

CHAPTER 1

INTRODUCTION

Acid gases such as CO_2 , H_2S and COS are the major impurities of natural and refinery gases, synthesis gas for ammonia production and synthetic natural gas. These impurities when present in the gas streams, lead to very serious problems in pipeline transportation and down stream processing of the gases. In case of ammonia synthesis gas, the acid gas impurities have to be removed to vanishing levels to avoid poisoning of synthesis catalyst. In natural gas processing, removal of acid gases, CO_2 and H_2S , to a level of about 2% for CO_2 and 4 ppm for H_2S is essential to avoid corrosion of pipeline and equipment and meet fuel gas specifications. In many cases bulk of the CO_2 and H_2S thus removed is recovered for reuse in the process such as for urea making and sulfur recovery. Removal of H_2S to a very low level from fuel gases is mandatory for environmental reasons. In natural gas liquefaction plants, gas sweetening processes are designed to meet more stringent standards with respect to the limiting concentrations of about 50 ppm and 4 ppm for CO_2 and H_2S , respectively, in the treated gas to avoid freezing in the cryogenic equipment.

Sweetening of sour gas streams is very often done by regenerative absorption of the acid gas impurities in a physical or chemical absorbent. Physical absorbents normally used for this purpose are chilled methanol (Rectisol process), N-methyl-2-Pyrrolidone (Purisol process), dimethylether of polyethylene glycol (Selexsol process) and propylene carbonate (Flour process), etc. The physical solvents usually have very good equilibrium loading capacity for the gaseous impurities and their regeneration is also relatively easier. But these solvents are expensive and they normally suffer from the disadvantage of co-absorption of hydrocarbons. When the rate of absorption and the economy is important as in the case of natural gas processing,

chemical solvents are mostly preferred though their regeneration is relatively difficult. Chemical solvents most widely used today are the amino alcohols or alkanolamines.

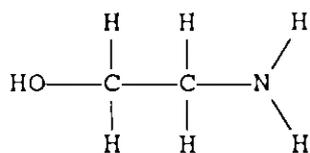
1.1 ALKANOLAMINES FOR ACID GAS ABSORPTION

Since the introduction of triethanolamine (TEA), a tertiary alkanolamine, by Bottoms (1930) as an absorbent in gas treating plants, other members of the alkanolamine family have been commercialized subsequently. In general these amines are used today in aqueous solutions, in combined solvents e.g. water and sulfolane as in Shell Sulfinol process, or as promoters in hot carbonate process.

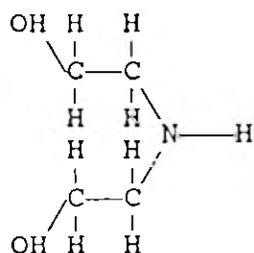
Industrially important alkanolamines are monoethanolamine (MEA), diethanolamine (DEA) di-2-propanolamine (DIPA), N-methyldiethanolamine (MDEA) and β, β' -hydroxy-aminoethylether known as Diglycolamine (DGA). Structural formulas for these alkanolamines are presented in Fig. 1.1.

Several proprietary formulations of alkanolamine solutions containing, besides the amine, corrosion inhibitor, foam depressants and activators are being offered under various trade names such as UCARSOL, Amine Guard (Union Carbide Corporation), GAS/SPEC IT-1 Solvents (Dow Chemical Company) and Activated MDEA (BASF Aktiengesellschaft), etc.

