Tirtiaux Fractionation: Analytical Data of End-Products through HPLC

ETIENNE DEFFENSE, S.A. Fractionnement Tirtiaux, 601, Chaussée de Charleroi, B-6220 Fleurus, Belgium

INTRODUCTION

The Tirtiaux process, with its accurate crystallization control and its continuous horizontal vacuum filter, provides a variety of products of different physical and chemical properties. The ability to refractionate any one of the end-products gives a wide range of specific qualities with commercial potential.

This paper describes the use of high performance liquid chromatography (HPLC) as a rapid routine technique for monitoring the fractionation of fats like palm oil and beef tallow.

CHARACTERISTICS OF PALM OIL FRACTIONS

Although fatty acid methyl ester (FAME) analysis is used for identification purposes, fractionation of palm oil can be more sensitively monitored on a triglycerides basis than by classical analysis of FAME, since it is a direct analysis of the fat affected by the fractionation treatment. However, gas liquid chromatographic (GLC) techniques give insufficient information to provide a complete triglyceride composition of the different fractions of palm oil even when combined with the fatty acid composition.

HPLC on reverse-phase column, separating triglycerides by chain length and by degree of unsaturation, provides more detailed information about triglyceride composition. The triglycerides of palm oil (Fig. 1.) were clearly separated into 16 peaks on two columns C 18-5μ in series, with acetone/acetonitrile (62.5:37.5) mixture as eluent. The 4 major peaks represent triglycerides POL (5), PLP-MOP (6), POO (9) and POP (10). Other palm oil triglycerides are OLL (1), PLL-MOL (2), MLP-MOM (3), OOL (4), MPP (7), OOO (8), PPP (11), SOO (12), POS (13), PPS (14). Two minor peaks are attributed to SOS (15) and PSS (16). The designation POO does not imply the triglyceride POO but a mixture of all isomers POO, OPO and OOP.

With solvent mixture acetone/acetonitrile (62.5:37.5), it has been found that fats and hard fractions like stearins are difficult to elute and tend to crystallize in the column. At 50 C, the solubility of the fat in the eluent is increased,

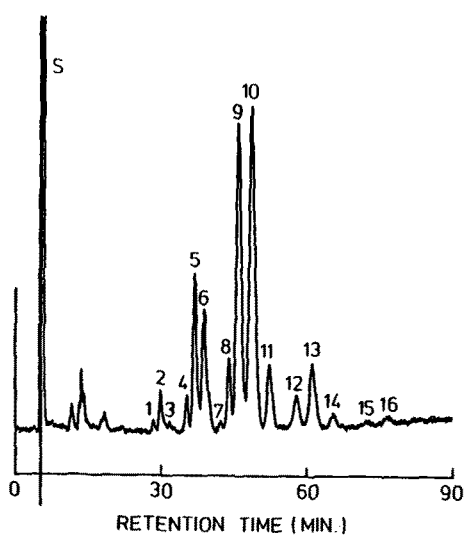

FIG. 1. HPLC of palm oil. P = palmitic acid, S = stearic acid, O = oleic acid, M = myristic acid, and L = linoleic acid.

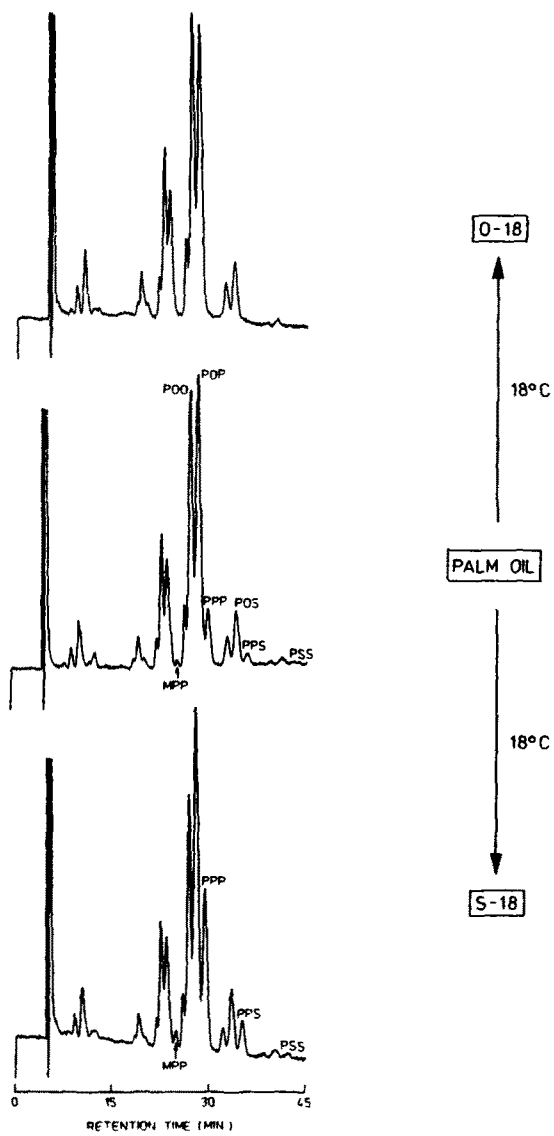

FIG. 2. HPLC of palm oil fractions.

