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Solubility of Sulfur Dioxide in a Commercial Hydrocarbon Mixture in the Temperature Range of (303 to 343) K: Effect of Hydrocarbon Composition on Absorption

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ABSTRACT: The sulfur dioxide solubility in hydrocarbon mixtures mostly depends on the chemical composition of the solvent. The changes in noncyclic−cyclic and saturated−unsaturated parts affect not only the physical properties of the liquid but also the gas solubility. This work evaluates the solubility and average enthalpy of dissolution of sulfur dioxide in a commercial hydrocarbon mixture (heat transfer oil) in the temperature range of (303 to 343) K and at gas loading pressures below 250 kPa. The investigated commercial hydrocarbon mixture mainly consists of aliphatic alkanes according to the ^{1}H NMR, Fourier transform infrared (FT-IR), and gas chromatography/mass spectrometry (GC-MS) analyses. Gas 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 three different pressures including solubility−temperature relations in the working temperature range. The effect of solvent chemical composition on sulfur dioxide solubility is also discussed with respect to the previous studies on hydrocarbon mixtures. The presented solubility data provide essential knowledge for development of modified hydrocarbon mixtures with high-boiling points to create absorption solvents suitable for high flue gas temperatures based on thermally stable hydrocarbon matrices. The hydrocarbon matrices can be further improved by absorption capacity modifying agents to create novel solvent systems for cost-effective desulfurization processes.

INTRODUCTION

Sulfur dioxide emissions from the combustion of sulfur containing fuels build major pollution problems. Although there are some commercial scrubbing processes for sulfur dioxide, none has yet been proven to be totally effective in removing sulfur dioxide under all circumstances. Most of the desulfurization processes containing alkali substances such as metal hydroxides, amines, or limestone have discharges resulting in other pollution problems.

Most of the research related to sulfur dioxide absorption are based on polar hydrophilic chemicals having strong chemical interactions. Sulfur dioxide absorption in aqueous electrolyte solutions of sodium chloride,[1] ammonium chloride,[1] sulfuric acid,[2] or ammonia[3] gives ionic species which are difficult to regenerate. In fact these solutions are not suitable for elevated gas temperatures.

In recent years, ionic liquids have attracted more attention in sulfur dioxide absorption with their solubility capacity and stability. However, they also tend to lose their absorption capacity with the increase in temperature, and most of them degrade below 550 K.[4,5] Even if it is possible to synthesize tailored ionic liquids, thermal stability remains as a problem to be solved. Therefore, development of thermally stable new solvents or solvent systems is needed which show not only strong interactions with sulfur dioxide but are also possible to regenerate by cost and energy efficient processes. In this sense, solvent systems may offer reasonable solutions to sulfur dioxide absorption. Solvent systems based on thermally stable liquids as the main matrix and modifiers as the absorption capacity improvers might provide better solutions for effective and environmentally friendly scrubbing solutions.

The aim of this paper is to introduce the solubility of sulfur dioxide in commercial heat transfer oil which represents a thermally stable hydrocarbon mixture model for such solvent systems. The oil has been chosen for its nonpolar hydrophobic solvent character, hydrocarbon distribution, and broad operating temperature range. The influence of hydrocarbon composition on sulfur dioxide solubility is discussed and compared with literature data to throw light on the solubility interactions between sulfur dioxide and saturated hydrocarbons. Sulfur dioxide absorption capacity of the oil has been determined in the temperature interval of (303 to 343) K with 10 K temperature interval and at (80, 101.3, and 150) kPa sulfur dioxide gas pressures. Empirical relations have been established, and the average enthalpy of sulfur dioxide dissolution has been given.

EXPERIMENTAL SECTION

Materials. The heat transfer oil, MobilTherm 605, is a commercial product of Exxon Mobil, and it is a mixture of

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saturated hydrocarbons without any additives. Several analyses have been performed to determine the chemical composition, average empirical formula, and average molecular weight of the oil to be used in related calculations. The physical properties of the oil are given in Table 1.[6]

Table 1. Physical Properties of MobilTherm 605[6]

relative density at 288 K	0.872 g·cm^{-3}
viscosity at 313 K	29 cSt
viscosity at 373 K	5.1 cSt
flash point, open cup	483 K
pour point	258 K
maximum operating temperature	593 K
boiling point	>589 K

Sulfur dioxide has been supplied by Linde Gas AG at 99.99 % purity.

