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# Reaction Kinetics of Carbon Dioxide with Nonaqueous Solutions of Sterically Hindered Amines

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**Abstract:** Due to existing energy intensive $CO_2$ capture processes and rising fuel costs, alternative and affordable solvents or technologies for $CO_2$ capture have gained importance in the research of reducing global warming. To that effect, sterically hindered amines have been introduced which have high $CO_2$ absorption capacities by means of the formation of unstable carbamate ions. Another factor leading to an affordable solvent for $CO_2$ capture could be using nonaqueous solvents instead of aqueous solvents in the process. This is important because nonaqueous solvents can eliminate some of the problems of aqueous solvents such as corrosion and high thermal requirements. To that end, in this study, we investigated the reaction kinetics of $CO_2$ and two sterically hindered amines; 2-amino-2-methyl-1,3-propanediol (AMPD) and 2-amino-2-ethyl-1,3-propanediol (AEPD) in ethanol. The pseudo–first-order reaction rate constants of the reactions between $CO_2$ and sterically hindered amines were measured in ethanol at 288, 298 and 308 K by using direct stopped-flow technique. The measured rate constants were then analyzed by using the equations of termolecular reaction mechanism. The orders of the reactions ($n$) between $CO_2$-AMPD and $CO_2$-AEPD were found as 1 and 2, respectively.

**Keywords:** Carbon dioxide absorption; fast reaction kinetics; global warming; sterically hindered amines; stopped-flow technique.

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## INTRODUCTION

The enhanced greenhouse effect resulting from the extensive use of fossil fuels to produce energy is one of the universal concerns nowadays. Therefore, there is a vital need to control the emission of greenhouse gases into the atmosphere, especially carbon dioxide ($CO_2$), due to its high abundance, so as to mitigate their greenhouse effect [1-3]. It is projected that the increase in energy efficiency and the use of renewable energy sources will not provide the required reductions in $CO_2$ emissions. On the other hand, the capture of $CO_2$ from industrial flue gases prior to its release into the atmosphere and subsequent storage of the captured $CO_2$ away from the environment seems to be a promising approach towards reducing global climate change due to the enhanced greenhouse effect [4, 5]. Today, the most effective method for the separation and capture of $CO_2$ from a gas mixture is the absorption of $CO_2$ into amine solutions with a reversible reaction [6, 7]. The $CO_2$ capture technology conventionally used in large scale is the absorption/desorption process in which aqueous monoethanolamine (MEA) solution is frequently used as solvent [8-10]. Among conventional amines, MEA is often used in industry due to its high $CO_2$ absorption rate. However, MEA has a low $CO_2$ absorption capacity (amine/$CO_2$ < 0.5) and a high thermal requirement for its regeneration due to stable carbamate formation. MEA also has a high degradation rate and a corrosive property [11]. For these reasons, sterically hindered amines which can absorb high amounts of $CO_2$ (amine/$CO_2$ = 1) and form unstable carbamates have been introduced [12] and obtaining rate constants of the reactions between $CO_2$ and sterically hindered amines to estimate the efficiency of the $CO_2$ absorption have gained importance in recent years [13-18]. Sterically hindered amines which are proposed as alternatives to conventional amines can be designed for specific applications, have increased resistance to degradation and can also be produced from renewable resources. In addition, compared to aqueous systems, the use of non-aqueous systems (*i.e.*, ethanolic or methanolic system) is considered to be a promising alternative method for $CO_2$ capture, due to high solubility and capacity, low corrosiveness and low energy consumption for the regeneration of used solvents [19-22].

In this study, reaction kinetics of $CO_2$ with different concentrations of two sterically hindered amines namely, 2-amino-2-methyl-1,3-propanediol (AMPD) and 2-amino-2-ethyl-1,3-propanediol (AEPD) in ethanol were investigated at 288, 298 and 308 K. The pseudo-first order reaction rate constants $k_o$ ($s^{-1}$) were directly measured using stopped-flow technique. Termolecular reaction mechanism was used to model the kinetics of the system and to calculate forward reaction rate constants. Interest in AMPD and AEPD comes from the fact that both of them can be produced from renewable sources, that is from 1,2-propanediol and 1,3-propanediol which are abundant as side products of biodiesel process [23, 24].

