Selective Absorption of H_2S from Gas Mixtures Contains CO_2 into New Sterically Hindered Amine DTBP for Natural Gas Purification

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Abstract. In this work, an experimental investigation for selective absorption of H_2S from gas mixtures into aqueous solutions of new sterically hindered amine for natural gas purification at atmospheric pressure was performed. The new sterically hindered amine with two tertiary butyl alkyls respectively attaches to two N atoms was called DTBP synthesized and characterized by IR and 1HNMR. The effect of concentration and temperature of solutions on absorption performance were investigated by means of the selectivity factor and sour gas load. The performance of simultaneous absorption of CO_2 and H_2S in to the aqueous solution of DTBP is compared with that of the aqueous solutions of MDEA and AMP. The aqueous solution of DTBP has been found to be an efficient solvent for selective H_2S removal. The experimental results also testified the advantages of severe sterically hindered amines (e.g., DTBP and AMP) over traditional amines in selective H_2S absorption. This work provides the results for sterically hindered amine to be extensively applied in the field of selective H_2S removal.

1. Introduction

Natural gas purification is a very important part in the natural gas industry. The removal of H_2S and CO_2 from industrial gas streams is a significant operation in natural gas processing, hydrogen purifying, refinery off-gas treating and synthesis gas for ammonia and methanol manufacturing. The exploited natural gas composition is very complicated, and most of which contain hydrogen sulfide and carbon dioxide.

At present, one of the methods that the natural gas commonly used for purification is alcohol amine method. Methyldiethanolamine (MDEA) has been widely used in production for its selectivity of H_2S, good desulfurated efficiency and not easy foaming [1].

However, the content of H_2S and CO_2 are low in many gas fields, especially the content of CO_2 is under standard of commodity natural gas in China (< 2%). But the proportion of CO_2/H_2S is high (more than 10, even more than 100). In view of this kind of situation, it's necessary to fully strengthen the solvent selected absorption function in order to reduce energy consumption and

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Improve the quality of sour gas into the sulfur recovery unit on condition of ensure purification degree of H_2S.

Since the mid-80s of 20th century, lots of research on selective desulfurizing solvent has been developed [2]. In this paper, sterically hindered amine which is a kind of selectivity desulfurizing solvent is researched. Recently, Exxon Research and Engineering Company have developed a kind of sterically hindered amine-based H_2S-selective gas treating processes. The processes have been commercially applied. They claimed that the new hindered amine-based processes would be potentially attractive replacements for the existing selective H_2S removal processes including MDEA based and direct conversion processes [3].

Sterically hindered amine belong to the compounds that one or two replace groups attached to the nitrogen atoms in their molecules, which can produce sterically hindrance effect of the new type of organic amine [4]. The effect makes amino-group more chemical activation[5]. For example, 2-amino-2-methyl-1-propanol(AMP), menthane diamine(MDA) and 2-piperidinealcohol(PE).

Since the sterically hindered amine was reported, many of the researches have been studied by domestic and foreign scholars. Presented a process development work using hindered amine as the promoter of hot carbonate solutions for simultaneous absorption of H_2S and CO_2 Say et al. [6]. Numerically interpreted the simultaneous mass transfer of CO_2 and H_2S into aqueous blends of MDEA and DEA Rascol et al. [7]. The work of Mandal presented an experimental and theoretical investigation of the simultaneous absorption of CO_2 and H_2S into aqueous blends of AMP and DEA [8]. Simulated selective H_2S absorption in aqueous amine solutions using a rate-based mode Nadhir et al. [9] and Markus et al. [10]. However, in the current literature, studies on a sterically hindered amine, especially a severe hindered amine (e.g., AMP) for high selective removal of H_2S from gas streams containing CO_2 and H_2S are rarely reported.

This work synthesized a kind of new sterically hindered amine with two tertiarybutyl alkyls respectively attaches to two N atoms which called DTBP. H_2S-selective absorption into the aqueous solution of MDEA, AMP and DTBP were investigated. The experiments dealt with the system of simultaneous absorption of tri-component gases (CO_2, H_2S, N_2) into the aqueous solution of absorbents (MDEA, AMP, DTBP) in a absorber at atmospheric pressure in this work. The effects of temperature, amine concentration were studied. Furthermore, experimental data from the solution of BTAP was compared with that from the aqueous solution of MDEA and AMP. Sterically hindered amine selectivity of H_2S(S) was determinated. The emphasis of this work focused on the selective removal of H_2S from gas mixtures containing both CO_2 and H_2S using the DTBP aqueous solution.