Determination of the Chemical Composition of the Heat Transfer Oil. Several analyses have been conducted to identify the chemical composition of the commercial oil, such as elemental analysis, Fourier transform infrared spectroscopy (FT-IR), gas chromatography/mass spectrometry (GC-MS), and ^{1}H NMR instrumental analyses. Elemental analysis was conducted with LECO Truspec CHN-S analyzer. FT-IR spectra were recorded on Perkin-Elmer FT-IR Spectrum 100 spectrometer with universal ATR accessory between (4000 and 650) cm^{-1} wavelengths. ^{1}H NMR data were recorded on a Bruker NMR spectrometer (500 MHz) in $CDCl_3$. GC-MS analysis was performed using Agilent HP 6890 N gas chromatography (GC) connected with 5975 B series mass spectrometry (MS). The HP-5MS capillary column with 30 m × 0.32 mm inner diameter (i.d.) 0.25 μm dimensions was used with the given GC temperature program: hold at 150 °C for 2 min, increase at 5 °C/min heating rate to 250 °C, and hold at 250 °C for 20 min. The lowest quantitation limits and sensitivities of the CHN-S analyzer for the elements were: C: 50 ppm, 25 ppm; H: 200 ppm, 100 ppm; N: 80 ppm, 40 ppm; and S: 10 ppm, 5 ppm, respectively. The precisions of analyses were 0.5 %, 1 %, 0.5 %, and < 1 % for C, H, N, and S, respectively. The average molecular weight and average empirical formula of the heat transfer oil were calculated with the help of GC-MS and elemental analyses data.

Solubility Measurement Setup. The solubility measurements were carried out using a gastight glass autoclave of Buchi AG which is illustrated in Figure 1. The system involved sulfur dioxide cylinder (A), HAAKE Phoenix II P2-B5 heating circulator (B), glass vessel with heating jacket (C), V-700 series Buchi vacuum pump (D), pressure sensor with ± 1 mbar sensitivity up to 3 bar (E), temperature sensor with ± 0.1 K sensitivity (F), and five four-bladed stirrers on a common shaft (G) with a controller (H) to ensure rapid physical equilibrium between the gas and the liquid phases. At least one stirrer rotated in the liquid. The system was connected to a PC controller with a transmitter (I), and pressure changes were monitored online using the software "Buchi bls 2".

The experimental procedure is similar to Olson,[7] Cukor and Prausnitz,[8] Graff et al.,[9] and Breman et al.[10] Briefly, the vessel is initially loaded with a known amount of liquid and gas at room temperature. Then, temperature is increased stepwise, and the gas−liquid system is allowed to reach equilibrium.

Heat transfer oil is a high-boiling point liquid, and it gives us the opportunity to load the vessel under vacuum at room temperature. Thus, a pure sulfur dioxide load is guaranteed with a minimized air ratio in the system.

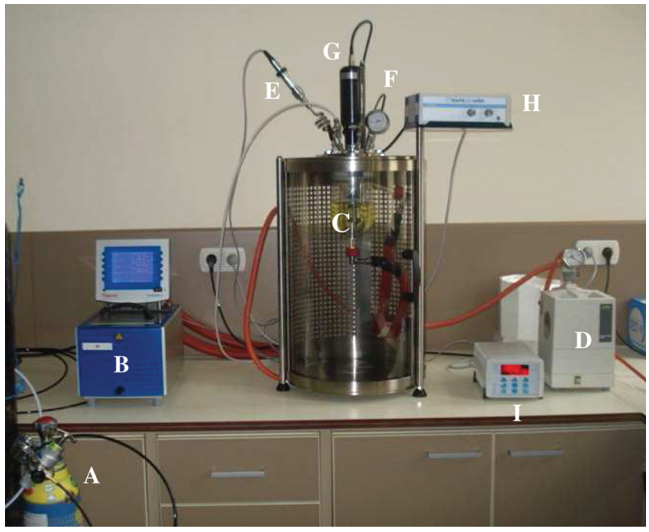

Figure 1. Experimental setup of solubility measurements.

