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# Ionic Liquid (1-Butyl-3-Methylimidazolium Methane Sulphonate) Corrosion and Energy Analysis for High Pressure CO<sub>2</sub> Absorption Process

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**Abstract:** This study explores the possible use of ionic liquids as a solvent in a commercial high-pressure CO<sub>2</sub> removal process, to gain environmental and energy benefits. There are two main constraints in realizing this: ionic liquids can be corrosive, specifically when mixed with a water/amine solution with dissolved O<sub>2</sub> & CO<sub>2</sub>; and CO<sub>2</sub> absorption within this process is not very well understood. Therefore, scavenging CO<sub>2</sub> to ppm levels from process gas comes with several risks. We used 1-butyl-3-methylimidazolium methane sulphonate [bmim][MS] as an ionic liquid because of its high corrosiveness (due to its acidic nature) to estimate the ranges of expected corrosion in the process. TAFEL technique was used to determine these rates. Further, the process was simulated based on the conventional absorption–desorption process using ASPEN HYSYS v 8.6. After preliminary model validation with the amine solution, [bmim][MS] was modeled based on the properties found in the literature. The energy comparison was then provided and the optimum ratio of the ionic liquid/amine solution was calculated.

**Keywords:** CO<sub>2</sub> absorption; ionic liquids; amines process simulation

## 1. Introduction

Anthropogenic carbon dioxide (CO<sub>2</sub>) removal systems are used in various chemical industries and in various processes or to gain carbon credits [1]. In many processes, its absorption from synthesis gas is a crucial parameter in order for downstream catalysts to work (usually allowing slippage <500 ppm) [2]. Major design constraints include the loading rate, CO<sub>2</sub> slippage limit and energy extensiveness. Absorption by solvents is a common practice employed in various industries [3].

Although there are many techniques reported in literature to capture CO<sub>2</sub> [4–6] until now only chemical solvent-based processes promise to achieve low slippage at high loading rates commercially. Many amines were studied for their CO<sub>2</sub> absorption capacity, but amines like methyl ethanolamine (MEA), diethanolamine (DEA), methyl diethanolamine (MDEA), piperazine (PZ), triethanolamine (TEA), and 2-amino-2-methyl-1-propanol (AMP) show better results in terms of stability and efficacy [7]. Commercial solvents are made up of concoctions of various chemicals that include optimum combinations of amines, corrosion inhibitors, antifoaming agents, and other proprietary additives. These amines mixtures have been commercialized under different names [8–13]. CO<sub>2</sub> is captured by reversible chemical reactions within these amines. The rate of reaction depends on the bulk concentration of CO<sub>2</sub>. A simple solvent-based CO<sub>2</sub> absorption scheme consists of a stripper and an absorber at low and high pressure respectively; however, this kind of scheme is extensive in terms of energy/cost [14]. Regeneration of these solvents requires steam and is thus a major energy consuming unit operation. Current literature focuses on two techniques in response to this problem: discovery of

a solvent that requires less decomposition energy to liberate CO<sub>2</sub> [15,16], or restructuring the process to economize pressure and heat energy recovery [17–22]. Therefore, development of new solvents requiring less drastic conditions without compromising CO<sub>2</sub> slippage remains an attractive area of research [23].

Pure ionic liquids are also reported to absorb CO<sub>2</sub> [24,25]. Furthermore, amine groups are functionalized with ionic liquids to synergize the effects of reaction and absorption in the same molecule [26,27]. This is reported to enhance the CO<sub>2</sub> recovery [28]. Ionic liquids also offer the advantage of low vapor pressure, decreasing unwanted loss in the stripped stream at high temperatures. However, ionic liquids have process issues related to corrosion and crystallization.

Corrosion of metals in amine solutions is well known [22,29]. The commercial amine mixture solvents usually have some corrosion inhibitor or passivators to deal with the detrition of metallic parts [30]. The corrosion by amine/ionic liquid mixtures is foreseen [31]. Ionic liquids are salts and can extremely affect the corrosion of various metals, as their hydrolysis can be significantly different [32]. In this study, 1-butyl-3-methylimidazolium methane sulphonate [bmim][MS] was chosen as a model molecule in the design of an ionic liquid-based process. At 60 °C, absorption of 0.25 mol CO<sub>2</sub>/mol IL at 5 MPa is reported [33]. The addition of water to the solution has mixing and transfer benefits that can result in energy efficiency due to decreases in viscosity. The surface tension of [bmim][MS] decreased when mixed with water, thus increasing the mass transfer coefficient [34].

## 2. Materials and Methods

1-Butyl-3-methylimidazolium methanesulfonate [bmim][MS] was provided by Ionic Liquid Technologies, Helibronn, DE (io.li.tec) with a purity of 99.6–99.9% and 98.5–99.9% respectively. Diethylethanol amine and piperazine were purchased from Sigma Aldrich, Al-Khobar, KSA. Three-electrode system was used to carry out electrochemical tests on a PG STAT-101 potentiostat, provided by AUTOLAB and powered by NOVA software. Ag/AgCl was used as a standard electrode with a platinum electrode as the counter electrode. The sample coupon of carbon steel was grinded and polished. The sample coupon was also washed with distilled water and ethanol before testing. Temperature was controlled manually by oil circulation in the cell jacket. All experiments were done at atmospheric pressure. Pure carbon dioxide was bubbled through the solution continuously ( $\approx 1$  mL/min). Electrodes were immersed and left for 30 min to achieve a steady state before any measurement. Open circuit potential was first measured and  $\pm 100$  mv range to open circuit potential is adjusted for Tafel scan. Scan rate was fixed at 1 mv/s. Each experiment was repeated thrice. Uncertainty in the results was less than  $\pm 7\%$ .

