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By Takatsugu Endo




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2 To cite this article: Takatsugu Endo, Mai Tatsumi, Kosuke Kuroda, Heri Satria, Yumiko Shimada, Kazuaki Ninomiya & Kenji Takahashi (2017) Efficient recovery of ionic liquid by electrodialysis in the acid hydrolysis process, Separation Science and Technology, 52:7, 1240-1245, DOI: 10.1080/01496395.2017.1281957

9 To link to this article: <http://dx.doi.org/10.1080/01496395.2017.1281957>

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9  Accepted author version posted online: 19 Jan 2017.
Published online: 19 Jan 2017.

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Efficient recovery of ionic liquid by electrodialysis in the acid hydrolysis process

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1 ABSTRACT

Electrodialysis (ED) has been recently known as a highly effective technique to remove and recover ionic liquids from aqueous solution. When a conventional electrolyte solution for the ED process containing Na₂SO₄ was used, a recovery ratio of an acidic IL, 1-butyl-3-methylimidazolium hydrogen sulfate ([Bmim][HSO₄]), was 90%. On the other hand, the value clearly increased to 96% when we employed [Bmim][HSO₄] as the electrolyte solution. In an acid hydrolysis of bagasse using the IL under microwave irradiation, the recovery ratio maintains 96%, irrespective of reaction time. This demonstrates the applicability of the proposed ED system in biomass processing.

ARTICLE HISTORY

Received 9 June 2016
Accepted 10 January 2017

KEYWORDS

Acid hydrolysis;
electrodialysis; ionic liquids

Introduction

Since the current chemical productions and energy resources unambiguously depend on fossil fuels with the problems of global warming and depletion, it is urgent to find renewable resources to replace with them for sustainable society. Lignocellulosic biomass, mainly composed of three biopolymers such as cellulose, hemicellulose, and lignin, is a promising candidate due to the abundant nature, however, its recalcitrance makes the utilization of lignocellulosic biomass challenging. To overcome the recalcitrance, conventional chemical pretreatments and corrosive chemicals with harsh reaction conditions. Ionic liquids (ILs) that are salts with the melting point around room temperature recently emerged as pretreatment solvents for lignocellulosic biomass under relatively mild conditions.^[1–3] Not only high efficiency in the pretreatment, but also environment-friendly nature that originated from practical non-volatility and non-flammability have attracted considerable interest. Additional significant character is their designability. ILs are composed of only cations and anions where functions are easily introduced. Since realization of one-pot biomass processing is highly desired due to its cost-effectiveness, ILs with an acidic function or less toxicity for biological reactions have been developed, aiming to achieve one-pot biomass processing of acid hydrolysis and enzymatic hydrolysis (and subsequent fermentation), respectively.^[4–8]

Separation and recovery of ILs are necessary in such processes, the latter of which is particularly important to minimize cost of biomass processing. However, it is problematic due to their non-volatile and hydrophilic nature. Electrodialysis (ED), a technique to transport ions through ion exchange membranes under an applied voltage, is strongly effective for this purpose. Actually, ED has been already applied to remove and recover ionic liquids from aqueous solution,^[5,9–15] even in the presence of hydrolyzates. Even though 99% removal of IL from an aqueous solution with ED is feasible,^[12–14] the recovery ratio of IL, which is the most crucial value in terms of IL recycle, is far from 100%. For instance the values of 85.2% and 76.9% for 1-butyl-3-methylimidazolium chloride ([Bmim][Cl]) were reported by two groups^[11,12] as the optimized conditions, and the former one is considered the highest value ever reported among all the ILs applied in the ED process.