RESULTS AND DISCUSSION

Chemical Composition of the Commercial Hydrocarbon Mixture. The commercial heat transfer oil sold by Exxon Mobil under the trade name MobilTherm 605 represents a hydrocarbon mixture model to observe the solubility behavior of sulfur dioxide in predominantly saturated hydrocarbon mixtures. According to the technical specifications of the product, it is stable up to 593 K which also defines its maximum operating temperature. The stated temperature limit is suitable for flue gas desulfurization processes as the common flue gas temperature interval is between (383 and 473) K.

The chemical composition of the oil has been determined by ^{1}H NMR, FT-IR, GC-MS, and elemental analyses, and gas solubility data have been presented between (303 and 343) K with a 10 K temperature interval and at pressures of (80, 101.3, and 150) kPa.

According to the elemental analysis and GC-MS data, the heat transfer oil does not contain N compounds in the detection limits but contains negligible amounts of S containing moieties and consists of mainly saturated hydrocarbons. Consequently, it has nonpolar and hydrophobic character. The elemental analysis indicates that the mass fraction of the oil is: C, 0.8454; H, 0.1530; N, 0.0000; S, 0.0031, and the empirical formula is: $(CH_{2.155})_n$.

Regarding the ^{1}H NMR, IR, and GC-MS data, MobilTherm 605 is a homogeneous mixture of mainly saturated hydrocarbons between 16 and 30 carbon atoms. It contains aromatic hydrocarbons in a 1:400 ratio approximately. The FT-IR spectrum shows absorptions (cm^{-1}) at 2921 and 2855 (s, C−H stretch), 1458 (m, C−H bend), 1377 (m, C−H rock), and 724 (w, C−H rock). Olefinic and aromatic (=C−H) bands around 3030 cm^{-1} and C=C vibrations around (1660 to 1590) cm^{-1} which belong to the aromatic hydrocarbons do not appear well. However, ^{1}H NMR data obtained at 500 MHz indicate the presence of aromatic compounds in the approximate ratio of 1:400. Aromatic protons and aliphatic saturated hydrocarbon protons have been observed as multiplets between chemical shifts (δ) of (6.76 to 7.18) ppm and (0.60 to 2.70) ppm, respectively. The FT-IR spectra are given in Figure 2.

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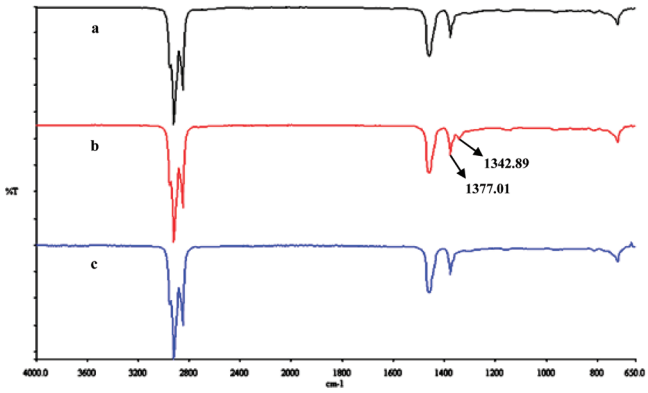

Figure 2. FT-IR spectra: (a) heat transfer oil, (b) after absorption, and (c) after scrubbing.

The average molecular weight and the average empirical formula of the heat transfer oil which were calculated according to the data of GC-MS and elemental analyses are as follows: average molecular weight, 330.67 g·mol^{-1}, and average empirical formula, $C_{23.33}H_{50.28}$.

The gas solubility is directly related to the composition of the liquid phase. Therefore, the determination of the chemical composition presents valuable data to clarify the effect of the carbon distribution on solubility. The chemical composition of the investigated hydrocarbon mixture is significantly different than the previously investigated heat transfer oil in literature,[11] and the influence of saturated hydrocarbon composition on solubility is clear in the presented solubility data comparison.

