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Concentration and purification of lycopene from watermelon juice by integrated microfiltration-based processes

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ABSTRACT

The concentration of thermosensitive bioactive compounds remains an important challenge. A gentle integrated process including enzymatic maceration, crossflow microfiltration, diafiltration and centrifugation was applied to watermelon juice to obtain a natural extract enriched with lycopene. Microfiltration was performed using ceramic membranes with different pore diameters (0.2 to 1.4 μm), transmembrane pressures (50 to 200 kPa) and temperatures (50 °C and 60 °C). Using the best operating conditions during microfiltration, the permeate flux was close to 110 $L \cdot h^{-1} \cdot m^{-2}$, and the lycopene concentration increased 11-fold in the retentate. Diafiltration allowed for the purification of lycopene up to 25-fold. Centrifugation enhanced the lycopene concentration up to 4-fold. *cis*-Isomerization remained low and similar to that of raw juice. This integrated process obtained a natural extract of lycopene that was 41 times more concentrated and 34 times purer than the initial juice, yielding an extract with up to 2% all-*trans*-lycopene on a dry basis.

1. Introduction

Lycopene is a carotenoid and an important lipo-soluble food component with health benefits. Numerous epidemiologic studies provide strong evidence that lycopene and its metabolites are active in several biological activities (Bramley, 2000; Cruz, González, & Sánchez, 2013; Waliszewski & Blasco, 2010; Wang, 2007). Anese, Mirolo, Fabbro, and Lippe (2013) reported that a number of epidemiological studies have concluded that lycopene-rich foods are associated with a lower risk of some degenerative diseases. Additionally, assays with living cells and dietary intervention have provided additional evidence that lycopene may play a specific role in preventing prostate cancer (Wertz, Siler, & Goralczyk, 2004).

The major diet intake of lycopene comes from the consumption of tomato and tomato products. Another dietary source in some countries is watermelon. Lycopene is responsible for the colour of red-flesh watermelon and is the main bioactive component (Wen'en, Pin, & Huihui, 2013).

According to Tarazona-Díaz, Alacid, Carrasco, Martínez, and Aguayo (2013), watermelon juice is naturally rich in lycopene and L-citrulline, an amino acid that is a very efficient hydroxyl radical scavenger and thus a strong antioxidant. Both compounds make watermelon an excellent option for designing functional foods. Perkins-Veazie and Collins (2006) reported that the concentration of lycopene in watermelon was approximately 50 $mg \cdot kg^{-1}$. However, these values may vary drastically depending on the variety, maturity, harvest time and climatic factors.

Lycopene ($C_{40}H_{56}$) is an open-polyene chain lacking the β-ionone ring structure with 13 double bonds. Lycopene is a powerful scavenger of singlet oxygen (a reactive oxygen-free radical precursor) (Shi, Dai, Kakuda, Mittal, & Xue, 2008). According to Boon, McClements, Weiss and Decker (2010), in fresh vegetable products, approximately 95% of lycopene is in all-*trans* conformation, which is thermodynamically the most stable form and exists in food matrices trapped in the pigment-protein complex localized in chromoplasts. However, lycopene isomers, such as 5 *cis*, 9 *cis*, 13 *cis* and 15 *cis*, have been reported in processed foods due to the effect of the application of high temperatures, exposure to light or oxygen incorporation. Although these forms are mostly found in plasma, for better absorption and stability it is advisable to avoid isomerization and to use soft-processing technologies (Van Buggenhout et al., 2010).

Crossflow microfiltration is a membrane process that is largely used to clarify or to stabilize without heating fruit juices, such as tomato juice (Razi, Aroujalian, & Fathizadeh, 2012) or even watermelon juice, and other plant extracts, such as roselle extract (Cissé, Vaillant, Pallet & Dornier, 2011). During the microfiltration of these products, hydrophobic compounds are generally retained by the membrane because they are associated with insoluble solids. Using this property, (Vaillant et al., 2005)

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and Abreu et al. (2013) applied microfiltration for the concentration of carotenoids from respectively melon juice and an aqueous extract of cashew apple. This process increased the carotenoid concentration in the retentate by 10-fold, obtaining a final product with high amount of carotenoids and with significant potential as a natural yellow colourant.

The aim of this paper was to propose and evaluate an environmentally friendly process (without organic solvent) of the concentration and purification of lycopene from watermelon juice. This process was based on a microfiltration step to obtain lycopene extracts without modifying its native structure.

