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# Pressurized liquid extraction and low-pressure solvent extraction of carotenoids from pressed palm fiber: Experimental and economical evaluation

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ABSTRACT

In this work, a comparison of Soxhlet extraction (LPSE–SOX), percolation (LPSE–PE) and pressurized liquid extraction (PLE) for the recovery of carotenoid-rich extracts from pressed palm fiber (PPF) was carried out in terms of yield, carotenoid profile and economic viability to evaluate the methods' industrial applicability. An optimization study was performed for each extraction technique with ethanol at a solvent/feed ratio of 20. The independent variables were temperature (35–55 °C), pressure (0.1–8 MPa) and flow rate (1.6, 2.4 g/min). The results showed that the global extraction yield obtained using LPSE–SOX (96 ± 4 mg extract/g PPF d.b.) after 6 h was higher than that obtained using LPSE–PE (74 ± 5 mg extract/g PPF d.b., 35 °C, 2.4 g/min) or PLE (44 ± 3 mg extract/g PPF d.b., 55 °C, 4 MPa, 2.4 g/min) after dynamic extraction time of 17 min under optimized conditions. On the other hand, the carotenoid yield obtained using PLE (305 ± 18 μg α-carotene/g extract and 713 ± 46 μg β-carotene/g extract) was higher than the obtained by LPSE–SOX (142 ± 13 μg α-carotene/g extract and 317 ± 46 μg β-carotene/g extract). PLE technique showed the highest selectivity for carotenoids than LPSE techniques. The lowest cost of manufacturing (COM) were obtained for LPSE–PE and PLE with values of US$13.4 and US$29.2/kg extract for a 0.5 $m^3$ vessel capacity.


## 1. Introduction

Current climate change interest and environmental preservation has promoted the development of sustainable technologies for the renewable exploited resources and the extracted and isolated bioactive compounds (O'Connor, 2013). Agricultural and agro industrial residues are renewable, low-cost and abundant resources. They could provide a wide range of value-added chemicals such as antioxidants, sugars, fibers, protein, phenolic compounds and lignin from a view of lignocellulosic-based biorefinery. These chemicals can be recovered by chemical, physical or biological treatments of these residues (Werpy et al., 2007; Cherubini, 2010; Babbar and Oberoi, 2014). One of the most abundant residues in Brazil is pressed palm fiber (PPF). It is obtained after the palm oil extraction process with an annual production of 123,000 tons in 2010 (Eisentraut, 2010). This residue presents significant amounts of carotenoids (4000–6000 ppm) (Choo et al., 1996).

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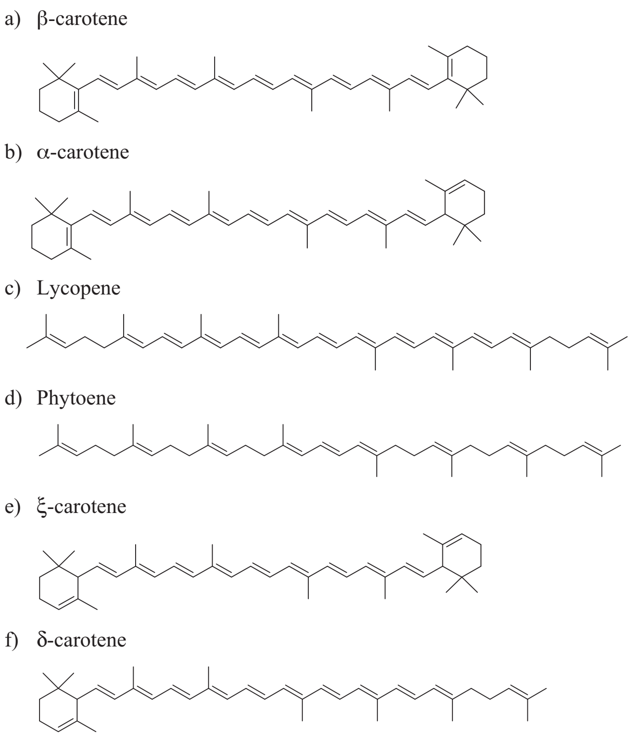


