

## RESPONSE SURFACE HISTORICAL METHOD, SIMULATION OF CO<sub>2</sub> ABSORPTION PROCESS BY DEA IN FFR USING COMSOL MULTIPHYSICS

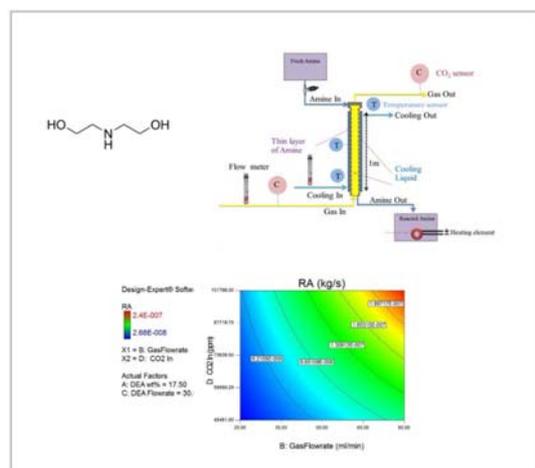
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Absorption of carbon dioxide as a greenhouse gas from a mixture of CO<sub>2</sub> and N<sub>2</sub> in a continuous falling film reactor (FFR) has been designed and developed. The reactor length and inner diameter were 1m and 0.021m, respectively. In this paper, the absorption of CO<sub>2</sub> by DEA (Diethanolamine) in FFR is studied by means of COMSOL Multiphysics and Design expert. Design expert is used to developing an empirical equation for CO<sub>2</sub> absorption when reacting with DEA. The simulation is performed with COMSOL Multiphysics for a second-order reaction and the velocity profile is considered the effect of shear stress. The result shows that the penetration depth of CO<sub>2</sub> in the falling film reactor is 0.006mm and the absorption rate increases with increasing gas flow velocity and inlet CO<sub>2</sub> concentration. Also, this study is performed on low Reynolds number ( $1 \leq Re_G \leq 6$  and  $4 \leq Re_L \leq 40$  at 298 K) which has not been considered by other researchers.



### INTRODUCTION

“The ultimate objective determined by the United Nations Framework Convention on Climate Change (UNFCCC) is to achieve stabilization of greenhouse gas concentrations in the atmosphere at a level that would prevent dangerous anthropogenic interference with the climate system”.<sup>1</sup> Therefore, reducing greenhouse gases such as CO<sub>2</sub> is of significant interest from an environmental point of view.<sup>1,2</sup>

To reduce CO<sub>2</sub> emission from various sources, chemical absorption has been used. Chemical absorption of CO<sub>2</sub> is considered as a process where CO<sub>2</sub> is absorbed by a liquid phase that contained reaction and absorptive mass transport. Aqueous amine solvents absorb CO<sub>2</sub> by chemical reaction.

These amines are classified as primary, secondary and tertiary. The reactivity order of amine to CO<sub>2</sub>; primary>secondary>ternary. DEA (Diethanolamine) aqueous solution is a secondary amine-based system that is used to sweeten sour gas and chemical absorption of CO<sub>2</sub> from flue gases.<sup>3</sup>

Chemical absorption needed gas-liquid contact that can be achieved in a falling film reactor (FFR) or contactor. No compression is needed before feeding it into an FFR, so it can be used to absorb CO<sub>2</sub> from flue gases.<sup>4</sup>

Numerous effort has been done to predicate the mass transfer process and obtaining knowledge of the absorption process for an FFR.<sup>5-19</sup> The mass transfer rates are investigated for the wavy and smooth falling film. The investigation has shown

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that the mass transfer rate was bigger for the wavy film and mass transfer characteristics varied substantially with the wave regime.<sup>6-9</sup> The film hydrodynamics, such as the velocity distributions and the vorticity variations at different positions of the wavy falling films, have been enhanced the mass transfer process.<sup>10</sup>

The analytical and numerical model has been applied to model FFR<sup>6-12</sup> and FFR could be simulated by means of COMSOL Multiphysics.<sup>13,14</sup>

Generally, the two-film theory is used to describe gas absorption. In this theory, two resistances have existed to the CO<sub>2</sub> diffusion into an amine solution. First, a stagnant film existed in the gas phase and the second another stagnant film can be considered in the liquid phase. Absorption can be calculated using the mass transfer coefficient, the mass, and heat transfer conversation equation. Since solving the coupled equations are very complex and some assumption is needed to do so, the results are not very accurate.<sup>16,17</sup>

Since little data are available for CO<sub>2</sub> absorption by means of DEA using FFR, in this paper, DEA is used to absorb CO<sub>2</sub> from the gaseous mixture (CO<sub>2</sub> and N<sub>2</sub>) and the operating parameters of FFR are investigated. Also, the absorption process in FFR for low Reynolds numbers, has been investigated and simulated using COMSOL Multiphysics. Finally, using the statistical method (Response Surface Historical Method (RSHM)) and the laboratory results, an

empirical equation has been developed that could calculate CO<sub>2</sub> absorption.

To investigate the absorption of the flue gases from the 200-MW boiler firing low-S fuel oil, CO<sub>2</sub> concentration was 5% and 10%. Predicated gaseous emission of CO<sub>2</sub> from the 200-MW boiler firing low-S fuel oil is about 10%.<sup>20</sup>

## MATERIALS AND METHODS

### 1. Materials

CO<sub>2</sub> and N<sub>2</sub> were procured from Fanavaran Petrochemical CO. Iran. DEA for CO<sub>2</sub> absorption was procured from Merck Company.

### 2. Procedure

The schematic diagram of the experimental setup and coordinate system are shown in Figure 1 and Figure 2. The setup is designed as a falling film reactor. The inner diameter of the reactor was 0.021m and the length of 1m. The flow in the reactor was counter current. The aqueous amine flowed from the reactor top. CO<sub>2</sub> gas was diluted to the desired concentration with nitrogen in a mixing chamber before entering the reactor from the bottom.

