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Effects of Na_2SO_4 or NaCl on sonochemical degradation of phenolic compounds in an aqueous solution under Ar: Positive and negative effects induced by the presence of salts

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ABSTRACT

Sonochemical degradation of 4-chlorophenol, phenol, catechol and resorcinol was studied under Ar at 200 kHz in the absence and presence of Na_2SO_4 or NaCl. The rates of sonochemical degradation in the absence of salts decreased in the order 4-chlorophenol > phenol > catechol > resorcinol and this order was in good agreement with the order of log *P* (partition coefficient) value of each phenolic compound. The effects of salts on the rates of sonochemical degradation consisted of no effect or slight negative or positive effects. We discussed these unclear results based on two viewpoints: one was based on the changes in pseudo hydrophobicity and/or diffusion behavior of phenolic compounds and the other was based on the changes in solubility of Ar gas. The measured log *P* value of each phenolic compound slightly increased with increasing salt concentration. In addition, the dynamic surface tension for 4-chlorophenol aqueous solution in the absence and presence of Na_2SO_4 or NaCl suggested that phenolic compounds more easily accumulated at the interface region of bubbles at higher salt concentration. These results indicated that the rates of sonochemical degradation should be enhanced by the addition of salts. On the other hand, the calculated Ar gas solubility was confirmed to decrease with increasing salt concentration. The yield of H_2O_2 formed in the presence of Na_2SO_4 or NaCl decreased with increasing salt concentration. These results suggested that sonochemical efficiency decreased with decreasing gas amount in aqueous solution: a negative effect of salts was observed. Because negative and positive effects were induced simultaneously, we concluded that the effects of salts on the rates of sonochemical degradation of phenolic compounds became unclear. The products formed from sonochemical degradation of 4-chlorophenol were also characterized by HPLC analysis. The formation of phenol and 4-chloro-1,3-dihydroxy benzene was confirmed and these concentrations were affected by the presence of salts.

1. Introduction

Phenol and substituted phenols are important organic intermediates for the products of industry and agriculture. However, they are high toxic and create serious problems to human health and aquatic species. The conventional methods to deal with such phenolic compounds include activated carbon adsorption, biological treatment, and chemical oxidation [1–5], however, the application of these methods has its limitations and disadvantages. Actually, high toxicity and stability of phenolic compounds hamper the treatments of wastewater.

Advanced oxidation processes (AOPs) have been developed to degrade organic pollutants in water. AOPs include ozonation, UV/photocatalysis, UV/H_2O_2, Fenton reaction, radiolysis, sonolysis and their combinations [6–19]. When a solution containing organic pollutants is irradiated by ultrasound, organic pollutants are degraded by OH radicals and/or direct pyrolysis reactions during acoustic cavitation. Although unique reaction conditions consisting of extremely high temperatures and pressures, rapid cooling, shock waves, micro-jets, etc. are generated in an aqueous solution [20,21], the rate of sonochemical degradation of organic pollutants is still slow for practical use.

Therefore, a new technique has to be developed to enhance the rate of sonochemical degradation. So far, effects of various types of salts on sonochemical degradation of organic compounds have

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been investigated, where NaCl or Na_2SO_4 are often used. However, it seems that the experimental results observed often conflict each other. For example, it was reported that the rates of sonochemical degradation of chlorobenzene, p-ethylphenol and phenol were enhanced by the addition of NaCl under 20 kHz ultrasound irradiation [14,16,17]: a positive effect of NaCl was observed. On the other hand, Chen and Smirniotis reported that the rates of sonochemical degradation of phenol decreased by the addition of NaCl or Na_2SO_4 under 20 kHz ultrasound irradiation [13]. The effects of NaCl or Na_2SO_4 on the rates of sonochemical degradation of Rhodamin B [22,23], malachite green [24] and crystal violet [25] have been investigated, but all cases showed no effect of NaCl or Na_2SO_4.

In this study, we investigated the effects of Na_2SO_4 or NaCl on sonochemical degradation of several phenolic compounds by using a standing wave type 200 kHz ultrasound irradiation system under Ar atmosphere. We chose phenolic compounds as probe compounds because they are the most common and recalcitrant pollutants present in industrial wastewaters.