2. Theory

2.1. Characteristics of molecular structure of sterically hindered amines

Amines are commonly subdivided into primary, secondary and tertiary amines according to the number of alkyl groups attached to N atom in the molecule of amines. MDEA belongs to tertiary amines, AMP belongs to primary amine and DTBP belong to secondary amines, and their molecule structures are shown in Figure 1.

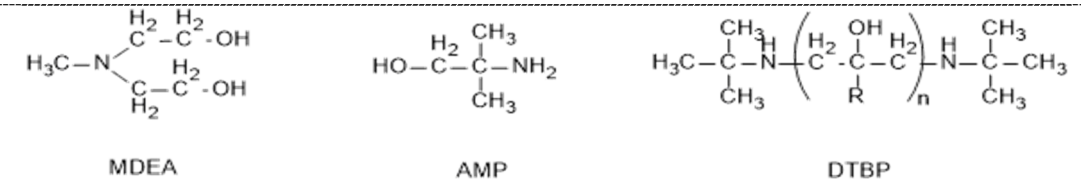


Figure 1. Molecule structure of MDEA, AMP and DTBP.

A nonlinear alkyl group, tertiary butyl alkyl group, attaches to N atom in AMP molecule, and two tertiary butyl alkyls respectively attaches to two N atoms in DTBP molecule. R stands for H atom or methyl molecule and n is greater than or equal to 1. It occupies bulky volume in space, and hinders activity of amino-group. The hindrance of nonlinear alkyl group gives amino-group of AMP and DTBP rise to higher chemical activation than that of non-hindered amines. The chemical activation is the so-called sterically hindered effect.

2.2. Reaction mechanism

The reaction mechanism for systems involving H_2S, MDEA, AMP, DTBP is as follows:

$$H_2S(g) \leftrightarrow H_2S(l) \quad H_2S \text{ phase change} \tag{1}$$

$$2H_2O \leftrightarrow OH^- + H_3O^+ \quad \text{dissociation of } H_2O \tag{2}$$

$$H_2S + H_2O \leftrightarrow H_3O^+ + HS^- \quad \text{dissociation of } H_2 \tag{3}$$

$$HS^- + H_2O \leftrightarrow H_3O^+ + S^{2-} \quad \text{dissociation of } HS^- \tag{4}$$

$$MDEAH^+ + H_2O \leftrightarrow H_3O^+ + MDEA \tag{5}$$

$$AMPH^+ + H_2O \leftrightarrow H_3O^+ + AMP \tag{6}$$

$$DTBPH^+ + H_2O \leftrightarrow H_3O^+ + DTBP \tag{7}$$

The reaction rate constants *k* values of Eq. (5), (6) and (7) are more than 10^9L/(mol·s) (25°C)[11]. These reactions are completed instantaneously. according to the theory of double film of gas-liquid mass transfer, everywhere in the liquid phase as well as the interfacial liquid film, H_2S-amine equilibriums exist always [12].

But the reaction of alcohol amine and carbon dioxide have a very different situation.

Carbon dioxide reaction with AMP and DTBP is as follows：

$$CO_2 + H_2O + RNH_2 = RNH_3^+ + HCO_3^- \tag{8}$$

$$-CH_2 - OH + OH^- + CO_2 = COCOO^- + H_2O \tag{9}$$

$$CO_2 + R_1R_2NH \quad + H_2O = R_1R_2NH_2^+ + HCO_3^- \tag{10}$$

$$CO_2 + 2R_1R_2NH \quad = R_1R_2NH_2^+ + RNHCOO^- \tag{11}$$

The overall reaction is

$$CO_2 + R_1R_2NH + H_2O = R_1R_2NH_2^+ + HCO_3^- \tag{12}$$

For the sterically hindered amine (such as AMP and DTBP), its carbamate is very unstable and the reaction mechanism indicates that the ultimate product of the reaction is bicarbonate.

