

Experimental Studies of CO₂ Capturing from the Flue Gases

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Abstract

CO₂ emissions from combustion flue gases have turned into a major factor in global warming. Post-combustion carbon capture (PCC) from industrial utility flue gases by reactive absorption can substantially reduce the emissions of the greenhouse gas CO₂. To test a new solvent (AIT600) for this purpose, a small pilot plant was used. This paper presents the results of studies on chemical methods of absorbing CO₂ from flue gases with the new solvent, and evaluates the effects of operating conditions on CO₂ absorption efficiency. CO₂ removal rate of the AIT600 solvent was higher in comparison to the conventional monoethanolamine (MEA) solvent. The optimized temperature of the absorber column was 60 °C for CO₂ absorption in this pilot plant. The overall absorption rate (Φ) and the volumetric overall mass transfer coefficient ($K_G a_V$) were also investigated.

Keywords: Operating Conditions, CO₂, Air Flow Rate, Chemical Absorption, Efficiency

1. Introduction

CO₂ is a greenhouse gas and its emissions substantially contribute to global warming. Therefore, the interest in techniques which allow reducing CO₂ emissions has tremendously risen over the past years. According to the International Energy Agency (IEA), approximately one third of all CO₂ emissions come from the combustion of fossil fuels to generate electricity (IEA, 2007). CO₂ capture processes represent allowable options for the abatement of greenhouse gas emissions, especially from fossil fuel power generation (IPCC, 2005).

CO₂, a major greenhouse gas, may need to be removed from flue gas produced by combustion of fossil fuels in order to enable the management of the future climate changes. Carbon capture and storage (CCS) is one of the most effective mitigation measures to reduce CO₂ emissions and to stabilize the atmospheric concentration of CO₂ in the mid to long term (2030–2100) (Steenveveldt et al., 2006). CCS is a process consisting of the separation of CO₂ from industrial and energy related sources, transporting it to a storage location, and its long-term isolation from the atmosphere. From this definition, CCS consists of three basic stages: (a) separation of CO₂, (b) transportation, and (c) storage (Hattenbach et al., 1999).

There are three major approaches for CCS: post-combustion capture, pre-combustion capture, and oxy-fuel process. The capture of CO₂ in the downstream of a carbonaceous fuel based combustion unit is referred to as post-combustion capture process. Conventional process heaters and industrial utility boilers fit into this category. In this processes, the fossil fuels are combusted in excess air, resulting in a flue gas stream, which contains a lean concentration of CO₂ (12-15 % (v/v) for modern

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coal-fired power plants and 4-8 % (v/v) for natural gas fired plants). Because nitrogen is the major component of flue gas in power plants that burn fossil fuels in the air, post-combustion capture is essentially a nitrogen-carbon dioxide separation. If there were no nitrogen, CO₂ capture from flue gas would be greatly simplified. This is the thinking behind oxy-combustion capture: instead of air, the power plant uses a high purity ($\geq 95\%$) oxygen stream for combustion of the coal. The oxygen is produced onsite in an air separation plant, which represents the largest cost component in the capture process (Howard, 2011).

The pre-combustion process is basically a de-carbonization of carbonaceous fuels. As the name implies, this refers to the capture of CO₂ prior to combustion. It is an option for integrated coal gasification combined cycle (IGCC) plants. In these plants, coal is first gasified to form a synthesis gas (syngas, a mixture whose key components are carbon monoxide and hydrogen). The syngas then undergoes the water-gas shift, in which the CO reacts with steam to form CO₂ and additional H₂. The CO₂ is then removed, and the hydrogen is diluted with nitrogen and fed into a gas turbine combined cycle (Erlach et al., 2011).

Post-combustion capture is important because:

It is compatible and can be retrofitted to the existing coal-fired power plant infrastructure without requiring substantial changes in the basic combustion technology.

It is the leading candidate for gas-fired power plants. Neither the oxy-combustion nor the pre-combustion approaches are well suited for gas plants.

It offers flexibility. If the capture plant shuts down, the power plant can still operate. The other two capture options are highly integrated with the power plant: so if capture fails, the entire plant must shut down (Wang et al, 2011).

The chemical method has been widely used due to its advantages of simple operation, high absorption efficiency, high economic value, and mature technology. The chemical method is one of the most widespread methods of removing CO₂ from combustion flue gases in the industry process (Dugas and Rochelle, 2009).

Aqueous mono-ethanol-amine (MEA) solutions have been commonly used for the commercial production of CO₂ and have been tested on pilot scale for carbon dioxide capture from power generation. The CO₂ absorption using aqueous MEA solutions has the advantage of high rates of absorption. However, the solvent entails degradation and corrosion issues and a large heat requirement for carbon dioxide desorption (Liqiang et al., 2012).

Eimer (2005) identified three alternative solvents which have about 30% lower energy consumption and significantly lower degradation rates compared to MEA, i.e. Mitsubishi's KS-1, University of Regina's PSR and Praxair's solvent. KS-1 is already implemented commercially at a fertilizer plant in Malaysia while PRS and Praxair's solvents are still in testing stage but they could soon be available commercially (IEA, 2008). KS-1 solvent is also not a corrosive substance in contrast to MEA. Hence it does not require a corrosion inhibitor (Gronvold et al., 2005). Meanwhile, integrated projects supported by the European Commission have so far developed several versions of new solvents such as CASTOR1, CASTOR 2, CESAR1, and CESAR 2 (Wang et al, 2011).

