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Sulfur Dioxide Solubility in Heat Transfer Oil in the Temperature Range of (293.15 to 313.15) K: Experimental Results and Related Correlations

Ahmet Alper Aydın, Ahmet Tuncer Erciyes, and Hasancan Okutan*

Department of Chemical Engineering, Faculty of Chemical and Metallurgical Engineering, Istanbul Technical University, Istanbul, Turkey

This work evaluates the solubility and average enthalpy of dissolution of sulfur dioxide in heat transfer oil in the temperature range of (293.15 to 313.15) K at 101.3 kPa. The heat transfer oil is a commercial product, consisting primarily of noncyclic and cyclic alkanes. In this study, solubility is expressed in four different units; Henry's constant, Ostwald's coefficient, sulfur dioxide mole fraction, and grams of solute in unit volume of solvent at 101.3 kPa pressure with solubility−temperature relations in the working temperature range.

Introduction

Sulfur dioxide is one of the major pollutants arising from the combustion processes, and today it is probably the most widespread air pollutant. The major source of sulfur dioxide is derived from the combustion of high sulfur fuels. Although many processes have been proposed for the removal of sulfur dioxide from stack gases, none has yet been proven to be superior to all others, and none has been proven to be totally effective in removing sulfur dioxide under all circumstances.

In some of the sulfur dioxide removal processes, sulfur dioxide streams are scrubbed with an alkali solution, performing a chemical reaction with an alkaline substance such as alkali metal hydroxides, amines, and solid limestone or dolomite, by which other pollution problems may arise. Although the conversion of sulfur dioxide to elemental sulfur has environmental advantages, no processes exist that are environmentally acceptable and economically viable. Alternative removal processes for sulfur dioxide are based on the physical absorptions with organic solvents such as dimethylaniline,[1] tetraethylene glycol dimethyl ether,[2] *N*-methylpyrrolidone,[3] *N*,*N*-(dimethylpropylene) urea,[3] and methyl diethanolamine.[3]

The studies on sulfur dioxide removal from flue gases and its solubility mostly focus on polar hydrophilic chemicals having strong chemical interactions with sulfur or oxygen atoms of sulfur dioxide. In hydrophilic solvents, such as pure water[4] or aqueous electrolyte solutions of sodium chloride,[5] ammonium chloride,[5] sulfuric acid,[6] hydrochloric acid,[6] seawater,[7] ammonia,[8] and ethylenediamine−phosphoric acid,[9] sulfur dioxide gives ionic species, in which the dissolution mechanisms differ. Although the sulfur dioxide solubility increases in the presence of polar groups, the regeneration process of the solvent becomes more difficult and expensive. However, limited data are reported for hydrophobic absorbents with different polarity, such as 1,2-dichloroethane,[10] nitrobenzene,[10] and diethyl ether.[10]

In the present work, a nonpolar hydrophobic absorbing solvent has been selected for sulfur dioxide removal that can be used at high flue gas temperatures because of its high flash, fire, and boiling points in practice. The aim of this research is to determine the sulfur dioxide absorption capacity of heat transfer oil in the temperature range of (293.15 to 313.15) K with 5 K intervals. Empirical relations have been established, and the average enthalpy of sulfur dioxide dissolution in the temperature range has been given.

Table 1. Typical Characteristics of the Heat Transfer Oil[11]

relative density at 288.5 K	875 kg·m^{-3}
flash point, open cup	494 K
fire point	516 K
auto-ignition point	623 K
neutralization value	< 0.05 %, mass fraction
normal operating range of bulk temperature	−263 K to 593 K

Experimental Section

Materials. The heat transfer oil, Transcal N, is a commercial product of BP Company. Transcal N is a mixture of saturated noncyclic and cyclic hydrocarbons without any additives. Several analyses have been performed to determine the chemical structure, average empirical formula, and average molecular weight of the oil to be used in related calculations. Physical properties of the heat transfer oil are given in Table 1. Sulfur dioxide was provided with a sulfur dioxide gas cylinder at $w = 0.99$ purity.

Instrumentation in the Determination of the Heat Transfer Oil Chemical Composition. To determine the chemical composition of the oil, several analyses have been performed, such as elemental analysis, IR, GC−MS, and ^{1}H NMR instrumental analyses. IR spectra were recorded on Shimadzu FT-IR spectrophotometer model 8300, and GC−MS data were recorded on Hewlett-Packard model 5890 series GC connected with a 5972 mass selective detector. HP 5 MS column, 30 m × 0.32 mm i.d. 0.25 μm was used. ^{1}H NMR data were recorded on Varian Mercury−V$_x$−400 MHz. Average molecular weight of the heat transfer oil is evaluated with the help of GC−MS spectral and elemental analyses data.

