ENRICHMENT OF LYCOPENE IN WATERMELON JUICE BY ULTRAFILTRATION: TECHNICAL ASSESSMENT AND FOULING ANALYSIS

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Abstract. Watermelon juice is proven to be a potential source of lycopene, but its exploitation and consumption are currently constrained. The research focused on lycopene enrichment in watermelon juice by ultrafiltration (UF) with 1000 Da of molecular weight cut-off (MWCO). The selectivity of UF membrane for the constituents in watermelon juice was investigated from 2 to 8 bar by dead-end mode combined with stirring. The 1000 Da UF membrane showed a high potential to recover and separate lycopene from other components. However, membrane deposition and capillary occlusion could occur, affecting the overall efficiency of the enrichment process. To better understand kinetics, phenomena and efficiency, the study was evaluated in cross-flow mode with full recirculation of retentate. The enrichment efficiency was found to increase the concentration and purity of lycopene by several times.

Keywords: watermelon juice, lycopene, ultrafiltration, membrane separation, membrane fouling, concentration, carotenoids.

Introduction. Watermelon (*Citrullus lanatus*) is an important horticultural crop characterized by its high moisture content, pleasant sensory properties, and considerable nutritional value. In addition to water and soluble sugars, watermelon contains bioactive compounds, particularly lycopene, which contributes to the characteristic red color of the fruit. Lycopene is a lipophilic carotenoid with a conjugated polyene structure that gives it strong light-absorbing properties and contributes to its antioxidant activity. Consequently, watermelon juice may serve as a valuable raw material for developing natural colorants, functional beverages, and ingredients for the food-processing industry.

Despite its potential, the industrial utilization of watermelon as a source of concentrated lycopene remains technologically challenging. Fresh watermelon juice contains a large proportion of water, soluble carbohydrates, organic acids, minerals, suspended particles, and other naturally occurring constituents. These components complicate the selective recovery of lycopene and may influence the stability, purity, and quality of the final concentrate. Conventional concentration methods, particularly those involving

elevated temperatures, may also affect heat-sensitive compounds, color, and sensory characteristics if processing conditions are not adequately controlled.

Membrane separation technologies have attracted considerable interest in food processing because they can separate or concentrate selected components through controlled transport across a semipermeable barrier. Depending on membrane characteristics and operating conditions, microfiltration, ultrafiltration, nanofiltration, and reverse osmosis can be applied to different stages of food processing. Ultrafiltration is particularly useful for separating macromolecular substances and colloidal particles from smaller dissolved components. Its performance depends on membrane material, pore structure, molecular weight cut-off, transmembrane pressure, feed composition, temperature, and hydrodynamic conditions.

The application of ultrafiltration to lycopene enrichment requires consideration of the physicochemical properties of the target compound. Lycopene has a molecular mass of approximately 537 g/mol and is poorly soluble in water. Therefore, its behavior during filtration cannot be predicted from molecular mass alone. In aqueous watermelon juice, lycopene may be associated with suspended particles, cell fragments, lipid-containing structures, and other colloidal components. These associations may influence whether the pigment is retained by the membrane or transported through it.

A membrane with a molecular weight cut-off of 1000 Da provides a potentially restrictive separation barrier for the aqueous juice matrix. Nevertheless, the MWCO value is a nominal membrane characteristic rather than an absolute molecular-size threshold. Lycopene retention depends not only on the membrane's nominal selectivity but also on the physical state of the pigment, its interactions with other juice constituents, and the development of deposits on the membrane surface.

The main objective of this study is to assess the technological potential of a 1000 Da ultrafiltration membrane for enriching lycopene in watermelon juice and to analyze the influence of operating pressure and filtration configuration on separation performance and membrane fouling. The study considers dead-end filtration combined with stirring at pressures ranging from 2 to 8 bar and cross-flow filtration with full recirculation of the retentate. This approach provides a basis for understanding the relationship between membrane selectivity, hydrodynamic conditions, concentration efficiency, and operational stability.