For the MDEA, because it is a kind of tertiary amine, CO_2 cannot directly react with MDEA to form carbamate, but with H_2O to form bicarbonate. Representative reactions are following:

$$CO_2 + H_2O = H^+ + HCO_3^- \tag{13}$$

$$R_1R_2R_3N + H^+ = R_1R_2R_3NH^+ \tag{14}$$

The overall reaction is

$$CO_2 + H_2O + R_1R_2R_3N = R_1R_2R_3NH^+ + HCO_3^- \tag{15}$$

Comparing Eq. (12) with Eq. (15), it shows that the ultimate products of MDEA, AMP and DTBP reaction with CO_2 are all bicarbonate. The reaction rate constants k values of Eq. (12) and (15) are less than 7 L/(mol·s) (25°C) [13]. The liquid-film is dominant resistance of CO_2 mass transfer [14].

This shows that the reaction of H_2S and CO_2 are different in kinetics. This is the theoretical foundation of severe hindered amine (such as AMP and DTBP) can using as the absorbent to attain higher H_2S selectivity.

2.3. Evaluation of absorption performance

L is sour gas load. The value of L_{H_2S} and L_{CO_2} are calculated by following equation:

$$L_{H_2S} = \frac{x_{H_2S}}{x_{amine}} \tag{16}$$

$$L_{CO_2} = \frac{x_{CO_2}}{x_{amine}} \tag{17}$$

S is selectivity factor of sterically hindered amine. Selectivity of amine solvents for H_2S may be defined as the tendency for the ratio of H_2S to CO_2 contents to be larger in the liquid phase than it is in the gas phase. Selectivity factor is used as a yardstick for the H_2S selectivity [15]. The value of selectivity factor equates the ratio of H_2S/CO_2 in liquid-phase to H_2S/CO_2 in gas-phase and is expressed as:

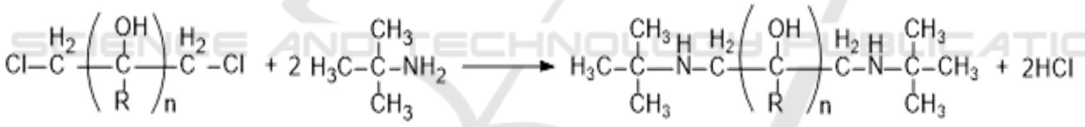
$$S = \frac{x_{H_2S}/x_{CO_2}}{y_{H_2S}/y_{CO_2}} \tag{18}$$

Where x is mole fraction of component i in the liquid bulk. *y* is mole fraction of component i in the gas bulk.

3. Experimental

3.1. Synthesis experiments of DTBP

Reaction equation is given as:



(19)

Tert-butylamine reacted with chlorinated alcohol in ratio of 4:1, absolute ethyl alcohol 50% from the total content of reactants was added as an azeotropic solvent. The mixture was heated at 120°C with continuous stirring for 4 hours. Poured out after cooling, add a certain amount of NaOH, reflux reaction under 70 °C for 50 min. Keep the filtrate after the suction filter, then vacuum distillation and cooling crystallization.

The chemical structure was confirmed by the infrared absorption spectroscopy (FTIR) and nuclear magnetic resonance (NMR) test. The results are shown in figure 2 and figure 3.

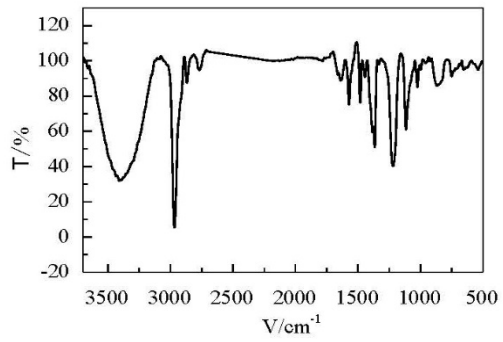


Figure 2 The infrared spectra of DTBP.