Chemical Composition of the Heat Transfer Oil. The heat transfer oil, Transcal N, is a commercial product having saturated noncyclic and cyclic hydrocarbon composition without any additives. It is very stable at high temperatures due to its usage area, such as at (383 to 473) K, which are common flue gas temperatures. Additional instrumental analysis and boiling point data are given as follows: Boiling point: (628 to 643) K. Elemental analysis, mass fraction: C, 0.8389; H, 0.1804; N, 0.0000; S, 0.0000. Empirical formula: $(CH_{2.562})_n$. Infrared

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* Corresponding author. E-mail: okutan@itu.edu.tr.

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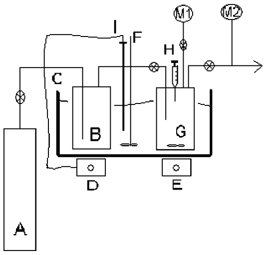

Figure 1. Experimental setup: A, SO_2 gas cylinder; B, preheating flask; C, water bath; D, electrical heating device; E, magnetic stirrer; F, mechanical stirrer; G, absorption flask; H, injector; I, contact thermometer; M1, mercury manometer; M2, digital manometer.

spectral data (NaCl, cm^{-1}): 2921 (strong, C−H stretching); 2853 (s, C−H str, cycloalkane); 1460, 1377 (m, C−H deformation); 722 (w, C−H deformation). ^1H NMR: Multiplets at (0.5 to1.0) ppm and (1.20 to 1.44) ppm (CH_3, CH_2, and CH protons); (6.8 to 7.2) ppm (aromatic protons, as noise).

According to the elemental analysis and GC−MS data, the heat transfer oil consists of saturated hydrocarbons and does not have any N or S containing moieties. Consequently, it has nonpolar and hydrophobic character.

Transcal N is a homogeneous mixture of hydrocarbons in the carbon atom range of 14 to 30. It is a liquid at room temperature, and its boiling point is around (628 to 643) K. Approximately 20 % of the oil consists of hydrocarbons with less than 21 carbon atoms, and 15 % of the total hydrocarbon content is cyclic alkanes.

In the IR data, the absence of olefinic (═C−H) bands near 3030 cm^{-1}, C═C vibrations around (1660 to 1590) cm^{-1}, and carbonyl vibrations show its saturated hydrocarbon composition along with the ^1H NMR spectrum obtained in chloroform solution, which has two multiplets between (0.5 to 1.0) ppm and (1.20 to 1.44) ppm assigned to CH_3, CH_2, and CH protons and aromatic protons, at noise level, observed at (6.8 to 7.2) ppm.

The average molecular weight and average empirical formula have been calculated from the GC−MS data and are given as follows: Average molecular weight: 367.31 g·mol^{-1}. Average empirical formula: $C_{25.17}H_{64.48}$.

Solubility Measurement. The solubility of sulfur dioxide in heat transfer oil was measured using the experimental setup shown in Figure 1. The experimental setup consisted of preheating and absorption flasks with water pump on the gas exit line, digital and mercury manometers, an electrical heating device with contact thermometer, magnetic and mechanical stirrers, and a water bath. The absorption process was performed in the absorption flask of 311.50 cm^3. Sulfur dioxide was fed into the system from a sulfur dioxide gas cylinder. The heat transfer oil was injected through the septum at the top of the absorption flask.

The basic approach in these experiments was to charge a closed system with a known amount of gaseous solute, in terms of pressure, and solvent, in terms of mass of injection, and then to measure the system pressure change at a constant temperature.[12]

The procedure for making a solubility measurement was as follows: The water pump, at the end of the gas exit line, was used to suck the present air in the system. Although the system was kept under vacuum, it was also necessary to let sulfur dioxide flow during suction in order to sweep out all air in the absorption flask. During suction, the mercury manometer line was closed in order to prevent any counter flow of mercury into the absorption flask, being under vacuum, as a precaution. The digital manometer, TESTO 506 electronic pressure meter, measured the vacuum pressure and helped setting the system pressure to a starting value. The precise pressure inside the absorption flask was measured with the mercury manometer after the inlet and outlet taps were closed and the gate to the manometer was opened. The pressure changes during absorption process were recorded after a known amount of oil was injected into the system at a constant temperature. To get more precise results at the evaluation of records, three injections with different amounts of oil were done in each experiment, resulting in a decrease in the inside pressure below 101.3 kPa. The amount of oil injected into the absorption flask was around 1.4 g for the first injection, and decreasing amounts were introduced in further injections at each experiment.

