Destruction of carbon disulfide in aqueous solutions by sonochemical oxidation

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Abstract

Carbon disulfide (CS_2) is toxic to animals and aquatic organisms, and can also decompose to carbonyl sulfide (OCS) and hydrogen sulfide (H_2S) in aqueous environment. The kinetics of the sonochemical degradation of aqueous CS_2 was studied in a batch reactor at 20 kHz and 20 °C, and the effects of process parameters (e.g. concentration, ultrasonic intensity, irradiating gas) investigated. The concentrations of unbuffered CS_2 solutions used were $(6.4$–$7.0) \times 10^{-4}$, 10.5×10^{-4} and $(13.2$–$13.6) \times 10^{-4}$ M and the intensities were varied from 14 to 50 W. The reaction rate was found to be zero-order and the rate constant for the degradation at 20 °C and 14 W in air was 21.1 μM/min using the largest initial concentration range studied. At the same initial concentration range but at 50 W (39.47 W/m^2) the degradation rate of CS_2 was 46.7 μM/min, more than two times that at 14 W (11.04 W/m^2). The rate of CS_2 sonochemical degradation in the presence of the different gases was in the order of He > air ≥ N_2O > Ar; the rate with helium was found to be about three times that of argon. The formation of sulfate (SO_4^{2-}) as reaction product with air as the irradiating gas was enhanced in the presence of hydrogen peroxide (H_2O_2) and inhibited in the presence of 1-butanol. The sonochemical oxidation of CS_2 may prove to be an efficient and environmentally benign way for the removal of this hazardous pollutant from natural water and wastewater. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Carbon disulfide; Sonochemical oxidation; Ultrasound; Ultrasonic irradiation; Degradation; Kinetics; Reaction pathways

1. Introduction

Organic and inorganic compounds containing sulfur and nitrogen are the main causes of odor in natural waters and process wastewater. Control of biogenic sulfur (or reduced

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sulfur) emissions is a key environmental concern in the purification of natural gas, viscose rayon manufacture, tanneries and in kraft pulp and petroleum refining industries. These compounds include hydrogen sulfide (H_2S), carbon disulfide (CS_2), alkyl sulfides (RSR), alkyl disulfides (RSSR), and alkylthiols or mercaptans (RSH) [1–3]. CS_2 is a volatile liquid with pungent smell. CS_2 is also classified as a hazardous air pollutant under Title III of the 1990 Clean Air Act Amendment (CAAA) of the United States [4]. This has triggered an increased interest to find reliable, cost effective technologies for its control.

The kinetics and mechanisms of oxidation of CS_2 in aqueous solutions by hydrogen peroxide (H_2O_2) were studied in detail by previous investigators [1–3]. Entezari et al. [5] investigated the sonochemical degradation of CS_2 liquid and the effects of frequency, temperature, intensity and gases on the rate of its dissociation. They found that ultrasonic irradiation of the CS_2 liquid at 20 kHz resulted in the formation of a heterogeneous mixture of black particles (amorphous carbon) in a yellow solution (monoclinic sulfur). Sonochemical techniques involve the use of sonic or ultrasonic waves to produce an oxidative environment via cavitation that yields localized microbubbles and supercritical regions in the aqueous phase. The collapse of these bubbles leads to surprisingly high local temperatures and pressures. Locally the high temperature and pressure may reach up to and above 5000 °C and 500 atm, respectively [6]. These rather extreme conditions are very short lived, but have shown to result in the generation of highly reactive species including hydroxyl ($^\bullet$OH), hydrogen ($H^\bullet$) and hydroperoxyl ($HO_2^\bullet$) radicals, and H_2O_2 [7–10].

Adewuyi [7] recently published a comprehensive review of sonochemistry and environmental applications. To further understand the chemical transformation of reduced sulfur compounds and their relevance in advanced environmental remediation systems, we have investigated sonochemical oxidation as an environmentally benign process for removing CS_2 from aqueous solutions. The kinetics of the sonochemical oxidation of aqueous CS_2 at 20 kHz and 20 °C in a batch reactor and the effects of process parameters (e.g. concentration, ultrasonic intensity, irradiating gas) are reported.

2. Experimental section

2.1. Reactor system

A simplified schematic of the experimental equipment used is illustrated in Fig. 1. The setup basically consists of a Branson model 450 sonifer, jacketed glass reactor, sound abatement enclosure box and circulatory water bath. The 20 kHz sonifer is capable of a maximum power output of 400 W. The 250 ml reactor (from Ace Glass Incorporated) was fitted with accessories including a gas dispersion tube, a mercury thermometer and a syringe for taking samples at regular time intervals. A Neslab constant temperature refrigerated bath circulator (Model RTE-100) was used to regulate reaction temperature by flowing tap water pre-set at desired temperatures in annular space of the jacketed reaction vessel.

