Performance comparison of ultrasonic-assisted and magnetic stirred absorption methods for CO₂ separation

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Abstract

Chemical absorption is the most matured and preferred separation process which is extensively used for CO₂ removal from natural gas. The current contactor systems used in the absorption process suffer from several drawbacks including excessive footprint, operating, and maintenance issues. Ultrasonic irradiation is a new alternative technique to assist the CO₂ absorption process without the aforementioned limitations. Thus, the aim of this paper is elucidating the potential of the ultrasonic-assisted CO₂ absorption system. To achieve this, the performance of the ultrasonic-assisted system was compared to that of the conventional stirring method. Two different solvents with dissimilar reaction mechanisms were chosen. The first part of the experiments was conducted in the ultrasonic-assisted batch vessel, while the second part was accomplished in the stirred batch cell. The parameters including ultrasonic power, ultrasonic frequency, stirring speed, and initial feed pressure were 18 W, 1.7 MHz, 500 rpm, and 11 bar, respectively. Besides, the operating temperature and the concentration were chosen based on the standard operating condition for each solvent. The mass transfer coefficient was calculated using the dynamic pressure-step method. The results revealed that in comparison with the stirring method, the ultrasonic-assisted absorption system significantly enhanced the CO₂ absorption process at similar operating conditions. By using ultrasonic irradiation, the volumetric absorption coefficient increased almost three times for MDEA and nearly six times for MEA. In the latter, this improvement can be related to the physical effect of the ultrasound. However, for the slow kinetic solvents, this improvement might be attributed to the chemical effect of the ultrasound.

Keywords Absorption · Stirring method · Ultrasonic irradiation · Sonophysical effect · Sonochemical effect

1 Introduction

The demand for natural gas is expected to rise by more than 60% until 2040 since it has less environmental issues among the other types of fossil fuels [1, 2]. Depending on the geological condition of the reservoir, natural gas contains a wide range of methane with heavier hydrocarbons such as ethane, propane, isobutene, normal butane, and significant amount of contaminating compounds including CO₂, H₂S, and CO [3].

Meanwhile, it is highly necessary to remove these impurities from the natural gas in order to increase the calorific

value, meet the gas pipelines specifications, optimize the operating and capital costs, environmental purposes, or even to raise the selling price [4–6].

Therefore, the removal of these acid gases is an essential and inevitable process during the sweetening of natural gas which can be considered as a steppingstone to the continuation of using this source of energy in various applications. There are some widely adopted processes to treat the natural gas, namely adsorption [7, 8], membrane [9, 10], cryogenic [11], and absorption [12, 13].

Up to the present, absorption is the most matured and preferred separation process which is extensively used for

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CO₂ removal from the natural gas. In many industrial processes, absorption has been extensively applied within the bulk of the material via a chemical or physical interaction [14].

The physical absorption is generally based on the solubility of CO₂ in the solvent instead of a chemical reaction with it. In other words, the interaction between CO₂ and the solvent is by nonchemical surface forces, which is Van der Waals interaction [15, 16]. In comparison with the physical absorption, chemical absorption (or reactive absorption) is defined as a process in which gas is absorbed by the liquid phase, with a combination of the chemical reaction and absorptive mass transport [17]. During this process, chemical absorbents react with CO₂, and covalent bond forms among the molecules. In the next step, the CO₂-rich absorbent regenerates using thermal regeneration and the captured CO₂ steam is released and then compressed for the subsequent transportation and storage [6, 7].

Typical solvents such as amine-based systems, carbonate-based systems, aqueous ammonia, and sodium hydroxide along with the existing contactor systems, including spray tower, bubble column, packed bed column, mechanical agitator, and membrane have been developed for intensifying the mass transfer of the absorption process [13, 18–20]. Good compatibility, maturity, and high efficiency are the main advantages of the chemical absorption process that make it the most promising and commercially used process for large-scale CO₂ capture [21]. However, significant energy requirement due to solvent regeneration, solvent loss, equipment corrosion, and large absorption column volume is considered as the main dilemma of this process [22, 23]. Since these drawbacks have posed major technical challenges for CO₂ capture, there is still a growing demand for the development of alternative technologies. The new technology must have high CO₂ removal capacity, low energy consumption, better-contacting equipment, smaller size, and lower cost.

