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# Solubility and Spectral Studies of Dilute COS in Ethylene Glycol + Water System

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In this work, the solubility data have been measured for COS + $N_2$ mixture gases in the aqueous solutions of ethylene glycol in the range of (298.15-318.15) K. Measurements were carried out by a saturation method using a glass absorption apparatus, which were controlled at constant temperatures using a thermostatic circulation bath with Beckmann thermometer. The solubility data were obtained with relative uncertainties within ± 3.5 % for COS concentration in gas phase. The measurement showed that the addition of water to ethylene glycol enhanced the solubility of dilute COS compared with pure ethylene glycol and that the 50 % volume fraction of ethylene glycol in aqueous solution of ethylene glycol (mass fraction is 52.24 %) is a more reasonable composition used in absorption processes of COS. When conventional UV, FTIR, fluorescence and $^1$H NMR spectroscopic techniques were used for inspection of spectral changes of COS in aqueous solutions of ethylene glycol, the spectral results suggest that COS can interact with ethylene glycol by hydrogen bonding and interaction. The results of this work can be used to provide important data for the design and operation of the absorption and desorption processes of COS with potential industrial application of aqueous solutions of ethylene glycol.

**Key Words: Ethylene glycol, Carbonyl sulfide, Hydrogen bond, Solubility.**

## INTRODUCTION

The removal of the sulfur compounds, such as hydrogen sulfide ($H_2S$) and carbonyl sulfide (COS), from natural and synthesis gases is a significant operation in industrial processes. These compounds are removed because of a need to reach specifications imposed in gas transport industries and to respect strict environmental standards[1]. After COS is the main sulfur compound found in coal-derived gases and the removal of COS has been largely reported[2-6]. The hydrolysis of COS has been studied since the 1950s as a secondary reaction in viscose ripening[7] and for the removal of sulfide in industrial products and waste gas[8]. The hydrolysis of COS is used as a test for Bronsted basicity, because the first step of the base interaction involves the nucleophilic attack of the basic hydroxyl groups on the COS carbon atom[9-14]. The process has been studied over a widely buffered range (of pH 4-10)[15]. Especially, Philipp and Dautzedberg[7] and Donaldson and Nguyen[16] studied the hydrolysis of COS in water and the COS analogies with $CO_2$ chemical absorption, respectively. However, a reaction mechanism that described the COS absorption was not proposed until the work by Al-Ghawas *et al.*[17], which included the estimation of physicochemical properties for COS-MEDA absorption, at 288.15-313.15 K and MDEA concentrations up to a weight fraction of 0.3. Afterward, the COS absorption by MEDA aqueous solutions, using a stirred reactor, under saturated bulk conditions, was studied by Littel *et al.*[18,19]. Ulteriorly, Amaraene and Bouallou[20] experimentally measured the rates for COS absorption by MEDA aqueous solutions at 313-353 K. However, they do reveal that a rigorous model that consider the chemical the reactions could validate the reaction mechanism that is proposed.

Ethylene glycol is an important industrial solvent, which has been used in the absorption processes for $SO_2$ in the previous works[21-23] because of its favorable properties, such as low vapor pressure, low toxicity, low viscosity and low melting point. Ethylene glycol presents native hydrogen bonding sites so that the potential desorption characters are presented in the absorption processes of solutions dissolving gases. Knowledge of the solubility data for dilute COS (< 2000 ppmv) with ethylene glycol and aqueous solutions of ethylene glycol under low total pressures is an indispensable requirement for the design of absorption and desorption processes of gases containing COS. But the solubility data are very lack in the current data, so we have to determine the solubility data for dilute COS in aqueous solutions of ethylene glycol for the future industrial application of the solutions containing ethylene glycol.

In addition, FTIR spectroscopy, UV-VIS spectroscopy and fluorescence spectroscopy are very successful methods to probe the molecular structure of association effects among molecules, since the FTIR spectroscopy gives precise information about water sensitive bonds[24,25] and the UV and fluorescence spectroscopy gives important information about various electronic transitions. Generally, FTIR spectral technique[26,27], UV and fluorescence spectroscopic technique offer the advantages to measure the association properties and hydrogen bonding capability and to assess interaction of alcohol with water by analyzing band shifts and changes of band shape. Furthermore, FTIR is also advantageous to evaluate the vibrational properties of bonds through very thin solution films, which are usually difficult to handle for the floating properties of solution.

