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Enrichment of minor components from crude palm oil and palm-pressed mesocarp fibre oil via sequential adsorption-desorption strategy

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ABSTRACT

Crude palm oil (CPO) and palm-pressed mesocarp fibre oil (PPMFO) consist of minor components such as Vitamin E, phytosterols, squalene, and carotene that play numerous health function in human. However, limitations are present in current separation processes to separate or enrich minor components from plant sources. In this study, interest is focusing on the enrichment efficiency of the minor components from CPO and PPMFO through proposed sequential adsorption-desorption strategy. The strategy started with the adsorption-desorption properties evaluation of six adsorbents. Both Diaion HP20 and Sepabeads SP850 showed better adsorption-desorption properties than silica gel, Florisil, Diaion HP2MG and Amberlite XAD-7HP. Diaion HP20 was selected as the suitable adsorbent to perform a series of selective desorption process using three different solvents: methanol, IPA, and *n*-hexane in Soxhlet extraction. Vitamin E, phytosterols, and squalene from CPO were obtained in the 1st fraction using methanol solution and their concentration increased from the initial concentration with the enrichment factor (EF) of 3.4, 3.9, and 1.8, respectively, which slightly higher than those minor components obtained from PPMFO, 1.2, 1.8, and 1.4, respectively. The carotene from both CPO and PPMFO was enriched in the 3rd fraction by using *n*-hexane solution with an enrichment factor of 1.1 and 1.5, respectively. In conclusion, the obtained result revealed the efficiency of the proposed sequential adsorption-desorption strategy to enrich the minor components from CPO and PPMFO.

1. Introduction

Crude palm oil (CPO) which represents one of the world's major vegetable oil, is the product of oil palm industry. It is extracted from the oil palm (*Elaeis guineensis Jacq.*) mesocarp mainly through mechanical screw pressing technique (Mba et al., 2015). Besides as vegetable oil, it serves as the raw material for the commercial production of Vitamin E, phytosterols, squalene, and carotene (Choo et al., 2009; Ho, 2009). Vitamin E (1000–1200 μg/g), phytosterols (326–527 μg/g), squalene (200–500 μg/g), and carotene (500–700 μg/g) are present as minor components in CPO (Mba et al., 2015).

Another potential source of minor components of oil palm is palm-pressed mesocarp fibre which is one of the solid wastes produced in the oil palm industry (Liu et al., 2008). Currently, palm-pressed mesocarp fibre is being utilized as fuel to generate steam and energy in the palm oil mill (Abdullah and Sulaiman, 2013). Studies show that residual oil from the fibre contains high amount of Vitamin E (1900–3000 μg/g), carotene (3800–7000 μg/g), phytosterols (4500–8500 μg/g), and squalene (1400–1700 μg/g) (Choo et al., 1996; Lau et al., 2006). Thus, palm-pressed mesocarp fibre oil (PPMFO) can be valorised as the natural source for extracting minor components.

Studies reported that Vitamin E shows anti-proliferative effect on human breast cancer cells (Loganathan et al., 2013; Nesaretnam et al., 1995) and hepatoprotective effect (Magosso et al., 2013). The phytosterols help in lowering cholesterol by inhibiting intestinal cholesterol adsorption (Fernandes and Cabral, 2007; Miettinen et al., 1995) whereas squalene demonstrates chemopreventive effect on cancer (Newmark, 1999; Rao et al., 1998). The carotene especially β-carotene possesses 100% provitamin A activity, which helps in improving eye vision (Saini et al., 2015). The growing interest in the minor components has led to development of nutrition, cosmetic, and pharmaceutical industry.

In order to fulfil the market demand of these minor components, attempts such as supercritical fluid extraction (Akgün, 2011; Mendes et al., 2005) and molecular distillation (Fregolente et al., 2006; Liu et al., 2008; Posada et al., 2007) were developed to separate the minor components from plant source. However, there are limitations in current separation processes including high capital investment to build up

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pressure resistant equipment, strict operation conditions, and multi-stage separation process. Chemical modification such as transesterification are required before separation process when using vegetable oil as the feedstock.

Adsorption-desorption concept is one strategy that can be applied as a separation process through enrichment of target components into one fraction. In the adsorption process, the components in the oil mixture bound on the adsorbent, and were further desorbed at a reverse condition in the desorption process (Ghosh, 2006). Separation through adsorption-desorption process is feasible as it uses simple equipment setup and requires no or less feedstock pre-treatment. Moreover, it can be performed at atmospheric pressure. The extraction solvent can be recovered or recycled (Gunawan et al., 2008). Latip et al. (2001, 2000) demonstrated the enrichment of carotene from CPO using adsorption-desorption technique in Soxhlet extraction system and yielded carotene enriched and carotene depleted fractions. Similar technique was applied to enrich the Vitamin E, phytosterols, and, squalene from soybean oil deodorizer distillate (Gunawan et al., 2008). Vitamin E and phytosterols were enriched into polar liquid fraction whereas squalene was enriched into non-polar liquid fraction. The study related to the enrichment of Vitamin E, phytosterols, squalene, and carotene from CPO and PPMFO using adsorption-desorption process is still very limited.

