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Effects of piperazine on carbon dioxide removal from natural gas using aqueous methyl diethanol amine

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ABSTRACT

CO_2 removal process by Amine absorption can be improved by adding an activator such as piperazine (a secondary amine) to the aqueous Methyl Diethanol Amine (MDEA) solution. The shuttle mechanism and the effect of Piperazine on CO_2 removal process have been studied and simulated through licensed simulation software, Unisim. The simulation was used to simulate amine absorption process for CO_2 removal and to study the effect of the process variables on CO_2 removal efficiency. An actual Process Flow Diagram (PFD) was built to simulate the absorption process based on an actual feed gas stream from Egyptian natural gas plant raw gas. The natural gas stream contained 10% CO_2 and is treated to reduce the CO_2 content to less than 1% by mole. CO_2 removal efficiency was investigated by changing process parameters, namely absorption process pressure and temperature, amine concentration and lean amine circulation rate. Piperazine addition, either on account of water or MDEA, increases the absorption efficiency but to a certain limit, when the reaction is no longer mass transfer limited. Temperature decrease improves absorption efficiency, unlike the common behavior of pure aqueous MDEA. Also, decreasing column pressure contributes in reducing CO_2 partial pressure in the feed gas and consequently decreases reaction rate with amine. Increasing circulation rate was found to increase the absorption efficiency till reaching the equilibrium.

1. Introduction

Carbon dioxide (CO_2) is a main source of corrosion and reduces heating value of natural gas. Besides, the contribution of carbon dioxide to global warming represents around 80% (Ratana, 2009). Therefore, it is required to completely or partially remove CO_2 before natural gas downstream processing or distribution. The bulk removal of carbon dioxide from gas streams is usually carried out via an absorption process operation (Derks, 2010a). CO_2 chemical absorption using an aqueous solution of amine-based absorbents is one of the most effective known methods (Muraia, 2013). Aqueous alkanolamines such as monoethanolamine (MEA), di-isopropanolamine (DIPA), 2-amino- 2-methyl-1-propanol (AMP), diethanolamine (DEA), and methyldiethanolamine (MDEA) are widely used for acid gases removal. Many amine mixtures were recently introduced to remove carbon dioxide with higher efficiency and at reduced expenses. The principle of mixing amines is to combine the favorable characteristics of different absorbents (Lu, 2011). The use of aqueous tertiary amine, such as MDEA, in gas treatment is popular for its H_2S removing high selectivity from streams containing H_2S, CO_2, and hydrocarbons (Xia, 2003). In general, the H_2S is thought to react almost instantaneously with MDEA by proton transfer as is the case with primary and secondary amines as illustrated in Eq. (1) (Kohl, 1999).

$$H_2S + R_2NH \xrightarrow{\text{Fast}} R_2NH_2^+ + HS^- \quad (1)$$

$$CO_2 + 2R_2NH \xrightarrow{\text{Fast}} R_2NH_2^+ + R_2NCOO^- \quad (2)$$

$$CO_2 + H_2O + R_2NH \xrightarrow{\text{Slow}} R_2NH_2^+ + HCO_3^- \quad (3)$$

$$R_2NCOO^- + CO_2 + H_2O \xrightarrow{\text{Slow}} R_2NH_2^+ + 2HCO_3^- \quad (4)$$

Eqs. (2)–(4) represent a summary of secondary amines reactions with CO_2. Eq. (2) is the predominant reaction and is faster than Eqs. (3) and (4) (Kohl, 1999). The drawback of this reaction is that two moles of amine are needed to be loaded by one mole of CO_2.

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Since MDEA is a tertiary amine and its Nitrogen atom does not have a hydrogen atom, the CO_2 reaction can only occur after the CO_2 dissolves in the water to form a bicarbonate ion. Then, an acid–base reaction occurs between the bicarbonate with the amine to yield the overall CO_2 reaction (Eq. (3)) (Kohl, 1999; Bullin and Polasek, 1990). The advantage of the tertiary amines over other amines is that the theoretical loading could reach 1 mol CO_2/mole amines (Kohl, 1999) as they undergo just to Eq. (3). However, the reaction is slow compared to the amine reaction with H_2S and as a result; tertiary amines are considered selective to H_2S.

