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Separation Science and Technology

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Recovery of Butyl Acetate in Wastewater of Penicillin Plant by Solvent Sublation II. Theoretical Modeling

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To cite this Article Sun, Xinghua , Chang, Zhidong , Liu, Huizhou and Gao, Renlong(2005) 'Recovery of Butyl Acetate in Wastewater of Penicillin Plant by Solvent Sublation II. Theoretical Modeling', Separation Science and Technology, 40: 4, 941 — 957

To link to this Article: DOI: 10.1081/SS-200044730

URL: http://dx.doi.org/10.1081/SS-200044730

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Separation Science and Technology, 40: 941–957, 2005
Copyright © Taylor & Francis, Inc.
ISSN 0149-6395 print/1520-5754 online
DOI: 10.1081/SS-200044730

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Abstract: In recent years, pilot-scale solvent sublation has been explored in the treatment of industrial wastewater containing dilute organics. In the present work, a mathematical model is proposed to describe the experimental results of semibatch sublation for recovering butyl acetate (BA) in the wastewater from solvent extraction of penicillin. Different from other models in recent literatures, our model took account of the variation of the volume of solvent, v_o, in the sublation. The model parameters of the sublation of BA in the wastewater are calculated. Calculation results show the volume of solvent could increase by more than 30% in 180 min under optimal

Received May 2004, Accepted October 2004.

This work was supported by a grant from North China Pharmaceutical Corporation Limited. We would like to thank the National Natural Science Foundation of China for financial support (No. 20406021, No. 20221603, and No. 20236050). We would also like to thank Prof. Mooson Kawuk and Prof. Chia-yung Chen for their revision of language.

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conditions. Calculation results with different operation parameters show that increasing the gas flow rate, Q_a, could improve the sublation efficiency, E_{ss}, which would be more than 70% under optimal conditions. Decreasing the bubble size, a, would increase the rate of the sublation. The initial solvent volume, v_{oi}, may affect the sublation weakly when initial ratio of water to solvent used is in the range from 50 to 70. Calculation results also show that the loss of butyl acetate to the atmosphere, E_{ls}, is minimal, though with high gas flow rate. The simulated results are in good agreement with experimental results.

Keywords: Solvent sublation, mathematical model, butyl acetate, penicillin, wastewater

INTRODUCTION

Solvent sublation, suggested first by Sebba (1), is an adsorptive bubble separation process combining the benefits of bubble fractionation and transport extraction, especially effective for treating wastewater containing dilute volatile or nonvolatile organics. The removals of inherent hydrophobic compounds by solvent sublation were explored in recent years (2, 3). Wilson et al. and Valsaraj et al. carried out many studies on the removal of refractory organics from water (4–7), to further our understanding of the process. Thoma et al. studied solvent sublation for treatment of oil-field-produced water by dissolved air precipitation (8). Bryson and Valsaraj published a pilot-scale study of solvent sublation to remove naphthalene from a process water stream (9). All of the studies show solvent sublation is an attractive separation process.

Many studies on the theoretical modeling of solvent sublation were carried out to provide parameters for the design of separation equipment or to predict sublation results with simple parameters. Karger and his associates first gave, qualitatively, the possible transport pathways in solvent sublation (10–12). They improved the assumption made by Sebba that sublate moved across the water-solvent interface in one direction only. Their alternative mechanism was that sublate moved in both directions across the liquid-liquid interface. They also proposed that as bubbles coalesced on the water-solvent interface and penetrated into the organic phase, thin water films around them were dragged into the solvent phase only to be turned back again into the aqueous phase as water droplets. This process of water drag-up by bubbles and return as droplets would maintain an eventual steady state. They assumed that bubble layer at water-solvent interface would prevent the solute from returning again to water. Nevertheless, mathematical model suitable for a theoretical and predictive treatment of these phenomena was not given. Wilson et al. proposed different outstanding mathematical models of batch or continuous sublation including the effect of the rate of solute mass transfer from the aqueous phase to the air-water interface

(4, 13, 14). Valsaraj and Thibodeaux compared sublation to the conventional liquid-liquid extraction, providing, at the same time, the mechanisms and a complete model for sublation (15). Unfortunately, the effect of gas holdup on the diffusive mass transport at the water-solvent interface driven by a concentration gradient between water and solvent has not been included. It remained for Smith et al. to account for this point in their models (16). Ososkov and his coworkers used gas chromatography to study the emission of organic compounds to the atmosphere and deduced that such emission could be significantly reduced by using solvent sublation instead of air stripping (17, 18). Valsaraj and Thibodeaux disregarded the amount of solute lost to the atmosphere in an overall solute mass balance across the organic solvent and aqueous phases in order to simplify the calculation (15). All of the mathematical models above ignored the change in the volume of solvent layer because the volume of removed solute could be insignificant.

