



# Carbon Dioxide Absorption By 1-Butyl-3-Methyl-Imidazolium Tetrafluoroborate [Bmim][BF<sub>4</sub>]

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## Abstract

The main objective of this thesis is to study the reduction of carbon dioxide emissions into the atmosphere as it leads to higher temperatures, increased pollution, health problems, smog and acid rain. In this work, the ionic liquid, such as 1-butyl-3-methyl-imidazolium tetrafluoroborate [Bmim][BF<sub>4</sub>] was used at a concentration of (0-3) mol/L and a temperature of (25-40) degrees Celsius. The ionic liquid is mixed with alkanolamines such as (MEA) (4 mol/L) and water to increase the absorption efficiency. 10 ml of aqueous solution (IL + MEA + j, absorber cell (50ml), water bath and CO<sub>2</sub> analyzer. The central composite design (CCD) method was applied to reduce the number of experiments needed to improve operating conditions. In absorption experiments, the effect of temperature of the solution (25-40)°C, the concentration of the solvent (0-3) mole/l and absorption time (60-120 min) were investigated to determine the optimum operating conditions on the performance of absorption process. In addition, the absorption rate of CO<sub>2</sub> were calculated. The experimental data have shown that The best conditions for obtaining the highest absorption rate were at a temperature of 40°C, a concentration of 1.5 mol/L and an absorption time of 90 min, with an absorption rate of approximately 0.077 mol/kg/min and absorption efficiency for solution ([Bmim][BF<sub>4</sub>], MEA and water) 88%.

## 1. Introduction

Pollution has increased over the previous century as a result of industrialization and population growth. Apart from contamination of water and soil, which has a negative effect on the ecosystem, air pollution is the most important concern in the environmental debate [1]. Carbon dioxide (CO<sub>2</sub>) is one of the most prevalent waste gases emitted by industries, industrial plants, and vehicles [2]. The most serious issue with carbon dioxide is the greenhouse effect, which causes rising temperatures. More than 60% of global warming is attributed to carbon dioxide [3]. CO<sub>2</sub> concentrations are presently approximately 400 parts per million, according to scientific estimations, a significant increase from pre-industrial values of fewer than 300 parts per million [4]. The Intergovernmental Panel on Climate Change (IPCC) predicts a 1.9°C increase in global temperature by 2100, implying that lowering CO<sub>2</sub> levels is critical for humans and all other life on the planet [5]. Health issues, urban smog, and acid rain are just a few of the major consequences of CO<sub>2</sub> pollution. For lowering total CO<sub>2</sub> in the atmosphere, several options are offered. The first option is to replace fossil fuels with non-carbon energy sources like wind, solar, and biomass, which necessitates a switch to non-fossil fuels (e.g. renewable energy and hydrogen). The second alternative is to improve energy efficiency in order to reduce greenhouse gas emissions per unit of energy consumed, which requires efficient energy use. The third alternative (CCS) [6] is carbon sequestration and storage.

Ionic liquids (ILs), for example, have been proposed as promising CO<sub>2</sub> capture possibilities. In 1914, Paul Walden created the first IL (ethyl ammonium nitrate), although he had no idea that over a century later, ILs would become a major scientific topic [7]. In actuality, ILs as creative fluids have only recently gained a lot of attention. The number of SCI articles published on ILs has increased at an exponential rate, from a few in 1996 to over 5000 in 2016, considerably surpassing the annual growth rates of other important research topics. This suggests that an increasing number of scholars are working on this fascinating topic, resulting in a multitude of findings. A multidisciplinary study encompassing chemistry, materials science, chemical engineering, and environmental science is being conducted on ILs. Some essential fundamental viewpoints have begun to vary from the initial principles as more knowledge regarding the core of ILs becomes available [8].

