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Analytical Methods

Extraction of β-carotenes from palm oil mesocarp using sub-critical R134a

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ABSTRACT

Sub-critical extraction of palm oil from palm mesocarp using R134a solvent was conducted via the dynamic mode to investigate the ability of R134a to extract β-carotene. The yield of palm oil and the solubility of β-carotene were investigated at 40, 60 and 80 °C and pressure range from 45–100 bar. The extracted oil was analysed for β-carotene content using UV–Vis spectrophotometry. The results showed that palm oil yield increased with pressure and temperature. The maximum solubility of β-carotene was obtained at 100 bar and 60 °C while the lowest solubility occurred at 80 bar and 40 °C. The higher concentration of extracted β-carotene ranging from 330–780 ppm as compared to that achieved through conventional palm oil processing indicates that extraction of β-carotene using R134a is viable.

1. Introduction

Palm oil is well-known to contain a high amount of carotenoids. Typical crude palm oil contains 500–700 ppm carotenes, which contribute to palm oil stability and nutritional value. Conventional processing of palm oil leaves about 3–7% residual oil in the fruit fibre (França & Meireles, 2000; Lau, Choo, Ma, & Chuah, 2008; Nik Norulaini et al., 2008) and a high content of carotenoids in the pressed palm fibres. Residual fibres from palm oil production contain between 4000 and 6000 ppm of carotenoids, about six times higher than that found in crushed palm oil (Franca & Meireless, 1997, 2000). Until now, palm oil fibre residues that are rich in valuable carotenoids are treated as a waste product, which is destined to be burnt together with empty fruit bunches (Birtigh, Johannes, Brunner, & Nair, 1995) or transported to the plantation for field mulching (Lau et al., 2008). Effective recovery of the carotenes would therefore give a significant advantage to the palm oil industry.

Supercritical fluid extraction (SFE) technology has become an increasingly popular method for the recovery of food ingredients and products over the last 20 years, due to its unique advantages, including low temperature use, selective extraction, simpler and cleaner (solvent-free) product recovery. SFE is also an environmentally benign technology since the process typically generates no waste. Supercritical fluid exhibits high density like liquids, which contributes to greater potential for solubilisation of materials, and low viscosity similar to gases, which enables its penetration into the solid.

Extraction of palm oil from its mesocarp using supercritical fluid technology has been considered a promising way to recover carotenes and prevent their high losses during palm oil milling. Application of SFE technology for crude palm oil extraction, and for the recovery of carotenes from palm mesocarp is expected to have a great future outlook (Bharath, 2003) was and is seen to have great potential for replacing the conventional screw-press extraction, clarification and vacuum drying processes (Lau, Choo, Ma, & Chuah, 2006). Therefore, the extraction of palm oil using supercritical extraction technology is set to become more popular in the next 10 years.

Various studies aimed to investigate the potential of some marker compounds to be extracted using supercritical solvents, such as carbon dioxide (Cygnarowicz, Maxwell, & Seider, 1990; Franca & Meireless, 1997; Lau et al., 2008; Markom, Singh, & Hasan, 2001; Nik Norulaini et al., 2008; Puah, Choo, Ma, & Chuah, 2005; Saldaña, Temelli, Guigard, Tomberli, & Gray, 2010; Sovová, Stateva, & Galushko, 2001; Škerget, Knez, & Habulin, 1995), ethane (Mendes, Nobre, Coelho, & Palavra, 1999) and nitrous oxide (Sakaki, 1992; Subra, Castellani, Ksibi, & Garrabos, 1997). Hansen, Harvey, Coelho, Palavra, and Bruno (2001) reported the solubility of carotene in halocarbon (R134a).

Carbon dioxide (CO_2) has been the most popular supercritical solvent, due to its non-toxic, non-hazardous and non-flammable properties, but typically requires pressure of up to 500 bar for satisfactory extraction of food products. In view of the economic and environmental needs, it is desirable to explore alternative SFE

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solvents that enable operation at less intense conditions (Catchpole & Proells, 2001; Wood & Cooper, 2003), thereby allowing designers to exploit the typical SFE benefits at more reasonable costs (Perrut, 2000).

R134a (1,1,1,2-tetrafluoroethane) is non-toxic, non-reactive, non-flammable, and non-ozone depleting. It also has a high volatility and a boiling point at atmospheric pressure of −26.2 °C, which means that it leaves negligible solvent residues in the products. The use of R134a as the extraction solvent at sub-critical conditions can address the shortcomings of supercritical CO_2 processes (Corr, 2002). Sub-critical fluid, which is also known as a high-pressure liquid, exhibits similar behaviour to, and can be exploited in the same manner as, supercritical fluids (Brunner, 1994; Brunner, 2005).

To-date, only Mustapa, Manan, Mohd Azizi, Nik Norulaini, and Mohd Omar (2009) have studied the use of sub-critical R134a as an alternative to CO_2 solvent for the recovery of palm oil. They performed the extraction of palm oil from palm mesocarp, using R134a at sub-critical conditions. The extraction of palm oil was found to increase with temperature and peaked at a maximum of 100 bar and 80 °C. Mustapa et al. (2009) also reported that the extraction of palm oil using R134a has great potential for application in the palm oil industry. The objective of this study is to investigate the potential of using R134a to recover valuable minor components by determining the yield of palm oil extracted from palm mesocarp and the solubility trend of β-carotene in R134a over the range of temperatures and pressures studied.