In the first of a two-part series, we described the experimental study on the semibatch sublation for recovering rather large concentrations of butyl acetate (BA) in wastewater from solvent extraction of penicillin. Results show that solvent sublation is a very efficient process for such recovery. As BA with initial concentration of about 1% in the wastewater is sublated into the solvent, the volume of solvent also increases, as much as by more than 30% at the end of the sublation. An improved theoretical model should be given in order to predict the sublation results with simple parameters. The usual simplification accepted in the previous literatures that the volume of solvent is taken to be invariable in the simulation of solvent sublation is considered unsuitable. In order to account for the actual variation of solvent volume during the sublation, our model takes into consideration the solvent volume, v_o, as they vary with sublation time, t. In the same way, the volume of aqueous phase, v_w is also treated as a variable. Though much less, the amount of BA emitted to the atmosphere by wake of the gas bubbles should not be neglected. Furthermore the ratio of loss is also calculated in the simulation.

MECHANISMS AND MODELING FOR THE SEMIBATCH SUBLATION

Mechanisms

In solvent sublation, there are three principal mass transport processes in which solute is transported from aqueous phase into organic solvent (16).

Transport by Bubbles

As shown in Fig. 1A, mass transport by bubbles is the most important transport pathway in solvent sublation. Solutes with surface activity or hydrophobic

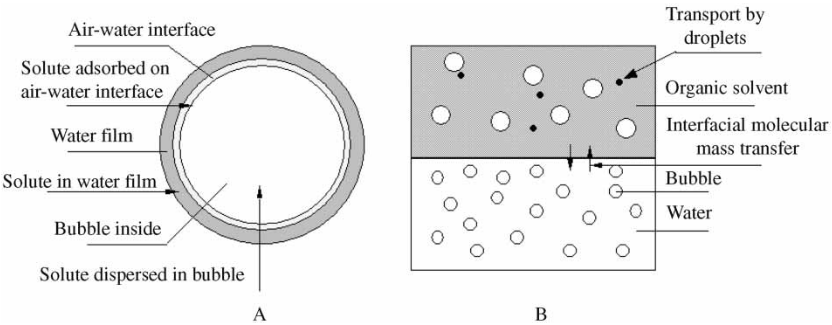

Figure 1. Schematic transport processes in solvent sublation.

property could be adsorbed or attached to the air-water interface of rising bubbles. For simplifying the simulation of solvent sublation, the extent of this adsorption is usually determined by linear Gibbs adsorptive isotherm equation. As bubbles ascend in the sublation column, some volatile solute determined by Henry's law also disperses into their interior. As bubbles traverse the water-solvent interface, penetrate into the organic solvent layer, and break, solute carried by them is released into the solvent floating on top of the aqueous phase.

Solvent-Water Interfacial Mass Transport

As shown in Fig. 1B, molecular mass transport at the water-solvent interface is usually driven by the concentration gradient of solute between the aqueous phase and organic solvent. In conventional liquid-liquid extraction, it is the only transport mechanism, but it is not the predominant transport process in solvent sublation because of the most significant mass transport pathway by adsorption to the air-water interface in sublation. The mass transport rate on water-solvent interface is characterized by the aqueous phase overall mass transfer coefficient K_1.

Transport by Water Film

Rising bubbles in the water column are not exactly spherical but are actually shaped more like semispherical caps. Located behind the bubble resides a small quantity of water called the wake. In the wastewater after solvent extraction of penicillin, the presence of biological materials and surfactants made the rising bubbles very small and almost spherical with thin films around them as shown in Fig. 1A. When bubbles penetrate into the solvent, the thin water films are also entrained into it. Then the water in the films disengages the bubbles and they become very small droplets descending to the aqueous phase because of gravity. In the process, the solute in water films is entrained into the solvent and the equilibrium amount of solute with organic solvent is returned to the aqueous phase by descending droplets. In our solvent sublation, we approximately calculated the volume of water film by the conventional wake's volume prediction as shown in Eq. (1).

$$V_{wk} = 4\pi a^2 d_i \tag{1}$$

In addition to these three mass transport mechanisms discussed above, a little solute could be emitted into the atmosphere in the gas wake, especially when the solute is a volatile compound. But in solvent sublation, the amount of the solute is very low because of the trapping of organic solvent on the top of column.