Gas−Liquid Equilibrium Determination and the Effect of Chemical Composition. Due to the physical interactions between sulfur dioxide and the heat transfer oil, the absorption mechanism is also reversible. Such a behavior is seen on the FT-IR spectra in Figure 2. The absorbed sulfur dioxide is seen at 1342.89 cm^{-1}, and the regenerated oil is free of sulfur dioxide after 30 min of nitrogen gas scrubbing under atmospheric conditions. Therefore, it can be stated that sulfur dioxide is physically absorbed in the solvent and the system equilibrium can be reset by changing temperature without charging fresh oil. This property is also favorable for the simple regeneration of the oil in applications of flue gas-desulfurization.

The physical interactions between sulfur dioxide and hydrocarbons allow the simultaneous measurement of solubility at different temperatures. The dynamic condition of physical-absorption equilibrium enables changing the system temperature to set a new balance between gas and liquid phases.

The net volume of the gas-tight glass system is 500 mL, and the introduced liquid volume is between (75 and 80) mL in each absorption experiment run. The experiment procedure is briefly as follows: the liquid is initially degassed under vacuum and at room temperature for 30 min. Then, the gas is introduced into the system at room temperature up to the desired starting pressure with online data logging. The initial pressure at room temperature is converted to the pressure at initial measurement temperature according to the ideal gas law, which represents the starting pressure condition of the system. Such an approach is meaningful because absorption takes place continuously at each temperature after the gas is introduced into the system. So that, the absorbed gas amount during the time needed to heat up the system to 303 K is also taken into consideration in calculations to estimate the total absorbed gas at the desired temperature correctly with the pressure difference.

When the loaded system reaches the desired temperature, both liquid and gas phases are stirred vigorously at 500 rpm to enhance the optimum contact. The equilibrium pressure is recorded after constant equilibrium pressure is reached and the stirrer is stopped. The next equilibrium pressure is recorded when the system reaches the next desired temperature and the new balance is established.

The gas solubility data of sulfur dioxide in heat transfer oil are calculated at the desired pressures by using the equilibrium data of at least four experiments with different initial gas loading pressures. The initial gas loading pressures range from (85 to 240) kPa at 303 K, and the solubility experiments are conducted between (303 and 343) K with 10 K temperature interval. Since the heat transfer oil has high flash, fire, and boiling points, the vapor pressure of MobilTherm 605 is assumed to be negligible in the working temperature range, and the pressure drop by sulfur dioxide absorption is assumed to be insignificantly influenced by the expansion of the oil.

Solubility has been expressed in four different units including mole fraction of the absorbed gas, Henry's constant, Ostwald's coefficient, and gas solubility in unit volumes of liquid. The relationship between temperature and solubility is also given to clarify the thermal behavior of the gas−liquid system at different temperatures.

The mole fraction expresses the sulfur dioxide solute proportion on total molar bases of solute and solvent. If the moles of solute is n_n(oil) and total number of moles of solvent and solute is N_n(oil) + n_n(oil) in the liquid phase, then the mole fraction is expressed as follows:

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

The sulfur dioxide mole fractions were calculated between (303 to 343) K at three different pressures and presented in Table 2 with related statistical calculations. Since the highest average mole fraction is around 0.07 at 303 K and under 150 kPa, the system can be accepted to obey Henry's law.

From the definition of Henry's law, the Henry's constant is expressed as a proportion constant between the partial pressure of the gas and the mole fraction of the gas in the solution at a constant temperature and solubility equilibrium. The calculated constant of gas solubility should be specified with the partial pressure of the solute gas and its temperature. The Henry's constant of sulfur dioxide in the hydrocarbon mixture is tabulated in Table 2, and the relation between Henry's constant and temperature change is given in Figure 3 at 101.3 kPa.