In this work, the solubility data for COS + $N_2$ in aqueous solutions of ethylene glycol at 298.15, 303.15, 308.15, 313.15 and 318.15 K and low pressures were reported to optimize the composition of aqueous solution of ethylene glycol for the future removal processes of COS. Meanwhile, the work was focused on investigating the possible interaction mechanism of aqueous solution of ethylene glycol with COS in the absorption processes of COS by FTIR, UV, fluorescence and $^1$H NMR spectroscopic techniques.

## EXPERIMENTAL

The certified standard mixture, (COS + $N_2$, in which COS volume fraction is $1.94 \times 10^{-3}$), purchased from the Beijing Zhaoge Co. Ltd. (Beijing, China), were employed to determine the solubility data for COS in aqueous solutions of ethylene glycol in this work. The analytical grade ethylene glycol was purchased from Beijing Reagent Company (Beijing, China). It was used after drying over molecular sieves (type 4A) and decompression filtration before measurements. The purity of the final samples, as found by gas chromatograph, was better than 99.4 %. The density of ethylene glycol at 298.15 K was found to be 1.1096 g cm$^{-3}$, in good agreement with the literature[28-30]. Bi-distilled water was used.

**Apparatus and procedure:** The method of solubility measurement was similar to that previously used[31-37]. It was carried out by the static method and the experimental apparatus used in this work is shown in Fig. 1. The experimental apparatus was also evacuated by using the vacuum pump in order to remove air from the system. The volume of the equilibrium cell was determined by pure water prior to the experiment. The accuracy of the volume measurement of the solvent was ± 0.0001. The degassed aqueous solution of ethylene glycol was introduced into the equilibrium cell and the volume of aqueous solution of ethylene glycol was determined from the weight and the density of the solvent mixture. COS/$N_2$ mixtures from the gas cylinder (9) were poured into the absorption system through switching the regulating valve K3 and were recycled through jacketed vessel (1) and gas circulatory pump (4) for whole recycling process. Liquid temperatures were registered on a standard thermometer (7) at different points and the temperatures do not vary more than 0.02 K. Total pressures were recorded on pressure meter (8). When no pressure lowering occurs after these operations, absorption equilibrium has been reached. The total pressure under the equilibrium state was recorded at the same time. COS concentrations in gas phase were determined by GC with FPD detector. After experiments were performed, the mixture gas was discharged out by switching the regulating valve K1 and K2 and passing buffer (10) and absorption apparatus (11) containing alkaline solution. The concentrations of COS in gas phase were determined by gas chromatograph on a 2 m × 3.2 mm Porapak Q packed column using an Agilent 6890N gas chromatograph (GC) and a FPD detector linked to a HP6890 workstation. In all case, the injections were repeated at least seven times and the average results were reported. In order to calibrate the GC FPD detector, the external standard method was used. Experiments were carried out at various temperatures kept at a constant temperature using CS 501 thermostated bath with Beckmann thermometer purchased from Huanghua Meter Factory (Hebei province, China) with ± 0.02 K and inspected using an accurate thermometer purchased from Fuqiang Meter Factory (Hebei province, China) with the precision of ± 0.02 K and the total pressure inspected by a pressure gauge purchased from Fuqiang Meter Factory (Hebei province, China) with ± 0.133 kPa, using COS + $N_2$ mixtures ($F_{COS} = 1.94 \times 10^{-3}$) in low pressures.