MATERIALS AND METHODS: ULTRAFILTRATION AND MEMBRANE FOULING ANALYSIS

Watermelon juice is the primary raw material for lycopene enrichment. To obtain reproducible filtration results, the feed should be prepared under controlled conditions, including standardized juice extraction, homogenization, and removal of coarse particles where appropriate. The preparation procedure must be documented because differences in particle size, pulp content, and initial lycopene concentration can substantially influence membrane performance.

Before filtration, the juice should be characterized in terms of pH, soluble solids, turbidity, total suspended solids, and lycopene concentration. These parameters provide essential information about feed composition and allow the relationship between raw-material properties and filtration behavior to be evaluated. Samples should be protected from excessive light and unnecessary exposure to oxygen to minimize changes in pigment content during handling.

A 1000 Da molecular weight cut-off ultrafiltration membrane is selected for the separation process. Membrane characterization should include its material, effective filtration area, operating temperature range, and recommended pressure limits. The membrane must be conditioned according to the manufacturer's instructions before experimentation. Appropriate cleaning procedures and blank-water permeability measurements should be performed to establish a reference for evaluating subsequent flux decline.

2.1. Dead-end ultrafiltration with stirring

In the dead-end configuration, watermelon juice is directed toward the membrane surface, while the permeate passes through the membrane and the retained constituents accumulate on the feed side. Stirring is used to improve mixing near the membrane surface and reduce, to some extent, the formation of a highly concentrated boundary layer.

The filtration process is evaluated at transmembrane pressures of 2, 4, 6, and 8 bar, provided that these conditions fall within the membrane manufacturer's specified operating limits. The selected pressure range allows the influence of pressure on permeate flux, lycopene retention, and deposit formation to be investigated. At each pressure, the permeate volume should be recorded over time, and samples of both the permeate and retentate should be collected for compositional analysis.

Permeate flux is calculated using the following equation:

$$J = V / (A \times t)$$

where J is the permeate flux, V is the volume of collected permeate, A is the effective membrane area, and t is the filtration time.

The flux response to increasing pressure is an important indicator of process behavior. Under favorable conditions, increasing transmembrane pressure increases the driving force for liquid transport. However, this relationship may become nonlinear when concentration polarization, surface deposition, or membrane pore blockage becomes significant. Consequently, higher pressure does not necessarily produce a proportional increase in filtration productivity.

2.2. Cross-flow ultrafiltration with full retentate recirculation

Cross-flow ultrafiltration is investigated to assess whether improved hydrodynamic conditions can limit membrane fouling and support more stable separation. In this configuration, watermelon juice flows tangentially across the membrane surface. The retentate is continuously returned to the feed reservoir, while the permeate is collected separately. Full retentate recirculation allows the retained fraction to remain within the system during the experiment.

Compared with dead-end filtration, cross-flow operation generates shear forces along the membrane surface. These forces can reduce the accumulation of suspended particles and partially mitigate concentration polarization. However, the effectiveness of cross-flow filtration depends on circulation velocity, feed viscosity, membrane geometry, operating pressure, and the tendency of the juice constituents to form deposits.

The filtration system should be operated under controlled and documented conditions. Relevant variables include transmembrane pressure, cross-flow velocity, temperature, filtration duration, recirculation rate, and the initial volume of feed. The collected permeate and final retentate should be analyzed to determine their respective lycopene concentrations and total volumes.

The enrichment factor can be calculated as:

$$EF = C_r / C_0$$

where EF is the enrichment factor, C_r is the lycopene concentration in the retentate, and C_0 is the initial lycopene concentration in the feed.

Lycopene recovery in the retentate can be expressed as:

$$R_r\ (\%) = (C_r \times V_r) / (C_0 \times V_0) \times 100$$

where V_r is the final retentate volume and V_0 is the initial feed volume. This mass-balance-based calculation is essential because an increase in concentration does not necessarily mean that a high proportion of the initial lycopene has been recovered.

