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Separation of D, L-Lactic Acid by Filtration Process

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Abstract

The purpose of this research was to study the optimum conditions for separation of D,L-lactic acid isomer by filtration process using polytetrafluoroethene (PTFE) membrane filter. The study on suitable chiral selector by adding chiral selector into D,L-lactic acid and separate through membrane filter showed that the separation time of 30 min by using 2.67 g/l (v/v) \( \beta \)-cyclodextrin as chiral selector was the most suitable for separation of D,L-lactic acid isomer. Efficiency of carrier on separation of D,L-lactic acid isomer was studied. D,L-lactic acid was separated through the carrier impregnated membrane filter, the results showed that modified Aliquat\textsuperscript{TM}336 in carbonate form was the most efficient for separation of D,L-lactic acid isomer. In addition, efficiency improvement of D,L-lactic acid isomer separation was carried out by using \( \beta \)-cyclodextrin as chiral selector, the impregnated membrane filter with modified Aliquat\textsuperscript{TM}336 and 2 stages of separation by filtration process that separation time of 30 min each step. It was found that the first separation stage could be separate D-lactic acid from L-lactic acid in transmembrane solution, the D,L-lactic acid separation factor achieved was 2.93. High efficiency of D,L-lactic acid isomer separation was obtained from the second stage and separation factor was as high as 4.43 in this stage.

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Keywords: D,L-Lactic acid; Polytetrafluoroethene membrane; Filtration process; Chiral selector

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1. Introduction

Lactic acid is an organic acid produced either by chemical synthesis or by carbohydrate fermentation. It is used in a wide range of food, cosmetic, pharmaceutical and other industrial applications. Lactic acid can also be used as a feedstock monomer for the production of biodegradable polyactic acid (PLA), which is well-known as a sustainable bioplastic material [13], [16], [18]. There are three types of lactic acid: L-lactic acid, D-lactic acid and D,L-lactic acid that is a mixture of D-and L-isomer (racemic mixture) [9]. Racemic mixture form is always produced by chemical synthesis, whereas an optically pure L- or D-lactic acid can be obtained by microbial fermentation from renewable resources depending on the strain chosen [16], [17]. Nowadays, lactic acid is considered the most potential feedstock monomer for chemical conversions into potentially useful chemicals including PLA, which serves as biodegradable commodity plastic and biocompatible polymer [16]. The optical purity of lactic acid is an important properties of PLA, an optically pure L or D-lactic acid will be give better properties of PLA than racemic DL-lactic acid. PLA obtained from optical pure lactic acid can be polymerized to a high crystalline PLA that is suitable for commercial uses [10].

The increasing need for enantioseparation of racemic mixture is significantly important for many industries. Many studies concerning lactic acid separation methods have been conducted using different separation techniques such as ion exchange [1], [15], esterification and hydrolysis [13], supported liquid membrane system [3], [4], [17], and bipolar electrodialysis [14]. Enantioseparation methods using chiral selectors and filtration processes are very promising new processes for separation of lactic acid enantiomers. The aim of this work is to investigate the separation of D,L-lactic acid racemic mixture by filtration process in a solution system using β-cyclodextrin (β-CD) [6], [17] or bovine serum albumin (BSA) [2] as combining chiral selector.

2. Experimental Procedure

2.1. Materials

The D-lactic acid and L-lactic acid (Fluka, Switzerland) were used as feed solution. β-cyclodextrin (β-CD) (Cyclolab, Hungary) or bovine serum albumin (BSA) (Sigma, USA) were used as chiral selector in the feed phase to selectively bind and retain one of the enantiomer. Aliquat™ 336 (triocylmethylammonium chloride) (Aldrich, Singapore) and its modified form with carbonate were used as carriers in liquid membrane phases. Polytetrafluoroethylene (PTFE) membrane (Satorius, Germany) with diameter of 47 mm and average pore size of 0.2 μm was used as membrane filter.