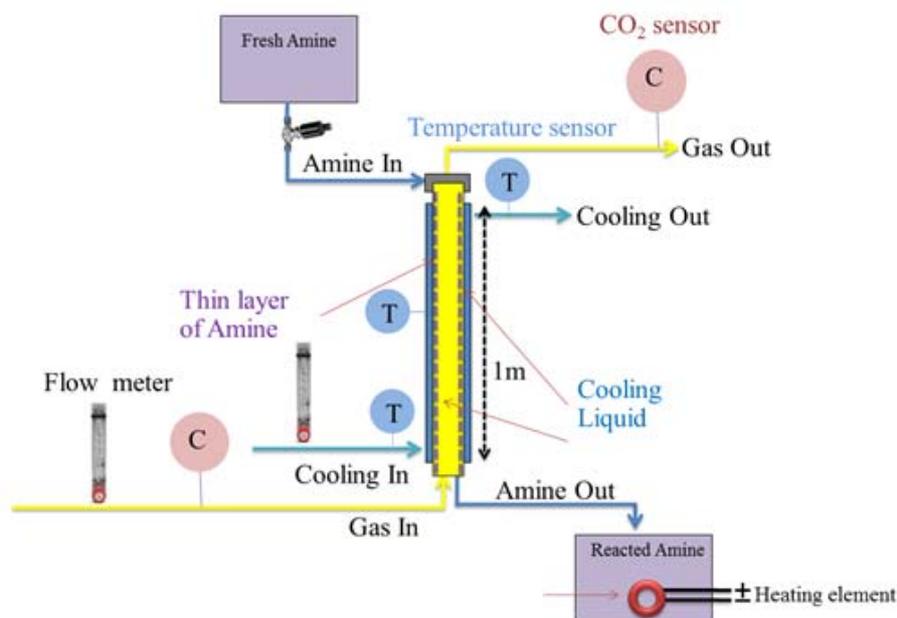


Fig. 1 – Experimental setup.

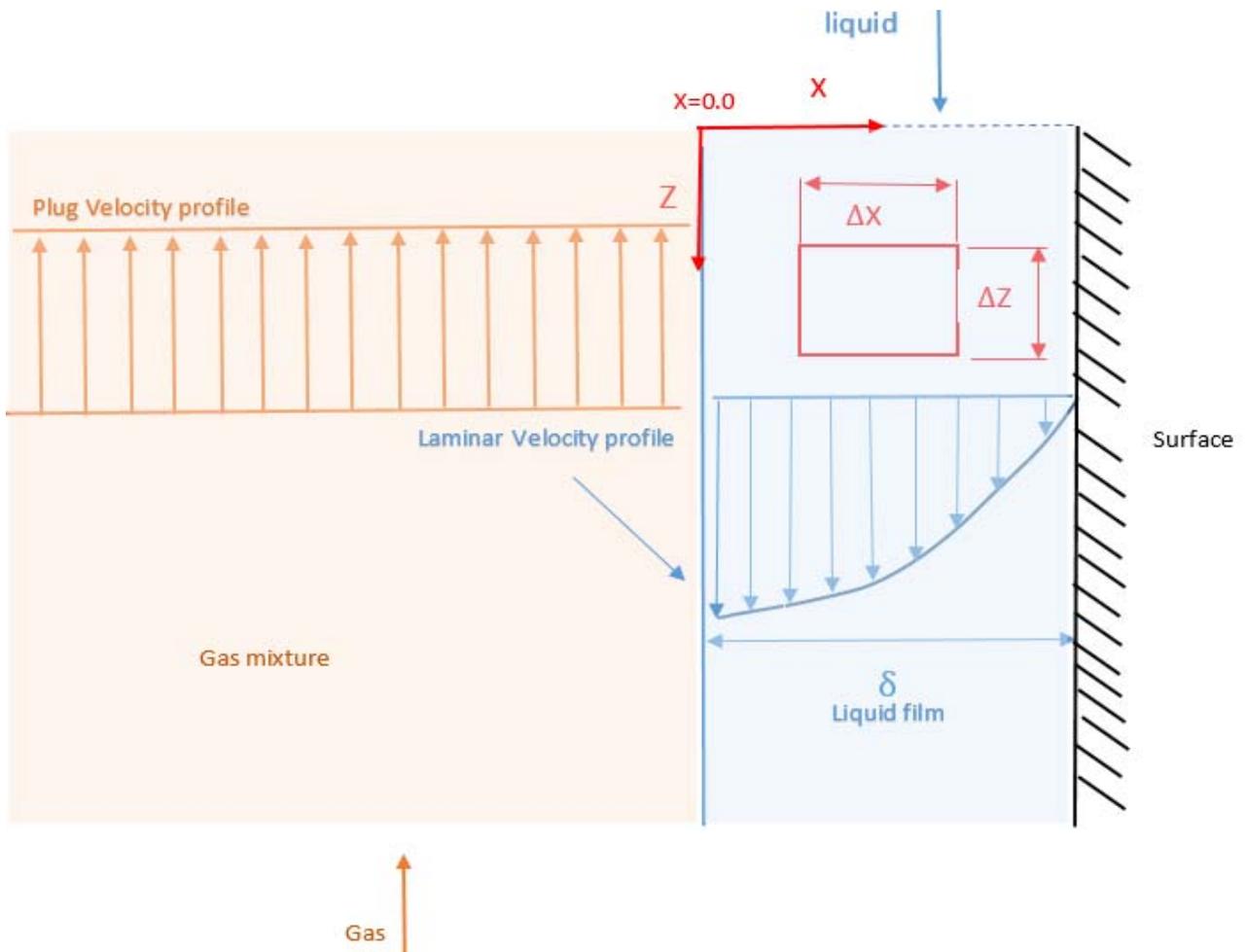


Fig. 2 – Flow model of the falling film reactor(x and z coordinate are in mm).

