

Spectral Investigation of Intermolecular Hydrogen Bonding and S...–O Interaction in Diethylene Glycol + H₂O + SO₂ Systems

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Diethylene glycol (DEG) + H₂O solutions (DEGWs) were used as the promising medium for the absorption processes of SO₂. In this work, when UV, FTIR, ¹H NMR, and fluorescence spectroscopic techniques were used for inspection of spectral changes of DEGWs with increasing SO₂ concentration, the results suggest that H₂O can interact with DEG by hydrogen bonds as the formation of –CH₂CH₂O(H)...–HOH...– and –CH₂–CH₂–O(CH₂–CH₂–)...–HOH...– in DEGWs. Furthermore, SO₂ can interact with DEG in various DEGWs by hydrogen bonds as the formation of –CH₂CH₂OH...–OSO...– and the intermolecular S...–O interaction in the absorption processes of SO₂, which was not discussed in the previous similar systems. The results of this work can be used to provide an important absorption mechanism for the design and operation of the absorption and desorption process in flue gas desulfurization with potential industrial application of DEG aqueous solutions.

1. Introduction

This paper is a continuation of the systematic program on the absorption processes of sulfur dioxide (SO₂) in the binary mixtures of diethylene glycol (DEG) + water (DEGW).¹ DEG is an important industrial solvent with potential gas scrubbing because of its favorable properties, such as low vapor pressure, low toxicity, high chemical stability, and low melting temperature. DEG and its similar solvents, including ethylene glycol (EG) and poly(ethylene glycol) (PEG), present native hydrogen-bonding sites so that their potential desorption characters are presented in the regenerative processes of solutions dissolving SO₂; therefore, in this work and in a number of previous studies, DEG and its similar solvents have been paid great attention to alcohol + water systems for SO₂ removal.^{2–6}

For the research of the absorption mechanism of SO₂ in DEG, FTIR, UV, and fluorescence spectroscopic techniques are used to probe the intermolecular hydrogen bonding^{7,8} and interaction among molecules, since the FTIR spectroscopy presents precise information about sensitive bonds of water^{9,10} and characteristic vibrational properties of DEG; furthermore, FTIR is also advantageous to evaluate vibrational properties of bonds through very thin solution films, which are usually difficult to handle for the floating properties of solution. UV and fluorescence spectroscopic techniques give important information about various electronic transitions. Generally, FTIR,^{11,12} UV, and fluorescence spectroscopic techniques offer the advantages to measure the association properties and hydrogen-bonding capability and to assess interaction among DEG, SO₂, and H₂O by analyzing band shifts and changes.

Previous work¹ shows that $w_1 = 0.60$ DEG (mass fraction of DEG in DEG) presents the extreme minimum solubility of 110 mg·L^{–1} to SO₂ when the SO₂ concentration in the gas phase was set at $\Phi_{\text{SO}_2} = 5 \times 10^{-4}$ (volume fraction of SO₂ in

the gas phase), and the solubility of SO₂ in pure DEG is 259 mg/L at the same composition. The study of absorption processes of SO₂ in DEG includes the following three steps: (1) gas–liquid equilibrium data for a gaseous mixture of SO₂ + N₂ with DEG aqueous solutions; (2) density, viscosity, and excess properties for DEG aqueous solutions; and (3) spectral properties of DEG + SO₂ + H₂O interactions. Previous articles cover the first two steps of this study. The present work is mainly focused on investigating intermolecular hydrogen bonding and S...–O interaction of DEG with SO₂ by UV, FTIR, ¹H NMR, and fluorescence spectroscopic techniques.

2. Experimental Section

The SO₂ gas (99.9%) and the N₂ gas (>99.9%) were purchased from the Beijing Gas Company, China. Analytical grade DEG was purchased from the Beijing Reagent Company (China), which was used after drying over molecular sieves (type 4A) and decompression filtration before measurements. The purity of the sample was checked by density determination at 298.15 K. The density of DEG at 298.15 K was found to be 1.1132 g·cm^{–3}, in good agreement with literature values of 1.113 03¹³ and 1.112 97.¹⁴ Double-distilled water was used.

UV–vis spectra were recorded on a Varian CARY 1E UV–vis spectrometer. 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 to 400 cm^{–1}. The spectrometer possesses autoalign energy optimization and a dynamically aligned interferometer, fitted with a constraining BaSO₄ pellet for the measurement of aqueous solution, an OPUS/IR operator, and IR source. A baseline correction was made for the spectra that were recorded in air, and then a 15 μ L solution was used on the FTIR spectrometer in every one of measurements; the thin layers of samples are less than typically 2 μ m thickness. ¹H NMR spectra were acquired using a Bruker ARX-400 nuclear magnetic resonance spectrometer, and (CH₃)₂SO (dimethyl sulfoxide, DMSO) was used as the NMR solvent. Fluorescence spectra were acquired using an F-4500 fluorescence spectrophotometer employing a 500 W Hg–Xe high pressure lamp.

