



Solvent sublation for waste minimization in a process water stream — a pilot-scale study

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Abstract

Solvent sublation, an adsorptive bubble separation process, was carried out on a pilot scale to separate dilute concentrations (30–100 ppmw) of naphthalene from a process water stream at a temperature of 140°F. The test was conducted at the Borden Chemicals and Plastics (BCP) acetylene plant site located in Geismar, Louisiana. A carbon steel column of 6' i.d. and 17' high was constructed. White mineral oil supplied by Texaco Inc., was used as the organic solvent for solvent sublation. A special annular shear sparger was used for nitrogen gas sparging into the vessel. The process was conducted in two-phase continuous and three-phase continuous modes. The naphthalene recovery from the process water was independent of the oil flow rate, but depended on the nitrogen-to-water flow rate ratio. The release of naphthalene to the overhead gas space during the solvent sublation process was substantially less than that during conventional gas stripping. The improved performance of solvent sublation over both conventional gas stripping and solvent extraction operations was apparent. © 2001 Published by Elsevier Science B.V.

Keywords: Solvent sublation; Pilot scale; Naphthalene removal

1. Introduction

Spurred on by the passage of the Pollution Prevention Act (PPA) of 1990, chemical and petrochemical companies have moved from end-of-the-pipe pollution treatment to pollution prevention at the source. Thus, chemical process industries (CPI) are pursuing process modifications in order to minimize wastes generated at the source. Process modifications take many forms. They may involve substitution of raw materials that lead to benign by-products, or modifications or additions of unit operations, or improved operating practices.

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In both bulk and speciality chemical manufacturing industries, typically the process water generated, will contain organic intermediates at low concentrations, that have to be separated before the water is recycled or reused within the plant. Borden Chemicals and Plastics (BCP), located in Geismar, Louisiana is one such chemical company; it manufactures polyvinyl chloride, vinyl chloride monomer, acetylene, methanol, urea, ammonia, and formaldehyde, and has air separation and co-generation facilities. The refining process water in the acetylene plant refining section contacts gases in vacuum-producing steam jet ejector direct contact coolers. Thus, a need exists to separate the organic intermediates from the refining water, so that naphthalene solids build-up in the refining water coolers is reduced to a tolerable level. The organic intermediates are generated at a high temperature, and are absorbed into the process water at very dilute concentrations. Therefore, many of the traditional unit operations (gas stripping, solvent extraction and activated carbon adsorption) are either infeasible or uneconomical. Thus, BCP engineers began to look for alternative means of separations. After evaluating several options, one that appeared most promising was the so-called *solvent sublation* process [1].

Solvent sublation is an adsorptive bubble separation process that combines the effectiveness of adsorptive and vapor transport on gas bubble surfaces from water, and subsequent extraction of the material into an organic solvent (oil) floated on top of the aqueous phase. The process is carried out in a conventional bubble column modified with a solvent reservoir at the top. The process takes advantage of the volatilization into the bubbles, and the adsorption of compounds at the bubble/water interface; both mechanisms are important for hydrophobic compounds at dilute concentrations in the aqueous phase. As the bubbles exit, the aqueous phase with the contaminant, they contact an immiscible, non-volatile, non-toxic organic solvent (oil) into which the solute is extracted. The process has several advantages over both conventional gas stripping in bubble columns and solvent extraction. In conventional gas stripping, only volatile materials can be separated. In solvent sublation, both volatile and non-volatile materials can be separated from water. A unique feature of the process is that mixing and turbulence at the oil/water interface is minimal, and the dominant transport is due to gas bubbles crossing the interface at velocities that are typical of the homogeneous flow regime. The process is not equilibrium-limited at the oil/water interface, involves little turbulence, and has only a minimal dissolution of the oil into the water phase. A key advantage of the process is that all three unit operations (stripping, extraction and solvent recovery) occur in a single vessel.

The two most important mechanisms in solvent sublation are (a) mass transfer of solute to the gas/water interface of the bubble in the aqueous phase and (b) the bubble wake entrainment and boundary layer separation at the oil/water interface. The mass transfer to the bubble from water is dependent on the driving force (concentration gradient between water and gas) and the mass transfer coefficient from water to the gas bubble. The bubble wake entrainment involves the solute transport with the water droplets dragged into the organic solvent as the bubble enters it. In other words, it involves the bubble boundary layer separation within the organic solvent and can be looked upon as the solvent extraction portion of solvent sublation. The overall separation depends on both mechanisms [1].