Ultrasonic irradiation has several applications in different research areas including food technology [24, 25], plastic or metal welding and cleaning [26, 27], medical treatment [28], synthesis of nanomaterial and biomaterial [29], or enhancement of multiphase reactions due to its physical and chemical effects [30, 31]. However, with such favorable applications, it is found that using ultrasound in gas separation processes, especially the absorption process, is limited [32]. Hence, the main objective of this work is to elucidate the potential of using ultrasonic irradiation in the CO₂ absorption process by comparing its performance with the stirring method which is a conventional tool used to study the reaction and mass transfer kinetics in gas–liquid systems. The outcome of the current work is

a guide for the proper solvent selection to investigate the physical or chemical effects of the ultrasonic irradiation.

2 Principles of the ultrasonic assisted absorption

Generally, CO₂ absorption involves the vapor–liquid mass transfer, liquid–liquid mass transfer, and liquid chemical reaction. Meanwhile, CO₂ transportation from the vapor phase into the liquid solvent is dependent on the vapor–liquid interfacial area and the vapor diffusivity. Then, the absorbed CO₂ in the solvent surface is then diffused into liquid by liquid diffusivity and the chemical reaction takes place with CO₂ in the liquid [19, 33]. Using high-frequency ultrasonic irradiation technique in the CO₂ absorption process empowers the process because of its sonochemical and sonophysical effects.

Sonophysical effects entail microstreaming, fountain formation, and atomization. Propagation of the ultrasonic wave through the liquid and vapor phase generates a small oscillation motion of the fluid around the fluid mean position known as microstreaming. Microstreaming phenomenon is able to generate turbulence within the fluid to enhance the mass transfer. Fountain formation, which forms under a sufficient ultrasonic power, is capable for enhancing the mass transfer process.

In addition, the gas–liquid interfacial area is forced to oscillate until pinching off into droplets by applying sufficient ultrasonic intensity. As a result, the generated fine liquid droplet provides a large surface area for the mass transfer process. This phenomenon is known as “ultrasonic atomization.” It is noteworthy that the higher frequency can generate smaller droplets which can increase the surface area and consequently affect the mass transfer process [34–37]. On the other hand, the most basic concept of sonochemical effect is free radicals generation (especially hydroxyl radicals), which emerge as a result of the cavitation or bubble formation and enhances the chemical kinetic reaction rate. The implosion of cavities, which are produced by ultrasonic irradiation, establishes an unusual environment for chemical reactions. The pressure and temperature in the imploding cavity can be several hundred atmospheres and several thousand degrees Kelvin. These high pressures and temperatures provide the activation energy needed for the chemical bond breakage and thus result in the production of highly reactive free radicals and various other species. The free radicals can react rapidly to form new molecules and other radical species or even diffuse into the liquid bulk and affect the chemical reaction and the mass transfer process [38, 39]. Figure 1 shows the schematic diagram of the physical and the chemical

Fig. 1 Schematic diagram of physical and chemical effects created by high-frequency ultrasound [32]

effects of the ultrasonic irradiation during the CO_2 absorption process.

Different gasses have been removed using ultrasonic technique; however, there are limited reports on the CO_2 absorption by this method. Table 1 summarizes the ultrasonic-assisted mass transfer process for the absorption process.

3 Method and material

3.1 Material

To commence the experimental part of this study, the CO_2 (99.9%) was provided by Air Sdn. Bhd., Malaysia, and the chemicals including monoethanolamine (MEA, purity $\geq 99\%$) and methyldiethanolamine (MDEA, purity $\geq 99\%$) were supplied by Merck Sdn. Bhd., Malaysia.

All the selected chemicals were used directly without additional purification. The specifications provided by their suppliers are shown in Table 2. The aqueous MEA and MDEA solutions were prepared using double distilled water. The operating parameter ranges were chosen based on the standard operating condition for each solvent such as temperature (30 °C for MEA and 60 °C for MDEA) and solvent concentration (30 wt% for MEA and 50 wt% for MDEA).

