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Role of pectin and haze particles in membrane fouling during cold microfiltration of apple cider

Dongjun Zhao, Evonne Lau, Olga I. Padilla-Zakour, Carmen I. Moraru*

Department of Food Science, Cornell University, USA

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ABSTRACT

The role of pectin in membrane fouling during cold MF of apple cider was investigated. Clarified apple juice with four different concentrations of pectin was subjected to cold MF, at pore sizes above 0.45 μm. The experimental data shows that pectin plays a significant role in membrane fouling, and its fouling effect increased with increasing concentration. The association of pectin with polyphenols and proteins, which results in colloidal haze particles with low surface electrical charge, seems to be the major factor in fouling during cold MF of apple cider. Depectinization was beneficial to MF with pore sizes below 0.45 μm, for which fouling is dominated by cake layer formation. Depectinization had a negative effect on MF flux for pore sizes above 0.8 μm, since the size reduction of haze particles accentuated pore constriction and blockage, which dominate fouling in large pore MF. These findings have practical implications for the development of efficient, commercially viable, cold MF processes for minimally processed apple cider.

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1. Introduction

Microfiltration (MF) is a pressure-driven membrane process that can be used to clarify liquid streams by removing suspended solids larger than the membrane pore size. MF is used in the manufacturing of apple juice primarily as an alternative to the conventional clarification methods such as filtration using sheets and diatomaceous earth. In addition to clarification, MF can remove many of the microorganisms of concern from the unfiltered apple juice (also known as "apple cider" in the US), including bacteria, yeast, mold and protozoa (Zhao et al., 2015).

A significant issue that limits the commercial use of membrane filtration is membrane fouling. In MF, membrane fouling mechanisms include pore constriction, pore blocking and cake layer formation (de Barros et al., 2003). Feed particles smaller than the membrane pore openings can get adsorbed onto the internal membrane channels and cause pore constriction, thus reducing the effective diameter of the pores. When particle diameters approach the effective membrane pore size, pore blocking can occur due to a convective drag of particles toward the membrane created by the permeate flux, and particles are adsorbed and/or deposited onto the membrane pores and surface. When particles are larger than membrane pores, they can be retained in a cake layer onto the membrane surface. A study that investigated the effect of pore size in membrane fouling during MF of apple cider revealed that the predominance of one of these mechanisms depends on the membrane pore size (Zhao and Moraru, 2015).

In membrane filtration of apple cider, haze particles and pectin are believed to have a significant role in membrane fouling. While pectin is considered to be the individual haze component that contributes the most to membrane fouling, the structure and interactions of the haze components are most critical to fouling (Riedl et al., 1998; Su et al., 1993). Colloidal haze particles in apple cider are formed of proteins, polyphenols and pectin (Beveridge and Wrolstad, 1997; Siebert, 2009). Proline-rich haze active proteins bind to polyphenols by hydrogen bonds and hydrophobic interactions (Siebert et al., 1996). Pectin can also interact with polyphenols via hydrogen bonds, hydrophobic and electrostatic interactions (Le Bourvellec et al., 2009).

Much of the data available on the MF of apple cider focuses on MF with a pore size of 0.2 μm, also known as sterilizing MF. Membranes with such small pore sizes retain some of the components that contribute to juice color and flavor, such as pectin and chemical compounds, thus stripping the final product of some of its most desirable properties (Wu et al., 1990). Therefore, using larger membrane pore sizes for clarification and microbial removal could

* Corresponding author.
E-mail address: cim24@cornell.edu (C.I. Moraru).

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result in increased retention of the nutritional, color and flavor components of the juice, as well as a potentially higher permeate flux as compared to 0.2 μm membranes. Nonetheless, the fouling mechanisms during large pore, cold MF of apple cider have not been yet fully elucidated. This study focuses specifically on understanding the role of haze particles and pectin in membrane fouling during large pore cold MF of apple cider. This can provide an insights into ways to mitigate fouling and make large pore size MF of apple cider a commercially viable process.

2. Materials and methods

2.1. Apple cider

Raw apple cider was obtained from Cornell Orchards (Ithaca, NY) and stored at 4 °C for a maximum of two weeks prior to use. Due to seasonality, some apple cider was stored frozen and thawed before being processed.

2.2. Pectin addition experiments

To evaluate the role of pectin in membrane fouling, a “base juice” was obtained by clarifying raw apple cider using a 0.2 μm pore size MF membrane, which helped remove most of the suspended solids. Apple pectin with molecular weight (MW) of 30,000–100,000 (Sigma Aldrich, St. Louis, MO) was added to this clarified juice, in a range of concentration typically found in apple cider. The target concentrations used were 0.05%, 0.10%, 0.15% and 0.20% w/w, denoted as concentration levels I to IV.

