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CO_2 Capture Using Amine-Based Solvents: Identification of Additives to Improve the Kinetics and Thermodynamics of CO_2 Sorption at High-Pressure Conditions

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Abstract: The increase in atmospheric CO_2 caused by human activities has driven the development of technologies to capture this gas before it reaches the atmosphere. This study analyzed CO_2 sorption using amine-based solvents, such as methyldiethanolamine (MDEA), diethylenetriamine (DETA), triethanolamine (TEA), and monoethanolamine (MEA) in 40 wt.% aqueous solutions, under high-pressure conditions (initial pressure: 500 psia) and room temperature (30 °C), in both non-stirred and stirred systems. Piperazine (PZ), a heterocyclic compound, was tested as an additive to improve the kinetics of the CO_2 sorption process. Kinetic and thermodynamic analyses were conducted to evaluate the efficiency of each amine-based solution in terms of reaction rate and CO_2 loading capacity. MEA and TEA exhibited higher reaction rates, while DETA and MDEA were the most thermodynamically efficient due to the highest CO_2 loading capacity. The PZ kinetic behavior depended on the equipment used; in the non-stirred system, no kinetic effect was observed, while in the stirred system, this effect was appreciable. Additionally, a corrosivity study revealed that MEA, a primary amine, was the most corrosive, whereas TEA, a tertiary amine, was the least corrosive.

Keywords: CO_2 capture; amines; MDEA; DETA; TEA; MEA; PZ; high pressure

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1. Introduction

Climate change is one of humanity's most significant challenges in the 21st century, with the accumulation of carbon dioxide (CO_2) in the atmosphere being a substantial driver of global warming [1]. A study on monitoring global carbon emissions in 2022 [2] reported that global CO_2 emissions increased by 1.5% in 2022 compared to 2021, following a more impactful rise of 7.9% and 2.0% relative to 2020 and 2019, respectively, bringing the total to 36.1 gigatons of CO_2 (36.1 $GtCO_2$). This increase consumed between 13% and 36% of the remaining carbon budget required to limit global warming to 1.5 °C, suggesting that the allowable emissions could be depleted within the next 2 to 7 years, with a 67% probability. The trend in increasing emissions has been linear for many years, which is why, in 2015, the United Nations launched the Sustainable Development Goals (SDGs) to eliminate poverty, protect the environment, and promote global peace and prosperity by 2030 [3,4]. Reducing CO_2 emissions is a crucial part of environmental protection goals, as it directly contributes to mitigating climate change, one of the most notable global challenges [5–7]. Reducing emissions by 45% by 2030 and achieving carbon neutrality by 2050 is essential to limit global warming to 1.5 °C, the threshold scientists consider necessary to avoid catastrophic consequences [8–10].

The urgent need to mitigate greenhouse gas emissions has fueled research into carbon capture and storage (CCS) technologies [11–15]. Within this context, CO_2 sorption has emerged as a promising strategy that enables the capture of this gas using sorbent materials that can be optimized for improved efficiency and capture capacity [16–19]. This technology is based on carbon dioxide capture from gas streams—natural gas, refinery gas, flue gas, fuel gas, or biogas—through interactions with chemical solvents, forming intermediate products via chemical reactions [20,21]. The chemical solvents include amines, amino acid salts, carbonates, ionic liquids, and ammonia [22–26]. This method is particularly well-suited for post-combustion CO_2 capture [14]. It is widely applied in various industrial settings, such as removing acid gases from natural gas and extracting CO_2 from reforming gases [27].