2. Experimental section

2.1. Experimental procedure

2.1.1. Chemicals

1-Octanol (purity > 99.5%) was purchased from Tokyo Chemical Industries. Catechol (purity 99.0%), resorcinol (purity 99.0%), phenol (purity 99.0%), 4-chlorophenol (purity 98%), Na_2SO_4 (purity 99%), NaCl (purity 99.5%), and methanol (purity 99.7%) were purchased from Wako Pure Chemical Industries. All the chemicals were reagent grade and used as received. Ar (purity 99.999%) was purchased from Osaka Sanso. All solutions were prepared with Milli-Q purified water (resistivity of 18.2 MΩ cm at 25 °C).

2.1.2. Measurement of log *P*

Hydrophobicity is commonly expressed by log *P*, where *P* is the octanol–water partition coefficient. *P* is determined by $C_{\text{octanol}}/C_{\text{water}}$, where C_{octanol} and C_{water} are the concentrations of a target organic solute in octanol and water, respectively. The stir-flask method [26] improved was used to measure *P*: 1-octanol (5 ml), aqueous solution with or without salt (4 ml), and 0.45 mM phenolic solution (1 ml) were added to a flat-bottomed flask. The contents were sealed and stirred at 500 rpm for 24 hours at room temperature (22 ± 1 °C). The contents were settled on the desk for 2–3 h at room temperature. 50 μl of aqueous layer was injected in a high performance liquid chromatograph (HPLC, Shimadzu LC-20ATvp with a VP-ODS C-18 reversed phase column (4.6 × 250 mm)) for analysis. A mobile phase of methanol and water (volume ratio = 30:70) was used for resorcinol, catechol and phenol, and methanol and water (volume ratio = 80:20) was used for 4-chlorophenol at flow rate of 0.70 ml min^{-1} where the detection wavelength of 254 nm was used for resorcinol and catechol, and of 280 nm for phenol and 4-chlorophenol.

2.1.3. Measurement of surface tension

When the concentration of 4-chlorophenol was less than 1.0 mM, the change in surface tension was too small to monitor the change in surface tension. Therefore, as a sample solution for surface tension measurements, 20 mM 4-chlorophenol aqueous solution was chosen. The changes in the surface tension of the sample solution in the absence and presence of 0.50 M Na_2SO_4 or 0.45 M NaCl were measured in the time range from 0 to 1800 s by using a Young–Laplace method for a pendant drop with an interfacial tension meter (Kyowa Interface Science DM-501), where the surface tension was monitored at 1 s interval. Three or five experimental runs were performed and the average data were used in figure, where the standard deviation was 0.4 (no salt), 0.4 (0.50 M Na_2SO_4) and 1.3 (0.45 M NaCl) at 1000 s, respectively.

2.1.4. Sonolysis

An ultrasonic generator (Kaijo 4021, Lot No. 1033) and an oscillator (Kaijo 4611, MFG. No. 34C3) of 65 mm diameter were used for ultrasonic irradiation and were operated at 200 kHz with a nominal power of 200 W (calorimetric power for 60 ml sample solution: 16 W) at 20 °C. A glass vessel with 60 ml sample solution was used for ultrasonic irradiation under Ar atmosphere, where the vessel was mounted at a constant position (4 mm from the oscillator). The vessel had flat bottom with 1 mm thick and a side arm with a silicon rubber septum for Ar gas bubbling and sample extracting by glass syringe (1 ml) without exposing the sample to air. The schematic diagram of the experimental setup was described in the literature [27]. The sonication was performed up to 30 min to understand the initial degradation behavior of phenolic compounds. When the degradation products were analyzed, the sonication was performed up to 60 min. The sonicated solutions were analyzed by a HPLC. The analysis conditions for resorcinol, catechol, phenol and 4-chlorophenol were the same as the measurement of log *P*. For determination of products formed from sonochemical degradation of 4-chlorophenol, a mobile phase of methanol and water (volume ratio = 40:60) was used at flow rate of 0.70 ml min^{-1}, where the detection wavelength of 224 nm was used. The changes in UV–vis spectra of the sample solutions were measured by a UV–vis spectrophotometer (Shimadzu UV-2450). The concentration of H_2O_2 formed in the sonolysis of aqueous solution with and without salts under Ar was measured by a KI method [28], where the absorbance at 352 nm was measured.