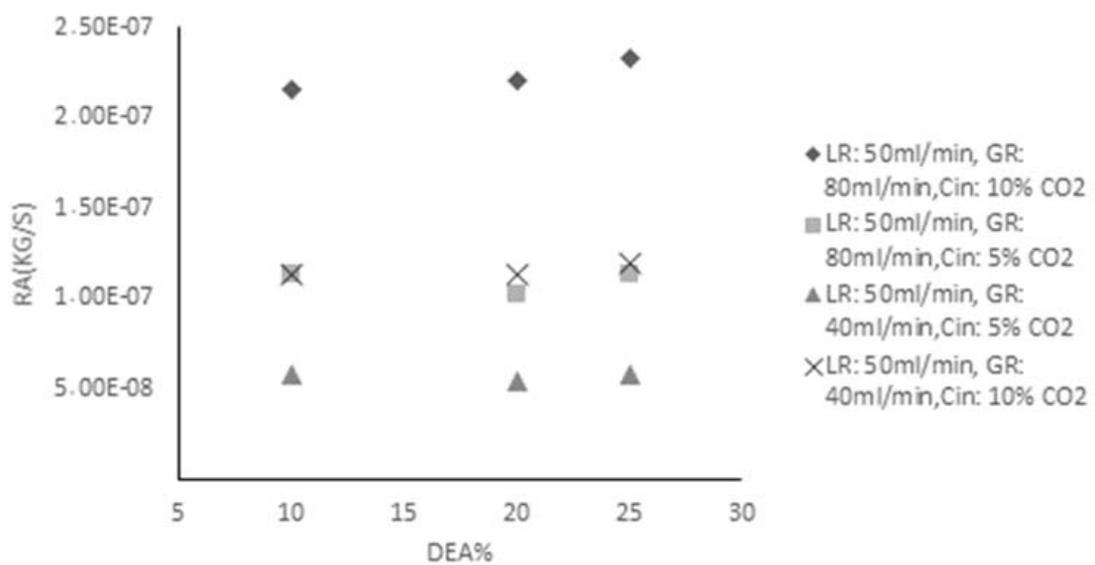


Fig. 3 – Comparison of gas absorption rate ( $R_A$ ) with DEA%.

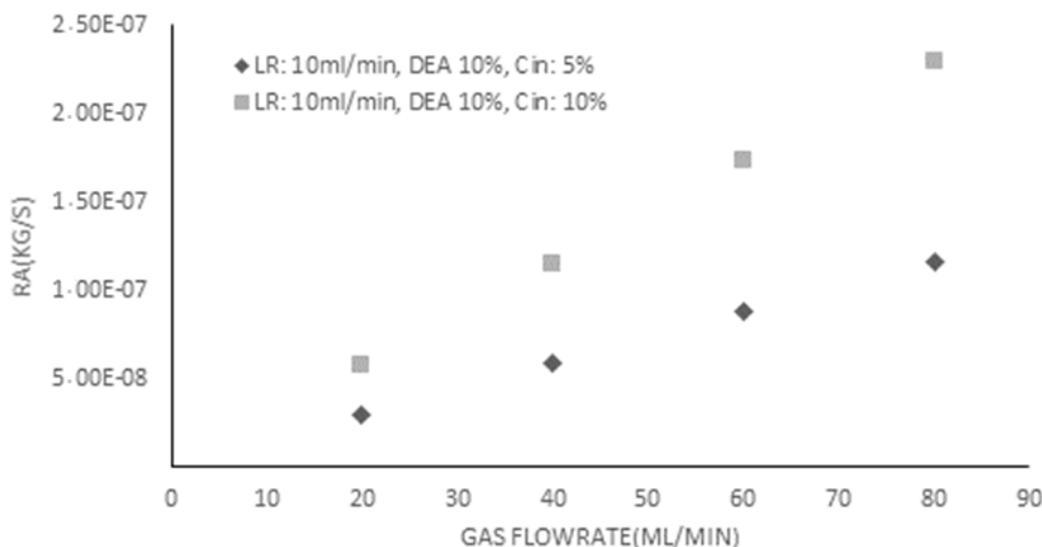


Fig. 4 – Comparison of gas absorption rate ( $R_A$ ) and a gas flow rate.

All the experiments were performed at a 298K temperature and 1atm pressure. The absorbent (DEA solution) selectively absorbs  $CO_2$  gas from the feed gas mixture and reacts simultaneously to form a stable carbamate in the reactor section. The input and output concentration of the gas were determined using two *K33 ICB 30%  $CO_2$  Sensor* from *CO<sub>2</sub> Meter Company*. The  $CO_2$  content can be monitored by *GasLab™* software online. The mass rate of absorption was calculated using the following equation:

$$R_A = C_{in}Q_{Gas} - C_{out}Q_{Gas} \quad (1)$$

where  $C_{in}$  and  $C_{out}$  were input and output concentration of  $CO_2$  to FFR respectively.

## RESULTS AND DISCUSSION

The input and output ppm of  $CO_2$  at the bottom and the top of FFR during the execution of 62 runs are tabulated in Table 1. According to the data, it is observed that with increasing amine concentration,  $CO_2$  concentration decreases. This is because as amine concentration increases, falling film thickness also increases (see Figure 5) and as a result driving force for mass transfer increases [4]. It is obvious that increasing the gas flow rate, causing an increase in  $CO_2$  output concentration. The absorption rate is increased slightly by increasing DEA percent (see Figure 3) but it is increased with increasing input gas flow rate and  $CO_2$  percent (see Figure 4). This is because there is

an increase in gas Reynolds number due to increasing gas flow rate.