Modeling

In order to simplify the simulation process, two assumptions are first made. The first one is that bubbles reaching the solvent-water interface are in equilibrium with the aqueous phase. The second one is that both phases are perfectly mixed.

For the semibatch sublation, mass balance equation of solute in organic solvent layer at time t is given by

$$\begin{aligned}\frac{d(v_o c_o)}{dt} = V_o\frac{dC_o}{dt} &= Q_a(H_c + \frac{3}{a}K_a + \frac{3}{a}d_i)\frac{C_w}{1+(M_s/\rho_s)C_w}\\ &\quad + \pi r_c^2(1-\varepsilon)K_1\left(C_w - \frac{C_o}{K_{ow}}\right)\\ &\quad - Q_a\left(H_c + \frac{3}{a}d_i\right)\frac{C_o/K_{ow}}{1+(M_s/\rho_s)(C_o/K_{ow})}\end{aligned} \tag{2}$$

Mass balance equation of solute in the bulk aqueous phase at time t is given by

$$\begin{aligned}\frac{d(v_w c_w)}{dt} = V_w\frac{dC_w}{dt} &= -Q_a\left(H_c + \frac{3}{a}K_a + \frac{3}{a}d_i\right)\frac{C_w}{1+(M_s/\rho_s)C_w}\\ &\quad - \pi r_c^2(1-\varepsilon)K_1\left(C_w - \frac{C_o}{K_{ow}}\right)\\ &\quad + Q_a\left(\frac{3}{a}d_i\right)\frac{C_o/K_{ow}}{1+(M_s/\rho_s)(C_o/K_{ow})}\end{aligned} \tag{3}$$

Overall volume balance equation of the aqueous phase at time t is given by

$$v_{wi} - v_w = \frac{M_s}{\rho_s}V_w(C_{wi} - C_w) \tag{4}$$

Overall volume balance equation of the solvent phase at time t is given by

$$v_{oi} - v_o = \frac{M_s}{\rho_s} V_o(C_{oi} - C_o) \tag{5}$$

The relationship of C_o and c_o is given by

$$c_o = \frac{C_o}{1 + (M_s/\rho_s)C_o} \tag{6}$$

The relationship of C_w and c_w is given by

$$c_w = \frac{C_w}{1 + (M_s/\rho_s)C_w} \tag{7}$$

The sublation efficiency of BA at time t, E_{ss}, is defined by Eq. (8) as discussed in the first of the two-part series.

$$E_{ss} = \frac{c_{wi} - c_w}{c_{wi}} \times 100\% \tag{8}$$

The loss ratio of BA emitted to the atmosphere, E_{ls}, is calculated by

$$\begin{aligned} E_{ls} &= \frac{(v_{wi} - v_w) - (v_o - v_{oi})}{v_{wi} - v_w} \times 100\% \\ &= \frac{V_w(C_{wi} - C_w) - V_o(C_o - C_{oi})}{V_w(C_{wi} - C_w)} \times 100\% \end{aligned} \tag{9}$$

Defining the initial conditions: at $t = 0$, $C_w = C_{wi}$, $C_o = C_{oi}$, $V_o = V_{oi}$, $V_w = V_{wi}$, the set of equations containing two differential Eqs. (2) and (3) can be solved by Runge-Kutta method, We can obtain the numerical values of C_w and C_o, at time t. Combining Eqs. (4)–(7), v_o, v_w, c_o, and c_w can be calculated. The sublation efficiency of BA, E_{ss}, and the loss ratio of BA emitted to the atmosphere E_{ls} also can be calculated.