In addition to Henry's constant, which defines the solubility in terms of pressure units, Ostwald's coefficient defines solubility in terms of volumetric ratio of absorption. If the volumetric expansion of solvent with dissolved solute is negligible, it can be assumed that ideal gas behavior is acceptable. If V^0_{oil} is the volume of pure oil at a specified temperature and V_{gas} is the volume of absorbed gas, then the Ostwald's coefficient (L_v^0) is calculated as follows:

$$L_v^0/\text{mL}_{SO_2}\cdot \text{mL}_{oil}^{-1} = (V_g/V_L^0)_{equil}$$

The Ostwald's coefficients of sulfur dioxide in hydrocarbon mixture are given in Table 3. The change in the volumetric solubility ratio with temperature at 101.3 kPa fits to the given equation in Figure 4.

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Table 2. Sulfur Dioxide Mole Fraction x and Henry's Constant H/(kPa) between (303 and 343) K

		x			H/(kPa)		
	pressure (kPa)	80.0	101.3	150.0	80.0	101.3	150.0
303 K	mean value	0.0348	0.0449	0.0680	2295.8	2254.8	2206.8
	std. deviation	0.0001	0.0003	0.0009	7.3	15.2	27.7
	95 % conf. interval	±0.0001	±0.0003	±0.0009	±7.7	±16.0	±29.0
313 K	mean value	0.0278	0.0358	0.0531	2873.6	2833.3	2824.8
	std. deviation	0.0002	0.0002	0.0008	19.5	16.2	40.3
	95 % conf. interval	±0.0002	±0.0002	±0.0008	±20.4	±17.0	±42.3
323 K	mean value	0.0224	0.0288	0.0429	3575.6	3518.7	3495.7
	std. deviation	0.0002	0.0002	0.0003	31.2	20.9	22.7
	95 % conf. interval	±0.0002	±0.0002	±0.0003	±32.7	±21.9	±23.9
333 K	mean value	0.0187	0.0237	0.0352	4280.2	4269.2	4256.5
	std. deviation	0.0002	0.0001	0.0003	47.2	14.7	39.7
	95 % conf. interval	±0.0002	±0.0001	±0.0003	±49.6	±15.4	±41.7
343 K	mean value	0.0159	0.0201	0.0299	5039.2	5027.4	5014.2
	std. deviation	0.0002	0.0001	0.0003	62.7	28.0	53.9
	95 % conf. interval	±0.0002	±0.0001	±0.0003	±58.0	±25.9	±49.9

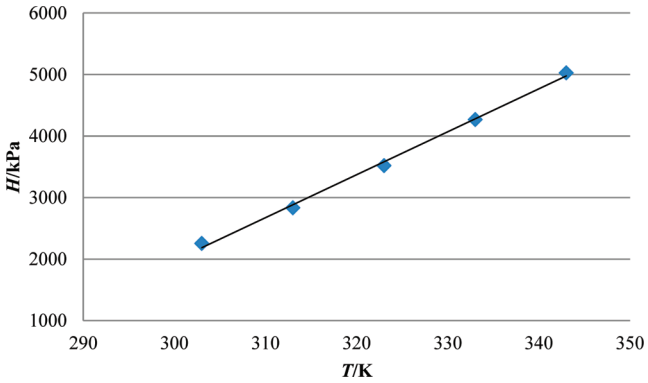

Figure 3. Temperature dependence of Henry's constant of sulfur dioxide in hydrocarbon mixture at 101.3 kPa, x = 69.811(T/K) − 18968, R^2 = 0.99.

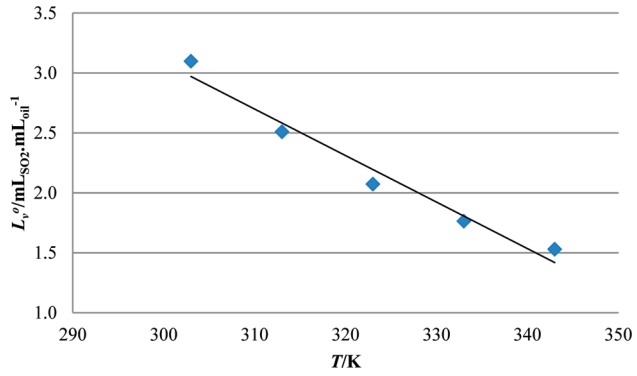

Figure 4. Temperature dependence of Oswald's coefficient of sulfur dioxide in hydrocarbon mixture at 101.3 kPa, x = −0.0388(T/K) + 14.7314, R^2 = 0.97.