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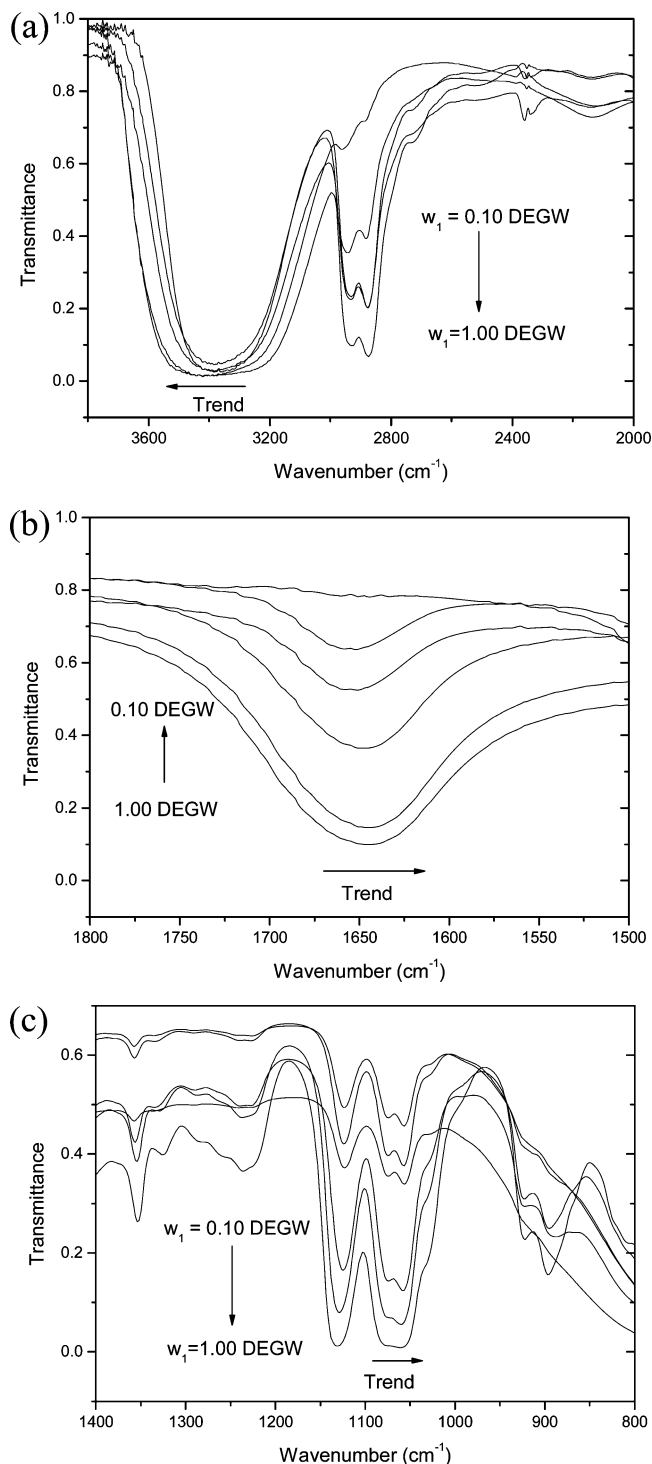


Figure 1. FTIR spectra of DEG at various mass fractions: (a) 3800–2000 cm^{-1} , (b) 1800–1500 cm^{-1} , and (c) 1400–800 cm^{-1} . The changing trend was found with increasing H_2O concentration.

All spectral experiments of DEG + SO_2 + H_2O were performed at room temperature and atmospheric pressure.

3. Results and Discussion

3.1. Spectral Properties of DEG + H_2O . The recorded FTIR spectra of DEG + H_2O are shown in Figure 1. In Figure 1a the stretching vibrational band of the hydroxyl group in DEG is found to shift toward higher frequency, from 3356 to 3422 cm^{-1} , with increasing H_2O concentration. The fact that the stretching vibrational band of hydroxyl in PEGW shifts

