N$_2$-bubbling Method for O$_2$ Removal from the Loaded MEA Solution in the CO$_2$ Capture Process

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Abstract

Flue gas stream contains full-oxidized (carbon dioxide), partially oxidized hydrocarbons, and some extent of oxygen. Carbon dioxide can be separated by CO$_2$ capture and separation (CCS) process using monoethanolamine (MEA). In this process, CO$_2$ accompanied with O$_2$ can be absorbed by the solution (MEA+H$_2$O). Dissolved molecular oxygen may explicitly participate in the cathodic reaction and generate sever corrosion problems, or it may induce amine degradation via irreversible oxidative reaction, reducing amine efficiency. In this research, the method of N$_2$-bubbling was investigated for deoxygenation of loaded aqueous MEA solution on a laboratory scale. N$_2$-bubbling set-up was simulated with a known volume glass vessel in which bubbling of N$_2$ with an available flow rate was applied through the loaded amine solution. A mathematical formula was developed to correlate the N$_2$-bubbling flow rate, F, the volume of amine solution in the bubbling column, V, and time taking the oxygen concentration decreases from saturated O$_2$-solution to less than 0.5 mg/L.

Keywords: CO$_2$ Capture, De-oxygenation, Dissolved Oxygen, Monoethanolamine Solution, N$_2$-Bubbling.

Introduction

Oxidative and thermal carbamate polymerization degradations of alkanolamine solutions are two crucial sources for the removal of monoethanolamine during the absorption/desorption cycling process. These two degradation processes are responsible for the increase in corrosion and foaming; at last, they decrease amine efficiency decrease. Thermal carbamate polymerizations are major at temperatures higher than 100° C in which monoethanol carbamate as a central substance is imposed to turn into oxazolidine. Oxazolidine repeatedly can take part in the same process and produce dimer and even polymer [1-6]. Moreover, some oxidative substances may get alkanolamines to participate in unpleasant processes. To exemplify, oxygen, active radicals [1,2], some metal ions [7-11], sulfur oxides [12-13], and nitrogen [14-16] are some main substances responsible for chain irreversible reactions. Oxidation degradation eliminates solvent components in gas processing, and creates other species, such as heat stable salts, which can

increase the corrosion rate [17]. Dissolved oxygen may directly participate in the cathodic reaction and intensify corrosion issues as well.

In the CO$_2$ capture process, due to imperfect fuel oxidation, the existence of oxygen in the gas feed is inevitable. To address this problem, there might be several chemical and physical methods. In the chemical method, some reagents may be added to the solvents, but in physical methods, no reaction is included in the deoxygenation process. Some physical deoxygenation methods include nitrogen bubbling, degassing by vacuum, thermal degassing, and degassing using a membrane [18-22]. Lausier et al. [19] utilized a nitrogen powered permeator and reached a reduction in *DO* from 8.37 to 0.7 ppm with one pass and down to 0.12 ppm with an another one, in fact, they reported that dissolved oxygen levels were reduced greater than 90% with a single pass through the permeator and were reduced more than 98% with a double pass. In other work presented by Polyanskii et al. [21], the dissolved oxygen reduced down to less than

0.01 ppm using a sorption membrane. Butler et al. [22] compared four typical methods including; boiling at 1 atm, boiling under reduced pressure, purging with nitrogen, and sonication under reduced pressure. They concluded that, although none of the methods used for comparison lead to total removal of oxygen, however, the method of nitrogen purging can achieve the residual DO down to 0.2-0.4 ppm after 2-40 minutes nitrogen purging at flow rate 25 mL/second, so it may be very effective and simple technique and the method of boiling at 1 atm was found to be the least effective.

In the present work, two possible scenarios exist for selecting an applicable method during the cycling operation in the CO_2 capture process. 1) adding an appropriate inhibitor / oxygen scavenger [23-27] in the solvent formulation. Using inhibitors as a chemical method comes with some problems, such as low efficiency and side effects due to the creation of byproducts. Meantime the amount of oxygen scavenger required for loaded solvent depends upon the amount of oxygen dissolved in the solution, temperature, solvent composition and rate of reaction.

