Bioresource Technology 101 (2010) 3859–3863

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Nutrient recovery from the dry grind process using sequential micro and ultrafiltration of thin stillage

Amit Arora [a], Bruce S. Dien [b], Ronald L. Belyea [c], Vijay Singh [a], M.E. Tumbleson [a], Kent D. Rausch [a,*]

[a] Agricultural and Biological Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA
[b] National Center for Agricultural Utilization Research, Agricultural Research Service, USDA, 1815 North University Street, Peoria, IL 61604, USA
[c] Animal Sciences, University of Missouri, Columbia, MO 65211, USA

ARTICLE INFO

Article history:
Received 24 July 2009
Received in revised form 23 December 2009
Accepted 31 December 2009
Available online 6 February 2010

Keywords:
Corn
Ethanol
Thin stillage
Membrane filtration

ABSTRACT

The effectiveness of microfiltration (MF) and ultrafiltration (UF) for nutrient recovery from a thin stillage stream was determined. When a stainless steel MF membrane (0.1 μm pore size) was used, the content of solids increased from 7.0% to 22.8% with a mean permeate flux rate of 45 L/m^2/h (LMH), fat increased and ash content decreased. UF experiments were conducted in batch mode under constant temperature and flow rate conditions. Permeate flux profiles were evaluated for regenerated cellulose membranes (YM1, YM10 and YM100) with molecular weight cut offs of 1, 10 and 100 kDa. UF increased total solids, protein and fat and decreased ash in retentate stream. When permeate streams from MF were subjected to UF, retentate total solids concentrations similar to those of commercial syrup (23–28.8%) were obtained. YM100 had the highest percent permeate flux decline (70% of initial flux) followed by YM10 and YM1 membranes. Sequential filtration improved permeate flux rates of the YM100 membrane (32.6–73.4 LMH) but the percent decline was also highest in a sequential MF + YM100 system. Protein recovery was the highest in YM1 retentate. Removal of solids, protein and fat from thin stillage may generate a permeate stream that may improve water removal efficiency and increase water recycling.

1. Introduction

In the corn dry grind ethanol industry, thin stillage is recycled as backset which is added in the slurry tank. The rest of the thin stillage is sent to evaporators for concentration which involves significant energy inputs and results in evaporator fouling problems. To decrease the load upon evaporators and reduce demand for fresh water, recycle of water in the thin stillage stream should be increased. However, there are concerns with recycling thin stillage above a certain percentage, generally ⩾30–50% depending on plant operating conditions. As percent recycle is increased, the concentrations of various compounds, especially lactic and acetic acids, inhibit yeast growth and reduce ethanol yields (Chin and Ingledew, 1993; Ingledew, 2003).

Ultrafiltration (UF) is an efficient process for selective removal of compounds by convective solvent flow through a membrane. Membrane filtration involves no evaporation of water; hence energy consumption is lower than with thermal methods. Application of UF membranes to process thin stillage obtained from conventional and an enzymatic corn dry grind (E-Mill) processes have been described (Arora et al., 2009, 2010). Total solids recovered through batch UF membrane separation were similar to solids levels obtained from commercial evaporators. The membrane fouling issue has also been addressed during thin stillage concentration and found to be primarily reversible using filtration parameters and cleaning methods (Arora et al., 2009). In small scale experiments, the flux rates of thin stillage through MF and UF membranes was high enough to appear economically feasible (Arora et al., 2009). However, application of microfiltration (MF) and sequential MF + UF processes have not been used to filter and recover nutrients from commercial thin stillage; distribution of yeast inhibitors in membrane retentate and permeate streams have not been investigated. Water recycling rates could be increased if MF and UF methods were effective in removing compounds that inhibit yeast growth and metabolism. We evaluated nutrient recovery using MF and UF membranes and evaluated permeate streams on the basis of organic compound removal. Specific objectives were to: (1) compare filtration characteristics of thin stillage for MF, UF and sequential MF + UF processes, (2) evaluate solids recovery and nutrient compositions of permeate and retentate streams and (3) evaluate the permeate streams for potential water recycling based on organic acid contents.

* Corresponding author. Address: Agricultural and Biological Engineering, University of Illinois at Urbana-Champaign, 1304 West Pennsylvania Avenue, Urbana, IL 61801, USA. Tel.: +1 217 265 0697; fax: +1 217 244 0323.
E-mail address: krausch@illinois.edu (K.D. Rausch).

0960-8524/$ - see front matter © 2010 Elsevier Ltd. All rights reserved.
doi:10.1016/j.biortech.2009.12.127

2. Methods

2.1. Experimental material

Thin stillage was collected from a commercial dry grind ethanol facility and stored at 4 °C. To characterize thin stillage and retentate streams, one 500 mL sample was analyzed for total solids (TS) using a two stage oven method (Approved Method 44-15A, AACC International, 2000). Composition of thin stillage and retentate streams obtained from MF and UF processes were analyzed for protein (total nitrogen × 6.25), fat and ash content using standard methods (AOAC, 2003) at the University of Missouri, Columbia. Compositional analyses were performed in duplicate.