The U-tube mercury manometer has a 0.5 mm graduated scale. The uncertainty of the differential pressure reading at the mercury manometer is ± 0.066 kPa, and the pressure changes are recorded at 3, 5, 10, 13, and 15 min after each injection. The thermometer has been calibrated against reagent grade diphenylamine as the reference substance. The uncertainties of mass of oil injected and the temperature of the water bath are ± 0.0001 g and ± 0.2 K, respectively.

Data Reduction. Since the heat transfer oil has high flash, fire, and boiling points, the vapor pressure of Transcal N is assumed to be negligible in the working temperature range. Pressure drop by sulfur dioxide absorption is assumed to be not significantly influenced by the expansion of the oil.

The amount of sulfur dioxide in gas and liquid phases at equilibrium is calculated with the help of ideal gas equation, concerning a decrease in gas volume after each injection. Therefore, a volumetric adjustment coefficient, z', is used in the calculations.

If the initial mole number of sulfur dioxide in gas volume of V_0 is n_{0g} and the unabsorbed sulfur dioxide moles in the gas phase is n_{1g} for the initial stage and first oil injection, respectively, then

$$\frac{P_0}{P_1}\frac{V_0}{V_1} = \frac{n_{0g}}{n_{1g}} = z \tag{1}$$

can be written. If, $V_0/V_1 = z'$, then the above equation can be written as

$$\frac{P_0}{P_1}z' = \frac{n_{0g}}{n_{1g}} = z \tag{2}$$

Thus, by use of the sulfur dioxide mole numbers in the gas phase before and after the injection, the number of moles of sulfur dioxide absorbed by oil, n_1(oil), can be calculated as follows:

$$n_{0g} - n_{1g} = n_1(\text{oil}) \tag{3}$$

$$n_{0g} - \frac{n_{0g}}{z} = n_1(\text{oil}) \tag{4}$$

$$n_{0g}\left(1 - \frac{1}{z}\right) = n_1(\text{oil}) \tag{5}$$

where

$$z = \frac{P_0}{P_1}z' \tag{6}$$

Sulfur dioxide mole fraction in oil is calculated by use of number

Table 2. Sulfur Dioxide Mole Fraction x at 101.3 kPa from T = (293.15 to 313.15) K

	x				
	T = 293.15 ± 0.2 K	T = 298.15 ± 0.2 K	T = 303.15 ± 0.2 K	T = 308.15 ± 0.2 K	T = 313.15 ± 0.2 K
mean value	0.140	0.124	0.113	0.103	0.093
standard deviation	0.006	0.002	0.003	0.002	0.001
95 % conf interval	± 0.015	± 0.005	± 0.007	± 0.005	± 0.002

Table 3. Sulfur Dioxide Mole Fraction x Data at Different Pressures

	T = 293.15 ± 0.2 K		T = 298.15 ± 0.2 K		T = 303.15 ± 0.2 K		T = 313.15 ± 0.2 K	
t/min	p/kPa	x	p/kPa	x	p/kPa	x	p/kPa	x
15	103.300	0.1469	106.632	0.1324	105.165	0.1181	105.365	0.0943
30	101.233	0.1461	102.966	0.1276	102.966	0.1175	102.566	0.0929
45	100.367	0.1456	100.234	0.1225	101.233	0.1170	100.967	0.0925

Table 4. Sulfur Dioxide Henry's Constants H at 101.3 kPa from T = (293.15 to 313.15) K

	H/(kPa)				
	T = 293.15 ± 0.2 K	T = 298.15 ± 0.2 K	T = 303.15 ± 0.2 K	T = 308.15 ± 0.2 K	T = 313.15 ± 0.2 K
mean value	726.19	814.83	894.30	987.03	1085.39
standard deviation	31.15	9.97	25.00	22.50	6.69
95 % conf interval	± 77.38	± 24.78	± 62.11	± 55.89	± 16.62
relative standard dev	4.28%	1.22%	2.79%	2.28%	0.61%

Table 5. Sulfur Dioxide Ostwald's Coefficients L_v^0 and Solubility Data s at 101.3 kPa from T = (293.15 to 313.15) K

