choice, since the differences found between this method and BF3 are not statistically significant. For DHA, provided that a solvent other than hexane is used in the extraction step (because,

according to some authors (2), the extraction of FAME with hexane does in fact lead to a decrease in DHA amounts), A1 could also be a suitable method. The use of acetyl chloride, instead of the standard method, has several advantages, viz. lower cost, higher shelf life without the need of refrigeration, higher simplicity of implementation arising from the lower number of preparation steps and smaller amount of catalyst used (5% solution instead of 12%).

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