It was found that MDEA is a superior amine as it is characterized by low corrosion rate and degradation rate resulting in an ability of using high solution concentrations (Polasek and Bullin, 2006). Also, the MDEA loading, theoretically, reaches 1 mol acid gas/mole amine (Kohl, 1999). All these features make it more attractive for CO_2 removal if the process could overcome the low reaction rate of MDEA with CO_2. CO_2 removal absorption process by MDEA shall be improved by adding an activator (Arkema Co., 2000) to the aqueous MDEA solution such as Piperazine (Pz). MDEA has an excellent capacity, but the absorption rate is limited by the slow bicarbonate forming reaction. Piperazine (Pz) is shown to be an effective promoter in MEA, DMEA, and potassium carbonate due to its rapid formation of carbamates with CO_2 (Tan, 2013). A blend of MDEA and Pz has the potential to combine the high capacity of MDEA with the attractive kinetics of Pz (Peter, 2014). Piperazine is a common absorbent, which is used principally as an active agent to activate ethanolamine solutions (Lu, 2014). Besides, there is solubility limitation for Pz in water, that's why Pz is usually used as the promoter to mix with other alkanolamines for the enhancement of reaction rate with CO_2 (Tan, 2013).

Piperazine has a high reaction rate towards CO_2 compared to other amines, as shown in Table 1. Aqueous solution of Piperazine-activated MDEA was disclosed and introduced to market by many leading companies such as BASF, Dow Chemical Company, UOP LLC, Huntsman Corporation and Shell (Peter, 2014). Piperazine acts as a shuttle that quickly reacts with CO_2 to reduce resistance to mass transfer and facilitates the movement into the liquid phase. It then passes on the CO_2 to react with an MDEA molecule. Piperazine is then available to react with another CO_2 molecule (Optimized Treating Inc., 2008).

Pz/MDEA blends absorb CO_2 faster than monoethanolamine (MEA) or diethanolamine (DEA) blends with MDEA at similar concentrations (Bishnoi, 2002).

Piperazine, is also used as an activator with other absorbents such as m hexamethylenediamine diaminobutane, bis(aminoethyl)ether, aminoethyl Piperazine, methylmonoethanolamine, and potassium carbonate. Also, it can be used a lot as the main absorbent (Ratana, 2009; Lu, 2011; Gary, 2013; Tim Cullinane, 2004; NETL, 2011). Piperazine is considered a promoter as being a cyclic diamine, each mole of Pz can theoretically absorb two moles of CO_2, and it may favor rapid formation of the carbamates (Bandyopadhyayb, 2011).

Lensen (2004) studied the reaction of Piperazine with MDEA as a trial to determine the order of reaction and he found that adding Piperazine to MDEA and increasing the temperature, regardless of MDEA concentration, will increase the reaction rate constant dramatically. That's why the blend combines the benefits of both Pz (high rate of reaction with CO_2) and MDEA (a low reaction enthalpy with CO_2) (Derks, 2010b).

Table 1
Reaction rate constants of amines toward absorption of CO_2 (Optimized Treating Inc., 2008).

Amine	Reaction rate constant ($L\ mol^{-1}s^{-1}$)
MEA	6000
DGA	4500
DEA	1300
DIPA	100
Piperazine	59,000
MMDEA	7100
MDEA	4

Lunsford (1996) studied the effect of temperature on mixed amine behavior, DEA and MDEA, and found that changing amine mixture concentration changes temperature effect on the absorption efficiency. He also found that for this amine mixture, every concentration has its optimum operating temperature.

CO_2 removal from Syngas using Piperazine/MDEA is also studied by Alvis et al. (2010) in their work, Alvis et al. showed the solvent temperature effect on CO_2 removal process. Increasing the temperature lowers the viscosity, and improves the CO_2 removal dramatically.