3. Results and discussion

Sonochemical degradation of 4-chlorophenol, phenol, catechol and resorcinol was investigated in the absence and presence of Na_2SO_4 or NaCl under Ar. The rate of degradation of phenolic compounds obeyed a pseudo first order rate constant as seen in Figs. S1–S3 in Supplementary data. Therefore, we analyzed the pseudo rate constant as a rate of degradation in this study. The result obtained in the absence and presence of Na_2SO_4 is shown in Fig. 1(a). The rate constant for sonochemical degradation of 4-chlorophenol in the absence of salt was determined by three times sonication experiments as shown in Fig. S1. The average value was plotted in Fig. 1(a), where the error bar corresponds to the standard deviation. Figs. 1(a) and S2 indicate that no effect or a slight positive effect of Na_2SO_4 were observed. We also investigated the effects of NaCl on the rate constant for sonochemical degradation. The result is shown in Figs. 1(b) and S3. Even in the case of NaCl addition, no effect for chlorophenol ($k = 0.0214\ min^{-1}$ at no salt, 0.0209 min^{-1} at 0.023 M NaCl, 0.0217 min^{-1} at 0.45 M NaCl), a slight negative effect for phenol ($k = 0.0180\ min^{-1}$ at no salt, 0.0168 min^{-1} at 0.023 M NaCl, 0.0160 min^{-1} at 0.045 M NaCl), or slight positive effects for catechol ($k = 0.0125\ min^{-1}$ at no salt, 0.0143 min^{-1} at 0.023 M NaCl, 0.0142 min^{-1} at 0.045 M NaCl) and for resorcinol ($k = 0.00977\ min^{-1}$ at no salt, 0.0128 min^{-1} at 0.023 M NaCl, 0.0134 min^{-1} at 0.045 M NaCl) were observed.

In the absence of salt, the results showed that the rate constants for sonochemical degradation decreased in the order 4-chlorophenol > phenol > catechol > resorcinol. It has been reported that the hydrophobicity of an organic solute is one of the most important parameters for the rate of sonochemical degradation, because highly hydrophobic compounds tend to accumulate

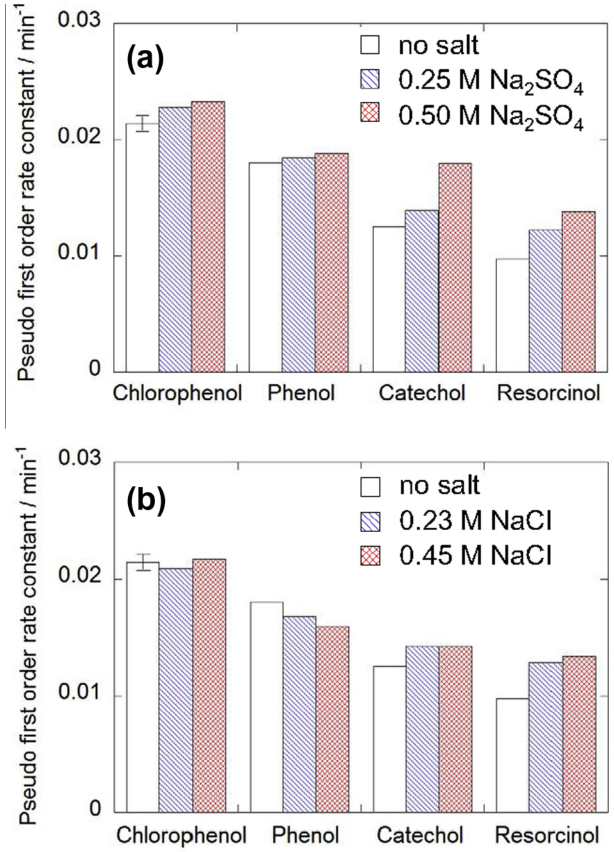

Fig. 1. Pseudo first order rate constants for sonochemical degradation of 4-chloropenol, phenol, catechol and resorcinol in the absence and presence of (a) Na_2SO_4 and (b) NaCl. Initial concentration of phenolic compound: 0.45 mM.

at the interface region of cavitation bubbles in which the concentration of OH radicals is higher than that in the bulk solution [29]. Nanzai et al. reported that the initial rates of degradation of aromatic compounds increased with increasing log *P* value, which was used as an indicator of hydrophobicity of an aromatic compound [30]. To confirm the relationship between the rate constant for sonochemical degradation and log *P* value, we measured *P* values of phenolic compounds experimentally. The results showed that log *P* values were 2.25 for 4-chlorophenol, 1.47 for phenol, 0.97 for catechol, and 0.87 for resorcinol, respectively. The values measured were in good agreement with the reported values [31]. The order of the rate constant for sonochemical degradation was in good agreement with the order of log *P*. In addition, the hydrophobicity of phenolic compound was reported in the order of 4-chlorophenol > phenol > catechol = resorcinol which was determined by the change in surface tension as a function of phenolic compound concentration in the range from 0 to 10 mM at 20 °C [32]. They also suggested that the rates of sonochemical degradation of these compounds should be this order. Our results were in good agreement with this order.