Amine-based solvents have gained significant attention in CO_2 sorption processes due to their unique ability to form reversible bonds with CO_2, making them highly effective chemical sorbents. With their tunable chemical structures, amines offer the potential to improve both absorption kinetics and CO_2 loading capacity, which are critical factors in the efficiency of carbon capture systems [22,28]. By studying various types of amines and how they interact with CO_2, researchers focus on identifying the best properties to optimize the carbon dioxide capture process [29]. Common solvents used in this process include alkanolamines such as monoethanolamine, diethanolamine, and methyl diethanolamine in aqueous solutions [16,30]. A standard reference for post-combustion CO_2 capture processes is based on a 30 wt.% aqueous monoethanolamine solution that usually reports 0.40–0.50 mmol of CO_2 removed/mmol of amine [31]. Although amine-based solvents for CO_2 capture are a mature technology, amines have challenges in capturing CO_2, which leads to chemical and thermal degradation that hinders their industrial application [24]. Since amines are exposed to high temperatures or oxide contaminants, they tend to degrade, reducing the CO_2 sorption efficiency. These processes require energy for solvent regeneration, which increases operating and maintenance costs in the implemented technologies, such as corrosion-resistant materials [32]. On the other hand, there are also reasons to continue with the research of amine-based solvents in the capture of CO_2 in which the efficiency of the processes can be improved, reduce degradation and toxicity, as well as create a hybrid or modified amine that is friendly to the environment [33]. Details related to the challenges in amine-based solvents used in CO_2 capture processes can be found elsewhere [34].

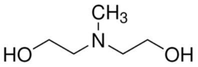
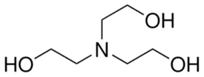
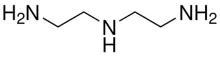
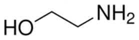
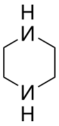
While it is true that current studies of CO_2 capture using amine-based solvents are popular, most of these are reported at low or atmospheric pressures, with few studies being carried out at high-pressure conditions [35–37]. This could be due to the limited availability of high-pressure equipment or the operational complexity of working and managing pressurized gases in low-scale laboratories. This experimental study aimed to investigate the role of some amines—methyldiethanolamine (MDEA), diethylenetriamine (DETA), triethanolamine (TEA), and monoethanolamine (MEA)—to identify the one with the best characteristics for the CO_2 sorption process, focusing on their impact on absorption kinetics and CO_2 loading capacity at high-pressure conditions (initial pressure: 500 psia) and room temperature, in both non-stirred and stirred systems. This will follow the detailed methodology outlined in Villarroel et al. [38] for the kinetic and thermodynamic analysis of high-pressure CO_2 sorption using ethylenediamine (EDA). Piperazine (PZ) was also tested as an additive to improve the kinetics of the CO_2 sorption process. Finally, the corrosion potential of amine-based solutions that have reacted with CO_2, industrially known as rich-amine solutions, was analyzed when in contact with wrought iron.

2. Materials and Methods

2.1. Amines

Table 1 summarizes the amines used in the study. All aqueous amine solutions were prepared using distilled water (conductivity: 0.5 µS/cm) at 40 wt.%. Piperazine was tested as a kinetic additive at 5 wt.% in addition to amine-based solutions. Alalaiwat et al. [39] reported that blends of MDEA + PZ have a synergistic effect on improving the CO_2 capture process.

Table 1. Amines used in this study.

Amine	Abbreviation	CAS Number	Molecule Structure	Purity (%)
Methyldiethanolamine	MDEA	105-59-9		>99
Triethanolamine	TEA	102-71-6		>99
Diethylenetriamine	DETA	111-40-0		>99
Monoethanolamine	MEA	141-43-5		>98
Piperazine *	PZ	110-85-0		>99

* Piperazine was tested as a kinetic additive.

2.2. CO_2 Sorption Tests: Experimental Equipment

Two different devices were used:

2.2.1. Non-Stirred Reactor

The Parr 4790 pressure vessel (Parr Instruments Co., Moline, IL, USA) is a batch reactor designed for handling corrosive substances under extreme pressure and temperature conditions, with a nominal capacity of 100 mL. It is made from a C-276 alloy, allowing it to operate at pressures up to 3000 psig and temperatures up to 225 °C in acidic environments. The reactor consists of a moving head and a sealed cylinder equipped with a pressure gauge, rupture disc, thermocouple, and three valves for gas release, liquid sampling, and gas injection. Its design ensures minimal exposure to hazardous materials [40].

2.2.2. Stirred Reactor

The Parr model 5100 batch reactor (Parr Instruments Co., Moline, IL, USA) is a 660 mL capacity vessel designed for reactions requiring stirring, with speeds ranging from 0 to 1700 rpm. It operates at pressures up to 1000 psig and temperatures up to 225 °C, thanks to its stainless steel construction, which makes it corrosion resistant. The reactor features valves for gas inlet/outlet, liquid sampling, and an internal thermocouple for precise temperature control. It also includes an O-ring and a split ring closure for secure sealing [41].