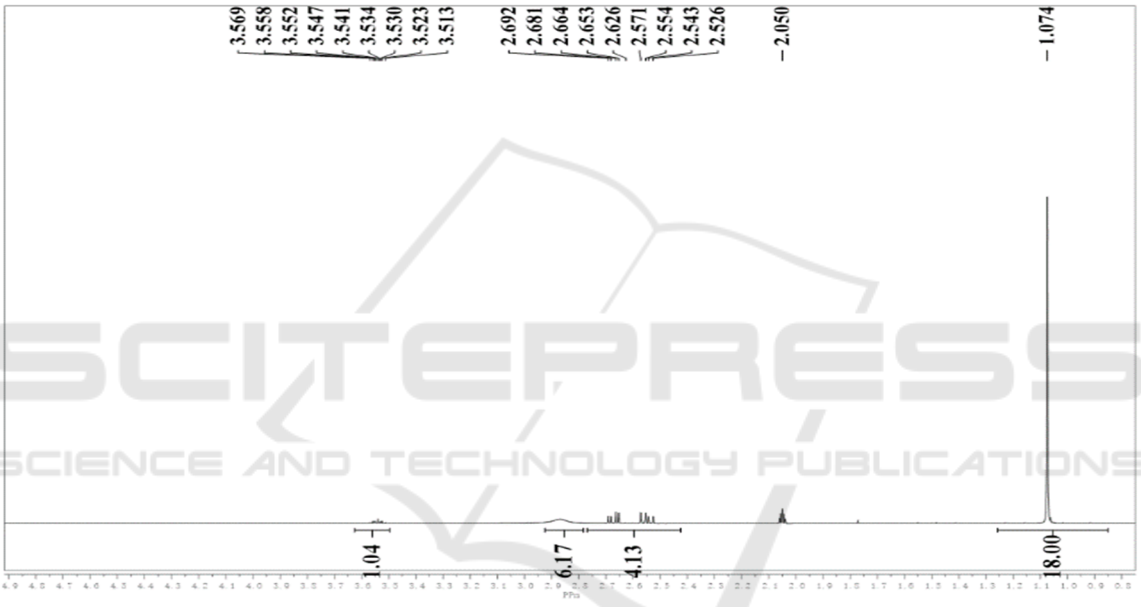


Figure 3 The Nuclear magnetic resonance spectra of DTB.

3.2. The experiment of H_2S selective evaluation of MDEA, AMP and DTBP

MDEA(clear colorless liquid with a purity of ≥ 99 mass %) and AMP (clear colorless liquid with a purity of ≥ 99 mass %) were supplied by Shanghai Aladdin biochemical Polytron Technologies Inc. DTBP (clear colorless liquid with a purity of ≥ 99 mass %) is for this work synthesis. The gas cylinder was supplied by Chemical Gas Company with the mole fraction purity of 10% CO_2, 1%H_2S and 89% N_2. Distilled H_2O was produced using Europe ultra water equipment.

As shown in figure 4, the absorption/desorption apparatus was used to determine the selective absorption of H_2S from gas mixtures into solution of MDEA, AMP and DTBP for the systems studied.

4. Results and discussions

This paper investigated the acid gas which has high carbon sulfur ratio (n_{CO_2}: $n_{H_2S} = 10:1$). The desulfurization performance of amine liquid (MDEA, DTBP, AMP) which a concentration of 10, 20,

30, 40, 50% have been tested at 20 to 60°C and compared with the same conditions of MDEA solution. The experimental results are shown in table 1 to table 3.

