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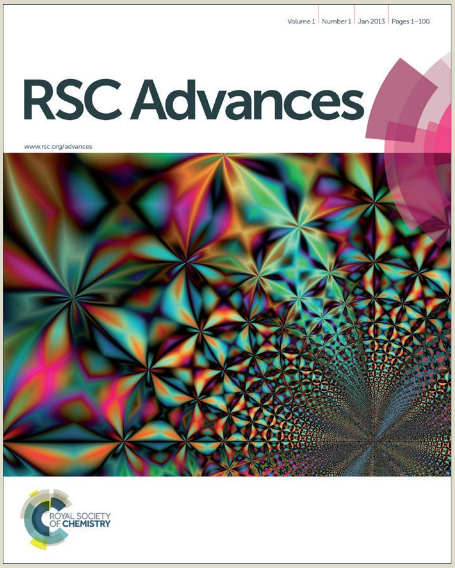

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Retrieval of CO_2 from carbonate solution for its sequestration by a novel electrochemical decarbonizing and ingathering method

Yang Yang[a], Yun Tian[a], Zucheng Wu*[a]

Many attempts at CO_2 recovery and sequestration have been made in recent years. The use of an electrochemical decarbonizing and ingathering method exhibits the ability to segregate targeted ions absorbed in a dilute solution. However, because of the low conductivity of the dilute solution, a great part of the energy is consumed to overcome the internal resistance. In this study, an "electro-dynamic inspissation (EDI)" system was introduced to examine the retrieval of carbonate ions from a sodium carbonate solution. The addition of an ion exchange resin serving as a conductor in the EDI device provides a new way to enable CO_2 recovery from the concentrated stream; thus, a purified CO_2 stream was obtained. The properties of the EDI system were characterized in detail, addressing its recovery effectiveness and providing a cost evaluation. The results indicate that EDI performs well, with the current efficiency reaching 75% and the energy consumption calculated as 1.41 MJ/kg of CO_2 captured. This system is alternative to most traditional technologies, such as amine absorption/thermal desorption.

1 Introduction

CO_2 emissions due to human activities are forcing climate change and have raised concerns during the past decades. According to statistical data from the global carbon project, worldwide carbon emissions from human activity reached 36 billion tons in 2013, which set a record. China, America and Europe deserve much of the blame for this emission level because they had the three highest carbon emission levels over this period. Therefore, these countries are working together to confront the problem of CO_2 emission reduction. However, the current economy is so heavily dependent on fossil fuel that energy switching is impossible in the near future. Hence, carbon capture and storage (CCS) technology is regarded as an effective way of achieving CO_2 emissions reduction in the near-term [1-2]. In the CCS process, CO_2 emitted from point sources is captured and transported to storage sites, where liquid CO_2 is injected either underground or into the sea. Currently, the CO_2 capture process is the most expensive step in the CCS scenario [3]. Therefore, the biggest challenge to implementing CCS is reducing the cost of the capture process.

With current technology, several methods have been evaluated for recovering CO_2, including membrane separation [4-8], absorption (pressure swing adsorption, temperature swing adsorption) [9-13], adsorption and [14-16] cryogenic separation [17-18]. Among these methods, chemical absorption using liquid absorbents has gained acceptance from an industrial viewpoint [1,19]. This method captures CO_2 using amine absorbents, which have a strong affinity to CO_2. Then, CO_2-amine form solvents are recovered in the form of a temperature swing, whereby a concentrated CO_2 gas stream is obtained. However, it is well-known that the thermal treatment requires a large amount of thermal energy, in the range of 3-4 MJ/kg-CO_2, of which 28% to 42% represents the thermal desorption energy [20]. Therefore, an alternate way to recover CO_2 from an absorbent solution is urgently needed.