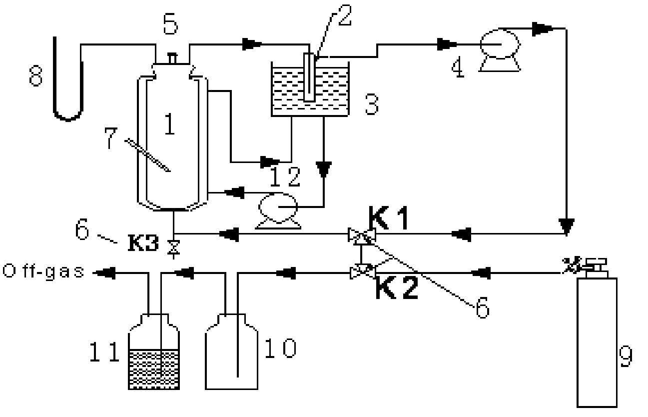


Fig. 1. Sketch of the experimental apparatus. (1) jacketed vessel; (2) cold trap; (3) thermostatic bath; (4) gas circulatory pump; (5) gas analysis outlet; (6) regulating valve (K1, K2 and K3); (7) thermometer; (8) pressure meter; (9) COS/$N_2$ gas cylinder; (10) buffer; (11) absorption apparatus; (12) liquid circulatory pump

The solubility of dilute COS in the liquid phase was calculated using the following equations:

$$p_1V_1 = n_1RT \tag{1}$$

where $p_1$ denotes the initial system pressure, $V_1$ denotes the initial gas volume, $n_1$ denotes the initial gas mole value and T denotes the measured temperature.

$$p_2(V_1 - V_2) = n_2RT \tag{2}$$

where $p_2$ denotes the system pressure under the absorption equilibrium, $V_2$ denotes the gas volume in the gas phase under the absorption equilibrium and $n_2$ denotes the gas mole value in the gas phase under the absorption equilibrium.

$$\Delta n = n_1 - n_2 = \frac{p_1V_1}{RT} - \frac{p_2(V_1 - V_2)}{RT} \tag{3}$$

where Δn denotes the absorption gas mole value in the whole absorption process.

$$\Delta n_{cos} = \frac{p_2V_1x_1 \times 10^{-6}}{RT} - \frac{p_2(V_1 - V_2)x_2 \times 10^{-6}}{RT}$$

$$= \frac{1}{RT} \times 10^{-6}[p_1V_1x_1 - p_2(V_1 - V_2)x_2] \qquad (4)$$

where $x_1$ denotes COS concentration in the gas phase before absorption and $x_2$ denotes COS concentration in the gas phase after absorption.

$$C_{COS} = \frac{\Delta n_{cos} \times M}{v_2}$$

$$= \frac{M}{RTV_2} \times 10^{-6}[p_1V_1x_1 - p_2(V_1 - V_2)x_2] \qquad (5)$$

where M is molar mass of COS and $C_{COS}$ is the absorption values of COS in aqueous solutions of ethylene glycol (mg/L).

FTIR spectra were recorded on a Bruker VECTOR22 FTIR spectrometer with a resolution of 1 $cm^{-1}$ at 298 K in the range from 4000-400 $cm^{-1}$. The spectrometer possesses auto-align energy optimization and a dynamically aligned interferometer and fitted with a constringent $CaF_2$ pellet for the measurement of aqueous solution, an OPUS/IR operator and IR source. A base line correction was made for the spectra that were recorded in air; and then 20 mL solution was used to performance on the FTIR spectrometer in every one of measurements and the thin layer of samples are less than typically 2 μm thickness. UV spectra were recorded using a Varian CARY 1E UV-VIS spectrometer with a resolution of 0.2 nm at room temperature in the region of 190-900 nm. A base line correction was made for the spectra recorded in deionized water. $^1$H NMR spectrum was recorded using a Bruker ARX-400 nuclear magnetic resonance and $(CH_3)_2SO$ (DMSO) was used as an NMR solvent. Fluorescence spectra were acquired using an F-4500 fluorescence spectrophotometer employing a 500 W Hg-Xe high pressure lamp. All spectral experiments of ethylene glycol + water + COS + $N_2$ were performed at normal temperature and pressure.

## RESULTS AND DISCUSSION

**Solubility data for aqueous solutions of ethylene glycol with dilute $SO_2$:** A series of solubility experiments of COS + $N_2$ in aqueous solutions of ethylene glycol (ethylene glycol (1) + $H_2O$ (2)) were performed at 298.15, 303.15, 308.15, 313.15 and 318.15 K and the absorption data are listed in Table-1. The solubility data were obtained with relative uncertainties within ± 3.5 % for COS concentration in the liquid phase. From the table, the solubility of dilute COS in $\Phi_1$ = 50 % aqueous solution of ethylene glycol (volume fraction of ethylene glycol in aqueous solution of ethylene glycol and mass fraction is 52.24 %) is the strongest in aqueous solutions of ethylene glycol.