	$L_v^0/\text{mL}_{SO_2}\cdot\text{mL}_{oil}^{-1}$				
	T = 293.15 ± 0.2 K	T = 298.15 ± 0.2 K	T = 303.15 ± 0.2 K	T = 308.15 ± 0.2 K	T = 313.15 ± 0.2 K
mean value	9.298	8.272	7.567	6.884	6.300
standard deviation	0.466	0.153	0.245	0.190	0.106
95 % conf interval	± 1.158	± 0.380	± 0.609	± 0.472	± 0.263

	$s/\text{g}_{SO_2}\cdot\text{L}_{oil}^{-1}$				
	T = 293.15 ± 0.2 K	T = 298.15 ± 0.2 K	T = 303.15 ± 0.2 K	T = 308.15 ± 0.2 K	T = 313.15 ± 0.2 K
mean value	24.760	21.658	19.486	17.240	15.704
standard deviation	1.242	0.401	0.630	0.418	0.264
95 % conf interval	± 3.086	± 0.996	± 1.565	± 1.038	± 0.656

of moles of solute, n_n(oil), and total number of moles of solvent and solute, $N_n(\text{oil}) + n_n(\text{oil})$, in the liquid phase:

$$X_{SO_2} = \frac{n_n(\text{oil})}{N_n(\text{oil}) + n_n(\text{oil})} \qquad (7)$$

Results

Sulfur Dioxide Mole Fraction in Transcal N at 101.3 kPa. The mole fraction expresses the sulfur dioxide solute proportion on total molar bases of solute and solvent. The sulfur dioxide mole fraction at 101.3 kPa pressure was calculated by interpolation with the help of graphs plotted between the pressure and mole fraction for each experiment. The sulfur dioxide mole fraction at 101.3 kPa and some of the actual experimental data at different pressures are given in Tables 2 and 3, respectively.

Henry's Constant. Henry's constant has been calculated from the equation:

$$H/\text{kPa} = (P/\text{kPa})/x \qquad (8)$$

at 101.3 kPa. The results are given in Table 4. The correlation between Henry's constant and temperature is observed to be linear, and the equation expressing the variation in Henry's constant H/kPa with temperature, T/K = (298.15 to 313.15) K is given in Figure 2.

Ostwald's Coefficient. Ostwald's coefficient can be defined as a volumetric ratio of sulfur dioxide absorption and it is calculated by assuming the acceptable ideal gas behavior in which solvent expansion upon dissolving gas is negligible. If V_{oil}^0 is the volume of pure oil at a specified temperature, total pressure is p, and V_{gas} is the absorbed gas volume, then Ostwald's coefficient (L_v^0) is calculated from the equation:

$$L_v^0/\text{mL}_{SO_2}\cdot\text{mL}_{oil}^{-1} = (V_{gas}/V_{oil}^0)_{equil.} \qquad (9)$$

The results for total 101.3 kPa pressure are given in Table 5. The correlation showing the decrease in Ostwald's coefficient by increasing temperature and the equation expressing the variation in Ostwald's coefficient, L_v^0, with temperature, T/K = (298.15 to 313.15) K are in Figure 3.

Sulfur Dioxide Solubility in Unit Volume of Solvent. Solubility values of sulfur dioxide in unit volume of oil were calculated from Ostwald's coefficient showing gram of sulfur dioxide absorbed in unit volume of heat transfer oil at 101.3 kPa pressure. The calculated values are given in Table 5.

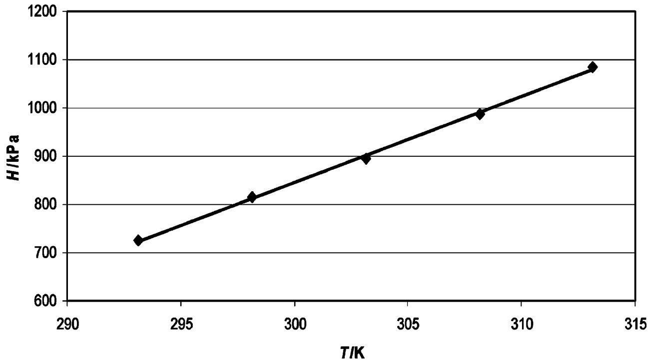

Figure 2. Temperature dependence of Henry's constant, $H/\text{kPa} = 17.812\,(T/\text{K}) - 4498$, se = 6.09 kPa.