the resolution is still maintained and the analysis time is reduced. The elution of SSS (54:0) required 52 min and a capacity factor k' less than 10, as opposed to a k' value of ca. 15 under the previous conditions (30 C).

The different fractions of palm oil taken from industrial Tirtiaux plants were analyzed by HPLC according to these conditions: chromatographic runs were performed with a Waters Associates liquid chromatograph 6000A equipped with two columns in series: a μ-Bondapak C 18, 5 μ (Waters) and a Lichrosorb RP-18, 5 μ (Merck). Samples were injected as methanol/chloroform solution by means of a U6K septumless loop injector. A Waters R401 differential refractometer was used as detector. Samples were run isocratically at 50 C using a mixture of acetone/acetonitrile (62.5: 37.5) with a flow rate of 1.1 mL/min. Separations were recorded with the aid of a Hewlett Packard 5880 A electronic integrator with retention times printed automatically.

Chromatograms of palm oil and its most common fractions, (olein and stearin separated at 18 C, Fig. 2.), reveal that the major difference lies in the trisaturated triglycerides (PPP, PPS, MPP) which disappear completely in the olein 0-18. The palmitodiolein (POO) tends to increase in the olein. In fact, when the increase in POO is more substantial, we have noted a significant decrease of the olein cloud point.

BEEF TALLOW

Figure 3 shows HPLC curves of an industrial fractionation of a titer 42.5 beef of European origin.

The effect of fractionation on the triglyceride composition of beef fat is prominently shown in the trisaturated triglycerides content S3 where the 44% observed for SSS, PSS, PPS and PPP in stearin SS-36-43 is reduced to 3% in the olein 00-36-20. A major difference occurs also in the increase of the POO in the olein.

The large difference in triglyceride composition observed between the oleins and the stearins of the beef reflects the wide variety of products which can be obtained by this commercial process.

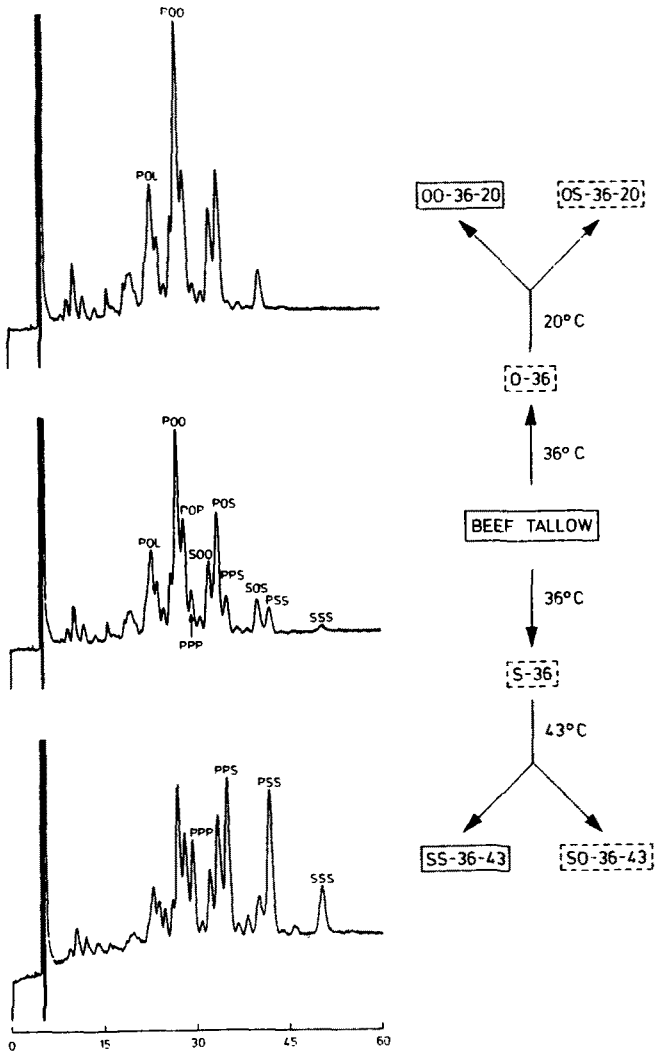

FIG. 3. HPLC of beef tallow fractions.