1- Control of all these contributing factors within the operational condition may be a complicated task.

2- Employing a physical process such as N_2-bubbling [8] just after the absorption tower. The problem with this conventional method is that the deoxygenation may not meet a lower ppb level. The reason for this problem may be related to the low mass transfer, the inadequate contact between gas and liquid phase and distribution of bubble size, which none of them are the interest issues in this work. N_2-bubbling seems to be an appropriate technique in which it may achieve an economical, rapid and simple deoxygenation procedure [28] for loaded monoethanolamine just after the absorption tower. Generally, carbon dioxide capture and separation units are used in fossil fuels power plants instead of sending gases to the chimney. On the other hand, due to the fact that the absorption of small amounts of oxygen for amine solutions, especially monoethanolamine, causes its degradation, as a result, the use

of nitrogen flow right after the absorption tower (contactor) can help in reducing the destruction of amine.

Considering the temperature of the absorption tower being about 40 – 60 °C, in this work, the loaded MEA (20 wt%) solution (loading = 0.35 mol CO_2/mol MEA) at the absorption temperature ($T = 50$ °C) was simulated in laboratory scale. The flow rate of N_2-bubbling trial was from (35 to 360) mL/min and two different solution holdups, which is an indicator for batch size, were designed. It is worth noting that MEA (20 wt %) may be regarded as a benchmark in CO_2 capture processing. The aim is to obtain a correlation between the bubbling time taking O_2-riched saturated solution to reach to less than 0.5 milligram per L and F/V, in which F is the flow rate of nitrogen and V is solution holdup. Therefore, regarding a particular residential time of the loaded solution in the known volume of the bubbling column, one reaches an optimum flow rate of N_2.

Materials and Methods

Materials

Deionized water was degassed in an ultrasonic bath (FUNGILAB, model UA10MFD) at temperatures of 353.15 K and wave frequency of 50 kHz about half an hour before use. The amount of water impurity was quantified by conductivity measurement. The total amount of water impurities was recognized as less than 10 ppm.

In Table 1 the required official documents including the chemical abstract service (CAS) registry number, purity, analysis method for purity and source of preparation of the chemicals were summarized. Calibrated balance (Mettler model AE 200 with a standard deviation of ± 0.001 g) was employed to weigh and prepare the solution. The dissolved oxygen concentration in the loaded MEA solution was measured by IP67 COMBO D.O. METER 8603 with temperature resolution and accuracy of 0.1 and ±0.5 °C and DO resolution and accuracy of 0.1 and ±0.3% (reported by supplier).

Table 1 Specifications and sources of chemicals were used in this work.

Chemical name	Molecular formula/ Relative molar mass g.mol^{-1}	CAS registry number	Purity	Analysis method	Source
Distilled water has been deionized twice	H$_2$O/18.015	[7732-18-5]	The total dissolved substance (TDS) < 10 ppm[1]	Conductometer[2]	ION EXCHANGE RESIN (RIPI), and MILLIPORE S.A.S., 67120 MOLSHEIM, FRANCE
Carbon dioxide	CO$_2$/44.01	[124-38-9]	99. 5 mol%	GC[3]	ROHAM GAS COMPANY
Oxygen	O$_2$/15.999	[7782-44-7]			
Nitrogen	N$_2$ / 14.007	[7727-37-9]	99.999 mol%	GC	FARAFAN GAS
MonoEthanolamine	MEA / 61.08	[141-43-5]	> 99 wt%	GC	MERCK

1. The total dissolved substance (TDS) has been measured by conductivity method <10 ppm (Mettler Toledo).

2. The total amount of impurities in water was obtained less than 10 ppm by conductivity measurement.

3. The gas chromatography and its information are given by the manufacturer.

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IP67 COMBO is waterproof instrument and auto temperature and manually altitude compensation (maximum operating temperature is 50 °C) with three-functional purposes pH meter / Conductometer and dissolved oxygen meter. It is switchable among three parameters pH/ Cond./*DO* detected in LCD display. *DO* probe is just plugged into the meter and the electrolyte solution is refilled. *DO* can be red and switch between *DO* in mass percent and in mg per L (ppm). This instrument needs to be calibrated before each experiment. Calibration will be attained by holding the *DO* probe in the air and waiting for few minutes until the reading on LCD gets stabilized. After a couple of seconds when the reading is stable, the calibration gets finished by pressing the ENTER.