2.2.3. Experimental Setup

Figure 1 shows the experimental setup tuned up for the CO_2 sorption tests using the non-stirred and stirred reactor. The equipment is detailed below:

In CO_2 sorption processes using high-pressure systems, the CO_2 storage cylinder (A) supplied the gas necessary to carry out the said processes. During the process, the gas was regulated by a pressure valve or gas regulator (B), controlling the amount of CO_2 that entered the experimental setup to maintain optimal pressure conditions. Once the system was pressurized, the gas was injected into the reactor, which can be unstirred (C) or equipped with stirring (D), and it additionally contains a heat exchanger (E), depending on the objective of the experiment. However, in both cases, the efficiency of the process will be evaluated under non-stirred and stirred conditions. In loading and unloading gas to the reactors, the gas extraction system (F) is activated to minimize any risk to the health of the technical personnel. This system was responsible for eliminating possible gas leaks that may escape during operations, keeping the work environment safe, and avoiding the accumulation of gases that could be harmful if inhaled in high concentrations. This is especially important in high-pressure systems, where the possibility of an accidental leak may increase due to the high density and pressure of the gas. During the process, key variables, such as pressure and temperature, were constantly monitored at defined time intervals. These parameters were observed through each reactor's controller (G), ensuring the conditions were maintained within the established limits and optimizing the CO_2 sorption process's efficiency. This monitoring secured the system's stability and facilitated real-time decision-making to adjust any deviation that could affect the operation. At the end of the experiments, a detailed evaluation of the results was performed using a computer (H). In the evaluation, the adsorption capacity and process efficiency were analyzed as a function of time, allowing the system's performance to be determined under the established operating conditions. These data were essential to adjust future parameters and optimize adsorption efficiency in upcoming experiments or industrial applications.

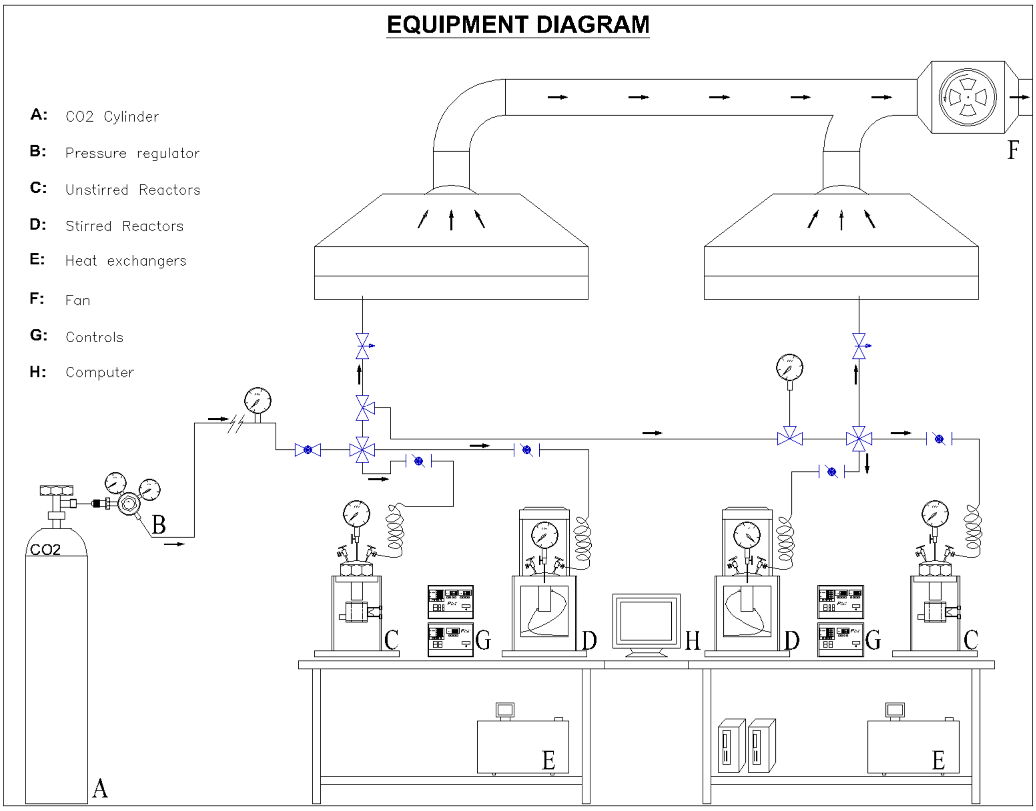


Figure 1. The experimental design setup.