Ionic liquids (ILs) at room temperature have lately been suggested as a possible CO<sub>2</sub> capture candidate. The effects of SO<sub>2</sub> and O<sub>2</sub> on CO<sub>2</sub> capture were investigated using an aqueous amine solution mixed with IL in an absorption-desorption loop system in this work. The feed gas was chosen as a gas mixture containing CO<sub>2</sub>, O<sub>2</sub>, SO<sub>2</sub>, and N<sub>2</sub> in the composition range of flue gas from a coal-fired power plant following flue gas desulfurization. By reducing the saturated vapour pressure of the mixed absorbent, it was discovered that adding hydrophilic IL of 1-butyl-3-methylimidazolium tetrafluoroborate ([Bmim][BF<sub>4</sub>]) to a monoethanolamine (MEA) aqueous solution reduced MEA and water losses. Because carbonate, which was produced by MEA reacting with CO<sub>2</sub>, was insoluble in [hmim][Tf<sub>2</sub>N] at the absorber working temperature of 323 K, the MEA loss for 30 wt% MEA + 70 wt% [hmim][Tf<sub>2</sub>N] increased considerably with the system running [9].

### 1.2 Carbon Dioxide Capture Technologies

Many technologies have been developed to absorb CO<sub>2</sub> and reduce its emissions into the environment. The most important technologies used are cryogenic process, adsorption technique, membrane technique and absorption method [10].

#### 1.2.1 Absorption Technology

For more than a few decades, absorption technology has been the commercial technique for CO<sub>2</sub> capture. CO<sub>2</sub> absorption can take place either physically or chemically.

There is no reaction between the solvent and the absorbed solute in the physical absorption process (CO<sub>2</sub>). The Henry's law governs the transfer of CO<sub>2</sub> from gas to solvent, therefore absorption is influenced by pressure and temperature. When the CO<sub>2</sub> partial pressure is high and the temperature is low, the solubility of CO<sub>2</sub> in the solvent

increases. The output solution is then forced through a succession of flash towers at various pressures. CO<sub>2</sub> is released as a result of the flash process [11]. Many solvents are utilized in this process, including union carbide selexol (selexol process), methanol (rectisol process), and propylene carbonate (propylene carbonate process) (flour process). Selexol process is used union carbide selexol (dimethylether polyethylene) as solvent since 1969 to sweetening the natural gas in order to absorb carbon dioxide and hydrogen sulfide. Rectisol process is widely used in natural gas plants to remove carbon dioxide and it is used the methanol as a solvent because of its high vapor pressure, does not form the spume, does not erosion problems and degradation of the solvent. Flour process is used to CO<sub>2</sub> absorption at higher partial pressure > 4.137 bar and it used propylene carbonate as solvent. In general, the physical absorption process has several disadvantages are more low-cost at high pressure, need for cooling of the lean seloxe solution [12].

### 1.3 Aims of the Work

The primary objectives of this study are listed below:

- Investigate the behavior of ionic liquids and their effect on CO<sub>2</sub> capturing from synthetic gas mixture.
- Quantify the effect of variables such as solvent temperature, solvent concentration and absorption time on the capture of carbon dioxide during the absorption process.
- Identify the best operating conditions for CO<sub>2</sub> capturing.

## 4. Materials and Methods

### 4.1 Chemicals

The ionic liquid 1-butyl 3-methylimidazolium tetrafluoro[Bmim] [BF<sub>4</sub>] was imported from China. The basic properties of [Bmim] [BF<sub>4</sub>] are summarized in Table 1 [13]. Monoethanol and water were mixed with the ionic liquid to increase the absorption efficiency.

**Table 1:** Basic characteristics of [Bmim][BF<sub>4</sub>]

| Property          | Detailis  |
|-------------------|---|
| Short name        | [bmim][BF <sub>4</sub> ]                                      |
| Molecular Formula | C <sub>8</sub> H <sub>15</sub> BF <sub>4</sub> N <sub>2</sub> |
| Molecular Weight  | 226.02  |
| Purity            | ≥98%  |
| Melting Point     | -71.0° C  |
| Poling Point      | 100° C  |
| Flash Point       | 288° C not applicable   |
| Density           | 1.21 g/cm <sup>3</sup> at 20° C                               |
| pH Value          | 5 (H <sub>2</sub> O 20° C )                                   |
| Vapor Pressure    | <9.375×10 <sup>-5</sup> mmHg                                  |
| Water             | ≤ 1.0 %   |
| Storage           | below +30°C   |