Usually, the concentration of the MEA solution used in industrial processes is between 15 and 30%. For the MDEA aqueous solution, the widely used concentration is between 35 and 60 wt%. Besides, the most reported concentrations of MEA and MDEA for commercial processes are 30 wt% and 50 wt%, respectively [44–47]. Furthermore, chemical absorbent vapor pressure and thermal stability are also key parameters that can be affected by the operating temperature. Although higher temperature

Table 1 Ultrasonic-assisted system for the absorption process

Operation mode	Gas	Absorbent	Ultrasonic effect	Ultrasonic power	Ultrasonic frequency	Findings	Ref
Semi-batch	O_2	Water	Physical	600	22 kHz	Mass transfer coefficient increases with an increase in the power	[40]
Semi-batch	O_3	Water	Physical	260	20 kHz	The presence of ultrasound-enhanced ozone mass transfer rate	[41]
Batch	N_2	Water	Physical	62.6	20 kHz	Ultrasound greatly improves gas liquid mass transfer	[42]
Semi-batch	NO	Water	Chemical	110	20 kHz	Both physical and chemical effects are dominant in the mass transfer enhancement	[43]
Batch	CO_2	Water	Physical	18	1.7 MHz	The streaming force and fountain formation are the dominant factors for the absorption enhancement	[32]

Table 2 Specifications of the chemicals used in the current work

Name of chemical	Formula	Molecular wt (gr)	purity%	Method of purification	Supplier
Monoethanolamine	C ₂ H ₇ NO	61.08	≥ 99%	–	Merck
Methyldiethanolamine	C ₅ H ₁₃ NO ₂	119.16	≥ 99%	–	Merck
Distilled water	H ₂ O	18.015	99%	Distillation	–
Carbon dioxide	CO ₂	44.01	99.99%	–	Air product

1	CO ₂ gas tank	8	Pressure sensor
2	Pressure gauge	9	Ultrasonic power system
3	Back pressure regulator	10	Ultrasonic transducer
4	Mass flow controller	11	Temperature sensor
5	Compressor	12	Ultrasonic vessel
6	Water bath	13	Conductivity sensor
7	Gas storage vessel	V1, V2	Valve 1, Valve 2

Fig. 2 Ultrasonic-assisted absorption system schematic diagram [52]

may enhance chemical absorption (especially for the slow kinetic solvent), but if the solvent is easily volatile, its vapor may even penetrate into the gas phase and cause some problems. In addition, the solvent should have good thermal stability and be chemically stable within a wide temperature range, so that its thermal degradation can be avoided. Since MEA has higher vapor pressure and degradation tendency than MDEA, it cannot be used in high-temperature ranges [48–51].

4 Ultrasonic cell

The first part of the experiments was conducted in the stainless steel batch vessel, as shown in Fig. 2. The vessel had two main parts, including a top cover and a cylindrical body of 5 cm in diameter, 17.8 cm in height and a total volume of 250 ml. The top cover also had five different outlet ports including the temperature sensor, the pressure sensor, the gas inlet and outlet, and the conductivity probe. Since the formation of OH radicals would affect the conductivity, in situ conductivity measurement can be a

possible option to clarify the role of radical generated during the CO₂ absorption process.

The transducer that converts the electric power to ultrasonic irradiation, with the frequency of 1.7 MHz and a diameter of 1.5 cm, had to be mounted at the bottom of the reactor due to close contact with the chemical solution. In the previous work, the calorimetric method was used to measure the ultrasonic power [52]. Moreover, based on the limitation of the ultrasonic power system, the highest ultrasonic power is 18 W.

Then, 100 ml of various aqueous solutions with different concentrations were injected into the ultrasonic reactor. Because of the power dispersed by the ultrasonic irradiation, the temperature of the solution could be slightly increased; therefore, the reactor was settled in the water bath to constraint the temperature variation in the solvent.

