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THE REMOVAL OF BROMOPHENOL BLUE FROM WATER BY SOLVENT SUBLATION

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ABSTRACT

Bromophenol blue (BB), an anionic dye, was removed from aqueous solution by solvent sublation of a BB hexadecyl-pyridium-chloride (HPC) complex (sublate) into isopentanol. A stoichiometric amount of surfactant (surfactant-dye = 2:1) was the most effective compound for the removal, with over 95% BB removed from the aqueous solution in 10 minutes by solvent sublation. The removal rate was somewhat enhanced by high airflow rates and was also more or less independent of the organic solvent volume floated on the top of the aqueous column. The effects of electrolytes (e.g. KCl), nonhydrophobic organic compounds (e.g. ethanol), and pH of the solution upon the process were studied. The infrared spectra showed that the interaction between sublate and

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organic solvent was weak. The comparison of air stripping and solvent extraction with the solvent-sublation process was made. The solvent-sublation process was consistent with first order kinetics. A characteristic parameter, apparent activation energy of attachment of the sublate to bubbles, was determined as 4.77 KJ/mol.

INTRODUCTION

Solvent sublation, originated by Sebba (1) for ionic-surfactant complexes, has shown a promise for removing hydrophobic molecular compounds and ion-pair complexes from aqueous systems. Solvent sublation is one of several adsorptive bubble-separation techniques, in which a hydrophobic compound is levitated on a bubble surface to the top of an aqueous column where it encounters a solvent layer (e.g., mineral oil, lauryl alcohol) to which the material is transferred as the bubbles move through the solvent layer (2,3).

The solvent-sublation process is different from solvent extraction. When fine gas bubbles (of radii 0.01–0.05 cm and in laminar flow) pass through an aqueous column (with an overlaying organic layer) that contains hydrophobic compounds, they inherently concentrate at the air-water interface. These hydrophobic materials are collected on the bubble surface by diffusion through the thin boundary layer that surrounds the air bubble. As the bubble transits the aqueous column and moves through the organic solvent layer, the adsorbed phase gets stripped into the organic phase. Thus, the solvent-sublation process improves the efficiency of a bubble aeration column. Moreover, the presence of the organic solvent also reduces the eventual redispersion of the material into the aqueous column upon bubble bursting, which usually occurs in conventional bubble aeration columns (4).

Most sublation-technique studies have focused on the removal of organic pollutants from aqueous systems, such as alkyl phthalate, volatile chlorinate organic compounds, dichlorobenzenes, nitrophenols, polynuclear aromatics, chlorinated pesticides (5–11), and the emission of volatile organic compounds (volatile chlorinated organics and toluene) to the atmosphere in solvent-sublation processes (12,13). Other studies were conducted on the solvent sublation of dyes in aqueous solution, such as the selective separation of BB from dye solution by solvent sublation with quaternary ammonium into octanol (14), the separation of methyl orange from Rhodamine B (15,16), the solvent sublation of the methyl orange–hexadecyltrimethylammonium ion pair (17), Magaenta (a cationic dye) with sodium lauryl sulfate (18), and Direct Red and Acid Red (two anionic dyes) with hexadecyltrimethylammonium ion (HTA) (19,20).

Recently, both the mechanism and kinetics of solvent sublation were investigated by Palagyi (21–25), who proposed an ionic-associate formation mechanism and second order kinetics in the separation of iodide from water by solvent sublation with hexadecyl-pyridiumchloride (HPC) into benzene. However, the studies of both kinetics and thermodynamics of solvent sublation are few. Although BB was studied previous by Sebba and Lovell and Sebba (1,14), we propose a modified method of BB separation by solvent sublation with HPC into isopentanol. The kinetics and thermodynamics of the solvent sublation of BB-HPC were investigated and the efficiencies of solvent sublation with some conventional separation methods were compared. The effects of parameters, such as the airflow rates and the volume ratio of the aqueous phase to the organic solvent, were studied, and the effects of certain co-solutes were also tested.

EXPERIMENTAL

The solvent-sublation system was similar to that previously described (26). The glass column was 90 cm in length with an i.d. of 3.7 cm and had 3 access ports. A titanium plate was used to introduce air bubbles into the aqueous phase. To obtain a representative concentration of the aqueous phase, the sample solution was withdrawn from the middle access port for analysis.