## MATERIALS AND METHODS

### Chemicals

Sterically hindered amines, AMPD and AEPD of purities ≥ 99.8%, and ethanol of purity ≥ 99.9% were supplied by Merck (Darmstadt, Germany) and J.T. Baker, respectively. Carbon dioxide with a purity of 99.9% was supplied by Linde (Munich, Germany). The experiments were carried out in ethanolic solution using 0.25–1.00 kmol/m$^3$ of AMPD and 0.25–1.25 kmol/m$^3$ of AEPD, at 288, 298 and 308 K.

### Apparatus

The stopped-flow apparatus (model SF-61SX2; Hi-Tech Scientific, Salisbury, UK) with a conductivity detector is a direct standard technique used for kinetic study of the fast reactions between amines and $CO_2$. The equipment consists of four main units: a sample handling unit, a conductivity cell, an A/D converter, and a microprocessor unit. The entire flow circuit is encased in a thermostat and maintained at a constant temperature by an external water bath within ± 0.1 K. "Kineticasyst" software was used to activate the pneumatically controlled drive plate, which pushes separately the amine and $CO_2$ solutions in equal volumes into the conductivity detection cell through a mixing loop. For each experimental run, equal volumes of solution were suddenly pushed into and mixed in the stopped flow mixer cell. In the conductivity cell, solutions react to generate ions that conduct electricity through platinum electrodes placed across the cell. The conductivity detection system directly measures the intrinsic rate of the rapid homogeneous reaction. The reaction software automatically calculates the observed pseudo–first order rate constant ($k_o$) based on least square regression. To satisfy the pseudo–first order condition, concentration ratios of the amine (AMPD or AEPD) to that of $CO_2$ were maintained at about 10:1 for all experiments. Further details of the equipment and the experimental procedure can be found elsewhere [19, 25, 26].

## Experimental procedure

According to termolecular reaction mechanism, which was first proposed by Crooks and Donnellan [27], there is a loosely bound encounter complex formed as initial product in the reaction between amine and $CO_2$. In this mechanism, the bonding between amine and $CO_2$ and the proton transfer take place simultaneously, involving only a single step as represented by Eq. (1). Here, carbamate formation involves the transfer of a proton from the amine functionality.

$$\mathrm{CO_2 + RNH_2 \cdots B \leftrightarrow RNHCOO^- \cdots BH^+} \qquad \textbf{(Eq. 1)}$$

It should be noted that for sterically hindered amines the carbamate ion is unstable, leading to the following reaction [26]

$$\mathrm{CO_2 + RNH_2 + H_2O \leftrightarrow HCO_3^- + RNH_3^+} \qquad \textbf{(Eq. 2)}$$

The observed carbon dioxide absorption rate in amine solutions is given as

$$r_{obs} = k_o[\mathrm{CO_2}] \qquad \textbf{(Eq. 3)}$$

The observed pseudo–first order reaction rate constant ($k_o$) in aqueous systems is given as

$$k_o = k_{H_2O}[\mathrm{H_2O}][\mathrm{RNH_2}] + k_{RNH_2}[\mathrm{RNH_2}]^2 \qquad \textbf{(Eq. 4)}$$

According to Eq. 1, in nonaqueous systems such as in ethanol (EtOH), $k_o$ will be

$$k_o = k_{EtOH}[\mathrm{EtOH}][\mathrm{RNH_2}] + k_{RNH_2}[\mathrm{RNH_2}]^2 \qquad \textbf{(Eq. 5)}$$

Note that the base (B) which is weakly bonded to amine in Eq. (1) could be any base in the environment such as amine or ethanol in a nonaqueous system. Considering that EtOH concentration remains constant for dilute nonaqueous amine solutions, then, introducing constant *k*, where

$$k = k_{EtOH}[\mathrm{EtOH}] \qquad \textbf{(Eq. 6)}$$

Thus, the observed pseudo-first order reaction rate constant in ethanol system will be

$$k_o = k[RNH_2] + k_{RNH_2}[RNH_2]^2 \qquad \textbf{(Eq. 7)}$$

## RESULTS AND DISCUSSION

The reaction kinetics of carbon dioxide with AMPD or AEPD in ethanol were investigated at 288, 298, and 308 K. Table 1, Figure 1 and Figure 2 show the experimentally obtained pseudo–first order reaction rate constants for the sterically hindered amines dissolved in ethanol. It is clearly seen that the reaction rate constants increased with increasing temperature at a constant amine concentration. Similarly, the rate constants also increased with increasing amine concentration at a constant temperature. Due to the limited solubility of AMPD in ethanol, the maximum 1.00 kmol/m$^3$ concentration of solution could be successfully prepared.