The applications of this technology for removing a variety of organic compounds from the aqueous phase have been documented by a number of laboratory studies. A recent review of the applications is available [2]. Since the process exploits the surface active nature

of compounds at the gas bubble/water interface, many hydrophobic compounds typically encountered in industrial applications are amenable to this separation technology. Some of these are polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and chlorobenzenes (ClBz), all of which are highly hydrophobic and, therefore, have very low solubilities in the aqueous phase. Whereas most of the studies reported in the literature have been on small laboratory bench-scale columns or small pilot scale columns, no large industrial applications, either pilot or full scale, have been reported using this technology. The work reported here is the first of its kind and demonstrates the use of the technology on a real process water stream and on a large pilot scale. This effort was undertaken in order to obtain relevant information for the scale-up of the process to full scale.

2. Experimental

2.1. Description of the pilot plant

A pilot plant was constructed at BCP, Geismar, Louisiana for conducting a series of test runs. Fig. 1 is the flow sheet for the process. A slip stream from the refining process water circulation at the plant was diverted to the top of the solvent sublation column and fed at a height of 10 feet from the bottom of the column. The treated water exited from the column at the bottom and was returned to the refining process water circulation stream. Nitrogen from the process plant was introduced at the bottom of the sublation column at rates up to a maximum of 50 SCFM through a porous shear sparger (Model 8501) supplied by Mott Metallurgical Corporation, Farmington, CT. This unique sparger had an effective porous element area of 15.3 in.², had holes 2 μm in diameter, and was made of stainless steel. A pump (Burks Model T5GA4) for sparger water recirculation was provided at the bottom for the sparger with a maximum flow rate of 20 gal/min at a maximum pressure drop of 10 psi. At the top of the column, a pressure relief valve and an outlet to atmosphere for the exiting nitrogen stream was provided.

The oil was pumped into the top of the sublation reservoir from a separate tank. The oil/water interface was controlled using a robust level controller. The level controller consisted of a Level Multicap Probe (E & H Model # DCIITAS-KR-1355G-K82) and an automated valve package consisting of a 2" KTM ball valve (Model WO 401-62.3 L), and a KTM actuator provided by Iscola Inc., Baton Rouge, LA. The rod probe and capacitance level switch were provided by Fogerty Engineering, Baton Rouge, LA.

Water flow rate was measured using a Brooks flowmeter; the nitrogen flow rate was measured using a Wallace and Tiernan flowmeter, and the oil flow rate was measured using a Brooks flowmeter. Process water was delivered to the top of the sublation column in a slip stream from the large process water pumps. The oil was delivered using a pneumatic powered sidewinder pump (Model 164) supplied by Sidewinder Pumps Inc., Lafayette, LA.

The main sublation column consisted of two sections. The lower section was a carbon steel pipe (6" diameter and 11' high) with a 4" and a 6" opposing sight port on the side half-way up. The upper section consisted of a reservoir (24" in diameter and 6' high) also constructed of carbon steel. A sight glass on the reservoir allowed visual tracking of the oil/water interface. The function of the reservoir was to prevent the oil from being entrained