Reagent-grade HPC (Fluke AG, Buchs SG, Switzerland) was used as a collector without further purification. Reagent grade BB (Shaihai Chemical Agents Factory, China), and the other reagents were all analytical grade.

For the solvent sublation runs, HPC was added to the sample solution (350 mL) to form the dye-surfactant complex; the solution containing 1.0×10^{-5} mol/L BB was poured into the sublation column; and 5 mL isopentanol was added immediately; then the timer was started and the samples of aqueous solution were taken for analysis at a specified times.

The solution pH was measured with a pHs-3C (Shaihai Rex Industry, China). UV-visible spectra of the sample solutions were measured with a Unico PC2100 UV/Vis Spectrophotometer (Unico Co, China) at the maximum peak of 592 nm. The infrared (IR) spectra of the top layer, which contains of the BB-HPC sublate or pure isopentanol, was analyzed with the IR Spectrophotometer (Nicolet, USA).

RESULTS AND DISCUSSION

The Effect of the Molar Ratio of HPC to BB on Solvent Sublation

The effect of HPC concentration on the solvent sublation of BB is shown in Fig. 1. A 2:1 mole ratio of surfactant-dye gave the fastest rate of separation and the lowest residual dye concentration, with over 95% of BB removed in 10 minutes. At smaller concentrations of surfactant, the rate of removal was slower

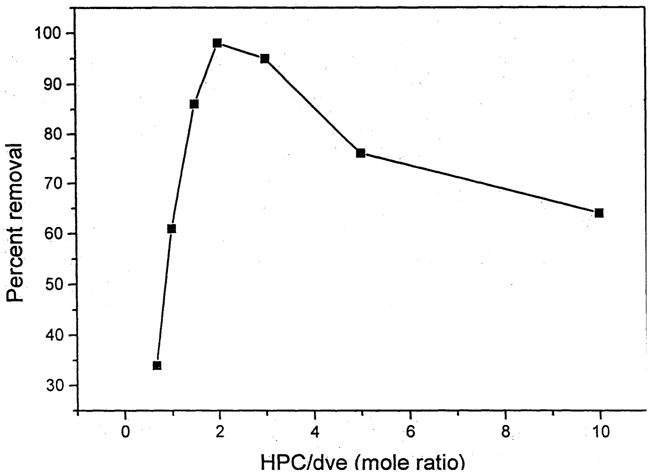


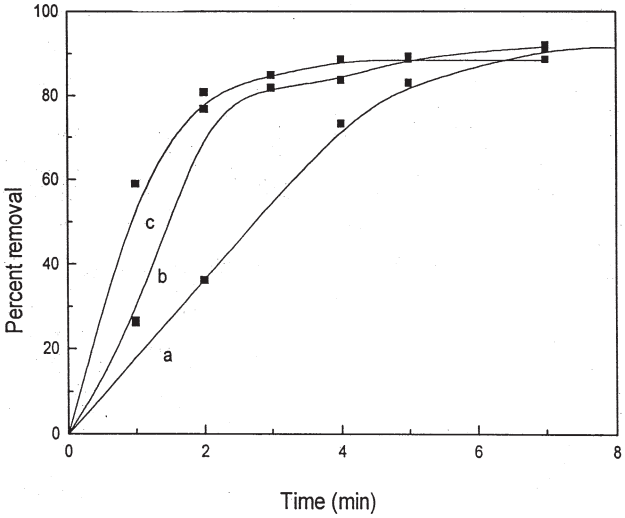
Figure 1. Effect of HPC dose on solvent sublation. Air flow rate = 75 mL/min, and air flow duration = 10 minutes.

and the level of the residual dye was greater, presumably due to incomplete formation of a dye-surfactant complex. However, when the ratio was higher than 2:1, the rate of solvent sublation was smaller and the removal efficiency was lower, presumably due to the competition of excess surfactant ion with the dye-surfactant complex for the bubble surface. In the process of solvent sublation of HPC without BB, the recovery efficiency was higher than 98% (27), which indicated that the surfactant was competing for adsorption spots on the bubbles. The excess surfactant also caused the emulsification of the isopentanol (which was observed during the solvent-sublation process with a large excess of surfactant), such that the dye-surfactant complex in the isopentanol was constantly dispersed back into the solution and the separation efficiency decreased greatly. The optimum separation efficiency with surfactant dosage at the stoichiometric amount was also observed for the solvent sublation of HTA-Acid Red (20). The finding with HTA-Red was quite different from the results of solvent sublation experiments of methylene blue and methyl orange studied by Carger and Karger (15–17) and Womack, Lichter, and Wilson (28), who found that the rate of removal of dyes increased with increased surfactant concentration when it was in great excess of the stoichiometric amount. This contradiction was probably due to the difference in the formation constant of the varied surfactant-dye complexes and may also be due to the altered molar ratio of the surfactant and dye in the complexes (20).