Sample Preparation

Monoethanolamine is loaded with CO_2 via the isochoric saturation method (ICS). The details of the ICS method are accessible in other works [29-34]. This method bases on measuring the gas pressure swing and volume of the gas container. KELLER model PA-33X pressure transmitter (0 to 3) MPa within 0.01% of full scale was employed as pressure sensor.

The Vacuum Pump BS 5000-11 Type: BS2208 A21042003 (up to 0.1 kPa) was hired to make the cell vacuum. Then, a known quantity of MEA 20 wt% solution was introduced to the cell, and then a quantified CO_2 was brought to meet the MEA solution in the equilibrium cell. The amounts of CO_2 injected into the equilibrium cell were calculated with accurate PVT data obtained from the National Institute of Standards and Technology (NIST) for pure CO_2 [35].

$$n_{CO_2} = \frac{V_{gc}}{RT_a}\left(\frac{P_i}{Z_i} - \frac{P_f}{Z_f}\right) \qquad (1)$$

where V_{gc}, Z_i and Z_f are the volume of the gas container, the compressibility factors associated to the initial and final state in the gas container, respectively, and T_a is the ambient temperature. Owing to this fact, the whole CO_2 injected into the cell would enter the liquid phase; the CO_2 loading in the liquid phase was obtained as:

$$\alpha_{CO_2} = \frac{n_{CO_2}^l \ / \ mol}{n_{MEA} \ / \ mol} \qquad (2)$$

in which, $n_{CO_2}^l$ and n_{MEA} are the amount of acid gas and amine in the liquid phase, respectively. Pressure swings would be adjusted in a way that the loading of the solution was obtained ∼ 0.35 (the value of loading just at the end of the absorption tower). The loaded solution quenches under the atmosphere temperature and is prepared for the interest test.

It's worth noting that, the loading of amine solutions with the ICS method has been widely carried out in the open literature, and the accuracy of this method has been approved by scientific societies in the references [29 – 34]. In Azam Najaflo's PhD thesis [36], Some methods of measuring the solubility of carbon dioxide in amine solutions at low loading were considered from the point of view of accuracy. They carried out their experiments at two temperatures of 313.15 and 348.15 K with four different methods, i.e., ICS, potentiometry, chemical method and gas chromatography. On the base of their results, the average error percentage for all data in temperature 313.15 K in the ICS method equal to 1.57, in the gas phase analysis method equal to 3.06, in the chemical method 22.7 and in the potentiometric method was obtained 65.37. Average error percentage for all data in temperature 348.15 K in the static method equal to 2.76, in the gas phase analysis method equal to 10.7 in the chemical method 21.26 and 29.42 was obtained in the potentiometric method.

N_2-bubbling experiments for O_2 removal may make CO_2 removal or even evaporation of the amine solution which results in concentration change in MEA solution, in addition to change in CO_2 loading. The concentration of the MEA solution as well as CO_2 loading were remeasured respectively by gas chromatography and Orsat Gas Analysis (OGA) (https://www.sciencedirect.com/topics/engineering/orsat). In the OGA, a known amount of loaded-amine solution introduced into a vessel which is connected to a leveling bottle. The vessel contains slightly was acidulated by concentrated acid sulfuric with a trace of chemical indicator (typically methyl orange) to liberate gas from the absorption media. The liberated gas transfer to the leveling bottle to enable readings to be taken at constant pressure. The loading amount of 20% monoethanolamine solution was subjected to Orsat test before and after the nitrogen-bubbling tests, and the deviation values of loading were about 1% compared to the initial amount obtained by ICS method. In the meantime, the composition of the CO_2 – loaded MEA 20 wt% solution was checked by GC analyses before and after the N_2- bubbling and the chromatograms show that the N_2- bubbling have no prominent effect on compositions of solutions.