**Spectral properties of $\Phi_1$ = 50 % aqueous solution of ethylene glycol + COS:** Table-1 shows that solubility of COS in $\Phi_1$ = 50 % aqueous solution of ethylene glycol is the strongest in various aqueous solutions of ethylene glycol. The interaction between aqueous solution of ethylene glycol and COS, which is related with the hydrogen bonding and interactions among molecules, plays a more important role in the absorption and desorption processes. Therefore, UV and fluorescence spectral changes were used to investigate the properties of bonding among ethylene glycol, $H_2O$ and COS.

UV spectral changes in the absorption processes of COS in $\Phi_1$ = 50 % aqueous solution of ethylene glycol were shown in Fig 2. The figure shows that the electronic transitions red shift from 197-202 nm with increasing COS concentration in aqueous solution of ethylene glycol. Meanwhile, the UV spectra of pure water with increasing COS concentration keep constant. In Fig. 2, the absorption band at 197-202 nm can be due to the n → σ* electronic transition of unshared electronic pair of hydroxyl oxygen in ethylene glycol[38] because the n →σ* electronic transition of $H_2O$ is often found at the vacuum ultraviolet

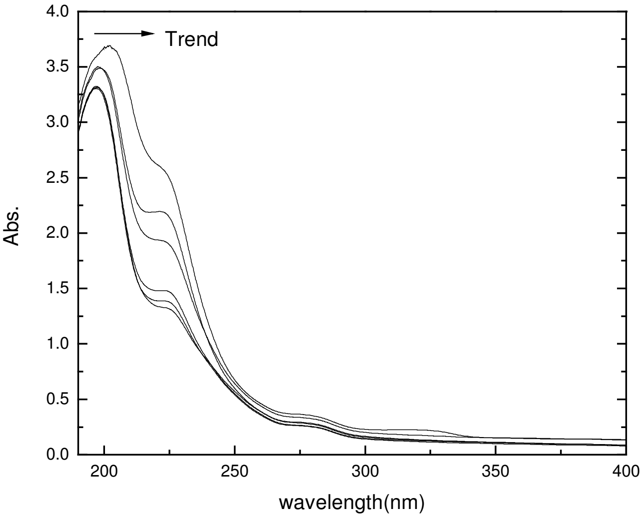


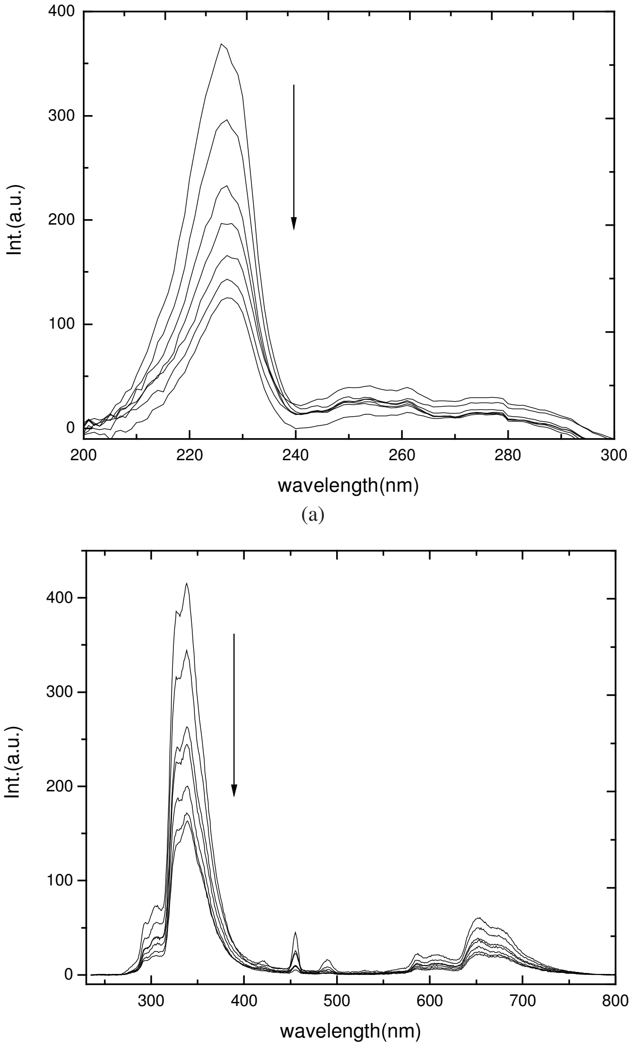
Fig. 2. Absorption spectral changes of $\Phi_1$ = 50 % aqueous solution of ethylene glycol with increasing COS concentration