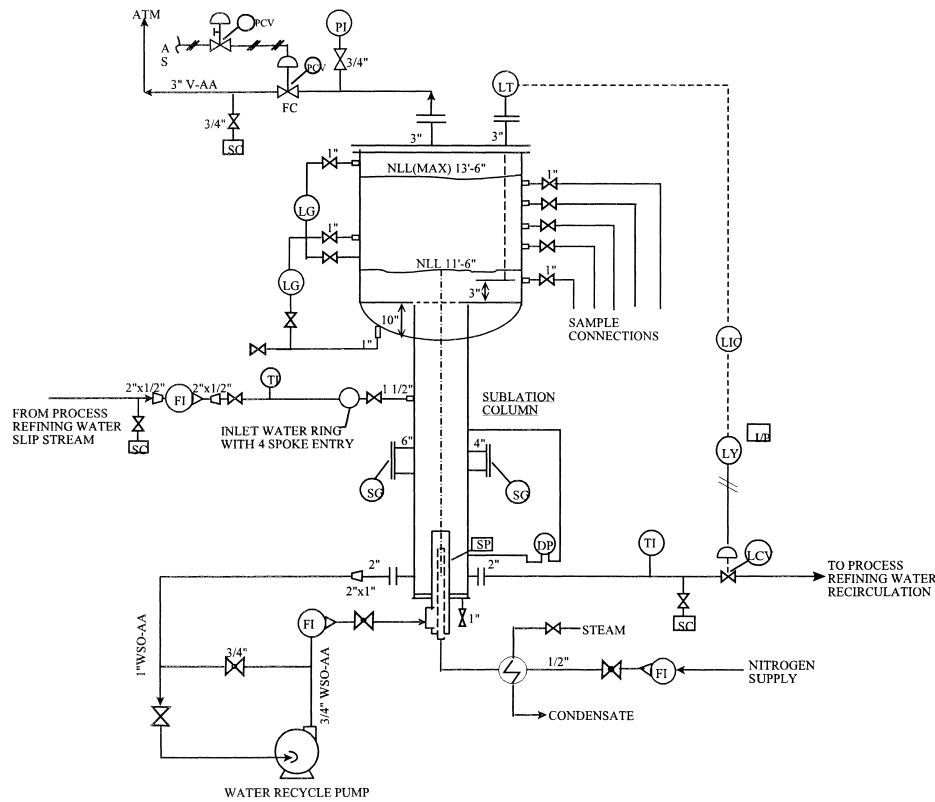


Fig. 1. Flow sheet of the pilot scale solvent sublation process: D — differential pressure indicator; FI — flow indicator; SC — speed frequency controller; SG — sight glass; SP — sparger; LIC — level indicator control; LT — level transmitter; LG — glass viewing device; LCV — level control valve; LY — level relay; PI — pressure indicator; PCV — pressure control valve; TI — temperature indicator.

and mixed throughout the column. The column protruded about 6'' into the upper reservoir to facilitate the expansion and smooth entry of the gas bubble stream through the oil/water interface. This also provided a 9'' annulus of a 6–7'' layer of stagnant water in the bottom of the reservoir which provided added stability to the floating oil layer. Since the stagnant unaerated water had a higher density than the aerated water in the main column, this acted as a buffer zone preventing the circulation and mixing of the organic solvent with the water phase. Differential pressure between the two top and bottom taps was measured using a Dwyer Capsuhelic Model 4330 DP meter.

The column was run in two different modes: (1) two-phase continuous, where the aqueous and nitrogen streams were in the continuous countercurrent mode, but with a stagnant oil layer, and (2) three-phase continuous, where all three streams (nitrogen, water and oil) were in the continuous flow mode. The nitrogen stream and water flowed countercurrent, whereas the oil flowed across the top reservoir at a low flow rate. Fig. 2 is a schematic of the two modes of operation.

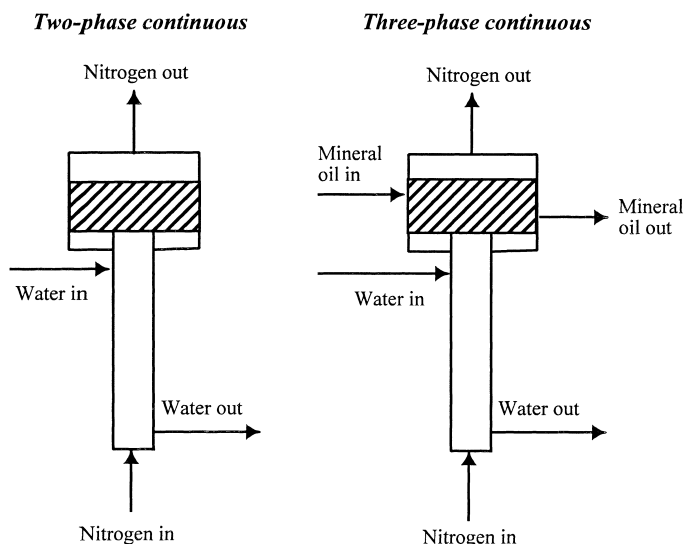


Fig. 2. Schematic of the two-phase and three-phase continuous modes of operation.

Water was first added to the proper level near the interface level probe. The organic solvent (mineral oil) was transferred to the column using a drum pump. The nitrogen supply was started to establish pressure control and set the flow of nitrogen into the sparger to 5 SCFM. Then, the refining water supply was started at 5 gal/min with the level control in automatic to obtain a steady state operation of the level controller. Thereupon, the water flow rate was adjusted to the desired value. The nitrogen flow was adjusted to the desired flow conditions. Once the experiment was over, the nitrogen was turned down to a minimum to keep liquids out of the sparger. The supply refining water flow was then stopped. The organic solvent was re-used for subsequent experiments under different water and nitrogen flow conditions.

2.2. Experimental run conditions

Typical experimental flow rates varied from 5 to 50 gal/min for the refining water slip stream. The nitrogen flow rate varied from 0.5 to 5 ACFM, whereas the sparger water flow rate was kept at approximately 3 gal/min. For the three-phase continuous experiments, the oil flow rate was maintained at 4 gal/h.