Next, the CO₂ gas was then compressed to the gas storage tank. The adjustment of the position of valve 1 and 2 along with a back-pressure regulator enables us to pressurize and control the pressure of the ultrasonic vessel. The same procedure can be used for different solvents and different operating parameters.

5 Stirred cell

Magnetic stirred cells are widely used as conventional tools to investigate the reactions and kinetics of the mass transfer process in the gas–liquid systems. The stirred cell structure is simple which can be operated easily. In these cells, the reaction rate can be determined by pressure measurements [53].

The second part of the experiments was conducted in a stainless steel stirred batch cell. As shown in Fig. 3, the whole unit had a mixing vessel (MV) with a total volume of 3 L and four magnetic stirred cells (EC1–EC4) with a total volume of 50 ml. However, the volume of the stirred cell is less than that of the ultrasonic cell, but in both cells, it was tried to maintain the vapor-to-liquid ratio. The mixing vessel and also stirred cells were equipped with a digital thermometer and a digital pressure sensor. In addition, computer-integrated software was used to record data for all of the process parameters. The internal diameter, height, and the stirred cell volume were

Fig. 3 Schematic diagram of the stirred cells

30 mm, 70 mm, and 50 ml, respectively. An electric water bath was applied to control the stirred cells temperature.

First, the stirred cells were washed by using acetone and water, after that purged with the N_2 for a few minutes for removing any chemical traces or even unwanted gasses. Then, CO_2 gas was supplied to the mixing vessel and pressurized at the desired pressure by using an air-driven booster pump (2 to 25 bar). The stirred cells were filled with around 20 ml of the fresh aqueous solution. Once system achieved the chosen temperature and pressure, CO_2 was then transferred to the stirred cells from the mixing vessel. For increasing the mass transfer rate between the gas and the liquid phase, the magnetic stirrers under the stirred cells were switched on at a speed of 500 rpm. Concurrently, the time was recorded until the pressure in the ECs became zero. It is worth to note that only one of the stirred cells was used for the current work and the highest rpm at which the magnetic stir bar could rotate without jumping around was chosen.

6 Results & discussion

The potential of using ultrasonic-assisted CO_2 absorption system is evaluated by comparison of its performance with the conventional stirring method under batch process. The gas-to-liquid ratio was the same in both systems. Moreover, the absorption rate and, consequently, volumetric absorption coefficient were measured base on the pressure drop profiles using the following equation:

$$\dot{n}_{CO_2} = \frac{V}{ZRT} \frac{dp}{dt} \quad (1)$$

where V indicates the volume of the vapor in the reactor, T is the temperature, R is the real gas constant, and Z is the compressibility factor. In addition, for calculating the compressibility factor, the Peng–Robinson equation of state was used [54]:

$$P = \frac{RT}{V_m - b} - \frac{a}{V_m^2 + 2V_m b - b^2} \quad (2)$$

where R is the gas constant, T is temperature, P is pressure, and V_m is molar volume of the gas. The a and b can be calculated according to the following equations:

$$a = \left(0.457235 \frac{R^2 T_c^2}{P_c} \right) \alpha \quad (3)$$

$$b = 0.077796 \frac{RT_c}{P_c} \quad (4)$$

In Eq. (3), α can be calculated using:

$$\alpha = [1 + k(1 - T_r^{0.5})]^2 \quad (5)$$

and the k is a function of acentric factor only.

Considering all the above-mentioned equations, Z can be determined according to Eq. (6):

$$Z = \frac{V_m}{V_m - b} - \frac{a}{RTV_m^2 + 2RTb - \left(\frac{RTb^2}{V_m}\right)} \quad (6)$$

7 CO₂—MEA system

Comparison of the CO₂ pressure drop profiles among the ultrasonic-assisted absorption system and the conventional stirring system using an aqueous MEA solution is shown in Fig. 4. The initial pressure, the MEA concentration, and the temperature were 11 bar, 30wt%, and 303 K, respectively. The ultrasonic power was adjusted to 18 W, and the stirrer speed was 500 rpm. The power consumption for the current stirrer speed range is around 16 W, which is slightly lower than the ultrasonic-assisted system. In this study, the absorption rate using stirring method was not able to be increased using higher rotation speed of the stirrer.