2. Methodology

2.1. Materials and sample preparation

Fresh palm fruits were provided by MALPOM Industries Sdn. Bhd palm oil mill in Nibong Tebal, Penang, Malaysia. The fresh fruits were put in vacuum plastic bags and stored at −20 °C. Prior to extraction, a bag of the sample was randomly selected and was left at room temperature for about 30 min. Commercial liquefied R134a gas (purity 99.7%) was supplied in a gas cylinder by Malaysia Oxygen (MOX), Penang. The mesocarp of fresh palm fruits was peeled, sliced and chopped to reduce the sample size using a Kiwi cutter. Samples of chopped mesocarp were oven-dried to reduce its moisture content to a range of about 5–6%. The sample was named as Uncooked-Chopped (UC) sample. Mustapa et al. (2009) reported that the UC sample palm fruits resulted in higher oil yield than cooked fruits. They also reported that the chopped samples gave higher extraction rate as compared to sliced samples. Lau et al. (2006) peeled off the palm mesocarp and dried in the oven at 60 °C until a constant weight was obtained. Prior to the extraction the dried mesocarp was ground into pieces of 1–3 mm length.

2.2. Sub-critical R134a extraction

The dynamic extraction is a superior SFE method when the solid sample has a high oil concentration (De Castro, Valcárcel, & Tena, 1994). Fresh liquid solvent at ambient temperature is charged and compressed to the desired pressure using high-pressure pump at the beginning of extraction. The fluid then flows through an extractor vessel containing the solute and the solvent. The fluid then leaves the vessel and is depressurised to atmospheric conditions, using a heated metering valve, or restrictor. At the same time, the extracted solute will precipitate or fall out of the fluid solution, to be collected and quantified gravimetrically, or by some conventional analytical technique. The solvent volume can either be measured using a gas totaliser or by measuring the flow rate of the solvent and the collected sample (Taylor, 1996).

Palm oil extraction experiments were carried out in a laboratory scale extraction apparatus shown schematically in Fig. 1. The apparatus consists of HPLC Pump (Model PU-980, Jasco, Japan) with a maximum capacity of 50 MPa (10,000 psi) which was used to deliver and regulate the R134a flow. Previous study by Mustapa et al. (2009) showed that the flow rate of sub-critical R134a resulted in a consistent oil solubility and shorter extraction time. Therefore, the R134a flow rate was maintained at 3 ml/min for all extraction conditions. For each extraction run, 1 ± 0.001 g of UC sample was placed in a 10-ml extractor (1.5 cm i.d. × 8 cm) and the extractor was placed in an oven (Model Memmert), which was used to control the operating temperature to within ±2 °C of the set point temperature for each run. The system pressure was controlled by a back-pressure regulator (Model 880–81, Jasco, Japan) with an

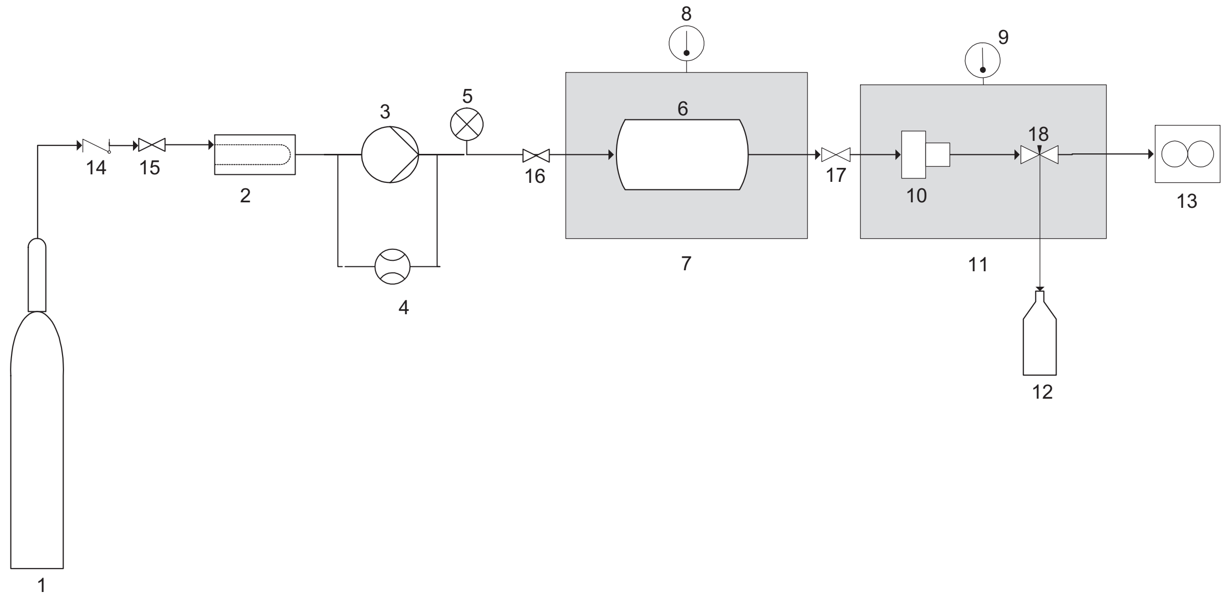

Fig. 1. Schematic diagram of the R134a extraction apparatus (1) R134a cylinder, (2) Chiller (set at −5 °C), (3) HPLC pump, (4) flow meter indicator, (5) pressure meter, (6) extraction cell, (7) oven (set the extraction temperature 40–80 °C), (8–9) temperature indicator, (10) heater (60–70 °C), (11) back-pressure regulator (BPR), (12) vial collector, (13) mass flow meter, (14–18) valve).