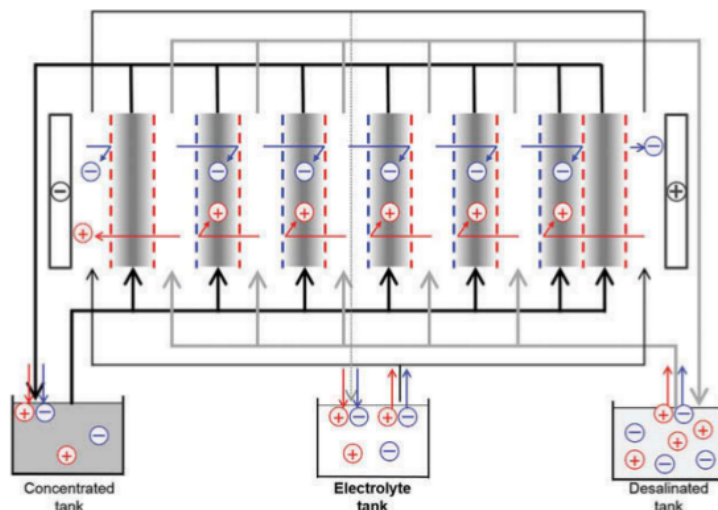
The aim of the present work is to establish an ED process with high IL recovery ratio and subsequently apply the process to an acid hydrolysis biomass process. To improve the efficiency of recovery, we particularly focus on undesirable ion exchanges through the ion exchange membranes between electrolyte solution and desalinated/concentrated solutions, which possibly decreases the recovery ratio. Figure 1 shows a schematic diagram of the electrodialysis system used here (basic principle is the same for other ED apparatuses). There are three solution tanks in ED system. Ions of

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Figure 1. A schematic diagram of the used electrodesialysis system.

interest first exist in the desalinated solution tank and are migrated to the concentrated solution tank during ED. The electrolyte solution flows past both an anode and cathode. This flow is to carry current across the stack, therefore, ion exchanges between the electrolyte solution and the desalinated/concentrated solution tanks through the membranes occur in principle. However, the effect of the ion exchanges on IL recovery with ED has ever been totally overlooked. To improve the recovery ratio, in this study we employed the same IL for the electrolyte solution as used for recovery, which may cancel practical ion exchanges between the solutions.

Experimental section

Materials and analyses

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An acidic ionic liquid, 1-butyl-3-methylimidazolium hydrogen sulfate ([Bmim][HSO₄], 99% purity) purchased from Kanto Chemical CO., was used through whole the experiments as received. IL concentrations before and after ED were determined with quantitative ¹H NMR spectroscopy (JEOL JNM-ECS400 or JNM-ECA600) or high-performance liquid chromatography (HPLC, SHIMADZU system). Our HPLC system unfortunately gives the peaks of [Bmim][HSO₄] and Na₂SO₄ at the same retention time but that of glucose at different retention time. On the other hand, the qualitative ¹H NMR measurements does not suffer from the presence of Na₂SO₄ whereas signal sensitivity is low compared to HPLC. These techniques were used depending on circumstances. It was confirmed that

both measurements provided consistent results within experimental error (ca. ±1%). Qualitative ¹H NMR measurements were carried out with 3-(trimethylsilyl)-1-propanesulfonic acid-d₆ sodium salt (DSS-d₆ material reference, 92.3% purity) purchased from WAKO, as an internal reference in D₂O solution with a recycle delay of 60 s and averaging of collections of 16 free inductions decays. Representative data for qualitative ¹H NMR and HPLC measurements are displayed in Supporting Information.

Electrodialysis

Electrodialysis was performed using a Selemion electrodesialyzer (DW-Lab, AGC Engineering Co., Ltd, Tokyo, Japan). The system comprised of a membrane stack, three tanks for desalinated, concentrated, and electrolyte solutions, and a DC power supply (PMC18-3A, Kikusui Electronics Co., Yokohama, Japan). The membrane stack comprised of five pairs of Selemion AMV anion exchange and CMV cation exchange membranes (Fig. 1). The thickness of the membranes is 100–200 μm and the effective membrane area is 30 cm² in all compartments. Each membrane is placed at the interval of 0.75 mm. The desalinated, concentrated, and electrolyte solutions were circulated at 4 L·min⁻¹ using pumps (RD-05V24, Iwaki Co., Ltd, Tokyo, Japan) with applied voltage of 8 V. The amount of solution in each tank was initially 250 g, and after ED, the change in the solution amount was weighted. The desalinated solution contained 1 mol·L⁻¹ of the IL while pure water was used for the concentrated solution. For the electrolyte solution, Na₂SO₄/H₂SO₄ or [Bmim][HSO₄] aqueous solution with an appropriate concentration was employed (vide infra).