**Table 1.** Measured pseudo-first order rate constants of the reactions between $CO_2$-AMPD and $CO_2$-AEPD in ethanol at 288, 298 and 308 K

| [AMPD] (kmol/m$^3$) | $k_{o(288\ K)}$ (s$^{-1}$) | $k_{o(298\ K)}$ (s$^{-1}$) | $k_{o(308\ K)}$ (s$^{-1}$) |
|---|---|---|---|
| 0.25 | 2.9 | 6.0 | 8.5 |
| 0.50 | 6.9 | 11.0 | 17.0 |
| 0.75 | 10.2 | 19.9 | 31.6 |
| 1.00 | 19.2 | 32.3 | 45.0 |
| [AEPD] (kmol/m$^3$) | $k_{o(288\ K)}$ (s$^{-1}$) | $k_{o(298\ K)}$ (s$^{-1}$) | $k_{o(308\ K)}$ (s$^{-1}$) |
| 0.25 | 2.3 | 3.0 | 3.9 |
| 0.5 | 8.3 | 9.6 | 10.5 |
| 0.75 | 19.4 | 21.6 | 26.2 |
| 1.00 | 30.2 | 35.6 | 44.2 |
| 1.25 | 53.8 | 61.4 | 73.4 |

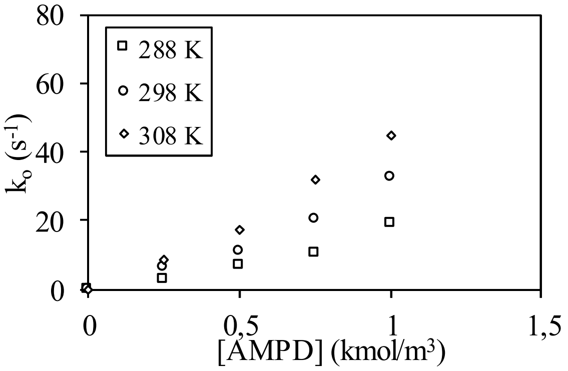

**Figure 1.** Effect of AMPD concentration on the measured pseudo-first order rate constants of the reaction between $CO_2$-AMPD in ethanol at 288, 298 and 308 K.

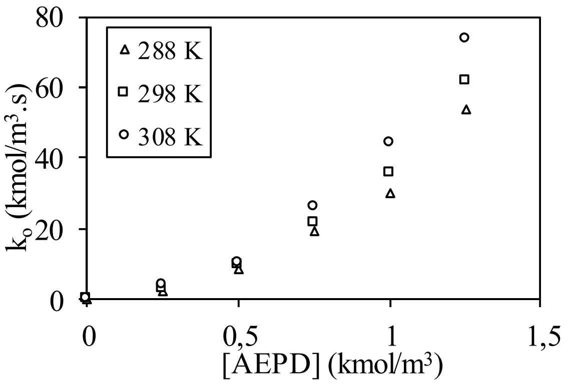

**Figure 2.** Effect of AEPD concentration on the measured pseudo-first order rate constants of the reaction between $CO_2$-AEPD in ethanol at 288, 298 and 308 K.

The obtained values of pseudo-first order reaction rate constants ($k_o$) were generally low in comparison to that of MEA systems. Considering that AMPD and AEPD are sterically hindered amines, this trend is comparable to that of other hindered amines [16-18, 25]. In addition, $CO_2$-amine reaction rates in nonaqueous solution such as ethanolic solution used in our study were observed to be lower than the reaction rates in aqueous solution. For example, in our previous study [16] the pseudo–first order reaction rate constant ($k_o$) measured between $CO_2$ and aqueous AMPD [0.5 kmol/m$^3$] at 298 K was found to be 55.9 s$^{-1}$. Compared to that, the measured rate constant ($k_o$) of the reaction between AMPD [0.5 kmol/m$^3$] and $CO_2$ in ethanol at 298 K was found to be 11.0 s$^{-1}$ (Table 1). Similarly, the measured reaction rate constants for $CO_2$-AEPD in ethanol were lower than the rate constants obtained for the same reaction in aqueous solution [17]. This trend can be linked to the lower basicity of ethanol compared to that of water.