2. Materials and methods

2.1. Raw material and processing

Watermelon fruits (*Citrullus lanatus* L.) were purchased from the local market in Clapiers (Hérault, France). After washing with tap water, they were disinfected with chlorinated water (200 mg·kg^{-1} active chlorine), cut and peeled manually and then subsequently refined in a horizontal pulper Auriol PH3 (Marmande, France) with 0.5 mm mesh. The average mass of the extracted watermelon juice was 62%. The epicarp and seeds represented 30% and 8% of the final product, respectively. The juice was stored at −18 °C until processing. Before each test, the juice was macerated at 40–45 °C for 45 min with 100 mg·kg^{-1} of the commercial enzyme blend Ultrazym AFP-L or Pectinex Ultra SP-L (Novozymes, Bagsvaerd, Denmark). According to the supplier, these preparations are obtained from selected strains of *Aspergillus niger* and *Aspergillus aculeatus* and mainly consist of cellulase and pectinase. They have already been used successfully in the processing of a large variety of fruit pulps (Silva, Della, Penha, da Matta & Corrêa, 2005; Laorko, Li, Tongchitpakdee, Chantachum, & Youravong, 2010; Nattaporn & Pranee, 2011; Machado, Haneda, Trevisan, & Fontes, 2012).

Two processes for the purification and concentration of lycopene from watermelon juice were evaluated. In the first process, the juice was microfiltered, and subsequent centrifugation was performed with the obtained retentate. In the second process, a diafiltration step with pure water was included prior to centrifugation in order to increase the purity of the final extract (Fig. 1).

2.2. Equipment used

Microfiltration experiments were performed in pilot equipment (3 L feeding tank) manufactured by TIA (Bollène, France) with 4 tubular α-alumina membranes in series (Membralox T1–70 (Pall Exekia, Bazet, France)), each with a 55 cm^2 effective area. The temperature was between 50 and 60 °C, and the crossflow velocity was 6 m·s^{-1} (Fig. 2). The pore diameter and transmembrane pressure for the microfiltration process were selected according to the results of the

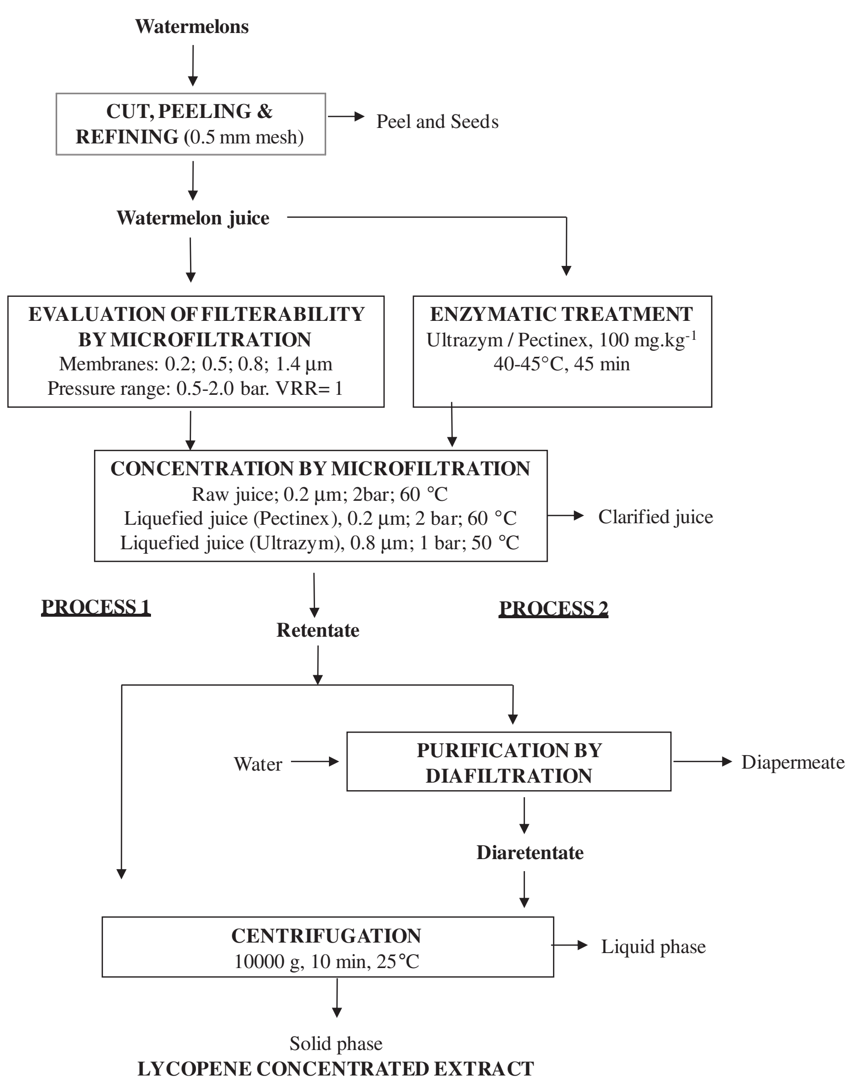

Fig. 1. Flow diagram for the lycopene concentration and purification processes.