2.2. Reagents

Stock solutions were obtained from appropriate amounts of reagent-grade CS_2, H_2O_2 and 1-butanol (from Sigma–Aldrich Company) and de-ionized water (from a Barnstead
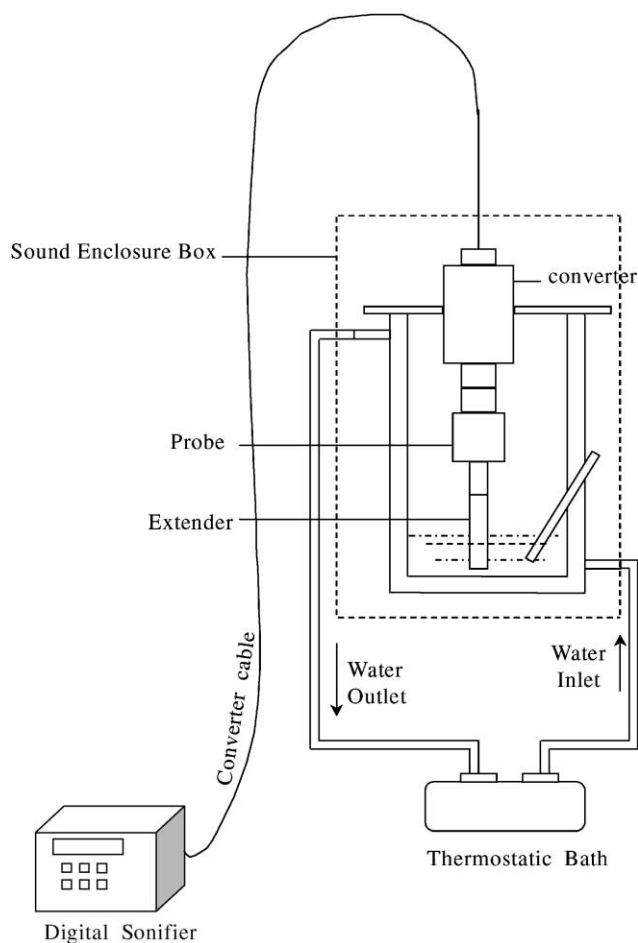
Fig. 1. Experimental setup of sonochemical reacting system.

mega-pure system) in 1000 ml flat bottom flask. The procedure was first to pipette the required amount of CS_2 using an eppendorf reference pipette (100–1000 µl) into the flask, and fill the flask with about 500 ml of de-ionized water. A stirrer was dropped into the solution and the flask placed on a magnetic stirrer. Strips of parafilm (MA) laboratory film was wrapped around the mouth of flask to prevent any of the sample from escaping since CS_2 is volatile. Stirring was then initiated to mix the two liquids and after achieving a homogenous solution, de-ionized water was added to make it to the 1000 ml mark. The stirring was done for a time period exceeding 6 h to ensure uniform mixing and a homogenous solution. After stirring was complete, the solution was left overnight to attain dynamic equilibrium. Ultra-pure grade (UPC) air, nitrous oxide (N_2O), helium or argon used as saturating gas was obtained from Air Products Company, Ltd.

2.3. Experimental procedures

A stock solution (50 ml) with known concentration of CS_2 (0.02, 0.03 0.04 mol/l) was poured into the reactor. The reactor was immediately sealed to prevent any CS_2 from volatizing from the solution. A gas dispersion tube was inserted in the reactor below the surface of the solution and UPC air or the nucleating gas was bubbled into the sample for a period of 30 min to saturate the solution. The absorbance of the solution was read using a Beckman DU-7000 spectrophotometer set at 314 nm. The wavelength of 314 nm was used for analyzing the sample solutions for all the experimental runs in this study. The bubbling process reduced the prepared 0.02, 0.03 and 0.04 M solutions to typically $(6.4–7.0) \times 10^{-4}$, 10.5×10^{-4} and $(13.2–13.6) \times 10^{-4}$ M, respectively. Kinetic runs were carried out by initiating the sonication of the solution for the desired length of time. After the time had elapsed, a syringe was used to withdraw about 4 ml of sample into UV-silica cuvette for spectrophotometric analysis. Typical absorbance versus time curves for the concentrations of interest are shown in Fig. 2. The concentrations of sample solutions were determined from a calibration curve prepared from stock solutions. The reactor was then washed thoroughly with de-ionized water to remove any traces of contaminant or any intermediates formed during sonolysis. The edge of the extender was also cleaned and the procedure was repeated
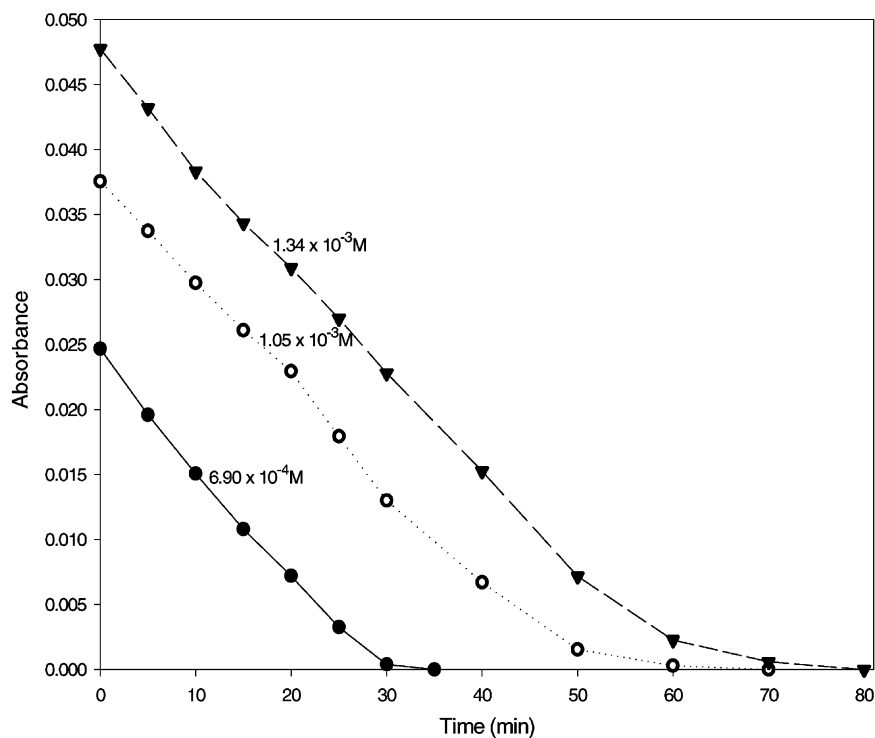