2.3. CO_2 Sorption Testing Protocols

The system involved loading 30 ± 0.2 cm^3 of the aqueous solution to be tested into the non-stirred reactor. The vessel was integrated into the experimental setup, which was set to a temperature of 30 °C, controlled by a heating unit and a temperature controller. Pure CO_2 was then supplied up to 500 psia (initial pressure) directly into the liquid phase, regulated by a pressure regulator connected to a pure CO_2 cylinder. The experiment was conducted over 20–24 h, during which the system was maintained in continuous operation, and data recording proceeded uninterrupted. The pressure and temperature parameters were monitored in real-time using SpecView 32 SCADA software.

The experimental protocol was applied similarly in the experimental setup for the stirred reactor. However, unlike the non-stirred reactor, the solution's volume was higher (250 mL) in the stirred reactor, and an additional variable of stir was introduced, set at 300 rpm. It was controlled by a, which also controlled temperature and pressure for the stirred reactor. Figure 1 shows the experimental setup used in the study, which includes all the essential components for performing the CO_2 sorption tests.

2.4. Data Processing

The data collected by SpecView 3.1 software provided information on time, pressure (P), temperature (T), and stir (specifically for the stirred reactor), which was used to carry out the kinetic and thermodynamic study. The Peng-Robinson equation-of-state [38] was utilized to calculate the compressibility factor (Z). Equation (1) allowed us to determine the number of gas moles in the gas phase at any time $\left(n_t^{gas}\right)$. V is the gas phase volume.

$$PV = n_t^{gas} ZRT \tag{1}$$

Additionally, the CO_2 removed $\left(n_{CO_{2\_removed}}\right)$ was measured to quantify the amount of gas transferring from the gas to the liquid phase (Equation (2)). This was achieved by subtracting the remaining CO_2 in the gas phase $(n_{t_f}^{gas})$ from the initial amount $(n_{t_0}^{gas})$.

$$n_{CO_{2\_removed}} = n_{t_0}^{gas} - n_{t_f}^{gas} \tag{2}$$

Subsequently, these values allowed time-dependent graphs to facilitate kinetic and thermodynamic analyses. The kinetics issues were based on estimating (i) the capture rate (dn/dt) in the first moments of the experiments by the initial slope method [42], and (ii) the time required to capture 10%, 25%, 50%, and 90% of the total CO_2 removed in each test, identified as t_{10}, t_{25}, t_{50}, and t_{90}, respectively. Moreover, the thermodynamic analysis focused on the $CO_{2\_loading}$, i.e., the CO_2 sorption capacity at equilibrium per mole of amine (Equation (3)). There are different units for CO_2 loading. However, in the context of this study, it is expressed as mmol CO_2/mmol amine.

$$CO_{2\_loading} = \frac{n_{CO_{2\_removed}}}{n_{amine}} \tag{3}$$

2.5. Corrosion Potential Testing Protocol

The rich-amine solutions, i.e., the aqueous amine solutions pre-absorbed with CO_2, were used for the corrosion potential tests. Wrought iron specimens of approximately 2.5 cm in size were prepared and weighed before being immersed in the solutions (m_i) for 4 months (t). At the end of the immersion, each specimen was sanded smoothly on all sides to remove surface corrosion, and then its mass was measured again $\left(m_f\right)$. The mass loss during the tests allowed the evaluation of the corrosion rate (R) induced by the rich-amine solutions, using Equation (4). R is expressed as grams per month.

$$R = \frac{\left(m_i - m_f\right)}{t} \tag{4}$$

3. Results and Discussion

3.1. CO_2 Sorption Using Non-Stirred Equipment

The partial pressure and CO_2 removed versus sorption time from the non-stirred reactor are presented in Figure 2. Both graphs demonstrate that each solution tended to stabilize at different times, reflecting the varying reaction rates of the reactants, with some reacting more quickly than others.