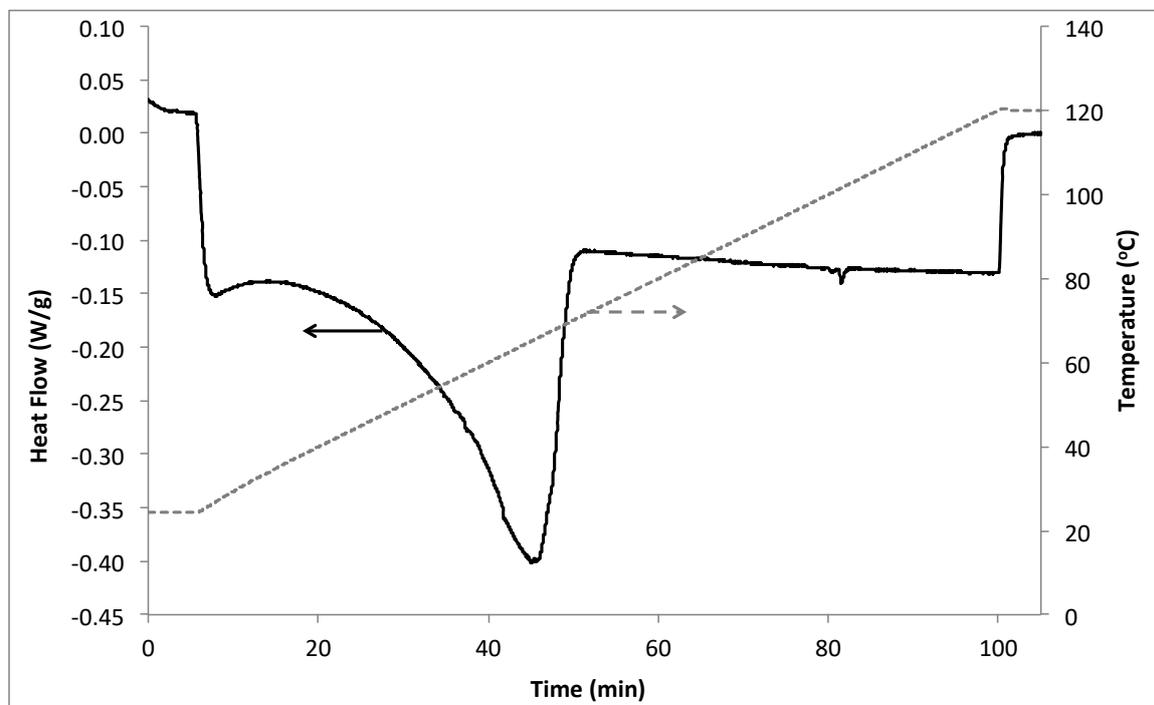
Differential Scanning Calorimeter was provided by Netzsch, Selb, DE (DSC 200 F3). The samples (about 10–15 mg) were initially heated to 100 °C to erase the thermal history, then cooled to room temperature, and finally heated at 1 °C per min from  $\approx 25$  °C to 120 °C. Aluminum crucible with pierced lid was used. The obtained thermograms were used to determine the glass transition/melting temperature of the ionic liquids.

## 3. Results & Discussion

### 3.1. Differential Scanning Calorimetry

The differential scanning calorimetry (DSC) results are shown in Figure 1. As [bmim][MS] is a strong hydrophilic ionic liquid and can absorb moisture from the environment, care was taken to keep contact between the ionic liquid and the environment to a minimum, to avoid water ingress after sample preheating. However, it cannot be completely avoided. The valley started immediately after heating began. This was attributed to the vaporization of the absorbed water. The exact melting point for the ionic liquid is hard to measure but [bmim][MS] must be kept above 65 °C to avoid crystallization in the CO<sub>2</sub> absorption process. The heat capacity data (above 65 °C) obtained from the DSC was also used to model the process, as described in the next section. No decomposition

of the ionic liquid was physically observed below 100 °C. Moreover, after cooling of DSC samples there was no/negligible weight loss. Kärkkäinen discussed the stability of the ionic liquids and reported [bmim][MS] decomposition onset temperature as 335 °C. Therefore, it is assumed that no decomposition of the ionic liquid occurs at the process conditions described in the simulation section [35].