### 1. Model reaction

#### 1.1. Physical properties of DEA

Density and viscosity of DEA solution were estimated from the work of Arachchige *et al.*<sup>21</sup> and Han *et al.*<sup>22</sup> The effect of DEA concentration and temperature on density and viscosity was considered in their experiments. The heat capacity of DEA solution was estimated from the work of Shokouhi *et al.*<sup>23</sup>

The diffusivity of DEA in DEA solution was estimated by the following equation.<sup>24</sup>

$$\ln(D) = -13.268 - 2287.7/T - 19.699 \times 10^{-5}C$$

$$\text{For } 9 \geq C \text{ (mol/m}^3\text{)} \geq 4013 \quad (2)$$

$$298 \geq T \text{ (K)} \geq 348$$

The diffusion coefficient of  $CO_2$  in DEA solution and Henry's constant was estimated from the work of Abu-Arabi *et al.*<sup>25</sup> In the model, the thermal conductivity of the DEA solution and the heat transfer coefficient are equal to 0.4893 (W/m K) and 900(J/m<sup>2</sup> s) respectively.

#### 1.2. Physicochemical properties of $CO_2$

The physicochemical properties of  $CO_2$  and the experimental conditions of the system required for the model are given in Table 2.

Table 1

Data from Lab work

Run	DEA%	Gas Rate (mL/min)	Amine Rate (mL/min)	Input CO2 (ppm)	Output CO2 (ppm)
1	10	20	10	49194	174
2	10	40	10	49570	347
3	10	60	10	49677	464
4	10	80	10	49532	819
9	10	40	50	50021	1550
10	10	80	50	50293	2769
11	10	60	50	50196	2139
12	10	20	50	50062	798
20	20	20	10	45303	182
21	25	40	10	49649	55
22	25	60	10	49803	23
23	25	80	10	49278	65
24	25	20	10	49019	30
33	10	80	10	98101	1903
34	10	80	20	98044	3559
35	10	40	20	97964	1952
42	10	60	10	98333	1119
43	20	40	20	98067	1117
44	20	80	20	98217	3239
45	20	80	50	98181	5557
46	20	40	50	97690	2118
47	20	40	10	97522	339
53	25	80	10	101355	267
54	25	20	10	100892	174
55	25	20	20	100770	316
61	25	40	50	101798	1686
62	25	20	50	101678	1146

Table 2

Experimental parameters and physiochemical properties of CO<sub>2</sub>-DEA system

Temperature (K)	298
Pressure (atm)	1
Height of the column, L (m)	1
The inner diameter of the column, d (m)	0.021
Gas flow rate, Q <sub>G</sub> (m <sup>3</sup> /s)	3.33 × 10 <sup>-7</sup> -1.33 × 10 <sup>-6</sup>
Liquid flow rate, Q <sub>L</sub> (m <sup>3</sup> /s)	1.67 × 10 <sup>-7</sup> -8.33 × 10 <sup>-7</sup>
Diffusivity of CO <sub>2</sub> in gas, D <sub>G</sub> (m <sup>2</sup> /s) <sup>4</sup>	1.67 × 10 <sup>-5</sup>
Viscosity of gas mixture, μ <sub>G</sub> (kg/m.s) <sup>4</sup>	1.72 × 10 <sup>-5</sup>
Heat of reaction (kJ/kmol) <sup>26</sup>	57200
Heat of solution (kJ/kmol) <sup>26</sup>	13240

### 1.3. CO<sub>2</sub>/DEA system: reaction kinetics

The overall reaction which accounts for the reaction between CO<sub>2</sub> and DEA is



The rate of the above equation can be described as:

$$r_{\text{CO}_2} = -k\text{C}_{\text{CO}_2}\text{C}_{\text{DEA}} \quad (4)$$

and the second-order rate constant of the overall reaction between CO<sub>2</sub> and DEA was determined by:<sup>27</sup>

$$\ln k \left( \frac{\text{m}^3}{\text{kmol}\cdot\text{sec}} \right) = 24 \cdot 515 - \frac{5411 \cdot 3}{T} \quad (5)$$

The following correlation is used to calculate the mass transfer coefficient for gas (k<sub>G</sub>):

$$\text{Sh}_G = 0.02922 \text{Re}_G^{0.7151} \text{Sc}_G^{0.5} \quad (6)$$

This correlation is based on 62 data points and predicts the experimental data with R<sup>2</sup> of 0.97. The coefficients of Eq (6) are estimated using non-linear least square regression in Excel by fitting the expression to the experimental data. The correlation was valid for 1 ≤ Re<sub>G</sub> ≤ 6 and 4 ≤ Re<sub>L</sub> ≤ 40 at 298K. The gas side mass transfer coefficient depended on the gas flow rate and its properties.

In the simulation, because of the calculated Hatta number, it was assumed that the reaction between

CO<sub>2</sub> and DEA was instantaneous, and interpretation for the kinetic regime was performed based on the published literature for an instantaneous chemical reaction.<sup>27</sup> The Hatta number in the present study, is greater than 3 for a film thickness of 0.2 mm and in the fast reaction region, indicating that the reaction can be assumed as an instantaneous reaction. Based on this, and due to the high solubility of CO<sub>2</sub> in DEA solution, the liquid-phase mass transfer resistance was neglected.

#### 1.4. Model equation

The following table shows the model equation.

#### 1.5. Solution procedure and Output

To create a 2D falling film model in COMSOL Multiphysics 5.1 environment, Transport of Diluted Species (tds), Heat Transfer in Fluids (ht), Coefficient Form Boundary PDE are required. After creating the model, the parameters and boundary conditions are defined for each physics. Then, a stationary study is used to solve the coupled equations (see Table 3).

