



The photodegradation of trichloroethylene with or without the NAPL by UV irradiation in surfactant solutions

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ABSTRACT

The photodegradation of trichloroethene (TCE) with or without nonaqueous phase liquids (NAPL) by ultraviolet irradiation in surfactant solutions was examined in this study. The photodecay of TCE was studied at monochromatic 254 nm UV lamps. The effects of the type of surfactants, initial surfactant concentrations, pH levels and NAPL concentrations were examined to explore the photodecay of TCE. All the photodegradation of TCE followed pseudo-first-order decay kinetics at various conditions. It was found that Brij 35 overdose and higher initial pH levels may retard or inhibit the photodecay of TCE, mainly due to protons, intermediate generation and change of surfactant structure in the processes. The optimal condition for TCE photodecay was suggested based on the analysis of kinetics data, from which the reaction mechanism of TCE in the presence of NAPL form was also studied. In general, the reactions of TCE in micellar solution and NAPL pool can be considered as independent pathways due to the low molecule diffusion between the two phases.

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1. Introduction

Chlorinated organic compounds, such as trichloroethylene (TCE), are the most prevalent contaminants found in soil and groundwater and pose serious health risks even at trace levels. TCE is also a volatile solvent, it has been used extensively in dry cleaning, in the metal and glass industries as a solvent and degreasing agent, and in household products such as spot removers, rug cleaners, and air fresheners [1,2]. The US Environmental Protection Agency has classified TCE as a priority pollutant on the basis of its widespread contamination, its possible carcinogenicity, and its anaerobic bio-conversion to a more potent carcinogen, vinyl chloride (VC). TCE, a problematic groundwater contaminant and a suspected human carcinogen, is considered to be long-lasting in the environment [3].

TCE becomes a health hazard after being in the soil for a period of time. It is chemically converted to vinyl chloride by reductive dehalogenation, where the product vinyl chloride is a potential carcinogen. EPA drinking water standards proclaim that the maximum contaminant level (MCL) for TCE is 0.005 mg L^{-1} , and the maximum contaminant level goal (MCLG) is zero. Major environmental releases of TCE are due to air emissions from metal degreasing plants. Wastewater from metal finishing, paint and ink produc-

tion, electrical components, and rubber-processing industries also contains TCE.

Various types of organic pollutants including nonaqueous phase liquids (NAPLs) are known to accumulate in the groundwater of Hong Kong and many places throughout the world. The remediation of the polluted groundwater will be an environmental concern because underground hydrophobic pollutants will impair public health by bioaccumulating in the food-chain through the media of crops. The presence of NAPLs in the subsurface represents a threat to groundwater supplies and has been the focus of considerable attention over the last 20 years [4]. NAPLs mostly consist of chlorinated and aromatic solvents, which are released in the environment through spillage or leakage from pipelines, storage tanks, or industrial facilities. TCE is present in dense nonaqueous phase liquids (DNAPLs), especially in the form of pools in the subsurface.

In general, DNAPLs have a low aqueous solubility. A major barrier to the successful implementation of soil treatment technologies is the hydrophobic nature of hazardous compounds, which limits the concentration level in the aqueous phase. Low aqueous-phase contaminant levels lead to low conversion rates, resulting in longer treatment times and therefore, higher cost. In the case of TCE, aqueous solubility of 1000 mg L^{-1} and log octanol–water partition coefficient (K_{OW}) of 2.42 are not as hydrophobic as corresponding values for PAHs and PCBs, but can still be rate controlling in soil treatment systems. Typically, higher K_{OW} values are an indication that the substance has a lower affinity for the aqueous phase and not due to a higher solubility in the organic phase.

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Because TCE is slightly soluble in water, a low concentration of TCE is generally observed and it has become a long-term source of aquifer contamination. Since TCE has been widely used and abundant in the soil; much research focus has been devoted to remediating TCE-contaminated soil. Many remedial technologies of groundwater and soil contaminated by DNAPLs and/or TCE have been developed [5–7]. Treatment alternatives including bioremediation, thermal treatment, dehalogenation, chemical oxidation, advanced oxidation processes and electrokinetic remediation efforts are being used to develop and demonstrate *in situ* treatment methods that are rapid and extensive, as well as less sensitive to contaminant character and concentration. Pump-and-treat technologies can prevent the migration of the contaminant, but have been ineffective in restoring sites to precontamination levels, partly because of the slow dissolution and desorption rates of NAPL. To remediate NAPL contaminated sites, it is necessary to achieve the complete mass removal or destruction of the pooled liquid. Several remediation technologies are currently being studied to mitigate this problem through enhanced solubilization in the aqueous phase and/or the physical mobilization of NAPL.