2.2. Microfiltration equipment

Microfiltration (MF) was carried out using a bench top membrane unit. A tubular stainless steel module with six tubes having 0.61 m length, nominal diameter of 0.64 cm, 0.1 μm pore size, 0.31 cm wall thickness and 0.07 m^2 membrane area (Scepter model 2.5-250A-2P6, Graver Technologies, Glasgow, DE) was used in a crossflow filtration arrangement. The unit was equipped with a batch tank of 15 L capacity, a heat exchanger and positive displacement pump (model M-03, Hydra-Cell, Minneapolis, MN). The unit was operated in total recycle mode (permeate returned to tank) or batch concentration mode (permeate collected separately and retentate recycled) with 4.75 m/s crossflow velocity. The permeate that passed through the membrane was termed MFP and the material that was retained and returned to tank was termed retentate. Permeate was collected in a graduated cylinder during batch concentration. Thin stillage (15 L) was used for each batch experiment; permeate flux rates measurements were taken during concentration and presented as LMH (L/m^2/h) until batch material was exhausted. Five replicates were performed for concentration profiles. Permeate flux rate was determined manually, with graduated cylinder and stopwatch. The selected operating conditions were 690 ± 13 kPa transmembrane pressures (TMP) with a crossflow velocity and fluid temperature of 4.75 m/s and 75 ± 2 °C, respectively. Thin stillage temperatures in an ethanol plant are 60–75 °C. Therefore, the operating temperature chosen for the MF filtration study was 75 ± 2 °C. Average permeate flux rate values were calculated using Eq. (1) as described in the section describing membrane performance.

2.3. Stirred cell ultrafiltration unit

A stirred ultrafiltration cell (400 mL Amicon, model 8400, Millipore Corporation, Bedford, MA) was used for concentrating thin stillage at room temperature. An argon gas cylinder was used to apply pressure to the stirred cell. A magnetic stir bar was used to simulate crossflow filtration. Two regenerated cellulose membranes, YM10 and YM100 (Millipore Corporation, Bedford, MA) with pore sizes of 10 and 100 kDa, respectively, and effective membrane area of 41.8 cm^2 were used. Five replicates of thin stillage (300 mL) were used in filtration.

2.4. Membrane selection

Since thin stillage contains a range of molecular weight compounds such as amino acids, peptides, proteins, fat and minerals (Jones and Ingledew, 1994; Kim et al., 2008; Arora et al., 2009), it is important to choose a membrane or membranes that recover solids and produce cleaner permeate stream with higher flux rates. Therefore, four membranes with different pore sizes were chosen to evaluate permeate flux rates and solids recovery. In phase I filtration experiments (Fig. 1), thin stillage batches were filtered through stainless steel MF (0.1 μm pore size) and regenerated cellulosic UF membranes YM1, YM10 and YM100 with 1, 10 and 100 kDa molecular weight cutoff (MWCO), respectively. Permeate flux rates were measured for all membranes. Thin stillage, retentates and permeates were analyzed for compositions. Five replications were used for each treatment.

In phase II, permeates obtained from MF runs were filtered further using YM100, YM10 and YM1 membranes (Fig. 2) and analyzed for lactic and acetic acid concentrations. UF experiments were conducted at 25 °C (room temperature) and used pressures recommended by the manufacturer for each membrane (380, 207 and 70 kPa for YM1, YM10 and YM100 membranes, respectively). Both MF (15 L batch) and UF (350 mL/batch for phase I, 300 mL/batch for phase II) experiments were operated in batch concentration mode and experiments were continued until the material was exhausted. Permeate flux rates were measured using a graduated cylinder during MF experiments. During UF, permeate flux rates were determined gravimetrically by measuring the cumulative weight permeated, collected from the bottom of the cell as a function of time using an electronic balance. In sequential filtration, MF permeate was further filtered using a UF membrane. Permeate flux rates for UF and MF + UF membranes were calculated from start of the UF experiments.

2.5. Measurement of membrane separation performance

The average permeate flux rate (J_{av}) was calculated by

$$J_{av} = \frac{V}{At} \tag{1}$$

where J_{av} was the average flux rate (LMH), V was the total volume (L) of permeate, A was the effective area of the membrane, and t (h) was the permeate collection time.

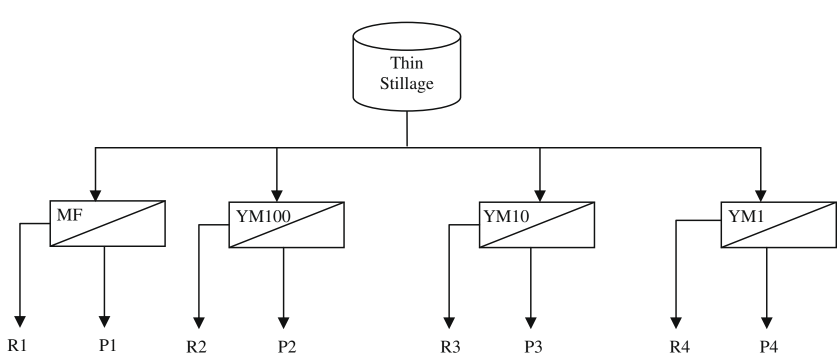

Fig. 1. Thin stillage filtration through various membranes (phase I).