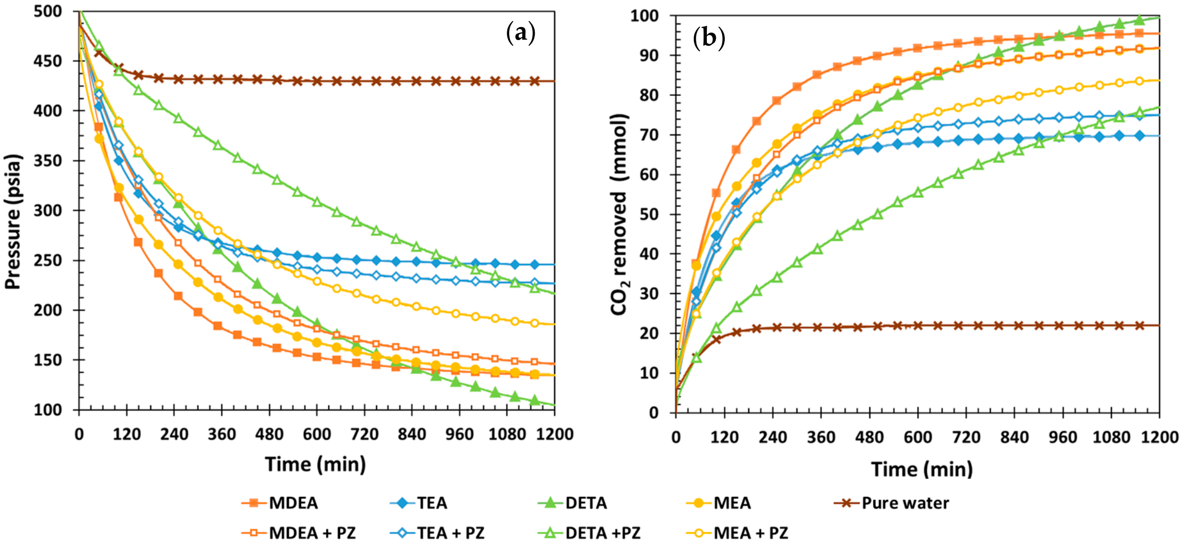


Figure 2. CO_2 sorption testing in the non-stirred equipment: (**a**) pressure drop; (**b**) CO_2 removed.

The kinetic data in Table 2 show that the MEA solution had the highest reaction rate in the first 120 min in the non-stirred system, followed by MDEA, TEA, and DETA. The reaction rate would be directly influenced by the formation of carbamates or bicarbonate ions, with carbamates leading to faster reaction kinetics due to their more excellent stability [43,44]. Primary and secondary amines, which tend to form carbamates, exhibited faster reaction rates than tertiary amines, which form bicarbonates [45]. dos Santos et al. [46] reported the formation of monoalkyl carbonate—a hemiester of carbonic acid and the alcohol group of the alkanolamine—with formation constants much higher than carbamates. This difference in reaction mechanisms contributed to the slower equilibrium times observed for TEA and MDEA compared to MEA and DETA. Moreover, the distinct behavior of MDEA, which did not show any reduction in its reaction rate with piperazine, suggests that the molecular structure of MDEA may interact uniquely with piperazine, possibly due to steric effects or differing pathways in carbamate versus bicarbonate formation.

Table 2. Kinetic data for CO_2 sorption using the non-stirred reactor.