The numerical solution must be applied to the coupled equations. In the numerical solution, predefined distribution type mesh is used (Distribution 1 in x-direction: Number of element 200 and Element ratio 0.01. Distribution 2 in z-direction: Number of element 50 and Element ratio 0.01). Therefore, memory and calculation time could be optimized. The film thickness of amine in the reactor was estimated by performing momentum balance in the liquid phase.<sup>4</sup> Figure 5 shows the film thickness and velocity profile for DEA solution. A more viscous DEA solution presents a lower velocity value at the interface and higher film thickness. "This behavior can be explained by Nusselt theory, which predicts lower velocity values and thicker liquid films as the viscosity increases".<sup>28</sup>

Finite element method and Non-linear solver is used to solve the non-symmetric matrix and the results are showed in the following figures. Figure 6 shows the concentration variation of CO<sub>2</sub> in the falling film of DEA.

Table 3

Mathematical model<sup>4</sup>

Chemical equation:



Equations

Liquid Phase:

$$u_L = \frac{g}{2\nu_L} \delta^2 \left( 1 - \left( \frac{x}{\delta} \right)^2 \right) - \frac{\tau_G \delta}{\mu_L} \left( 1 - \left( \frac{x}{\delta} \right) \right) \quad \tau_G = f \rho_G u_G^2 \quad (8)$$

Mass Balance  
Component A

$$u_L \frac{\partial C_A}{\partial z} = \frac{\partial}{\partial x} \left[ D_A \frac{\partial C_A}{\partial x} \right] - k C_A C_B \quad (9)$$

Component B

$$u_L \frac{\partial C_B}{\partial z} = \frac{\partial}{\partial x} \left[ D_B \frac{\partial C_B}{\partial x} \right] - k b C_A C_B \quad (10)$$

Heat Balance

$$u_L \frac{\partial T}{\partial z} = \frac{\partial}{\partial x} \left[ \alpha \frac{\partial T}{\partial x} \right] + \frac{\Delta H_R}{\rho c_p} k C_A C_B \quad (11)$$

Gas-Phase:

Mass Balance  
Component A

$$\frac{dw_G C_{AG}}{dz} = k_G (C_{AG} - H_0 C_A) \quad (12)$$

Heat Balance

$$\frac{dw_G c_G T_G}{dz} = h_G (T_{x=0} - T_G) \quad (13)$$

## Boundary Conditions

## Liquid Phase:

$$\text{At } x = \delta: \quad \frac{\partial C_A}{\partial x} = 0 \quad \frac{\partial C_B}{\partial x} = 0 \quad -k_\lambda \frac{\partial T}{\partial x} = U(T_{x=0} - T_R) \quad (14)$$

$$\text{At } x=0: \quad k_G(C_{AG} - C_{AH_0}) = -D_A \frac{\partial C_A}{\partial x} \quad \frac{\partial C_B}{\partial x} = 0 \quad h_G(T - T_G) - k_\lambda \frac{\partial T}{\partial x} = (-\Delta H_s) D_A \frac{\partial C_A}{\partial x} \quad (15)$$

$$\text{At } z = 0: \quad C_A = 0 \quad C_B = C_B^0 \quad T = T_0 \quad (16)$$

Also, Figure 7 shows the concentration variation of CO<sub>2</sub> in the falling film of DEA at various height (z) of the reactor (Amine flowrate:  $8.33 \times 10^{-7} \text{ m}^3/\text{s}$ , CO<sub>2</sub> concentration at the input gas: 10%, DEA aqueous solution Concentration: 25% – zoom mode). The reaction of CO<sub>2</sub> with amine occurred in a very small section of the falling film thickness (0.006mm). The driving force of mass

transfer at the bottom of the reactor is greater so the slope change is also large.

Simulation results show that the lowest concentration of DEA was  $2400 \text{ mol/m}^3$  so many moles of the DEA didn't react with CO<sub>2</sub> and could be reused. The highest concentration of CO<sub>2</sub> in DEA was  $0.24 \text{ mol/m}^3$  at the interface.

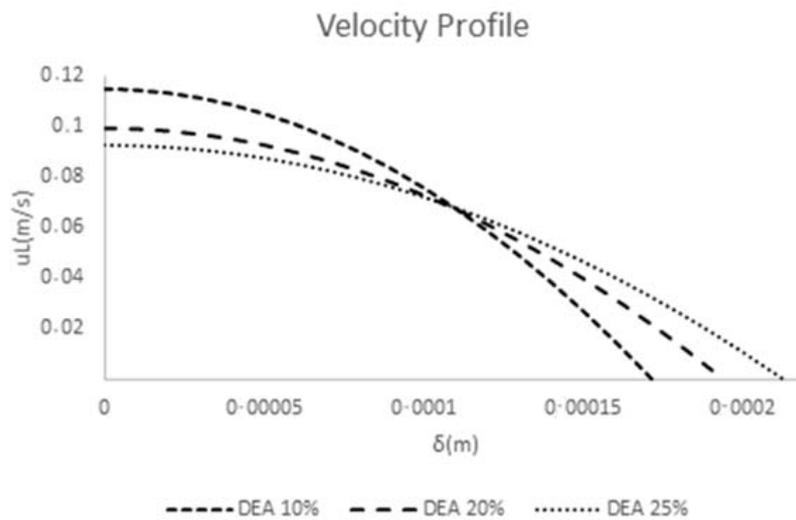


Fig. 5 – DEA velocity profile with various concentration, DEA flow rate:  $8.3 \times 10^{-7}$  Gas flow rate:  $6.7 \times 10^{-7}$ .