Fig. 2. Absorbance of CS_2 solution as a function of time during degradation at different initial concentrations, $[CS_2]_0$ ($T = 20\,^{\circ}C$, $P = 14\,W$)

for a different length of time until CS_2 is completely degraded. The products of degradation (nitrite, nitrate, sulfate (SO_4^{2-}), sulfite (SO_3^{2-}), thiosulfate ($S_2O_3^{2-}$)) were monitored with DIONEX DX 500 ion chromatograph, a DIONEX IonPac® AS11 2 mm analytical column, and a DIONEX EG 40 Eluent Generator using 1 M potassium hydroxide as eluent. Calibration curves for nitrite, nitrate, SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$ were obtained from 100 to 1000 mg/l standards purchased from High-Purity Standards Inc. The solution pH was measured with Fisher scientific pH/ion/conductivity meter (model 50).

3. Results and discussion

In order to determine the feasibility of decontaminating wastewater contaminated with reduced sulfur compounds and the rate constants for oxidation, aqueous solutions of CS_2 were irradiated with ultrasound in neutral, unbuffered aqueous medium at a frequency of 20 kHz and different intensities, initial CS_2 concentrations and gaseous media. The experimental conditions and results are summarized in Table 1. As shown in Figs. 2 and 3, CS_2 is effectively removed by ultrasonic irradiation under the conditions studied. Figs. 2 and 3 illustrate the changes in the absorbance readings and concentrations of CS_2, respectively with time at 20 °C and power of 14 W (or intensity of 11.05 W/cm^2). Using initial concentrations of $(6.4–7.0) \times 10^{-4}$, 10.5×10^{-4} and $(13.2–13.6) \times 10^{-4}$ M, CS_2 was found to be completely depleted after irradiation times of 30, 60 and 70 min, respectively.

3.1. Effect of experimental variables

Different irradiating gases were used in the experiments in order to investigate the effect of the physical and chemical properties of these gases on the sonochemistry of the reaction. The dissolved gases act as nucleation sites for cavitation. The physical properties of CS_2 and the irradiating gases are shown in Table 2. Although helium and argon have the same polytropic ratio, the thermal conductivity of helium is ten times greater than that of argon. Also, helium has a relatively low solubility in comparison to argon. The results obtained for air, argon, helium and N_2O at the same experimental conditions are shown in Table 1

Table 1
Summary of results for aqueous degradation of CS_2

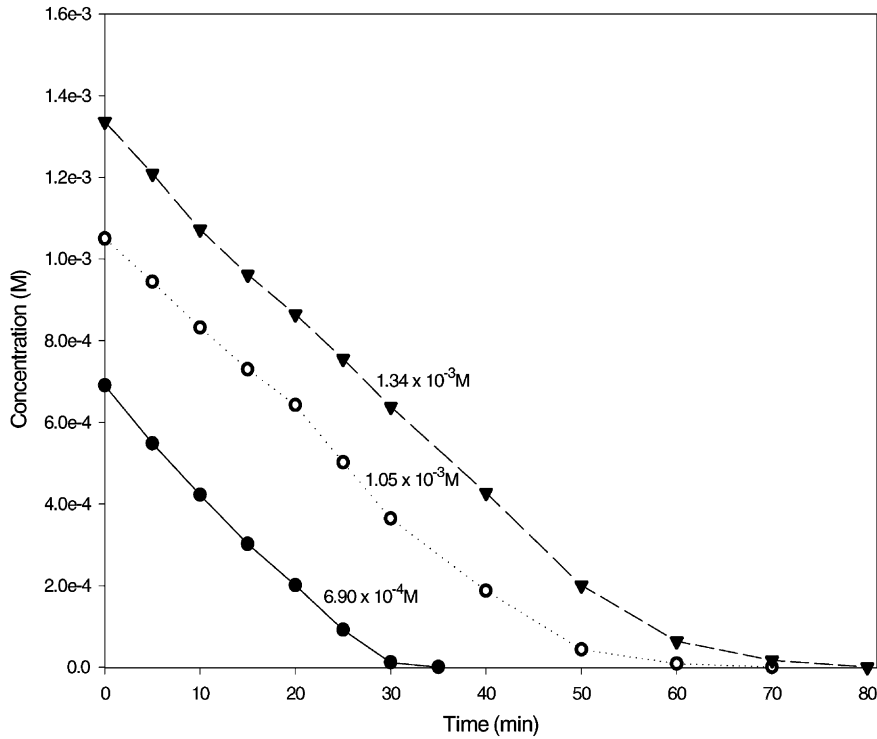| Initial concentration ($\times 10^4$ M)	Temperature (°C)	Power (W)	Gas medium	Rate constant (k) ($\times 10^5$ M/min)	R^2
6.90	20	14	Air	2.27	0.9981
10.51	20	14	Air	2.07	0.9919
13.36	20	14	Air	2.11	0.9966
13.51	20	14	N_2O	2.03	0.9982
13.56	20	14	He	3.07	0.9539
13.44	20	14	Ar	1.20	0.9928
13.36	20	30	Air	3.62	0.9510
13.31	20	50	Air	4.67	0.9689