Prior to obtaining data, the (preliminary) ED was carried out 5 times to replace counter-ions initially existing in the ion exchange membranes with ions that were transported. Transport behavior of the IL was monitored by measuring electronic conductivity in the desalinated solution using a conductivity meter (LAQUA DS-70, HORIBA Scientific Ltd, Kyoto, Japan), and ED experiments were terminated when the conductivity became constant reaching almost zero. The calibration of conductance was performed with $0.01 \text{ mol}\cdot\text{L}^{-1}$ KCl aqueous solution at room temperature.

The recovery ratio of the IL (%) was determined by qualitative ^1H NMR spectroscopy or HPLC as,

$$\frac{C_C V_C}{C_D V_D} \times 100 \quad (1)$$

where C is the concentration and V is the volume for the solutions. Subscripts C and D stand for the concentrated tank (after ED) and the desalinated tank (before ED), respectively.

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Acid hydrolysis of lignocellulosic biomass

Acid hydrolysis of lignocellulosic biomass (bagasse) was performed with [Bmim][HSO₄] under microwave irradiation (Start SYNTH, Milestone). Bagasse powder (approximately < 3 mm) was purchased from Sanwa Cellulose Co., Ltd. (Yokkaichi, Japan). The bagasse powder was grinded in a mill and then sieved to obtain a powder of 250–500 μm . One gram of dried bagasse containing ca. 40% cellulose was immersed into a 50 ml aqueous solution of $1 \text{ mol}\cdot\text{L}^{-1}$ [Bmim][HSO₄], then the solution was poured into a tightly sealed container for microwave irradiation. Microwave power and temperature were set to be 500 W and 190°C, respectively. A tiny amount of the solution was taken in a time lapse

(10, 20, 30, 40, 50, and 120 minutes) to determine glucose yield by HPLC, the procedure of which was done in triplicate. ED experiments were performed for the obtained hydrolysate solution including the IL with the abovementioned condition after the solution was filtrated several times to remove solid residues.

Results and discussion

Electrodialysis of [Bmim][HSO₄]

Figure 2 shows ED results of [Bmim][HSO₄] using three electrolyte solutions. The electrolyte solution of a 4.0 wt% aqueous Na₂SO₄ solution (black in the figure) with a slight amount of H₂SO₄ is regarded as reference. The role of acid in the solution is to adjust the pH of 2–3 to neutralize hydroxide ions formed in the cathode during the ED process. When [Bmim][HSO₄] is utilized as the electrolyte solution (red), the concentration becomes 0.2 wt% because the IL is acidic and the pH falls in 2–3 with this concentration. Therefore, 0.2 wt% of Na₂SO₄ with a drop of H₂SO₄ (black) was also tested for comparison. In Fig. 2(a) displays conductivity changes in the desalinated solutions against ED time. The change is the most rapid for 4.0 wt% Na₂SO₄ while that for 0.2 wt% Na₂SO₄ shows the slowest. The ED processes were terminated when the conductivity becomes almost constant, the values of which were ca. 1% with respect to the initial values. The observation of 99% removal of the IL ions with ED is in line with the previous results.^[12–14]

Figure 2(b) displays the recovery ratios of ILs after ED. The ED with the conventional electrolyte solution provides the recovery ratio of ca. 90%. This value is somewhat larger than the optimized value for the [Bmim][Cl]