Empirical power law plots for the $CO_2$-AMPD system (Figure 3) gave reaction orders of 1.31, 1.20 and 1.21 at 288, 298 and 308 K, respectively (Table 2). In other words, according to termolecular reaction mechanism, the base which is weakly bonded to amine was either EtOH or amine itself for all temperatures investigated. However, due to the fact that the degrees obtained were very close to 1, the base was mostly EtOH. The measured rate constant $k_o$ (s$^{-1}$) of the reaction between $CO_2$ and AMPD in ethanol solution can be shown as below:

$$k_o = k_{EtOH}[EtOH][AMPD] + k_{AMPD}[AMPD]^2 \qquad \textbf{(Eq. 8)}$$

**Table 2.** Forward reaction rate constants and reaction orders (*n*) of $CO_2$-AMPD and $CO_2$-AEPD in ethanol at 288, 298 and 308 K.

| Temperature (K) | $k$ ($m^3$/kmol.s) | $k_{AMPD}$ ( $m^6/kmol^2.s$) | $n$ |
|---|---|---|---|
| 288 | 6 | 12.4 | 1.31 |
| 298 | 14 | 17.7 | 1.20 |
| 308 | 27 | 18.0 | 1.21 |
| Temperature(K) | | $k_{AEPD}$ ( $m^6/kmol^2.s$) | $n$ |
| 288 | | 33 | 1.93 |
| 298 | | 38 | 1.90 |
| 308 | | 46 | 1.85 |

In the literature, the $CO_2$-AMPD reaction was examined indirectly in aqueous media and related solubility or kinetic data were reported [13-15]. To the best of our knowledge, no kinetic studies in ethanol media was reported for $CO_2$-AMPD system. The kinetic studies for $CO_2$-AMPD system performed in aqueous media at different temperatures (278-298 K) has revealed that the reaction order of $CO_2$ with AMPD varies between 1.12 and 1.25 ([AMPD] = 0.10-1.50 kmol/$m^3$). This leads to the conclusion that the base which is weakly bonded to amine in $CO_2$-AMPD system is the solvent used in the system.

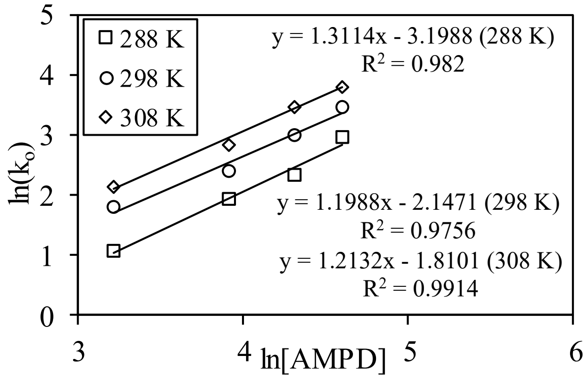

**Figure 3.** Empirical power law plots for $CO_2$-AMPD system in ethanol at 288, 298, and 308 K.

When $CO_2$-AEPD reaction in ethanolic media was investigated, the empirical power law plots for the system (Figure 4) gave reaction orders of 1.93, 1.90 and 1.85 at 288, 298 and 308 K, respectively (Table 2). It implies that, for $CO_2$-AEPD system, the base which is weakly bonded to amine was mostly amine itself for all temperatures investigated.