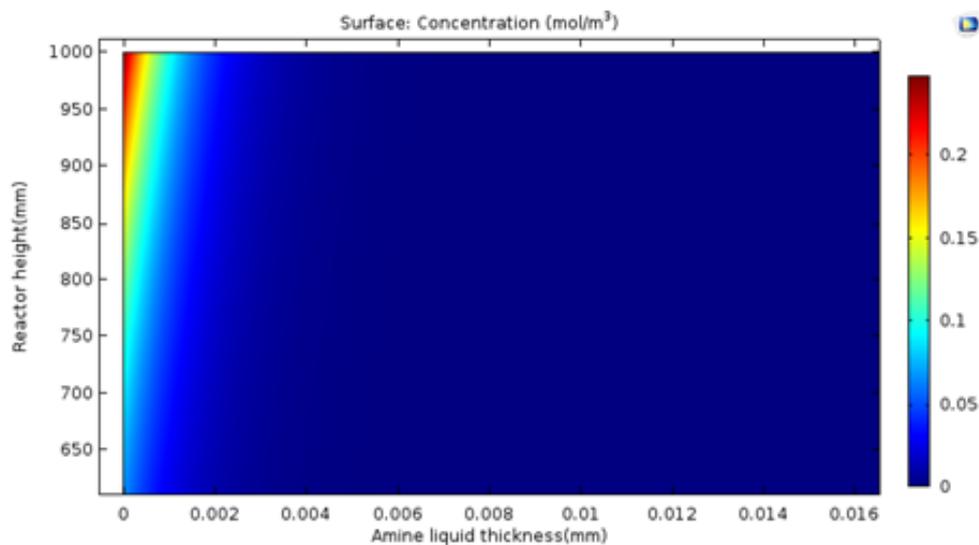


Fig. 6 – Liquid concentration of CO<sub>2</sub> in the falling film reactor in zoom condition.

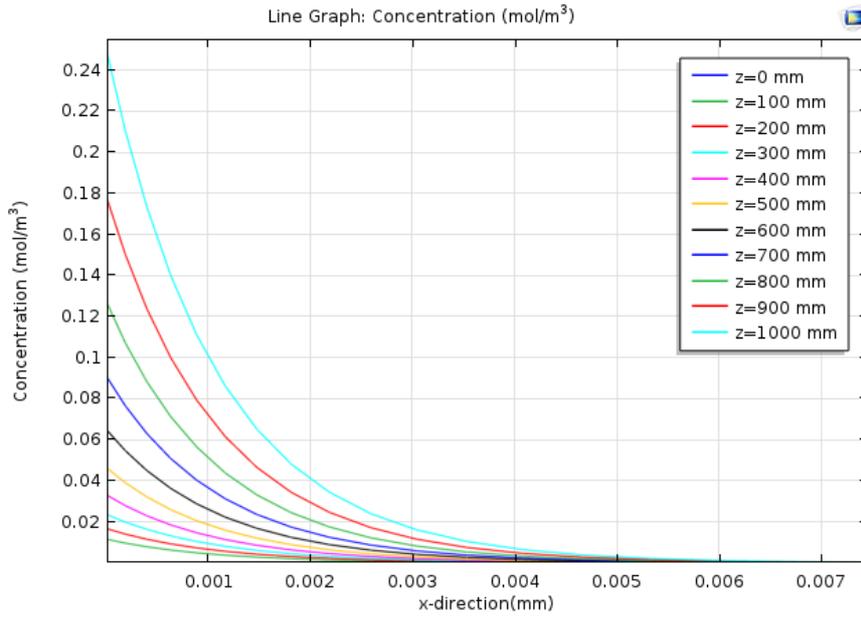


Fig. 7 – CO<sub>2</sub> Concentration in DEA at the various height of the reactor.

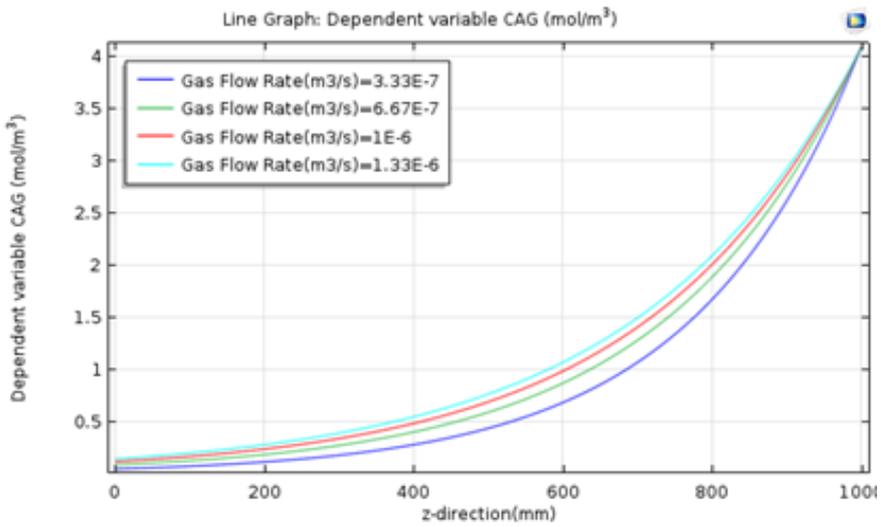


Fig. 8 – CO<sub>2</sub> Concentration for various gas flow rate in the gas phase.

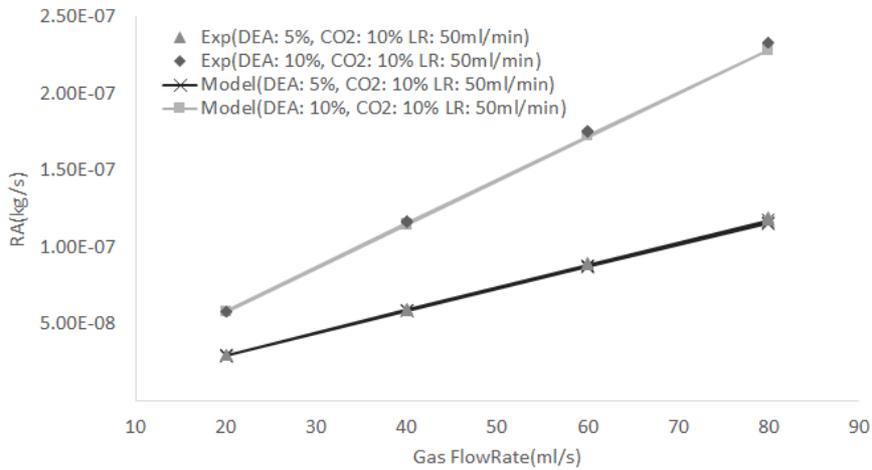


Fig. 9 – comparison of CO<sub>2</sub> absorption rate using DEA by model and the result from setup.