## 4.2 Equipments

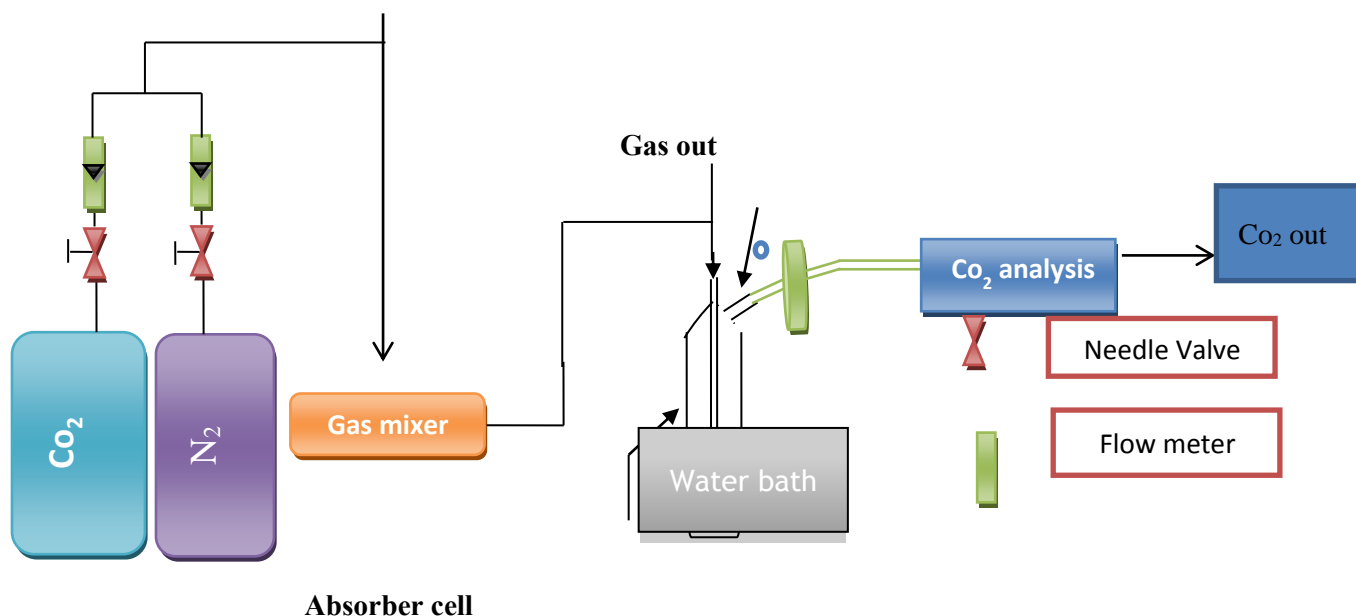
The equipment used in this work are listed in Table 2.

**TABLE 2:** List of tools and devices used in this work.

| No. | Equipment  | Source   |
|-----|--|--|
| 1   | Bottles CO <sub>2</sub> & N <sub>2</sub>   | Biladi plant for gases/Iraq                          |
| 2   | Pressure gauge (0-50)bar   | Visto/ German  |
| 3   | Needle Valve   | Visto/ German  |
| 4   | Flow meters/Air(0-150)ml/min   | Flutech / China                                      |
| 5   | Water bath   | England  |
| 6   | Absorber cell (50ml) (It is a glass cell that contains two holes, one for gas input and one for gas removal) | China  |
| 7   | Analyzer (vol.%)   | Atmocheck<br>O <sub>2</sub> /CO <sub>2</sub> / U.S.A |
|     |  | DOUBLE   |

## 4.3 Absorption Process

An aqueous solution (IL + MEA + Water) of 10 mL was applied and heated to the desired temperature (25-40)°C using a water bath at a specified concentration. The gas mixture (15% CO<sub>2</sub> and 85% N<sub>2</sub>) was allowed to contact the solution to absorb carbon dioxide. Gas flow rates were controlled by calibrated flow meters. The operating conditions of the absorption process are atmospheric pressure and the flow rate of a gas mixture of 133 mL/min (20 mL/min for CO<sub>2</sub> and 113 mL/min for N<sub>2</sub>). The gas exists of the cell uptake was analyzed by a carbon dioxide analyzer every five minutes to find out the saturation of the solvent (the solvent does not absorb more carbon dioxide). The run time (60 - 120) min was taken during the period in which there is a marked change in the carbon dioxide concentration which is measured with a carbon dioxide analyzer. Figures (1) and (2) show the schematic diagram and photo of the experimental setup for the absorption process used in this work.