Next, we discuss the effects of salts on the rate of degradation. As described above, the addition of Na_2SO_4 or NaCl did not affect the rate of degradation clearly. Several researchers reported that the presence of NaCl resulted in the enhanced sonochemical efficiency [14,16,17,33]. However, our result was different from their results. Before experiments, we had expected that the addition of salts will enhance the rate of degradation by moving phenolic compounds from bulk solution to the interface region of bubbles (here becomes high temperature and high OH radical concentration after collapsing bubbles). To guess whether the movement of phenolic compounds is enhanced or not by the presence of salts during acoustic cavitation, we measured the change in log *P* value of 4-chlorophenol, phenol, catechol and resorcinol at different Na_2SO_4 concentrations. The result is shown in Fig. 2. It was confirmed that the pseudo log *P* values measured slightly increased with increasing Na_2SO_4 concentrations, suggesting that all phenolic compounds behave as more hydrophobic compounds in a higher Na_2SO_4 concentration solution. Since more hydrophobic compounds are degraded more quickly [30], the addition of salts should induce a salting out effect of phenolic compounds from bulk solution to the interface region of bubbles which should enhance the rate of degradation. However, Fig. 2 shows that the log *P* value does not increase largely by the addition of Na_2SO_4. We considered that the log *P* value was the information of equilibrium so that the real diffusion behavior of phenolic compounds was unclear.

To confirm the effect of salt on the diffusion behavior of phenolic compounds, we measured the dynamic surface tension of 4-chlorophenol aqueous solutions in the absence and presence of 0.50 M Na_2SO_4 or 0.45 M NaCl, where dynamic changes in the surface tension of a pendant drop were measured as a function of time. The results are shown in Fig. 3. The surface tension gradually decreased with time. This meant that 4-chlorophenol molecules accumulated at the gas-liquid interface with time. Fig. 3 shows three results: (1) at the initial stage of equilibrium, the surface tension in the presence of salts is smaller than that in the absence of salt, (2) the slope in the change in the surface tension in the presence of salts is steeper than that in the absence of salt, and (3) the surface tension reached after equilibrium in the presence of salts is smaller than that in the absence of salt. All results indicated that 4-chlorophenol molecules accumulate at the gas-liquid interface faster in the presence of salt. This quick accumulation behavior by the presence of salt should act as a positive effect for degradation of phenolic compounds.

However, as seen in Fig. 1, some results do not show such a positive effect. To discuss this contradiction phenomenon, we considered a further aspect regarding the effects of salts. For example, in the absence of organic solutes, when the concentration of salts is increased, various changes in parameters such as changes in the surface tension of solution, vapor pressure of water, viscosity of solution, and gas solubility in a solution occur simultaneously (Table 1).

Based on the literature [34], the surface tension of aqueous solutions increases linearly with increasing salt concentration.

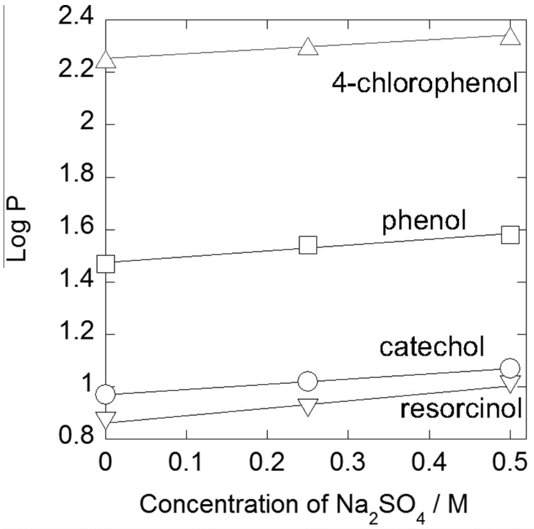

Fig. 2. Relationship between concentrations of Na_2SO_4 and log *P* of 4-chloropenol, phenol, resorcinol, and catechol.