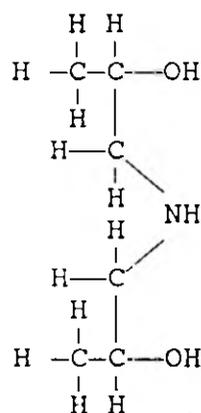
A typical amine absorption process is shown in Fig. 1.2. The sour feed gas stream with the acid gas impurities is contacted with the solvent at elevated pressure in the absorber. The solvent, loaded with the absorbed acid gas components, flow via a heat exchanger to the regenerator in which the acid gases are desorbed from the solvent at reduced pressure with steam, generated by reboiling. The acid gases



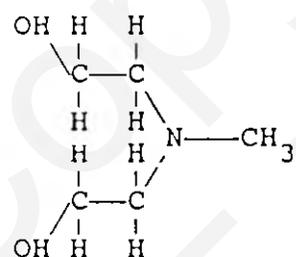
monoethanolamine (MEA)



diethanolamine (DEA)



di-2-propanolamine (DIPA)



N-methyldiethanolamine (MDEA)

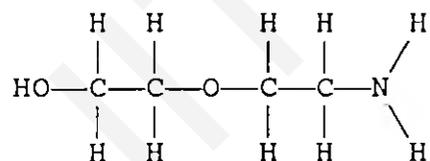

 β, β' -hydroxyaminoethylether
or
diglycolamine (DGA)

Fig.1.1. Structural formulas for important alkanolamines.