Solution	***dn/dt*** **(mmol/min)**	**t_{10} (min)**	**t_{25} (min)**	**t_{50} (min)**	**t_{90} (min)**
MDEA	0.41	4.50	36.17	123.83	557.70
DETA	0.34	2.17	53.17	217.50	842.83
TEA	0.37	2.17	20.17	63.50	293.17
MEA	0.55	0.17	17.50	84.50	541.17
MDEA + PZ	0.33	4.50	36.17	123.83	557.50
DETA + PZ	0.18	21.83	89.83	334.83	1043.83
TE + PZ	0.35	3.50	26.17	83.17	399.83
MEA + PZ	0.26	3.17	36.50	147.17	691.17
Pure water	0.13	0.08	0.50	31.50	140.50

The CO_2 removal performance of the tested amines—MDEA, DETA, TEA, and MEA—is summarized in Table 3. Unlike the other compounds, DETA exhibited the highest CO_2 loading capacity, although it did not fully reach equilibrium. MEA and MDEA followed, with TEA removing three times more moles of CO_2 than pure water. The CO_2 loading obtained by each compound is also presented in Table 3, highlighting that DETA and MDEA achieved the highest CO_2 loading, with a value of 0.920 mmol CO_2 per mmol of amine.

Table 3. Thermodynamic data for CO_2 sorption using a non-stirred reactor.

Solution	Amine	CO_2 Removed (mmol)	CO_2 Loading (mmol/mmol Amine)	
	(mmol)		This Study	Referential Data
MDEA	104.53	93.859	0.898	(0.90–1.00) [47]
TEA	90.41	69.994	0.774	(0.57–1.18) [48]
DETA	111.08	102.188	0.920	(0.66–1.03) [49]
MEA	198.88	92.489	0.465	(0.23–0.56) [31,50,51]
MDEA + PZ	122.61	92.631	0.756	(0.19–0.49) * [39,52]
TEA + PZ	109.98	75.450	0.686	-
DETA + PZ	127.71	80.921	0.634	-
MEA + PZ	216.51	85.278	0.394	-
Pure water	-	22.023	0.013	0.0128 [38]

* Data obtained from simulation (Aspen HYSYS and Aspen Plus).

The color variation of aqueous amine solutions after CO_2 sorption is shown in Figure 3. A notable observation across all studies was the uniform colorless appearance of the initial solutions. This initial characteristic provides a baseline for assessing the extent of reaction with CO_2. The progression in color intensity correlates with an increase in the number of moles of CO_2 reacting with the amine. Notably, TEA exhibited no significant color changes, which is consistent with the thermodynamic analysis, which identified TEA as having the lowest CO_2 sorption capacity. The greatest intensity in coloration was obtained by DETA, which was the one with the highest CO_2 loading.

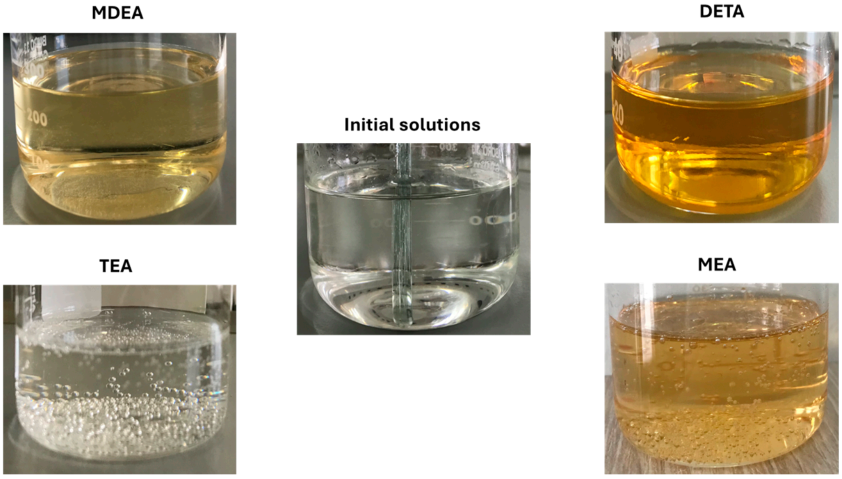


Figure 3. Changes in amine-based solutions color due to CO_2 sorption.

3.2. CO_2 Sorption Using Stirred Equipment

For the amines that demonstrated the most favorable thermodynamic and kinetic performance, taking into account the analytical approach applied at each stage, experiments were conducted in a stirred reactor under controlled conditions of 30 °C and 500 psig of initial pressure (Figure 4). Initially, it was hypothesized that 6 h would suffice to achieve equilibrium.