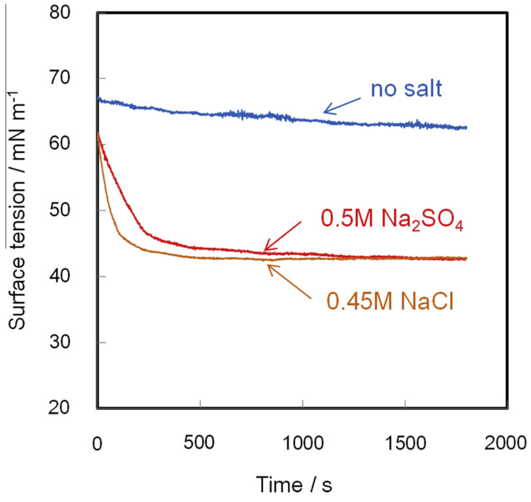

Fig. 3. Changes in surface tension of 4-chlorophenol aqueous solutions in the absence and presence of 0.50 M Na_2SO_4 or 0.45 M NaCl as a function of time. Concentration of 4-chlorophenol: 20 mM.

However, the increment of the surface tension at 20 °C is as small as from 72.5 mN m^{-1} (pure water) to 73.9 mN m^{-1} (Na_2SO_4: 0.51 M) and to 73.4 mN m^{-1} (NaCl: 0.50 M) [34,35]. As is well known, the vapor pressure of water decreases with increasing salt concentration. However, based on the literature [35,36], the decrement of the vapor pressure of water at 20 °C is as small as from 17.5 mmHg (pure water) to 17.2 mmHg (Na_2SO_4: 0.50 M). In the case of NaCl at 18 °C, 15.5 mmHg (pure water) decreases to 15.2 mmHg (NaCl: 0.62 M). In addition, based on the literature [35,37], the viscosity of solutions at 20 °C changes from 1.00 mPa s (pure water) to 1.29 mPa s (Na_2SO_4: 0.70 M) and 1.07 mPa s (NaCl: 0.84 M). The change in the viscosity may look like a relatively larger value than those in the surface tension and vapor pressure, but the change in the viscosity could be considered as not so large change, because the viscosity of 0.70 M Na_2SO_4 solution at 20 °C (1.29 mPa s) is almost the same as that of pure water at 10 °C (1.31 mPa s). Therefore, we assumed that the effects of changes in the surface tension, vapor pressure of water and viscosity of solution are negligibly small for sonochemical degradation.

It has also been suggested that the amount of dissolved gas in water affects sonochemical efficiency [38], because the number of acoustic cavitation bubbles is affected by the amount of dissolved gas. To discuss the amount of dissolved Ar gas in water, we calculated the saturation solubility of Ar gas in the presence of Na_2SO_4 or NaCl. The calculation method is shown in Supplementary data. Table 1 shows the solubility of Ar gas at 20 °C decreasing with increasing salt concentrations: 2.75×10^{-5} (no salt) > 1.87×10^{-5} (0.25 M Na_2SO_4) > 1.28×10^{-5} (0.50 M Na_2SO_4), and 2.75×10^{-5} (no salt) > 2.55×10^{-5} (0.23 M NaCl) > 2.37×10^{-5} (0.45 M NaCl). This tendency is in good agreement with the literature [39].

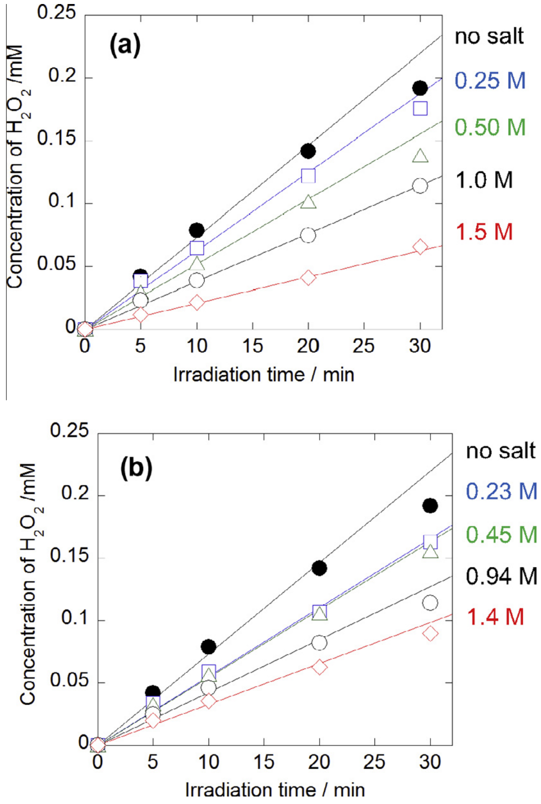

Fig. 4. Sonochemical formation of H_2O_2 in the absence and presence of (a) Na_2SO_4 and (b) NaCl.