An alternative method is to recover CO_2 by acidification of the absorbent solution with electrodialysis (ED) technology. Theoretically, the ideal power consumption for carbonate recovery from alkaline absorbent (self-regeneration of NaOH) is approximately 0.8 MJ/kg, which is far less than for recovering CO_2 from amine absorbents. Recently, electrodialysis with bipolar membranes [20-22] has been utilized for recovering CO_2 from chemically absorbed solutions. Electrodialysis is able to provide protons and simultaneously remove the metal ions from solution. Because carbonate is a weak acid, gaseous CO_2 is obtained when protons are provided. As opposed to the thermal desorption (same as amine absorbent to first capture CO_2 gas), water was electrochemically dissociated to acidify the solution and then to release the CO_2 via an ED device, which means that less energy was needed. Moreover, the regeneration of the alkaline solution can be recycled to capture carbon dioxide in flue gases. However, the current efficiency was only approximately 30% [20], which may be caused by the leak transport of hydroxyl ions across the anion membrane. The lower current efficiency suggested that the impact of the leakage of hydroxyl ions from the alkaline compartment to the regeneration compartment should be reduced for the case where the hydroxyl ions decreased the pH value in the regeneration compartment, which was unfavorable for CO_2 recovery. Electro-deionization (EDI) technology was originally developed for purifying water and optimized on the basis of electrodialysis by filling the dilute chamber with resin. This technology should have a higher efficiency than the ED process because it reduces the resistance [23] across the cell pairs, and ions produced by water splitting are mainly used for resin regeneration. From this point of view, EDI shows substantial

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[a] *Department of Environmental Engineering, Laboratory of Electrochemistry and Energy Storage; State Key Laboratory of Clean Energy Utilization, Zhejiang University, Hangzhou 310027, China Tel: +86-571-85660839. E-mail: wuzc@zju.edu.cn*

possibility for further increasing the current efficiency of carbonate recovery and reducing power consumption.

To the best of our knowledge, electro-deionization technology has not been fully investigated for recovery of carbonate from alkaline solutions. In our previous work, we employed this technology to recover some metal ions as useful chemicals instead of discarding them [24-28]. Therefore, the technology is not only used to "de-ionize" water to purify it but also is a novel desalination technology for gathering or recovering ions under electro-forces, namely "electro-dynamic inspissation (EDI)", same abbreviation as EDI. In this study, a modified EDI apparatus was innovated and examined. The object of this study was to demonstrate the utilization of the apparatus to recover CO_2 from an alkaline solution, to examine its CO_2 recovery performance and to evaluate its power consumption over a range of conditions. The properties of EDI were characterized in addressing the costs and availability of the process to obtain pure CO_2, which can be used as feedstock for fuels and chemical synthesis rather than disposing of it as waste [29-30].

2 Experimental

2.1 Setup

The bench-scale electro-dynamic inspissation (EDI) apparatus used in this study is shown in Figure 1. It consisted of four chambers: the cathode compartment, the dilute compartment (resin chamber), the concentrated compartment (regeneration chamber), and the anode compartment. We named them chambers 1-4, respectively. Membranes served as separators between the compartments. For each membrane, the effective surface area of the membrane was 38.4 cm^2 and the spacer channel width between two membranes was 0.7 mm. There was only one cell pair in this stack, with two pieces of anion ion exchange membrane (AEM) and one piece of cation ion exchange membrane (CEM) used. The commercially available membranes used in this experiment were purchased from Tianwei, China. The D201 strongly alkaline styrene-divinylbenzene anion exchange resin (AER) (ZhengGuang, China) was packed into the dilute chamber before conventional pretreatment. A Ti substrate RuO_2 electrode and a stainless steel electrode, which have the same effective area as the membrane, are used as the anode and cathode electrode, separately. A DC adjustable power supply (SiLing) was used to supply constant current across the electrodes.

All the chambers were connected to a separate external container for continuous recirculation by four peristaltic pumps (Longer pump BT100-2J). The concentrated compartment was connected to a vapor-liquid equilibrium vessel that is transformed from a conical flask. The measurement of flow rate was accomplished by a rotameter from the feed compartment. Three parallel water samples and gas samples were taken from the concentrated feed water for analysis at an appropriate interval. The pH and conductivity measurements were performed on every sample using a Metter Toledo 320-S pH meter and a LeiCi conductivity meter.