The effect of various operational parameter on the absorption process is simulated with COMSOL Multiphysics. Figure 8 shows the concentration variation of CO<sub>2</sub> in the gas phase for FFR (Amine flowrate:  $8.33 \times 10^{-7} \text{ m}^3/\text{s}$ , CO<sub>2</sub> concentration at the input gas: 10%, DEA aqueous solution Concentration: 25%). It has been observed that most of the CO<sub>2</sub> absorption and mass transfer occurred in a short section of FFR. When the gas flow rate has changed from  $3.33 \times 10^{-7}$  to  $1.33 \times 10^{-6} \text{ (m}^3/\text{s)}$ , the output concentration increased. As the gas flow rate increased, the gas velocity increased and the resident time decreased, so the CO<sub>2</sub> moles had less time to react with DEA.

The simulation shows that the temperature of the falling film has been approached to the cooling water temperature very soon because the falling film thickness was very thin (approximately 0.2mm), and close agreement between simulation results and experimental data is observed (Figure 9).

$$R_A = 1.068E-7 + 1.156E-9A + 6.266E-8B - 1.686E-9C + 4.055E-8D + 9.13E-10AB + 4.201E-10AC + 5.585E-10AD - 1.978E-9BC + 2.416E-8BD - 9.026E-10CD \quad (17)$$

In the linear terms, A, B, D and in the interaction terms, AB, AC, AD, and BD have a significant synergistic effect on the response, since they have a positive coefficient, whereas the negative coefficient of C, BC and CD show a significant antagonistic effect.

The model has an F-value of 18391.39 (much greater than unity) and a p-value of 0.0001 (<0.05), which also implies that the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. In this case, A, B,

## 2. RSHM & CO<sub>2</sub> absorption using DEA in FFR

Design expert can be used to model many chemical processes and is an effective tool in experimental design.<sup>29,30</sup> The variables studied in this work are DEA (wt%) (A), gas flow rate (mL/min) (B), DEA flowrate (mL/min) (C) and input CO<sub>2</sub> concentration (ppm) (D), to determine absorption rate or gain ( $R_A$  (kg/s)). The range of these parameters was selected based on laminar flow in the liquid phase, gas phase ( $1 \leq Re_G \leq 6$  and  $4 \leq Re_L \leq 40$  at 298 K) and setup specification. 2FI(two-factor interactions) model is used to determine response( $R_A$ ) according to effective factors(A, B, C, D) on the absorption process.

### 2.1. 2FI Model and ANOVA Analysis

The experimental data were analyzed and a 2FI model was developed to correlate process parameters with the response, as presented in the following equation in terms of coded factors.

C, D, AB, AC, AD, BC, BD, CD are significant model terms. The  $R^2$  value for the 2FI model is 0.9997. The “Pred R-Squared” of 0.9995 is in reasonable agreement with the “Adj R-Squared” of 0.9997, expressing that the model is significant. An adequate precision value of 440.864 (>4) implies an adequate signal ratio. So this model can be used to navigate design space.

Normal Plot of Residuals is shown in Figure 10 and it is observed that the values on this graph are very close to red lines and this is due to a low error.

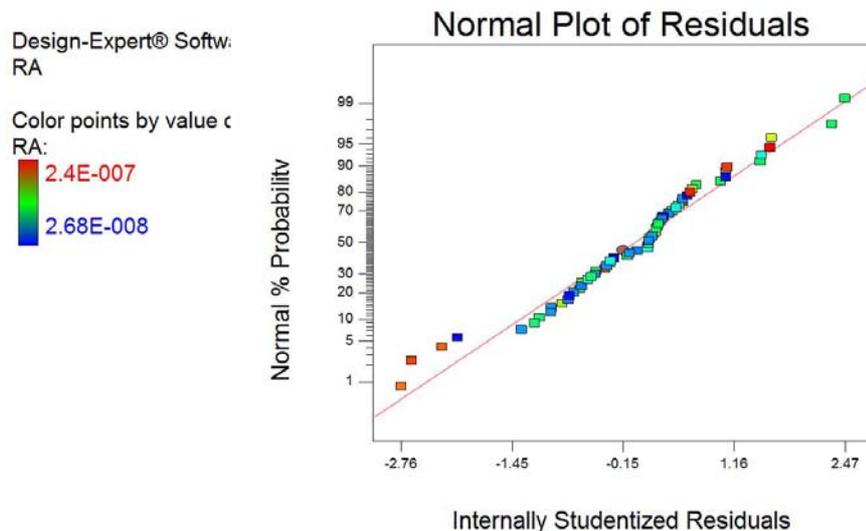


Fig. 10 – Normal Plot of Residuals for Eq. 7.