2.2. Filtration device

Filtration experiments were carried out using a dead-end stirred filtration device holding a flat sheet polytetrafluoroethylene (PTFE) membrane filter [Fig. 1]. The system was performed with a transmembrane pressure of 3.5 bar and a rotation speed of 200 rpm.
2.3. Comparison of D, L-lactic acid enantioseparation using β-CD or BSA as the chiral selector

D-lactic acid and L-lactic acid were mixed at a ratio of 1:1 and used as feed solution. β-CD or BSA was added to the feed solution to give a final chiral selector concentration of 2.67 g/l. The resulting solution was gently stirred overnight and then filtered through PTFE membrane in filtration device. The D- and L- lactic acid concentrations in the collected filtrate were determined by high performance liquid chromatography (HPLC). The lactic acid flux (mol/cm²/min) was calculated based on lactic acid concentration as a function of time using in the feed solution and membrane area [3],[4]

\[ J = \frac{C}{A \times T} \]  \hspace{1cm} (1)

where \( C \) is mole of lactic acid, \( A \) is the area of membrane (cm²) and \( T \) is operation time (min). The characterization of the enantioseparation process can be described by the separation factor (\( \alpha \)) [4],[11]

\[ \alpha = \frac{J_{D-L}}{J_{L-L}} \]  \hspace{1cm} (2)

where \( J_{L-L} \) and \( J_{D-L} \) denote the flux of L- and D-lactic acid, respectively.

2.4. Comparison of D, L-lactic acid enantioseparation using the Aliquat™ 336 and its modified form as the carrier

In this study, carrier modification was carried out by vigorously mixing 200 ml Aliquat™ 336 with excess amount of 1 M Na₂CO₃ at 600 ml in a shaker over 3 days at ambient temperature. The extended period of mixing was to ensure it was sufficient to obtain equilibrium of exchanging the chloride anion of Aliquat™ 336 with a carbonate anion [17]. Aliquat™ 336 and its modified form were used to impregnate PTFE membrane filters before filtration experiments. In this part of work, the feed solution was D-lactic acid and L-lactic acid mixture at a ratio of 1:1 with 2.67 g/l β-CD. The feed solution with β-CD addition was gently stirred overnight and then filtered through impregnated PTFE membrane in filtration device. The D- and L- lactic acid concentrations in the collected filtrate were determined by HPLC. The lactic acid flux (mol/cm²/min) and the separation factor (\( \alpha \)) were calculated.
2.5. Enantioseparation of D, L-lactic acid by two stages filtration process

Mixture of D-lactic acid and L-lactic acid at a ratio of 1:1 was used as feed solution. Suitable chiral selector (β-CD) was added to the feed solution to give a final concentration of 2.67 g/l. The feed solution was gently stirred overnight and then filtered through impregnated PTFE membrane with modified Aliquat™ 336 in filtration device. The filtrates from the first filtration stage were collected and added with β-CD to give a final concentration of 1.33 g/l. The resulting solution was gently stirred overnight and then filtered through impregnated PTFE membrane again. The D- and L- lactic acid concentrations in the collected filtrate from the second filtration stage were determined by HPLC.

2.6. Lactic acid analysis

The D-lactic acid and L-lactic acid concentrations were determined by HPLC equipped with Ultra Aqueous C18 column (RESTEX, USA) and UV-Vis detector operating at 210 nm. The column was eluted with 5 mM H₂SO₄ at 0.5 ml/min.

3. Results and Discussion

In comparison of using β-CD and BSA as the chiral selector for D, L-lactic acid enantioseparation by adding 2.67 g/l chiral selector into the feed solution and filtered through PTFE membrane in filtration device, the results show that D, L-lactic acid separation factors (α) are always higher using β-CD than BSA (Table 1). D, L-lactic acid with β-CD and filtration time of 30 min, separation factor achieved is 2.67. Furthermore, the addition of β-CD into the feed solution, the D-lactic acid transmembrane fluxes are lower than the L-lactic acid transmembrane fluxes. This is due to the fact that the favorable formation of a stronger diasteromer complex between D-lactic acid and β-CD [8], [17]. It is also reported that chiral resolution of the aliphatic α-hydroxy acids such as lactic acid can be accomplished using β-CD [7]. From this experiment, β-CD was selected to be chiral selector for enantioseparation of D, L-lactic acid.