MODEL PARAMETERS

Solute Partition Constant K_{ow}

In the conventional liquid-liquid extraction, the solute partition constant, K_{ow}, can be obtained from Eq. (10). Under a series of different volumetric ratios of solvent to water, V_o/V_w, the term of the extraction efficiency, $E_{se}/(1 - E_{se})$, may be calculated. Therefore, K_{ow} is given by plotting the extraction efficiency, $E_{se}/(1 - E_{se})$, vs. the volumetric ratios of solvent to water, V_o/V_w. The process to calculate the partition constant of BA between the simulated

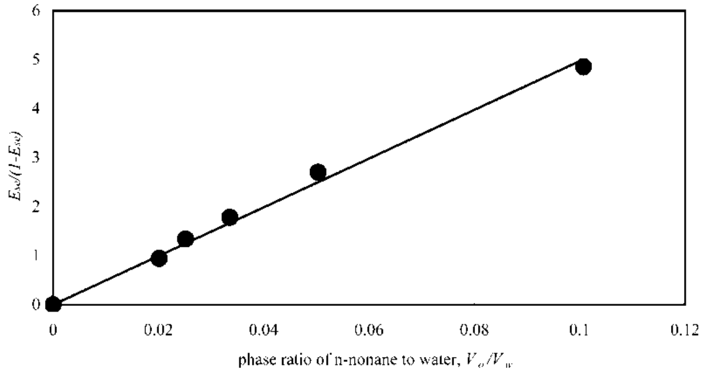

Figure 2. $E_{se}/(1-E_{se})$ vs. phase ratio V_o/V_w.

wastewater and n-nonane at about 288.15 K is shown in Fig. 2.

$$\frac{E_{se}}{1-E_{se}} = K_{ow}\frac{V_o}{V_w} \tag{10}$$

Aqueous Phase Overall Mass Transfer Coefficient K_1

In conventional laminar liquid-liquid extraction, a mass balance equation of solute in organic solvent layer is given by

$$V_o\frac{dC_o}{dt} = \pi r_c^2 K_1\left[C_w - \frac{C_o}{K_{ow}}\right] \tag{11}$$

An overall mass balance equation of solute is given by

$$V_w C_{wi} = V_w C_w + V_o C_o \tag{12}$$

Substituting Eq. (12) into Eq. (11), we obtain

$$-\ln\left(1-\frac{C_o}{A}\right) = pt \tag{13}$$

where

$$A = \frac{C_{wi}}{(V_o/V_w)+(1/K_{ow})} \tag{14}$$

and

$$p = \frac{K_1\pi r_c^2}{V_o}\left(\frac{V_o}{V_w}+\frac{1}{K_{ow}}\right) \tag{15}$$

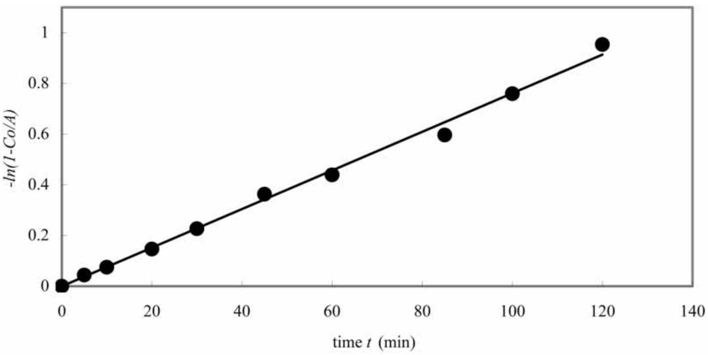

Figure 3. $-\ln(1 - C_o/A)$ vs. time t.

Constant p is obtained by plotting $-\ln(1 - C_o/A)$ vs. t. Then K_1 could be calculated from Eq. (15). The process to calculate K_1 at about 288.15 K is shown in Fig. 3.

Mean Thickness of Thin Water Film d_i

The mean thickness of the thin water film around bubble, d_i, can be approximately calculated with Eq. (1) and the following Eq. (16) as given in the literature (16).

$$\frac{V_{wk}}{V_b} = 1.5 \times 10^{-4}\, V_b^{-0.741} \tag{16}$$

Gas Holdup ε

The gas holdup, ε, was calculated by measuring the change of liquid height, Δx, with and without gas flow. The gas holdup, ε, can be thought of as a constant to simplify the simulation, since the gas flow rates in our sublation work were low.

Linear Adsorption Constant K_a

Gibbs isotherm equation is usually used to calculate the linear adsorption constant K_a in simulated aqueous solution. But it is unlucky that the wastewater was a real industrial system, and the real value of K_a could not be calculated properly from the simulated solution. The reason may be that diverse surfactants, biological materials and penicillium chrysogenum in the wastewater have a rather complex effect on the sublation, or the wastewater was inherently an emulsion, which requires further study. In order to simplify the simulation, it is a good way to treat K_a as an adjustable parameter.