Alternative solvents are therefore being investigated. In this work, a novel amine solvent (AIT600) was used. The influence of temperature, solvent concentration, CO₂ mole fraction, and the flue gas flow rate on the rate of CO₂ absorption was measured using an absorption/desorption pilot plant. The

goal of the paper is to present the plant itself, to critically discuss its operation, to evaluate the resulting data, and to describe extensive pilot plant studies with the AIT600 solvent. As most of this is not plant specific, the main conclusions should also hold for other plants of this type, many of which are presently being built or has recently been put in operation.

2. Experiments

2.1. Material

The following materials are used in the experiments: A CO₂ cylinder (mass fraction purity 0.999), a blended amine solution (AIT600), which is a proprietary solvent with mass fraction purity of 30 wt.% prepared by Abadan Refinery Faculty Research Center, and a potassium hydroxide, KOH, solution in ethanol (Merck, C (KOH) =0.5 mol/l). All of the solutions were prepared with deionized water.

2.2. Apparatus and experimental procedure

The process flow scheme of the absorption/desorption process and the picture of the pilot plant for post-combustion CO₂ capture by reactive absorption are shown in Figures 1 and 2 respectively. The main specifications of the pilot plant are given in Table 1. The flow sheet represents a continuous absorption regeneration cycling process. CO₂ is captured in the absorber column at the temperature of 50 °C and it is released from the regenerator at the temperature of 120 °C. In our investigation, the experimental apparatus consists of a packed column absorber to promote gas liquid contact and reaction in a counter current flow pattern.

Table 1
Main specification of pilot plant.

Parameter	Data
Flue gas source	Air compressor and CO ₂ gas Cylinder
CO ₂ content in the flue gas (vol.%)	5-20
Flue gas flow rate(m ³ /hr)	2-5
Solvent flow rate (ml/min)	100-400
Type of packing in the absorber and stripper	Metal grid
Total height of packing in the absorber (m)	1.5
Total height of packing in the stripper (m)	1.5
Absorber pressure (bara)	1

The flue gas was produced by an air compressor and the CO₂ gas cylinder. The flue gas flow rate can be set approximately between 2 m³/hr and 5 m³/hr. The flue gas enters the absorber at the bottom with a temperature of approximately 30 to 50 °C.

The absorber consists of five sections, each equipped with 0.3 m of the random packing rushing rings. The regenerated solvent (lean solvent) is fed to the absorber top, typically at a temperature of 60 °C. Upon the CO₂ absorption into the liquid phase, absorption enthalpy is released, which leads to a temperature increase. For steady state operation, the liquid level in the absorber bottom is controlled by a pump. The rich solvent is pumped into the desorber through the rich lean heat exchanger, where the CO₂ rich solvent is heated to higher temperatures through the lean solvent from the desorber

bottom. The bottom of the desorber contains electrical heating elements for partial evaporation of the solvent. For aqueous amine solutions, mainly water is evaporated. The vapor at the top of the desorber consists of water, CO₂, and some traces of amine. The vapor in the desorber top is led into the condenser, where most of the water is removed so that almost pure CO₂ is obtained.

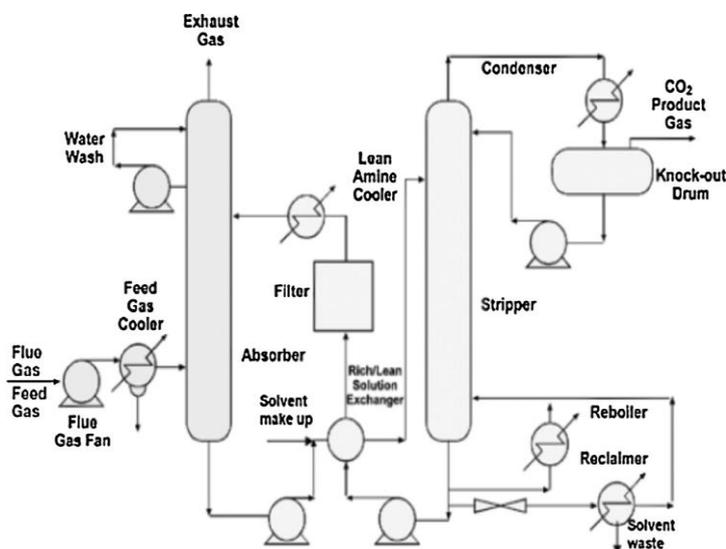


Figure 1

Typical process diagram of CO₂ capture using solvent absorption in industry.