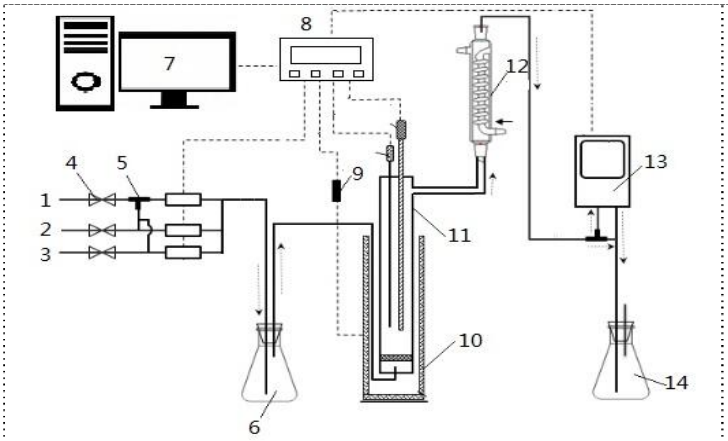


Figure 4. Schematic diagram of the experimental setup: 1, H_2S input unit; 2, CO_2 input unit; 3, N_2 input unit; 4, cut-off valve; 5, three-way valve; 6, buffer tank; 7, Record and control unit; 8, Data acquisition unit; 9, temperature sensor; 10, micro controlled heating furnace; 11, absorption/desorption reactor; 12, condenser pipe; 13, gas analyzer; 14, alkali liquor absorption tank..

Table 1. The results of different concentrations of MDEA solution absorption acid gas at different temperature

T (°C)	w(MDEA) (%)	x_{H_2S} (g/L)	x_{CO_2} (g/L)	y_{H_2S} (%)	y_{CO_2} (%)	L_{H_2S}	L_{CO_2}	L	S
20	10	0.1049	0.2558	1.0061	9.9886	0.0637	0.1552	0.2188	4.07
20	20	0.1978	0.4907	1.0018	10.0041	0.0600	0.1488	0.2088	4.03
20	30	0.2552	0.6472	0.9957	9.9926	0.0516	0.1309	0.1825	3.96
20	40	0.3390	0.8613	0.9986	9.9980	0.0514	0.1306	0.1820	3.94
20	50	0.4329	1.1246	1.0002	10.0021	0.0525	0.1364	0.1890	3.85
30	10	0.1023	0.2559	0.9977	10.0018	0.0621	0.1553	0.2173	4.01
30	20	0.1831	0.4565	1.0027	9.9980	0.0555	0.1385	0.1940	4.00
30	30	0.2542	0.6364	0.9993	9.9975	0.0514	0.1287	0.1801	4.00
30	40	0.3188	0.8122	0.9982	10.0015	0.0483	0.1232	0.1715	3.93
30	50	0.4013	1.0259	1.0037	9.9993	0.0487	0.1245	0.1732	3.90
40	10	0.0939	0.2357	0.9977	10.0018	0.0570	0.1430	0.2000	3.99
40	20	0.1766	0.4504	1.0027	9.9980	0.0536	0.1366	0.1902	3.91
40	30	0.2420	0.6315	0.9993	9.9975	0.0489	0.1277	0.1766	3.83
40	40	0.3058	0.7883	0.9982	10.0015	0.0464	0.1196	0.1659	3.89
40	50	0.3565	0.9407	1.0037	9.9993	0.0433	0.1141	0.1574	3.78
50	10	0.0807	0.2031	1.0004	9.9956	0.0490	0.1232	0.1722	3.97
50	20	0.1530	0.3974	0.9963	10.0020	0.0464	0.1205	0.1669	3.87
50	30	0.2347	0.6081	0.9973	10.0042	0.0475	0.1230	0.1704	3.87
50	40	0.2723	0.6959	0.9990	9.9958	0.0413	0.1056	0.1468	3.91
50	50	0.3216	0.8386	0.9991	9.9999	0.0390	0.1017	0.1408	3.84
60	10	0.0758	0.1953	0.9984	10.0037	0.0460	0.1185	0.1645	3.89
60	20	0.1243	0.3253	0.9969	10.0042	0.0377	0.0987	0.1364	3.84
60	30	0.1774	0.4610	1.0010	9.9987	0.0359	0.0932	0.1291	3.84
60	40	0.2347	0.6221	1.0037	10.0038	0.0356	0.0943	0.1299	3.76
60	50	0.2760	0.7453	0.9986	10.0004	0.0335	0.0904	0.1239	3.71

Table 2. The results of different concentrations of AMP solution absorption acid gas at different temperature

