10th Eco-Energy and Materials Science and Engineering Symposium


On December 5-8, 2012
Sunee grand hotel,
Ubon-ratchathani

Organized by

Co-organized by
PREFACE:
Message from the President of
Rajamangala University of Technology Thanyaburi

Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is please to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This international conference is not only giving an opportunity for Thai and foreign researchers to present and discuss their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Our program consists of six research tasks: (1) Energy Technology, (2) Environmental and Social Impact, (3) Nanotechnology and Materials Science, (4) Energy Economics and Management, (5) New Energy Technology and (6) Nuclear Technology.
I would like to take this opportunity to express our sincere gratitude to our two distinguished Plenary Speakers for kindly accepting our invitation. I deeply appreciate of the very strong support given by Kyoto University. Thanks to the tireless works of the Organizing Committee, the Technical Program Committee, the invited speakers and paper and poster contributors, and excellent program been assembled to cover a broad spectrum of interesting topic.
We warmly welcome you to the 10th EMSES on December 5-8, 2012, Ubon Ratchathani, Thailand.

Numyoot SONGTHANAPITAK, Ph.D.
President of Rajamangala University of Technology Thanyaburi
andConference Chairman of 10th EMSES 2012
PREFACE:
Message from the Director of
Institute of Advanced Energy, Kyoto University

It is my great pleasure to have the 10th Eco-Energy and Materials Science and Engineering Symposium (EMSES) with Rajamangala University of Technology Thanyaburi (RMUTT) under the long-term collaboration between RMUTT and Kyoto University. The 1st EMSES was held in 2001 in Thailand and the symposium has been expanded in its scientific contents as well as the academic network. I believe that the 10th EMSES gives a good opportunity to all participants to exchange their knowledge and idea to realize eco-friendly energy system in society. I would like to express my welcome to all participants and sincere thanks to the 10th EMSES organizing committee and all supporting organizations to make us having this symposium.
I hope that the symposium will be successful and lead to further progress in energy science and technology and also in friendships of participants.

Professor Yukio Ogata, Ph.D.
Director of Institute of Advanced Energy, Kyoto University
PREFACE:
Message from the Former Dean of
Graduate School of Energy Science, Kyoto University
Program Leader,
Global COE “Energy Science in the Age of Global Warming”

I want to express my hearty welcome to all participants of Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This symposium is aiming the realization of importance of energy and materials technology through the academic, science and technology network among the world communities. The symposium gives an opportunity for researchers to discuss their research works and also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the further cooperation work will be developed.

I would like also extend my sincere thanks to all who made the meeting possible, including the 10th EMSES organizers, the SEE forum committee members, and the Japanese Government, JSPS, for their kind support. I am looking forward to seeing you in Ubon Ratchathani, Thailand.

Y. Yao

Professor Takeshi YAO, Ph.D.
Former Dean of Graduate School of Energy Science, Kyoto University
and Program Leader, Global COE “Energy Science in the Age of Global Warming”
Message from the Chairperson of 10th EMSES Organizing Committee

Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is pleased to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES).

RMUTT has a major mission on encouraging and supporting all areas of research. One of the key reasons is to assist in developing capability in science and technology in order to cope with recent rapid change in this field. We have jointly set up an academic symposium on the 10th EMSES with the perception on the significance of exchanging knowledge and research experiences between researcher in the field of energy, materials technology and environmental science. This symposium is not only giving an opportunity for Thai and foreign researcher to present and discussion their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the closer future cooperation incorporate with joint-research works will be developed. Thus, to support the aforesaid role, the symposium working committee would like to invite you to participate in this academic symposium.

I would like to express our sincere thanks to the organizing committee, participants and contributors for your kind corporation to this symposium. I wish this symposium proceeding will be a useful reference for future scientific research development.

Sommai PIVSA-ART, Ph.D.
Dean of Faculty of Engineering, RMUTT
Director of CoE on Sustainable Energy System (Thai-Japan)
Organizing Chairman of 10th EMSES 2012
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# Conference Program of 10th Eco-Energy and Materials Science and Engineering

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<td>09:45-10:45 am</td>
<td>Keynote Speaker I: Japan Power Generation Mix and Energy Security after Fukushima Nuclear Accident, presented by Professor Dr. Keiichi N. Ishihara, Graduate School of Energy Science, Kyoto University, Japan</td>
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## 10th EMSES 2012