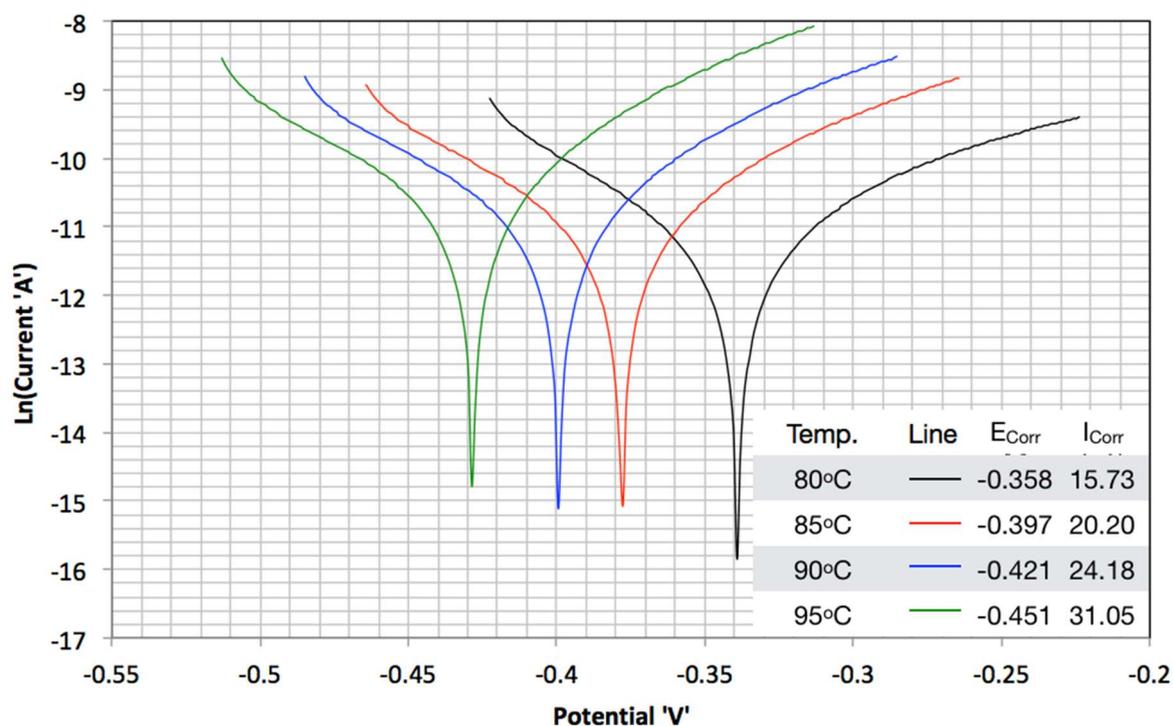
**Figure 1.** Differential scanning calorimetry (DSC) results for the 1-butyl-3-methylimidazolium methane sulphonate ([bmim][MS]), scan rate is 1 °C/min. Gray dotted line—Temperature of the sample (°C), Black continuous line—Heat Flow (W/g).

### 3.2. TAFEL Curves

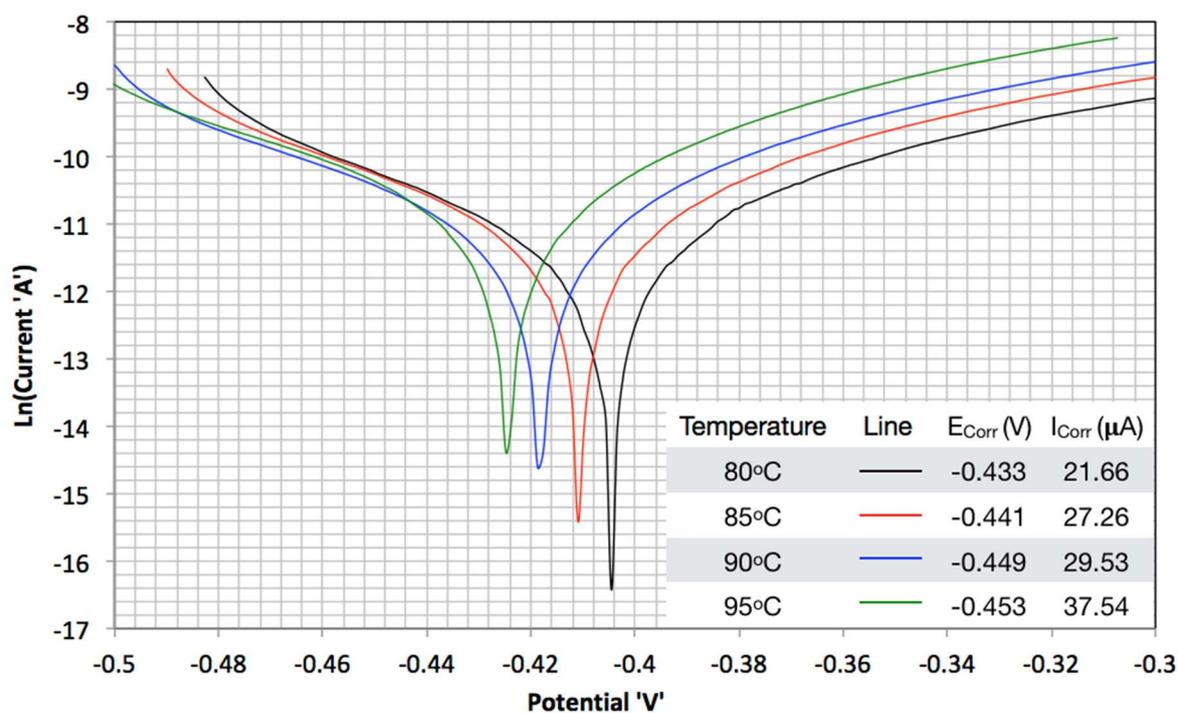
Figures 2 and 3 present the Tafel curves obtained without and with carbon dioxide for pure [bmim][MS] respectively. Corrosion current is reported per unit area ( $\text{cm}^2$ ) of the exposed surface. Corrosion rates are obtained by the standard procedure of Tafel slopes. The rate of corrosion increased with temperature because of the enhanced mobility of the ions and charge transfer. It is also shown that carbon dioxide absorption in water lowers the pH of water, thus increasing the oxidation rates. The same was observed for [bmim][MS]. As there is no water present the increase in corrosion rates is attributed to the enhanced charge mobility by [bmim][MS].  $\text{CO}_2$  was physically absorbed on the ionic liquid, structural modification of imidazole cation is shown by the published data of Jung et al. [33]. Increase in  $\text{CO}_2$  absorption by longer chain cations than [bmim] further strengthens the view that the cation is a major source of  $\text{CO}_2$  absorption. The slopes of the Tafel curves suggest that cation charge transfer controls the oxidation rates. This is true both with and without  $\text{CO}_2$ . Results suggest that the  $\text{CO}_2$  absorption on cation enhances its mobility in the solution.