The Effect of Different Air Flow Rates on Solvent Sublation

The solvent sublation of BB into isopentanol was investigated at 3 different air flow rates (35, 75, 150 mL/min). The removal rates increased with increased air flow rates, as shown in Fig. 2. However, the increase in removal rate was not proportional to the increase of air flow rates; this finding is similar to the results of research by Valsaraj et al. (4). This result is probably due to the mean bubble-radius increase associated with increasing air flow rates. As the interfacial area per unit volume of air (3/*r*) decreased, the bubble residence time also reduced because the larger bubbles have higher rise velocities. Furthermore, the axial dispersion certainly increased with the increase of the air flow rates, which would impair the performance of the sublation process.

At higher flow rates, the oil-water interface was drastically disrupted and some drops of the top layer could return to solution. Although the increased air flow rate can improve the removal rate of solvent sublation, the removal effi-



	Air Flow Rate (mL/min)	Rate Constant K $(s^{-1}) \times 10^4$	SE of *k* $(s^{-1}) \times 10^4$
a	35	0.55	0.05
b	75	0.83	0.11
c	150	1.38	0.06

Figure 2. Effect of rate of air flow on solvent sublation.

ciency decreases if the airflow rate is quite high because the air currents arising from the high air flow rates disrupt the oil-aqueous interface.

The Effect Organic Solvent-Aqueous Phase Volume

Sebba (1) showed that in the case of the solvent sublation of ion-surfactant complexes from aqueous solution into 2-octanol, the removal efficiency is independent of 2-octanol volume. Caballero concluded that the sublation efficiency is independent of the organic solvent volume only until the phase is saturated by the sublate (29). We obtained the same result as these researchers through the experiments, the results of which are shown in Fig. 3. No significant improvement in removal efficiency was observed as a result of changing the aqueous phase-solvent (isopentanol) ratio from 175:1 to 35:1. The results showed that sublation was independent of the organic solvent volume. For the most part, mass-transfer occurs from gas bubbles crossing the aqueous phase–solvent interface and not from diffusion of solute across this interface; the amount of material transferred should depend only on the amount of air crossing the interface and not on the organic volume. While the volume ratio of the 2 immiscible phases is a very important parameter in liquid-liquid extraction, it is not critical to the solvent-sublation process. However, if the organic volume used in solvent sublation is too low, the oil-water interface will be drastically disrupted at a high air flow rate and the process will lose its efficiency. Hence, the air flow rates and solvent volume must be chosen to minimize disruption of the interface.

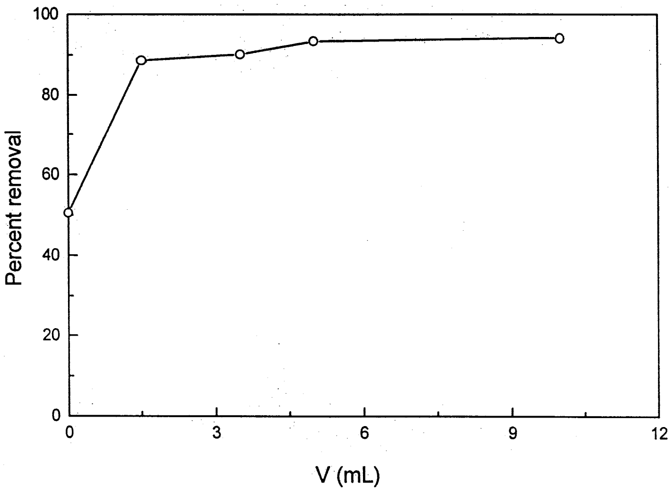
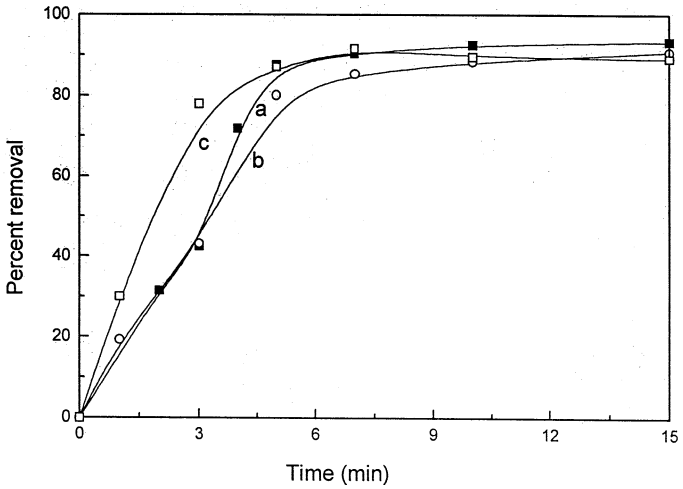