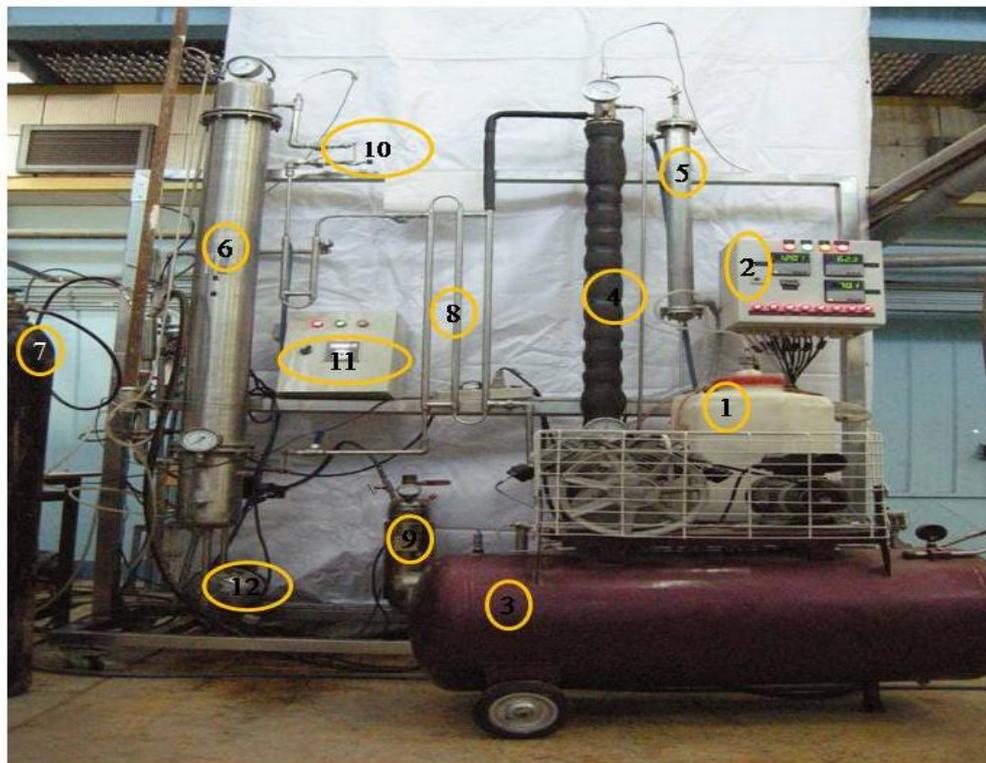


Figure 2

The pilot plant with all equipment employed in the experiments; Different parts of pilot plant which are shown in Figure 2 are as follows: 1-feed tank, 2-control switch, 3-compressor, 4-desorption column, 5-condenser, 6-absorption column, 7- CO₂ cylinder, 8-heat exchanger, 9-boiler, 10-liquid flow meter, 11-termocoupl, 12-pump.

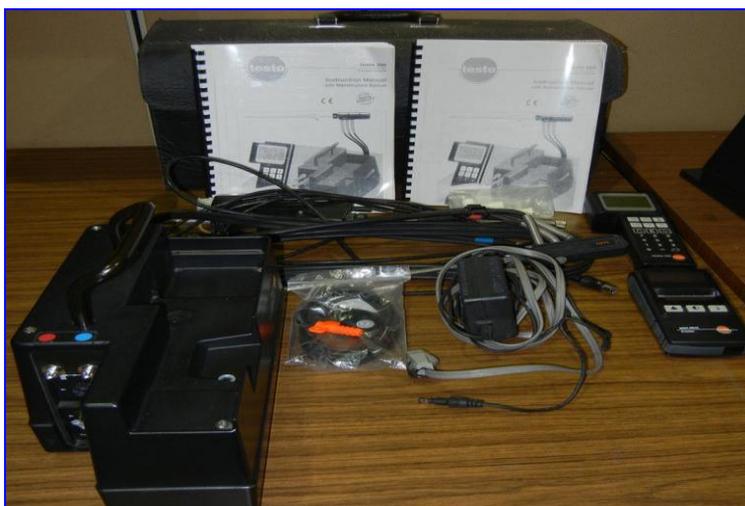


Figure 3

Picture of testo350 flue gas analyzer.

The basic operating conditions for the pilot plant are given as below:

Gas flow rate: 3 m³/hr, CO₂ concentration in flue gas: 10%, absorber column temperature: 50 °C, and liquid solvent flow rate: 300 ml/min. The concentrations of CO₂/O₂ were measured with a gas analyzer (testo350 flue gas analyzer).

Y represents the result of the measurement, i.e. Y is the efficiency of the CO₂ capture

$$Y_i = \frac{y_{CO_2, in} - y_{CO_2, out}}{y_{CO_2, in}}$$

2.3. Determination of the CO₂ content in the liquid by KOH titration

The amount of CO₂ in liquid samples is determined by titration with a KOH solution. The analysis is carried out at least twice to judge the reproducibility and to reduce the measurement error by averaging.

3. Results and discussion

3.1. Effect of operating conditions on the CO₂ absorption efficiency

3.1.1. Absorbent concentration

The effect of absorbent concentration on CO₂ absorption efficiency is shown in Figure 4. The flow rate of flue gas, the flow rate of liquid solvent, the input temperature of absorbents, and the CO₂ concentration of flue gas were 3 m³/hr, 300 ml/min, 60 °C and 10% respectively. The ratio of liquid to gas (L/G) was kept constant with the value of 6 l/m³.

We can see from Figure 4 that the CO₂ absorption efficiency increases with the concentration of absorbent increasing, then plateaus. When the absorbent concentration is low, increases in absorbent concentration lead to a higher growth rate in CO₂ absorption efficiency. However, when the absorbent concentration is high, CO₂ absorption efficiency growth is not obvious. These phenomena are determined by reversible equilibrium conditions and the gas-liquid two-phase mass transfer conditions

(Mansourizadeh et al., 2010). In the view of chemical dynamics, increasing absorbent concentration is equivalent to increasing the reactant concentration, resulting in the response moving to the positive direction, improving the reaction rate, and the CO₂ absorption efficiency (Khan et al., 2011). Thus in different operating conditions, it is feasible to improve CO₂ absorption efficiency by increasing the absorption concentration.