According to Fig. 4, the pressure drop in ultrasonic-assisted absorption system with a single transducer was significantly faster than the magnetic stirring method with the same operating conditions.

Fig. 4 Comparison of the magnetic stirring method and ultrasonic-assisted absorption using 30% MEA

Table 3 Comparison of the absorption rates between ultrasonic and stirring methods using MEA

Applied method	Specifications	P _{CO₂} (kPa)	T(K)	Absorption rate * 10 ⁻³ mol.s ⁻¹	Volumetric absorption coefficient * 10 ⁻⁵ mol.pa ⁻¹ .m ⁻³ .s ⁻¹
Stirring	500 rpm	1100	303	0.054	0.098
Ultrasonic	18 W	1100	303	1.56	0.574

Table 3 shows the calculated CO₂ absorption rate and the volumetric absorption coefficient for both systems. According to the results, the volumetric absorption coefficient is nearly six times greater than the stirring method when the ultrasonic-assisted absorption system was used. Since MEA is a fast kinetic solvent by itself, this improvement can be attributed to the good mixing, which is created by the ultrasonic irradiation [55]. As aforementioned, a good mixing can be created by the ultrasonic physical effect including microstreaming, fountain formation, and atomization. This perfect mixing can enhance the gas–liquid interfacial area, improve mass transfer process, and subsequently lead to a higher absorption rate and a higher volumetric absorption coefficient [34–37].

8 CO₂—MDEA system

The interest in using MDEA has increased dramatically over the last decade. Low reaction enthalpy with CO₂, relatively high capacity, and low vapor pressure are MDEA main advantages over the widely used MEA. Other valuable properties of MDEA consist of a higher resistance to degradation and fewer corrosion problems compared to MEA. One of its main disadvantages, however, is that since it is a tertiary amine, it does not directly react with CO₂ [56, 57]. It is assumed that MDEA low reactivity to CO₂ might be affected by ultrasound irradiation.

Figure 5 shows the comparison of the CO₂ pressure drop profiles among the ultrasonic-assisted method and the conventional stirring method using 50% MDEA. The temperature, pressure, solvent concentration, ultrasonic power, and the stirrer speed were 333 K, 11 bar, 50wt%, 18 W, and 500 rpm, respectively.

According to Fig. 5, it can be seen that not only for the fast kinetic solvents like MEA, but even for the slow kinetic solvents such as MDEA, the ultrasonic-assisted absorption system can be seen as an alternative technique to assist the CO₂ separation process. As shown in Fig. 5, by using 50wt% of MDEA at 333 K, the pressure drop in ultrasonic-assisted absorption system was faster than the conventional stirring method. As summarized in Table 4, by applying the ultrasonic irradiation and the stirring method, following CO₂ absorption rates and volumetric absorption coefficients using 50wt% MDEA are obtained.

Fig. 5 Comparison of the magnetic stirring method and ultrasonic-assisted absorption using 50% MDEA

The volumetric absorption coefficient by using ultrasonic irradiation was nearly three times bigger than the stirring method, as shown in Table 4. In comparison with MEA, the less improvement can be related to this fact that the CO_2 reaction mechanism with a slow kinetic solvent, such as MDEA, is totally different, which is not greatly affected by the physical effects created by the ultrasonic irradiation. However, in this case, the presence of the ultrasonic chemical effect might be more effective and dominant. It is believed that the sonochemical effect is able to improve the kinetic rate of the chemical reaction by changing the reaction pathway.

Generally, the primary alkanolamines react with CO_2 reversibly and directly through the formation of the zwitterion intermediate, which is deprotonated by the bases present in the solution to form a stable carbamate. Carbamate formation increases the reaction rate. In contrast, tertiary alkanolamines do not react with CO_2 directly to form carbamates. In aqueous solutions, tertiary amines catalyze CO_2 hydrolysis to form bicarbonate ions and the protonated amine. The bicarbonate ions formation is relatively slow compared to the formation of carbamate ion; thus, the CO_2 removal kinetics by tertiary amines, such as MDEA, is mostly slower than that for primary amines like MEA [56]. It seems, due to the smaller molecular and highly

reactive radical species formation, the reaction pathway between CO_2 and MDEA is changed as a result of ultrasonic chemical effect. Besides, because of these changes, the reaction rate and, consequently, the mass transfer process are enhanced.