Okitsu et al. reported that the yield of H_2O_2 formed in the sonolysis of pure water increases linearly with increasing amount of dissolved gases of He, Ne, Ar, Kr and Xe [38], where the yield of H_2O_2 corresponds to sonochemical efficiency. Based on the solubility of Ar gas in Table 1, sonochemical efficiency should decrease in a higher salt concentration. To confirm whether sonochemical efficiency changes or not by the addition of salts, the H_2O_2 yield was measured in the absence and presence of salts without phenolic compounds. Fig. 4(a) and (b) indicate the H_2O_2 yield as a function of irradiation time. The change in concentration of H_2O_2 could not be fitted linear lines between 0 and 30 min. This is because the change in the irradiation condition occurs with irradiation time. Taking into account the change in the irradiation condition, linear lines were drawn between 0 and 20 min with the least squares method through zero point. It was confirmed that the H_2O_2 yield decreased with increasing concentration of salts, where the

Table 1
Surface tension of solution, vapor pressure of water, viscosity of solution and saturation solubility of Ar in pure water, Na_2SO_4 aqueous solution and NaCl aqueous solution.

	Surface tension of solution [34]/mN m^{-1} at 20 °C	Vapor pressure of water/mmHg [36] at 20 °C[a] or 18 °C[b]	Viscosity of solution [37]/mPa s at 20 °C	Ar solubility in solution at 20 °C
Pure water	72.5	17.5[a], 15.5[b]	1.00	2.75×10^{-5} [38]
Na_2SO_4 aqueous solution (conc. of Na_2SO_4)	73.9 (0.51 M[c])	17.2[a] (0.50 M[c])	1.29 (0.70 M[c])	1.87×10^{-5} (0.25 M) 1.28×10^{-5} (0.50 M)
NaCl aqueous solution (conc. of NaCl)	73.4 (0.50 M[c])	15.2[b] (0.62 M[c])	1.07 (0.84 M[c])	2.55×10^{-5} (0.23 M) 2.37×10^{-5} (0.45 M)

[a] The data at 20 °C.
[b] The data at 18 °C.
[c] The concentration of salt was converted from mol kg^{-1} to mol L^{-1} by using the specific gravity of solutions in [35].

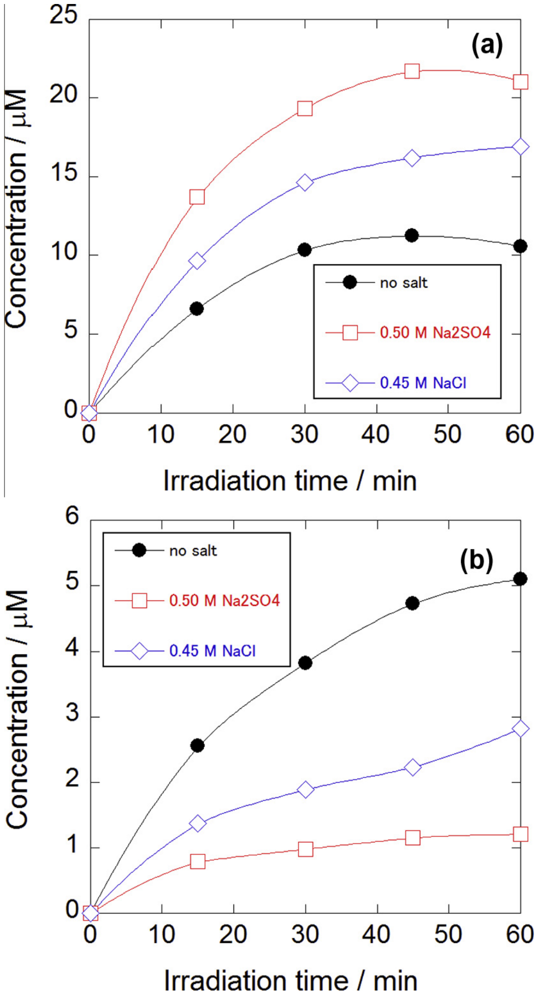

Fig. 5. Changes in concentration of (a) phenol and (b) 4-chloro-1,3-dihydroxy benzene as a function of irradiation time. Initial concentration of 4-chlorophenol: 0.45 mM.