Fig. 3. Concentration of CS_2 as a function of time during degradation at different initial concentrations, $[CS_2]_0$ ($T = 20\,^{\circ}C$, $P = 14\,W$)

and Fig. 4. The rate of sonochemical degradation of CS_2 in the presence of the different gases was in the order of He > air $\geq$ N_2O > Ar; the rate with helium is about three times of argon. The cavitation bubbles generated by ultrasound contain these gases in addition to CS_2 vapor. The nature of the gas in the bubble can have dramatic effects on the cavitational collapse.

There are several properties of gases that can affect sonochemical activities. These include polytropic ratio (γ) or ratio of heat capacity (C_p/C_v), thermal conductivity and solubility. The polytropic ratio, γ, of a gas affects the cause of reaction. Higher temperatures and

Table 2
Physical properties of CS_2 and dissolved gases

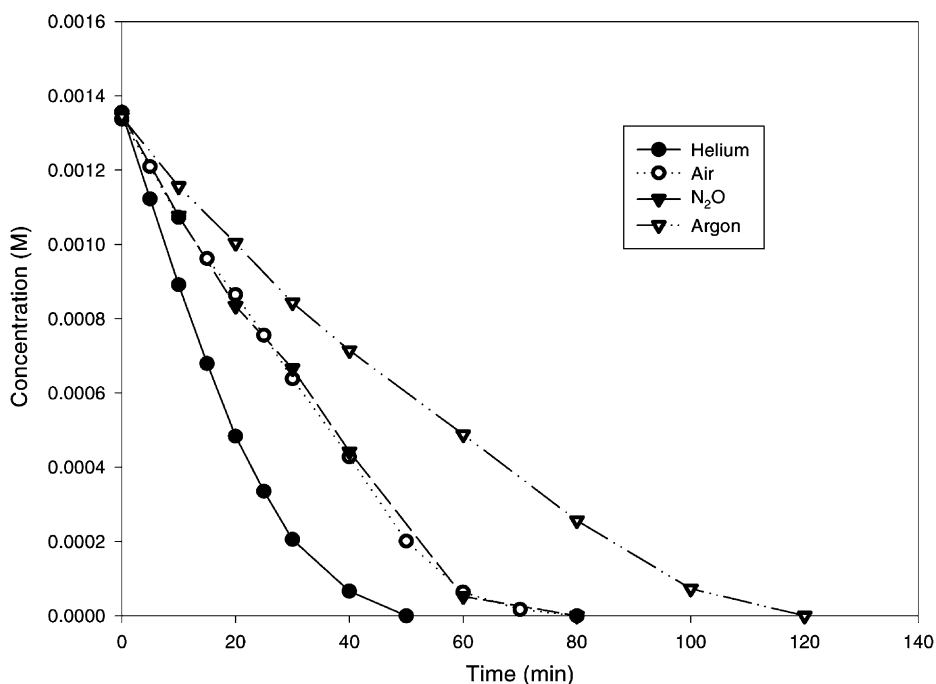| Gas medium	Heat capacity ratio at 298 K	Thermal conductivity at 300 K (μCal/s cm K)	Solubility at 273 K (ml/100 ml H_2O)
Air	1.40	58.3	2.89
Ar	1.67	42.4	5.60
He	1.67	357.5	0.94
N_2O	1.27	41.2	124.5
CS_2	1.22	16.5	60.10

Fig. 4. Concentration of CS_2 solution as a function of time during degradation in the presence of different gases, ($[CS_2]_0 = 1.33 \times 10^{-3}$ to 1.36×10^{-3} M, $T = 20\,^\circ$C, $P = 14$ W).

pressures are generated with monatomic gases with higher γ than with polyatomic gases with lower γ. The gas with low thermal conductivity reduces heat dissipation from cavitation site following adiabatic collapse, favoring high collapse temperature. The solubility of the gas in the liquid used is also an important aspect; the more soluble the gas, the more likely it is to diffuse into the cavitation bubble and cushion the implosion. Since dissolved gases form the nuclei for cavitation, soluble gases are expected to result in the formation of larger number of cavitation nuclei and extensive bubble collapse since these gases are readily forced back to the aqueous phase. In the study of the rate of dissociation of CS_2, Entezari et al. [5] obtained results similar to ours, with the rate decreasing in the order: He > H_2 > air > Ar > O_2 > CO_2. Since γ for argon is greater than hydrogen and argon has lower heat conductivity than helium, they concluded that the predominating factor in the system was gas solubility. The "hot spot" theory would suggest that the sonochemical activity should be higher under Ar than He due to the lower heat conductivity of Ar than He [7,9,10]. Our results indicate the opposite, suggesting that the cushioning effect of the more soluble gas (i.e. argon) predominated.