The Ostwald's coefficient provides the data to calculate the amount of sulfur dioxide in unit volume of hydrocarbon mixture. Thereby, the sulfur dioxide solubility which represents grams of absorbed sulfur dioxide in unit liters of hydrocarbon mixture is given in Table 3. The relative standard deviation of the presented data in the tables is less than 1.50 %, which shows the repeatability of the solubility experiments.

Based on the solubility data, the enthalpy of dissolution of sulfur dioxide in heat transfer oil can be calculated with respect to equilibrium constant K of the system at different equilibrium

Table 3. Sulfur Dioxide Ostwald's Coefficient L_v^0/(mL_{SO_2}·mL_{oil}^{-1}) and Solubility s/(g_{SO_2}·L_{oil}^{-1}) between (303 and 343) K

		L_v^0/(mL_{SO_2}·mL_{oil}^{-1})			s/(g_{SO_2}·L_{oil}^{-1})		
	pressure (kPa)	80.0	101.3	150.0	80.0	101.3	150.0
303 K	mean value	2.993	3.098	3.210	6.112	7.965	12.199
	std. deviation	0.012	0.010	0.087	0.009	0.034	0.113
	95 % conf. interval	±0.015	±0.012	±0.107	±0.011	±0.042	±0.140
313 K	mean value	2.440	2.509	2.545	4.837	6.232	9.419
	std. deviation	0.015	0.023	0.034	0.036	0.034	0.108
	95 % conf. interval	±0.019	±0.028	±0.043	±0.044	±0.043	±0.134
323 K	mean value	2.014	2.073	2.121	3.879	4.991	7.533
	std. deviation	0.005	0.013	0.011	0.036	0.022	0.075
	95 % conf. interval	±0.007	±0.016	±0.013	±0.045	±0.028	±0.093
333 K	mean value	1.710	1.764	1.802	3.220	4.120	6.176
	std. deviation	0.001	0.002	0.010	0.028	0.003	0.058
	95 % conf. interval	±0.002	±0.003	±0.012	±0.035	±0.004	±0.072
343 K	mean value	1.487	1.530	1.566	2.738	3.485	5.191
	std. deviation	0.002	0.002	0.007	0.033	0.009	0.048
	95 % conf. interval	±0.002	±0.002	±0.009	±0.041	±0.011	±0.060

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temperatures. Due to the nonvolatile character of the oil, K can be calculated at each temperature as follows:

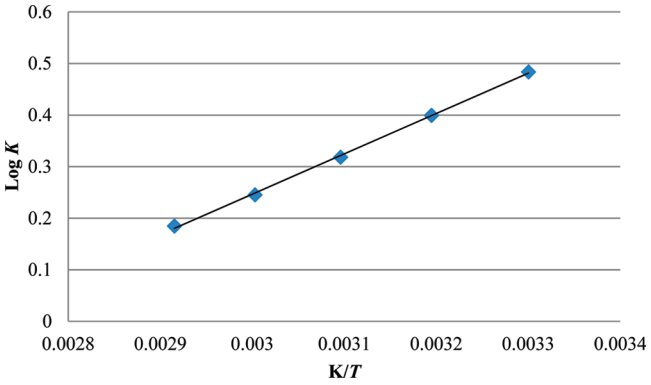

Figure 5. Plot of log K vs K/T.

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

The slope of log K versus K/T plot is used to calculate the average enthalpy of dissolution in Figure 5. The following equation expresses the correlation between the logarithm of equilibrium constant and inverse of the temperature with a slope of 783.084:

$$\log K = 783.084(K/T) - 2.1028$$

with $R^2 = 0.99$. The average enthalpy of sulfur dioxide dissolution in the temperature range of (303 to 343) K is calculated to be −14.99 kJ·mol^{-1}.