additional experiments at higher concentrations (Na_2SO_4; 1.0 M and 1.5 M, NaCl; 0.94 M and 1.4 M) were carried out to confirm the effect of salts more clearly. Wakeford et al. reported that the H_2O_2 yield decreased with increasing concentration of NaCl under Ar at 35 kHz ultrasound irradiation [40]. They suggested that the lower yield of H_2O_2 in higher NaCl concentration may be due to Cl^- acting as an OH radical scavenger. They do not consider the occurrence of a gas solubility change when the salt concentration increases. Based on the gas solubility at different salt concentration (Table 1), the number of the formed cavitation bubbles with high temperatures would decrease by the presence of Na_2SO_4 or NaCl in water, resulting in the lower H_2O_2 yield in the higher salt concentration. It was also confirmed that Na_2SO_4 was more effective for the decrement in the H_2O_2 yield than NaCl. This phenomenon would be explained by the difference of Ar gas solubility between Na_2SO_4 and NaCl solutions.

In spite of the lower H_2O_2 yield in the presence of Na_2SO_4 or NaCl, some results in Fig. 1 show that the rate constants for degradation do not change or slight increase when the concentration of Na_2SO_4 or NaCl increases. To explain these results, we proposed for the first time that the effects of salts on sonochemical degradation of phenolic compounds consisted of the mixture of a positive and a negative effect so that the effects of salts became unclear.

To discuss the effect of salts on sonochemical degradation of phenolic compounds, we investigated the degradation products. If phenolic compounds were pushed to the interfacial region from bulk solution by the presence of salts, the degradation mechanism of phenolic compounds would be different between in the absence and presence of salts: the contribution ratio of pyrolysis reactions to radical reactions may be changed. Fig. S4 shows the changes in absorption spectra of 4-chlorophenol solutions during irradiation, where the sonication was performed up to 60 min. From Fig. S4, it was confirmed that the degradation of 4-chlorophenol occurred to form any products which had absorption spectra in the UV region. From HPLC analysis, the formation of various types of products with higher hydrophilic compounds than 4-chlorophenol was confirmed. The peaks at the retention time of 15.6 and 14.2 min were determined as phenol and 4-chloro-1,3-dihydroxy benzene. Fig. S5 shows the changes in concentration of 4-chlorophenol as a function of irradiation time. Fig. 5 shows the changes in concentration of phenol and 4-chloro-1,3-dihydroxy benzene formed. By the addition of salts, the concentration of phenol formed increased and that of 4-chloro-1,3-dihydroxy benzene decreased, although the rates of degradation of 4-chlorophenol were not affected significantly. The reason why this phenomenon was observed was unclear at present, but this result suggested that the addition of salts should affect the degradation mechanism. Recently, Brotchie et al. [39] reported that the size of bubbles decreases by the presence of salts. Various changes in acoustic cavitation phenomenon may be induced by the presence of salts, therefore, further studies must be required to discuss in details.

4. Conclusions

The rates of sonochemical degradation of phenolic compounds in the presence of salts were affected by the changes in (1) the pseudo hydrophobicity and (2) diffusion behavior of phenolic compounds and (3) the solubility of Ar gas. The rates of sonochemical degradation of phenolic compounds in the absence and presence of Na_2SO_4 or NaCl were in the order 4-chlorophenol > phenol > catechol > resorcinol. This order was explained by the order of log *P*. The effects of Na_2SO_4 or NaCl on the rates of sonochemical degradation were investigated. We suggested that a positive effect due to increasing hydrophobicity induced by the addition of salts and increasing diffusion behavior of phenolic compounds would be offset by a negative effect due to decreasing sonochemical efficiency which was induced by the decrease of gas solubility at high concentration of Na_2SO_4 or NaCl. As described in introduction section, many studies for the effects of Na_2SO_4 or NaCl on the sonochemical degradation have been reported, where the obtained results have often conflicted each other. Our results suggested that the balance between negative and positive effects may sensitively depend on the sonication condition so that the effects of salts reported so far showed unclear effects.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ultsonch.2015.06.028.

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