Alvis et al. worked with 45 wt. % MDEA plus 5 wt. % Piperazine solution. Treated gas CO_2 content droped rapidly with temperature but eventually experienced a precipitous rise. In this case, a temperature of just over 50 °C presents an operational cliff. As temperature rises, of course, the vapor pressure of CO_2 over the solvent goes up as well, and near 50 °C the point is reached where solubility reduced to prevent absorption of enough CO_2 even to come close to meet <1000 ppmv CO_2 specification. The CO_2% rises suddenly because of solvent's capacity which has been reduced to a level insufficient to absorb enough CO_2 (Alvis et al., 2010).

The following reactions represent the absorption of CO_2 by MDEA/Pz (Frederick, 2011; Bandyopadhyayb, 2011).

$$MDEA + CO_2 + H_2O \rightarrow MDEAH^+ + HCO^{3-} \tag{5}$$

$$Pz + CO_2 + MDEA \rightarrow MDEAH^+ + PzCOO^- \tag{6}$$

$$PzCOO^- + CO_2 + MDEA \rightarrow MDEAH^+ + Pz(COO)_2^{2-} \tag{7}$$

$$MDEA + PzH^+ \rightarrow MDEAH^+ + Pz \tag{8}$$

$$H^+PzCOO^- + MDEA \rightarrow MDEAH^+ + PzCOO^- \tag{9}$$

This piece of work aims to explore this technology by modeling the whole process on a simulator to study the effect of using piperazine as an activator on the absorption process in addition to the effect of different parameters on absorption efficiency. These parameters include the lean solution temperature, absorber pressure, lean solution composition and concentration and lean solution circulation rate. Studying these parameters will contribute to reach the optimum operating conditions for the removal process based on the tested Egyptian natural gas composition.

2. Modeling and simulation

Simulation software, Unisim, was used to simulate an actual CO_2 removal absorption process using a natural gas stream from Egypt contains 10% CO_2 to study the effect of Piperazine and operating conditions on absorption efficiency.

2.1. Simulator

Unisim Design Suite R400, provided by Honeywell Process Solutions (HPS), was used in this simulation. DBR amine package was used as a property package to estimate, properties, compatible thermodynamics and binary coefficient for the used components specially Piperazine which is not recognized by other packages (Aspen Tech, 2006).

2.2. Process flow diagram

An actual PFD was used to simulate the whole process of absorption, as illustrated in Fig. 1. The raw gas from the gas wells goes directly to an inlet separator then enters a knockout drum to ensure the removal of any liquid entrainment. The gas then enters the absorber with roughly the same conditions to go countercurrent the lean solution. Finally the gas passes through a water wash vessel to recover any Piperazine carryover because of the high volatility of Piperazine (Ralph and Hatcher, 2010). The rich solution then enters a flash vessel as a primary step to remove carbon dioxide and hydrocarbons by reducing the pressure to 5–10 bar. The rich solution is then heated by a heat exchanger with the lean solution out of the regeneration column. The rich solution then enters the regeneration then carbon dioxide is being removed through a high temperature and low pressure approximates vacuum. The regeneration column is equipped by a reflux condenser and bottom re-boiler. The lean solution then goes back to the absorber after passing through booster pump, heat exchanger, cooler and a main pump respectively.

2.3. Feed gas

The feed gas is an Egyptian natural gas stream that contains 10% CO_2. It was required to lower the CO_2 content to less than 1%. The actual operating conditions and composition of the raw gas are given in Table 2.

2.4. Base case

A base case was built and used to simulate the absorption and regeneration processes using customary operating parameters and solution composition to insure that the model is flawless. Consequently, these different parameters were changed to study their effects on absorption efficiency. The base case operating conditions and results are illustrated in Table 3. The power consumption for this case is calculated to be 47 MW.

2.5. Process parameters

CO_2 removal efficiency is affected by process operating parameters such as:

Table 2
Actual feed gas conditions and composition in molar basis.