The apparent lycopene retention can also be evaluated by comparing its concentrations in the retentate and permeate. However, a complete assessment should include pigment recovery, losses to membrane deposits, and analytical uncertainty. If the objective is to determine purity, the lycopene content should be expressed relative to the total mass of the relevant concentrate or dry matter, with the chosen definition clearly specified.

2.3. Analytical assessment and fouling mechanisms

Lycopene concentration should be measured using a validated analytical method, such as spectrophotometry or high-performance liquid chromatography (HPLC). HPLC is particularly useful when the separation of lycopene from other carotenoids and potentially interfering compounds is required. The analytical protocol should specify extraction conditions, calibration procedures, wavelength or chromatographic detection settings, and the basis for reporting concentration.

Membrane fouling is evaluated by monitoring permeate flux as a function of time and comparing membrane permeability before and after filtration. Several mechanisms may contribute to the decline in filtration performance.

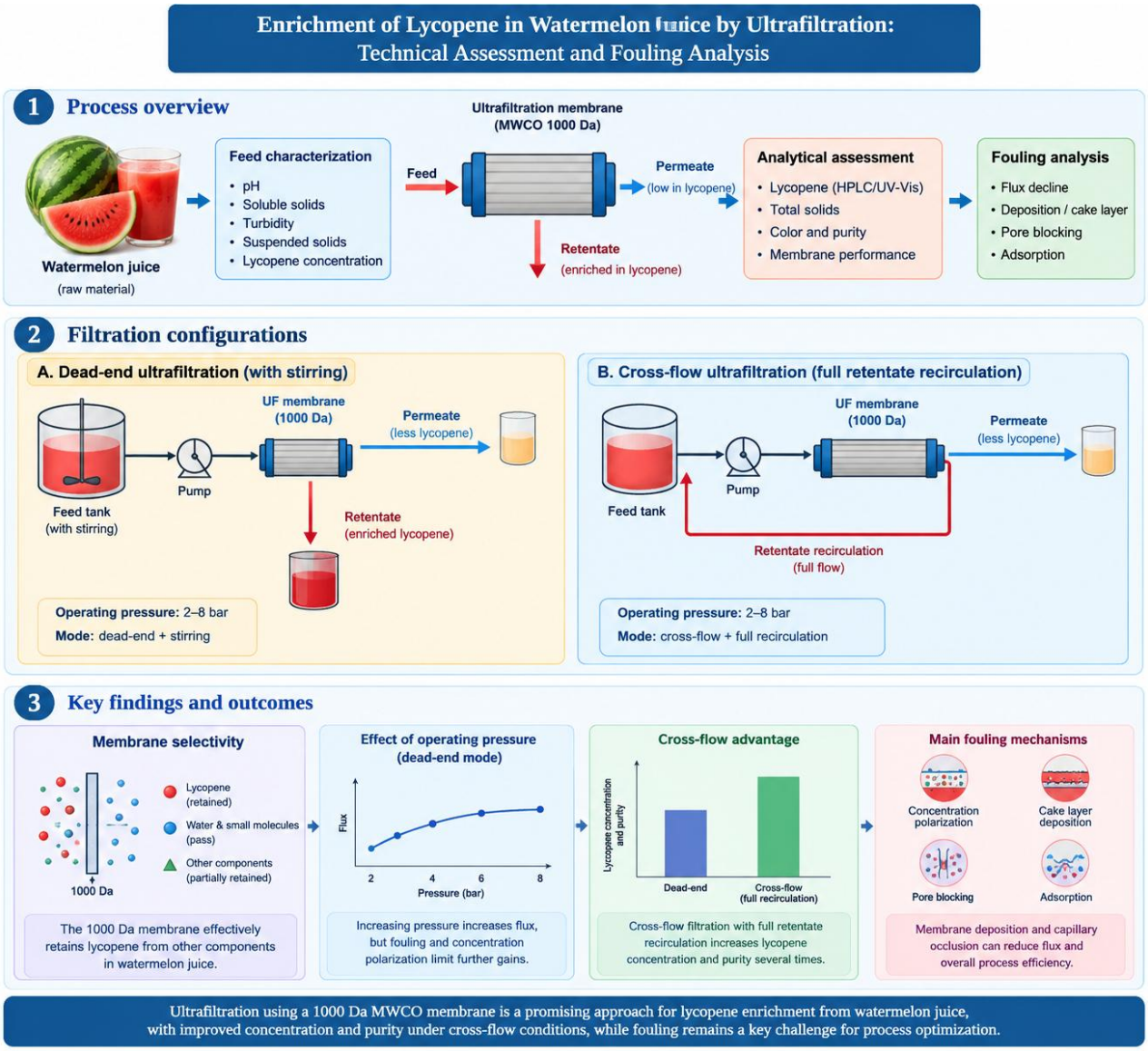
First, concentration polarization occurs when retained compounds accumulate near the membrane surface, producing a local concentration gradient. Second, surface deposition may form a cake layer containing suspended particles and pigment-associated material. Third, pore blocking may occur when sufficiently small constituents enter or obstruct membrane pores. Finally, adsorption of organic compounds onto the membrane material may change its surface properties and hydraulic resistance.