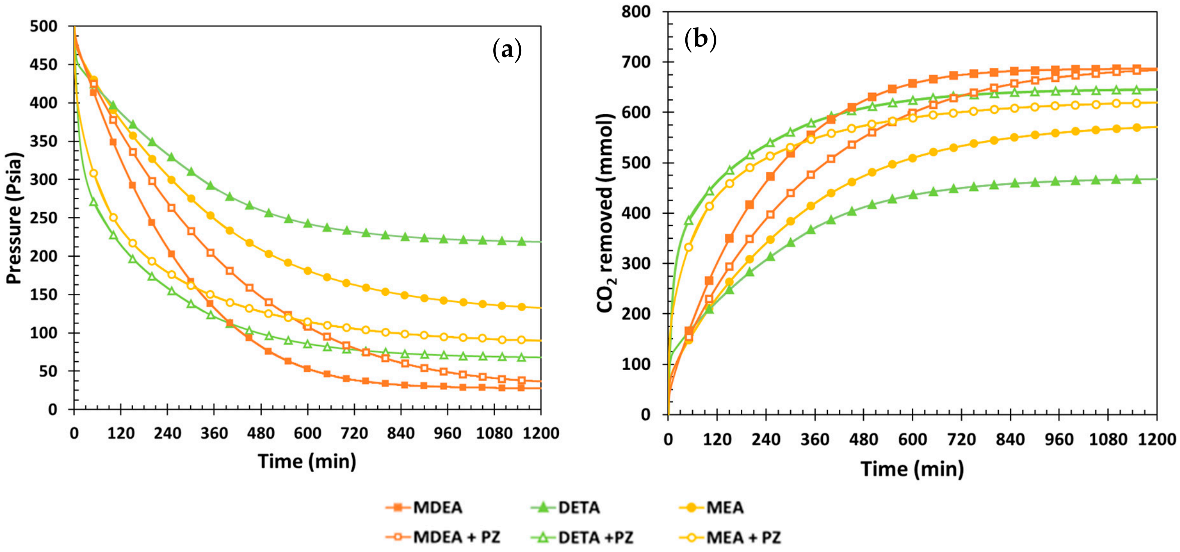


Figure 4. CO_2 sorption testing in the stirred equipment: (**a**) pressure drop; (**b**) CO_2 removed.

The MDEA solution exhibits the highest CO_2 sorption rate, followed by MEA and DEA, during the first two hours of experiments. Additionally, solutions containing piperazine significantly improve CO_2 loading capacity and sorption rate, as evidenced in Tables 4 and 5. MEA + PZ was the solution with the fastest CO_2 sorption rate. However, at equilibrium, MDEA and MDEA + PZ solutions reached the same amount of CO_2 removed, suggesting that for this amine, adding PZ did not influence its capacity for CO_2 removal.

Table 4. Kinetic data for CO_2 sorption using the stirred reactor.

Solution	dn/dt (mmol/min)	t_{10} (min)	t_{25} (min)	t_{50} (min)	t_{90} (min)
MDEA	2.33	10.83	52.50	145.83	468.17
DETA	1.05	0.50	7.83	130.5	514.69
MEA	1.46	3.17	45.83	174.17	514.61
MDEA + PZ	1.65	4.50	60.50	193.83	658.17
DETA + PZ	1.63	0.50	6.17	30.50	543.81
MEA + PZ	2.59	0.17	3.83	40.83	404.50

Table 5. Thermodynamic data for CO_2 sorption using a stirred reactor.

Solution	Amine	CO_2 Removed (mmol)	CO_2 Loading (mmol/mmol Amine)	
	(mmol)		This Study	Referential Data
MDEA	871.08	686.700	0.788	(0.9–1.0) [47]
DETA	925.66	467.644	0.505	(0.66–1.03) [49]
MEA	1657.33	571.037	0.345	(0.232–0.56) [31,50,51]
MDEA + PZ	1021.71	685.979	0.671	(0.19–0.49) * [39,52]
DETA + PZ	1064.25	645.423	0.606	-
MEA + PZ	1804.24.	621.952	0.345	-

* Data obtained from simulation (Aspen HYSYS and Aspen Plus).