Pressure, bar	95
Temperature, °C	60
Volume flow MMSCFD	128
Component	Mole fraction
Methane	0.838
Ethane	0.034
Propane	0.009
i-Butane	0.002
n-Butane	0.002
i-Pentane	0.002
Hexane	0.004
Heptane	0.002
Octane	0.000
Benzene	0.000
Toluene	0.002
H_2O	0.004
CO_2	0.100
H_2S	0.000
COS	0.000
CO	0.000
Nitrogen	0.002

1. Acid gas partial pressure.
2. Temperature.
3. Type of amine.
4. Amine concentration.
5. Circulation rate (Kohl, 1999).

More than 380 runs were made through the simulator and the base case by changing these parameters to reach the optimum operating conditions, which obey sales specifications with minimum operating cost. Table 4 illustrates the ranges of each process parameter which were tested.

3. Results and discussion

In this section, results obtained from the simulation model by studying the operating parameters are illustrated and discussed.

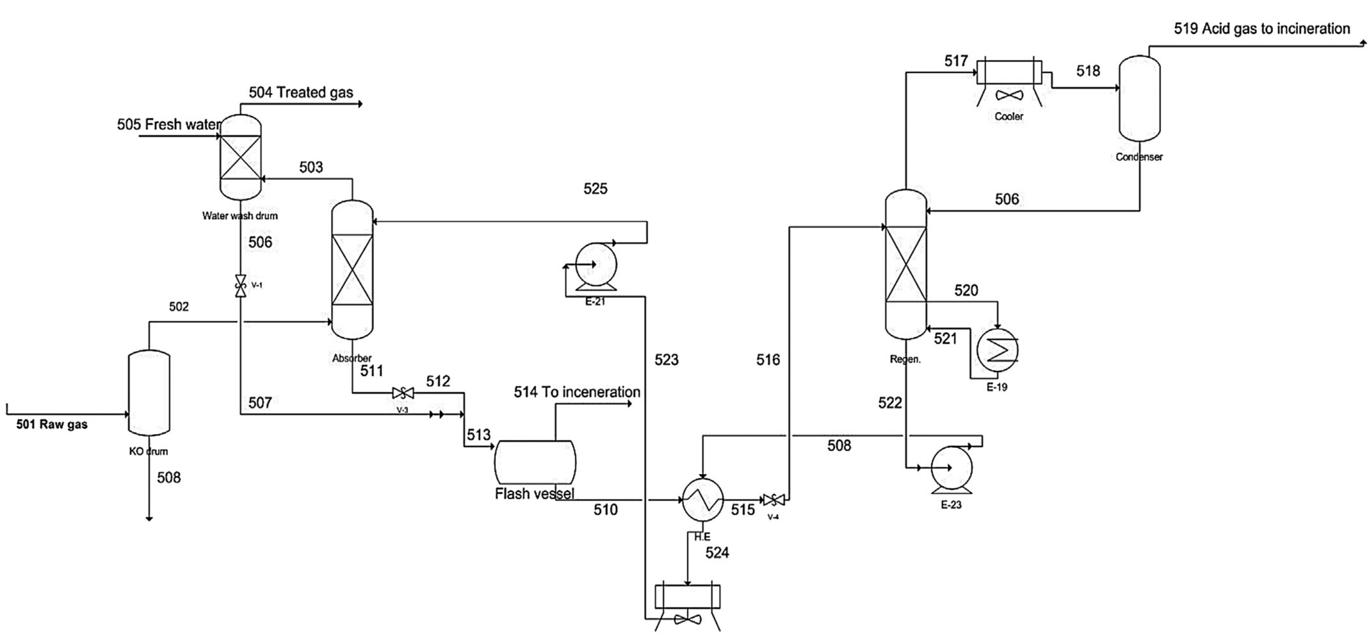

Fig. 1. A process flow diagram of amine absorption CO_2 removal unit.

Table 3
CO_2 removal process base case data.