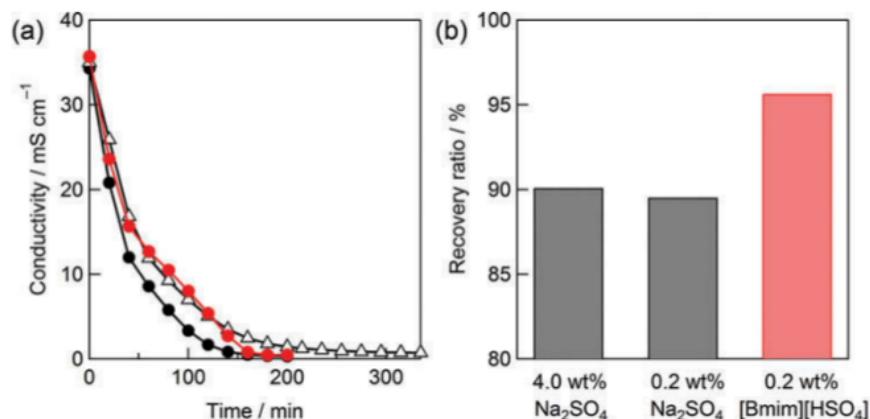


Figure 2. (a) Conductivity change in the desalinated solutions (4.0 wt% Na₂SO₄: black circle; 0.2 wt% Na₂SO₄: open triangle; 0.2 wt% [Bmim][HSO₄]: red circle) during ED. (b) Recovery ratios of the IL with ED using different electrolyte solutions, determined by qualitative ^1H NMR measurements.

recovery ratio of 85.2%,^[11] which is the highest one previously reported. Although the reason that our value is the higher could partly originate from the difference in apparatus used, a possible explanation is higher electrochemical stability of HSO_4^- anion than Cl^- anion.^[16] The Cl^- anion could be partially consumed in anode and turned into Cl_2 gas, which reduced the recovery ratio of $[\text{Bmim}][\text{Cl}]$. The recovery ratio with 0.2 wt% Na_2SO_4 was the same as that with 4.0 wt%. Then, the effect of the Na_2SO_4 concentration only appeared in desalination speed. The lowered desalination speed is caused by smaller current across the stack due to low ion concentration. However, when using $[\text{Bmim}][\text{HSO}_4]$ that is the same IL to recover for electrolyte solution, the ratio clearly increases and reaches to ca. 96%. This demonstrates that there is a non-negligible loss of IL during ED with the conventional electrolyte. The reason for the increase is, as expected, that the ions in the $[\text{Bmim}][\text{HSO}_4]$ electrolyte solution are transported into the concentration solution which compensates the leaking of IL from the concentrated/desalinated solutions.

Even though the recovery ratio reaches to 96–7%, there is still a slight gap between removal and recovery. Table 1 lists the mass balance of the IL for the ED process. Since the weight of the IL in electrolyte solution does not change, 2–3% loss exists in the total mass. Trinh *et al.* reported^[12] IL ions deposition on ion exchange membrane surface during ED of ILs,

famously known as the fouling phenomenon, which could be the main reason for the unbalanced mass. It should be noted that the fouling of ILs may occur because the size of IL ions is competitive to the size of pores of the ion exchange membranes where ions pass through.^[15] Another possibility is an electrochemical decomposition of the IL on electrodes due to long ED time (3–4 hours) despite of high electrochemical stability of the IL^[16] compared to H_2O .