T (°C)	w(AMP) (%)	x_{H_2S} (g/L)	x_{CO_2} (g/L)	y_{H_2S} (%)	y_{CO_2} (%)	L_{H_2S}	L_{CO_2}	L	S
20	10	0.1748	0.1511	0.9978	9.9989	0.1433	0.1237	0.2671	11.60
20	20	0.3314	0.2862	1.0017	9.9966	0.1358	0.1173	0.2531	11.56
20	30	0.4897	0.4237	1.0021	9.9956	0.1338	0.1158	0.2496	11.53
20	40	0.6347	0.5551	0.9957	10.0006	0.1301	0.1137	0.2438	11.49
20	50	0.8056	0.7011	1.0024	9.9984	0.1321	0.1149	0.2470	11.46
30	10	0.1636	0.1418	0.9982	9.9970	0.1341	0.1162	0.2503	11.55
30	20	0.3255	0.2813	0.9992	10.0040	0.1334	0.1153	0.2487	11.58
30	30	0.4567	0.3960	0.9985	10.0043	0.1248	0.1082	0.2329	11.56
30	40	0.5997	0.5270	1.0017	9.9968	0.1229	0.1080	0.2309	11.36
30	50	0.7462	0.6551	1.0041	10.0004	0.1223	0.1074	0.2297	11.35
40	10	0.1589	0.1377	0.9991	9.9963	0.1303	0.1129	0.2432	11.54
40	20	0.3005	0.2605	0.9969	10.0027	0.1232	0.1068	0.2300	11.57
40	30	0.4359	0.3789	0.9975	9.9991	0.1191	0.1035	0.2226	11.53
40	40	0.5806	0.5044	1.0040	9.9956	0.1190	0.1034	0.2224	11.46
40	50	0.7242	0.6311	1.0033	9.9964	0.1187	0.1035	0.2222	11.43
50	10	0.1503	0.1310	1.0027	9.9978	0.1231	0.1074	0.2305	11.43
50	20	0.2776	0.2442	0.9992	10.0030	0.1137	0.1001	0.2138	11.37
50	30	0.3997	0.3526	1.0000	9.9999	0.1092	0.0963	0.2055	11.34
50	40	0.5306	0.4691	0.9983	10.0025	0.1087	0.0961	0.2048	11.34
50	50	0.6741	0.5944	0.9989	9.9982	0.1105	0.0974	0.2080	11.35
60	10	0.1334	0.1173	1.0037	10.0019	0.1094	0.0961	0.2055	11.35
60	20	0.2501	0.2208	1.0019	9.9981	0.1025	0.0905	0.1930	11.30
60	30	0.3604	0.3198	0.9958	9.9986	0.0985	0.0874	0.1858	11.32
60	40	0.4803	0.4274	0.9995	10.0011	0.0984	0.0876	0.1860	11.24
60	50	0.5914	0.5278	1.0039	9.9993	0.0969	0.0865	0.1835	11.16

From table 1to table 3 it can be seen that MDEA, DTBP, AMP of H_2S and CO_2 absorption load is reduced as the temperature increases, and the total acid gas load is reduced; with the increase of concentration of amine liquid, acid gas load is also decreased.

Analysis the load of H_2S, CO_2 and total acid gas load of MDEA, AMP and DTBP under different concentration at 40°C. The results are shown in Figure 5.

From figure 5 it can be seen that as the concentration of amine increased, the content of L_{H_2S}, L_{CO_2} and L kept less decreased. L_{H_2S}(DTBP)> L_{H_2S}(AMP)> L_{H_2S}(MDEA), L_{CO_2}(MDEA)> L_{CO_2}(DTBP)> L_{CO_2}(AMP) and L(DTBP)>L(AMP)>L(MDEA) at amine solution concentration of 10%~40% at 40°C. Meanwhile, The same results can be obtained at 20~60°C. Under the same concentration and temperature, H_2S load of AMP and DTBP are greater than the MDEA, L_{H_2S}(DTBP) is the biggest of the three amine and L_{H_2S}(DTBP) is about 3 times than L_{H_2S}(MDEA). But L_{CO_2}(MDEA) is the biggest of the three amine, L_{CO_2}(DTBP) is closed to L_{CO_2}(AMP) at 40°C.