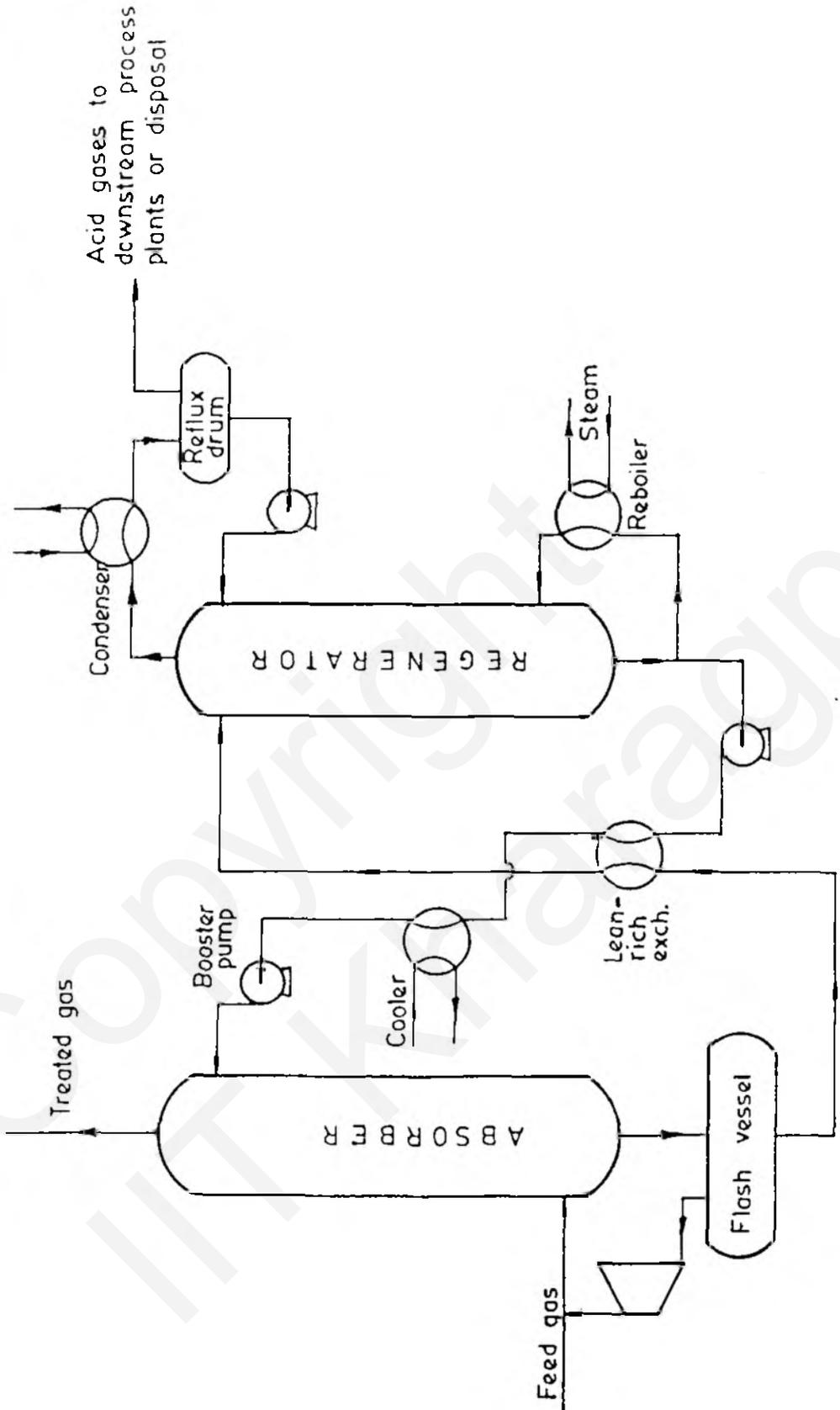


Fig.1.2 Basic flow scheme for an amine process for the removal of acid gases.

leave the regenerator overhead system after condensation of the stripping steam. The stripped solvent is returned to the absorber via heat exchanger and cooler.

The alkanolamines in solution undergo chemical reaction with acid gas impurities, thus giving rise to enhanced rate of absorption compared to absorption into the physical solvents. The reactions are discussed in detail in Chapter 2 (sec.2.1).

The reaction between H_2S and the amine involves a proton transfer and can be regarded as instantaneous and reversible. The primary, secondary and tertiary amines undergo the same reactions with H_2S forming amine sulfide and hydrosulfide. With CO_2 the primary and secondary amines react to form the relatively stable carbamate. It can be seen from Sec.2.1 that the maximum loading of CO_2 in the primary and secondary amines is limited by stoichiometry to 0.5 mol of CO_2 /mol of amine. A certain amount of carbamate hydrolysis occurs with all amines so that even with MEA and DEA the loading may marginally exceed 0.5, particularly at high pressures. With tertiary amines, which are unable to form carbamates, a loading of 1 mol of CO_2 /mol of amine can theoretically be achieved. But the CO_2 -tertiary amine reactions leading to the formation of bicarbonate are very slow, giving rise to low rate of absorption.

1.2 STERICALLY HINDERED AMINES :

A new class of amines, the sterically hindered amines, has been introduced recently by Exxon Research and Engineering Company. These amines are claimed as potential absorbents with high capacity for CO_2 absorption (Say et al., 1984; Kohl and Riesenfeld, 1985; Brown et al.,

1984). However, prior to this, Sharma (1964) highlighted the advantage of using highly branched amines such as 2-amino-2-methyl-1-propanol (AMP) for the absorption of CO_2 .

Hindered amines are formed by placing a bulky substituent group close to the amino nitrogen site to lower the stability of the carbamate ion. Steric hindrance plays a significant role in amine- CO_2 reactions owing to the bulky nature of the hindered amine molecule. It is suggested that due to the instability of the carbamate ion these carbamates readily undergo hydrolysis forming bicarbonate and releasing free amine (Alper, 1990), which again reacts with CO_2 thus leading to a stoichiometric loading capacity of 1 mol of CO_2 /mol of amine with appreciable rate of absorption for moderately hindered amines, particularly at higher loading of CO_2 (CO_2 -hindered amine reactions are discussed in detail in Sec. 2.1 of Chapter 2). This leads to a much reduced circulation rate and corresponding reduced regenerator heat load which translate into considerable energy saving (Goldstein et al., 1986).

A hindered amine is defined as belonging to either of these classes (Sartori et al., 1987) :

- i) a primary amine in which the amino group is attached to a tertiary carbon atom,
- ii) a secondary amine in which the amino group is attached to at least one secondary or tertiary carbon atom.