Figure 3. Effect of solvent volume on solvent sublation. Air flow rate = 75 mL/min.



	Mole Fraction of Ethanol	Rate Constant K (s^{-1}) × 10^4	SE of *k* (s^{-1}) × 10^4
a	0	0.49	0.07
b	0.006	0.50	0.06
c	0.06	0.61	0.04

Figure 4. Effect of ethanol mole fraction on solvent sublation. Air flow rate = 75 mL/min.

Effects of Co-Solute

The influence of 0.006–0.06 mole fractions of the ethanol the co-solute upon the BB removal rates is shown in Fig. 4. The low mole fraction of ethanol had no significant effects on solvent sublation, and the ethanol with high mole fraction enhanced removal rates.

The removal rate enhancements might be due to 2 factors. First, the addition of ethanol changed the bubble properties considerably; more very small bubbles were evident upon addition of ethanol than when ethanol was absent. This is a well-known effect that arises from the lowering of water surface tension and prevents the bubbles from growing larger. These smaller bubbles provide a very large surface area per unit volume of air, which enhances mass transfer from the liquid phase to the bubbles. At the same time, smaller bubbles also have slower rise velocities (30). Second, according to Ben-Naim (31), ethanol mole fractions up to 0.03 increase the hydrogen bonding of water and thus make the aqueous phase "less comfortable" for organic molecules. Under this concept, the BB molecules would prefer the air-water interface of the rising bubbles to the organic phase.

The effect of the inorganic salt, KCl, used as a co-solute on the sublation of the BB-HPC complex is shown in Fig. 5. Increasing the KCl concentration is correlated with a decrease in the removal rate and the removal efficiency. This finding was attributed to an ion-pair equilibrium that existed in the aqueous solution between the dye and surfactant molecules. In aqueous solution, the following equation is applicable:

$$HP^+B^- + K^+Cl^- \rightleftharpoons HP^+Cl^- + K^+B^-$$

The increase of the salt concentration (i.e., KCl) drove the equilibrium toward a larger concentration of K^+B^-, which was hydrophilic, and less mol of HPC-BB complex (i.e., HP^+B^-) existed in the aqueous phase. Thus, the rate of removal decreased.

The Effect of pH

The pH effect HPC-BB removal in the solvent-sublation process is shown in Fig. 6. The removal rate and removal efficiency of BB increased at lower pH. Over 90% of BB was removed from the solution by solvent sublation within 10 minutes at acidic pH. But at higher pH (10.58), the removal rate and efficiency de-

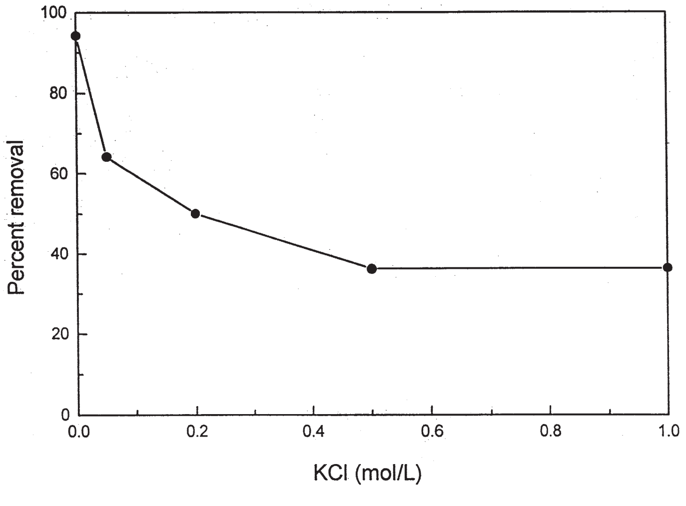


Figure 5. Effect of KCl concentration on solvent sublation. Air flow rate = 75 mL/min, and air flow duration = 10 minutes.

