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Whey recovery using forward osmosis – Evaluating the factors limiting the flux performance

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ABSTRACT

The focus of whey processing has been changed from waste treatment to the production of valuable products owing to the advancement of membrane technology. Producing high quality whey products at low energy consumption entails novel techniques for whey concentration. This study proposes forward osmosis (FO) as a potential approach to concentrate whey protein solutions, which is superior to the concentration via reverse osmosis (RO), mainly owing to the low hydraulic pressure during FO process. In particular, this work validated the feasibility of FO-based whey concentration using high-performance hollow fiber membranes that were fabricated in-house. The investigation focused on the effects of various operating conditions on the concentration performance. The experimental results reveal that optimal concentration efficiency would be achieved by appropriately choosing the cross-flow velocity, draw solution concentration and the maximum attainable concentration of whey protein solution. Especially, the flux decline behaviour as a function of the whey protein concentration in the feed solution indicated rapid formation of a polarized or gel foulant layer at the membrane surface. The proposed mechanism was supported by the investigation of physical cleaning with water, showing that the post-process cleaning after the concentration cycle was able to recover the membrane performance, whereas the intermediate cleaning during one concentration cycle had little impact on the concentration efficiency enhancement. This study has practical significance for the application of FO in the concentration of dairy streams and other liquid foods.

1. Introduction

Whey processing is one of the most important sectors in dairy industry. Over the years, whey processing has transformed from waste treatment to the production of valuable products such as whey protein concentrates. This change is to a great extent attributed to the advances of membrane technology and its application in the field of dairy product processing [1,2]. Pressure driven membrane processes, including reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF), have been used to remove bacteria from whey, concentrate and demineralize whey, fractionate whey proteins, and recover proteins from dairy industry process waters, and others [2–6]. In addition to low pressure UF, relatively high pressure driven NF and RO are also reported as a tool for recovering proteins in whey processing [2,6], where higher rejection of small solutes and salts can be achieved. However, the high pumping energy and the high viscosity of the liquors limit the maximum attainable concentration factor [2,5]; moreover, the irreversible fouling under high pressure usually renders membrane flux recovery difficult despite of cleaning [7,8].

As an osmotically driven process, forward osmosis (FO) utilises a concentrate draw solution (DS) and a dilute feed solution (FS) that are separated by a semi-permeable membrane, where the osmotic pressure difference drives the water to flow from the FS to the DS. In contrast to the pressure driven membrane processes, the minimal pressure requirement in FO operation is more advantageous for treating viscous liquor with high solid content [9]. In addition, previous work revealed that FO had a relatively low fouling tendency and rarely resulted in irreversible fouling [10,11]. Therefore, FO has recently been gaining more research interests in low-energy separation processes [9,12–15], including liquid foods concentration [16–22].

The conceptual design of using FO for liquid foods concentration dates back to 1960s [23], but it attracted substantial research interests only a decade ago due to the recent advancement in FO membrane fabrication [18,22,24]. A few studies on utilizing FO for the concentration of fruit juices demonstrated that FO could treat high solid content liquids in lab-scale tests [16,18,25]. Nevertheless, relatively low water

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fluxes (e.g., below 10 L/m^2 h) were obtained in most of these studies [16,18,21,26] in spite of the highly concentrated DS used (e.g., NaCl of 4–6 mol/L). Low permeate fluxes would result in a low concentration efficiency, which may render these applications unrealistic. Another concern on the FO-based concentration is the solute leakage from the DS to the liquid food, which can deteriorate the food taste and cause health problems [18,23,27]. Therefore, in addition to the selection of appropriate draw solutes, a high performance membrane exhibiting high FO water flux and low solute flux is the prime requirement for the FO-based concentration.

Only a few investigations have been reported on the utilization of FO for dairy products concentration [1,28,29]. All these studies were performed with flat-sheet cellulose triacetate FO membranes, which yielded lower water flux than that obtained from the recently developed thin film composite (TFC) polyamide FO membranes [30,31]. Although the influencing factors such as temperature, cross-flow velocity (CFV) and membrane orientations have been investigated [29], the dominant factors accounting for the severe flux decline during whey concentration remain unclear. Moreover, the ineffectiveness of membrane cleaning during whey concentration reported in prior work [1] deserves a further investigation.

The objectives of current study were to demonstrate the feasibility of FO application in dairy industry by carrying out whey protein concentration experiments using an in-house fabricated TFC hollow fiber FO membrane with high salt rejection, and to understand how the operating conditions affect the FO flux performance during the whey protein concentration. In particular, the effect of feed solution concentration alone was isolated from the long term fouling effect for better understanding the causes of flux drop. The physical cleaning with water was also investigated to verify its effectiveness in different cleaning modes.