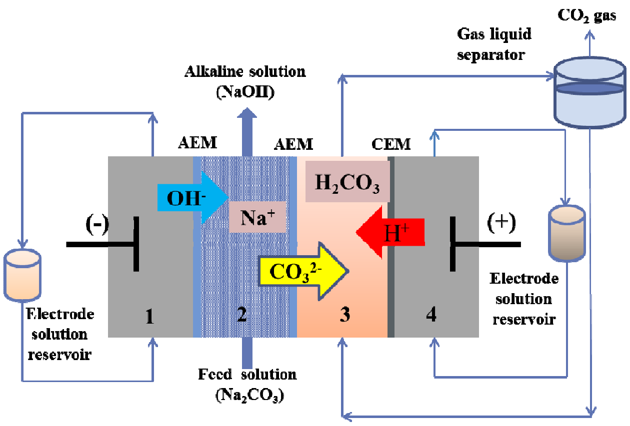

Figure 1. EDI system

The gas-phase CO_2 concentration was analyzed using a FuLi gas chromatograph (9790) with a thermal conductivity detector TCD detector and a TDX-01 packed column. The liquid-phase CO_2 concentration was determined by inorganic carbon analysis using a TOC-VCSN carbon analyzer.

2.2 Method

In the EDI process, anion and cation membranes were severally arranged in the cathode and anode to isolate the chamber. The resin chamber was isolated by two anion membranes, and the regeneration chamber was placed between one cell pair (CP, a pair of anion and cation ion exchange membranes). Under applied current, protons and hydroxyl ions migrated out of the electrode interface to the adjacent chamber in opposite directions.

The concept behind the method is as follows [31-32]. First, carbonate-absorbed solution is passed upwards through the cation resin exchange compartment. Carbonate ions in the solution exchange with mobile ions in the ion exchange resin and are then transported to the concentrated compartment through the anion membrane. Sodium ions are repelled by the fixed negative charge in the anion membrane (AEM) and cannot permeate through the AEM; they remain in the resin compartment and combine with hydroxyl ions detached from the resin. When the resin is saturated with carbonate ions, it can be regenerated by hydroxyl ions produced by water dissociation.

$$CO_3^{2-} + R\text{-}OH^- = R\text{-}CO_3^{2-} + OH^- \quad (1)$$

$$Na^+ + OH^- = NaOH \quad (2)$$

$$OH^- + R\text{-}CO_3^{2-} = R\text{-}OH^- + CO_3^{2-} \quad (3)$$

Then, the protons produced by water electrolysis in the anode electrode move to the concentrated compartment. Because the hydrogen ions have the fastest migration rate of all the ions present, the pH of the concentrated chamber is quickly lowered to less than pH 7. Because carbonate is a weak acid, at a pH less than 6, dissolved carbonate in the absorption solution reconverts to gaseous CO_2. Therefore, gaseous CO_2 is generated in the concentrated compartment according to the following reaction:

$CO_3^{2-} + H^+ = HCO_3^-$;

$HCO_3^- + H^+ = H_2CO_3$;

$H_2CO_3 = H_2O + CO_2$ (4)

2.3 Calculation

The system was operated in a once-through mode where a volume of water is passed through the EDI unit only once.

The current efficiency (CE) of ion A (here, the carbonate ion) was calculated as the ratio of the electrical charge used for the transport of ion A to the total electrical current charge. The applied current being constant, the current efficiency of ion A (η_A) can be calculated as

$$\eta_A = \frac{r_{CO2} \times z \times F}{n \times i \times S} \quad (5)$$

where z is the charge number of ion A, F is the Faraday constant, i is the current density (A/m^2), S is the area of the membrane (m^2), and n is the number of cell pairs in the EDI stack.

The CO_2 recovery rate, r_{CO2} can be expressed by the following equation:

$$r_{CO2} = \eta_A \times \frac{I \times n}{F} \quad (6)$$

where F is the Faraday constant, n is the number of cell pairs in the EDI stack, η_A is the current efficiency, and I is the applied current.

The energy consumption (EC) is defined as the energy required for transporting ions from the feed stream to the concentrated stream (kwh/mol). It is also an important technical specification to measure the practical feasibility for CO_2 recovery. It is calculated using the following equation:

$$E = \frac{U \times i \times S}{m_{CO2} \times r_{CO2}} = \frac{U \times F}{m \times \eta \times n} \quad (7)$$

Where U is the component voltage of the feed chamber (V), i is the current density (A/m^2), and S is the area of the membrane (m^2).

