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Analytical Methods

A high performance liquid chromatography method for determination of furfural in crude palm oil

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ABSTRACT

A modified steam distillation method was developed to extract furfural from crude palm oil (CPO). The collected distillates were analysed using high performance liquid chromatography (HPLC) coupled with an ultraviolet diode detector at 284 nm. The HPLC method allowed identification and quantification of furfural in CPO. The unique thermal extraction of CPO whereby the fresh fruit bunches (FFB) are first subjected to steam treatment, distinguishes itself from other solvent-extracted or cold-pressed vegetable oils. The presence of furfural was also determined in the fresh palm oil from FFB (without undergoing the normal extraction process), palm olein, palm stearin, olive oil, coconut oil, sunflower oil, soya oil and corn oil. The chromatograms of the extracts were compared to that of standard furfural. Furfural was only detected in CPO. The CPO consignments obtained from four mills were shown to contain 7.54 to 20.60 mg/kg furfural.

1. Introduction

Palm oil research institutions have continuously strived to discover new and functional compounds in the extracted palm oil, i.e. the CPO. Some of the natural minor compounds discovered include vitamin E, phenolics, sterols, squalene and terpenoids (Goh, Choo, & Ong, 1985) and these compounds have been embodied as important dietary and nutritional compounds affecting human health. Meanwhile, thermal extraction of CPO from fresh fruit bunches (FFB) produces new compounds due to hydrolysis of complex material or chemical interaction at high temperature.

CPO, the crude oil extracted from the mesocarp of palm fruits in the conventional mill, employed high pressure steam to sterilise the FFB. The steam at 40–45 psi (or 275.8–310.3 kPa) sustained at 140–145 °C is supplied into the steriliser for 1–1½ hr. The sterilisation process not only serves to inactivate the fruit and infecting microorganism lipases but has a more crucial role in that it maximises the hydrolysis of carbohydrates within the FFB (Ariffin, 1991). Specific carbohydrates, namely hemicelluloses, bind fruits to the bunch stalk. An effective and optimised sterilisation ensures hydrolytic degradation of these carbohydrates and maximises detachment of fruits from the bunch (Ariffin, 1991; Poku, 2002). The number of detached fruits will affect the mill oil extraction ratio or rate (OER) (Wahid, Abdullah, & Henson, 2004).

Unlike alpha-cellulose, which is chemically stable, hemicelluloses are prone to chemical breakdown and yield various simple sugars; the 6-carbon glucose, fructose, galactose and the 5-carbon xylose, ribose and arabinose. Studies on conifer and other softwoods reveal that xylose can be further dehydrated to form furfural (Gandini & Belgacem, 1997).

The presence of furfural and its related compounds has been detected in various processed foods and beverages such as infant formulas (Ferrer, Alegria, Farre, Abellan, & Romero, 2002), Italian chestnuts (Krist, Unterweger, Bandion, & Buchbauer, 2004), brandy (Barroso, Rodríguez, Guillén, & Pérez-Bustamante, 1996), citrus juice (Lee, Rouseff, & Nagy, 1986), and apple juice (Kermasha, Goetghebeur, Dumont, & Couture, 1995). Furfural has also been detected in cocoa, coffee, tea, beer, wine, and bread (Maga, 1979). All these food products have been exposed to thermal treatment during processing and this has led to the breakdown of carbohydrate to furfural (IPCS, 2000).

The information on the formation and occurrence of breakdown of carbohydrates during processing of palm fruit bunch to oil is lacking. This scarcity has led to the investigation of the hydrolytic effect of carbohydrate towards the formation of furfural. Furfural formation in oil is determined via the high performance liquid chromatography (HPLC).

2. Materials and methods

2.1. Materials

Crude palm oil samples were obtained from different palm oil mills. Refined palm oil fractions (refined palm olein (RBDPOL)

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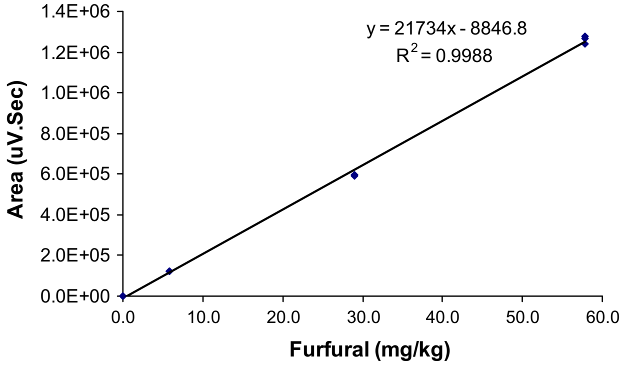

Fig. 1. Standard furfural calibration curve via the modified steam distillation method.

and refined palm stearin (RBDPSt)), olive oil, coconut oil, sunflower oil, soya oil and corn oil were obtained from a local supermarket. Fresh palm oil from FFB was obtained using soxhlet extraction and hexane as the solvent.