Photoreduction of chlorinated organic contaminants by using ultraviolet in surfactant solution is an emerging process that has become increasingly important recently [8]. For example, the use of surfactant in soil-washing operations followed by UV-destruction is a promising soil remediation technology and has good potential for extracting and degrading the chlorinated soil-contaminants, such as trichloroethene, in one step. Surfactants can increase the solubility of organic compounds and decrease the interfacial tension, which allows for more effective *in situ* or pump-and-treat technologies. To improve the soil treatment performance, new additives and technologies are being developed to increase contaminant mobilization by increasing the aqueous solubility of the contaminant [9]. Chu and Choy [10] demonstrated that a faster photodegradation rate of TCE was observed in the presence of non-ionic surfactant Brij 35 than in water, because the surfactant could be used as an additional hydrogen source in the photolysis reaction.

It is theoretically possible to further improve the photodegradation rates of NAPL-TCE in the surfactant solution. In this study, the application of photolytic irradiation techniques for NAPL-TCE degradation in Brij 35 solutions was studied and optimized through the examination of reaction kinetics at different reaction conditions.

2. Materials and methods

2.1. Chemicals

The chemicals used in this study were trichloroethene (TCE; >99%) from Aldrich, non-ionic surfactant Brij 35 from Warena-chen der ICI America and Anionic surfactant SDS from Aldrich. Because most soil carries cationic adsorption capacity, the application of cationic surfactants to soil-washing activities should be restricted. Non-ionic surfactants usually carry lower critical micelle concentration (cmc), so lower surfactant doses may be expected for field applications. In addition, non-ionic surfactants are safer to use than cationic ones due to their higher biodegradability in natural environments. Thus, two commonly used surfactants were chosen in this study, and some of their physical and chemical properties were shown in ATSDR, 1995 [11]. Acetonitrile of HPLC grade (from Labscan Asia Co. Ltd.) was degassed before being used in liquid chromatography (LC). Inorganic acids/base (A.R. grade) including sulphuric acid and sodium hydroxide were used to adjust the initial pH of solutions to the predetermined

levels. All chemicals were used without further purification. All solutions were prepared using distilled deionized water (resistivity 18 M Ω cm) by a Barnsted NANO pure ultra-pure water system.

2.2. Methods

2.2.1. Solubility experiments

The solubility of TCE in different concentrations of surfactant Brij 35 solutions was investigated. Equal volumes of solutions containing Brij 35, TCE and water were prepared in dark vials. Concentrations of surfactant Brij 35 solutions were varied at 0, 0.00531, 0.01063, 0.02125, 0.03453 and 0.04781 mol/L, with a solution volume of 8 ml. The total volume of each solution was 10 mL, after 2 mL of TCE was added to each solution. The vials were sealed with Teflon-lined caps to prevent any TCE loss from escaping the system via volatilization. The vials were then placed on a rotary shaker and allowed to mix for 6 h. Samples were taken from the vials and analyzed by Liquid Chromatography (LC), which comprised a Waters 515 HPLC pump, a Waters 717plus Autosampler with a 20 μ L injection loop, a Restek pinnacle octyl amine (5 μ m, 4.6 mm \times 250 mm) column, and a Waters 2487 Dual λ Absorbance Detector. The mobile phase consisted of 50% acetonitrile with 50% distilled-deionized water and was delivered at a flow rate of 1.5 ml min⁻¹, which resulted in a TCE peak at 7.1 min.

In addition, the maximum absorbance of TCE aqueous solution was about 213 nm scanned by Spectronic Genseys 2 UV/vis spectrophotometer, which is similar to the data reported by Zuo [12]. Zuo mentioned that the UV absorption spectrum of TCE vapor was measured by a UV-vis spectrometry (HP8453), which showed that TCE have a strong adsorption band at 206 nm and a weaker one at 232 nm. A same measurement was also conducted by using alcohol as solvent, the two adsorption bands were also clearly observed, and that when the concentration of TCE decreased, the 232 nm peak weakened till it vanished as the concentration below 2×10^{-4} M. On the other hand, the maximum adsorbance would transfer a little as the concentration changed. As a result, the maximum absorption wavelength of TCE was pre-selected at 213 nm in this study.