As the search for references was conducted to corroborate the thermodynamic data, it was found that adding piperazine has a promoting effect on the reaction [53–55]. The kinetic study in the non-stirred reactor revealed an unexpected outcome: piperazine reduced the sorption rate compared to amine-only solutions. As shown in Table 2, this behavior highlights a decrease in sorption efficiency when piperazine is added under non-stirred conditions. In contrast, experiments in the stirred reactor demonstrated that piperazine effectively enhances the reaction rate when stir is introduced, as evidenced by the data in Table 4. This discrepancy can be attributed to the lack of proper mixing in the non-stirred reactor, which likely hindered the interaction between piperazine and the amine solutions, thus diminishing its expected catalytic effect. These findings emphasize the critical role of mixing in maximizing the performance of piperazine as a reaction promoter. It also indicated that the behavior of the CO_2 capture process using chemical solvents depends significantly on the equipment used. In non-stirred systems, the kinetics would be slower; in stirred systems, the kinetics can be faster. Still, we must consider the energetic cost of keeping the system under agitation.

3.3. Corrosion Potential Tests

Table 6 presents the loss in mass and corrosion rate of wrought iron specimens due to exposure to rich-amine solutions. When comparing DETA, a secondary amine, with TEA, a tertiary amine, both in aqueous solutions, it was observed that the corrosion rate of DETA is nearly double that of TEA, confirming the tendency of secondary amines to corrode more than tertiary ones. Additionally, the results from tests conducted on mixtures of amine solutions with piperazine, considering that MEA is a primary amine, DETA is a secondary amine, and MDEA is a tertiary amine, indicate that the primary amine (MEA) exhibits the highest corrosion rate. This finding aligns with studies by Fleury et al. [56] and Gunasekaran et al. [57], demonstrating that MEA is the most corrosive among the amines tested.

Table 6. Corrosion potential rate.

Solution	Initial Mass (g)	Final Mass (g)	Corrosion Rate × 10^3 (g/month)
DETA	4.051	4.035	4.00
MEA	3.929	3.920	2.25
MDEA + PZ	4.173	4.150	5.75
DETA + PZ	4.195	4.184	2.75
MEA + PZ	3.910	3.858	13.00
Environmental	3.868	3.862	1.50

4. Conclusions and Perspectives

The study aimed to identify the optimal amines for CO_2 sorption. The experiments conducted in the non-stirred reactor demonstrated that DETA and MDEA achieved the highest thermodynamic performance, with CO_2 removal amounts of 102.188 and 93.859 mmol, respectively, and an absorption capacity of ~0.90 mmol of CO_2 per mmol of amine.

Among the tested solutions in the stirred reactor, MDEA exhibited the highest overall absorption capacity. At the same time, MEA + PZ proved to be the most efficient in balancing high capacity and short processing times. The addition of piperazine significantly enhanced the performance of both MEA and DETA, especially in the initial stages of sorption, demonstrating its effectiveness as a promoter under appropriate conditions. However, MDEA + PZ, while achieving the highest total capacity, required longer processing times, indicating a slower kinetic performance. These findings underscore the importance of tailoring the choice of absorbent to the specific operational priorities, such as maximizing absorption capacity or optimizing reaction speed, and highlight the necessity of adjusting experimental parameters to ensure equilibrium is achieved.

The comparison between stirred and non-stirred systems demonstrated that proper mixing is crucial for enhancing reaction kinetics and accurately assessing corrosion tendencies. In the absence of agitation, the uneven distribution of reagents, including piperazine, likely contributed to the unexpected reduction in corrosion rates for certain amine mixtures. The results suggest a stirred system improves CO_2 sorption rates and impacts the amine-based solutions' corrosivity. Adding piperazine to amine solutions was expected to enhance CO_2 absorption and potentially influence corrosion rates. However, unlike stirred systems, the piperazine did not act as an effective promoter in non-stirred systems.

Subsequent studies will be carried out on the rich-amine solutions to identify ionic complexes or C-N bond-forming complexes resulting from reactions between CO_2 and aqueous amine solutions. This could be achieved using Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, or other spectroscopic techniques. Furthermore, additional studies will allow the identification of corrosion products and the corrosion mechanism that occurs in wrought iron and other alloys exposed to rich-amine solutions using electrochemical techniques.

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