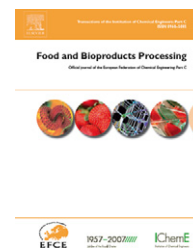


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Lactic acid enrichment with inorganic nanofiltration and molecular sieving membranes by pervaporation

Mikel. C. Duke^{a,*}, Agnes Lim^b, Sheila Castro da Luz^c, Lars Nielsen^d

^a Institute for Sustainability and Innovation, Victoria University, Werribee Campus, PO Box 14428, Melbourne, Vic. 8001, Australia

^b School of Life Sciences and Chemical Technology, Ngee Ann Polytechnic, Singapore

^c Faculdade Cenecista, Facensa University, Gravataí, Brazil

^d Australian Institute of Bioengineering and Nanotechnology, The University of Queensland, St Lucia, Qld 4072, Australia

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ABSTRACT

Lactic acid is a valuable product in the food industry, but requires expensive complex systems to purify. Porous inorganic membranes have high fluxes and water separation potential and are driven only by pressure difference without the need for added chemicals. Here we show the application of readily available γ -alumina (nanofiltration), and the more advanced molecular sieve silica membranes, to enrich lactic acid for product use by selectively depleting water through the membrane. The alumina membranes showed flux starting at $6 \text{ kg m}^{-2} \text{ h}^{-1}$, reducing to $1 \text{ kg m}^{-2} \text{ h}^{-1}$ after 250 min due to pore blocking of lactic acid. The membrane acted to remove water from the 15 wt% feed, with permeate lactic acid concentration at 2 wt% corresponding to a water selectivity factor of 9. Silica membranes on the other hand exhibited a water selectivity factor up to 220 (a rejection coefficient of 0.995) with lactic acid in the permeate as low as 0.08 wt% after regeneration with an overall stable flux of $0.2 \text{ kg m}^{-2} \text{ h}^{-1}$. The strong surface charge and wider pore size of the alumina membrane enabled a slow pore blocking mechanism, with flux dropping towards that of the silica membrane. The silica membrane was therefore the choice technology as the tight pore spaces inhibited lactic acid from entering and the charge-neutral surface leading to a more stable separation not subject to pore blocking. Performance results allowed calculation of membrane area for industrial separation. Flux improvements and longer term studies are needed to improve silica membrane commercial attraction.

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1. Introduction

The use of lactic acid (LA) in industries including foods is growing rapidly. In a review by Lee (1997), the importance of LA to flavour, food quality and as a preservative in the preparation of perishable foods was presented. Traditionally, LA is fermented within the food, but can also be produced at large scales for improving product versatility. The majority of LA plants use fermentation (Kašćák et al., 1996), but have high capital costs due mostly to the removal and purification stages (González et al., 2007). Solvent extraction removes LA

from fermentation and is a popular choice for industry today (Marinova et al., 2004; Urbas, 1984; Yankov et al., 2004), but relies on the use of added chemicals which introduce further complexities so alternative processes are under investigation. González et al. (2007) proposed a purification process involving ultrafiltration, ion exchange, reverse osmosis and vacuum evaporation. Their costing revealed the final vacuum evaporation stage took 47% of the separation capital costs and 18% of the separation operating costs. Electrodialysis is another option for LA purification but is still undergoing research to reduce fouling (Habova et al., 2004; Thang et al.,

* Corresponding author.

E-mail address: mikel.duke@vu.edu.au (Mikel.C. Duke).

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Nomenclature

A	membrane active area (m ²)
C _P	lactic acid concentration in the permeate
IEP	Iso-electric point
J	total membrane flux (kg m ⁻² h ⁻¹)
LA	lactic acid
m _P	mass permeate vapour caught in trap (kg)
t	time of which trap is collecting vapour (h)
x _{F,w}	feed weight fraction water (kg kg ⁻¹)
y _{P,w}	permeate weight fraction water (kg kg ⁻¹)
Greek letter	
α _{Proc}	water selectivity factor for LA pervaporation

2005). Inorganic membrane pervaporation has the potential to reduce energy requirements of the final purification stage and may be less sensitive to the fouling impurities.

Inorganic membranes are a class of membranes, differentiated from their organic counterparts, which are used commercially in industries such as water purification and treatment. Inorganic γ -alumina membranes are widely available, are a mature product, and have pores \sim 4 nm in diameter (Leenaars et al., 1984). Recently, Polom and Szaniawska (2006) explored optimal conditions for ultrafiltration of LA using inorganic membranes but LA-surface interactions were not considered.