Methods

In the N_2-bubbling process, an experimental system was developed using double-layer cylindrical glass. The ceramic fine bubble sparger was installed at the lower part of the glass column and connected to the regulated N_2 and O_2 capsules. The sparger diameter is 8 cm, made from sintered glass with a porosity of 40-100 μm (grade 2). It can make tiny bubbles and efficiently introduces gas into the liquid phase, and even facilitates the gas mass transfer through the liquid phase. Furthermore, the other advantages of the sparger are to increase gas-liquid contact area and to enhance the rate of diffusion as well as to homogenize gas distribution through the liquid. The system's temperature was fixed by a water recirculation bath (PMT Tamson model T 2500) at the temperature of 323.15 K and was measured by a Lutron model TM-917 digital thermometer with a 0.01 K resolution using a Pt-100 sensor as the temperature indicator.

The diagram of the apparatus is shown in Fig. 1. Bubbling glass columns with two constant volumes (1900 and 700 mL) and constant cross-section (diameter of 6.8 cm) were employed to consider the solution holdup. Flow rates of N_2 were adjusted from (35 to 360) mL/min by mass flow controller (MFC) (Brooks instrument 3903 AZ- the Netherlands, N_2 S/N=F13056-005 with the flow range of 500 mL/min and operating pressure of 70-67 bar). To make O_2-saturated amine solution, the interest solution was bubbled with an adjusted flow rate of pure O_2 (the MFC used for this purpose is Brooks instrument 3903 AZ- the Netherlands, O_2 S/N=F13052-050 with flow range 333 mL/min and operating pressure of 50-47 bar).
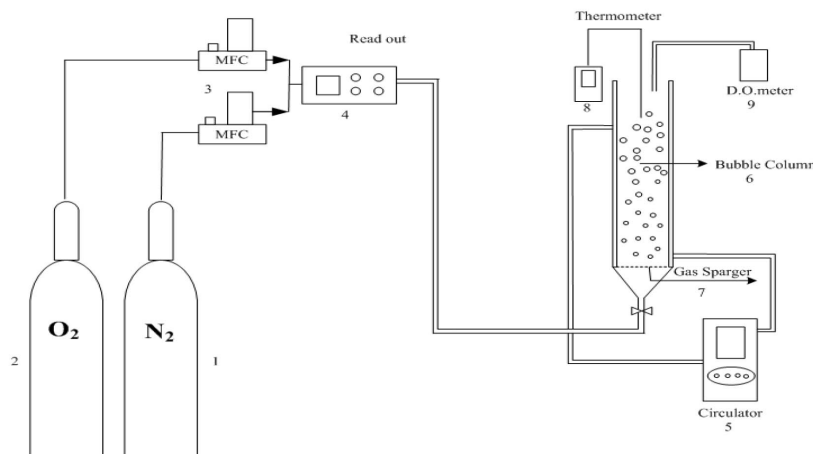
Fig. 1 Schematic diagram of lab scale N_2-bubbling apparatus.

The glass column was carefully washed and blow-dried then a known amount of CO_2-loaded solution (1900 and 700 mL) was poured into the glass column. First, the pipeline at the button of the column was connected to the O_2 cylinder. With a proper flow of O_2, the solution was saturated with O_2, and then the oxygen valve became closed and the line connected to the N_2 cylinder with an interest flow rate. The chronometer started with valve opening located just below the column and kept track of time within the test. At different times, *DO* drops are indicated by the *DO* meter in the middle of the column.