2.2.2. Photodegradation experiments

All photolysis experiments were conducted using an RPR-200 RayonetTM photochemical reactor purchased from the Southern New England Ultraviolet Company. In each experiment, a cylindrical quartz vessel filled with 250 ml mixture of TCE, NAPL of TCE and surfactant was placed inside the reactor and illuminated with monochromatic UV lamps. A magnetic stirrer was located at the reactor's base to achieve sufficient recirculation of the solution in the quartz vessel throughout the reaction; a cooling fan was also installed at the reactor base such that the experimental temperature maintained at 24–25 °C. In each experiment, sixteen 254 nm phosphor-coated low-pressure mercury lamps were installed in the reactor. The intensity of incident UV light, I_0 , was equivalent to 1.44×10^{-3} Einstein L⁻¹ min⁻¹.

Samples were taken from the quartz vessel at different predetermined reaction times until the total degradation of TCE achieved 80% or higher. The samples then were analyzed by LC. From previous studies [10,13–15], the initial TCE photodegradation was postulated to follow pseudo-first-order reaction and could be determined by:

$$C_t = C_0 e^{-kt} \quad (1)$$

where C_t and C_0 are the concentration of TCE (M) at time t and time zero, respectively, and k is the pseudo-first-order decay rate constant (s⁻¹).

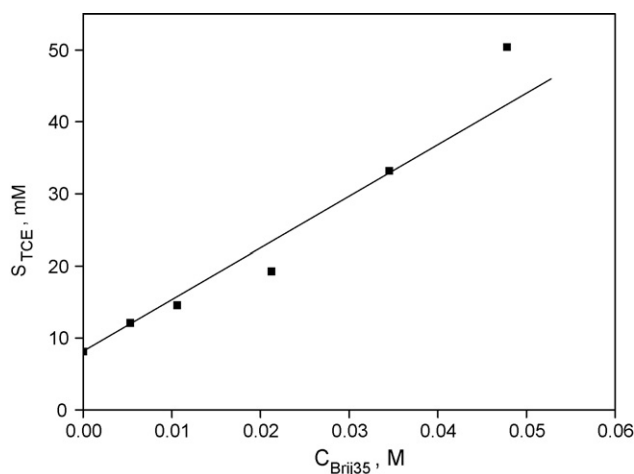


Fig. 1. The solubility of TCE in Brij 35 solutions at various concentrations.

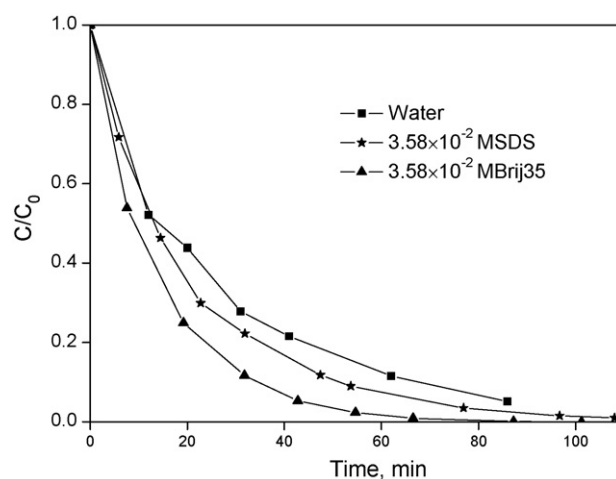


Fig. 2. The photodegradation of 8.14×10^{-3} M TCE in surfactant micelles and water at 254 nm. The initial pH is 4 and light intensity is 1.44×10^{-3} Einstein $L^{-1} min^{-1}$.