Silica has more recently been used as a membrane material, with researchers still uncovering gas (de Vos and Verweij, 1998; Duke et al., 2004; Nair et al., 2004) and liquid (Peters et al., 2005; Sekulic et al., 2005; Sommer and Melin, 2005) separation properties. The mechanism of separation, like alumina, can be governed by surface interactions with permeating species, but also have an extra molecule sieving ability due to the molecular dimensioned (\sim 0.3 nm) amorphous silica structure (de Vos and Verweij, 1998; Tsai et al., 2000; Uhlhorn et al., 1992). This means He, H₂ and H₂O are among the only molecules able to pass through the material when made into a membrane film. Recent progress in silica membrane hydrostability by carbonised template molecular sieve silica (CTMSS) (Duke et al., 2006) allow application in aqueous LA separation. Both alumina and silica materials could therefore separate LA from water.

For volatile organic solutions, pervaporation is a convenient and widely utilised process to perform separation with

inorganic membranes (Campaniello et al., 2004; Peters et al., 2005; Sekulic et al., 2005; Sommer et al., 2002; Yang et al., 2006). The key benefit of pervaporation lies in its ability to separate mixtures before distillation, overcoming thermodynamic limitations and potentially offering energy reductions. In this work, we developed alumina and silica membranes and apply them to remove water from (or concentrate) LA solutions containing glucose in a pervaporation setup. The startup performance will be observed to establish diffusion mechanisms. Testing will also allow for developing understanding into the unique surface interactions of permeating species. Results are then used to develop a unit operation model to compare technologies and give insight into industrially orientated development of the inorganic membrane technology. The membrane's ability to be regenerated will also be shown.

2. Methods and materials

Inorganic membranes in this work were made by our standard sol-gel process described elsewhere (Duke et al., 2004). Membranes were prepared on α -alumina supports (Rojan Ceramics, Australia) with pores with a diameter of 0.5–1 μ m and porosity of 30%. The support surface was prepared by sanding to 2000 grade sandpaper. After sanding, substrates were immersed in water and treated in an ultrasonic bath to remove stray particles. The γ -alumina membrane was then prepared by dip coating substrates into a Locron (Clariant, GmbH) solution followed by calcination to 600 °C. The process was repeated to repair defects, leaving an intact film with a pore size of \sim 4 nm (Leenaars et al., 1984). Some γ -alumina membranes were taken out at this stage for testing. The silica membrane was prepared on top of the remaining γ -alumina membranes by dip coating with silica sols made by the acid catalysed sol-gel process. Hexyl triethyl ammonium bromide (Aldrich) was added to the silica sols before dip coating and membranes were calcined in vacuum at 500 °C, preventing carbon oxidation thus preserving surfactant templates, forming the hydrostable (Duke et al., 2006) carbonised template molecular sieve silica membranes.

The characteristics of the membrane were determined by gas permeation of He and CO to ensure intact films. Permeation was conducted in a dead-end setup at 200 °C and with 2 bar pressure drop across the membrane. The setup of the process is described elsewhere (Duke et al., 2004).

LA separation was performed at room temperature (25 °C) using pervaporation, a process with a liquid feed and vapour

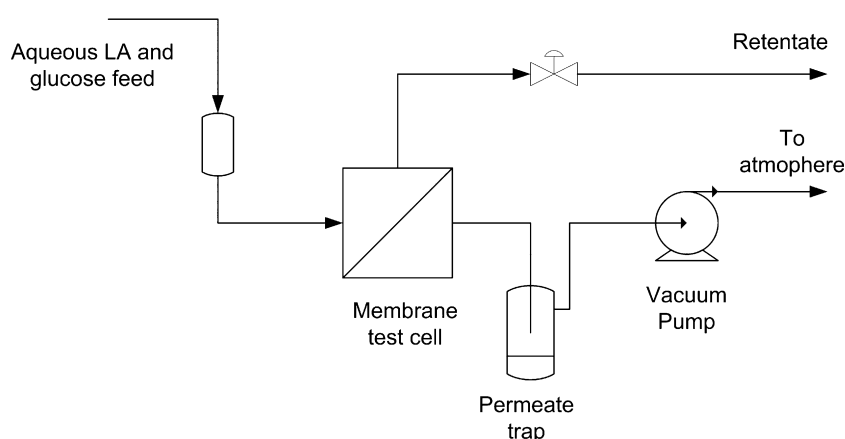


Fig. 1 – Experimental setup of membrane pervaporation.

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