Results and Discussion

To show the effluence of the N_2-bubbling on the O_2 removal, three experimental trials were carried out. 1) A CO_2-loaded MEA 20 wt% was bubbled with O_2 to be saturated. It was left in the atmosphere temperature, and its O_2 concentrations were traced by DO-METER. 2) A CO_2-loaded MEA 20 wt% (loading = 0.12) was bubbled with O_2 to be saturated, and then via the N_2-bubbling process (flow rate of 100 mL/min), its O_2 concentrations were traced by DO-METER. 3) To see the effect of CO_2-loading on the O2 removal the second test was repeated with unloaded MEA 20 wt%. Fig. 2 shows the quality of the N2 bubbling operation in which, MEA 20 wt% solution was exposed to N_2-bubbling and as can be seen, the dissolved O_2 is remarkably removed from the solution in comparison with the case of non-bubbling operation. As it is shown in Fig. 2, at the atmosphere temperature and flow rate of 100 mL/min, N_2-bubbling in 15 minutes reduced the O_2 concentration of loaded and unloaded MEA to about 0.2 and 0.4 of the initial one, respectively, and the O_2 concentration in the case of without N_2-bubbling would not be remarkably reduced during a long time.

Oxygen solubility in electrolyte solution is less than that in non-electrolyte one and that is the reason why O_2 is removed easier in loaded MEA solution in comparison to unloaded one. In the loaded solution, due to strong reaction between the dissolve CO_2 and MEA, the solution undergoes speciation and ionization, which causes the ionic strength of the solution to increase, and this, in turn, causes a sharp decrease in molecular species such as oxygen. At the next step, loaded MEA solution (loading~0.35) at the constant temperature of T=323.15 K was experienced a different flow (from 30 to 360 mL/min), and the time needs *DO* value of solution reached to 0.5 mg per liter (L) was measured. To study the effect of volume of loaded solutions in the cylindrical holdup, the experiment was repeated with two series of initial amount of solution (700 and 1900 mL). In Figs. 3 to 4, the effect of the flow rate of N2-bubbling on *DO* for two series experiments was presented. As can be seen in both cases, the more the flow rate value is, the more oxygen removal attains.
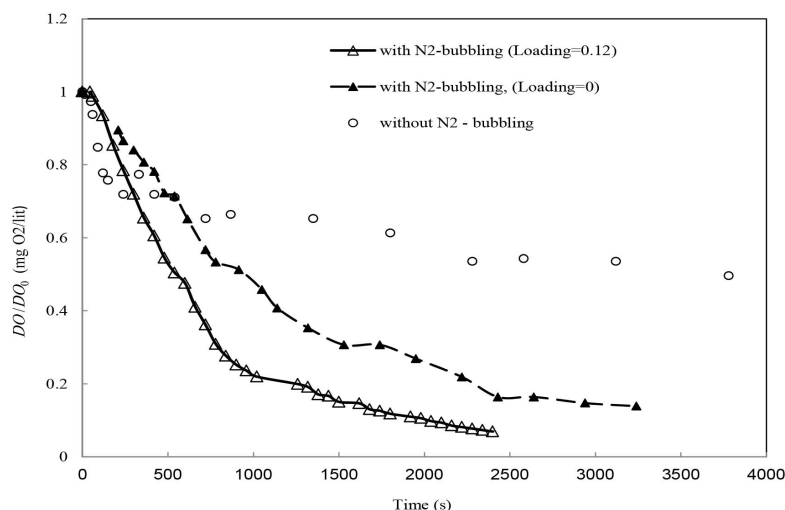

Fig. 2 The effect of CO_2 loading and N_2-bubbling on the O_2 removal (loading = 0.12, flow rate of nitrogen=100 cc/min, T=26 °C, volume of solution=300 cc).
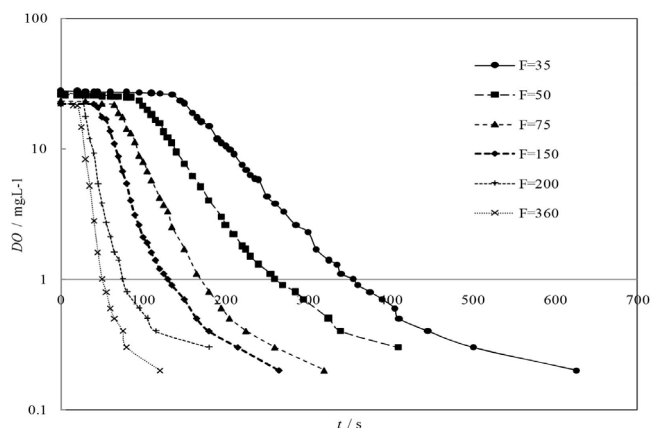
Fig. 3 Dissolved O_2 (mg·l⁻¹) versus time of N_2-bubbling at those given flow rate ($V = 700$ cm³, loading = 0.35 mol CO_2/mol MEA, h = 22.5 cm, d = 6.8 cm, T = 50 °C).