3. Results and discussion

3.1. Solubility of TCE in Brij 35 solutions

The solubility of TCE in different concentrations of surfactant Brij 35 solutions was investigated as shown in Fig. 1. Concentrations of Brij 35 were 0, 0.00531, 0.01063, 0.02125, 0.03453 and 0.04781 mol/L, respectively. From Fig. 1, it can be found that the solubility of TCE (S_{TCE}) is strongly dependent on the concentrations of surfactant Brij 35 in solutions, i.e., the higher the concentrations of Brij 35, the higher the solubility of TCE, which is mainly because the solubility of TCE in hydrophobic surfactant micelles are considerably higher than that in hydrophilic aqueous phase [16]. In addition, the linear relationship between S_{TCE} (mM) and concentrations of Brij 35 (C_{Brij35}) (M) can be formulated as below,

$$S_{TCE} = 8.14 + 724.90C_{Brij35} \quad (2)$$

Fig. 1 shows that the solubility of TCE without Brij 35 in water is 8.14×10^{-3} M, which is similar to the data reported by others [17].

3.2. Selectivity of surfactants

The photodegradation of 8.14×10^{-3} M TCE by 254 nm UV in solutions containing different types of surfactants (3.58×10^{-2} M Brij 35 and SDS) and distilled water was investigated and compared (see Fig. 2), and the related information of surfactants physical and chemical characteristics was discussed by Frenkler and Fendler [18]. The decay rates of TCE in surfactant solutions were reported in Table 1. From Table 1, significant rates of improvement were observed in Brij 35 and SDS surfactant solution compared to that in distilled water. The Brij 35 demonstrates the best performance in terms of TCE decay rate and is about 1.6 times faster than that of SDS. This is likely due to cage effect causing by the anionic hydrophilic head in the SDS, which might slow down the diffusion of ionic intermediate in/out the micelle or the hydrophobic chain of SDS is not as a good hydrogen source as the Brij 35 for the photodechlorination reaction.

Table 1
Decay rate of TCE in surfactant solutions

Solution	Surfactant concentration (mol/L)	First-order decay rate (min^{-1})	R^2
Distilled water	0	0.0358	0.9920
SDS	3.58×10^{-2}	0.0436	0.9981
Brij 35	3.58×10^{-2}	0.0694	0.9988

This evidence suggests that the photodegradation of TCE in surfactant micelles is a useful process to increase the reaction rate, but the process is very sensitive to the types of surfactant micelles adopted. It is therefore recommended that only pre-selected surfactants can be used to improve the degradation rate of pollutants in surfactant aided soil-washing/photodegradation applications [8]. In addition, it should be noted that the rates of TCE photodegradation slowly declined (i.e. tailing) after 90–95% of the TCE is removed in the SDS and water phases as show in Fig. 2. The gradual decrease of reaction rates may be due to several reasons: (a) the photoproducts may act as internal light filters that reduce the light available for parent compound TCE degradation; (b) the photoproducts may act as quenchers to retard the photoreaction; and (c) the TCE photoreactions is known to be dominated by photodechlorination process, where chloride ion is one of the end-products. At the later part of the reaction, the chloride has accumulated to a higher level and activated hydrogen source (from surfactant) is relatively at a lower level. Under these circumstance, a minor reversible reaction can be initiated (i.e. photochlorination) and the tailing of TCE is observed. However, no tailing of the TCE is observed in the Brij 35 solution, indicating the reaction is too fast to be hindered by the above retardation mechanisms when Brij 35 is used, so 100% of TCE removal is observed after 75 min of illumination. Since Brij 35 is among the best surfactant for TCE decay, it will be used as the sole surfactant for the rest of this study.

3.3. Optimization of TCE photodecay in Brij 35 surfactant solutions

The pseudo-first-order photodecay of saturated TCE in various Brij 35 surfactant concentrations from 0 to 9.37×10^{-2} mol/L was studied, and the decay curves are shown in Fig. 3(a). This process not only improves the TCE photodecay rate but also reduces the time required for the termination of the process depending on the required removal performance.

The TCE degradation rate in water alone without adding Brij 35 is found to be $3.58 \times 10^{-2} min^{-1}$, while in the presence of Brij 35, the maximum decay rate increases to $9.90 \times 10^{-2} min^{-1}$, that is, nearly 3 times the rate improvement is achieved at the maximum tested concentration of 9.37×10^{-2} mol/L, solely due to the adding of Brij 35. It is interesting to note that the rate increment is not exactly a linear pattern; this can be observed as the TCE decay rate constants are correlated to Brij 35 concentrations. An inflexion