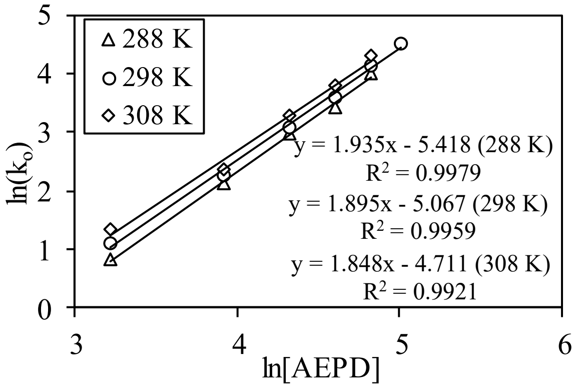

**Figure 4.** Empirical power law plots for $CO_2$-AEPD system in ethanol at 288, 298, and 308 K.

If the effect of EtOH as a base is neglected, then the measured rate constant $k_o$ ($s^{-1}$) of the reaction between $CO_2$ and AEPD in ethanol solution can be shown as below:

$$k_o = k_{AEPD}[AEPD]^2 \qquad \textbf{(Eq. 9)}$$

Previously we have found that the reaction order of $CO_2$ with aqueous AEPD varies between 1.04 and 1.14 for temperatures between 278 and 298 K ([AEPD] = 0.03-1.00 kmol/m$^3$) [17]. Similarly in another study [18] the reaction order of $CO_2$-AEPD in aqueous solution was reported to be as 1 at temperatures between 303 and 318 K ([AEPD] = 0.41-2.15 kmol/m$^3$). To conclude, the reaction orders and reaction rate constants of the $CO_2$-AEPD system in aqueous and nonaqueous solutions are different from each other. The results obtained in this respect are important in order to estimate the efficiency of the $CO_2$ absorption to occur, since they are not previously found in the literature and are obtained directly using the stop-flow technique.

The forward reaction rate constants for $CO_2$-AMPD reaction, $k$ ($k_{EtOH}[EtOH]$, m$^3$/kmol.s) and $k_{AMPD}$ (m$^6$/kmol$^2$.s), and for $CO_2$-AEPD reaction, $k_{AEPD}$ (m$^6$/kmol$^2$.s) were obtained by polinomial regressions (Table 2). For both reactions, the forward rate constants increased with increasing temperature. However, all rate constants obtained for $CO_2$-sterically hindered amine reaction are very slow relative to the rate of an amine solution commonly used in the industry, such as MEA. In the case of large-scale $CO_2$ absorption processes, activator-associated solutions of sterically hindered amines will need to be used.

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### Türkçe Öz ve Anahtar Kelimeler

# Sterik Olarak Engelli Aminlerin Susuz Çözeltileriyle Karbon Dioksidin Reaksiyon Kinetikleri

F. Pınar Gördesli Duatepe, Erdoğan Alper

**Öz:** Şu anda var olan $CO_2$ tutuklama süreçleri ve artan yakıt maliyetlerinden ötürü, küresel ısınmayı azaltacak araştırmalarda $CO_2$ tutuklama için ucuz çözücüler veya teknolojilerin kullanılması önem kazanmıştır. Bu anlamda, sterik olarak engelli aminler, kararsız karbamat iyonlarının oluşumu yoluyla yüksek $CO_2$ soğurma kapasitelerine sahip moleküller olarak bilim dünyasının dikkatine sunulmuştur. $CO_2$ tutuklanması için ucuz bir faktör de süreçte sulu çözücüler yerine susuz çözücülerin kullanılmasıdır. Bu değişiklik önemlidir, çünkü sulu çözücülerin korozyon ve yüksek ısıl gereksinimleri gibi bazı sorunları giderilmektedir. Bu bakımdan çalışmada $CO_2$ ve iki sterik olarak engelli amin (2-amino-2-metil-1,3-propandiol, AMPD ve 2-amino-2-etil-1,3-propandiol, AEPD) reaksiyon kinetikleri etanollü çözeltide çalışılmıştır. $CO_2$ ve sterik olarak engelli aminler arasındaki yalancı birinci mertebeden reaksiyon hızı sabitleri etanol içinde 288, 298 ve 308 K sıcaklıklarda doğrudan durdurulmuş akış tekniği ile ölçülmüştür. Ölçülen hız sabitleri üç moleküllü reaksiyon mekanizması kullanılarak analiz edilmiştir. $CO_2$-AMPD ve $CO_2$-AEPD arasındaki reaksiyonların mertebeleri (*n*) sırasıyla 1 ve 2 olarak tespit edilmiştir.

**Anahtar kelimeler:** Karbon dioksit soğurulması; hızlı reaksiyon kinetikleri; küresel ısınma; sterik olarak engelli aminler; durdurulmuş akış tekniği.

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