As shown in Table 1 and Fig. 5, using the same probe area, reaction rate is greater at higher power and, hence, higher intensity. With the same initial concentrations and temperature, and in the presence of air, the degradation rate of CS_2 at 50 W (39.47 W/m^2) is more than two times that at 14 W (11.04 W/m^2). The results are consistent with the sonochemical cavitational theories and the results obtained by other investigators [7,8]. An increase in
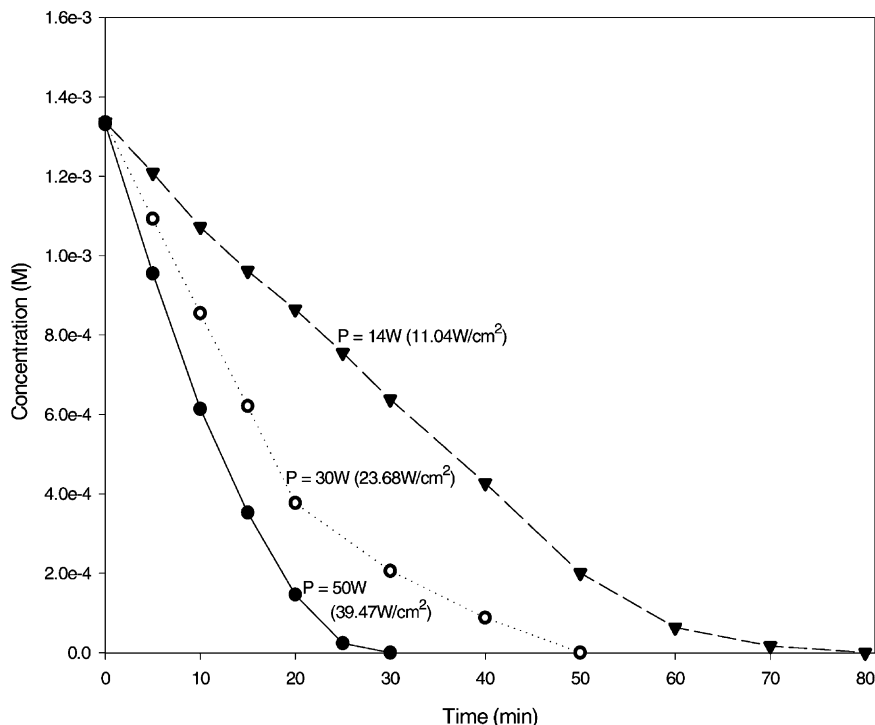
Fig. 5. Concentration as a function of time for CS_2 degradation at different powers or intensities ($[CS_2]_0 = (1.33–1.34) \times 10^{-3}$ M, $T = 20\,^\circ$C)

ultrasound intensity means an increase in the acoustic amplitude. The collapse time, the temperature, and the pressure on collapse are all dependent on acoustic amplitude. Hence, the cavitation bubble collapses more violently at higher intensities or acoustic amplitudes. An increase in intensity will, thus, result in greater sonochemical effects, resulting in higher CS_2 degradation rates.

3.2. Reaction kinetics and product analysis

The experimental runs at $20\,^\circ$C were analyzed using Eq. (1):