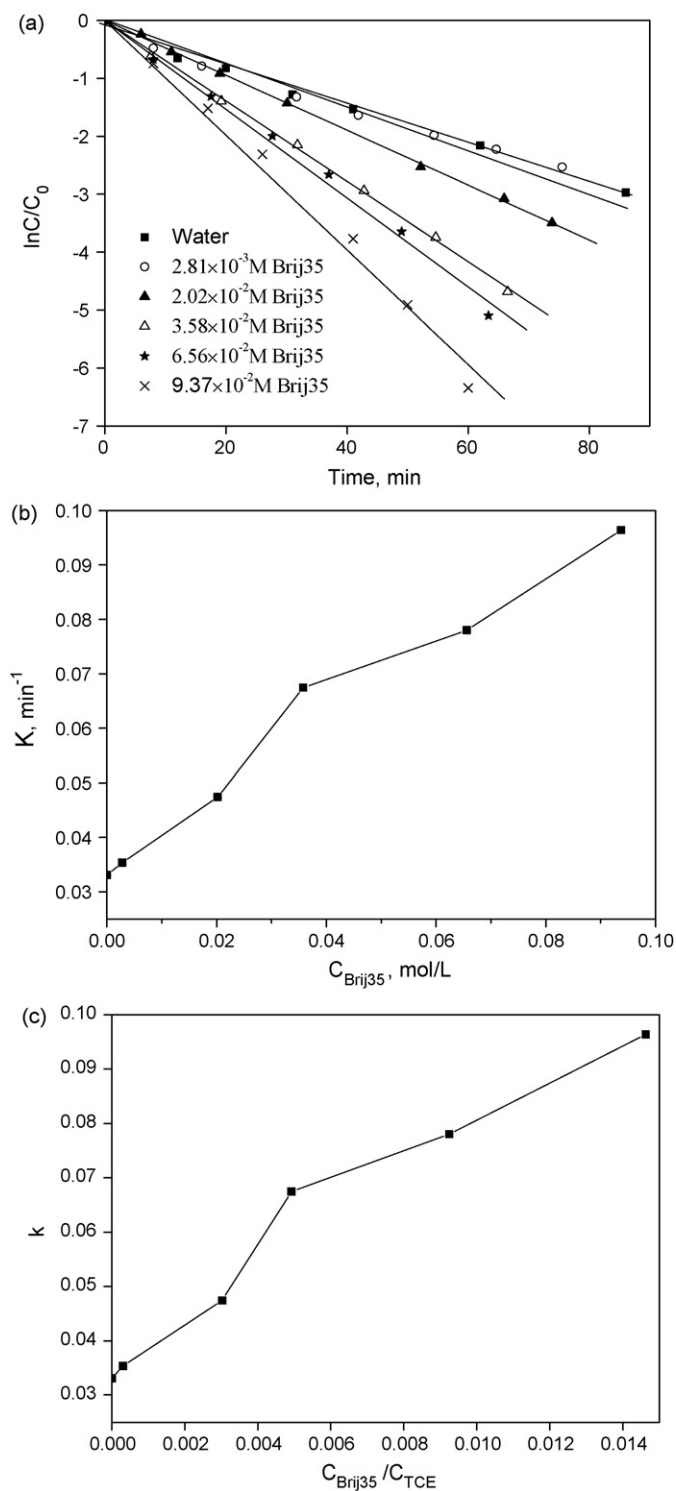


Fig. 3. The photodegradation of saturated TCE in the presence of Brij 35 with different concentrations, at an UV illumination at 254 nm and the initial pH level of 4. (a) The pseudo-first-order decay of saturated TCE in the presence of Brij 35 with different concentrations. (b) Rate constants of TCE photodegradation at various Brij 35 concentrations. (c) Variation of pseudo-first-order rate constants with various concentrations of Brij 35 and TCE.

peak of the overall reaction rate is observed as shown in Fig. 3(b); indicating when the Brij 35 is overdosed in the reaction, the reaction will be retarded slightly. The reason for this retardation effect might be due to the light attenuation by higher concentration of Brij 35. In addition, higher Brij 35 will increase the viscosity of the solution, which is unfavorable to the molecule diffusion between or within the micellar core and aqueous phase and therefore lower the reaction rates. Alternatively, as the TCE photodegradation rate constants are normalized to the ratios of $[\text{Brij 35}]/[\text{TCE}]$ according to the saturated TCE under various Brij 35 (as indicated in Fig. 1), a similar inflexion peak of the overall reaction rate can be observed as shown in Fig. 3(c). For practice purpose, the optimal concentration of Brij 35 at 3.4×10^{-2} mol/L is therefore suggested for avoiding the retardation and getting better cost-effectiveness, which gives a k of $6.6 \times 10^{-2} \text{ min}^{-1}$.