### Conference Program of 10th Eco-Energy and Materials Science and Engineering

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### 8th December 2012

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Prof. Dr. Takeshi Yao, Leader of GCOE Program/Professor, Graduate School of Energy Science, Kyoto University

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Separation of D, L-Lactic Acid by Filtration Process

Anan Boonpan*, Sommai Pivsa-art, Sirikhao Pongsawat, Atsadawut Areesrisuk and Piyamas Sirisangawong

1Division of Biology, Faculty of Science and Technology, Rajamangala University of Technology Thanyaburi, Khlong 6, Thanyaburi, Pathumthani, Thailand
2Department of Chemical and Materials Engineering, Faculty of Engineering, Rajamangala University of Technology Thanyaburi, Khlong 6, Thanyaburi, Pathumthani, Thailand

Abstract—The purpose of this research was to study the optimum conditions for separation of D,L-lactic acid isomer by filtration process using polytetrafluoroethylene (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) β-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™ 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 β-cyclodextrin 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. 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.

Keywords—D,L-lactic acid, polytetrafluoroethylene membrane, filtration process, chiral selector

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 poly(lactic acid) (PLA), which is well-known as a sustainable biobased 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-isomers (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 parameter for 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 use [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], esterification and hydrolysis [13], supported liquid membrane system [3, 4, 17], and bipolar electro dialysis [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

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 enantiomers. Aliquat™ 336 (trietylphosphonium 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.

Fig.1. Schematic of filtration device for D, L-lactic acid enantioseparation

* Corresponding author: fax: (662) 549-4119
E-mail address: ananboonpan@hotmail.com
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]

\[
J = \frac{C}{A \cdot \Delta t}
\]

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

\[
a = \frac{\left(\Delta t \Delta t + \Delta \lambda \Delta \lambda \right)}{J_{L,L} - J_{D,L} - J_{D,L}}
\]

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 (a) 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 (a) 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 diastereomer 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,L} ) (mol/(cm²/min))</th>
<th>( J_{D,L} ) (mol/(cm²/min))</th>
<th>( \alpha )</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>
</tbody>
</table>

| Racemic mixture of D- and L-lactic acid with BSA |
| 10        | 0.0078                        | 0.0097                        | 0.80       |
| 30        | 0.0071                        | 0.0066                        | 1.08       |
| 60        | 0.0036                        | 0.0035                        | 1.03       |
| 100       | 0.0022                        | 0.0021                        | 1.02       |

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:

\[
2 \text{NR}_2\text{Cl} + \text{CO}_3^{2-} \leftrightarrow (\text{NR}_2\text{Cl})_2\text{CO}_3 + 2\text{Cl}^{-}
\]

Where \( \text{NR}_2\text{Cl} \) and \((\text{NR}_2\text{Cl})_2\text{CO}_3 \) represents Aliquat™ 336 in chloride and carbonate form, respectively. Aliquat™ 336 and its modified form could be combined with lactic acid form and 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 2 Enantioseparation of D, L lactic acid using the Aliquat™ 336 and its modified form as the carrier.

<table>
<thead>
<tr>
<th>Chiral selector and carrier</th>
<th>J_{L\rightarrow A} (mol/cm²:min)</th>
<th>J_{D\rightarrow A} (mol/cm²:min)</th>
<th>( \alpha )</th>
<th>( \frac{J_{L\rightarrow A}}{J_{D\rightarrow A}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>β-CD</td>
<td>0.0067</td>
<td>0.0025</td>
<td>2.67</td>
<td></td>
</tr>
<tr>
<td>β-CD and Aliquat™ 336</td>
<td>0.0069</td>
<td>0.0024</td>
<td>2.83</td>
<td></td>
</tr>
<tr>
<td>β-CD and modified</td>
<td>0.0071</td>
<td>0.0024</td>
<td>3.04</td>
<td></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 3 Enantioseparation of D, L lactic acid by two-stages filtration process

<table>
<thead>
<tr>
<th>Stage*</th>
<th>J_{L\rightarrow A} (mol/cm²:min)</th>
<th>J_{D\rightarrow A} (mol/cm²:min)</th>
<th>( \alpha )</th>
<th>( \frac{J_{L\rightarrow A}}{J_{D\rightarrow A}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0070</td>
<td>0.0024</td>
<td>2.93</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.0066</td>
<td>0.0015</td>
<td>4.43</td>
<td></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 Aliquat™ 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.

5. ACKNOWLEDGEMENT

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6. REFERENCES