		Lean solvent	Feed gas	Sales gas
Pressure, bar		100	95	94.5
Temperature, °C		68	60	72
Flow		343,000 kg/h	128 MMSCFD	116 MMSCFD
		Mass fraction	Mole fraction	Mole fraction
Composition	Methane	0.0000	0.8400	0.9266
	Ethane	0.0000	0.0340	0.0376
	H_2O	0.4976	0.0030	0.0051
	CO_2	0.0040	0.1002	0.0058
	MDEA	0.4287	0.0000	0.0000
	Piperazine	0.0698	0.0000	0.0000

3.1. Solution concentration

The effect of Lean solution with constant total amine concentration and replacing MDEA with piperazine (Pz) on the treated gas purity and on losses is plotted in Fig. 2 at four different temperatures (65, 70, 75, 80 °C).

Fig. 2 indicates that replacing MDEA with Pz increases absorption efficiency but this effect gradually disappears which confirms that there is enough Pz to handle the mass transfer step and any further increase in Pz concentration will not have any significant effect on CO_2 removal.

Changing piperazine concentration with constant MDEA concentration was also studied and it was found that adding more piperazine improves absorption efficiency dramatically as illustrated in Fig. 3. The addition of piperazine to fixed MDEA concentration reduces the equilibrium partial pressure of CO_2 (Bishnoi and Rochelle, 2002). The process is limited to 60% wt. amine concentration to avoid corrosion and evaporation losses (Kohl, 1999). The economic study for any actual case could put limits for this increase as Piperazine is more expensive than MDEA ($60/kg for MDEA vs. $80/kg for Piperazine) (Sigma-Aldrich Corporation, 2012).

3.2. Temperature

Increasing lean solution temperature will decrease absorption efficiency unlike pure MDEA, which efficiency increases with increasing temperature. Temperature increase will increase Piperazine carry over, decrease solution pH, decrease solubility, increase CO_2 equilibrium partial pressure and increase reaction rate, which greatly enhances the efficiency of CO_2 removal.

The effect of temperature on absorption efficiency is illustrated in Fig. 4. The starting point for plotting is 65 °C as the gas inlet temperature is 60 °C and it is recommended for lean solution to enter the tower not less than 5 °C above feed gas temperature (Arnold and Stewart, 1999; Mokhatab et al., 2006). As seen in Fig. 4, 3% of Pz is enough to obtain 1% CO_2 in sales gas and it is applicable to decrease lean solution circulation rate by increasing Pz concentration. Also, it was reported by Peter Thomoson that at low loadings and temperatures the absorption rate of CO_2 is limited by reaction kinetics; at high loadings and temperatures the diffusion of reactants and products is limiting (Peter, 2014). This was enhanced and justified by Alvis et al. as illustrated before.

Table 4
Process parameters ranges.

	From	To
Temperature, °C	65	80
Pressure, bar	20	95
MDEA, wt.%	36	45
Pz, wt.%	3	14
Flow rate, kg/h	245,000	390,000

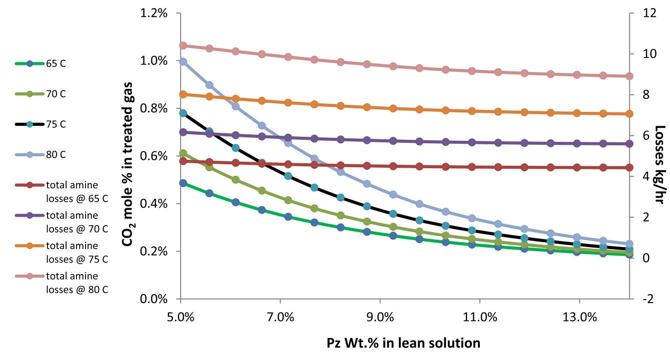

Fig. 2. Effect of replacing Pz with MDEA on total 50% amine in lean solution on CO_2 content in treated gas @ 1500 GPM (357,700 kg/h).

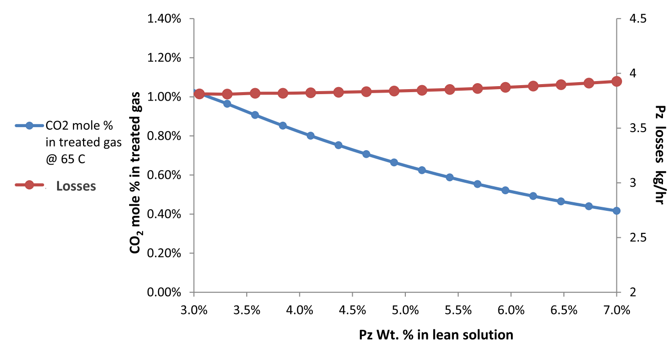

Fig. 3. Effect of Pz Wt. % on CO_2 in treated gas and Pz losses @ 65C Constant 38% MDEA @ 1500 GPM (357,700 kg/h).