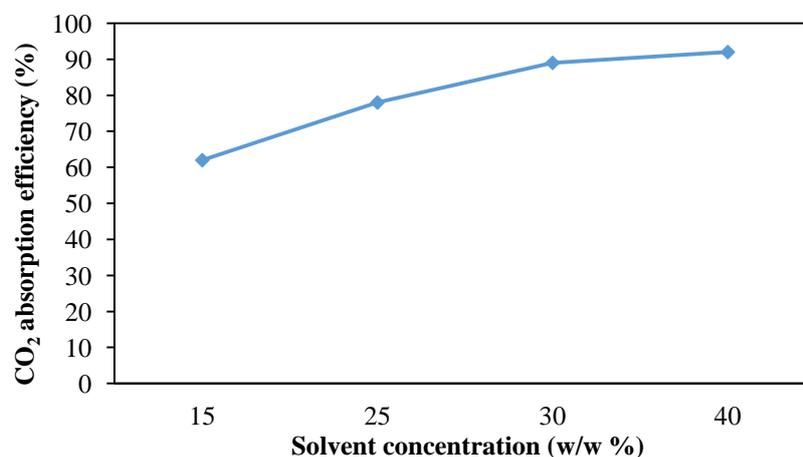


Figure 4

Effect of absorbent concentration on absorption efficiency.

3.1.2. Absorbent flow rate

The effect of the absorbent flow rate on the CO₂ absorption efficiency is shown in Figure 5. The other operation conditions are the same as basic operation conditions except the ratio of the flow rate of liquid.

It can be seen from Figure 5, the CO₂ absorption efficiency increases when the absorbent flow rate rises. It can be explained when the flow rate increased, the interfacial area between gas-liquid places increased. Therefore, the result is to enhance gas-liquid mass transfer rate and improving the CO₂ absorption efficiency (Kohl and Nilson 1997).

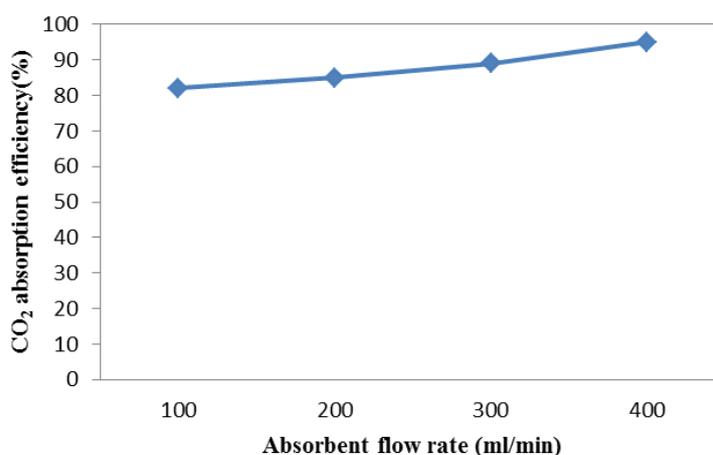


Figure 5

Effect of absorbent flow rate on absorption efficiency.

Combining two aspects of the effect, CO₂ increases when absorbent flow rate rises (Kohl and Nielson, 1997). Therefore, in certain experimental conditions, it is possible to improve CO₂ absorption efficiency by increasing the absorbent flow rate.

3.1.3. CO₂ concentration in flue gas

The effect of CO₂ concentration in flue gas on absorption efficiency is shown in Figure 6. It can be seen in Figure 6 that, CO₂ volume concentration changes in the range of 5% to 20% and other operating conditions are the same as the basic operation conditions. As the CO₂ concentration increases, the CO₂ absorption efficiency decreases; but the decline is small. There may be two reasons for this trend: on the one hand, as the CO₂ concentration increases, the unit volume of gas liquid contact time gets shorter in the tower; on the other hand, increasing CO₂ concentration promotes the reaction rate of CO₂ absorption to a certain extent (Tan et al., 2012). The combination of these two factors makes CO₂ absorption efficiency slowly decrease when the CO₂ concentration rises.

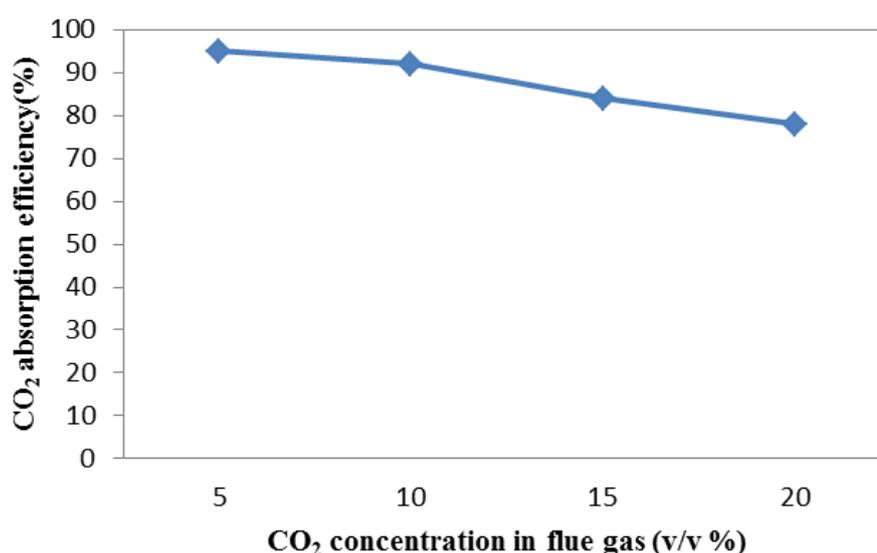


Figure 6

Effect of CO₂ concentration on absorption efficiency.