Structural formulas for some of the sterically hindered amines are shown in Fig. 1.3. Although all sterically hindered amines are not necessarily alkanolamines, their characteristics, as gas purification agents, are mostly similar to those of the alkanolamines. Out of the family of sterically hindered amines only the aliphatic and

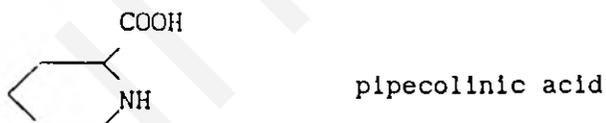
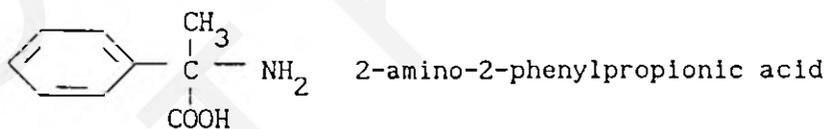
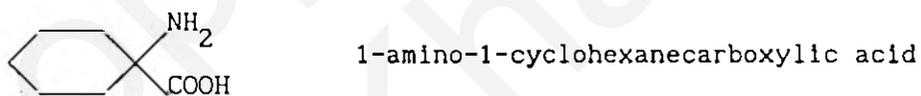
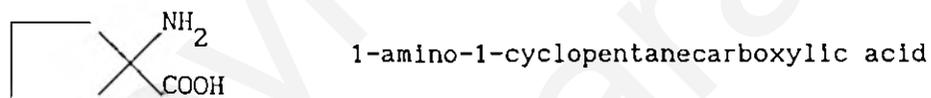
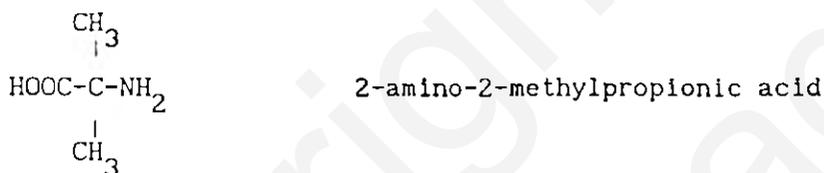
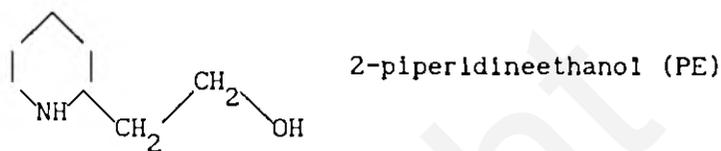
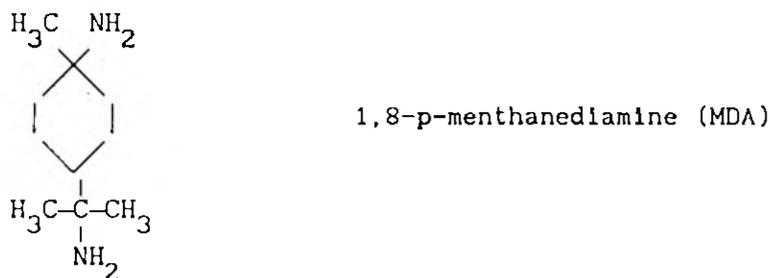
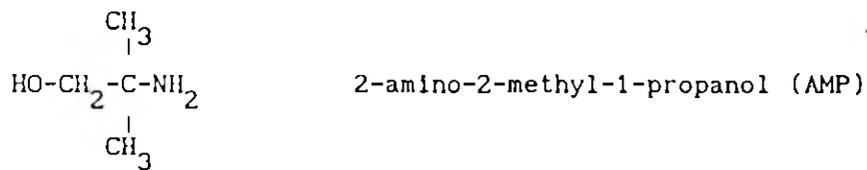


Fig.1.3. Structural formulas for some sterically hindered amines.

cycloaliphatic amines are suitable for gas treating. Aromatic amines, due to their lower basicity, lead to low absorption capacities and rates. Besides the amino-group, the amine must contain another functional group, e.g., a hydroxyl or carboxylic group, to increase solubility and reduce volatility.