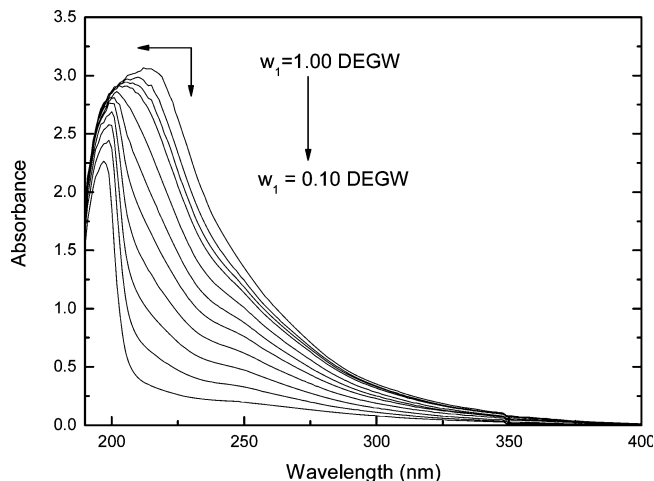


Figure 2. Absorption spectral changes with increasing H_2O concentration at various concentrations of DEG.

toward higher frequency indicates that the interactions of DEG with H_2O are due to the variational property of the hydroxyl group in DEG. In Figure 1b the bending vibrational frequency of H_2O changes from 1652 to 1645 cm^{-1} , which has been reported to appear at 1645 cm^{-1} in H_2O saturated low density polyethylene.⁹ The shift of the H–O–H bending vibrational band indicates that the interactions of DEG with H_2O result from the property of the hydrogen atom in H_2O . Meanwhile, from Figure 1c the stretching vibrational band of C–O–C in DEG is found to shift toward lower frequency from 1061 to 1057 cm^{-1} , which indicates that interactions can be related to the property of the ether oxygen atom of DEG. Based on the above results, the possible interactions of DEG with H_2O were due to the following two ways: (1) intermolecular hydrogen bonding and interaction of the hydrogen atom in H_2O with the hydroxyl oxygen atom in DEG by cross-linking, forming $-\text{CH}_2\text{CH}_2\text{O}(\text{H})\dots\text{HOH}\dots-$, and (2) weak intermolecular hydrogen-bonding interaction of hydrogen atom in H_2O with ether oxygen atom in DEG, forming $-\text{CH}_2-\text{CH}_2-\text{O}(\text{CH}_2-\text{CH}_2)\dots\text{HOH}\dots-$.

Figure 2 shows that the electronic transitions blue shift from 208 to 195 nm with increasing H_2O concentration in DEG. The absorption band is assigned to the $n\rightarrow\sigma^*$ electronic transition of the unshared electronic pair of the hydroxyl oxygen atom in DEG and the $n\rightarrow\sigma^*$ electronic transition of the ether oxygen atom in DEG because the $n\rightarrow\sigma^*$ electronic transition of H_2O is often found at the vacuum ultraviolet region. With increasing H_2O concentration, the intermolecular hydrogen-bonding interaction of hydroxyl oxygen atom in DEG with hydrogen atom of H_2O happened easily; however, the hydrogen bonding and interaction make the $n\rightarrow\sigma^*$ electronic transition of hydroxyl oxygen in DEG become more difficult. The present results show that the intermolecular hydrogen-bonding interaction in DEG formed as the formation of $-\text{CH}_2\text{CH}_2-\text{O}(\text{H})\dots\text{HOH}\dots-$.

3.2. Spectral Properties of DEG + SO_2 . The recorded FTIR spectra of DEG and DEG + SO_2 are shown in Figure 3. In Figure 3a, an asymmetry stretching band (V_{as}) of SO_2 was observed at 1327 cm^{-1} , and the C–O–C asymmetry stretching band at 1061 cm^{-1} does not change, which shows that the C–O–C asymmetry stretching band of DEG was not affected by SO_2 . The absorption peak at 1327 cm^{-1} , which is reported at 1344 cm^{-1} for SO_2 in noncomplexing CCl_4 ,¹³ can be attributed to the V_{as} of SO_2 because the IR and Raman spectra indicate the following values for the fundamental frequencies: symmetry