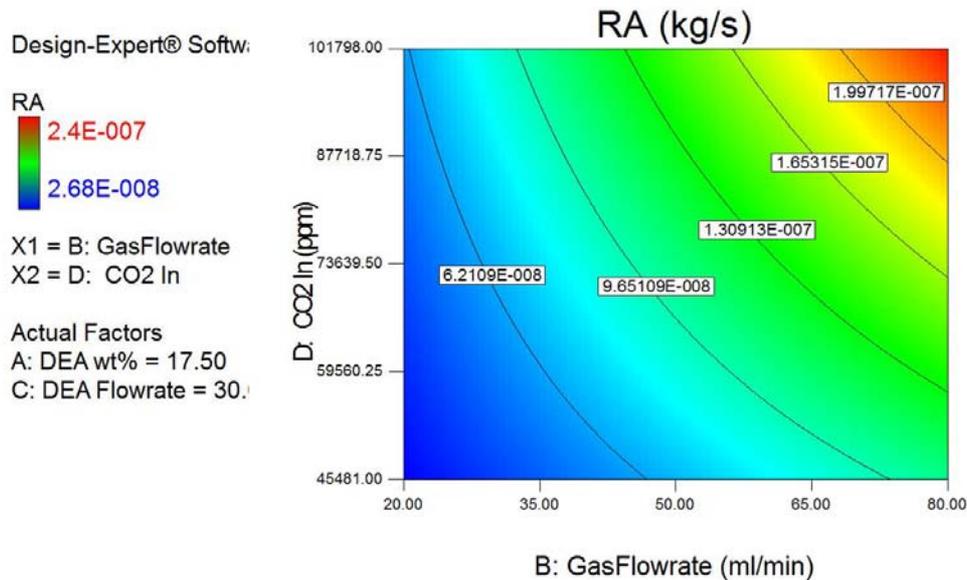


Fig. 11 – Contour plot for CO<sub>2</sub> absorption ( $R_A$  (kg/s)) as a function of gas flow rate (mL/min) and CO<sub>2</sub> input ppm at a DEA % of 17.5 and a DEA flowrate of 30 mL/min.

## 2.2. Response surface analysis

To understand the behavior of various process variables, Contour plots were generated. The effect of two parameters, keeping the third and fourth constant, is depicted in Figure 11. It is observed that when DEA percent and DEA flowrate were constant, an increase in CO<sub>2</sub> input concentration and gas flow rate has been increased the absorption rate. At a fixed gas flow rate, the CO<sub>2</sub> absorption rate was a strong function of CO<sub>2</sub> input ppm.

## CONCLUSIONS

DEA can be used to reduce the CO<sub>2</sub> concentration from the flue gas. Falling film reactor is a very useful tool for this purpose and operating parameter could be controlled to get optimum result. The numerical modeling of the CO<sub>2</sub> absorption process in FFR is simulated by COMSOL Multi-physics software environment 5.1 and a clear picture of the reaction occurred in the reactor is shown. The model has predicted the setup data precisely. The penetration depth of CO<sub>2</sub> into the falling film of DEA was 0.006 mm. Since most of the CO<sub>2</sub> absorption and mass transfer occurred in a short section of FFR, it is suggested that the size of the reactor could be reduced.

In a falling film reactor, 2FI model can be used to predict the absorption rate of CO<sub>2</sub> in terms of the operating parameters, and for low Reynolds number, gas flowrate and CO<sub>2</sub> input concentration can be the most effective parameters.

*Acknowledgements.* Mahshahr Branch, Islamic Azad University is gratefully acknowledged.

## Nomenclature

A	gaseous reactant
$a_c$	correction factor for the interfacial area, $(d - 2\delta)/d$
B	liquid reactant
$C_A$	concentration of dissolved gas A (kmol/m <sup>3</sup> )
$C_{AG}$	concentration of A in bulk gas (kmol/m <sup>3</sup> )
$C_B$	concentration of Reactant B (kmol/m <sup>3</sup> )
$C_B^0$	inlet concentration of Reactant B (kmol/m <sup>3</sup> )
d	inner diameter of the column (m)
$D_G$	diffusion coefficient of CO <sub>2</sub> in gas (m <sup>2</sup> /s)
$H_o$	Henry's constant
$h_G$	heat transfer coefficient in gas phase (J/s m <sup>2</sup> K)
k	second order rate constant at a temperature $T_r$ (m <sup>3</sup> /kmol s)
$k_G$	mass transfer coefficient (m/s)
$k_\lambda$	thermal conductivity (W/m K)
L	height of the column (m)
$Q_L$	volumetric flow rate of liquid (m <sup>3</sup> /s)
$Q_G$	volumetric flow rate of gas (m <sup>3</sup> /s)
r	rate of reaction (kmol/m <sup>3</sup> s)
$R_A$	rate of absorption (kg/s)
$Re_G$	gas-phase Reynolds number, $(d - 2\delta) u_G \rho_G / \mu_G$
$Re_L$	liquid-phase Reynolds number, $4\Gamma / \mu_L$
$Sc_G$	Schmidt number, $\mu_G / \rho_G D_G$
$Sh_G$	Sherwood number, $k_G a_c d / D_G$
T	liquid temperature (K)
$T_G$	gas-phase temperature (K)

$T_o$	inlet liquid temperature (K)
$T_R$	temperature of cooling water (K)
$U$	overall heat transfer coefficient for cooling water ( $J/s\ m^2\ K$ )
$u_G$	velocity of gas film (m/s)
$u_L$	axial velocity of liquid film (m/s)
$w_G$	molar flow rate of gas per unit wetted perimeter ( $kmol/(m\ s)$ )
$x$	radial coordinate (m)
$z$	axial coordinate (m)
$-\Delta H_R$	Heat of reaction ( $J/kmol$ )
$-\Delta H_S$	Heat of solution ( $J/kmol$ )

## Greek letters

$\Gamma$	volumetric liquid flow rate ( $m^3/s$ )
$\delta$	liquid film thickness (m)
$\mu$	liquid viscosity ( $kg/m\ s$ )
$\nu$	kinematic viscosity of liquid ( $m^2/s$ )
$\rho$	liquid density ( $kg/m^3$ )
$\tau$	shear stress ( $N/m^2$ )
$\alpha$	thermal diffusivity ( $m^2/s$ )

## Subscripts

$G$	gas phase
$i$	interface
$in$	inlet
$L$	liquid phase
$out$	outlet

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