3.1.4. Flue gases flow rate

In this part, the flow rate of flue gas changes from 2-5 m³/hr, and the other operating condition are the same as the basic operation conditions. As can be seen from Figure 7, in the flue gas flow rate change scope, the CO₂ absorption efficiency decreased when the flue gas flow rate increased. Increasing the flue gas flow rate is equivalent to reducing the gas liquid two phase residence time in the absorption column so the reaction rate of CO₂ absorption slowed down (Kohl and Nielson, 1997). It was also observed that the increasing flue gas flow rate carried spray absorbent out from the tower, and led to the loss of the absorbent. Therefore, an increase in the flue gas flow rate would lead to a drop in the CO₂ absorption efficiency.

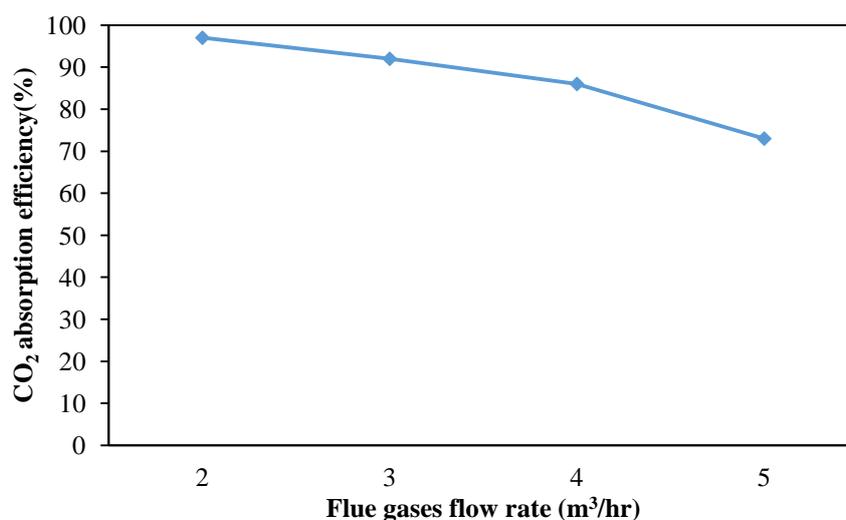


Figure7

Effect of flue gas flow rate on absorption efficiency.

3.1.5. Effect of temperature on absorption efficiency

In general, temperature is an important parameter influencing reaction kinetics. For CO₂ absorption kinetics, the reaction rate constant is normally based on the Arrhenius expression:

$$k = A e^{-\frac{E_a}{RT}} \quad \text{or} \quad \ln k = -\frac{E_a}{RT} + \ln A \quad (1)$$

where,

k = chemical reaction rate

A = pre exponential factor

E_a = activation energy

R = gas constant

T = temperature in kelvin

This indicates that the reaction rate constant is temperature dependent and that elevated temperatures lead to a greater reaction rate constant, k . It was observed that too high temperature caused efficiency deterioration. The effect of CO₂ concentration in flue gas on absorption efficiency is shown in Figure 8. When the temperature of the liquid solvent was raised from 30 to 60 °C, the mass transfer coefficient increased. Nevertheless, when the temperature was further increased to 70°C, the absorption efficiency dropped. This was due to the thermodynamics of the exothermic CO₂ absorption system which could cause reversible reactions when the temperature was too high (Vibeke et al., 2012). The increase in temperature could also increase the CO₂ vapor pressure over the solution which leads to decrease of the physical solubility of CO₂ in the solvent. Hence there is a need to strike a balance between physical solubility and reaction kinetics while increasing temperature to enhance the absorption efficiency. The maximum efficiency was observed at 50 °C for this liquid solvent.

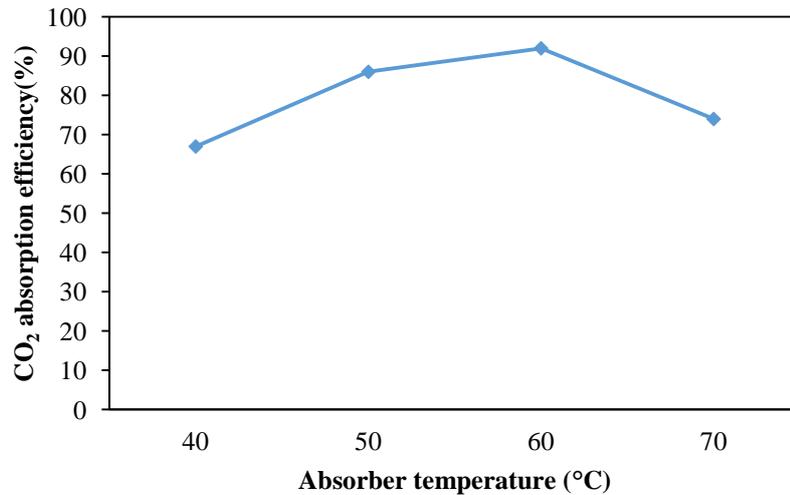


Figure 8

Effect of temperature on absorption efficiency.