$$C = C_0 - kt \tag{1}$$

where k is the reaction rate constant. The plots obtained using Eq. (1) for different initial CS_2 concentrations at $20\,^\circ$C are shown in Fig. 6. These plots indicate the reactions are zero-order at the experimental conditions studied. As shown in Table 1, the zero-order rate constant ranges from 20.3 to 46.7 μM/min depending on the ultrasonic power or intensity and the irradiating gas used. However, the effect of initial CS_2 concentration appears not to be significant in the $[CS_2]_0$ range of $(6.90–13.36) \times 10^{-5}$ M. The zero-order kinetics is typical of hydroxyl radical reactions, since free radicals are generated at a constant rate under ultrasonic irradiation [11].
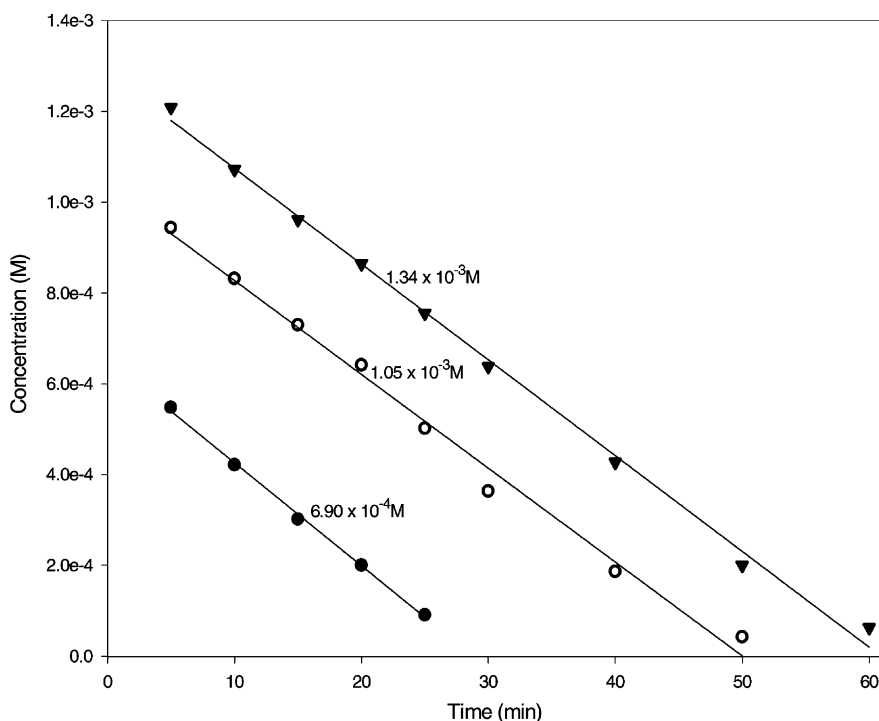
Fig. 6. Zero-order kinetic degradation of CS_2 at different initial concentrations, $[CS_2]_0$ ($T = 20\,^{\circ}C$, $P = 14\,W$).

As shown in Table 3, CS_2 appears to be oxidized to SO_4^{2-} by $^{\bullet}OH$ radical reactions at the interface of the bubble or in the liquid phase as the radical diffused out of the cavitation bubble. The formation of SO_4^{2-} as reaction product with air as the irradiating gas is enhanced in the presence of H_2O_2 while it is drastically inhibited in the presence

Table 3
SO_4^{2-} production at 20 °C and $P = 50\,W$: effect of H_2O_2 or 1-butanol addition[a]

$[CS_2]_0$ ($\times 10^3$ M)	$[H_2O_2]_0$ (mg)	$[1\text{-Butanol}]_0$ (mg)	pH_0	pH_f	Irradiation time (h)	SO_4^{2-} ($\times 10^3$ M)
1.364	0	0	7.0[b]	4.8	6	1.039
1.394	0	0	7.1	4.3	6	1.182
1.370	0	200	7.1	4.8	6	0.483
1.375	100	0	7.1	4.7	6	1.039
1.388	200	0	7.1	4.6	6	1.545
1.368	600	0	7.1	4.4	6	1.615
1.368	200	0	7.1	4.3	10	1.898
1.364	200	0	7.1	4.2	16	2.025

[a] 0: initial value; f: final value.
[b] Buffered at pH of 7.
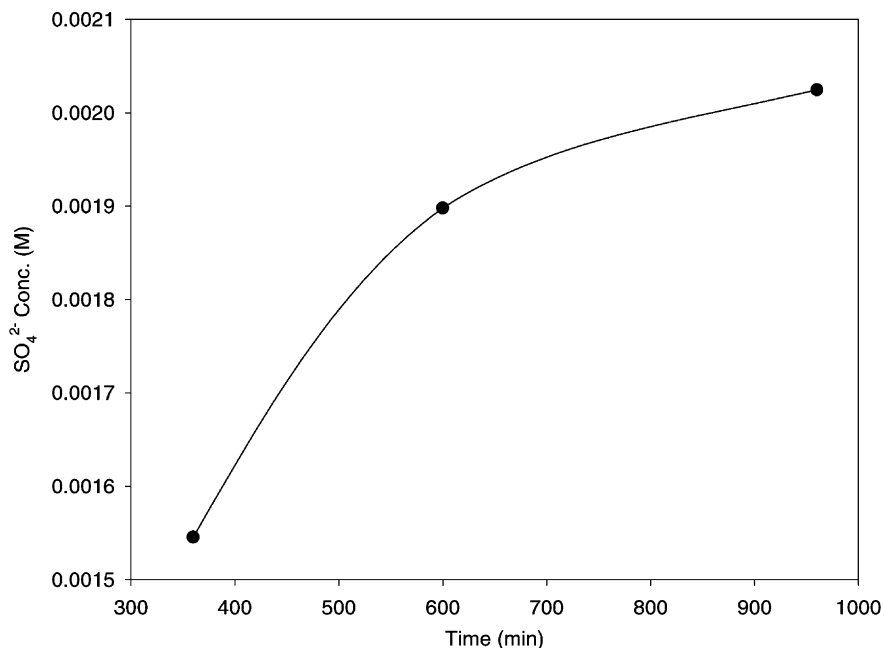
Fig. 7. SO_4^{2-} formation as a function of time in the presence of H_2O_2 (initial concentration, $[CS_2]_0 = 1.36 - 1.39 \times 10^{-3}$ M, $[H_2O_2]_0 = 200$ mg, T = 20 °C, P = 50 W).