This study proposed a sequential adsorption-desorption strategy related to the enrichment efficiency of the minor components from CPO and PPMFO. The aim of this study is to obtain optimized conditions to enrich any single minor components. These includes the study on the effect of adsorbent types, desorption solvents, and desorption time by subjecting CPO as the feedstock. Then, it was repeated with PPMFO as the feedstock, performed at the optimum condition obtained from the CPO study.

2. Materials and methods

2.1. Materials

Palm-pressed mesocarp fibre and CPO were obtained from Felda Sungai Tengi Palm Oil Mill, Selangor, Malaysia. The fibre sample was dried in an oven, at 60 °C for 12 h and placed into a Soxhlet apparatus to extract the oil using *n*-hexane. For CPO, it was filtered with filter paper (Whatman, Type 1) to remove insoluble material before use.

Tocopherols (α-, β-, γ-, δ-) and tocotrienols (α-, γ-, δ-) in a purity > 93% were purchased from Chromadex, USA. *n*-Tetradecane (99%), 5α-cholestane (97%), squalene (99.3%), β-sitosterol (85%), and *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (MSTFA) were purchased from Sigma-Aldrich Co. LLC. (Saint Louis, USA). CPO served as an in-house standard for monoacylglycerols, diacylglycerols, and triacylglycerols were quantified by Malaysia Palm Oil Board (MPOB), Malaysia before use. All chemicals used were reagent grade except for those used in the analysis were analytical grade.

Adsorbents used in this study were silica gel (Scharlau Chemical, Spain), Florisil (Merck KGaA, Germany), Diaion HP2MG, Amberlite XAD-7HP, Diaion HP20, and Sepabeads SP 850 (Sigma-Aldrich Co. LLC., USA). Adsorbents were treated prior to use by washing with 2-propanol for 15 min and drying in an oven at 60 °C for 12 h. The adsorbents characteristic are shown in Table 1.

2.2. Static adsorption and desorption test

The static adsorption-desorption process was carried out in an orbital shaker. Two grams of CPO was dissolved into 100 mL isopropyl alcohol (IPA). The sample solution and adsorbent (6.0 g) were mixed in a 250 mL conical flask and agitated at 200 rpm, at room temperature for 2 h. Adsorbent was filtered and the filtrate was taken for analysis. The adsorbent was further used for the 1st desorption process by adding 100 mL of ethanol into the conical flask and agitated for 2 h at room temperature. The adsorbent was filtered and the 1st filtrate was taken for analysis. The adsorbent was taken again and repeated for the 2nd and the 3rd desorption process, using the same amount of IPA and *n*-hexane, respectively. Each fraction was taken for the analysis. Adsorption and desorption efficiency (%) were then calculated based on Eqs. (1) and (2), respectively:

$$A = \frac{M_0 - M_e}{M_0} \times 100 \quad (1)$$

$$D = \frac{M_d}{M_0 - M_e} \times 100 \quad (2)$$

where A is the adsorption efficiency (%); D is the desorption efficiency (%); M_0 and M_e are the initial and equilibrium mass (μg) of the target component in the oil, respectively, and M_d is the target component mass (μg) in the oil after desorption.

2.3. Selective desorption process in Soxhlet extraction

CPO (5 g) was dissolved in 250 mL IPA. Diaion HP20 (15 g) was added into the solution and magnetically stirred at 200 rpm for 1 h at room temperature. Then, the IPA solution was removed from the adsorbent by vacuum rotary evaporation at 150 mbar and 60 °C. The CPO-filled adsorbent was packed with filter papers and placed into a 200 mL Soxhlet extractor for selective desorption process to yield three fractions according to desorption solvents as depicted in Fig. 1. Oil from each fraction was obtained after removal of solvent by vacuum evaporation at 150 mbar and 60 °C. Enrichment factor was calculated based on Eq. (3) to evaluate the efficiency of the method.

$$EF = \frac{C_a}{C_i} \quad (3)$$

where EF is the enrichment factor; C_a and C_i are the concentration of component in oil fraction and its initial concentration in CPO (μg/g), respectively.

2.3.1. Effect of methanol and ethanol as desorption solvents in the 1st fraction

The selective desorption process was carried out with a sequence of desorption solvents by using Soxhlet extractor as: methanol or ethanol was conducted at 90 °C (4 h) for obtaining the 1st fraction; IPA was conducted at 95 °C (2 h) for obtaining the 2nd fraction and *n*-hexane was conducted at 80 °C (4 h) for obtaining the 3rd fraction.

2.3.2. Effect of ethanol and IPA as desorption solvents in the 2nd fraction

The selective desorption process was carried out with a sequence of

Table 1
Characteristics and properties of adsorbents.

Adsorbent	Matrix	Polarity	Particle Size (μm)	Surface Area (m^2/g)	Pore Radius (Å)
Silica Gel	Silicon dioxide	Polar	40–60	450	60
Florisil	Activated magnesium silicate	Polar	150–250	500	86
Diaion HP2MG	Polymethacrylate	Weak Polar	297–707	500	170
Amberlite XAD-7HP	Acrylic	Weak Polar	590–710	380	300–400
Diaion HP20	Styrene-diviynlbenzene	Non Polar	250–850	500	260
Sepabeads SP850	Styrene-diviynlbenzene	Non Polar	250–850	1000	38