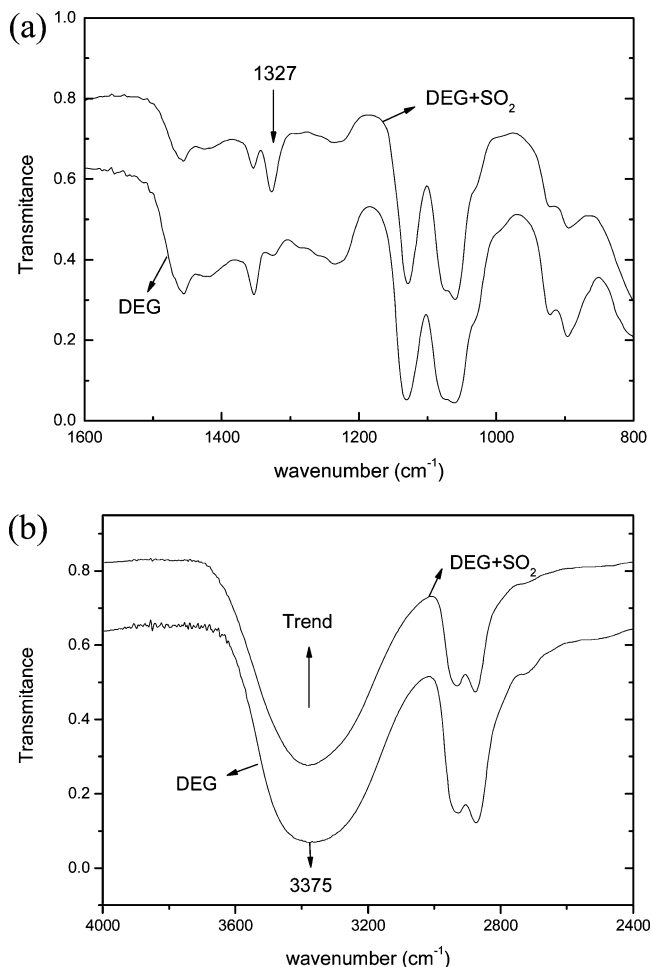


Figure 3. FTIR spectral changes of DEG with increasing SO_2 concentration in the range of $1600\text{--}800\text{ cm}^{-1}$ (a) and the range of $4000\text{--}2400\text{ cm}^{-1}$ (b).

stretching band (V_s) = 1151.38 cm^{-1} , bending vibrational band (δ) = 517.69 cm^{-1} , and V_{as} = 1361.76 cm^{-1} . Meanwhile, the phenomenon that the V_{as} of SO_2 shifts toward lower wavenumber than 1361.76 cm^{-1} can be due to the interaction of the sulfur atom in SO_2 with other atoms. The SO_2 molecule is known to be polar and the sulfur atom to be electropositive; therefore, SO_2 behaves as an electron acceptor by the sulfur atom, and its interaction with the hydroxyl oxygen atom (electronegative) in DEG should occur by the way of intermolecular $\text{S}\cdots\text{O}$ interaction, which was not discussed in the previous $\text{EGW} + \text{SO}_2$ system¹⁶ and $\text{PEGW} + \text{SO}_2$ system.¹⁷ Unfortunately, the V_s of SO_2 at 1151 cm^{-1} is strongly masked by the vibrations of DEG. In Figure 3b a phenomenon was displayed. In the absence of SO_2 , the stretching vibrational band of hydroxyl in DEG was observed at 3375 cm^{-1} . In the presence of SO_2 , the band was changed into a more peaked band. The phenomenon could be due to the fact that the addition of SO_2 affects the original hydrogen-bonding interaction among DEG molecules and forms the new intermolecular hydrogen bonding of the hydroxyl hydrogen atom in DEG with the oxygen atom in SO_2 and intermolecular $\text{S}\cdots\text{O}$ interaction.

In Figure 4, the characteristic bands of DEG and SO_2 were respectively identified, but no information on a complexing reaction could be obtained. From the figure the absorption band of the $n\rightarrow\pi^*$ electron transition of the oxygen atom in SO_2 (Π_3^4) is observed at 276 nm , and the absorption intensity of the band increases with increasing SO_2 concentration. Another absorption band, which was mainly due to $\pi\rightarrow\pi^*$ electron transition of the sulfur atom in SO_2 (Π_3^3) and the $n\rightarrow\sigma^*$ electron transition

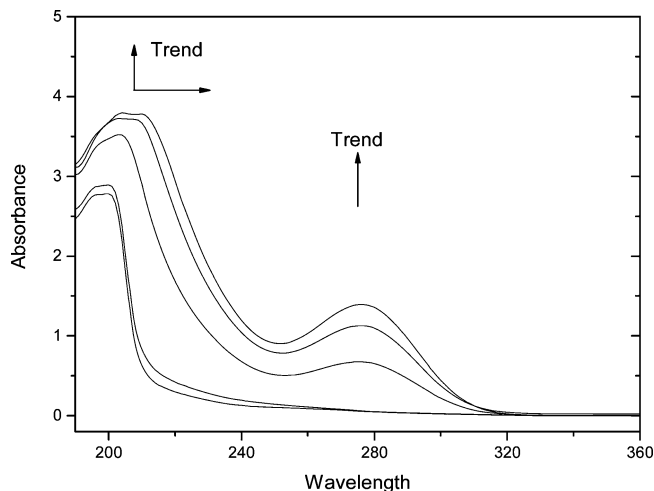


Figure 4. Absorption spectral changes of DEG with increasing SO_2 concentration. The changing trend was found with increasing SO_2 concentration.