It is claimed that in addition to the lowering of energy consumption, hindered amines can increase the output of existing plants, i.e., they can be used to retrofit and debottleneck a plant (Sartori et al., 1987). Besides saving energy and capital in gas treating processes significantly, the hindered amines used, have much better stability than conventional amines, since hindered amines have low or no amine degradation. In addition to the use of hindered amines in CO₂ removal, hindered amine-based processes (Flexsorb SE, Flexsorb PS, Flexsorb HP) for selective removal of H₂S and for non-selective removal of CO₂ and H₂S have also been commercialized by Exxon Research & Engineering Co. recently (Goldstein et al., 1986; Gas Process Handbook'92). It is indicated (Goldstein et al., 1986) that hindered amine-based process, Flexsorb SE, is a potentially attractive replacement for selective H₂S removal approaches e.g., MDEA-based and direct conversion processes which are in commercial use now. In fact, results of a commercial test reported by Goldstein et al. (1986) showed 40% energy saving when the MDEA based H₂S-selective absorption process was replaced by Flexsorb SE. As of today, about twenty commercial units are using hindered amine based absorption processes (Gas Process Handbook'92) for CO₂ removal and for H₂S-selective and non-selective removal of H₂S and CO₂ from sour gas streams.

The introduction of hindered amines represents new advances in industrial gas treating. But, it will be evident from a review of

literature, presented in section 1.3, that in spite of the dimensions and importance of the new area represented by the hindered amines, published information on the kinetics and mechanism of absorption of CO_2 in these amines is very limited. With respect to simultaneous and selective absorption of H_2S and CO_2 in hindered amines the available information is even more scanty.

1.3 LITERATURE REVIEW ON ABSORPTION IN HINDERED AMINES

Information available in the open literature on acid gas absorption in hindered amines is limited. With the exception of Sartori and Savage (1983), who have reported results on gas absorption in 2-amino-2-methyl-1-propanol (AMP) and few other hindered amines as well, most of the other investigations reported so far have dealt with only AMP, the most important member of the hindered amine family.

Sharma (1965) has observed that steric effects influence the stability of the carbamates formed by the amines with CO_2 . From absorption studies in a laminar jet absorber he has found out a second order rate constant of $1045 \text{ m}^3/\text{kmol s}$ for the CO_2 - AMP reaction. Sharma (1964) has also proposed the use of highly branched amines such as 2-amino-2-methyl-1-propanol (AMP) for CO_2 absorption because he thought these amines, due to steric hindrance, could show considerable advantages over conventional amines with respect to cyclic absorption capacity .

Sartori and Savage (1978, 1983) have developed the concept of using hindered amines for gas sweetening processes. From thermodynamic point of view they have explained as to how steric hindrance and basicity control the amine- CO_2 reactions. Sartori and Savage(1983)

have proposed that in aqueous amino alcohols steric hindrance is the dominant factor giving rise to high thermodynamic capacity and fast absorption rates at high CO_2 loading despite some reduction of the rate constant owing to steric hindrance. They have reported the results of vapor liquid equilibria studies of CO_2 -amino alcohol-water and CO_2 -amino alcohol-potassium carbonate-water systems performed in an autoclave under batch mode, between 313-393 K. In all the cases sterically hindered amines showed higher capacities for CO_2 than unhindered primary and secondary amines. Sartori and Savage (1983) and Sartori et al. (1987) have reported the carbamate stability constants determined by C^{13} NMR for hindered and conventional amines at 313K. For AMP the reported value of the carbamate stability constant was as low as less than 0.1 (Table 2.1, Chapter 2). From the measurements of the rates of absorption in a single sphere absorber they have showed that while the CO_2 absorption rates in conventional amines such as MEA, DEA and DIPA reduce drastically on approaching a loading of about 0.5 mol of CO_2 per mol of absorbent, hindered amines (e.g. AMP and PE) maintain appreciable absorption rates even at higher loadings. However, no detailed kinetic data have been presented .