3 Results and Discussion

3.1 Effect of resin

Table 1 lists the difference in the solution resistance for the apparatus with resin and without resin. The comparison was conducted by adding resin to the dilute compartment. The corresponding condition was fixed: 0.15 A current and 10 mmol/L influent solution.

Table 1 show that adding resin will remarkably reduce the resistance of the dilute chamber, and accordingly, the component voltage is significantly decreased. This is mainly because ion is transported by resin when working in the low-salt solution. The conductivity of the migration channel formed by adjacent resins is 2-3 orders of magnitude higher than that formed by aqueous solution[33]. As power consumption is mainly affected by the component voltage of the feed chamber, power consumption should be decreased by adding resin. In previous studies, the current efficiency of the ED process was low, possibly due to the resistance consumption. An obvious resistance reduction is shown in Table 1, which suggests that the current efficiency will be improved. Therefore, the results show a promising prospect for recovering carbonate alkaline solution compared with previous work, and the result is likely to be superior to those of previous work without resin addition in terms of EC and CE.

Table 1. Comparison performance of EDI apparatus with resin and without resin

	Resistance of feed solution chamber (Ω)	Component voltage percentage of total voltage (%)
Without resin	29.33	34.64
With resin	19.87	25.04

3.2 Effect of current

It is well-known that working current is important in ion exchange membrane processes [34-36]. Figure 2 shows the I-V relationship of cation and anion electrodes, where the feed concentration is 600 mg/L and the flow rate of the feed solution in chamber 2 is 0.6 L/h. We examine the current variation by varying the voltage from 2V to 40V every 10 seconds (supporting Information, Figure S1, and Table S1). It was found that three main regions appear in the I-V relations: a low current density region (region 1), where current increases linearly with voltage; a high current density region (region 3) where current increases steeply; and a curvature region (region 2) between the above two regions. The transition region (region 2) was determined by drawing tangents to the decreasing resistance and increasing resistance curves in the plots. Region 2 is between the two cut-off points, and the limiting current obtained corresponds to the point of intersection of these two lines [37].

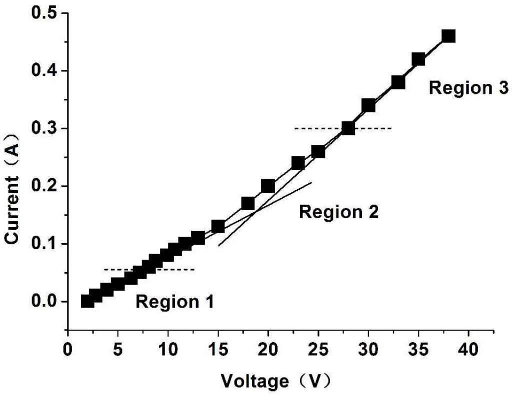

Figure 2. I-V curve in the EDI process

The linear curve reveals that the ion removal mechanism is dominant in region 1 for the reason that the applied current is a driving force for ion transport. In region 3, the curve changes rapidly and the resistance decreases greatly, which indicates that the water dissociation mechanism is dominant. Water dissociation produces large quantities of hydrogen and hydroxide ions that reduce the resistance between the electrodes and accelerate the electro-regeneration process, which indirectly facilitates the ion removal. However, the current efficiency is reduced when water dissociation occurs, which may induce the increase of energy consumption. This result indicates that region 3 should be excluded for the shortage of energy reduction (large exceeding hydrogen generated when higher voltage was applied to overcome the whole cell resistances). In brief, ion removal and water dissociation both exist in region 2, so region 2 is selected as a suitable choice for the working current.