Compared with the research work conducted by Tay et al. [32], the current experimental results indicated that, in addition to the physical solvent, the ultrasonic irradiation is an efficient technique for assisting the absorption process using chemical solvents, as well.

9 Solvents performance comparison using magnetic stirrer

Figures 4 and 5 illustrate that besides the type of technology used, the applied solvent also has various impacts on the CO_2 absorption process. These impacts could be due to different reaction mechanisms and therefore various absorption regimes. Typically, the CO_2 pressure versus absorption rate profile is the base for the definition of the absorption regime.

The experimental CO_2 pressure drop profiles by using 500 rpm magnetic stirrer in the pressure range between 3 and 11 bar by using 30 wt% MEA at 303 K are demonstrated in Fig. 6a, while Fig. 6b presents the respective absorption rate.

Based on the results, despite pressure raise, the initial absorption rate has been enhanced slightly. This insignificant raise can be related to the instantaneous absorption regime in which the absorption rate is usually controlled by the transportation of CO_2 and MEA and effective surface area. Therefore, while using fast kinetic solvents like MEA, providing a good mixing which can be created by the magnetic stirrer or even ultrasonic irradiation can enhance the effective surface area and improve the absorption process. However, according to Fig. 7b, by using MDEA, the absorption rate has been proportional to the CO_2 pressure. The absorption is considered to be in the pseudo-first-order regime, because of the linearly raise of absorption rate by increasing CO_2 pressure. In this regime, the presence of excess reactant species is more than the CO_2 concentration. This mostly happens for the slow kinetic solvents and means chemical reaction is much slower than the CO_2 and reactant species diffusion. Therefore, for the

Table 4 Comparison of absorption rates between ultrasonic and stirring methods using MDEA

Applied method	Specification	P_{CO_2} (kPa)	T (K)	Absorption rate $\times 10^{-3} \text{ mol.s}^{-1}$	Volumetric absorption coefficient $\times 10^{-5} \text{ mol.pa}^{-1}$ $\text{.m}^{-3}.\text{s}^{-1}$
Stirring	500 rpm	1100	333	0.047	0.086
Ultrasonic	18 W	1100	333	0.667	0.248

Fig. 6 **a** Experimental pressure drop profiles, **b** experimental initial absorption rate under different initial pressure for 30 wt% MEA at 303 K

Fig. 7 **a** Experimental pressure drop profiles and **b** experimental initial absorption rate under different initial pressure for 50 wt% MDEA at 333 K

slow kinetic solvents which yet are not widely used in the industry, there is a chance of further study on the chemical reactions and their changes with the application of ultrasound and, especially, its chemical effect.

10 Conclusion

The main purpose of this experimental work was to elucidate the potential of using ultrasonic irradiation as an alternative option to assist the absorption process based on its chemical and physical effects which are still under

further development. In the current work, the ultrasonic-assisted absorption system performance was compared with the conventional stirring method. For this purpose, two different aqueous solutions of MEA and MDEA were examined under the batch process, and the CO₂ absorption process was analyzed according to the pressure drop profiles.

The results revealed that in comparison with the stirring method, the ultrasonic-assisted absorption system significantly enhanced the CO₂ absorption process at similar operating conditions. The comparison was performed based on the volumetric mass transfer coefficient which

increased nearly six times for MEA and almost three times for MDEA. According to the results, the improvement is highly dependent on the solvent properties and different absorption regimes. By using fast-kinetic solvents, this improvement can be resulted from the physical effect of the ultrasound. However, the possible existence of the sonochemical effect is guessed to be more dominant in the slow-kinetic solvents. Moreover, from the economical point of view, this enhancement might lead to a significant reduction in the column size with a similar absorption performance. At the end, it is believed that extensive research is required for commercializing this new technology.

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Compliance with ethical standards

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

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