

## Vapor liquid equilibria and heat of absorption of CO<sub>2</sub> in aqueous 2-(diethylamino)-ethanol solutions



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### ARTICLE INFO

#### Article history:

Received 23 December 2013

Received in revised form 1 July 2014

Accepted 31 July 2014

#### Keywords:

DEEA

Vapor liquid equilibrium

Heat of absorption

eNRTL modeling

### ABSTRACT

The carbon dioxide (CO<sub>2</sub>) solubility in aqueous 2-(diethylamino)-ethanol (DEEA) solutions were measured at (60 and 80 °C), with the DEEA concentrations of 3 M and 4 M and pressure range of (3 to 192 kPa). The absorption heat of CO<sub>2</sub> with (1, 3, and 4 M) aqueous DEEA solutions at different temperatures is also presented in this work. A thermodynamic model based on the electrolyte non-random two-liquid (eNRTL) theory within Aspen Plus<sup>®</sup> has been developed to correlate and predict the vapor liquid equilibrium and heat of absorption of CO<sub>2</sub> in aqueous DEEA solutions. Meanwhile, the speciation of the solution at different loadings was also predicted using the developed eNRTL model.

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### 1. Introduction

Post combustion CO<sub>2</sub> capture by chemical absorption based on amines is proven to be a suitable technology (Rochelle, 2009). Many amines, such as the primary amine monoethanolamine (MEA), secondary amine diethanolamine (DEA), tertiary amine methyldiethanolamine (MDEA) and the diamine piperazine (PZ), have been applied to capture CO<sub>2</sub> (Bishnoi and Rochelle, 2002; Derks et al., 2005; Rinker et al., 2000). However, this process is found to be energy and capital intensive. Among all the amines, tertiary amines have lower absorption heat than primary and secondary amines due to the fact that there is no carbamation reaction when CO<sub>2</sub> reacts with a tertiary amine, thus having a potential for reducing the energy consumption during regeneration (Wang et al., 2011; Svendsen et al., 2011). 2-(Diethylamino)-ethanol (DEEA) is a promising tertiary amine that can be prepared from renewable resources (Konduru et al., 2010). Considering its relatively slow reaction rate with CO<sub>2</sub>, PZ and BDA can be used as additives to promote its reaction rate (Littel et al., 1990b; Vaidya and Kenig, 2008; Xu et al., 2013).

Vapor liquid equilibrium (VLE) is of great importance for the design and optimization of industrial gas treating process as it determines the minimum solution circulation rate and the maximum CO<sub>2</sub> concentration left after regeneration, as well as the best operation conditions during absorption and regeneration. Therefore, the VLE of many promising amines were studied in literature, including MEA, MDEA, AMP, etc. (Aronu et al., 2011; Kundu and Bandyopadhyay, 2006; Tong et al., 2012). The CO<sub>2</sub> partial pressure is always calculated by the product of total pressure multiplying CO<sub>2</sub> concentration, in which, the CO<sub>2</sub> concentration is measured by a FTIR CO<sub>2</sub> analyzer (Zhou et al., 2010). As of today, there is much literature presenting kinetics data of CO<sub>2</sub> absorption by DEEA (Littel et al., 1990b; Vaidya and Kenig, 2008; Xu et al., 2013; Kim and Savage, 1987; Li et al., 2007), but seldom reporting the VLE data. The only available VLE data are from Sutar et al. (2013) and Monteiro et al. (2013), which listed several VLE data of 2 M DEEA at 30 °C with a pressure range of (0.5–10 kPa), and 2 M and 5 M DEEA at 40–120 °C, respectively.

Heat of absorption is directly related with the regeneration energy requirement. Direct calorimeter measurements can give the accurate heat of absorption values, indicating a combination of the effect of the chemical reactions and physical dissolution, as well as the dependency of the absorption heat on temperature (Liu et al., 2012; Kim and Svendsen, 2011). Kim studied the heat of absorption of 32 wt% (~2.68 M) and 37 wt% (~3.11 M) DEEA at 40, 80 and 120 °C (Kim, 2009), while