Mean Bubble Radius *a* and Henry's Law Constant H_c

The mean bubble radius may be estimated by comparing the bubble with a scale located in the sublation column. The Henry's law constant of BA, H_c, has been given in the literature (19). The model parameters are summarized as shown in Table 1.

RESULTS AND DISCUSSION

The relationship between the calculated and experimental results of sublation efficiency, E_{ss}, under different gas flow rates, Q_a, is shown in Fig. 4. We can see that the calculated results are in good agreement with the experimental ones. The similar agreement of the effect of initial volume of solvent, v_{oi}, also can be seen.

Effect of Gas Flow Rate

Gas flow rate, Q_a, is a very important operation parameter in solvent sublation. Figure 4 also shows the simulation results of the effect of gas flow rate on the sublation efficiency in our sublation. At a high Q_a, we can get better sublation efficiency. The gas flow rate has a direct effect on the air-water interface area

Table 1. Model parameters

	Thermodynamics		
	$H_c = 8.29 \times 10^{-3}$ (19)	$K_a = 3.0 \times 10^{-3}\,\mathrm{cm}$	$K_{ow} = 49.7$
Kinetic	$K_1 = 5.42 \times 10^{-2}\,\mathrm{cm/min}$		
Hydrodynamic	$a = 0.03\text{–}0.04\,\mathrm{cm}$	$d_i = 12.61\text{–}8.87 \times 10^{-4}\,\mathrm{cm}$	$\varepsilon = 0.01563$
Design	$r_c = 5\,\mathrm{cm}$	$Q_a = 127\text{–}380\,\mathrm{ml/min}$	$v_{wi} = 15{,}000\,\mathrm{mL}$
Physics	$\rho_s = 0.88\,\mathrm{g/ml}$	$M_s = 0.11616\,\mathrm{g/mmol}$	

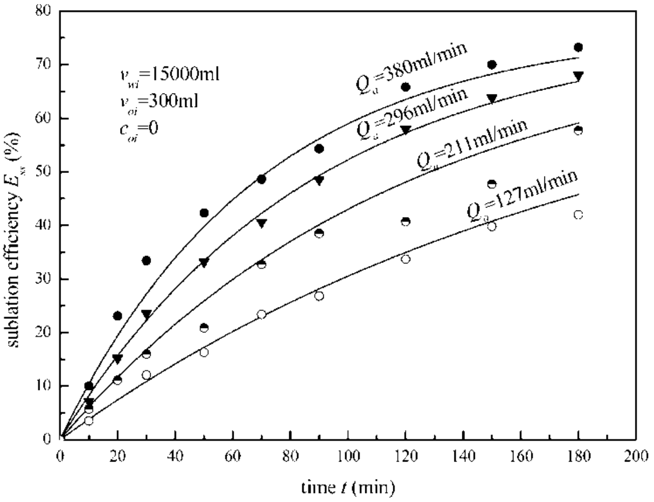

Figure 4. Comparison of experimental results with calculations under different gas flow rates. The solid lines represent simulation; points, experiments.

per unit volume of the aqueous bulk in unit time. The more area of water-gas interface, the more solute is brought into the solvent phase at the same time interval. In Eq. (3), varying rate of the concentration of BA in water, $-dC_w/dt$, is almost directly proportional to gas flow rate, Q_a. The simulation results are in agreement with the explanation. As sublation time increases, the increasing rate of the sublation efficiency decreases, since the concentration of BA in water becomes low and accordingly high in organic solvent. Under high gas flow rate, surfactants and biological materials in the wastewater would lead to unpleasant emulsification at the solvent-water interface or in solvent layer, which would aggravate back mixing of organic solvent and result in recontamination of water. Therefore, desired gas volumetric flow rate is typically 300 ml/min.