of the hydroxyl oxygen atom in DEG, shifts from 208 to 219 nm , and the absorption intensity of the band increases. The shift results from the intermolecular hydrogen bond of oxygen atoms in SO_2 with hydroxyl hydrogen atoms in DEG and intermolecular $\text{S}\cdots\text{O}$ interaction. The bonding of oxygen atoms of SO_2 with hydroxyl hydrogen atoms of DEG results in the following two results: (1) the decreasing effects of oxygen atoms on the sulfur atom of SO_2 make the $\pi\rightarrow\pi^*$ electron transition of the sulfur atom in SO_2 change easier; (2) the decreasing effects of hydroxyl hydrogen atoms on hydroxyl oxygen atoms in DEG make the $n\rightarrow\sigma^*$ electron transition of the hydroxyl oxygen atom in DEG change easier also.

^1H NMR spectra of DEG in the presence and absence of SO_2 are shown in Figure 5. Figure 5a shows that the chemical shifts of hydrogen in $-\text{CH}_2-$ appear at $\delta = 3.420\text{--}3.604\text{ ppm}$ (8 H), and the chemical shift of hydroxyl hydrogen appears at $\delta = 4.640, 4.626,$ and 4.612 ppm (2H) in the ^1H NMR spectrum of pure DEG. However, with increasing SO_2 concentration in DEG, because the bond length of $\text{O}\text{--}\text{H}$ in EG molecules become longer and the electron cloud of hydroxyl hydrogen atoms in DEG molecules becomes thinner, the signal changes into a single peak and the chemical shift of hydrogen atoms in $-\text{OH}$ groups shifts from $\delta = 4.640\text{--}4.612\text{ ppm}$ to 4.507 ppm in DMSO (Figure 5a–c). The phenomena are because the interaction of oxygen atoms in SO_2 with hydroxyl hydrogen atoms in DEG increases the shielding effect of hydroxyl hydrogen atoms in DEG, so that the signal changes into a single peak and the signal of the chemical shift of the hydroxyl hydrogen in DEG moves toward higher magnetic field.

Stable state fluorescence spectra with selective excitation of DEG with increasing SO_2 concentration are shown in Figure 6. Upon excitation at 205 nm , which is due to the $n\rightarrow\sigma^*$ electron transition of the oxygen atom of DEG, strong fluorescence with emission positions at $290\text{--}320$ and $580\text{--}620\text{ nm}$ was observed. The fluorescence intensity of the $\sigma^*\rightarrow n$ electron transition of the oxygen atom of DEG decreases with increasing SO_2 concentration. The phenomena can be due to the intermolecular interaction of the oxygen atom in DEG with the sulfur atom in SO_2 with the formation of $\text{S}\cdots\text{O}$ interaction.

According to the above IR, UV, ^1H NMR, and fluorescence spectral results, it is expected that DEG bonds with SO_2 by the intermolecular hydrogen bonds of $-\text{CH}_2\text{CH}_2\text{OH}\cdots\text{OSO}\cdots-$ and intermolecular $\text{S}\cdots\text{O}$ interaction.