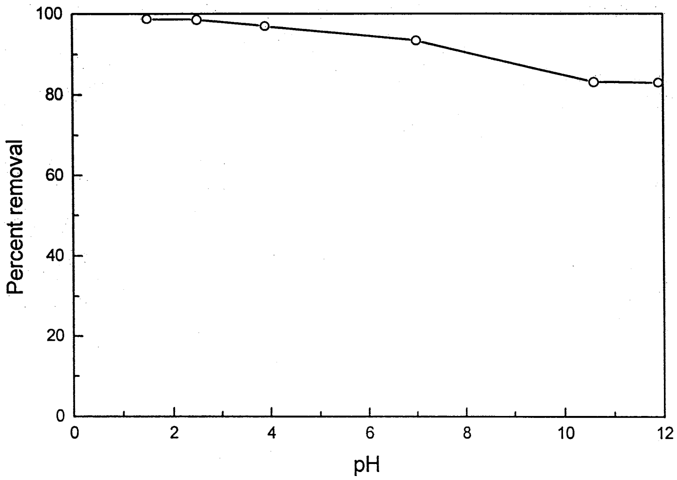


Figure 6. Effect of pH on solvent sublation. Air flow rate = 75 mL/min, and air flow duration = 10 minutes.

creased. In aqueous solution, the following equation can be applied:

$$B + A^- \underset{OH^-}{\overset{H^+}{\rightleftharpoons}} HA + BH^+ \rightleftharpoons [HA\text{-}BH^+]$$

Where B represents HPC, and HA is BB. The increase of the acidic pH drives the equilibrium toward a larger concentration of BH^+, which easily binds with HA to form ($HA-BH^+$). By contrast, the increase of the basic pH drives the equilibrium toward a larger concentration of B, which does not easily form [$HA-BH^+$].

Comparison with Air Stripping and Solvent Extraction

To investigate the effect of solvent on sublation, the IR spectra of the solvent and the top layer after sublation were compared. The results of IR spectra demonstrated that the force between the solvent and the sublate (BB–HPC) was very weak, which may indicate that the solvent only dissolved the sublate and the solvent was not a decisive factor in the solvent- sublation process. Furthermore, the air stripping process of BB (lack of HPC) was observed. The rate of the air stripping was very low ($6.7 \times 10^{-5} s^{-1}$), but the rate of the solvent sublation increased largely ($2.8 \times 10^{-3} s^{-1}$) under the same experimental conditions. Based on the results of the above experiments, one can conclude that the surface activity could accelerate the removal rate greatly; that is, the surface activity is a decisive factor in the solvent-sublation process.

To obtain more accurate results for comparative purposes, a separate extraction process was carried out using 250-mL extraction funnels. Solvent extraction of 50 mL BB, in the absence of HPC, into 10 mL isopentanol was completed. The result showed that only 3.6% of BB, which was the same as removal due to the air stripping process (3.9%). Fifty milliliters of BB-HPC solution and 10 mL isopentanol were respectively poured into the funnel in which the aqueous layer was continuously stirred. The equilibrium was reestablished in approximately 3 days. The removal efficiency of the solvent-extraction process was similar to that of the solvent-sublation process. But the former process required more time for completion; the latter reached equilibrium in only 10 minutes. The solvent extraction consisted of immiscible drops, which made the equilibrium proceed slowly. At the same time, the rate of solvent extraction was dependent on the volume ratio. From the above comparisons, we show that the solvent-sublation process is more rapid and more convenient than that of solvent extraction, and the addition of surfactant is very important to the removal of BB.