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>J_{L,-A} (mol/cm² min)</th>
<th>J_{D,-A} (mol/cm² min)</th>
<th>α (J_{L,-A}/J_{D,-A})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Racemic mixture of D- and L-lactic acid with β-CD</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>0.0153</td>
<td>0.0074</td>
<td>2.05</td>
</tr>
<tr>
<td>30</td>
<td>0.0067</td>
<td>0.0025</td>
<td>2.67</td>
</tr>
<tr>
<td>60</td>
<td>0.0036</td>
<td>0.0021</td>
<td>1.68</td>
</tr>
<tr>
<td>100</td>
<td>0.0022</td>
<td>0.0016</td>
<td>1.37</td>
</tr>
<tr>
<td>Racemic mixture of D- and L-lactic acid with BSA</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>0.0078</td>
<td>0.0097</td>
<td>0.80</td>
</tr>
<tr>
<td>30</td>
<td>0.0071</td>
<td>0.0066</td>
<td>1.08</td>
</tr>
<tr>
<td>60</td>
<td>0.0036</td>
<td>0.0035</td>
<td>1.03</td>
</tr>
<tr>
<td>100</td>
<td>0.0022</td>
<td>0.0021</td>
<td>1.02</td>
</tr>
</tbody>
</table>

Comparison of D,L-lactic acid enantioseparation using the Aliquat™ 336 and its modified form as the carrier, Aliquat™ 336 and its modified form were used to impregnate PTFE membrane filters before filtration experiments. Modified Aliquat™ 336 is the conversion of chloride form of Aliquat™ 336 into a carbonate form according to reaction:
2 NR₄⁺Cl⁺ + CO₃²⁻ \rightleftharpoons (NR₄)₂CO₃ + 2Cl⁻

Where NR₄Cl and (NR₄)₂CO₃ represents Aliquat™ 336 in chloride and carbonate form, respectively. Aliquat™ 336 and its modified form could be combined with lactic acid and form lactate-carrier complex that diffuse easily through membrane filters [17]. This study found that D, L-lactic acid separation factors are higher using impregnated PTFE membrane with modified Aliquat™ 336 than Aliquat™ (Table 2). In addition, using impregnated PTFE membrane with modified Aliquat™ 336 or Aliquat™ give D, L-lactic acid separation factor higher than non-impregnated PTFE membrane. At filtration time of 30 min, separation factor achieved from using impregnated PTFE membrane with modified Aliquat™ 336 is 3.04.

<table>
<thead>
<tr>
<th>Chiral selector and carrier</th>
<th>Jᵢ-L,L₀ (mol/(cm² min))</th>
<th>Jᵢ-D,L₀ (mol/(cm² min))</th>
<th>α (Jᵢ-L,L₀/Jᵢ-D,L₀)</th>
</tr>
</thead>
<tbody>
<tr>
<td>β-CD</td>
<td>0.0067</td>
<td>0.0025</td>
<td>2.67</td>
</tr>
<tr>
<td>β-CD and Aliquat™ 336</td>
<td>0.0069</td>
<td>0.0024</td>
<td>2.83</td>
</tr>
<tr>
<td>Aliquat™ 336</td>
<td>0.0071</td>
<td>0.0024</td>
<td>3.04</td>
</tr>
</tbody>
</table>

Improvement of D,L-lactic acid enantioseparation was carried out by using 2.67 g/l β-CD as chiral selector, the impregnated membrane filter with modified Aliquat™ 336 and 2 stages of separation by filtration process that separation time of 30 min each stage. Experimental results for enantioseparation of D, L-lactic acid were shown in Table 3. It was found that the first stage of filtration could be separate D-lactic acid in transmembrane solution, the separation factor achieved was 2.93. In the second stage of filtration, the filtrates from the first filtration stage were collected and added with β-CD to give a final concentration of 1.33 g/l, high efficiency of D, L-lactic acid separation was obtained and separation factor achieved was 4.43.

<table>
<thead>
<tr>
<th>Stage*</th>
<th>Jᵢ-L,L₀ (mol/(cm² min))</th>
<th>Jᵢ-D,L₀ (mol/(cm² min))</th>
<th>α (Jᵢ-L,L₀/Jᵢ-D,L₀)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>0.0070</td>
<td>0.0024</td>
<td>2.93</td>
</tr>
<tr>
<td>2</td>
<td>0.0066</td>
<td>0.0015</td>
<td>4.43</td>
</tr>
</tbody>
</table>

* stage 1: concentration of β-cyclodextrin = 2.67 g/l
* stage 2: concentration of β-cyclodextrin = 1.33 g/l
4. Conclusion

In this study, enantioseparation of D, L-lactic acid racemic mixture by filtration process has been performed. β-CD was found suitable to be chiral selector for enantioseparation D, L-lactic acid racemic mixture. Modified AliquatTM 336 in carbonate form was found to be effective to enhance transmembrane flux through PTFE membrane. In addition, the two-stages filtration process could be applied to practical D, L-lactic acid enantioseparation with separation factor of 4.43.

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References