Corrosion rates drop sharply when amine solution (40%MDEA, 2%PZ, 58% $\text{H}_2\text{O}$ ) is mixed with [bmim][MS], as presented by Figures 4 and 5. Amine saturated with  $\text{CO}_2$  has corrosion current rates for carbon steel in the range of 3 to 4  $\mu\text{A}$  [28]. The perturbation in Figure 5 can be attributed to noise. As the solution becomes more conductive, more noise is expected due to increase in the sensitivity levels. Amine introduces a strong inhibition effect on corrosion for ionic liquids. However, this is not true if the concentration of the amine solution is increased beyond a certain threshold. This can be

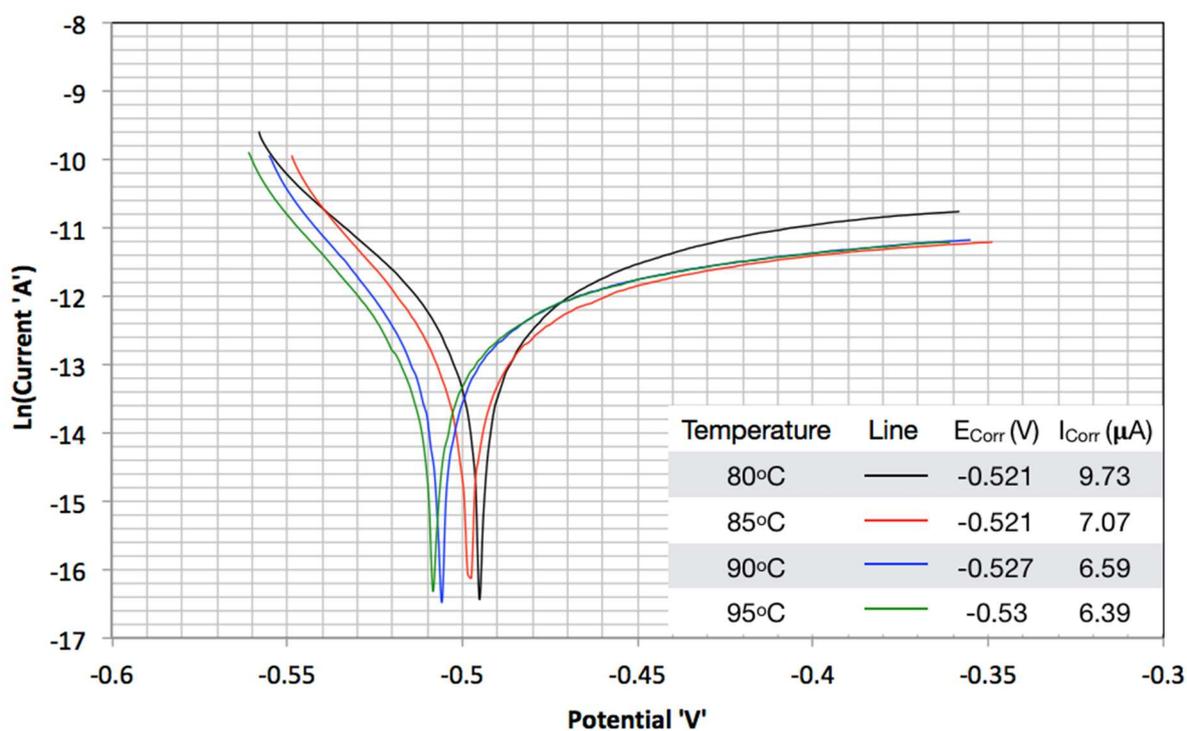
observed from the results presented in Figures 6 and 7. For any commercial process, such corrosion rates are usually not acceptable. Furthermore, the corrosion rates drop with increase in temperature.