2.3. Pectinase treatments

The effect of pectin hydrolysis on fouling during apple cider MF was evaluated by conducting pectinase treatments on apple cider prior to MF. Three pectinases were pre-screened by monitoring their effect on the particle size in apple cider: commercial pectinase blend ClariSEB RL (Specialty Enzymes & Biotechnologies, CA), pectolyase (E.C. 3.2.1.15) (PL) from *Aspergillus japonicus* and polygalacturonase (E.C. 3.2.1.15) (PG) from *Aspergillus niger* (Sigma Aldrich, St. Louis, MO). PG was selected for further use. PG at a level of 0.012% (w/w) was added to raw cider, and the PG treated cider was kept for 6 days at 4 °C prior to MF processing.

2.4. Microfiltration processing

A pilot scale MF unit consisting of a 50 gal (189.3 L) feed tank connected to a variable-speed centrifugal pump, a tubular heat exchanger and ISOFLUX™ tubular ceramic membrane of TAMI design (GEA Filtration, WI) placed inside a stainless steel housing was used. The membrane had an outside diameter of 25 mm, length of 1200 mm, 23 internal channel of 3.5 mm hydraulic diameter each, and a membrane surface area of 0.35 m^2. The membrane pore sizes (the nominal pore diameter per the membrane manufacturer) used in this study were 0.2 μm (used only for clarification prior to pectin addition), 0.45 μm, 0.8 μm and 1.4 μm.

Feed inlet pressure (P_1), retentate outlet pressure (P_2) were recorded, and transmembrane pressure (TMP) was calculated as:

$$TMP = \frac{(P_1 + P_2)}{2} - P_p \quad (1)$$

where permeate pressure (P_p) is the atmospheric pressure.

All pressures were expressed in kPa.

The permeate flux data was obtained gravimetrically using an electronic scale. Permeate flux (J) was calculated as:

$$J = \frac{M}{A \times t \times \rho} \quad (2)$$

where: J: permeate flux (L/m^2h); M: amount of permeate (kg) collected in the time interval t (hours); A: surface area of the membrane (m^2); ρ: density of the permeate at the filtration temperature (kg/m^3). The initial flux value was taken at 2 min after starting the pump, and every 5 s thereafter. To eliminate the “noise” generated by the multitude of data points, one point every 100 s was used when plotting the flux data.

The relative flux (J/J_0) was calculated as follows:

$$Relative\ flux = \frac{J}{J_0} \times 100\% \quad (3)$$

where: J: permeate flux at any time point (L/m^2h); J_o: initial flux (L/m^2h).

This normalized parameter allows direct comparisons among MF experiments that have different permeate flux values. A lower J/J_0 indicates a more pronounced fouling of the membrane than a higher J/J_0 value.

The MF process was conducted at a cross-flow velocity of 5.5 m/s and a TMP of 159 kPa, which were selected based on optimized conditions from a previous study (Zhao and Moraru, 2015). MF was conducted at a temperature of 6 ± 1 °C, which was maintained by circulating chilled water in the counter current tubular heat exchanger. This temperature was chosen to preserve the quality of the juice and minimize browning during processing. Running MF cold also keeps the potential of a cold pasteurization process as an alternative to the conventional heat pasteurization (Zhao et al., 2015).

2.5. Membrane cleaning

After each MF run, a chemical cleaning cycle was carried out. The cleaning procedure consisted of a rinse with reverse osmosis (RO) water for 10 min, followed by alkaline cleaning with Ultrasil-25 at a concentration of 20 g/L at 80 °C for 30 min and a second RO water rinse for 10 min or until neutrality. Acid cleaning with 5 mL/L HNO_3 at 50 °C for 20 min was then performed, followed by a third RO water rinse for 10 min or until neutrality. The effectiveness of cleaning and change in the membrane performance with time were monitored by determining the water flux of the clean membrane.

2.6. Physico-chemical analysis

The physico-chemical properties of the product were measured before and after MF.

pH was measured at 20 °C using a Fisher Scientific Accumet Excel XL20 pH meter (Fisher Scientific, Pittsburgh, PA).

Soluble solids content (°Brix) was measured with a MISCO® digital probe refractometer (MISCO® Products Division, Cleveland, OH), at room temperature.

The *suspended insoluble solids (SIS)* content was determined by centrifuging 10 mL of apple cider or juice at 2200 ×g for 15 min. After discarding the supernatant, the pellet, representing the SIS, was weighed and SIS calculated in g/L (Vaillant et al., 2008). All measurements were carried out in duplicate.

Viscosity was measured at 6 °C using a Brookfield DV-II+ Pro viscometer with a ULV adapter (Brookfield Engineering Laboratories Inc., Middleboro, MA). Measurements were performed in triplicate.

Turbidity was measured using a 2020wi turbidimeter (LaMotte Company, Chestertown, MD) in Formazin Nephelometric Units (FNU). Measurements were performed in triplicate.

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