2. Materials and methods

2.1. Chemicals and solution chemistry

All reagents and chemicals were used as received. Sodium chloride (NaCl) was used for the preparation of the draw solution (DS) and feed solution (FS). Ultrapure water with a resistivity of 18.2 MΩ.cm (Milli-Q water, Millipore Integral system) was used to prepare all working solutions. The whey protein adopted in this study was received from Optimum Nutrition (USA) in powder form. As shown in Table 1, the powder consists of mainly proteins (81.6%) and a small quantity of carbohydrate, fat and electrolytes. For simplicity, the term “whey protein concentrate (WPC)” in this study was referred to as a mixture whose key component was whey protein (in spite of a small amount of other contents). The WPC solution was prepared by adding the powder to Milli-Q water to form a suspension. The WPC solution of 6% (w/v%) solid content was used for the whey protein concentration experiments. It had a total organic carbon (TOC) content of ~31.84 g/L. The solution conductivity and osmolarity were ~1150 μS/cm and ~20 mOsm/L, respectively, which are equivalent to those of 10 mM NaCl solution. Hence, 10 mM NaCl solution was used as the control FS for baseline tests (i.e., the tests in the absence of whey proteins). The DS was 0.5 M NaCl solution unless otherwise specified. The pH of all the solutions in the current study was unadjusted (~pH 6).

Table 1
Composition of whey protein powder and prepared WPC solution.

Compositions	Dry weight basis (%)[a]	WPC solution (6% solid content) (g/L)
Protein	81.6%	48.96[a]
Carbohydrate	10.2%	6.12[a]
(Sugar content)	(3.47%)	(2.04)[a]
Fat	3.4%	2.04[a]
TOC	–	31.84[b]
Na	0.204	0.122[a]
K	–	0.250[c]
Ca	–	0.193[c]

Note:
[a] Given by supplier.
[b] TOC measurement (TOC-V_{CSH}, Shimadzu).
[c] Analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES). Whey protein powder was added to Milli-Q water and the suspension was filtrated with 0.45 μm membrane. The membrane permeate was collected for the ICP analysis.

2.2. FO membrane

An in-house fabricated hollow fiber FO membrane was used in the current study. It is a thin film composite (TFC) membrane, consisting of a polyethersulfone (PES) substrate and an ultrathin polyamide active layer. The detailed fabrication method of this membrane can be found in our previous study [32]. In brief, a polyamide skin layer was formed on the inner surface of a PES hollow fiber substrate via interfacial polymerization. The inner and outer diameters of the fibers were about 0.98 mm and 1.34 mm. The hollow fiber membrane module used for both FO and RO experiments consisted of 15 fibers and had a total active area of 106 cm^2. The water permeability (A) and salt permeability (B) were measured in a cross-flow RO setup. Details on the calculation of A, B value and structural parameter (S) are provided in Appendix A.

2.3. FO concentration experiments and cleaning

FO concentration experiments were carried out using a bench scale cross-flow FO system similar to what we have reported previously [33,34]. The FS (3-L) and DS (8-L) were circulated by two peristaltic pumps (Cole-Parmer, USA) in a counter-current cross-flow mode. The FS tank was placed on a digital balance (Mettler Toledo, Germany) that was connected to a computer for water flux acquisition. In all the whey protein concentration experiments, the membrane active layer was facing the FS (i.e., in the active layer-facing-FS (AL-FS) orientation, see Appendix B), which is also called the FO mode. The FS conductivity was monitored using a conductivity meter (Thermo Scientific). The cross-flow velocity (CFV) in the DS flow channel (shell side) was maintained at ~22 cm/s in all the tests, while the CFV in the FS flow channel (lumen side) was 55 cm/s or 15 cm/s. All the FO concentration experiments were performed at room temperature of 22.5 ± 1.5 °C.

Physical cleaning was carried out immediately at the end of each concentration cycle (post-process cleaning) or at the end of each stage within a concentration cycle (intermediate cleaning). In both cleaning modes, the fouled membranes were flushed with Milli-Q water at a CFV of 55 cm/s in the feed fluid channel for 30 min. After the cleaning, the FO concentration experiment was resumed using the previous FS or a newly prepared FS (i.e., WPC solution of 6% solid content). The former case is referred to as intermediate cleaning while the latter is the post-process cleaning. More details about the two cleaning modes will be explained in Section 3.5.

2.4. Experiments of concentration via RO

For comparison purposes, whey protein concentration using cross-flow RO was also performed. The hollow fiber FO membrane module used for this RO-based concentration had the same active area as the ones used for the FO experiments. The 3-L FS was circulated at a CFV of 15 cm/s using a gear pump (Cole-Parmer, USA). The applied pressure was maintained at 5 bar to attain 13 L/m^2 h water flux (similar to the FO flux) with 10 mM NaCl feed solution (see Appendix D). The feed temperature was controlled at 22.5 ± 1 °C using a water bath with a temperature controller (PolyScience, USA). The RO permeate was collected in a container without recycling to the feed tank. The permeate flux was measured using a digital mass flow meter (Brooks,