To investigate the effect of current on EDI operation, currents that are limited to region 2 (including 0.05, 0.1, 0.15, 0.2, 0.25 and 0.3 A, respectively) are chosen as samples based on the reasoning above. From Figure 3, it can be seen that the CO_2 recovery rate always increases as a function of current. The carbonate recovery rate is 0.05, 1.0, 2.0, 2.4, 3.0 and 3.5 mmol/h for the respective currents. Correspondently, the recovery efficiency is calculated to be near 90% (insert of Figure 3). The recovery rate increases by 2.0 mmol/h with a current increase from 0.05 A to 0.15 A but then increases by only 1.0 mmol/h with a current increase to 0.25 A. As a whole, the recovery rate increases steeply at low current and increases slowly at high current. The reason for such a trend is as follows: ion migration is driven by electric force and the migration rate of CO_3^{2-} increases with the current, which leads to an increasing carbonate recovery rate. The result is in agreement with the rising removal percentage of carbonate ions. However, high current not only accelerates ion removal but also results in the appearance of water dissociation, which produces a large number of hydroxide ions that have competitive transference with carbonate ions, thus leading to the reduction in the growth rate of the recovery rate.

Current efficiency variation is also obvious in Figure 3; the current efficiency increased to 74.68% and then decreased as a function of current. The efficiency reached its maximum point when the current was 0.15 A. Likewise, power consumption is shown in Figure 3; the system consumes the least energy when the current is 0.15 A. Here, power consumption decreases to 1.41 kwh/kg when the current is 0.15 A. It seems that power consumption is related to current efficiency. This is because power consumption augmentation is caused by heat generation or water dissociation on the surface of the electrode and the resin interval, and the decrease of current efficiency is also related to these factors. Consequently, we conclude that 0.15 A is the optimal current. In this case, the energy reduction and efficiency increase is satisfactory, although the recovery rate is maximal.

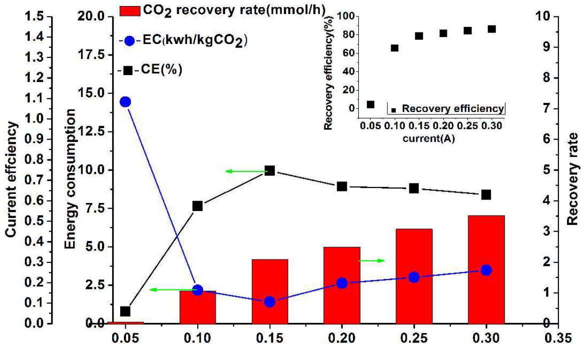

Figure 3. Effect of current on CO_2 recovery rate, current efficiency and energy consumption. Feed solution: 10 mmol/L; electrode solution: 0.5 g/L Na_2SO_4; feed flow rate: 0.6 L/h

3.3 Effect of influent concentration

As shown in Figure 4, the recovery rate is 0.38, 0.64 and 1.12 mmol/h when the feed concentration is 1.0, 2.0 and 4.0 mmol/L, respectively. This relationship is approximately linear. We speculate that this relationship is due to the variability in the mobility of CO_3^{2-} ions within the resin bed, which is in accordance with the theory[38] that as carbonate is replaced by two hydrogen ions, the osmotic pressure between the inside of the particle and the bulk solution is increased, which causes the water content of the resin to increase. The increased water content of the resin results in an elevated mobility of the carbonate ions. Our speculation is successfully demonstrated by a theoretical calculation. Table 2 shows the calculated data for the resin composition. The percentage of $R\text{-}CO_3^{2-}$ is calculated based on the mass balance among the feed, effluent and accumulated ions in the concentrated chamber [39]. $R\text{-}CO_3^{2-}$ amounts decrease with feed concentration, which reflects the replacement of CO_3^{2-} by hydroxide ions, thus leading to an increase in the carbonate recovery rate. Figure 4 also reveals that the CO_2 recovery rate continues to increase when the feed concentration is less than 4.0 mmol/L but appears to decrease after exceeding this point. This phenomenon indicates that the maximum recovery rate is determined by the current; that is to say, the carbonate flux at the 4.0 mmol/L condition is the largest carbonate quantity that can be transported by this current.

The current efficiency in Figure 4 changes consistent with the carbonate recovery rate. This is because the current efficiency is calculated based on the carbonate migration flux as the carbonate was the target anion. Additionally, the recovery rate is dependent on the ion migration flux. Thus the current efficiency changes with the carbonate recovery rate variation.