TABLE-1
SOLUBILITY DATA FOR DILUTE COS IN ETHYLENE GLYCOL (1) + WATER (2) SYSTEM

| 100 $w_1$ (mass fraction of ethylene glycol in aqueous solution of ethylene glycol) | Solubility values of dilute COS in aqueous solution of ethylene glycol (mg $L^{-1}$) | | | | |
|---|---|---|---|---|---|
| | 298.15 K | 303.15 K | 308.15 K | 313.15 K | 318.15 K |
| 0.00 | 0.48 | 0.51 | 0.52 | 0.55 | 0.52 |
| 10.98 | 1.02 | 1.04 | 1.08 | 1.19 | 1.13 |
| 21.74 | 1.05 | 1.08 | 1.15 | 1.24 | 1.21 |
| 31.83 | 1.24 | 1.25 | 1.28 | 1.36 | 1.33 |
| 42.21 | 1.27 | 1.27 | 1.38 | 1.39 | 1.34 |
| 52.24 | 1.44 | 1.45 | 1.47 | 1.57 | 1.47 |
| 62.15 | 1.43 | 1.44 | 1.47 | 1.52 | 1.41 |
| 71.79 | 1.36 | 1.38 | 1.44 | 1.50 | 1.41 |
| 81.33 | 1.21 | 1.27 | 1.29 | 1.31 | 1.25 |
| 90.75 | 1.11 | 1.13 | 1.15 | 1.21 | 1.12 |
| 100.00 | 1.03 | 1.11 | 1.13 | 1.17 | 1.09 |

region, so the spectral shift can be due to the interaction of ethylene glycol with COS. In the other words, with increasing COS concentration, hydrogen bonding and interaction of hydroxyl hydrogen of ethylene glycol with oxygen of COS happened easily so that the n → σ* electronic transition of hydroxyl oxygen in ethylene glycol become easier.

Futhermore, the binding character of aqueous solution of ethylene glycol with COS can be distinguished by examining of the absorption spectrum of the fluorophore. The varieties of the fluorescence excitation and emission spectra of $\Phi_1 = 50$ % aqueous solution of ethylene glycol caused by gradually increasing the concentration of COS were shown in Fig. 3. Fig. 3(a) shows that a strong fluorescence excitation spectra of ethylene glycol at 228 nm in the range of 200-240 nm was observed at an emission wavelength of 307 nm; meanwhile, from Fig. 3 (b) a strong fluorescence emission spectra of ethylene glycol at 307 nm in the range of (300-400) nm was observed at an excited wavelength of 228 nm. The fluorescence emission and excitation spectral intensities of ethylene glycol decreased with increasing of COS concentration (Fig. 3), but not altering the emission maximum and shape of the peaks. These results show that there are the interaction of ethylene glycol with COS.



(a)

(b)

Fig. 3. Fluorescence excitation (a) and emission (b) spectral changes of aqueous solution of ethylene glycol with increasing COS concentration

According to overall results, it is expected that the solubility of COS is mainly due to the binding and interaction of ethylene glycol with COS.

Spectral properties of ethylene glycol + COS. From above spectral results, the binding and interaction of ethylene glycol with COS play important role in the solubility of dilute COS, so the UV, IR, fluorescence, $^1$H NMR spectra were determined to investigate the binding and interaction.

In Fig. 4, the characteristic band of ethylene glycol was identified in UV spectra, but no information on a complexing reaction could be obtained. From the figure, with increasing COS concentration, the special absorption band shifts from 200-207 nm and the absorption intensity increases. The special band was mainly related to n → σ* electron transition of oxygen atom in ethylene glycol. The shift can be due to the addition of COS, which destroys the original hydrogen bonding and interaction among ethylene glycol molecules and forms the new hydrogen bonding and interaction of hydroxyl hydrogen atoms in ethylene glycol with oxygen atoms in COS. In this situation, the n → σ* electron transition of oxygen atom in ethylene glycol become more easily with decreasing effect from hydroxyl hydrogen in ethylene glycol, so red shift phenomena occurred.