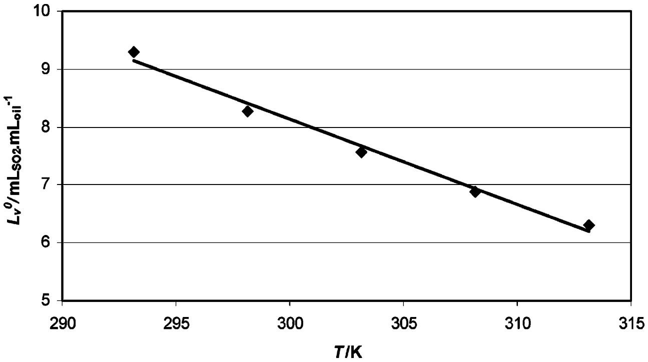

Figure 3. Temperature dependence of Ostwald's coefficient, $L_v^0/\mathrm{mL_{SO_2}{\cdot}mL_{oil}}^{-1} = -0.1477\ (T/\mathrm{K}) + 52.433$, $s_e = 0.15\ \mathrm{mL_{SO_2}{\cdot}mL_{oil}}^{-1}$.

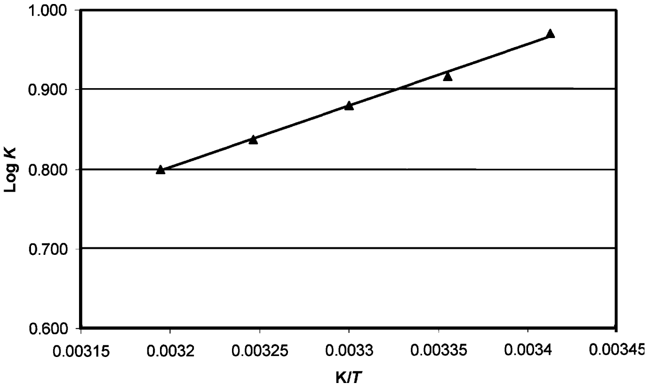

Figure 4. Plot of log K vs K/T.

The following equation expresses the variation in solubility $s/\mathrm{g_{SO_2}{\cdot}L_{oil}}^{-1}$ with temperature, $T/\mathrm{K} = (298.15\ \text{to}\ 313.15)$ K:

$$s/\mathrm{g_{SO_2}{\cdot}L_{oil}}^{-1} = -0.4506(T/\mathrm{K}) + 156.37 \qquad (10)$$

with $s_e = 0.49\ \mathrm{g_{SO_2}{\cdot}L_{oil}}^{-1}$.

Equilibrium between Gas and Liquid Phases and Enthalpy of Dissolution. At a specified temperature and pressure, sulfur dioxide dissolves in heat transfer oil until it reaches an equilibrium state. The mass transfer equilibrium can be written as:

$$SO_2(\mathrm{gas}) \leftrightarrow SO_2(\mathrm{oil})$$

At equilibrium, the initial mole number of sulfur dioxide n_0 is reduced by the absorption of n_{SO_2}(oil) moles of sulfur dioxide. Thus, the equilibrium constant K of heat transfer oil with nonvolatile character at working temperatures can be calculated as follows:

$$K = \frac{[SO_2]_{\mathrm{oil}}}{[SO_2]_{\mathrm{gas}}} = \frac{n_{SO_2}(\mathrm{oil})/V_{\mathrm{oil}}}{(n_0 - n_{SO_2}(\mathrm{oil}))/(V - V_{\mathrm{oil}})} \qquad (11)$$

The equilibrium constants $K_{1,2,..,5}$ are dependent on temperature and the slope of log K versus K/T plot has been evaluated in Figure 4 to calculate the average enthalpy of dissolution.

The following equation expresses the relation between logarithm of equilibrium constant and inverse of temperature with slope of 766.09:

$$\log K = 766.09(\mathrm{K}/T) - 1.6498 \qquad (12)$$

with $s_e = 0.009$. The average enthalpy of the sulfur dioxide dissolution in the temperature range of (293.15 to 313.15) K is calculated to be −14.7 ± 0.0002 kJ·mol^{-1}.