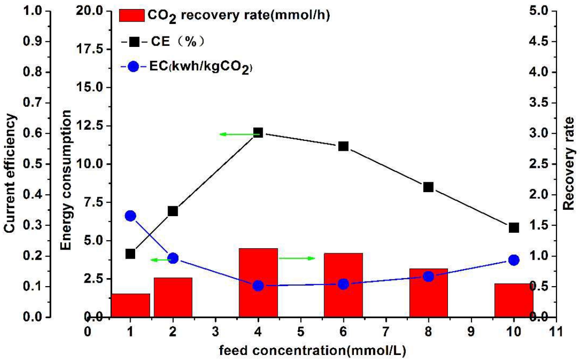

Figure 4. Effect of feed concentration on CO_2 recovery rate, current efficiency and energy consumption. Current: 0.1A; electrode solution: 0.5 g/L Na_2SO_4; feed flow rate: 1.2 L/h

Table 2. Fraction of resin at different feed concentrations

Current（A）	0	0.1	0.1	0.1	0.1	0.1	0.1
Concentration of feed solution		1	2	4	6	8	10
Fraction of R-CO_3^{2-} in resin（%）	100	35.49	29.63	25.82	15.95	9.11	5.00

In Figure 4, the power consumption seems to change with current efficiency when the feed concentration is under 4.0 mmol/L. However, when the feed concentration is above 4.0 mmol/L, the consumption appears to be different from the left part, where power consumption is related to current efficiency. It is odd that power consumption decreases along with the reduction of current efficiency. This relationship is explained by the simplified formula (7), which shows that the power consumption is affected by two factors: current efficiency and voltage. The power consumption variation depends on which factor that plays the leading role in the experiment. The voltage gradually decreases (Figure 5) as feed concentration increases from 1.0 mmol/L to 10.0 mmol/L at constant current density for the following reasons: On the one hand, because R-CO_3^{2-} has a higher resistivity than R-OH^-, the fraction of R-OH^- in the resin increases as the feed concentration increases, which induces a resistance change in the dilute chamber that lowers the voltage accordingly; on the other hand, the conductivity of the interstitial solution in the resin also increases with the influent feed concentration [40]. Therefore, the reduction of voltage and current efficiency both affect the power consumption. To conclude, from the point of view of energy saving, higher feed concentration is advantageous. However, from the point of view of EDI efficiency and recovery rate, a 4.0 mmol/L feed concentration is better.

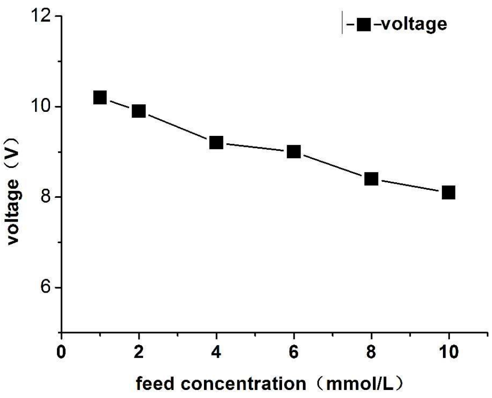

Figure 5. Voltage variation at different feed concentrations

3.4 Effect of flow rate

It can be seen from Figure 6 that the recovery rate and current efficiency show little change in the range of flow rates that we investigated. One possible reason for this lack of change is that the flow rate is lower than the ion migration rate; because the ion removal quantity at a certain current is fixed, increasing the flow rate is useless for increasing carbonate flux. Another possible reason is the elimination or decrease in the boundary layer thickness under high flow rate because concentration polarization at a low flow rate affected the vertical mole diffusion significantly [40].

The power consumption variations are similar to the recovery rates. Considering that the recovery rate, current efficiency and power consumption are the same at different flow rates, the removal percentage makes a difference. Meanwhile, the power requirement of the pump will increase for a high flow rate, which will increase the overall energy consumption. So, among these flow rates, 0.6 L/h is chosen as the rate for the best removal performance and the lowest energy consumption.