3.3. Pressure

Lean solution pressure increase had no significant effect on absorber pressure and the gas pressure has the great contribution on determining absorber pressure. The pressure decrease led to absorption efficiency decrease, as predicted. Pressure reduction contributes in reducing CO_2 partial pressure in the feed gas and consequently decreases reaction rate with amine, which lowers the removal efficiency of CO_2. Losses, also, are affected by pressure but within a limited range. Losses deterioration within 3 kg/h. represents less than 0.02% of piperazine mass flow rate according to Fig. 5. By decreasing column pressure, Pz volatility increases. At the same time, decreasing pressure will decrease absorption rate which accompanied by a decrease in heat generated by the reaction. As a result, absorber top temperature decreases and Pz volatility decreases. This dual effect leads to the mutable performance of losses.

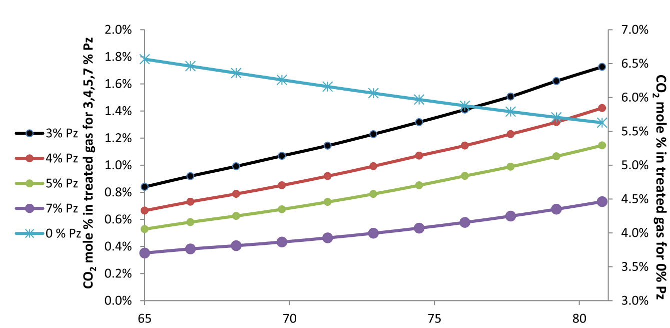

Fig. 4. Effect of temperature on CO_2 content in treated gas with fixed 45% Wt. MDEA @ 1500 GPM (357,700 kg/h).

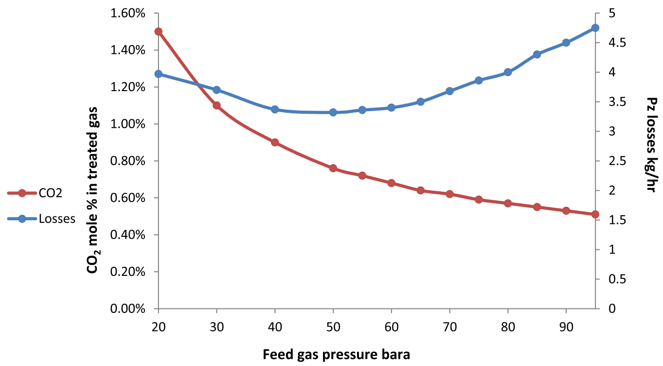

Fig. 5. Effect of feed gas pressure on CO_2 content in treated gas with fixed 45% Wt. MDEA and 5% Pz @ 1500 GPM (357,700 kg/h) with Pz losses.

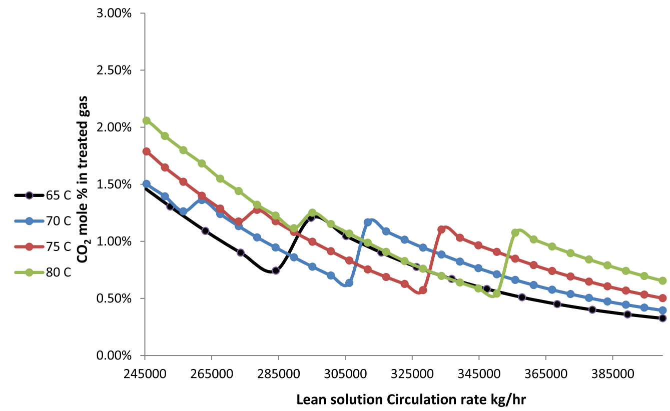

Fig. 7. Effect of lean solution circulation rate on CO_2 content in treated gas 45% MDEA, 5% Pz.