Fig. 4 Dissolved O_2 (mg·l⁻¹) versus time of N_2-bubbling at those given flow rate ($V = 1900$ cm³, loading = 0.35 mol CO_2/mol MEA, h = 54.0 cm, d=6.8 cm, T = 50 °C).

In Fig. 5, the time of N_2 bubbling (time lasts from DO=O_2-saturated to DO=0.5 mg per L) was depicted in terms of the value of nitrogen flow rate for two systems (with the V = 700 and 1900 mL for loaded solution) and also in Table 2 those values are numerically listed. The more the amounts of solution are, the more contact time is required to reach the DO = 0.5 mg per L. In other words, t is proportional to F where proportionality factor is dependent on the loaded solution volume. In Fig. 6, it was shown that the time of the N_2 bubbling for two loaded solution roughly does not depend upon the value of V/F. Fig. 7 shows the best correlation between the quantity of V/F for amine solution and the time of the O_2 removal down to the value of 0.5
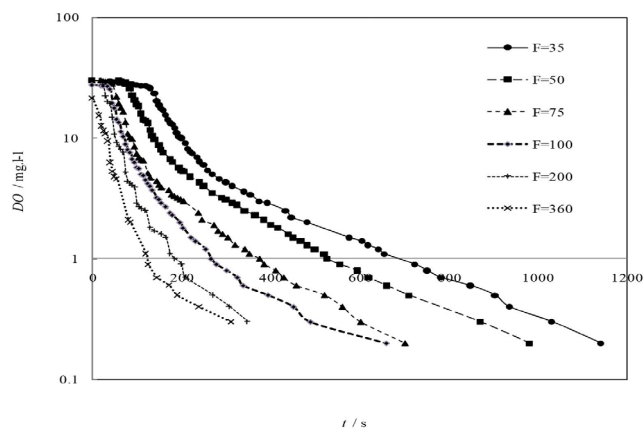
mg per L. As depicted, the third-order polynomial equation with a correlation coefficient of 0.995 was fitted through the experimental values,

$$t\left(DO=0.5\right)=0.001593\cdot\left(V/F\right)^3-0.211415\cdot\left(V/F\right)^2+22.845\cdot\left(V/F\right)^2+36.198$$

(3)

Equation 3 explicitly shows that, by using the volume of loaded solution (V) in the cylindrical volume of holdup and the flow of bubbling (F), one may be able to obtain the required time of bubbling for achieving appropriate O_2 concentration in solution. It should be noted that the height of loaded solution in the holdup depends on the amount of solution.

Table 2 Influence of N_2 flow rate and volume of amine on O_2 removal.

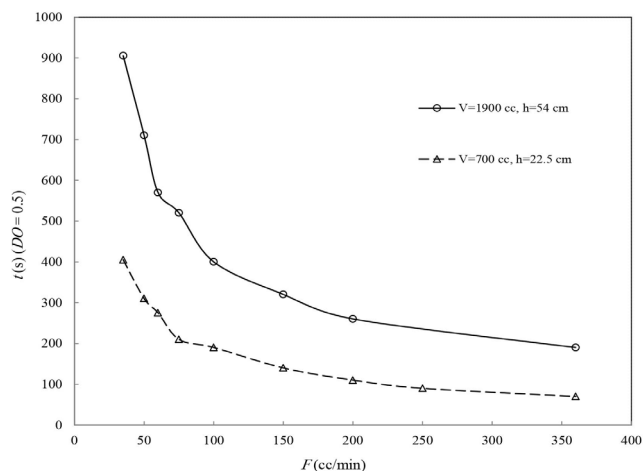| V (cm³)	h (cm)	d (cm)	T / K	F (cc/min)	V/F	t /s (DO/DO_0 = 0.5)
1900	54	6.8	323.15	35	54.29	905
				50	38.00	710
				60	31.67	570
				75	25.33	520
				100	19.00	400
				150	12.67	320
				200	9.50	250
				360	5.28	175
700	22.5	6.8	323.15	35	20.00	405
				50	14.00	310
				60	11.67	275
				75	9.33	210
				100	7.00	190
				150	4.67	140
				200	3.50	110
				250	2.80	90
				360	1.94	70