The Dynamics and Thermodynamics Mechanism of the Solvent Sublation

In the chemical reaction, the rate is followed by:

$$-dc/dt = kc^n \tag{1}$$

where c is the reaction concentration; k is the apparent rate constant; n is the order of chemical reaction. Upon the analysis of the experiment results, we found that the kinetics of solvent-sublation process was consistent with the equation

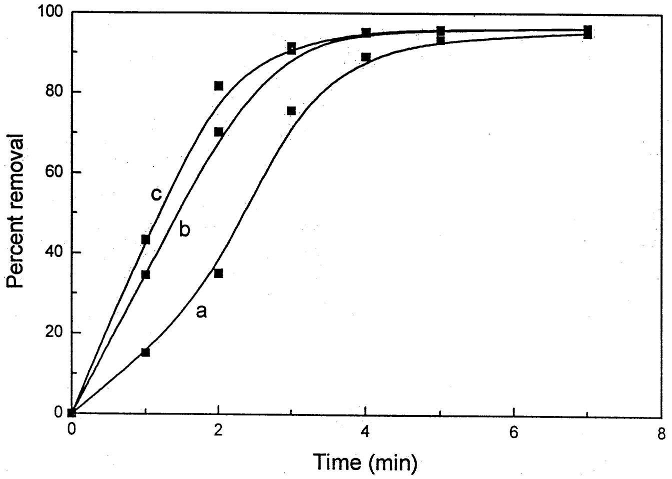
$$-\frac{dc}{dt} = kc \tag{2}$$

The kinetics mechanism of solvent-sublation process was consistent with the first order kinetics equation.

In the solvent-sublation process, the BB-HPC molecules and the bubbles attracted, and then the pulse forces increased, which resulted in boundary layer compression, thinning, and eventual eclipse. The energy changes of these processes were similar to those of the chemical reaction process (4). So, we used the Arrhenius equation to describe the process:

$$\ln k = -\frac{E_s}{RT} + B \tag{3}$$

where k is the rate constant; R is the common gas constant; B is the integrate constant; and Es is the solvent sublation activation energy. The relationship of $\ln k$ and $1/T$ is linear, and the value of the active energy can be calculated from the slope of the line. At a concentration of BB-HPC solution identical to that in previous experiments, the experiment temperature was varied from 288K to 337K. The re-



	Temperature (K)	Rate Constant K (s^{-1}) × 10^4	SE of *k* (s^{-1}) × 10^4
a	288	0.99	0.21
b	305	1.17	0.12
c	337	1.33	0.09

Figure 7. Effect of temperature on solvent sublation. Air flow rate = 75 mL/min.

moval efficiency of BB at different temperatures is shown in Fig. 7. The rate of solvent sublation was increased with the increase of temperature. The result was different from the ordinary phenomena (32). Three effects are responsible for the change of *k* with temperature: the change in bubble size distribution, the decrease of liquid from the cell, and the change in bubble surface excess. To compare the similarities of the solvation-sublation with the flotation process, one can use the following equation (32):

$$\Gamma_f(c) = \frac{V_o kc}{\phi_A} \tag{4}$$

where Γ_f is the surface excess carried by a bubble as it leaves the aqueous solution, V_0 is the aqueous volume; *A* is the amount of area carried through the column in unit time; *k* is the rate constant; and *c* is the concentration. Γ_f was proportional to *k*, thus ϕ increased with temperature increase. This result was consistent with that obtained in an activation-energy barrier-controlled process. The value of active energy was calculated as 4.77 KJ/mol through Eq. 3. This active energy rate was so slow that the solvent-sublation process was very rapid.

CONCLUSIONS

BB was removed effectively from the aqueous solution by solvent sublation with the cationic surfactant HPC. A stoichiometric amount of surfactant (dye-surfactant = 1:2) was found to be the most effective at removing the dye: Over 95% of BB was removed in 10 minutes. If the bubble sizes are kept small, the increased air flow rates enhance the process of solvent sublation. However, at higher air flow rates, the generation of axial dispersion compromises the efficiency of the solvent-sublation process.

Solvent sublation was somewhat independent of the organic volume when the organic volume is larger than that depicted at a critical value; that is, disruption of the organic-aqueous interface was minimal.

Smaller fractions of ethanol (0.006) made no significant effect on the efficiency of the solvent sublation; by contrast, large fractions of ethanol (0.06) improved the rate of removal. The increase of KCl concentrations drastically decreased the removal rate of sublation because more hydrophilic ionic species were generated. The acidic pH increased the rate, but the base pH decreased the rate of sublation.

The solvent sublation process followed first order kinetics. We investigated the thermodynamic mechanism of solvent sublation and found that the sublation process was controlled by an activation energy barrier and the apparent active energy (4.77 KJ/mol) was presented as a parameter of the solvent-sublation process.

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