Standard furfural was purchased from Merck (Darmstadt, Germany) and 5-hydroxymethyl-2-furfural (HMF) was purchased from Acros Organics (Geel, Belgium). Acetonitrile (HPLC grade) and hexane were obtained from BDH Prolabo (Poole, UK).

2.2. Extraction of furfural

Furfural extraction was carried out using a modified steam distillation method. A total of 20 g oil sample was placed into a 250 mL round bottomed flask and 100 mL of distilled water was added. A separation funnel containing 100 mL of distilled water was attached to the top of the round bottomed flask via a T-connector and water was allowed to drip at the rate of 1.5 to 2 mL/min. The water level in the flask was maintained at 100 mL throughout the extraction. The mixture was boiled, stirred on a hotplate magnetic stirrer and covered with aluminium foil to ensure consistent heating in the flask. A condenser was connected to the T-connector, bridging the flask to another distillate-receiving flask. The distillation process took 60 to 70 min to complete. The extraction was stopped once 100 mL of distillate was collected.

2.3. High-performance liquid chromatography (HPLC) analysis

A Hypersil ODS C_{18} 5 μm silica column (250 × 4.6 mm. i.d.) (Thermo Fisher Scientific, Waltham USA) was used as the solid phase. Separations were carried out isocratically at room temperature using a mixture of acetonitrile–water (5:95, v/v) at a flow rate of 1 mL/min as the mobile phase. Detection using a UV detector at wavelength 284 nm was carried out (Ferrer et al., 2002). The injection volume was 20 μL and ran for 16 min.

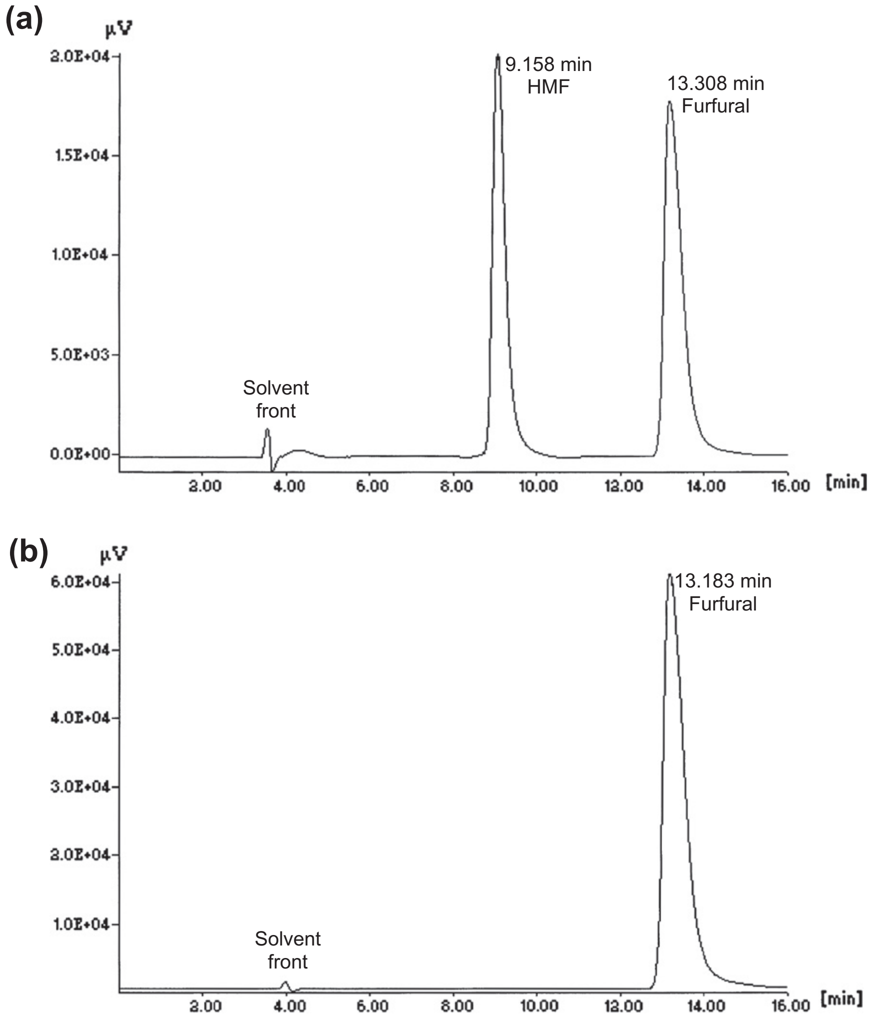

Fig. 2. Chromatograms (284 nm) of (a) standard furfural and 5-hydroxymethyl-2-furfural (HMF), and (b) furfural in crude palm oil (CPO) distillate.