Table. 3 The results of different concentrations of DTBP solution absorption acid gas at different temperature

T (°C)	w(DTBP) (%)	x_{H_2S} (g/L)	x_{CO_2} (g/L)	y_{H_2S} (%)	y_{CO_2} (%)	L_{H_2S}	L_{CO_2}	L	S
20	10	0.1575	0.1210	0.9987	10.0028	0.1620	0.1244	0.2864	13.04
20	20	0.3100	0.2377	1.0040	10.0037	0.1594	0.1222	0.2816	12.99
20	30	0.4553	0.3504	1.0016	10.0043	0.1561	0.1201	0.2762	12.98
20	40	0.6048	0.4659	1.0040	9.9966	0.1555	0.1198	0.2753	12.92
20	50	0.7576	0.5895	0.9992	10.0028	0.1558	0.1213	0.2771	12.86
30	10	0.1430	0.1104	0.9993	9.9998	0.1471	0.1134	0.2606	12.98
30	20	0.2781	0.2145	1.0011	9.9979	0.1430	0.1103	0.2533	12.95
30	30	0.4068	0.3174	0.9964	10.0015	0.1395	0.1088	0.2483	12.87
30	40	0.5258	0.4101	1.0033	10.0044	0.1352	0.1054	0.2406	12.78
30	50	0.6354	0.5044	0.9975	9.9988	0.1307	0.1037	0.2344	12.63
40	10	0.1291	0.0994	0.9961	9.9971	0.1328	0.1022	0.2350	13.03
40	20	0.2420	0.1868	0.9977	9.9997	0.1244	0.0961	0.2205	12.98
40	30	0.3726	0.2872	1.0045	9.9991	0.1277	0.0984	0.2262	12.92
40	40	0.4807	0.3764	0.9971	10.0036	0.1236	0.0967	0.2203	12.81
40	50	0.6015	0.4716	1.0028	9.9992	0.1237	0.0970	0.2207	12.72
50	10	0.1137	0.0886	1.0010	10.0023	0.1169	0.0910	0.2079	12.83
50	20	0.2157	0.1687	0.9958	9.9970	0.1109	0.0868	0.1977	12.83
50	30	0.3123	0.2448	1.0040	10.0024	0.1070	0.0839	0.1909	12.71
50	40	0.4017	0.3163	0.9999	10.0008	0.1033	0.0813	0.1846	12.70
50	50	0.5001	0.3925	1.0025	10.0008	0.1028	0.0807	0.1836	12.71
60	10	0.0986	0.0780	0.9987	10.0037	0.1014	0.0802	0.1815	12.67
60	20	0.1941	0.1534	1.0028	9.9956	0.0998	0.0788	0.1786	12.62
60	30	0.2726	0.2165	1.0011	10.0000	0.0934	0.0742	0.1677	12.57
60	40	0.3392	0.2721	0.9985	9.9998	0.0872	0.0699	0.1571	12.49
60	50	0.4031	0.3251	0.9968	10.0045	0.0829	0.0669	0.1498	12.44

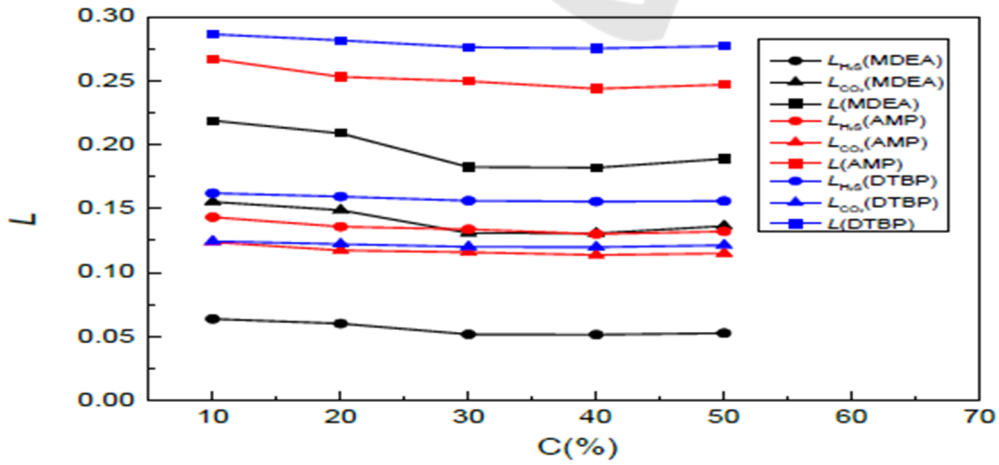


Figure 5. The L_{H_2S}, L_{CO_2} and L of different concentrations of MDEA, AMP and DTBP at 40°C.