Savage et al. (1984) have reported that some hindered amines, when used as promoters in the hot carbonate process, act as effective homogeneous catalysts for the CO_2 hydration reaction. It has also been reported that due to slow bicarbonate formation reaction hindered amines could show considerable selectivity for H_2S (Savage et al., 1985). However, no detailed work on this has been reported.

Chakraborty et al. (1986) have studied the absorption of CO_2 in AMP in a pressure decrease cell (PDC) and continuous flow cell (CFC) at 315 K. On the basis of C^{13} NMR spectra of liquid samples at

equilibrium, they have concluded the reaction to be amine catalyzed hydration of CO_2 , since they did not observe any carbamate peak in the spectrum. The rate data have been found out by them from the transient part of the absorption experiment in the PDC. For the CO_2 -AMP reaction they have reported the order with respect to both CO_2 and AMP as unity. The value of the second order rate constant have been found by them to be $100 \text{ m}^3/\text{kmol s}$ at 315 K. This value of the rate constant as found out by Chakraborty et al. (1986) is lower than the value reported by Savage et al. (1984) for hindered amine promoted hot carbonate process. But Chakraborty et al. (1986) have attributed this difference, to the difference in absorption temperature which was 363 K or higher in case of Savage et al. (1984).

Ziodas and Dadach (1986) have measured the absorption rates of CO_2 and H_2S into quiescent solutions of MEA and AMP in a cell at 298 K. The results of the experiments have been presented graphically as the total amount of gas absorbed versus time. They have correlated the gas absorption rates with the partial pressure of CO_2 and the amine concentration by a power law relation. The difference in behaviour between the two amines has been attributed to the steric hindrance in AMP, but no quantitative explanation or detailed kinetic data have been presented.

Yih and Shen (1988) have investigated the kinetics of the CO_2 -AMP system by gas absorption in a wetted wall column at 313 K. They have analyzed their results using the methodology of "gas absorption with fast pseudo-first-order reaction". They have found out the order with respect to both CO_2 and AMP for the CO_2 -AMP reaction as unity. The second order reaction rate constant has been reported by them as $1270 \text{ m}^3/\text{kmol s}$ at 313 K. They have concluded that the carbamate reaction

may still have significant effect on the overall CO_2 -AMP reaction even though the value of the carbamate stability constant as reported by Sartori et al. (1987) is very low.

Bosch et al. (1989) have analyzed the results of Chakraborty et al. (1986) and Zlodos and Dadach (1986) based on the numerical method developed by Versteeg et al. (1989) for parallel reversible reactions. They have demonstrated that absorption of CO_2 in hindered amines can be explained in terms of the established mechanism for the reactions of CO_2 with conventional alkanolamines. According to them no new reaction scheme is necessary to explain the observed behaviour. Bosch et al. (1990) have also studied the absorption of CO_2 into aqueous solutions of AMP under reaction controlled conditions in a stirred vessel at 298 K. They have observed that the CO_2 -AMP reaction could be described according to the generally accepted zwitterion mechanism. According to them, to derive the kinetics from absorption experiments it is necessary to use a numerically solved absorption model which describes absorption accompanied by several parallel chemical reactions and it is not possible to determine the kinetics in the well known fast reaction regime with the assumption of pseudo-first-order reaction derived for irreversible reactions.