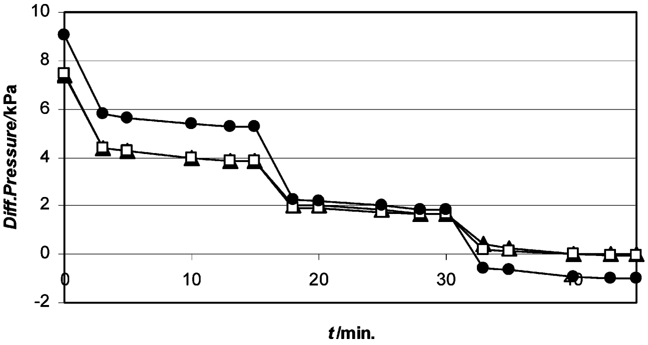

Figure 5. Change in sulfur dioxide absorption rate with three injections at 0, 15, and 30 min at 303.15 K: ▲, exp 1; □, exp 2; ●, exp 3.

Discussion

This paper presents the sulfur dioxide absorption capacity of heat transfer oil in the temperature range of (293.15 to 313.15) K with 5 K intervals. Empirical relations have been established, and the average heat of sulfur dioxide dissolution in the temperature range has been given.

The change in sulfur dioxide absorption rate has been followed by the differential pressure change with time. The graphs have been plotted by the data obtained during the experiments derived between (293.15 and 313.15) K. The graph for 303.15 K is given in Figure 5. As shown in the figure, sulfur dioxide absorption rate is very high in the first three minutes of each 15 min period, and most of the absorption capacity of the oil is being consumed. In the following minutes, the pressure change rate decreases in accordance with the decreasing absorption rate. The pressure change between 13 and 20 min is less than the pressure reading sensitivity, and a plateau has been observed at pre-experiments of each temperature. Therefore, the 15 min period is taken to reach the equilibrium state of absorption.

Since, the highest average mole fraction is around 0.14, the system can be accepted to obey Henry's law. The highest relative standard deviation for the Henry's constants is 4.28 % at 293.15 K, showing the reproducibility of the experimental work.

The established relations between the individual constants, such as Henry's constant, Ostwald's coefficient or solubility in unit volume of oil, and temperature have high confidence in evaluating the data in the working temperature range.

It can be stated that polar groups in organic solvents increase the sulfur dioxide solubility due to strong chemical interaction with sulfur or oxygen atoms of sulfur dioxide. In addition, in chemicals having unpaired electrons on oxygen atoms, such as diethyl ether,[10] diethylene glycol dimethyl ether,[13] or tetraethylene glycol dimethyl ether,[13] sulfur dioxide solubility increases with an increased number of oxygen atoms. Other chemicals which can act as Lewis bases also dissolve sulfur dioxide effectively. However, Transcal N is a mixture of saturated hydrocarbons and a nonpolar hydrophobic solvent. Therefore, comparing with the solvents such as 1,2-dichloroethane,[10] 1,2-ethanediol,[10] tetrahydrofuran,[14] diethyl ether,[10] diethylene glycol dimethyl ether,[13] tetraethylene glycol dimethyl ether,[13] dimethylacetamide,[13] or dimethylaniline,[15] Transcal N oil has a lower sulfur dioxide absorption capacity. Of the chemicals reported for sulfur dioxide absorption, 1,2-dichloroethane has the most hydrophobic character and performs higher absorption

Table 6. Henry's Constants *H* of Sulfur Dioxide in Different Organic Solvents and Transcal N at 298.15 K

solvent	*H*/kPa
Transcal N	814.83
1,2-dichloroethane[10]	427.86
1,2-ethanediol[10]	344.95
diethyl ether[10]	236.72
tetrahydrofuran[14]	39.19
diethylene glycol dimethyl ether[13]	26.79
tetraethylene glycol dimethyl ether[13]	21.79
dimethylacetamide[13]	11.89
dimethylaniline[15]	9.3

capacity than Transcal N. The sulfur dioxide Henry's constants of these solvents at 298.15 K and 101.3 kPa pressure are given in Table 6.

Sulfur dioxide absorption capacity of each organic solvent, given in Table 6, is higher than that of Transcal N. However, their physical properties are not suitable to be used at high flue gas temperatures or for regeneration. Most of them are low boiling and have low flash temperatures, affecting the maximum working temperature, and are soluble in water, making the regeneration process more difficult and expensive. Albeit its low sulfur dioxide absorption capacity, the superiority of Transcal N arises from its very high boiling and flash points, which makes its use safe at high flue gas temperatures. Its insolubility in water achieves an advantage to apply effective regeneration processes.

The experimental solubility values and equations in this study can be used as essential data. Further research for sulfur dioxide solubility at higher temperatures is being established along with regeneration studies.

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