of 1-butanol, a known scavenger of $^\bullet$OH radicals [11]. As shown in Fig. 7, SO_4^{2-} formation in the presence of 200 mg H_2O_2 (1.176×10^{-1} M) increases exponentially with irradiation time. After 16 h of irradiation, over 2.0×10^{-2} M SO_4^{2-} is produced, approaching twice the initial CS_2 concentration and indicating almost complete conversion of CS_2 to $SO_4{}^{2-}$. Kotronarou and co-workers [12,13] studied the rate of disappearance of H_2S or S(−II) solutions (i.e. $[S(-II)] = [H_2S] + [HS^-] + [S^{2-}]$ by ultrasonic irradiation spectrophometrically at pH = 10. They also found the disappearance of the reduced sulfur species to be rapid with a zero-order rate of 7.5 μM/min using $[S(-II)]_0 = 200$ μM and the formation of SO_4^{2-}, SO_3^{2-} as the main oxidation products and $S_2O_3^{2-}$ as minor product.

As shown in Table 3 and Fig. 8, the more the H_2O_2 added, the greater the oxidation efficiency, and hence, the greater the SO_4^{2-} formation. With the addition of 200 mg H_2O_2, the SO_4^{2-} formation observed after 6 h of ultrasonic irradiation is about 31% more than the amount observed with the control, i.e. without H_2O_2 addition. Also, addition of 200 mg of 1-butanol reduced the SO_4^{2-} formation by as much as 59% compared to the control. We attribute these results to an increase in $^\bullet$OH radicals generated on the addition of H_2O_2. However, the increase in oxidation rates is not significant between the additions of 200 mg H_2O_2 and 600 mg H_2O_2 (3.528×10^{-1} M), suggesting the concentration of $^\bullet$OH radical at the 200 mg level was adequate for the oxidation reaction. These observations coupled with the apparent zero-order dependence on CS_2 suggest that the rate-determining step in
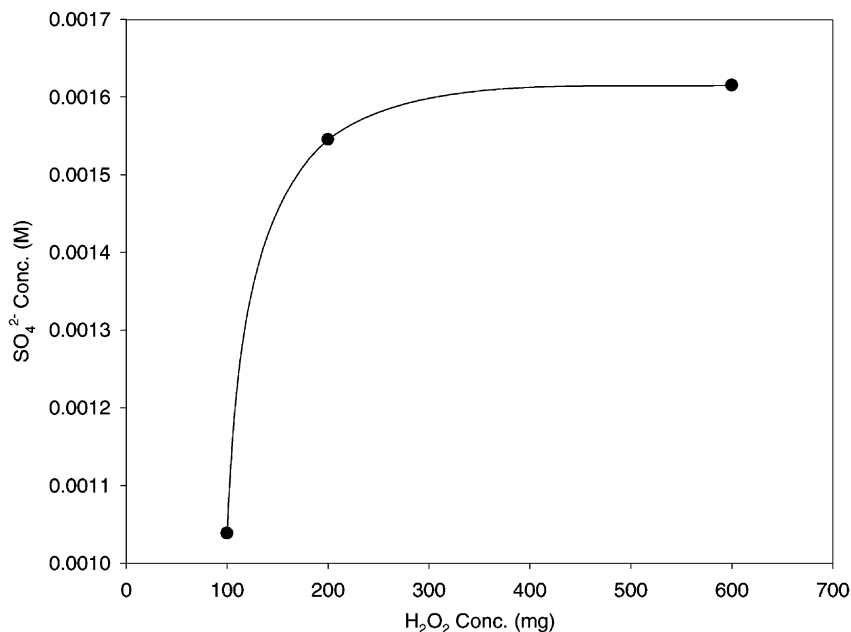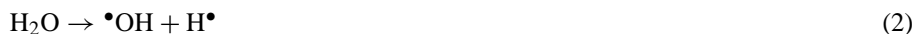
Fig. 8. $SO_4{}^{2-}$ formation as a function of initial H_2O_2 concentration after 6 h of ultrasonic irradiation (initial concentration, $[CS_2]_0 = 1.37 - 1.39 \times 10^{-3}$ M, T = 20 °C, P = 50 W).

the overall reaction is dependent on the availability of $^\bullet$OH radicals for oxidation and other reactions in the interface of the bubble or in the liquid phase. Lin et al. [14,15] found that the combination of ultrasound with H_2O_2 increased the efficiency of 2-CPOH decomposition significantly. They also studied the effect of H_2O_2 concentration on decomposition. It was shown that the more the H_2O_2 that was added, the greater the degradation efficiency. In a particular study with 500 mg/l of H_2O_2 and initial 2-CPOH concentration of 100 mg/l, they observed after a reaction time of 30 min., an improvement as great as 57% over the control, i.e. without H_2O_2 addition. The result was attributed to increase in $^\bullet$OH radicals generated on addition of H_2O_2. However, the increase in degradation rates was not significant between the additions of 200 and 500 mg/l H_2O_2. They explained these results by suggesting that the solutions were saturated with $^\bullet$OH radicals at the 200 mg/l H_2O_2 concentration level.