3.4. Circulation rate

Lean solution circulation rate is the most important factor in the economics of gas treating with chemical solvents. It influences the size of pumps, piping, heat exchangers and regeneration tower, thus has a large effect on the capital cost of gas treating plants. Circulation rate also influences the energy requirement for solvent regeneration because the reboiler heat duty is affected directly with liquid rate. In general, increasing circulation rate increases the volumetric mass transfer coefficient as illustrated in Fig. 6 (Chung-Sung, 2006), the greater the circulation rates of the solvent, the greater the amount of acid gas contaminants that can be removed. However when the point where sufficient solvent is added to allow the treated gas leaving the column to be in equilibrium with the lean solvent entering the column, a further increase in solvent rate will not reduce the level of contaminants in the treated gas. This fact was strengthened in the simulation and could be illustrated in Fig. 7. This conclusion was also enhanced by four temperatures plotted and compared (65, 70, 75, 80 °C).

On the other hand, the effect of Pz on amines absorption efficiency is reported by Chung in Fig. 6. The single amines have the lowest mass transfer rates.

The performance deviation found in Fig. 7 was investigated and interpreted as a disorder due to software calculation errors.

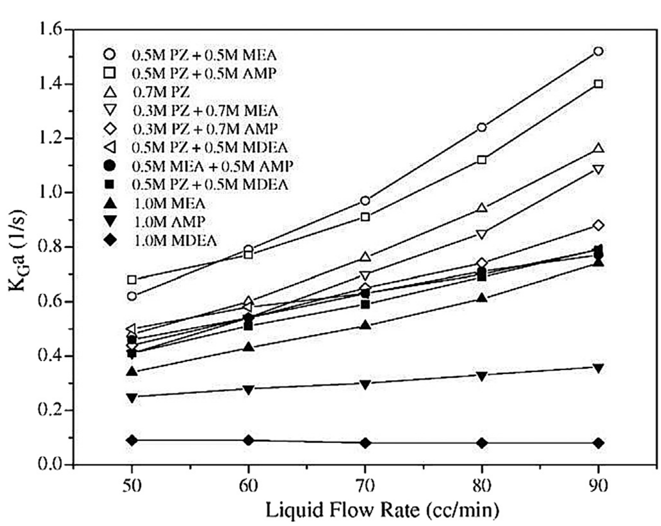

Fig. 6. Effect of circulation rate on mass transfer coefficient.

4. Conclusion

Aqueous solution of Piperazine activated MDEA was disclosed and introduced to market by many leading companies like BASF, Dow Chemical Company, and Shell. 3 percent of Piperazine has the ability of decreasing CO_2 in treated gas to less than 1% which cannot be reached by MDEA solution itself. Adding Piperazine either on account of water or MDEA will increase absorption efficiency but to a certain limit where the reaction is no longer mass transfer limited. Temperature increase will decrease absorption efficiency, unlike the behavior of pure MDEA solution and that is mainly because of Piperazine existence which makes the absorption process mass transfer limited instead of reaction limited, unlike absorption processes using MDEA or DIPA which are reaction limited and increasing temperature will increase CO_2 absorption efficiency (Lunsford, 1996). For instance, increasing temperature by 10 °C could increase CO_2 in treated gas up to 0.5% decreases gradually with increasing Pz concentration especially in low concentrations of Pz (i.e. 3%) as illustrated in Fig. 4. Column pressure decrease contributes in reducing CO_2 partial pressure in the feed gas and consequently decreases reaction rate with amine. However, the effect is not that significant as decreasing pressure from 95 bar to 20 bar will increase CO_2 percent by 1%. Circulation rate has a great economic concern in this process as it contributes in equipment design, operating power consumption and solution concentration needed to remove CO_2 to 1% in sales gas. Increasing circulation rate increases absorption efficiency as predicted till reaching the equilibrium so it is possible to compensate low concentration with increasing circulation rate which should be economically studied.

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