**Fig. 1 – Chemical structure of main carotenoid components in PPF (Choo et al., 1996; Barbosa-Filho et al., 2008).**

Some carotenoids have provitamin A activity and are of great interest for their various biological functions. They exhibit antioxidant and anti-inflammatory properties, prevent cell damage, premature skin aging, skin cancer and other cases related to the reduction of free radicals cancers (Minguez-Mosqueira et al., 2008; Meinke et al., 2013). A carotenoid quantification contained in the PPF residual oil showed that β-carotene, α-carotene, lycopene, phytoene, ξ-carotene and δ-carotene represented 80% of the total carotenoids, of which β-carotene was the major carotenoid found in the extract (Choo et al., 1996). Thus, the carotenoid-rich extract obtained from PPF contains mainly β-carotene and other highly beneficial carotenoids that can be included in functional foods as a natural alternative in food processing (Minguez-Mosqueira et al., 2008). Furthermore, the global market of carotenoids as high-value-added products reached US$1.2 billion in 2010, with an expected rise to US$1.4 billion by 2018; among these, β-carotene is the most prominent carotenoid, with a projected rise in consumption from US$261 million in 2010 to US$334 million by 2018 (BCC, 2011). Fig. 1 shows the chemical structure of the main carotenoids present in PPF.

Solid–liquid extraction of target compounds from plant materials is a unit operation that consists in the separation of solute (soluble compounds) from the vegetal matrix by using a liquid solvent. This operation involves three steps: (i) contact of the solvent with the vegetal matrix and transferring of the solute to the solvent, (ii) separation of the solvent from the matrix, and (iii) recovery of the solvent from the solute by evaporation or distillation (Lloyd et al., 2011). The extraction efficiency depends on the nature of the vegetal matrix and the solute to be extracted, the operational conditions of the extraction process such as pressure and temperature which modify the solvent physical properties to reduce the solvent surface tension, increase the solute's solubility and increment the solute diffusion rate (Mustafa et al., 2012).

Low-pressure solvent extraction (LPSE), which includes Soxhlet extraction (LPSE–SOX) and percolation (LPSE–PE), among others, uses solvents at low pressures (ambient pressure) for the selective dissolution of target compounds contained in the solid matrix by a liquid solvent. Several applications of LPSE in the food industry include the extraction of residual oil in vegetable oil processing with hexane, the extraction of lycopene from tomato peels and extraction of β-carotene from carrot by-products with ethanol which is recognized as a green solvent (Calvo et al., 2007; Takeuchi et al., 2008; Mustafa et al., 2012). Pressurized liquid extraction (PLE), also known as accelerated solvent extraction, is an emerging technology that uses liquid solvents such as hexane, ethanol and acetone to recover target compounds in a shorter extraction time compared to the LPSE process. Pressurized liquids have the advantage of enhanced solubility with increased temperature due to increased analyte diffusion from the solid matrix to the bulk solvent and the reduction of solvent viscosity, which facilitates the solvent's penetration into the matrix (Devanand et al., 2004). Experimental results from PLE and LPSE processes have differed in the recovery of carotenoids from different vegetal matrix sources. One study showed that PLE extraction presented a higher efficiency than LPSE extraction in the extraction of carotenoids from the green microalga *Chlorella vulgaris* (Cha et al., 2010). In addition, the PLE process required less time and consumed less solvent than LPSE in the recovery of β-carotene from carrots (Mustafa et al., 2012). Although the PLE process is a green extraction technique, there is no study for the carotenoids extraction from PPF using it. The extraction efficiency of carotenoids from pressed palm fiber was investigated in terms of global extraction yield and carotenoid yield. The global extraction yield refers to the amount of extract that can be recovered from a raw material over a specific time or S/F ratio (solvent mass/feed mass d.b. ratio) at a specified operational condition (Prado et al., 2013; Leal et al., 2010; Pereira and Meireles, 2010). The carotenoid yield refers to the carotenoid concentration in extract obtained after the extraction process. Therefore, the aim of this study was to compare the global extraction yield and carotenoid composition of extracts obtained by LPSE–SOX, LPSE–PE and PLE from PPF with ethanol as solvent. Subsequently, an economic evaluation of the PLE and LPSE processes was conducted using the software SuperPro Design 8.5®.

## 2. Materials and methods

### 2.1. Chemical and reagents

Ethanol (99.5%) was obtained from Chemco Ltda. (São Paulo, Brazil). Hexane (98.5%) was obtained from Dinâmica (São Paulo, Brazil). The analytical reagents used in carotenoid analysis, namely petroleum ether (≥99.5%), ethyl ether (≥99.5%), acetone (≥99.5%), methanol (≥98%) and potassium hydroxide (>90%), were obtained from Synth (São Paulo, Brazil). Acetonitrile (≥99.9%, HPLC grade) was obtained from JT Baker (New Jersey, USA). Methanol (≥99.9%, HPLC grade) was obtained from Merck (Darmstadt, Germany). Ultrapure water (18.2 mΩ) was obtained using a Direct-Q 3 UV ultrapure water system (Millipore Corporation, France). Magnesium oxide (97%) and Celite® Hyflo Supercel were obtained from Merck (Darmstadt, Germany).