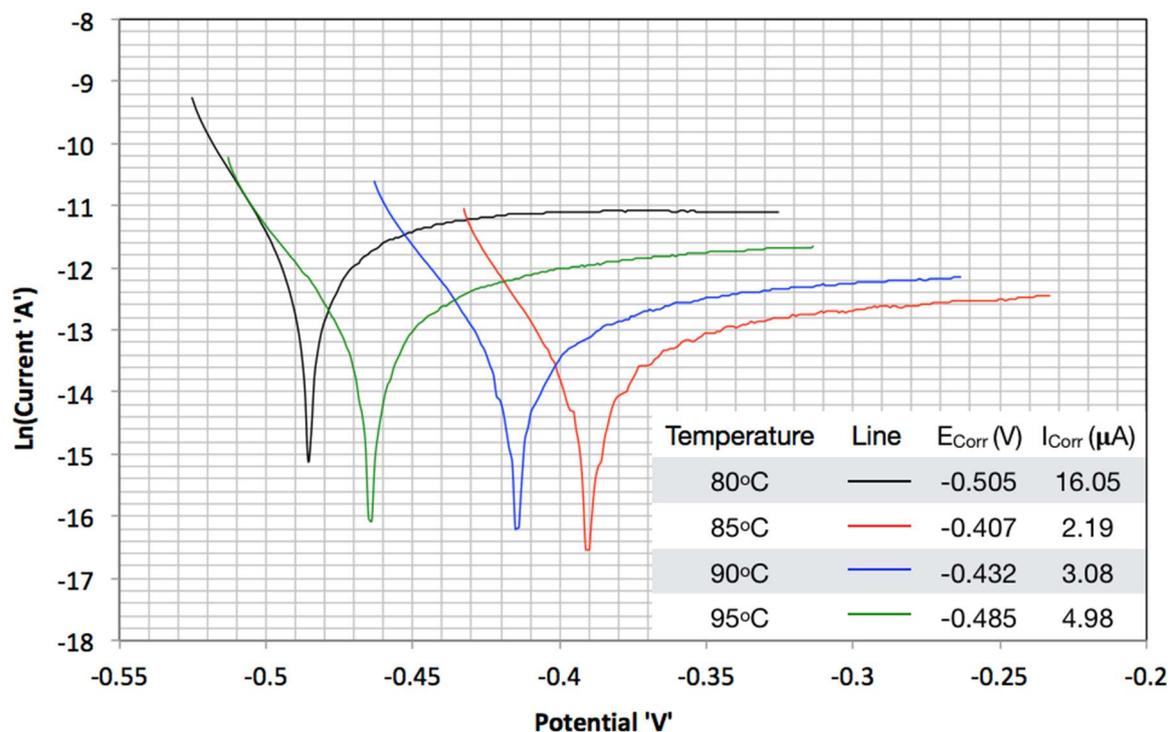
**Figure 2.** Tafel curves of pure [bmim][MS] without CO<sub>2</sub> at different temperatures. Scan rate = 1 mV/s. Fitted results for corrosion rates and potential are provided in the table.



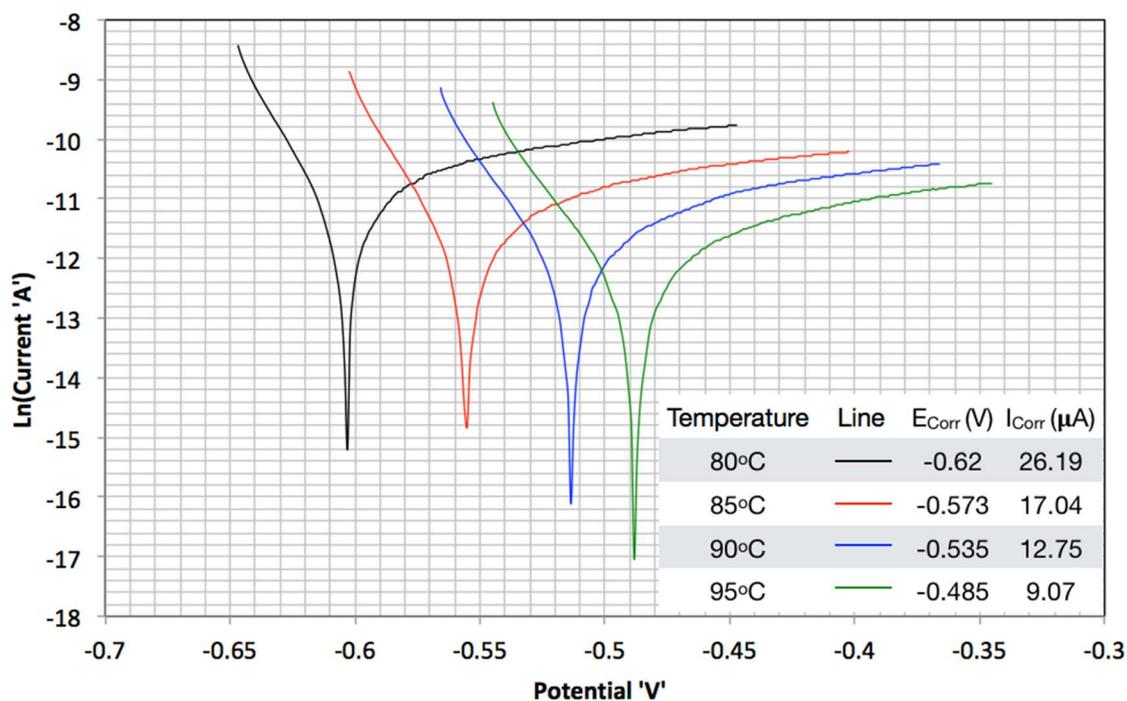
**Figure 3.** Tafel curves of pure [bmim][MS] with CO<sub>2</sub> at different temperatures. Scan rate = 1 mV/s. Fitted results for corrosion rates and potential are provided in the table.