Effect of Bubble Radius

Bubble radius, a, is another important operation parameter in solvent sublation. The simulation results of the effect of bubble radius in the sublation are shown in Fig. 5. In Eq. (3), the varying rate of the concentration of BA in water, $-dC_w/dt$, is almost inversely proportional to the bubble radius, a. As the bubble becomes smaller, such as $a = 0.01$ cm, a desirable sublation efficiency could be obtained in a short period of sublation. Such result also shows that sublation efficiency under large bubbles increases less slowly than that under small ones. On the other hand, as bubble radius decreases,

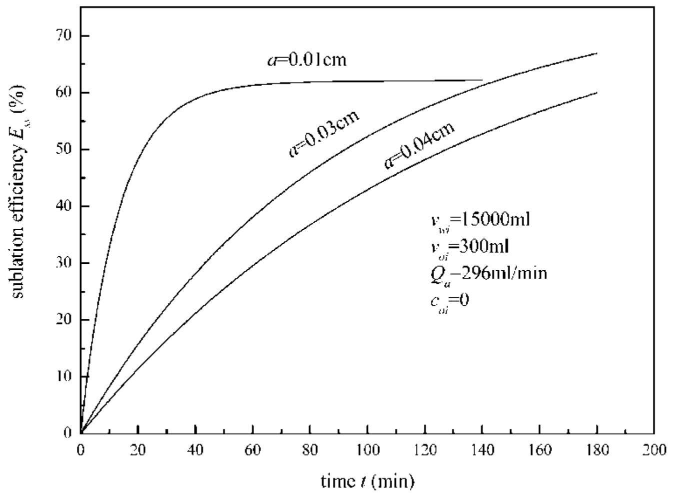

Figure 5. Effect of bubble radius, a, on sublation efficiency.

the thickness of water film d_i increases according to Eqs. (16) and (1), and then the value of the term of $3d_i/a$ increases, resulting in dragging more water into the solvent. In the process, phase partition constant of BA between water and n-nonane, K_{ow}, is small as shown in Table 1, therefore more BA would be carried back to the water because of more water in droplets under the supposition of the establishment of equilibrium between water droplets and solvent layer. It also resulted in low sublation efficiency relatively when the value of $3d_i/a$ was too large. Notice that for a bubble of radius 0.01 cm, value of d_i may be too large and unrealistic. On the whole, when the rate of mass transport of BA from water to n-nonane and the one from solvent layer to water established equilibrium, sublation efficiency, E_{ss}, would maintain almost invariable as shown in Fig. 5 when the bubble radius was 0.01 cm. When the mean bubble size is about 0.03 cm in the experimental study, the rate of increase of sublation efficiency slows down with the increase of time and approaches asymptotically its maximum value.

Effect of Initial Volume of Solvent

In solvent sublation, when the molecular diffusive transport at the water-solvent interface (characterized by K_1) is very low, the sublation efficiency, E_{ss}, is almost independent of the initial volume of solvent, v_{oi}. K_1 of BA in the sublation is relatively low ($K_1 = 9.03 \times 10^{-4}$ cm/s), so E_{ss} is almost independent of v_{oi} as shown in Fig. 6 when the initial solvent volume is more than 200 mL. It is one of the important advantages of solvent sublation over

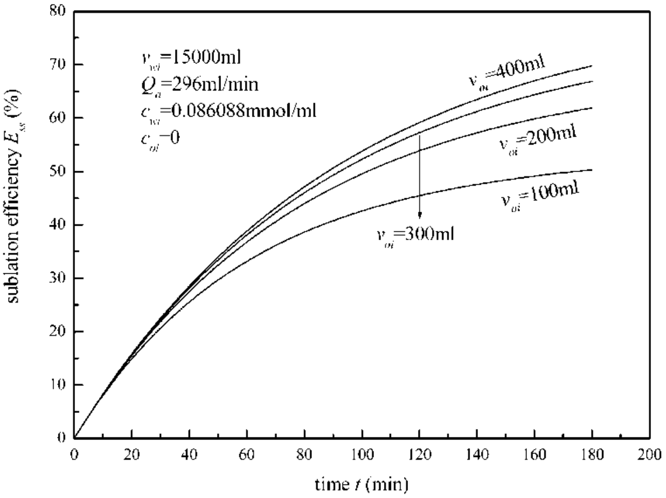

Figure 6. Effect of initial volume of solvent, v_{oi}, on sublation efficiency.

conventional liquid-liquid extraction. Figure 6 also showed that as sublation time increases, the sublation efficiency with more solvent used was a little higher, especially compared with the result when the initial solvent volume was 100 mL. The reason might be that as sublation time increases, the concentration of BA in the solvent, c_o, becomes much higher in the process of using less amount of solvent, then the reverse mass transports by molecular dispersion and dragged water may become important, resulting in a little lower sublation efficiency. So desired initial volumetric ratio of water to solvent used would be better in the range from 50 to 70.