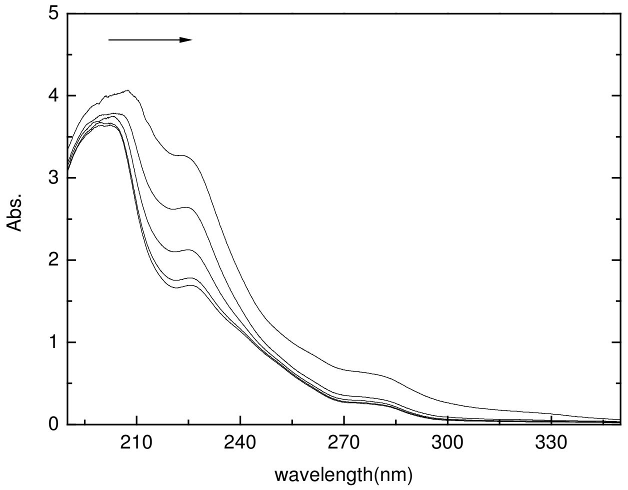
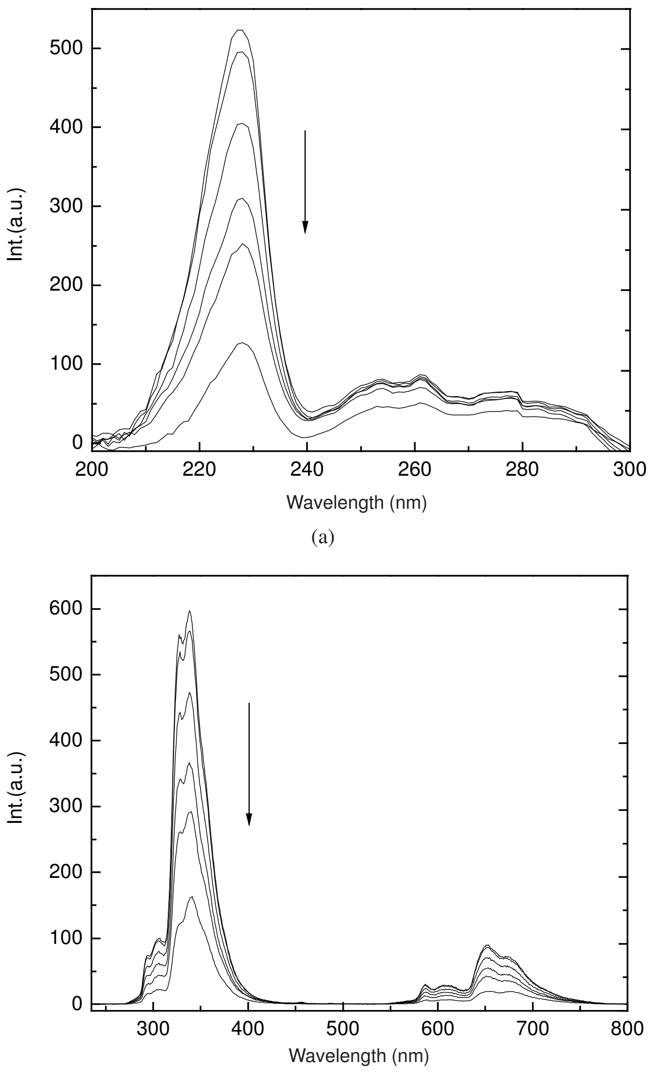


Fig. 4. Absorption spectral changes of ethylene glycol with increasing COS concentration

The binding of ethylene glycol with COS can be distinguished by examining of the absorption spectrum of the fluorophore. The collisional quenching only affects the excited states of the fluorophores, thus no changes in the absorption spectrum are predicted. In contrast, the ground state binding formation will frequently result in perturbation of the absorption spectrum of fluorophore. The fluorescence excitation and emission spectra of fluorophore can be treated as the same as the absorption spectra of the fluorophore. The quenching of the intrinsic fluorescence excitation and emission spectra of ethylene glycol caused by gradually increasing the concentration of COS were shown in Fig. 5. The special band was mainly related to σ*→n electron transition of hydroxyl oxygen atom in ethylene glycol. The quenching phenonenon can be due to the addition of COS, which affects the original hydrogen bonding and interaction among ethylene glycol molecules and forms the new hydrogen bonding and interaction between ethylene glycol and COS.