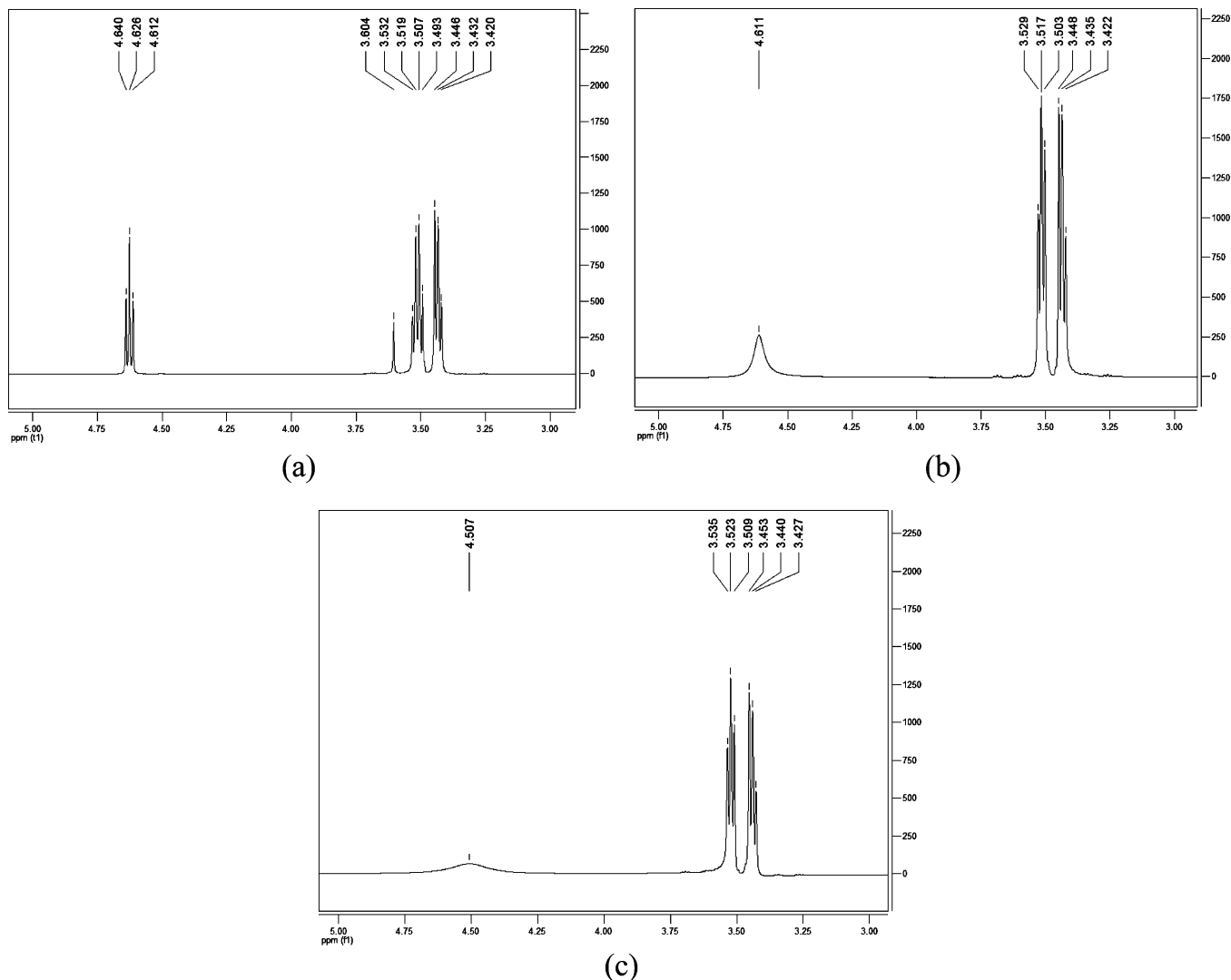


Figure 5. ^1H NMR spectra of DEG in the presence and absence of SO_2 : (a) ^1H NMR spectrum of DEG, (b) ^1H NMR spectrum of DEG after 5 min ventilation of SO_2 , and (c) ^1H NMR spectrum of DEG after 10 min ventilation of SO_2 .

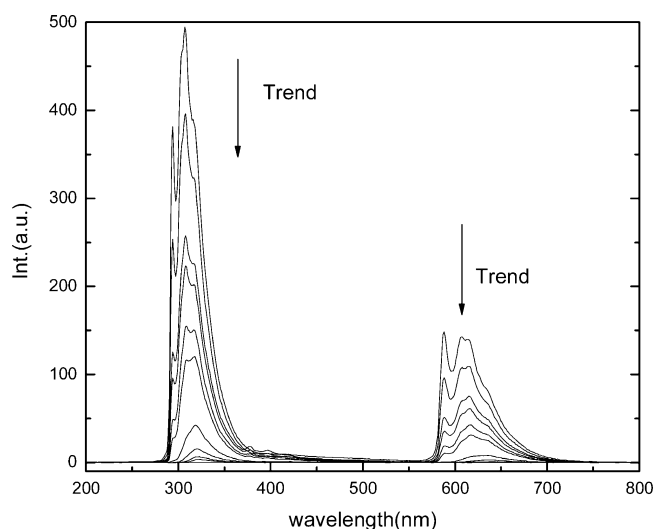


Figure 6. Fluorescence spectral changes of DEG with increasing SO_2 concentration. The changing trend was found with increasing SO_2 concentration.

3.3. Spectral Properties of $\text{DEGW} + \text{SO}_2$. The recorded FTIR spectra of H_2O and $\text{H}_2\text{O} + \text{SO}_2$ are shown in the previous work.¹⁶ From the spectra two special stretching bands are

observed at 1332 and 1151 cm^{-1} , which can be attributed to the V_{as} and V_{s} of SO_2 .¹⁵

The recorded IR spectra of $w_1 = 0.60$ DEGW and $w_1 = 0.60$ DEGW + SO_2 are shown in Figure 7. In Figure 7a the stretching band at 1329 cm^{-1} was observed. Meanwhile, the phenomenon that the V_{as} of SO_2 shifts to a lower wavenumber than $V_{\text{as}} = 1361.76$ cm^{-1} can be due to the interaction of the sulfur atom in SO_2 with the hydroxyl oxygen atom in DEG by the way of $\text{S}\cdots\text{O}$ interaction. In Figure 7b, in the absence of SO_2 , the stretching vibrational band of hydroxyl in DEG was observed at 3385 cm^{-1} and the band was broad; in the presence of SO_2 , the band was changed into a more peaked band. The phenomenon can be due to the new hydrogen bonding of the hydroxyl hydrogen in DEG with oxygen in SO_2 and intermolecular $\text{S}\cdots\text{O}$ interaction.