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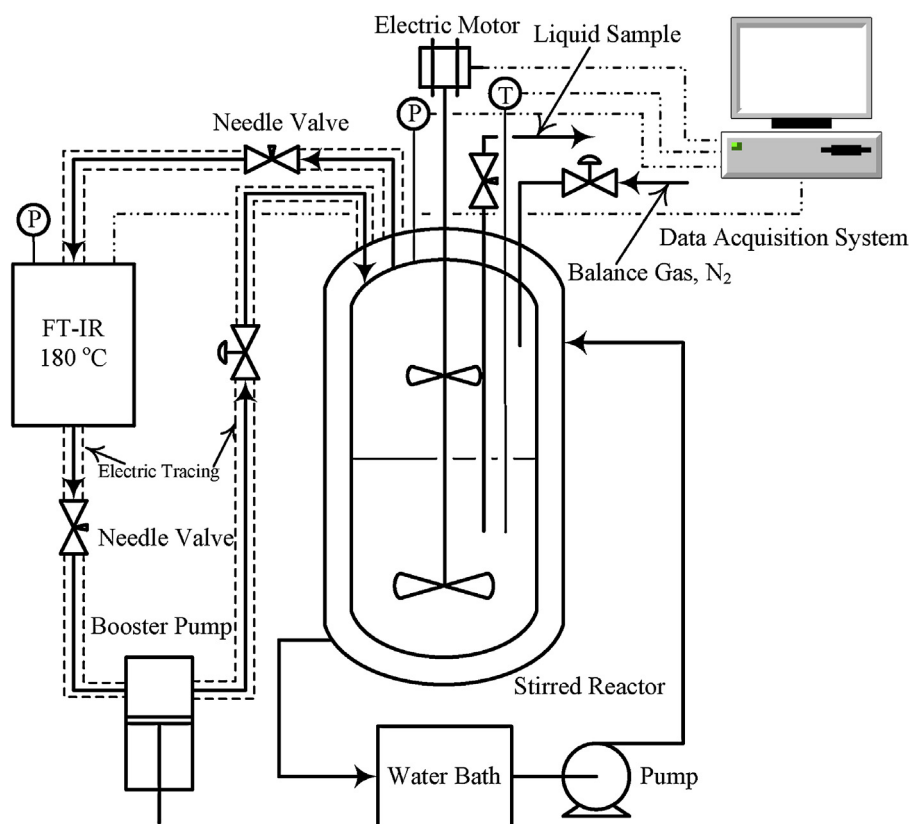


Fig. 1. Vapor liquid equilibrium measurement cell.

Arshad et al. (2013a,b) measured the absorption heat of 5 M DEEA at 40, 80 and 120 °C. Their reported DEEA concentrations were limited to around 3 M and 5 M. And also, considering that the reaction rate of DEEA is slow and DEEA usually used with other promotion amines together (such as PZ), and in the mixture the viscosity is related with DEEA's concentration, the concentration of DEEA should not be very high in the actual application. Therefore, we measure the heat of absorption of 1, 3, and 4 M DEEA.

In order to study the vapor liquid equilibrium of DEEA in a wider temperature and pressure range and to report the heat of absorption of CO<sub>2</sub> absorption by DEEA for other concentrations, this work therefore has measured the VLE of 3 M and 4 M DEEA at (60 and 80 °C), and the absorption heat of (1 and 4) M DEEA at (40, 60, 80 and 120 °C), as well as the absorption heat of 3 M DEEA at (60 and 80 °C). A thermodynamic model based on the electrolyte non-random two-liquid (eNRTL) theory within Aspen Plus<sup>®</sup> was applied to correlate the VLE and absorption heat data and to predict the speciation in the solution at different loadings.

## 2. Experimental method and apparatus

### 2.1. Vapor liquid equilibria

DEEA ( $\geq 99$  wt% pure) was obtained from Aladdin Reagent Company. CO<sub>2</sub> ( $\geq 99.9\%$  pure) from Beijing Huayuan Gas Company was used. Deionized water was used for preparing the solutions. The amine concentrations were determined by titration against 0.2 N H<sub>2</sub>SO<sub>4</sub> using a Metrohm 809 Titrando auto titrator.

The VLE measurements were conducted in a stirred stainless steel equilibrium cell, as shown in Fig. 1. The CO<sub>2</sub> loaded solution was first added into the stirred cell, then the system was heated by water circulation. After the temperature and pressure inside of the cell were constant, the needle valves were opened and the FTIR

measurement system was ready to record the CO<sub>2</sub> concentration in the gas phase. When the pressure, temperature and CO<sub>2</sub> concentration of the system were constant, these were recorded and a liquid sample taken from the specially designed tube and titrated for amine and CO<sub>2</sub> concentration. As studied by Qi et al., the error of FTIR measured CO<sub>2</sub> concentration in gas phase is less than 2.2%, and the range of pressure transducer is 0.5 MPa, with a accuracy of 0.25% of the measured value. The error of CO<sub>2</sub> concentration in liquid phase is about 1% according to titration. All the other details can be found in reference (Qi, 2013).

### 2.2. Heat of absorption

The experimental setup of the 2 L reaction calorimeter CPA-122 (ChemiSens AB, Lund, Sweden) is shown in Fig. 2.

The calorimeter was first flushed with N<sub>2</sub>, and then was vacuumed to avoid any contamination in the reactor. Fresh solution was then charged into the reactor and heated to the set temperature before a small amount of CO<sub>2</sub> bubbled into the solution. After each CO<sub>2</sub> injection, the CO<sub>2</sub> loading and the heat of absorption at this loading were calculated or measured. The other details of the experimental setup of the calorimeter, CPA-122, are described by Kim and Svendsen (Kim and Svendsen, 2007) and will not be repeated here. The total heat of absorption for each experimental point was found by integration of the heat flow with time. The total amount of CO<sub>2</sub> added from the accumulator cylinder into the reactor at each stage was calculated from the pressure changes in the cylinder. The amount of CO<sub>2</sub> remaining in the gas phase of the reactor after each stage was calculated from the pressure changes in the reactor. The Peng–Robinson equation of state was used to calculate the total amount of CO<sub>2</sub> added to the reactor and that remaining in the reactor. The heats of absorption were obtained in terms of kJ/mol CO<sub>2</sub>.

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