(a)

(b)

Fig. 5. Fluorescence excitation (a) and emission (b) spectral changes of ethylene glycol with increasing COS concentration

The recorded IR spectra of ethylene glycol + COS are shown in Fig. 6. From the spectra one stretching band is observed at 1704 cm$^{-1}$. The absorption peak at 1704 cm$^{-1}$ can be attribute to the stretching band of ν(C=O) (Vs) in COS because the IR spectra indicate that the fundamental frequencies of the stretching band of C=O often are found in the range of (1710-1820) cm$^{-1}$ [39]. However, the observed IR value is lower than the reference values. The phenomenon could be due to that the addition of COS forms the new hydrogen bonding and interaction of hydroxyl hydrogen atoms in ethylene glycol with oxygen atoms in COS. In this situation, the $\nu_s$ of C=O in COS become more easily with increasing effects from hydroxyl hydrogen in ethylene glycol, so $\nu_s$ shifts toward lower frequence. COS molecule is known to be similar with carbon dioxide and carbon disulfide; thus, COS behaves as an electron donor by the oxygen atom and its interaction with ethylene glycol should occur with the hydrogen of the hydroxyl group. Such an interaction should decrease the double bond character of COS and so induce a lower absorption frequency, as is observed. From the FTIR spectral results, one can suppose that the ethylene glycol-COS complex is the less stable, as suggested by its lower downshifted frequency. Such a shift observed in FTIR spectra was attributed to the hydrogen bonding and interactions of ethylene glycol with COS.

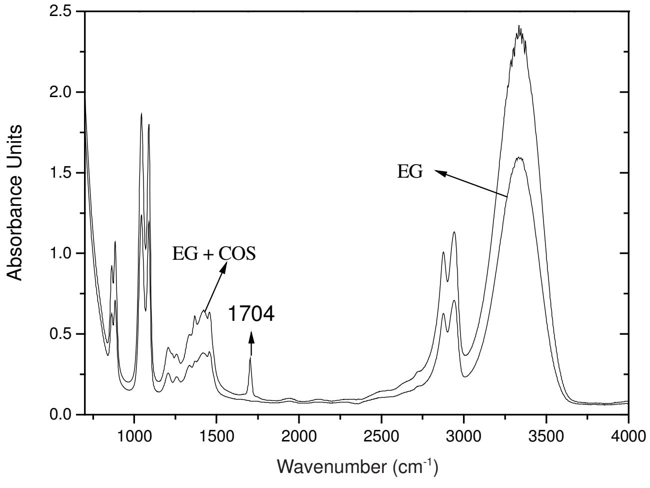
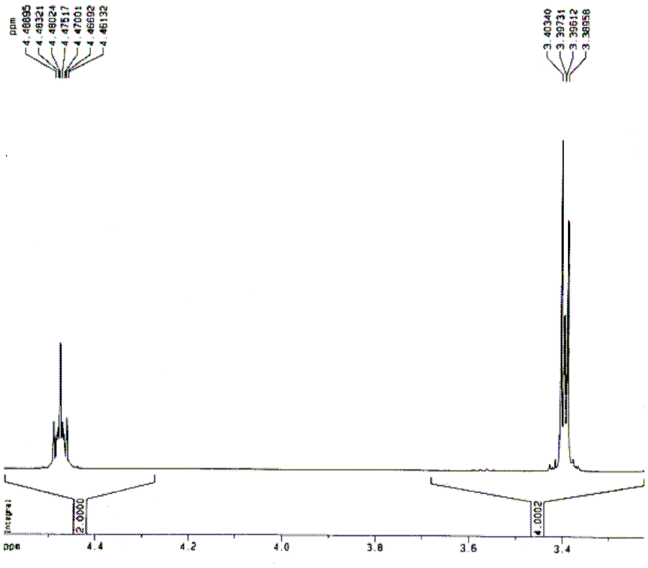
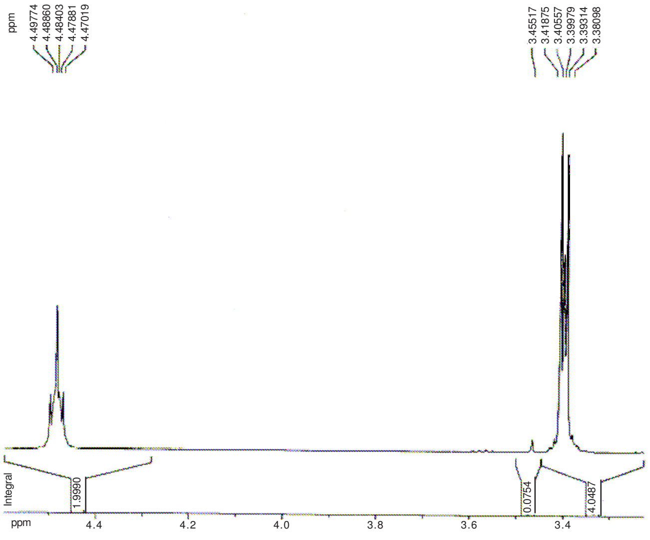