In Figure 8, the absorption band of the $n \rightarrow \pi^*$ electron transition of the oxygen atom in SO_2 is observed at 276 nm, and the absorption intensity of the band increases with increasing SO_2 concentration. Meanwhile, the special absorption band red shifts from 197 to 211 nm, and the absorption intensity of the band increases also. The results show the $\pi \rightarrow \pi^*$ electron transition of SO_2 and the $n \rightarrow \sigma^*$ electron transition of oxygen atom of DEG in $w_1 = 0.60$ DEGW with increasing SO_2 concentration.

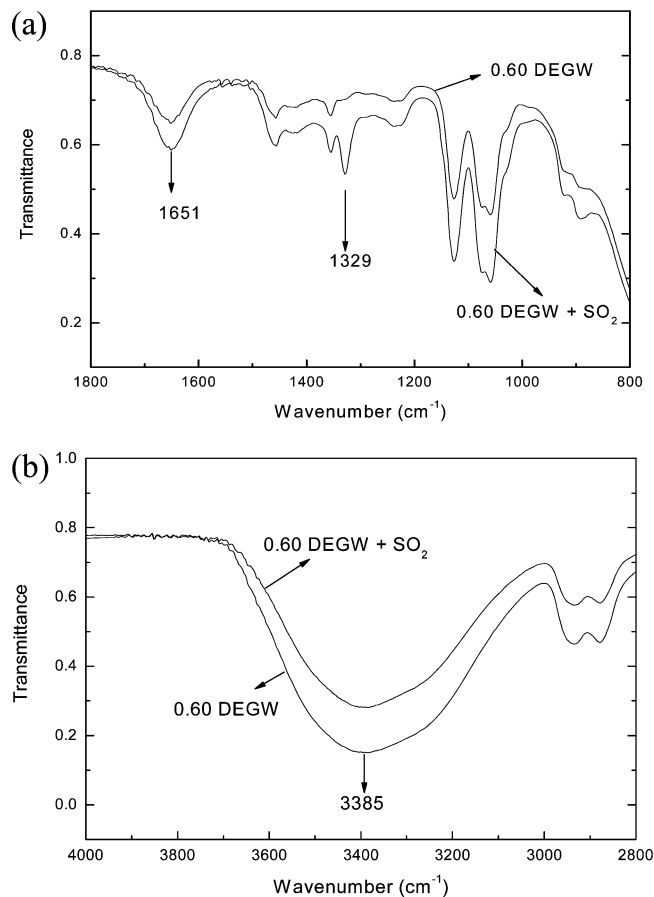


Figure 7. FTIR spectra of $w_1 = 0.60$ DEGW + SO_2 in the range of $1800\text{--}800\text{ cm}^{-1}$ (a) and $4000\text{--}2800\text{ cm}^{-1}$ (b).

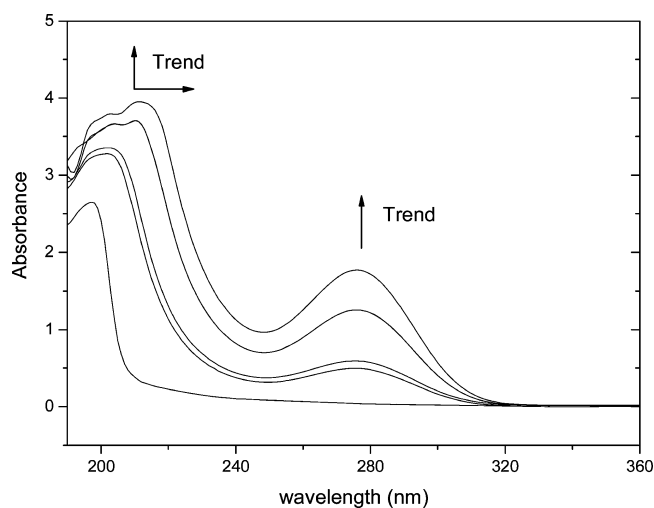


Figure 8. Absorption spectral changes of $w_1 = 0.60$ DEGW with increasing SO_2 concentration. The changing trend was found with increasing SO_2 concentration.