3.4. Photodegradations at different initial pH levels

TCE photodegradations at different initial pH levels in Brij 35 solutions were examined at the suggested $[\text{Brij 35}]$ of 3.4×10^{-2} mol/L. Fig. 4(a) shows the pseudo-first-order reaction of TCE photodegradation in Brij 35 at seven different initial pH levels ranged from 2.00 to 11.55. The first order rate constants at different initial pH levels are shown in Fig. 4(b), which reveals that the rate constants of k are dependent on the initial pH levels. An optimal peak at about pH 9.0 in terms of reaction rate is observed, and the curve can be divided into several sections. As the initial pH is in neutral or acidic ranges, the presence of proton (H^+) can slightly inhibit the TCE decay. As shown in Fig. 4(c), it is important to point out that during the photolysis process, the solution pH gradually decreases. As mentioned by Chu and Choy [10], it can be seen that the decline in pH is accompanied by a reduction in TCE concentration, which suggests that the proton is one of the major end products of the photoreduction reaction of TCE. In addition, the trend of pH decrease in the reaction is analogous to the photodecay of TCE, as shown in Fig. 4(a) and (c), i.e., the lower the solution pH, the lower the reaction rate. This strongly implies that proton generation may inhibit the photodegradation process, which agrees with the observations of lower reaction rates at lower pH levels. For the extreme acidic conditions, no significant pH variation was observed because the protons generated in the reaction are less significant compared to those originally exist in the solution.

When the pH is gradually increased from 8 to 9, the TCE decay is significantly increased with the initial pH levels, this is likely due to the involvement of additional reaction pathway—photohydrolysis of TCE in the presence of higher $[\text{OH}^-]$ in the solution. In addition, in an alkali medium, the generated proton is neutralized by OH^- in the solution, so the rate inhibition by proton can be minimized.

However, as the pH further increased beyond 9, the decay rate suddenly dropped. This is likely due to the chemical property of Brij surfactant. For Brij 35, it has an alcohol group as the hydrophilic head (ROH), in the presence of hydroxide ion at higher pH levels, the equilibrium of the alcohol and OH^- may promote the formation of alkoxide ion (RO^-) which will make the micelle partly carrying negative charge. This will result in the cage effect as discussed beforehand and the higher the pH (i.e. more the alkoxide ion formed), the lower the rate.

3.5. The photodecay of the TCE at different initial NAPL levels

The photodecay of TCE at different NAPL levels in Brij 35 solutions at optimal dosage of 3.40×10^{-2} mol/L and initial pHs 9.0 was examined, as shown in Fig. 5(a). The levels of NAPL were indicated by the additional dose other than the water solubility of TCE