**Figure 1.** The schematic diagram of the experimental setup.



**Figure 2.** Photograph of the whole experimental setup.

#### 4.4 Design of experiments

To investigate factors affecting the treatment process, RSM (Response Surface Methodology) and statistical experiment design approaches have grown popular [14,15]. 3-level factorial design, central composite design (CCD) [16,17], Box–Behnken design (BBD) [18], and D-optimal design [19] are examples of RSM designs. When compared to the other types of Response Surface Method (RSM) designs, the central composite design requires the fewest testing [20]. A response surface technique design is Central Composite Design (CCD). The CCD enables for the quantification of significant components and reactive factors, which is an advantage of this design. It also works to uncover the effect's nonlinear nature and to identify the ideal collection of experimental conditions for achieving the greatest outcome. While the CCD covers the same experimental area as the standard array design utilized in the primary study, it does so in a different way, it is less expensive and requires fewer tests in terms of cost. The Design Expert 11 version was utilized in this project. CCD design points



|    |    |      |     |     |        |
|----|----|------|-----|-----|--------|
| 14 | 4  | 32.5 | 1.5 | 120 | 0.0241 |
| 15 | 2  | 32.5 | 1.5 | 90  | 0.0673 |
| 16 | 11 | 32.5 | 1.5 | 90  | 0.0673 |
| 17 | 8  | 32.5 | 1.5 | 90  | 0.0673 |
| 18 | 9  | 32.5 | 1.5 | 90  | 0.0673 |
| 19 | 1  | 32.5 | 1.5 | 90  | 0.0673 |
| 20 | 5  | 32.5 | 1.5 | 90  | 0.0673 |

The experimental results were correlated with a second order polynomial as described below

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (1)$$

Where Y is the expected response, b<sub>0</sub> denotes the model constant, and X<sub>1</sub>, X<sub>2</sub>, and X<sub>3</sub> are independent variants. The linear coefficients are b<sub>1</sub>, b<sub>2</sub>, and b<sub>3</sub>; the interaction coefficients are b<sub>12</sub>, b<sub>13</sub>, and b<sub>23</sub>; and the quadratic coefficients are b<sub>11</sub>, b<sub>22</sub>, and b<sub>33</sub>. In this work, CCD was used to design 20 trials (Table 3), and all of the experiments were examined using analysis of variance (ANOVA). To measure the correctness of the relevant model, the coefficient of determination and its adjusted value were utilized. The F-test was also used to confirm the relevance of the linear and quadratic terms. In addition, the p-value is used to identify the final subset of variables.

## 5. Results and discussion

As indicated in Table 3, many tests were conducted under various experimental settings using Central Composite Design (CCD). The following operating parameters were used to attain a complete absorption rate: temperature 40°C, concentration 1.5 mol/l, and absorption time 90 minutes.

The coefficients were determined using a regression analysis based on the experimental data in Table 3 that were associated to quadratic response functions. With the calculated coefficients, After 90 minutes of absorption time, Equation (1) gives reaction functions to the percentage of absorption rate (Y).

$$Y = +0.0648 + 0.0055X_1 - 0.0163X_2 - 0.0194X_3 - 0.0013X_1X_2 + 0.0034X_1X_3 - 0.0096X_2X_3 + 0.0069X_1^2 + 0.0012X_2^2 - 0.0085X_3^2 \quad (1)$$

$$(R^2 = 0.93)$$

The absorption rate at the experimental sites was predicted using Equation (1). The response function predictions obtained are consistent with the experimental results. The ANOVA test was employed to measure the fit quality. The results of the ANOVA test based on the absorption rate are presented in Table 4. The model has been determined to be significant based on the F-value of absorption rate 14.74 presented in Table 4. The model conditions are also significant if the p-value is less than 0.05. The temperature of the solution, concentration, and absorption time all had a significant impact on CO<sub>2</sub> absorption.