A simplified resistance-in-series model can be used to interpret flux decline:

$$J = \Delta P / [\mu(R_m + R\boldsymbol{f})]$$

where ΔP is the transmembrane pressure, μ is the dynamic viscosity of the feed, R_m is the intrinsic membrane resistance, and $R\boldsymbol{f}$ represents the additional resistance associated with fouling and deposition.

This model provides a useful conceptual framework for distinguishing the clean-membrane resistance from the additional resistance developed during filtration. To identify individual fouling mechanisms more confidently, flux measurements should be supplemented with membrane cleaning tests, microscopic observations, or other suitable surface-characterization techniques.



3. RESULTS AND DISCUSSION: TECHNICAL PERFORMANCE, SELECTIVITY, AND PROCESS OPTIMIZATION

The technical assessment of ultrafiltration for lycopene enrichment must consider both separation efficiency and operational stability. The reported research indicates that a 1000 Da MWCO membrane has potential for retaining and separating lycopene from other watermelon juice constituents. The use of both dead-end and cross-flow configurations enables the influence of hydrodynamic conditions on this separation process to be examined.

In dead-end filtration, increasing pressure from 2 to 8 bar increases the driving force for permeate transport. Nevertheless, the actual increase in flux depends on the extent of concentration polarization and membrane fouling. At relatively low pressure, liquid transport may be limited by the available driving force. At higher pressure, the accumulation and compaction of deposits may become more pronounced, increasing hydraulic resistance. Therefore, pressure optimization should be based on measured permeate flux, lycopene retention, recovery, and process stability rather than pressure alone.

The presence of lycopene in a retentate fraction is technologically important because it may permit the production of a pigment-enriched ingredient from a large volume of dilute juice. However, the apparent selectivity of a 1000 Da membrane should be interpreted in relation to the physical state of lycopene. Since the pigment is hydrophobic and poorly soluble in water, its retention may be associated with colloidal

structures and suspended matter rather than exclusively with direct molecular-size exclusion. This distinction is essential when explaining the separation mechanism and assessing whether the same membrane performance can be expected for watermelon juice prepared under different conditions.

A further consideration is the separation of lycopene from soluble sugars, organic acids, and mineral components. These low-molecular-mass constituents may pass through an ultrafiltration membrane more readily than suspended or colloid-associated material. As a result, the retained fraction may become enriched in lycopene relative to some other constituents. Nevertheless, enrichment and purity are not equivalent. A higher lycopene concentration in the retentate does not automatically demonstrate higher lycopene purity unless the proportions of other retained substances are also quantified.

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