STUDIES ON CAROTENE RECOVERY FROM CRUDE PALM OIL BY ADSORPTION CHROMATOGRAPHY

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Introduction
Carotenes are the yellow to orange pigments found in carrots, leafy vegetables, milk fat and egg yolk. The alpha and beta-carotenes are associated with the synthesis of vitamin A in the liver and may offer some protection against cancer as well. Commercially, carotenes are used in food colouration, vitamin supplements, pharmaceutical and cosmetic products. Although palm oil is rich in carotenes (about 600 parts per million), carotenes are destroyed or discarded by bleaching and stripping operations in the oil refining process. Commercial production of carotenes by chemical conversion of palm oil results in loss of edible oil. Researchers in UPM have developed a physical method of carotene extraction without loss of edible oil. This method involves adsorption chromatography by synthetic polymer adsorbent. By employing this method, carotenes were concentrated up to 100,000 ppm in a two step chromatography operation. In this study, the effects of Crude Palm Oil (CPO) loading, mechanism of adsorption and FFA composition were investigated.

Materials and Methods
Crude Palm Oil (CPO) was obtained from Jomalina Sdn. Bhd. (Telok Panglima Garang, Malaysia). All solvents and chemicals used were of analytical grade. Synthetic porous polymer resin (Diaion HP-20) was obtained from Mitsubishi Chemicals (Tokyo, Japan). The chromatographic columns were glass tubes (3 cm i.d. by 35 cm length) with an outer jacket for circulating heated water. The adsorbent was slurried in the initial solvent and sonicated for 5 min in a Branson Sonicator (Danbury, CT) before packing into the glass tube to about 25 cm high and the column temperature was kept at 40 to 60°C. CPO, from 10 to 60 g, was dissolved in the heated initial solvent and then loaded onto the column bed. The initial solvent was 600 mL of isopropanol (IPA) and the second solvent was n-hexane at about 300 mL. Fractions were collected as IPA fraction and hexane fraction respectively. The oil content of each fraction was determined gravimetrically after removal of solvent by rotary evaporator. The carotene content was determined by spectrophotometric measurements. HPLC analyses was performed with a Shimadzu LC-10 apparatus. Fatty acid compositions were analysed by GLC method.

Results and Discussion
CPO loading on the column is an important condition because this largely affects carotene recovery. CPO loading was examined from 10 to 60 g at 40°C column temperature. Clearly, carotene recovery decreased with increasing CPO loading. The higher carotene recovery (85%) obtained at 10 g CPO loading dropped rapidly with a 60 g load to 22%. On the other hand, high oil recovery (89-96%) did not depend upon CPO loading. The carotene amount eluted by isopropanol reached 78% at 60 g CPO loading, in spite of the 15% level at 10 g CPO. These results suggest that carotene recovery depends mainly on two factors: (i) competitive adsorption between the oil and the carotene on the resin surface and (ii) the adsorption capacity of the resin for carotene in the presence of the solvent isopropanol. The adsorption capacity for oily materials (CPO and its fatty acid esters) on the resin has already been determined and a 0.2 to 0.5 g loading of oily materials is recommended. In this research, the capacity of resin at 10 g CPO loading was only 0.05 g per gram of resin and it was 0.3 at 60 g CPO. These differences depend on the kind of oily material, its loading quantity, the solvent system and column temperature. Carotene concentrations in the hexane fractions were affected by the amount of oil eluted at the same time, but carotene was concentrated to about 10 times when the CPO loading was below 30g. Thus, under the chromatographic conditions investigated, a 20 g loading was suitable for good recovery with a high concentration of carotene. HPLC analyses of carotene in the hexane fraction clearly showed almost the same pattern as that in CPO. The major components of the carotene fraction were similar to CPO, which contains α- and β-carotene. Carotenoids in CPO were almost equally concentrated in the hexane fraction. Fatty acid analyses were performed for the CPO and IPA fractions to check whether the fatty acid compositions were the same. The fatty acid compositions of both IPA and hexane fractions were almost similar to CPO data. These results indicate that the fatty acids of CPO do not change chemically during carotene recovery processes. The adsorption activity of the resin was determined in both IPA and hexane solvents. The resin tended to adsorb much more carotene in isopropanol than in hexane. The adsorption ratios of carotene by the resin were obtained as a percentage of carotene adsorbed to total carotene loaded, which were 93.7 and 43.7% in IPA and hexane respectively. The carotene that corresponds to the difference of these ratios can be recovered. The Langmuir adsorption model was found sufficient to describe carotene adsorption by the resin, suggesting that the process was favourable, saturable and equilibrated mechanism. A Scatchard transformation plot showed that the adsorption involves multiple binding sites.

Conclusions
HP-20 resin has been found to be a specific adsorbent for carotene and its adsorption process was favourable, saturable and equilibrated mechanism. A 20 g loading onto the column was found to be suitable with good recovery and a high carotene concentration. Fatty acid analyses showed that there was no chemical alteration to the oil during the recovery process. Carotene composition of the extract also showed that almost all carotenoids present in CPO was contained in it.

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