Fig. 6. FTIR spectra of ethylene glycol and ethylene glycol + COS in the range of (4000-400) cm$^{-1}$

The $^1$H NMR spectral results of ethylene glycol in the presence and absence of COS are showed in Fig. 7(a-b). Fig. 7(a) shows that the chemical shifts of hydrogen in -CH$_2$- appear at δ = 3.390, 3.396, 3.397 and 3.403 ppm (4H) and the chemical shift of hydrogen in -OH appears at δ = 4.461, 4.467, 4.470, 4.475, 4.480, 4.483 and 4.489 ppm (2H) in the $^1$H NMR spectrum of pure ethylene glycol. In the presence of COS, however, there appears at δ = (3.381-3.419) ppm (4H) and (4.470-4.498) ppm (2H) in Fig. 7(b). Meanwhile, the chemical shift of hydrogen atoms in -OH group shifts from δ = (4.461-4.489) ppm to (4.470-4.498) ppm in DMSO, which was due that the bond length of O-H in ethylene glycol molecules become longer and the electron cloud of hydrogen atoms in ethylene glycol become thinner. The results show that there is hydrogen bonding and interaction between ethylene glycol and COS so that shielding effect from oxygen atoms of COS makes the signal move to lower magnetic field.



(a)



(b)

Fig. 7. $^1$H NMR spectra of ethylene glycol in the presence and absence of COS

The above results suggest the hydrogen bonding and interaction between hydrogen atoms in ethylene glycol molecules and oxygen atoms in COS molecules occurred. When such hydrogen bonds are formed, hydroxyl hydrogen atoms in the ethylene glycol molecules are attracted by the oxygen atoms in COS and the bond length between hydrogen atom and oxygen atom in ethylene glycol molecules are elongated. In this situation, the $n \rightarrow \sigma^*$ electron transition of oxygen atom in ethylene glycol become more easily, so red shift phenomena occurred.

According to overall results and analyses, it is expected that hydrogen bonding and interaction among molecules is formed as …HO-$CH_2CH_2$O-H…OCS, which makes the desorption of COS from aqueous solution of ethylene glycol occur easier. The hydrogen bonding and interaction of oxygen atom in COS with hydroxyl hydrogen atom in ethylene glycol is very useful to desorb COS from aqueous solutions of ethylene glycol by pressure reduction, by temperature rise and by use of a carrier gas.

## Conclusion

This paper presents the results of fundamental investigations on the solubility data of dilute COS in various ethylene glycol aqueous solutions, which were determined as a function of composition in the range of (298.15-318.15) K. The data were obtained with relative uncertainties within ± 3.5 % for COS concentration in the gas phase, which showed that 50 % volume fraction of ethylene glycol in aqueous solution of ethylene glycol is a more reasonable composition used in absorption processes of dilute COS. Meanwhile, the various spectral results suggest that COS can interact with ethylene glycol by hydrogen bonds as …HO-$CH_2CH_2$O-H…OCS.

## ACKNOWLEDGEMENTS

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