On the basis of his results, Alper (1990) has speculated that the CO_2 -AMP reaction proceeds according to the accepted zwitterion mechanism (reactions (2.2) and (2.3) presented in Chapter 2) to form carbamate ion with the zwitterion formation reaction being possibly the rate controlling step. The carbamate ion is then hydrolyzed into bicarbonate ion (reaction (2.4) in Chapter 2) so that the final reaction mixture has no or little carbamate ion. This reconciles the finding of Chakraborty et al. (1986) through C^{13} NMR spectra of

CO₂-AMP equilibrium mixtures. To avoid the confusion of mass transfer in gas absorption, Alper (1990) has used the stopped flow technique to study the kinetics of the homogeneous reaction between aqueous solutions of CO₂ and AMP. On the basis of the results Alper (1990) has proposed a correlation for the second order reaction rate constant k_2 as a function of temperature. The corresponding value of the activation energy has been found to be 41.7 kJ/mol. Alper's predicted value of 1165 m³/kmol s for k_2 at 313 K agrees well with 1270 m³/kmol s reported by Yih and Shen (1988) at the same temperature. However, the k_2 value of 520 m³/kmol s predicted by Alper (1990) from the correlation at 298K, is somewhat smaller than 1048 m³/kmol s at 298K reported by Sharma (1965).

About an equal number of work as that of kinetic studies discussed above has been reported in the open literature on the thermodynamic and physico chemical properties of CO₂-AMP, H₂S-AMP and CO₂-H₂S-AMP systems as discussed below.

Roberts and Mather (1988) have presented data for equilibrium chemical solubility of H₂S and CO₂ in 2 kmol/m³ aqueous AMP solution at 373 K and up to a partial pressure of 2200 kPa and 6000 kPa for H₂S and CO₂ respectively. Teng and Mather (1989) have reported equilibrium chemical solubility data of H₂S, CO₂ and their mixtures in 3.43 kmol/m³ aqueous solution of AMP at 323 K. The partial pressure of the gas in their work has ranged between 4 and 5650 kPa. A mathematical model has also been developed to correlate the solubility data for individual gas as well as the influence of the presence of one acid gas on the solubility of the other. Teng and Mather (1990) have measured the solubility of CO₂ in 2 kmol/m³ aqueous solution of AMP at 313 K and 343 K at CO₂ partial pressures between 0.2 and 5279 kPa.

Tontiwachwuthikul et al. (1991) have measured the equilibrium chemical solubility of CO_2 in 2 and 3 kmol/m^3 aqueous solutions of AMP at 293, 313, 333 and 353 K and for CO_2 partial pressures ranging from approximately 1 to 100 kPa. They have interpreted their results by a modified Kent-Eisenberg model and showed that AMP solutions are superior to MEA solutions for the regenerative absorption of CO_2 because the CO_2 solubilities in AMP solutions are higher than those in MEA solutions at 313 K (at which absorption is normally done) but lower at 353K (which is closer to regeneration temperature), while the cross over takes place at about 333 K.

Xu et al. (1991) have estimated the physical solubility and diffusivity of CO_2 in 2 and 3 kmol/m^3 aqueous AMP solutions using " N_2O Analogy". N_2O analogy has also been used by Saha et al. (1993) to estimate the physical solubility and diffusivity of CO_2 in 0.5, 1.0, 1.5 and 2.0 kmol/m^3 aqueous solutions of AMP .

Erga and Lidal (1991) have developed a semi-empirical gas-liquid equilibrium model for CO_2 absorption in aqueous solution of AMP, based on their own experimental data.

Very recently Tontiwachwuthikul et al. (1992) have reported the absorption data of CO_2 in NaOH, MEA and AMP solutions in a packed column absorber (0.1 m i.d. column packed with 12.7 mm Berl Saddles up to a height of 6.55 m operated in counter current mode and near ambient conditions). They could not interpret the data of CO_2 -AMP system because of lack of physico-chemical and kinetic data of CO_2 -AMP system.