Analysis the *S* of MDEA, AMP and DTBP under different concentration and temperature. The results are shown in figure 6 and figure 7.

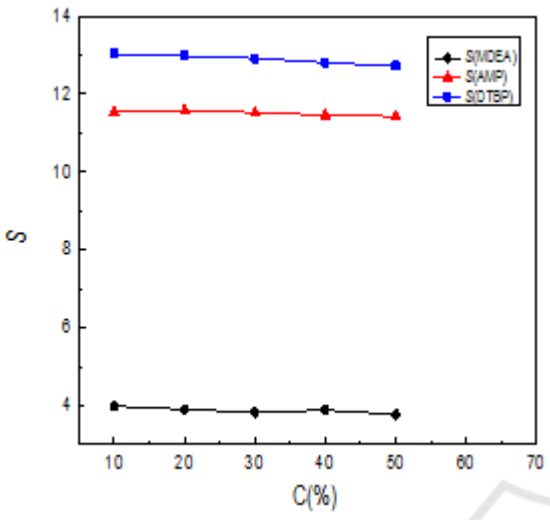


Figure 6. The *S* of different concentrations of MDEA, AMP and DTBP at 40°C

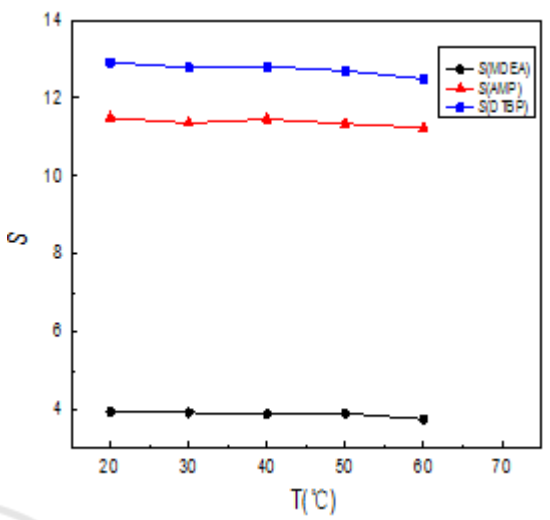


Figure 7. The *S* of 40% MDEA, AMP and DTBP at different temperature

Compared with the results in figure 6 and figure 7, the concentration and temperature had a much smaller effect on *S* . *S* of AMP and DTBP are greater than MDEA, *S* of DTBP is the biggest of the three amine.

5. Conclusions

In this work, a new type of sterically hindered amine DTBP was synthesized, and H_2S selective absorption from mixture gas into the aqueous solution of MDEA, AMP and DTBP have been studied. The absorption performance of DTBP solution was compared with MDEA and AMP solutions. The effect factors of temperature and the amine concentration on the performance were investigated. The experimental results testified the advantages of sterically hindered amines (eg., AMP and DTBP) over traditional amines in selective H_2S absorption.

MDEA, DTBP, AMP of H_2S and CO_2 absorption load is reduced as the temperature increases, and the total acid gas load is reduced; But with the increase of concentration of amine liquid, acid gas load is increased. Low absorption temperature and high concentration of amine are in favor of selection H_2S absorption. But selective (S) was little affected by temperature and concentration. The H_2S selectivity order is S(DTBP)>S(AMP)>S(MDEA), L_{H_2S}(DTBP)> L_{H_2S}(AMP)> L_{H_2S}(MDEA), L_{CO_2} (MDEA)> L_{CO_2} (DTBP)> L_{CO_2} (AMP), *L*(DTBP)>*L*(AMP)>*L*(MDEA) under the same absorption temperature and amine concentration. This suggests that the aqueous solution of DTBP is an efficient solvent for selective H_2S removal. The experimental results testified the advantages of severe sterically hindered (e.g., DTBP and AMP) over traditional amines (MDEA) in selective H_2S absorption. Sterically hindered amine like DTBP can be extensively applied in the field of selective H_2S removal.

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