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Removal of lactate from acid whey using nanofiltration

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ABSTRACT

The utilisation of acid whey by the dairy industry is limited by the high concentrations of lactate in the solution. In the present study, three commercially available nanofiltration membranes: HL, XN45 and DK were used in to evaluate the separation of lactose and lactate as a function of pH. Raw acid whey was used after microfiltration, while two laboratory prepared solutions comprising lactic acid and lactose; and sodium lactate and lactose were also tested for comparison. The rejection of lactic acid was found to increase with the degree of acid dissociation. About 50% of the lactate could be removed at pH ~3 due to the lower degree of dissociation. For all pH values tested, over 90% of the lactose was retained. The molecular weight cutoff and permeability of the membranes had little influence on the separation performance.

1. Introduction

The production of fermented dairy products involves the generation of waste streams termed "whey". Whey is the serum phase of milk and contains about 20% of the protein and most of the lactose from the original input milk (Blaschek et al., 2007; Fox et al., 2015b). It can be further classified based on processing conditions into sweet whey and acid whey. Sweet whey is a by-product of rennet cheese manufacture and is commonly spray dried to form high value dairy powders for use in infant nutrition and body building applications. Conversely, acid whey comes from the production of acid coagulated dairy products including fresh and cream cheeses and strained (Greek style) yoghurts (de Wit, 2001; Prazeres et al., 2012). A shift in market trends towards Greek style yoghurts has resulted in the production of very large quantities of this acid whey (Prazeres et al., 2012). Despite the profits generated from yoghurt production, the industry has been struggling to find solutions for such increasing volumes. In general, the disposal of waste whey is complicated due to its high biological oxygen demand that leads to the need for costly treatment facilities. Some small scale facilities use acid whey in manure as an organic fertiliser, as a supplement of cattle feed, or for conversion into biogas (Prazeres et al., 2012). However, these are all low value applications.

The presence of high concentrations of lactate, either as lactic acid or dissociated into lactate ions, reduces the potential for the use of acid whey in spray dried powders. Specifically, the presence of high concentrations of lactate has been shown to cause increased powder stickiness, which in turn causes operational problems in the dryer (Modler and Emmons, 1978; Prazeres et al., 2012; Shrestha et al., 2006). It has been shown that the temperature at which sticking occurs is directly related to the mass ratio of lactate to lactose (Knipschildt and Andersen, 1994); if there is more lactate present this occurs at a lower temperature. Therefore, a successful reduction in this ratio would allow acid whey to be converted to higher value powders using the conventional spray drying units that are widely available in the dairy industry.

Nanofiltration (NF) membrane technology is already extensively used in the dairy industry to demineralise and concentrate sweet whey streams prior to spray drying (Kentish and Rice, 2015; Okawa et al., 2015; Rice et al., 2009). NF has also been considered at laboratory scale for demineralisation of acid whey (Kelly and Kelly, 1995; Roman et al., 2010, 2009; Vasiljevic and Jelen, 2000). NF is also used for the recovery of lactic acid from fermentation broths where the lactate concentration is 10–20 times that in acid whey (Bouchoux et al., 2006; Li, 2006; Sikder et al., 2012; Wang et al., 2013).

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However, to the best of our knowledge, this approach has not yet been used to remove the lactate content from acid whey. Nanofiltration is an ideal candidate for this operation as separation is achieved without the use of additional chemicals; and as the whey solution would be simultaneously demineralised and concentrated. NF membranes reject molecules based on size exclusion or charge repulsion (Kentish and Rice, 2015). Under uncharged conditions, lactose (LT, 342.3 Da) can be retained preferentially over lactic acid (LA, 90.1 Da) due to its greater molecular weight. However, while lactose molecules are uncharged, lactic acid is a weak acid that can dissociate into its conjugate base (a lactate ion, $CH_3CH(OH)COO^-$) and protons. The dissociation of lactic acid is given by the Henderson–Hasselbalch equation:

$$pH = pK_a + \log\frac{[CH_3CHOHCOO^-]}{[CH_3CHOHCOH]} \quad (1)$$

The dissociation constant (pK_a) of lactic acid at 25 °C is reported to be 3.86 (Budavari, 1989). The association constant at a different temperature can be estimated by the following correlation (Perrin, 1964):

$$-d(pK_a)/dT = (pK_a - 0.9)/T \pm 0.004 \quad (2)$$

where T is the temperature of the system in Kelvin.