Variation of Solvent Volume in the Sublation

An obvious result of our solvent sublation was that the solvent volume, v_o, increased with the increase of process time. It can be explained that the initial concentration of BA in the wastewater, c_{wi}, was about 1% (by weight), which was much higher than other sublation work reported in recent literatures (7). So it is very useful to predict the variation of the solvent volume during the sublation. The typical results of simulation of the variation of solvent volume are shown in Fig. 7. At the end of the sublation, the volume of solvent would increase by more than 30%. It can be completed to some extent because BA and n-nonane can be dissolved in each other. The corresponding concentrations of BA in solvent, c_o, are also shown in Fig. 8. Both results show that solvent sublation is very effective to recover BA in the wastewater of penicillin plant. Therefore we can purify BA and recycle the sublation solvent by distillation.

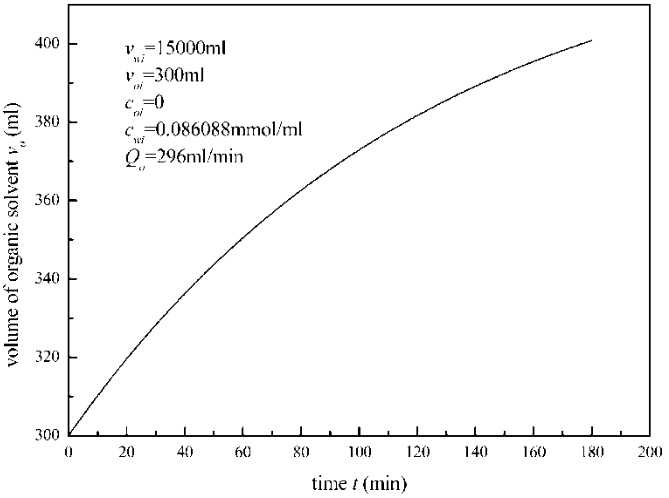

Figure 7. Variation of organic solvent, v_o, with time t.

Emission of BA to the Atmosphere

Few papers have been published to study the amount of the emission of solute to the atmosphere with simulation of solvent sublation, because the initial concentration of solute is very low. So it is a meaningful work to study such emission in our sublation. The difference between Eqs. (2) and (3) represents the amount of the emission of BA to the atmosphere. With simulation

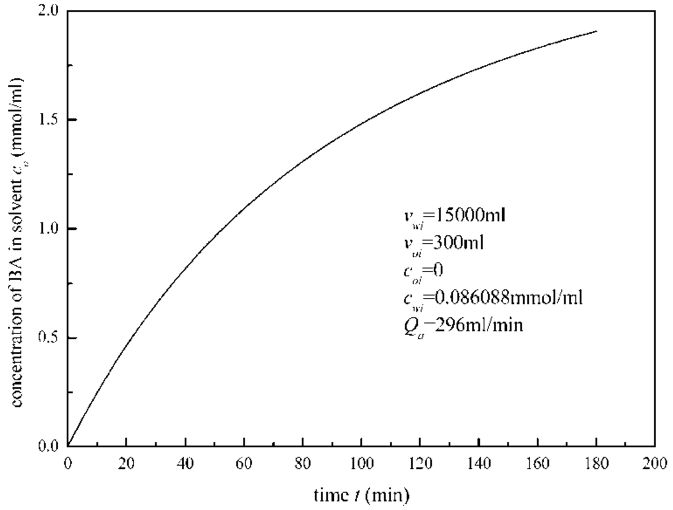

Figure 8. Variation of concentration of BA in organic solvent, c_o, with time t.

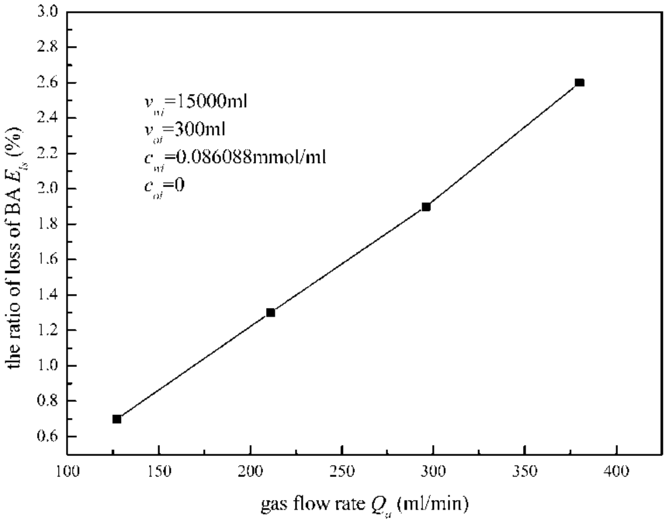

Figure 9. The ratio of loss of BA to the atmosphere at different gas flow rates.