3.2. CO₂ solubility comparisons of AIT600 solvent with MEA solution

CO₂ solubility obtained in this work is compared to the literature values of MEA solvent as a base in this section. Figure 9 shows that AIT600 has larger distances between the equilibrium curves than MEA according to data obtained by Neda (2012). Hence for AIT600 lower optimal solvent flow rates as well as somewhat lower regeneration energies compared to MEA can be expected. It must be considered that these expectations from equilibrium data, which are discussed above, do not take into account the mass transfer limitations. Slow mechanism leads to a need for high column and/or efficient packing.

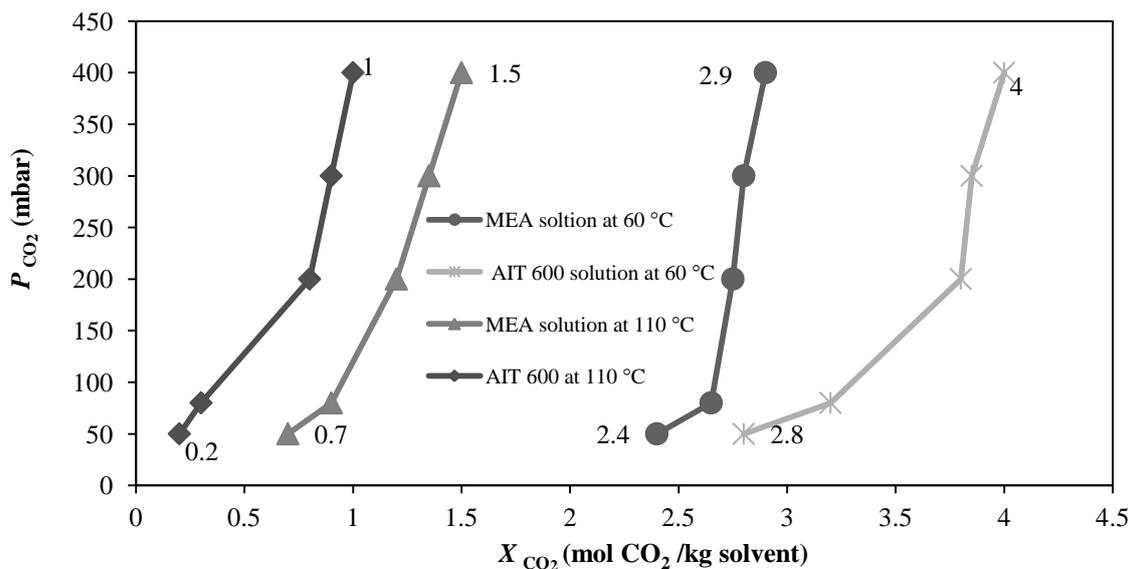


Figure 9

Calculated equilibrium data of CO₂ solubility at 60 °C and 110 °C for MEA in comparison with experimental data for AIT 600.

3.3. Determination of overall absorption rate of CO₂ into solvent

The specific rate of chemical absorption of component A into a reactant B can be expressed based on the two-film theory as:

$$N_A = K_G P (y - y^*) \quad (2)$$

where, N_A is the specific rate of absorption A into a reactant B, K_G is the overall mass transfer coefficient, P stands for the system pressure in kPa , y represents the mole fraction of the component A in the gas bulk, and y^* is the mole fraction of component A in equilibrium with the bulk liquid. When used for chemical absorption, y^* is assumed to be zero since the chemical reaction is fast. Thus:

$$N_A = K_G P_{CO_2} \quad (3)$$

where, the bulk gas partial pressure (P_{CO_2}) is represented as the logarithmic average of the inlet and outlet partial pressures.

$$P_{CO_2} = \frac{P_{CO_2,in} - P_{CO_2,out}}{\ln \left(\frac{P_{CO_2,in}}{P_{CO_2,out}} \right)} \quad (4)$$

The overall mass transfer coefficient K_G is based on the individual gas and liquid phase mass transfer coefficients.

$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{H}{\beta k_L} \quad (5)$$

where H , is the Henry's law coefficient, β is the enhancement factor, k_L and k_G are the liquid side mass transfer coefficients without chemical reactions and the gas-side mass transfer coefficient respectively. $\frac{1}{K_G}$, $\frac{1}{k_G}$ and $\frac{H}{\beta k_L}$ can be considered as the total mass transfer resistance, the mass transfer resistance in the gas phase and the mass transfer resistance in the liquid phase, respectively.

In a gas-absorption apparatus such as a packed column, the effective gas-liquid interfacial area (a_V) is considered as another important parameter in the mass transfer process in addition to the mass transfer coefficients, and it is very difficult to determine the effective gas-liquid interfacial area (a_V). Thus it is more practical to determine the volumetric overall mass transfer coefficients ($K_G a_V$) and the overall absorption rate (Φ). Then, the overall absorption rate is defined as the specific absorption flux per unit volume of the liquid phase. Finally, the overall absorption rate (Φ) can be described as:

$$\Phi = K_G a_V P_{CO_2} \quad (6)$$

$$\frac{1}{K_G a_V} = \frac{1}{k_G a_V} + \frac{H}{\beta k_L a_V} \quad (7)$$

Then, the expression of Φ can be obtained as follows:

$$\Phi = \frac{q_G (Y_1 - Y_2)}{V_r} \quad (8)$$

where, Y_1 is the mole fraction of CO₂ in the gas phase at the inlet of the absorber; Y_2 is the mole fraction of CO₂ in the gas phase at the outlet of the absorber, and V_r stands for the volume; q_G is the molar gas flow rate.