With respect to selective and non-selective simultaneous absorption of H_2S and CO_2 into solutions of hindered amines, available information is very scanty. Only a few literature is available

concerning the commercial test results and industrial applications of hindered amine-based solvents. Say et al. (1984), Goldstein et al. (1986) and Sartori et al. (1987) have reported the results of commercial tests of hindered amine based solvents, Flexsorb SE and Flexsorb PS, developed by Exxon Research and Engineering Company for selective and non-selective simultaneous absorption of H_2S and CO_2 . Say et al. (1984) and Goldstein et al. (1986) have claimed significant advantage of using Flexsorb SE in place of MDEA-based solvent for H_2S -selective absorption, while Flexsorb PS has been claimed to have resulted in 30% reduction in the liquid circulation rate compared to conventional amine solvent systems for the removal of both CO_2 and H_2S from sour gas streams.

1.4 OBJECTIVES OF THE PRESENT WORK

From the foregoing discussion it is evident that not much work has been done on the kinetics of CO_2 -AMP system in spite of its immense importance in industrial gas treating. Moreover, the agreement between the limited kinetic data, that are available, cannot, in general, be considered satisfactory. For instance, the reported values of the second order rate constant vary as widely as from $100 \text{ m}^3/\text{kmol s}$ at 313 K (Chakraborty et al., 1986) to $1270 \text{ m}^3/\text{kmol s}$ at the same temperature (Yih and Shen, 1988). Furthermore, it appears that there is a confusion about the exact reaction mechanism, since some of the authors presume no carbamate formation (Yih and Shen, 1988), while Alper (1990) has considered carbamate formation and subsequent hydrolysis of the carbamate ion into bicarbonate ion. On the other hand, Chakraborty et al. (1986) have presumed the reaction to be the

hydration of CO_2 which is catalyzed by AMP as in the case of tertiary amines. It was, therefore, thought desirable to undertake this work to investigate the kinetics of the CO_2 -AMP system in detail to resolve the contradictory information reported so far. In this work the kinetic studies, based on gas absorption experiments are presented in Chapter 3. The kinetic data on CO_2 -AMP system presented in Chapter 3 have been used to model the absorption of CO_2 in a mechanically agitated contactor. This is presented in Chapter 5.

Diffusivity and solubility data of CO_2 in aqueous AMP solutions at different temperatures were needed to analyze the results of absorption studies and also for the modeling work. These data have been determined by using " N_2O Analogy" (Saha et al., 1993). Determination of these properties is presented in Chapter 4.

With respect to simultaneous absorption of H_2S and CO_2 in hindered amines, available information is even more scanty although hindered amine based processes for H_2S -selective and non-selective simultaneous removal of H_2S and CO_2 have already been commercialized. An exploratory piece of work to study the H_2S -selective simultaneous absorption of H_2S and CO_2 into solutions of AMP was, therefore, undertaken to make a first-hand evaluation of AMP as a possible selective absorbent for H_2S in presence of CO_2 . Chapter 6 describes this exploratory work.

Another exploratory work, taken up and presented here (Chapter 7), involved the study of the effect of fine particles of activated carbon, present as a third phase in the gas-liquid system, on the absorption of CO_2 in aqueous solutions of AMP and other conventional alkanolamines e.g. MEA and DEA. It has been reported previously that activated carbon particles, when present as a third phase, can

significantly enhance the physical absorption rates of O_2 and CO_2 into water and chemical absorption rates of O_2 and CO_2 in Na_2S and $Na_2CO_3-NaHCO_3$ solutions respectively (Alper et al., 1980; Alper and Öztürk, 1986; Qulcker et al., 1989). But hardly anything has been reported so far about the effect of activated carbon particles on the rates of absorption of CO_2 into aqueous amine solutions. This piece of work was, therefore, directed towards making a preliminary assessment of activated carbon particles as absorption rate enhancing agents for CO_2 -amine systems (Saha et al., 1992). However, scope of these two exploratory studies, presented in Chapters 6 and 7, are essentially restricted to finding out future research directions in absorption of acid gases in AMP and other alkanolamines.

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