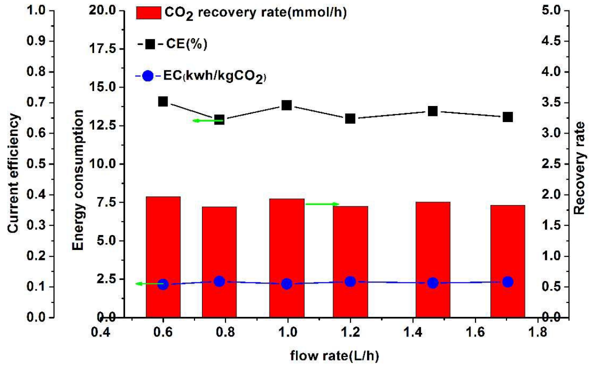

Figure 6. Effect of flow rate on CO_2 recovery rate, current efficiency and energy consumption. Current: 0.15A; electrode solution: 0.5 g/L Na_2SO_4; feed concentration: 10 mmol/L

3.5 The effect of absorption ratio

Here, the experiment was carried out on simulation wastewater (feed solution) by adding NaOH, and the effect on the absorption ratio is discussed. The absorption ratio varies from 100% to 9% with carbonate ions fixed at 10 mmol/L.

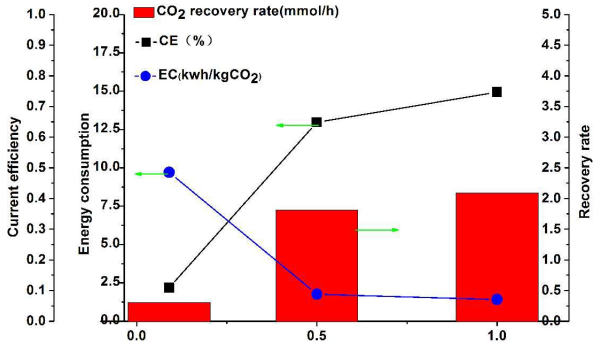

Figure 7. Effect of absorption ratio on CO_2 recovery rate, current efficiency and energy consumption. Current: 0.15A; electrode solution: 0.5 g/L Na_2SO_4; feed concentration: 10 mmol/L; feed flow rate: 0.6 L/h

When the absorption ratio is 100%, the maximum recovery rate can be reached. Decreasing the absorption is disadvantageous to CO_2 recovery. Such a trend is understandable for the following reason: the absorption ratio is adjusted by adding drops of NaOH solution; the competitive transference of OH^- through the membrane makes the carbonate flux decrease. Therefore, recovery performance is better at a neutral condition; thus, a pH adjustment is needed when using simulated wastewater. When there are no other anions that transport faster than carbonate ions, the EDI process is considered ideal for carbon dioxide recovery performance and energy reduction can be achieved.

Practically, the feed solution in the dilute chamber can be fully regenerated to its original alkaline condition (Figure 1), and this can be reused or recycled as an absorption solution, similar to the amine processes. Enlargement of the EDI device is feasible and the flow rate of the feed solution is flexible, the calculation of the EDI scaling up is shown in supporting Information.

4 Conclusion

The bench-scale EDI unit developed in this paper was demonstrated to be able to separate CO_2 gas from an alkaline solution. Current efficiency and power consumption are dependent upon the recovery rate. The operation can be optimized near the limiting current due to increase of the current applied to the unit is beneficial to CO_2 recovery but energy consumption per equivalent of carbonate removed will consequently increase. Flow rate has insignificant effects on CO_2 recovery within the flow rate range investigated. The CO_2 recovery rate increased with feed concentration and appeared to reach a limit at high concentration. A high absorption ratio is advantageous to CO_2 recovery, indicating that hydroxide ions have competitive transportation compared with carbonate ions.

Our method has a high current efficiency (near 80%), which shows that the addition of a resin to the feed compartment significantly reduces internal resistance and therefore increases effectiveness. In addition, the minimum power consumption for stripping carbon dioxide from the alkaline carbonate solution can be lessened to 1.41 MJ/kgCO$_2$, which provides a substitute method for an easy CCS process. It is believed that operational costs can be further reduced by employing multiple cell pairs working together. Further, the resin chamber (alkali-production chamber) can be employed to absorb carbon dioxide from flue gas or the atmosphere and make a full recycling process.

Acknowledgements

This work is financially supported by the National Natural Science Foundation of China (Nos. 21173188, 21473158) and partially 863 Program (No. 2013AA065900).

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Graphical abstract:

Under electrical force the migration rate of carbonate ions in low residence resin chamber was accelerated and CO_2 gas was released through acidic chamber.