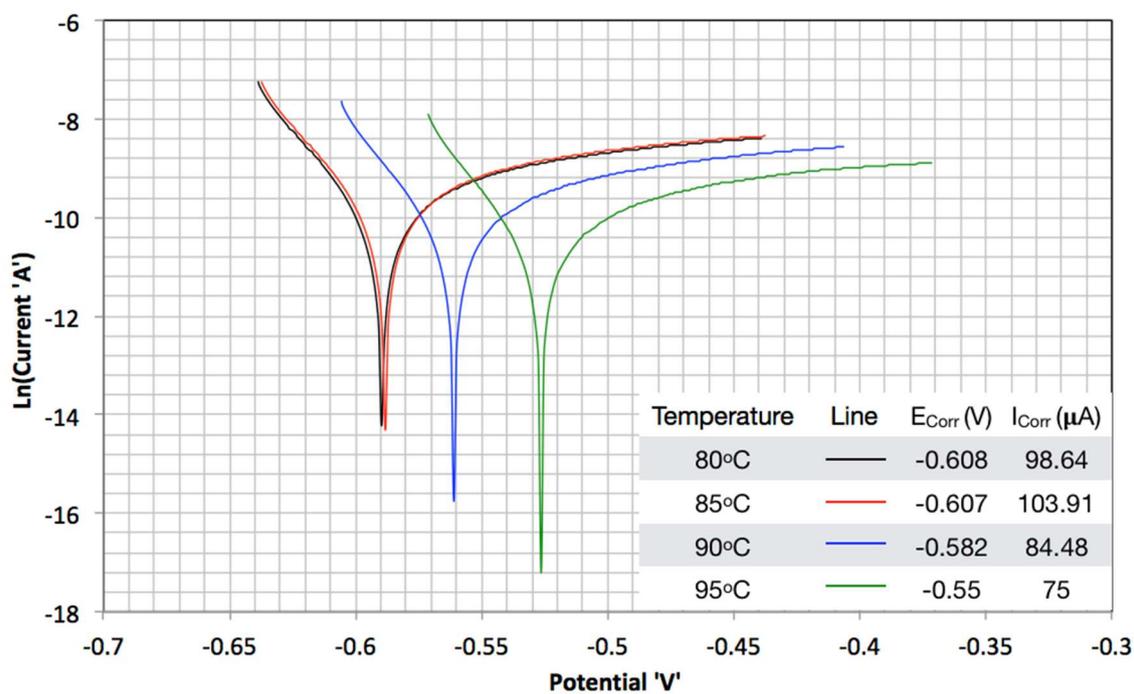
**Figure 4.** Tafel curves of [bmim][MS] and 10% methyl diethanolamine (MDEA) without CO<sub>2</sub> at different temperatures. Scan rate = 1 mV/s. Fitted results for corrosion rates and potential are provided in the table.



**Figure 5.** Tafel curves of [bmim][MS] and 10% MDEA with CO<sub>2</sub> at different temperatures. Scan rate = 1 mV/s. Fitted results for corrosion rates and potential are provided in the table.



**Figure 6.** Tafel curves of [bmim][MS] and 20% MDEA without CO<sub>2</sub> at different temperatures. Scan rate = 1 mV/s. Fitted results for corrosion rates and potential are provided in the table.



**Figure 7.** Tafel curves of [bmim][MS] and 20% MDEA with CO<sub>2</sub> at different temperatures. Scan rate = 1 mV/s. Fitted results for corrosion rates and potential are provided in the table.

### 3.3. Process Simulation and Validation

#### 3.3.1. Existing Facility Model

Zwitterion mechanism is considered responsible for CO<sub>2</sub> absorption by amines [36,37]. The reactions are provided in Table 1. The first five reactions are considered to be in quasi-state equilibrium. The rest of the reactions are used to model the CO<sub>2</sub> absorption based on the mass transfer coefficient and kinetics. These reactions along with the Electrolyte Non-Random Two Liquids (ENRTL) method form 'ACID GAS' package (specifically designed for amine-based solvents) for thermodynamic calculations using ASPEN HYSYS V 8.6 (Aspen Technology, Inc., Bedford, MA, USA). A similar technique was used by Greer et al. to simulate the CO<sub>2</sub> absorption process (solvent MEA) but using the Peng–Robinson model. Furthermore, absorption has been modeled using chemical reaction and Henry's law [38].