Electrodialysis of IL/hydrolysate mixture

Next question is whether this ED system is applicable to biomass processing, *i.e.*, recovery and separation of IL from hydrolysate solution after hydrolysis of lignocellulosic biomass. In Fig. 3 the change in glucose production from bagasse in the presence of $[\text{Bmim}][\text{HSO}_4]$ is displayed against the reaction time under microwave irradiation. Microwave irradiation for polar molecules is known to result in higher enhancement of chemical reactivity than conventional heating.^[17] This is caused by intermolecular frictions of polar molecules under alternative electric field, which leads to rapid heating. Utilization of an acidic IL as a catalyst instead of conventionally used inorganic acid is also advantageous.^[18,19] Amarasekara and Wiredu reported that glucose yield increased from 16.2% to 22.2% when switching used acid from H_2SO_4 to an acidic IL, possibly due to an adsorption^[16] of the IL ions on cellulose surface.^[18] In the current system, the maximum yield of roughly 20% was obtained at 30 min. The theoretical curve in the figure represents the assumption of the consecutive

Table 1. Mass balance of $[\text{Bmim}][\text{HSO}_4]$ before and after ED, determined by qualitative ^1H NMR measurements.

	Desalinated solution /g	Concentrated solution /g	Electrolyte solution /g	Total/g
Before ED	11.8	0.5	0.0	12.3
After ED	0.1	0.5	11.4	12.0

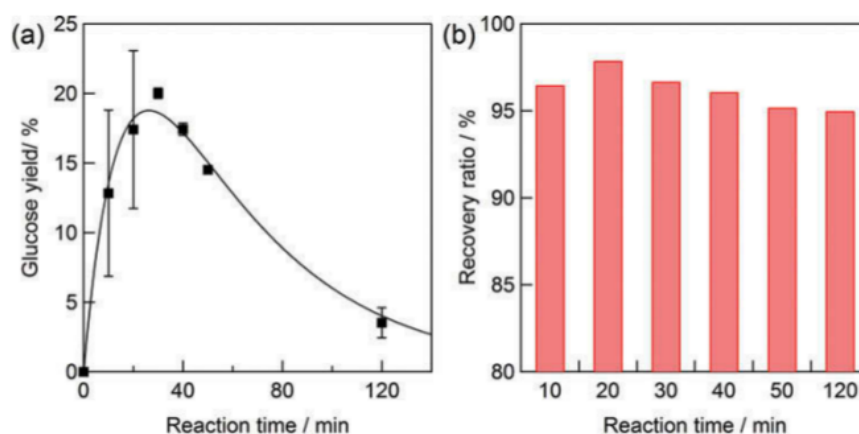


Figure 3. (a) Glucose yield of bagasse in acid hydrolysis against reaction time under microwave irradiation at 190°C . Error bars represent standard deviation of the triplicate experiments. The fitting curve was obtained assuming the consecutive first order reaction (Eq. 2). The rate constants of k_1 and k_2 were estimated to be $0.3 \times 10^{-3} \text{ s}^{-1}$ and $1.1 \times 10^{-3} \text{ s}^{-1}$, respectively, under the current conditions. (b) Corresponding recovery ratios of the IL, determined by HPLC.

first order reaction, that is, cellulose \rightarrow glucose \rightarrow degradation products.

$$[G] = A_0 \frac{k_1}{k_2 - k_1} (e^{-k_1 t} - e^{-k_2 t}) \quad (2)$$

$[G]$ is the concentration of produced glucose, A_0 is the initial concentration of cellulose, and k_1 and k_2 are the rate constants of the saccharification and the glucose degradation, respectively. After 40 minutes glucose yield decreases with time because the conversion of glucose to degradation products is dominant in this time domain.

Figure 3(b) shows recovery ratios of the IL with respect to each reaction time. The recovery ratio is hardly dependent on time, providing the average value of 96.3% which is consistent with the data shown in the former section (Fig. 2b). This result demonstrates that the ED technique, with the adoption of IL for electrolyte solution to improve recovery ratio, is also applicable to biomass processing. Since ^1H NMR spectra (Fig. 4) as well as FT-IR spectra and thermogravimetric analysis curves (Figure S2) of the recovered ILs do not differ from those of the original IL, it is strongly suggested that these recovered ILs are reusable.