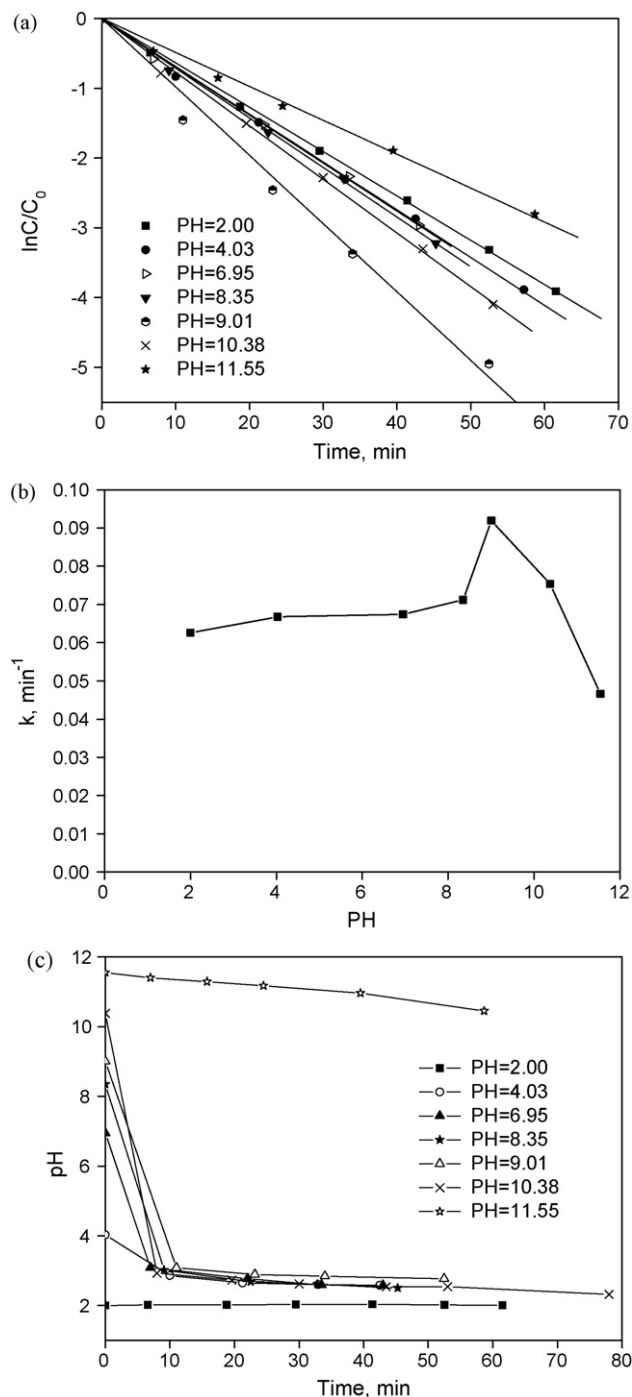


Fig. 4. The photodecay of saturated TCE in the presence of 3.4×10^{-2} mol/L Brij 35, under UV light exposure at 254 nm, at various initial pH levels. (a) The pseudo-first-order decay of TCE in the presence of 3.4×10^{-2} mol/L Brij 35 at various initial pH levels. (b) Rate constants of k at various initial pH levels in 3.4×10^{-2} mol/L Brij 35 solutions. (c) The variation of pH at different initial pHs in 3.4×10^{-2} mol/L Brij 35 solutions.

(S_0), e.g. $0S_0$ stands for the dosage of TCE at its water solubility of 8.14×10^{-3} M, while $1S_0$ stands for 1.63×10^{-2} M TCE in the. It was found that all the photodegradations of TCE-NAPL followed pseudo-first-order kinetics, while higher the NAPL level lower the reaction rate.

It was interesting to note that when the TCE levels were lower than $3S_0$, the photodecay decreased with the increment of [TCE]; while the decay rate approached to a constant as the [TCE] was