**Table 1.** Reactions involved in CO<sub>2</sub> absorption by methyl diethanolamine (MDEA)/piperazine (PZ) mixture.

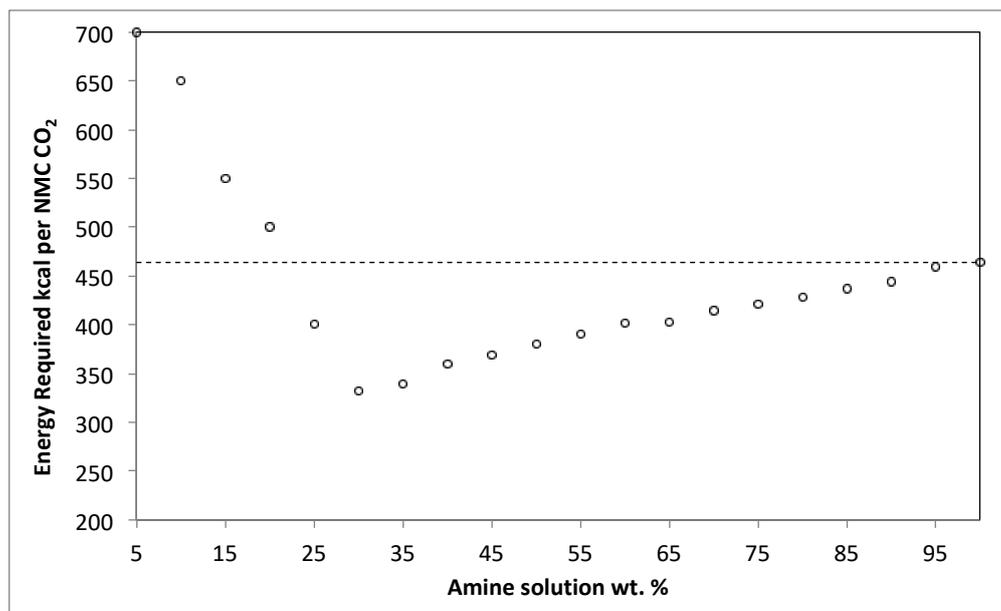
No	Reactions	Forward Reaction	Backward Reaction
1	MDEAH <sup>+</sup> + H <sub>2</sub> O ⇌ H <sub>3</sub> O <sup>+</sup> + MDEA		Quasi-state equilibrium
2	2H <sub>2</sub> O ⇌ H <sub>3</sub> O <sup>+</sup> + OH <sup>-</sup>		Quasi-state equilibrium
3	H <sub>2</sub> O + HCO <sub>3</sub> <sup>-</sup> ⇌ H <sub>3</sub> O <sup>+</sup> + CO <sub>3</sub> <sup>2-</sup>		Quasi-state equilibrium
4	PZ + H <sub>3</sub> O <sup>+</sup> ⇌ +H <sub>2</sub> O + PZH <sup>+</sup>		Quasi-state equilibrium
5	HPZCOO + H <sub>2</sub> O ⇌ H <sub>3</sub> O <sup>+</sup> + PZCOO <sup>-</sup>		Quasi-state equilibrium
4	CO <sub>2</sub> + OH <sup>-</sup> ⇌ HCO <sub>3</sub> <sup>-</sup>	$1.33 \times 10^{17} \exp\left(\frac{-13258}{RT}\right) [\text{CO}_2][\text{OH}^-]$	$6.63 \times 10^{16} \exp\left(\frac{-25674}{RT}\right) [\text{HCO}_3^-]$
5	MDEA + H <sub>2</sub> O + CO <sub>2</sub> ⇌ HCO <sub>3</sub> <sup>-</sup> + MDEAH <sup>+</sup>	$6.85 \times 10^{10} \exp\left(\frac{-9035.1}{RT}\right) [\text{MDEA}][\text{CO}_2]$	$6.62 \times 10^{17} \exp\left(\frac{-22146}{RT}\right) [\text{HCO}_3^-][\text{MDEAH}^+]$
6	PZ + H <sub>2</sub> O + CO <sub>2</sub> ⇌ H <sub>3</sub> O <sup>+</sup> + PZCOO <sup>-</sup>	$1.75 \times 10^{10} \exp\left(\frac{-319.21}{RT}\right) [\text{PZ}][\text{CO}_2]$	$3.4 \times 10^{23} \exp\left(\frac{-14169}{RT}\right) [\text{H}_3\text{O}^+][\text{PZCOO}^-]$
7	PZCOO <sup>-</sup> + H <sub>2</sub> O + CO <sub>2</sub> ⇌ H <sub>3</sub> O <sup>+</sup> + PZCOO <sup>2-</sup>	$1.04 \times 10^{14} \exp\left(\frac{-8043.3}{RT}\right) [\text{PZCOO}^-][\text{CO}_2]$	$3.2 \times 10^{20} \exp\left(\frac{-8697.9}{RT}\right) [\text{H}_3\text{O}^+][\text{PZCOO}^{2-}]$