Ideally, a NF membrane possessing a molecular weight cut-off (MWCO) between 90 and 340 and a positive surface charge could be used to retain lactose while removing lactate ions from acid whey (Bellona et al., 2004; Teixeira et al., 2005). However, almost all commercially available NF membranes are negatively charged at neutral conditions. This means that separation in the present case is likely to rely upon the differences in molecular size, with the larger lactose retained while lactate species are transmitted. However, for this to be effective, it would be necessary to ensure that the lactic acid is substantially undissociated, as the negatively charged lactate ions would not readily pass through the membrane.

Table 1
General composition of acid whey reported in the literature (Benitez and Ortero, 2012; Bylund, 1995; Durham and Hourigan, 2009; Nguyen et al., 2003; Peri and Dunkley, 1971; Rodriguez et al., 2012; Saffari and Langrish, 2014; Spreer, 1998; Waldron, 2007) and the acid whey (raw and microfiltered) used in the current study.

Properties	Literature	Present work	
		Raw	After MF
pH	4.0–4.6	4.3–4.6	
Total solids (%)	5–6.4	6.1	4.5–5.5
Fat (%)	0.003–0.38	–	0.02
Lactose (%)	3.9–4.9	3.5–4.5	3.7–4.4
Lactic acid (%)	<0.8	0.63	0.55–0.58
Ash (%)	0.5–0.7	0.76	0.61–0.64

Therefore, it was the purpose of this work to evaluate the potential for nanofiltration to separate undissociated lactose from charged lactate anions within an acid whey system, to increase the potential for spray drying of the product. Three commercial NF membranes were selected for this purpose, based on their differences in MWCO, isoelectric point and water permeability; so that the effect of these parameters on lactate/lactose separation could be evaluated. Experiments were conducted using both real acid whey and solutions prepared in the laboratory, across a range of pH values and temperature, which in turn varies the membrane permeability, the strength of the membrane surface charge and the degree of lactate dissociation. The separation performance of the different membranes was assessed and related to the underlying membrane characteristics and the operational conditions.

2. Materials & methods

2.1. Materials

Raw acid whey samples were obtained from a dairy processing company in Victoria, Australia. The two acid whey batches were microfiltered (MF) commercially in a pilot plant at Dairy Innovation Australia Ltd (Werribee, Victoria, Australia) using 1.4 μm Isoflex ceramic filters (Tami industries, Z.A. Les Laurons, NYONS CEDEX, France) with a transmembrane pressure of 1 bar. The approximate composition of acid whey was found to be consistent with that reported in the literature (Rodriguez et al., 2012; Waldron, 2007) (Table 1).

Artificial solutions were prepared in the laboratory by mixing lactic acid (85%, Ajax Finechem) or sodium lactate (70%, Ajax Finechem) with lactose (≥99.8%, Ajax Finechem) to mimic their concentrations in acid whey. The pH of the lactic acid-lactose (LA + LT) and the sodium lactate-lactose (Na-LA + LT) solutions were ~2.6 and ~7, respectively. The pH of these laboratory solutions was adjusted using an appropriate amount of 10 M NaOH or 2 M HCl. pH adjustment was only performed in one direction, in order to prevent unnecessarily high increases in ionic strength due to addition of both NaOH and HCl.

2.2. Nanofiltration

A cross flow GE Osmonics membrane filtration module was used for the NF experiments. The module has a maximum operating pressure of 69 bar and an effective membrane area of 0.014 m^2. Three commercially available flat sheet NF membranes with different MWCO, divalent rejection, isoelectric point and water permeability were supplied from Sterlitech (Kent, WA) (Table 2). Experiments were conducted with a feed flow rate of 2.4 L/min (cross flow velocity = 0.5 m/s) and a transmembrane pressure of

Table 2
Characteristics of NF membranes used in this work.

Membrane	Polymer[a]	Minimum salt rejection[a,b]	MWCO[a]	Water permeability[c]	Isoelectric point[c]	References
	–	%	Da	L/hm^{2}bar	–	–
Trisep XN45	Thin film composite - Polypiperazineamide	92% $MgSO_4$	500	4.9	~4.5	(Kappel et al., 2014; Mandale and Jones, 2010)
GE HL	Thin film composite - Polypiperazinamide	95% $MgSO_4$	150–300	3.2	~3.8	(Al-Amoudi et al., 2007; Ecker et al., 2012)
GE DK	Thin film composite - Polypiperazinamide	98% $MgSO_4$	150–300	2.8	~4.0	(Al-Amoudi et al., 2007; Ecker et al., 2012)

[a] Manufacturer supplied information.
[b] Testing conditions: 2,000 ppm MgSO4 solution at 760 kPa operating pressure, 25 °C, 15% recovery.
[c] Cited from the references shown.