**TABLE 4:** ANOVA test for the response function Y (absorption rate)

| Source         | Sum of Squares | Df | Mean Square | F-value | p-value  |
|----------------|----------------|----|-------------|---------|----------|
| Model          | 0.0079         | 9  | 0.0009      | 14.74   | 0.0001   |
| A-Temp.        | 0.0003         | 1  | 0.0003      | 5.19    | 0.0460   |
| B-Conc.        | 0.0027         | 1  | 0.0027      | 45.17   | < 0.0001 |
| C-Time         | 0.0038         | 1  | 0.0038      | 63.85   | < 0.0001 |
| AB             | 0.0000         | 1  | 0.0000      | 0.2154  | 0.6525   |
| AC             | 0.0001         | 1  | 0.0001      | 1.57    | 0.2381   |
| BC             | 0.0007         | 1  | 0.0007      | 12.55   | 0.0053   |
| A <sup>2</sup> | 0.0001         | 1  | 0.0001      | 2.24    | 0.1657   |
| B <sup>2</sup> | 3.871E-06      | 1  | 3.871E-06   | 0.0654  | 0.8033   |
| C <sup>2</sup> | 0.0002         | 1  | 0.0002      | 3.33    | 0.0981   |
| Residual       | 0.0006         | 10 | 0.0001      |         |          |
| Lack of Fit    | 0.0006         | 5  | 0.0001      |         |          |
| Pure Error     | 0.0000         | 5  | 0.0000      |         |          |
| Cor Total      | 0.0084         | 19 |             |         |          |

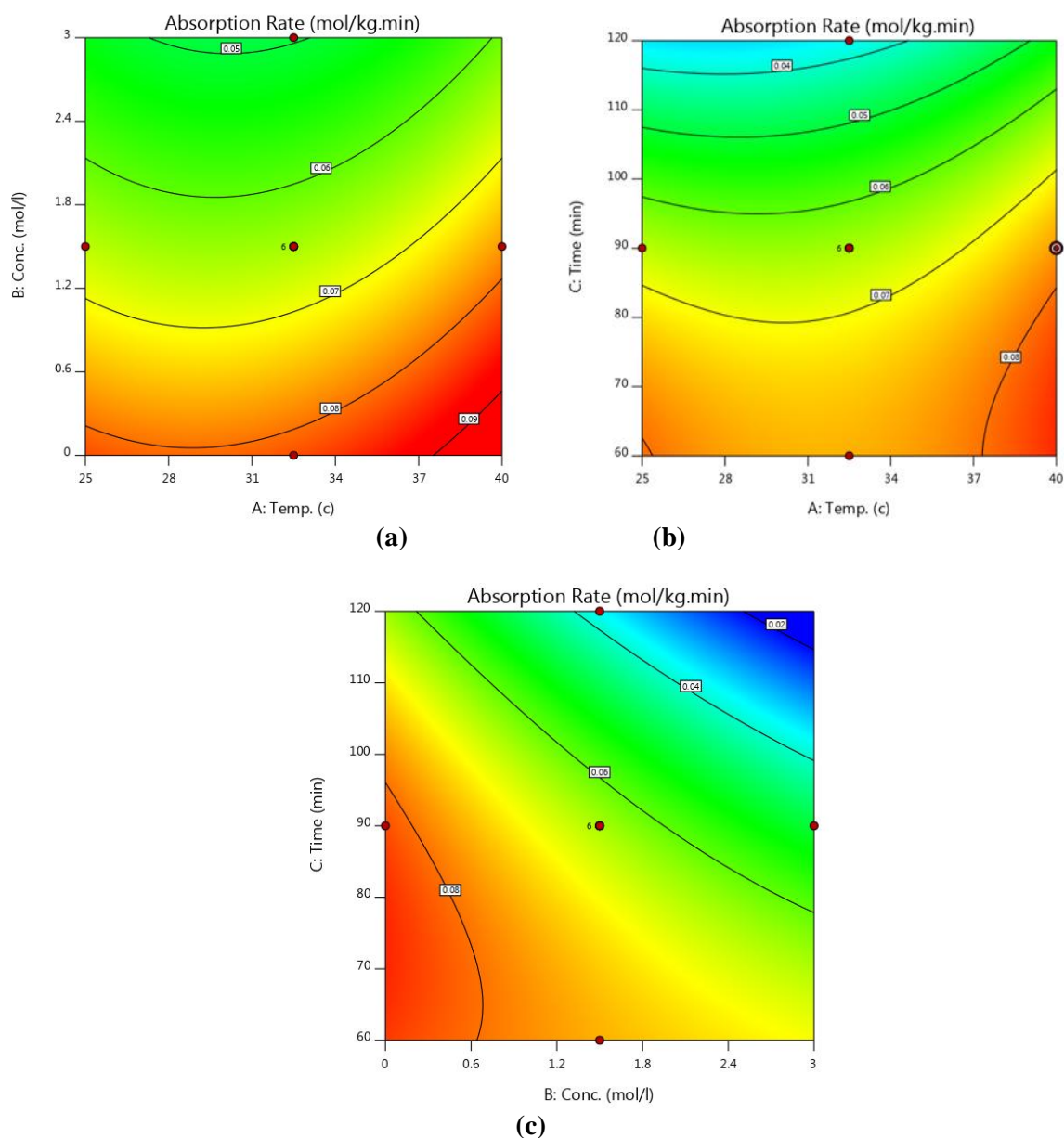
With p-values less than 0.05, X1, X2, X3, X12, X22, and X32 were identified as essential model parameters. According to the results of the ANOVA test on CO<sub>2</sub> removal efficiency, With a p-value less than 0.05, the variations X2, X3, X22, and X32 were essential model terms. The influence of influencing factors on the absorption rate is shown in Figure( 3-a, b, and c).