Fig. 5 Influence of N_2 flow rate and volume (700 cc and 1900 cc) of amine on O_2 removal (time of N_2 bubbling at which the concentration of O_2 in solution reaches down to 0.5 mg per liter). *h* is the height of solution in glass column.
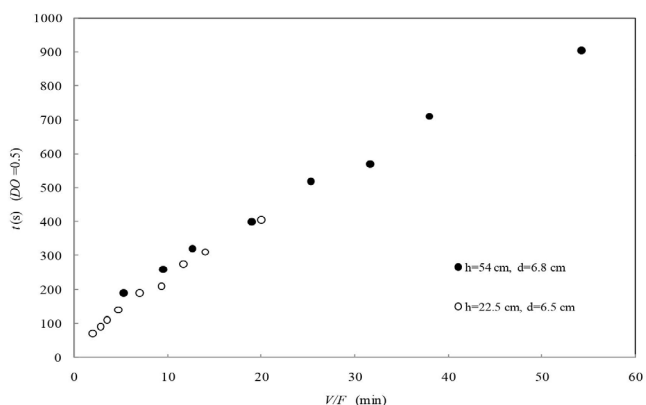

Fig. 6 Influence of *V/F* of amine solution on O_2 removal. *h* and *d* are the height of solution in glass column and inner diameter of glass column.
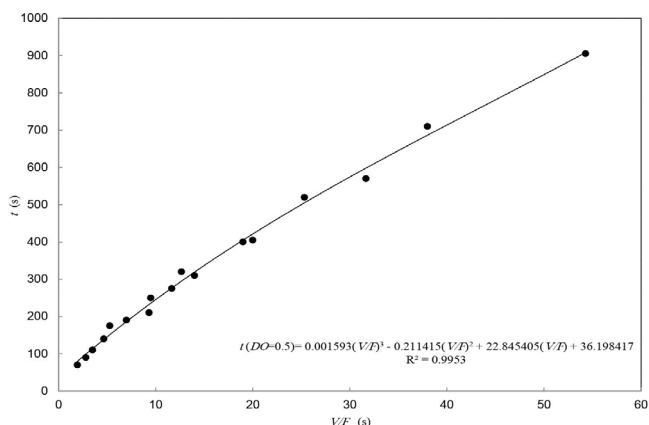

Fig. 7 The best correlation relationship between the quantity of *V/F* for amine solution and time of O_2 removal.

Conclusions

This work investigated the quality and quantity of the N_2-bubbling method for O_2 removal from the loaded MEA solution in the CO_2 capture process. To obtain a correlation between N_2-bubbling flow rate, F, the volume of amine solution, V, in cylindrical holdup, and time of N_2-bubbling, t, MEA solution was saturated with oxygen and then exposed

to N_2 bubbling with different flow rate from (30 to 360) mL/min. Time takes the dissolved oxygen of saturated solutions to fall to 0.5 mg/L was measured for different flow rates, *F*, and two values of the volume of loaded solutions, *V* or h (height of loaded solution in cylindrical holdup). Tests conducted on the apparatus revealed that:

1. N_2 bubbling is a sound method to reduce the O_2 concentration in a loaded MEA solution in the CCS process of the flare stream.

2. The efficiency of deoxygenation is enhanced by raising the N_2 bubbling flow rate and decreasing the volume of solution in the holdup. Efficiency is enhanced by designing a tall processing column.

3. Regardless of the value of the N_2-bubbling flow rate and the amount of solution in a holdup, bubbling time requires the value of *DO* to fall from saturated O_2 to the value of 0.5 mg per L depending on the value of *V/F*.

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