Table 2 lists distributions of the IL and glucose to desalinated and concentrated solutions after the ED process to evaluate separation efficiency for the current

Table 2. Distributions of the IL and glucose after ED, determined by HPLC. The weights prior to ED were set to be 100%. The average values of data between 10 to 120 minute microwave irradiations are displayed because the glucose distribution values have a relatively large error due to the low concentration (roughly 1/100 relative to the IL).

Ionic liquid/%		Glucose/%	
Desalinated solution	Concentrated solution	Desalinated solution	Concentrated solution
0.4 \pm 0.1	96.3 \pm 1.1	93.7 \pm 4.1	4.2 \pm 1.0

system. As already indicated above, 96.3% of the IL was recovered in the concentrated solution while less than 1% of the IL remained in the initial desalinated solution. Although most of the glucose remains in the desalinated solution, roughly 4% of glucose transports to the concentrated solution. Cheng *et al.* also observed hydrolysate loss during the ED process for acid removal.^[20] In the case of glucose their value was 3%, which is consistent with our data. Neutral molecules should not be affected by applied voltage, however, they could penetrate the membranes by molecular diffusions with time.

Conclusions

We have addressed a relatively low recovery ratio of ILs previously reported with ED. We focused on electrolyte solution where Na_2SO_4 aqueous solution with a drop of H_2SO_4 was conventional employed. The electrolyte solution was switched to the IL solution that was the same IL as the one used for recovery. With changing electrolyte solution the recovery ratio increased from 90% to 96%, demonstrating that the ion exchange through ion exchange membranes between electrolyte and desalinated/concentrated solutions clearly lowered the recovery ratio. The recovery ratio did not change when the developed ED system was utilized in the acid hydrolysis process for bagasse under microwave irradiation. The ^1H NMR spectrum of the recovered ILs was the same as that of the original IL. These results indicate that the developed ED system is applicable to other IL-assisted biomass processing, e.g., one-pot hydrolysis processes. Since ED is advantageous to separation of acid from hydrolysates compared to other methods, such as overliming and neutralization,^[21,22] our results will contribute further development of biomass processing with this technique.

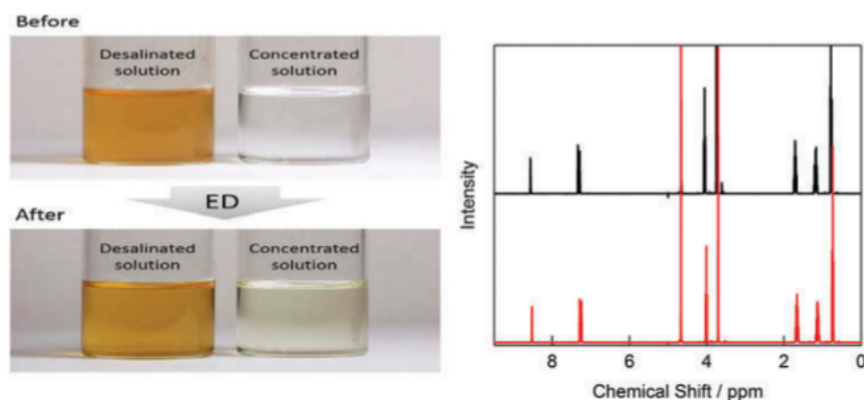


Figure 4. Appearance change of the desalinated and concentrated solutions (left) and ^1H NMR spectra of $[\text{Bmim}][\text{HSO}_4]$ in D_2O (right). These data were collected for the experiments with microwave irradiation time of 40 minutes. In the right figure black and red lines represent the original and recovered IL, respectively. For the peak assignments, please refer to Figure S1.

Funding

5 This study was supported in part by the Advanced Low Carbon Technology Research and Development Program (ALCA) (Grant number 2100040), the Cross-ministerial Strategic Innovation Promotion Program (SIP), and the Center of Innovation Program "Construction of next-generation infrastructure using innovative materials: Realization of safe and **8** pure society that can coexist with the Earth for centuries" from Japan Science and Technology Agency, and also by JSPS KAKENHI Grant Number 15H04193.

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