Figure.(3-a) depicts the impact of both the temperature and concentration on the absorption rate. With increasing temperature and decreasing concentration of ionic solvent [Bmim] [BF<sub>4</sub>], the absorption rate increase. The ability of the exothermic CO<sub>2</sub> absorption thermodynamics system to induce reversible responses was attributed to this [21]. And the size of the available cavities in [Bmim][BF<sub>4</sub>] is insufficient to hold a CO<sub>2</sub> molecule. When CO<sub>2</sub> is introduced, the [BF<sub>4</sub>]<sup>-</sup> anions are reorganized by a tiny angular displacement, which has no effect on the radial distribution functions, to generate larger voids that can store CO<sub>2</sub> molecules [22]. At a temperature of 32.5°C and concentration of 3 mol/l the absorption rate is 0.0369 mol/kg.min, but at a temperature of 40 °C and a concentration of 1.5mol/l, the absorption rate is 0.0795 mol/kg.min.

Figure.(3-b) depicts the impact of both the temperature and time on absorption rate The absorption rate increases with decreasing time and with increasing temperature at constant concentration. An increase in temperature may result in lower physical solubility, and therefore there should be a balance between reaction kinetic and physical solubility when temperature is a variable [23]. At a temperature of 32.5°C and after a time of 120 minutes, the absorption rate is 0.0241 mol/kg.min, but when the temperature is increased to 40°C and the concentration is stable, and after 90 minutes, the absorption rate is 0.0759 mol/kg.min.

Furthermore, Figure.(3-c) demonstrates the effect of both concentration and time on the rate of absorption. The absorption rate decreases with increasing time and increasing concentration with a constant temperature. This is because of two factors, the heat of the reaction increases as the concentration rises, causing the temperature to rise. As a result, the pressure of CO<sub>2</sub> vapor in the solution rises [22]. Second, for an equivalent acid-gas/amine mole ratio, the vapor pressure of acid-gas over the highly concentrated solution would be greater. Furthermore, raising the solvent concentration increases viscosity, which in many circumstances hampers the liquid's capacity to spread and reduces mass transfer [24]. At a concentration of 1.5mol/l and after 90 minutes, the absorption rate is 0.06 mol/kg.min, but at a concentration of 3mol/l and after 120 minutes and with constant temperature, the absorption rate is 0.0197 mol/kg.min





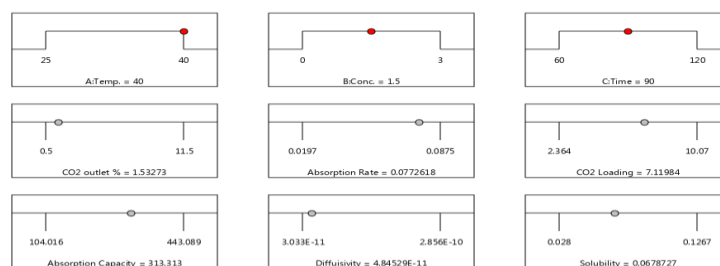
**Figure 3.** 3D response surface and contour plots: interactive effects of (a) Temperature and Concentration (b) Temperature and Time (c) Concentration and Time.