The sulfur dioxide solubility is highly dependent on the hydrocarbon composition of the oil. However, it is hard to find more adequate data to clarify the solubility behavior of sulfur dioxide and its relation with chemical structure differences in detail. Limited research in literature based on the determination of gas−liquid interactions between sulfur dioxide and hydrocarbon mixtures or specific sole hydrocarbons obstructs making more comments on sulfur dioxide−hydrocarbon interactions.

The presented solubility data introduce the sulfur dioxide solubility in predominantly saturated hydrocarbons between 16 and 30 carbon atoms in which approximately 25 % of it has less than 21 carbon atoms with average molecular weight of 330.67 g·mol^{-1}.

The effect of chemical composition on sulfur dioxide solubility is clearly seen when the data is compared with literature. The previously investigated oil, Transcal N, has carbon number range of 14 to 30. Around 20 % of the oil consists of hydrocarbons with less than 21 carbon atoms, and 15 % of the composition is cyclic alkanes. In addition to the differences in oil composition, Transcal N is also heavier than MobilTherm 605. It has an average molecular weight of 367.31 g·mol^{-1} and average empirical formula of $C_{25.17}H_{64.48}$.[11] Even if both of the hydrocarbon mixtures are predominantly saturated and show physical interactions with sulfur dioxide, the chain length and the structure of the saturated hydrocarbon composition directly affect the solubility.

It can be stated that the presence of cyclic alkanes and/or n-alkanes with higher carbon number enhances the solubility of sulfur dioxide. Sulfur dioxide is more than two times more soluble in Transcal N than in MobilTherm 605 at 101.3 kPa. The physical interactions between the hydrocarbon mixture and sulfur dioxide get stronger and the sulfur dioxide molecule is held more firmly as the average molecular size increases. The compared data are tabulated in Table 4 to show the significant differences in terms of Henry's constant and sulfur dioxide solubility in unit solvent volume. However, further research on sulfur dioxide solubility is still needed to clarify the effects of chain length and structure of the hydrocarbon on solubility in more detail.

Table 4. Sulfur Dioxide Solubility in Transcal N[11] and MobilTherm 605 at 101.3 kPa

	Transcal N[11]	MobilTherm 605
	Henry's Constant (kPa)	
303 K	894.30	2254.8
313 K	1085.39	2833.3
	Solubility in Unit Volume (g·L^{-1})	
303 K	19.486	7.965
313 K	15.704	6.232

Although the chemicals having unpaired electrons on oxygen atoms in their chemical structure like diethyl ether[12] or diethylene gylcol-dimethyl ether[13] have superior gas solubility, their physical properties are not suitable to be used at high flue gas temperatures. Most of them have low flash and boiling points affecting the maximum working temperature and are soluble in water which makes the regeneration process more difficult and expensive. Some give explosive mixtures at several limits which may cause explosion during absorption process. However, the hydrocarbon mixtures can operate safely at flue-gas temperatures between (383 and 473) K with their high flash and boiling points and thermal stability.

CONCLUSION

Based on the available data on hydrocarbon mixtures and sulfur dioxide solubility in literature, it can be stated that the nonpolar hydrophobic character of the hydrocarbons positively affects the sulfur dioxide solubility with an increase in average molecular structure and/or presence of cyclic alkanes.

The presented work indicated that sulfur dioxide, which is one of the major components of the flue gas generated from the combustion of fossil fuels, could be physically and reversibly absorbed in extremely high boiling hydrocarbon mixtures.

Further research on sulfur dioxide solubility in other commercial or particularly produced hydrocarbon mixtures with known compositions would be meaningful to clarify the relation between solubility and hydrocarbon composition. So that, suitable thermally stable matrix systems can be chosen to enhance the sulfur dioxide absorption capacity which can be further improved by absorption capacity modifying agents to create novel solvent systems. The physical absorption mechanism between the two phases provides simple regeneration of the oil which is cost- and energy-effective compared to other desulfurization processes. In this sense, a new cost-effective desulfurization process can be developed if more data on sulfur dioxide solubility in hydrocarbons are available.

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Notes
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