The effects of several operating parameters such as the operating temperature, the aqueous solvent flow rate, the aqueous solvent concentration, the total gas flow rate, and the CO₂ inlet concentration on the overall absorption rate of CO₂ were studied. Then, the overall absorption rate was determined by Equation 8.

3.3.1. Effect of temperature

Temperature is one of the important parameters in reaction kinetics. Figure 10 shows the profile of the overall absorption rate under different temperatures in the packed column. In these cases, the aqueous solvent concentration was 30 wt.%, the total gas flow rate was 2 m³/hr, the inlet CO₂ concentration was 15% (v/v), and the aqueous solvent flow rate was 200 ml/min. Increasing the temperature of the absorber column results in a shift of the Φ value. The overall absorption rate increases with the increasing temperature when the temperature is lower than 60 °C. Such behavior is attributable to the nature of the CO₂ absorption kinetics. The reaction rate constant of CO₂-amine based system increases with the reaction temperature. It is beneficial to enhance the absorption rate. However, when the temperature is higher than 60 °C, the overall absorption rate decreases by the increasing temperature. This phenomenon can be attributed to the reaction of CO₂ and the fact that the reaction is reversible.

According to the principle of molecular dynamics, the rate constant of reaction and the diffusion coefficient increase with the increase in the temperature, and this is beneficial to improve the absorption rate. Nevertheless, the CO₂ solubility coefficient decreases with the increase in the temperature. This gives rise to an increase in the mass transfer resistance in the liquid phase. Furthermore, the increase in the temperature also accelerates the decomposition of the reaction product of carbamate, which gives rise to the reduction of the absorption efficiency.

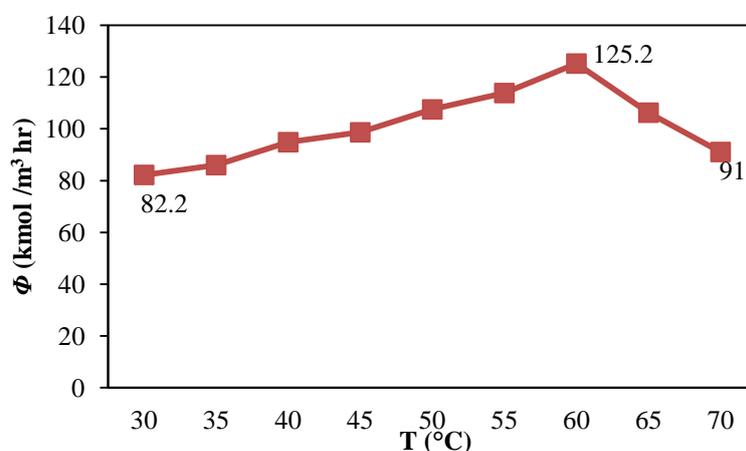


Figure 10

Effect of temperature on the overall absorption rate in the packed column. The total gas flow rate was 2 m³/hr, and the inlet CO₂ concentration was 15% (v/v).

3.3.2. Effect of liquid flow rate

The effect of the liquid flow rate on the overall absorption rate was investigated. In these cases, the gas flow rate was 2 m³/hr, the concentration of CO₂ at the inlet was 15% and the temperature was 60 °C.

Figure 11 shows that the overall absorption rate increases with the increase in the liquid flow rate. According to the two-film theory, the increase in the liquid flow rate leads to a higher liquid side mass

transfer coefficient (k_L), which directly results in the increase in the overall mass transfer coefficient (K_G) according to Equation 6. Besides, the interfacial area (a_V) generally increases with the liquid rate increasing in packed reactors of both random packing and structured packing. According to Equation 7, the absorption of CO_2 into an amine based solution is enhanced when the liquid flow rate increases.

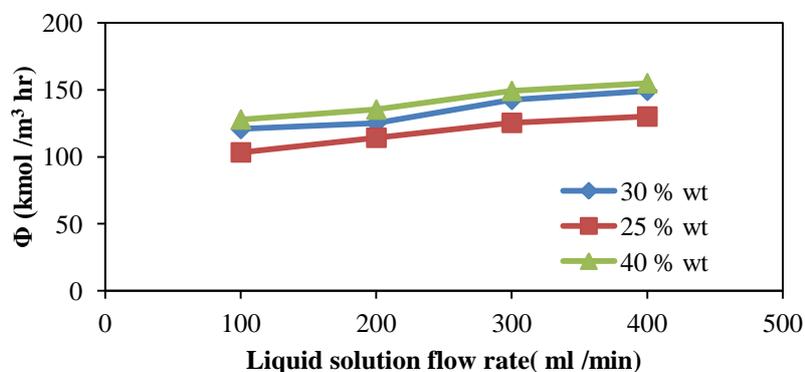


Figure 11

Effect of liquid solution flow rate on overall absorption rate of CO_2 in the packed column.