## 6. Optimization Choices

Response optimization helps identify the optimal factors that will give rise to an optimal response. The three factors were evaluated in this work to get the values that will give the maximum value of absorption rate. The essential objective of this study is to discover the best-operating conditions (temperature, concentration and absorption time) that give the highest absorption. Obtaining the optimal operating conditions as shown in Fig. (4) and Table (5), where the temperature, concentration of solvent and absorption time is 40 °C, 1.5 mol/l, 90 min for [Bmim][BF<sub>4</sub>], these values are economically good in comparison with other researchers and after these conditions the absorption efficiency decreases.

**Table 5:** optimal operating conditions

|    | Temp.  | Conc. | Time    | CO <sub>2</sub> outlet | Absorpti on Rate | CO <sub>2</sub> Loading | Absorption Capacity | Diffusivity             | Solubility |          |
|----|--------|-------|---------|------------------------|------------------|-------------------------|---------------------|-------------------------|------------|----------|
| 1  | 40.000 | 1.500 | 90.000  | 1.533                  | 0.077            | 7.120                   | 313.313             | $6.147 \times 10^{-11}$ | 0.068      | Selected |
| 2  | 32.500 | 1.500 | 60.000  | 1.833                  | 0.076            | 4.151                   | 182.625             | $4.5 \times 10^{-11}$   | 0.022      |          |
| 3  | 25.000 | 1.500 | 90.000  | 3.353                  | 0.066            | 5.964                   | 262.501             | $3.033 \times 10^{-11}$ | 0.023      |          |
| 4  | 40.000 | 0.000 | 60.000  | 0.606                  | 0.094            | 5.850                   | 257.395             | $4.576 \times 10^{-11}$ | 0.125      |          |
| 5  | 25.000 | 3.000 | 60.000  | 1.614                  | 0.076            | 4.784                   | 210.557             | $3.033 \times 10^{-11}$ | 0.033      |          |
| 6  | 40.000 | 0.000 | 120.000 | 1.514                  | 0.081            | 9.615                   | 423.052             | $4.576 \times 10^{-11}$ | 0.125      |          |
| 7  | 25.000 | 3.000 | 120.000 | 12.934                 | 0.011            | 1.559                   | 68.616              | $3.033 \times 10^{-11}$ | 0.033      |          |
| 8  | 32.500 | 1.500 | 120.000 | 8.553                  | 0.037            | 4.420                   | 194.484             | $4.5 \times 10^{-11}$   | 0.022      |          |
| 9  | 32.500 | 3.000 | 90.000  | 6.393                  | 0.050            | 4.305                   | 189.410             | $4.5 \times 10^{-11}$   | 0.041      |          |
| 10 | 40.000 | 3.000 | 60.000  | 1.294                  | 0.078            | 4.659                   | 204.986             | $6.147 \times 10^{-11}$ | 0.055      |          |

**Figure 4.** Process Optimization for the CO<sub>2</sub> removal by using absorption process for [Bmim][BF<sub>4</sub>]

### Conclusions

The absorption method was used to determine total CO<sub>2</sub> removal. The effect of independent variables on CO<sub>2</sub> absorption rate, such as temperature, concentration, and time, was investigated. Temperature and concentration were shown to be the most influential factors on absorption rate. It may be deduced that higher temperatures are required for higher absorption rates at lower concentrations of solvent, however increasing the concentration of solvent with increasing time at constant temperature also results in a high absorption rate. The best conditions for obtaining the highest rate of absorption are ( temperature 40°C, concentration 1.5 mol/l and absorption time 90 min) the absorption rate is 0.077 mol/kg.min.

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**Conflict of Interest:** The authors declare that they have no conflict of interest.

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