Stable state fluorescence spectra with selective excitation of DEGW with increasing SO_2 concentration are shown in Figure 9. Upon excitation at 205 nm strong fluorescence with emission positions at 290–320 and 580–620 nm were observed. The fluorescence intensity of the $\sigma^* \leftarrow n$ electron transition of the oxygen atom of DEG in DEGW decreases with increasing SO_2 concentration. The phenomena can be due to the intermolecular interaction of the oxygen atom in DEG with SO_2 as the formation of $\text{S}\cdots\text{O}$ interaction.

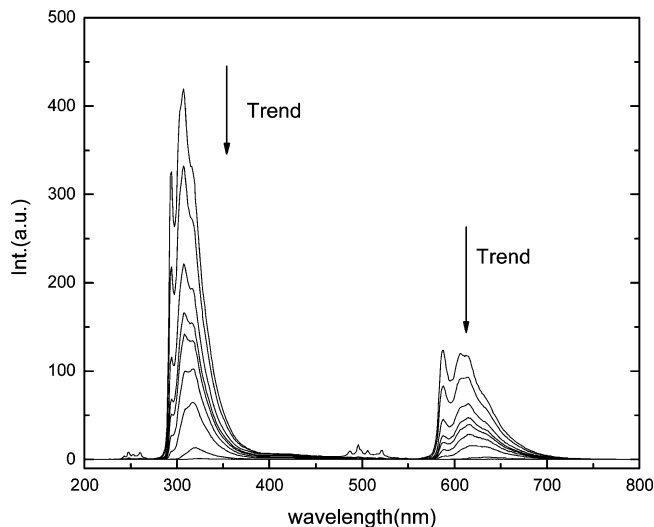


Figure 9. Fluorescence spectral changes of DEG in DEGW with increasing SO_2 concentration. The changing trend was found with increasing SO_2 concentration.

The above results suggest that the hydrogen bonding and interaction between hydrogen atoms in DEG and oxygen atoms in SO_2 occurred. When such hydrogen bonds are formed, hydroxyl hydrogen atoms in DEG are attracted by the oxygen atoms in SO_2 , and the bond length between hydrogen atom and oxygen atom in DEG is elongated. Such an interaction decreases the double bond character of SO_2 and so induces a lower absorption frequency, as is observed. From the FTIR spectral results recorded on the aqueous solution, one can suppose that the DEG– SO_2 complex is the less stable, as suggested by its lower downshifted frequency and its stronger desorption capacity. Such a shift observed in FTIR spectra was attributed to the interactions of DEG with SO_2 . Comparing the spectra of $w_1 = 0.60$ DEGW + SO_2 and $w_1 = 0.60$ DEGW, it is observed that the H–O–H bending band and the characteristic bands of DEG are not obviously shifted in the mixture under the influence of SO_2 (Figure 7). The constant H–O–H bending band in the absorption processes of SO_2 is mainly due to the hydrogen interaction of DEG and SO_2 rather than the reaction of water and SO_2 . Meanwhile, FTIR results support that there are $\text{S}\cdots\text{O}$ interactions of the sulfur atom in SO_2 with the hydroxyl oxygen atom in DEG. The hydrogen bonding and interaction of SO_2 with DEG is very useful to desorb SO_2 from DEGWs by pressure reduction, by temperature rise, and by use of a carrier gas in the following work. But the above results only give us the present information; the exact molecular mechanism of interactions requires further investigation. But in $w_1 = 0.06$ DEGW, which shows lower solubility to SO_2 ,¹ the hydrogen bonding and $\text{S}\cdots\text{O}$ interaction among molecules present passive effects on the absorption of SO_2 in DEGW.

4. Conclusion

DEGW presents native hydrogen-bonding sites for the absorption of SO_2 so that the absorption and desorption properties of SO_2 can be related to hydrogen bonding and intermolecular $\text{S}\cdots\text{O}$ interaction among molecules. Present results show that the possible interactions in DEGW result from the following two ways: (1) hydrogen bonding and interaction of the hydrogen atom in H_2O with the hydroxyl oxygen atom in DEG by cross-linking as the formation of $-\text{CH}_2\text{CH}_2\text{O}(\text{H})\cdots\text{HOH}\cdots$; (2) hydrogen bonding and interaction of the hydrogen atom in H_2O with the ether

oxygen atom in DEG as the formation of $-\text{CH}_2-\text{CH}_2-\text{O}(\text{CH}_2-\text{CH}_2-)\dots-\text{HOH}\dots-$. In addition, in the absorption processes of SO_2 in pure DEG or $w_1 = 0.60$ DEGW, the spectral analyses suggest that SO_2 can interact with DEG by hydrogen bonds as $\dots-\text{OSO}\dots-\text{H}-\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{O}-\text{H}\dots-\text{OSO}\dots-$, and intermolecular $\text{S}\dots-\text{O}$ interaction of the hydroxyl oxygen atom of DEG with the sulfur atom of SO_2 .

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