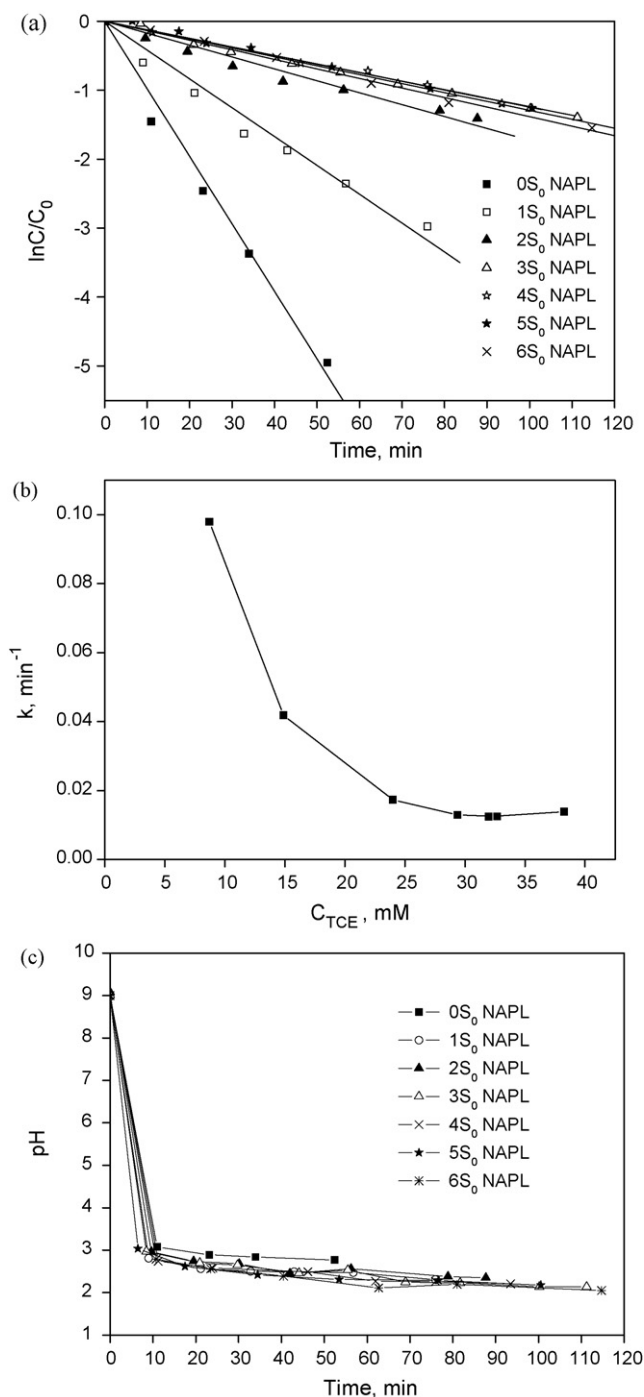


Fig. 5. The photodecay of TCE at different NAPL concentrations in Brij 35 solutions at optimal concentrations of 3.4×10^{-2} mol/L and initial pHs 9.0. (a) The pseudo-first-order decay of TCE at different NAPL concentrations in Brij 35 solutions at optimal concentrations of 3.4×10^{-2} mol/L and initial pHs 9.0. (b) Rate constants of k at various TCE concentrations in 3.4×10^{-2} mol/L Brij 35 solutions and initial pHs 9.0. (c) The variation of pH at various TCE concentrations in 3.4×10^{-2} mol/L Brij 35 solutions and initial pHs 9.0.

higher than $3S_0$ as shown in Fig. 5(b). This is most certainly related to the physiochemical states of TCE in the solution. From Fig. 1, it can be calculated that when the concentration of Brij 35 is 3.40×10^{-2} mol/L, the corresponding solubility of TCE is about 3.32×10^{-2} mol/L, which is closely equal to the dosage of [TCE] at $3S_0$. In another word, the $3S_0$ is the boundary of forming TCE-NAPL in the solution. When the level of [TCE] is lower than $3S_0$ all the

TCE is in the soluble form in micellar solution, while above that TCE-NAPL will present and co-exist with the TCE soluble form in the solution. In the former case, since no NAPL was involved, the reaction is mainly dominated by the concentration of TCE in the solution, higher the [TCE] lower the rate due to the competition of limited UV photons (a constant).

When the NAPL is present and becomes the second phase in the solution, the reaction become complicated. First, it is no doubt that the soluble TCE should decay as usual in the micellar solution, while the TCE-NAPL can be decayed within the NAPL pool simultaneously as a parallel pathway. Judging from the data, it is believed that the diffusion of TCE from NAPL pool to the solution is a slow and insignificant process within the reaction period, since the increase of NAPL in the solution will not affect the decay rate at all and all the TCE decay curves at high TCE levels almost merged together (see Fig. 5(a)). The decay of TCE-NAPL within the NAPL pool therefore can be considered as an independent pathway and will not interfere with the TCE decay in the micellar solution. Since LC analysis only reports the soluble form of TCE in the solution (i.e. in water and micelles) so the decay rate remain as a constant under these circumstances.

In addition, the variation of pH of solutions at different initial NAPL levels was also examined as shown in Fig. 5(c), where the initial pH is adjusted to 9.0. It is found that the proton generation is independent to the initial NAPL levels. This further verifies the assumptions that the decay of TCE in NAPL is an independent pathway and the diffusion of TCE (and its intermediates/end products) between micellar form and NAPL form is relatively insignificant.

4. Conclusion

The photodegradation of a TCE by UV in the presence of surfactants with or without the presence of NAPL was found to be a successful approach for soil remediation. Anionic surfactant SDS is found not an effective soil-washing agent as non-ionic surfactant Brij 35, because of cage effect and poor hydrogen source for the photodechlorination process. The TCE decay rates are strongly dependent on the initial concentration of Brij 35 and the initial pH levels, such information should be carefully considered before the design of such a system. The solubility of TCE in Brij 35 surfactant becomes predictable by using the proposed equation, which is very useful in determining the surfactant dosage and the process can be therefore optimized if cost-effectiveness is the concern in real applications.

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