3.3. Sonochemical oxidation mechanisms
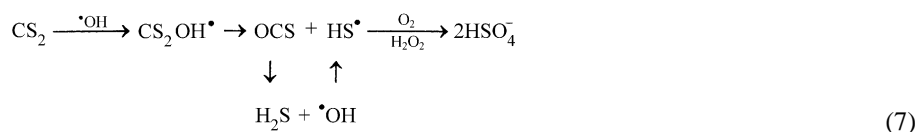
Ultrasound-induced bubble of cavitation can be considered a microreactor ejecting free radicals. Production of hydroxyl ($^\bullet$OH), hydroperoxyl ($HO_2{}^\bullet$) and hydrogen ($H^\bullet$) radicals, and H_2O_2 in water subjected to ultrasonic cavitation shown in Eqs. (2)–(6) is the subject of numerous publications [5–16]. The formation of $H^\bullet$ and $^\bullet$OH (Eq. (2)) is attributed to the thermal dissociation of water vapor present in the cavities during the compression phase. In the absence of scavengers, H_2O_2 is the main product of water sonolysis. In the presence

of air (i.e. O_2), the hydrogen radicals rapidly combine with O_2 to form $HO_2{}^\bullet$ as in Eq. (4).

$$H_2O \rightarrow {}^\bullet OH + H^\bullet \tag{2}$$

$${}^\bullet OH + {}^\bullet OH \rightarrow H_2O_2 \tag{3}$$

$$H^\bullet + O_2 \rightarrow HO_2{}^\bullet \tag{4}$$

$$HO_2{}^\bullet + H^\bullet \rightarrow H_2O_2 \tag{5}$$

$$HO_2{}^\bullet + HO_2{}^\bullet \rightarrow H_2O_2 + O_2 \tag{6}$$

The hydroxyl and hydroperoxyl radicals, and H_2O_2 are all oxidizing agents. However, the ${}^\bullet OH$ radical is a more powerful and efficient chemical oxidant both in the gas and liquid phase and its reactions with inorganic and organic substrates are often near the diffusion-controlled rate [17]. In aerated solutions, the hydroperoxyl radicals ($HO_2{}^\bullet$) formed by Eq. (4) will decay with generation of H_2O_2. The spontaneous disproprtionation of the hydroperoxyl radicals reduces its effectiveness to attack the pollutant [18,19]. However, the production of the $HO_2{}^\bullet$ radical increases the oxidation process due to further formation of H_2O_2 by its recombination reaction (Eq. (6)).

The sonochemical oxidation of CS_2 to $SO_4{}^{2-}$ in the presence of air (i.e. oxygenated aqueous solutions) is proposed to proceed via the initial reaction of ${}^\bullet OH$ in the interface of the bubble with CS_2 to produce the intermediate $CS_2OH^\bullet$ [20]. The rate constant at $23\,^\circ C$ for this reaction is reported to be $(8.0 \pm 2.0) \times 10^9\,M/s$ [21]. Subsequent intermediate steps leading to the formation of the H_2S radical ($HS^\bullet$) and its oxidation to $SO_4{}^{2-}$ may include a variety of radical reactions and H_2O_2, and are discussed elsewhere [12,13,20]. The oxidation of CS_2 to $SO_4{}^{2-}$ is proposed to proceed mainly through oxidation by ${}^\bullet OH$ radical and H_2O_2 produced from its recombination reaction. An overall reaction sequence consistent with the experimental data is

$$CS_2 \xrightarrow{\ {}^\bullet OH\ } CS_2OH^\bullet \rightarrow OCS + HS^\bullet \xrightarrow[H_2O_2]{O_2} 2HSO_4^- \\ \hspace{4.5cm} \downarrow \quad\ \uparrow \\ \hspace{4.5cm} H_2S + {}^\bullet OH \tag{7}$$

The details of the sonochemical oxidation pathways of CS_2 and the effects of temperature and pH on reaction kinetics and $SO_4{}^{2-}$ formation rates are presented elsewhere [20].

4. Conclusions

The potential applications of sonochemical techniques to water and wastewater treatment have not been fully explored and the applications to the detoxification of process wastewater containing reduced sulfur compounds are especially limited. The results of our study indicate the feasibility of effectively removing CS_2 from aqueous waste streams by ultrasonic irradiations under proper conditions. The reaction rate was found to be zero-order at $20\,^\circ C$. With the initial concentration of $[CS_2]_0 = 6.9 \times 10^{-4}\,M$, the rate constant for the degradation at $20\,^\circ C$, 14W and in air was $2.27 \times 10^{-5}\,min^{-1}$. At the same initial concentrations and

temperature, and in the presence of air, the degradation rate of CS_2 at 50 W (39.47 W/m^2) is more than two times that at 14 W (11.04 W/m^2). The rate of sonochemical degradation of CS_2 in the presence of the different gases was in the order of He > air ≥ N_2O > Ar; the rate with helium was found to be about three times that of argon. The formation of SO_4^{2-} as reaction product with air as the irradiating gas was enhanced in the H_2O_2 and drastically inhibited in the presence of 1-butanol. The results suggest CS_2 is oxidized to non-toxic SO_4^{2-} (with elimination of pungent smell) by $^\bullet$OH radical reactions at the interface of the bubble or in the liquid phase as the radical diffused out of the cavitation bubble.

Acknowledgements

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