Figure 8 shows the simple process flow diagram. Process gas is produced by reforming and is available at high temperature and pressure (78 °C, 33 bar). The gas contains 59% H<sub>2</sub>, 18% CO<sub>2</sub>, 20% N<sub>2</sub>, and the rest is water, methane and traces of H<sub>2</sub>S (mol basis). The process is designed to economize the process for stripping the rich amine solution (1–2). After cooling, this gas is passed through the absorber and the clean process gas is available (3–4). The downstream processes are designed based on the maximum CO<sub>2</sub> contents of 500 ppm. The absorber is divided into two sections. The upper section receives the lean amine solution from the stripper and works on approach to equilibrium (20–22). The lower section is at high temperature and receives comparatively hot semilean solution from intermediate flashing scheme (12–14). The lower absorber section thus enhances the CO<sub>2</sub> absorption kinetics, which is slower in the upper section. The high-pressure rich amine solution is then depressurized in a turbine to feed the high-pressure flash drum. This flash drum pressure is optimized to recover hydrogen and nitrogen that is absorbed along with CO<sub>2</sub>. As flash gas contains high contents of acid gas, it is therefore recycled back to the absorber (5–7). The amine solution then goes to the low-pressure flash drum. The pressure in this flash drum is equal to the stripper. The purpose of this flash drum is to split the amine flow to feed both the stripper and the lower part of the absorber (13–16). The acid gas is also recovered from this flash drum (10). Downstream stripper is used to produce the lean solution, almost free of acid gas, to feed the absorber upper section (17–22).



the existing process, but without mixing with amine solution (solution of amine and ionic liquid is assumed as an ideal mixture i.e., mixture properties are just the summation of pure species property with entropy adjustment with no chemical reaction between amine and [bmim][MS]). Task-specific ionic liquids (TSIL) are tailored—usually cation functionalized ionic liquids enhance CO<sub>2</sub> absorption. The imidazolium-based cation is functionalized and it is reported to increase CO<sub>2</sub> absorption by threefold for ionic liquids [40,41]. The functionalization mechanism usually involves specific radicals and ion exchangers. Furthermore, the imidazolium cation functionalization, with CO<sub>2</sub> absorption, increases the solution viscosity to a gel-like substance [42]. At the process conditions, it is highly unlikely to have a functionalization reaction between amine and [bmim][MS], as the exponential increase in viscosity was not observed in our corrosion experimentation. Any side reaction kinetics is assumed to be negligible due to the long chain diffusion limitations. Functionalization can increase the absorption of CO<sub>2</sub> but gravimetrically due to lower CO<sub>2</sub> loadings it is not feasible to use these TSIL alone in commercial processes [43].

Vapor pressure of the ionic liquid is taken as negligible. In the case of an ionic liquid mixed with the amine, the temperature of the process is kept above 65 °C. First, the carbon dioxide is absorbed in the ionic liquid according to the equilibrium relation obtained from the regression. Then the resulting gas is contacted with the amine solution. No dissolution of any other gas is considered in the process modeling of the ionic liquid, so a high-pressure flash drum is not required with ionic liquids. If only ionic liquid, without amine, is considered then in practice it is not possible to lower the amount of CO<sub>2</sub> in purified gas to 500 ppm. The pure ionic liquid has a tendency of crystallize. Furthermore, its high viscosity may result in extensive pressure drops. Therefore, amine/water solution is required to polish the gas by scavenging remaining carbon dioxide to acceptable levels, to avoid [bmim]z crystallization and to lower its viscosity. Viscosity is reported to decrease with the addition of water and is comparable with amine solution (30 wt. % ionic liquid, 20 °C, 0.1 MPa, amine viscosity  $\approx$  4 mPa·s, [bmim][BF<sub>4</sub>]  $\approx$  2 mPa·s) [44,45]. The ratio between amine solution and ionic liquid is fixed and their total quantities are adjusted to achieve the purity level of 500 ppm. Figure 9 shows the relation between the amount of energy required per NMC of the carbon dioxide removal and the percentage of amine solution mixed with the modeled [bmim][MS]. There exists an optimum mixture of  $\approx$ 27% with the minimum amount of energy required.



**Figure 9.** Amount of energy required per NMC of CO<sub>2</sub> removal. Dotted line presents the conventional process working with amine solution. The circles present the data directly obtained by simulation.

#### 4. Conclusions

The current study provides initial estimates for using ionic liquid [bmim][MS] for carbon dioxide removal. The corrosion rates of amines are considerably less than the ionic liquids, but the combined effect of both the solvents has a weight percent threshold limit. Extreme corrosion rates are expected and advanced corrosion mitigation techniques must be considered for the process design. Usually commercial solvent recipes for CO<sub>2</sub> removal come with corrosion inhibitors. The role of these inhibitors should be enhanced for any ionic liquid inclusion. The ASPEN HYSYS 'ACID GAS' package provides an excellent estimation of the amine properties and corresponds very well with the industrial data. Modeling of ionic liquids is not very mature and requires certain assumptions, because of the lack of available data. The built-in 'COSMOSAC' model (in ASPEN) does not provide good estimates in the case of [bmim][MS]. It is therefore modeled using primitive techniques, thus slight discrepancies in the results are expected that can be addressed with more experimental data input. Nevertheless, it can be concluded with sufficient evidence that an optimal mixing ratio exists, when using ionic liquid for high-pressure commercial CO<sub>2</sub> removal process under different conditions.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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