3.3.3 Effect of inlet CO_2 concentration

The effect of the CO_2 inlet concentration on the overall absorption rate was shown in Figure 12. In these cases, the operating temperature was 60°C , the solvent flow rate was 300 ml/min and the total gas flow rate was $2\text{ m}^3/\text{hr}$. Experimental results show that the Φ value increases rapidly as the CO_2 inlet concentration increases from 5% (v/v) to 20% (v/v). The increase in the CO_2 inlet concentration allows more CO_2 molecules to travel from gas bulk to the gas-liquid interface which is helpful for reducing the mass transfer resistance of the gas phase and enhancing the value of the bulk gas partial pressure (P_{CO_2}). According to Equation 7, the value of Φ is proportional to the bulk gas partial pressure (P_{CO_2}), thus the Φ value increases promptly as the CO_2 partial pressure increases. In order to further research the relationship between the Φ and the bulk gas partial pressure (P_{CO_2}), the variations of the Φ values are plotted versus the bulk gas partial pressure (P_{CO_2}) as shown in Figure 13. A linear relationship consequently leads to the following correlation:

$$K_{G aV} = \Phi / P_{\text{CO}_2} \quad (9)$$

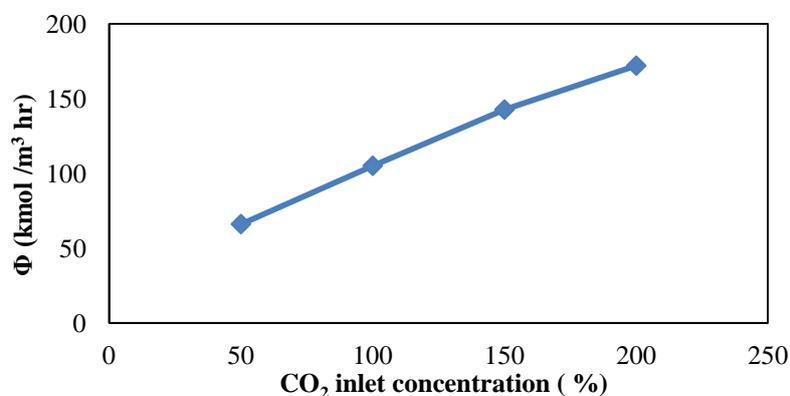


Figure 12

Effect of inlet CO_2 concentration on the overall absorption rate of CO_2 in the packed column; the operating temperature was 60°C , the aqueous solvent flow rate was 300 ml/min , and the total gas flow rate was $2\text{ m}^3/\text{hr}$.

Hence the value of $K_G a_V$ would be determined by calculating the slope of the straight line. The slopes of the straight lines in Figure 13 are 5.88. The volumetric overall mass transfer coefficient ($K_G a_V$) for this study is therefore 5.88 kmol/(m³.hr.mbar) in the 30 wt% AIT600 solution.

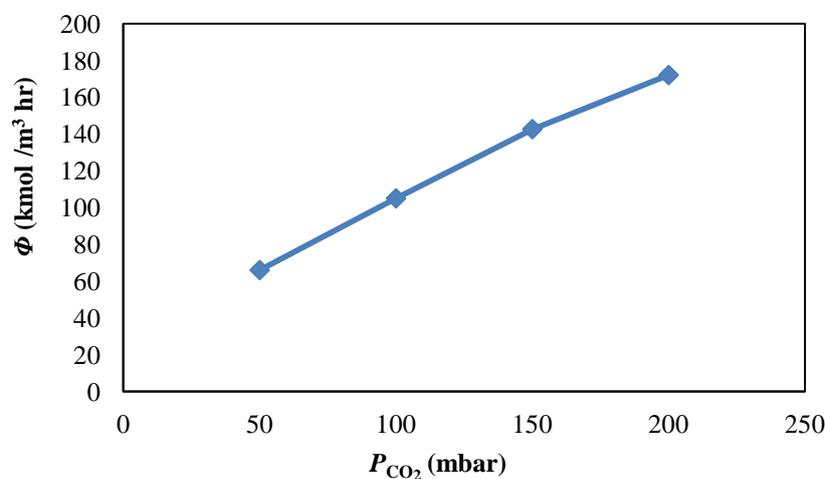


Figure 13

Variation of Φ with P_{CO_2} in the packed column; the operating temperature was 60 °C, the aqueous solvent flow rate was 300 ml/min, and the total gas flow rate was 2 m³/hr.

4. Conclusions

Various important operating factors affecting the CO₂ absorption efficiency were discussed in this paper. The experiments showed that the CO₂ removal efficiency increased with an increase in the liquid flow rate and solvent concentration, while the CO₂ mass transfer rate increased with an increase in the liquid flow rate, CO₂ volume fraction in the feed gas, solvent concentration, and the gas flow rate. The experimental studies on the overall absorption rate (Φ) of the absorption of CO₂ into aqueous solvent solution (AIT600) were also carried out. The experimental results show that the temperature plays an important role in the absorption of CO₂ into the aqueous solvent solution. The suitable manipulated temperature of the packed reactor is not supposed to beyond 60 °C.

The volumetric overall mass transfer coefficient ($K_G a_V$) determined by calculating the slope of the straight lines of the absorption rate against the bulk gas partial pressure was 5.88 kmol/(m³.hr.mbar).

It is hoped that with the continuous studies conducted worldwide currently, the challenges related to the absorption process can be overcome, and that CO₂ capture projects can be integrated and materialized in the industrial process.

Acknowledgments

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Nomenclature

A	: Pre-exponential factor
CCS	: Carbon capture and storage

<i>E_a</i>	: Activation energy
IEA	: International Energy Agency
IGCC	: Integrated coal gasification combined cycle
<i>K</i>	: Chemical reaction rate
L/G	: Liquid to gas ratio
MEA	: Mono-ethanol-amine
PCC	: Post-combustion carbon capture
<i>R</i>	: Gas constant
<i>T</i>	: Temperature in kelvin

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