numerical values of C_w and C_o at the end of the sublation, the loss ratio of BA emitted to the atmosphere, E_{ls}, could be calculated by Eq. (9). The calculated results under different gas flow rates, Q_a, are shown in Fig. 9. It can be seen that E_{ls}, is 2% or so and less than that without the adsorption of the solvent layer. The loss ratio is almost directly proportional to the gas flow rate, Q_a.

CONCLUSIONS

The transport mechanisms of solvent sublation for recovering butyl acetate (BA) in the wastewater during penicillin production were presented, and a mathematical model was proposed on the basis of the mechanisms proposed. Different from other models in recent literatures, it took account of the variation of the volume of organic solvent with sublation time. The model parameters of the sublation are calculated. The variations of sublation efficiency, E_{ss}, volume of solvent, v_o, and BA concentration in solvent, c_o, with sublation time are calculated and discussed. The solvent volume could increase by more than 30% in 180 min. Calculation shows that increasing the gas flow, Q_a, and decreasing the bubble size, a, could improve E_{ss} and increase the rate of the sublation respectively. The initial volume of the solvent may affect the sublation weakly when initial volumetric ratio of water to solvent used is in the range from 50 to 70. The loss ratio of BA emitted to the atmosphere, E_{ls}, is minimal, though with large gas flow rate, Q_a.

The model can be used to simulate the sublation for recovering BA in the wastewater of penicillin plant. It is a very promising trend that more of solvent sublations begin to be used in the treatment of industrial wastewater. If the organic has a large concentration in wastewater during solvent sublation for its recovery, such as wastewater from a pharmaceutical factory, the variation of the volume of solvent layer should be taken into account to predict the results and to provide parameters for the design of equipment. If the parameters of model equations are acquired correctly, the mathematic model will be solved easily. Nevertheless, further study should be carried out to improve the model to be better used in other solvent sublation.

NOMENCLATURE

a	bubble radius (cm)
A	constant in Eq. (13)
c_o	concentration of solute in solvent phase (mmol/ml)
C_o	concentration of solute based on pure solvent (mmol/ml solvent)
c_{oi}	initial solute concentration in solvent phase (mmol/ml)
C_{oi}	initial solute concentration based on pure solvent (mmol/ml solvent)
c_w	concentration of solute in aqueous phase (mmol/ml)
C_w	concentration of solute based on pure water (mmol/ml water)
c_{wi}	initial concentration of solute in aqueous phase (mmol/ml)
C_{wi}	initial solute concentration based on pure water (mmol/ml water)
d_i	thickness of water film around bubble (cm)
E_{se}	efficiency of liquid-liquid solvent extraction (%)
E_{ls}	loss ratio to the atmosphere (%)
E_{ss}	sublation efficiency (%)
H_c	Henry's law constant for solute between vapor and aqueous phase (—)
K_a	linear adsorption constant for solute between the air-water interface of bubble and aqueous phase (cm)
K_l	aqueous phase overall mass transfer coefficient (cm/min)
K_{ow}	solute partition constant between organic solvent and aqueous phases (—)
M_s	solute molecular mass (g/mmol)
p	constant in Eq. (13)
Q_a	gas flow rate (ml/min)
r_c	radius of column for sublation or laminar liquid-liquid extraction (cm)
t	sublation time (min)
V_b	volume of bubble (ml)
v_o	volume of organic solvent phase (ml)

V_o	volume of pure organic solvent (ml)
v_{oi}	initial volume of organic solvent phase (ml)
V_{oi}	initial volume of pure organic solvent (ml)
V_w	volume of pure water (ml)
v_w	volume of aqueous phase (ml)
v_{wi}	initial volume of aqueous phase (ml)
V_{wi}	initial volume of pure water (ml)
V_{wk}	volume of wake (ml)

Greek Letters

Δx	change in liquid height (cm)
ε	gas holdup (—)
ρ_s	solute density (g/ml)

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