2.3. Organic solvents tested and typical refining water composition

The organic solvent used for sublation in all the experiments was white oil 22 (CAS# 8042-47-5) supplied by Texaco Lubricants Co., Inc., Houston, TX. This was a white oil blended from highly refined mineral oil. It had a flash point of 270°F, specific gravity of 0.85 and a boiling point of 503°F at atmospheric pressure. This is an oil that is used in the plant for other applications. It was chosen because it is lighter than water, essentially

Table 1
Relevant physico-chemical properties for naphthalene^a

Property	Value
Molecular weight	128
Melting point (K)	353
Boiling point (K)	491
Aqueous solubility at 298 K (ppmw)	30
Solubility in refining water at 140°F (333 K) (ppmw)	100
Solubility in white mineral oil at 333 K (ppmw)	150000
Henry's constant at 333 K ^b (dimensionless)	0.18
Partition constant between water and white mineral oil at 333 K	1500

^a All parameters obtained by Borden Chemicals and Plastics in their laboratories. 140°F is the temperature of the refining water in the sublation vessel.

^b Determined using the Clausius–Clapeyron equation using $\Delta H_{\text{Henry}} = 33 \text{ kJ/mol}$. At 298 K the Henry's constant value is 0.019.

immiscible with water, non-toxic, non-odorous, and relatively inexpensive. It also has a high boiling point and is non-reactive with the hydrocarbons in the water stream.

The refining process water stream had temperatures between 120 and 140°F. It contained traces of several organic compounds, but the one of interest in the present work was naphthalene. Typically, the naphthalene concentration varied from 30 to 100 ppm. The actual concentrations varied depending upon the day-to-day operations within the plant. The relevant physico-chemical properties of naphthalene are given in Table 1.

2.4. Analysis of samples

Feed water, exit water from the sublation unit, and the inlet and outlet oil layer containing naphthalene were analyzed using a gas chromatograph by the analytical group at the BCP site according to the procedures developed in-house. In a few selected cases the nitrogen stream exiting the top of the sublation column was also analyzed for naphthalene using gas sampling tubes.

2.5. Experimental data analysis

The experimentally determined influent and effluent water concentrations were used to obtain the overall removal of naphthalene at steady state from the refining process water stream. Efficiencies are reported as $E = 1 - (C_{\text{out}}/C_{\text{in}})$.

3. Results and discussion

3.1. Gas phase hold up and bubble size

As outlined earlier, there are two mechanisms of solute transport between the water and the organic solvent in sublation. It has been found from work on the laboratory scale that both mechanisms are dependent on the average gas bubble size and gas/water interfacial area in the reactor. These two parameters are invaluable in the design of a bubble column reactor

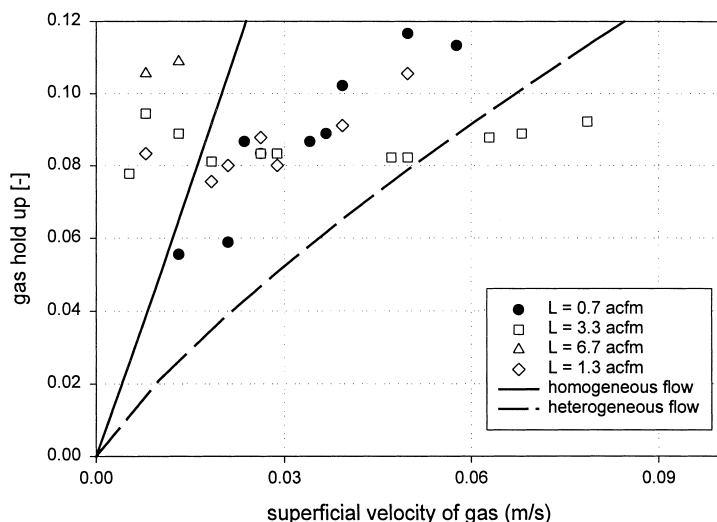


Fig. 3. Gas hold up versus superficial gas velocity for the annular shear sparger.

for any application including solvent sublation [3–5]. The gas/water interfacial area, a_v is determined primarily by the bubble size, d_b and gas phase hold up, ϵ_g within the reactor: $a_v = 6\epsilon_g/d_b$. The term ϵ_g represents the ratio of the overall gas volume to the total fluid volume at given gas and liquid flow rates. Fig. 3 shows the measured gas phase hold up in the sublation column as a function of the superficial nitrogen velocity, u_g through the column. In the low superficial gas velocity region the gas hold up varies linearly with u_g , however, at higher u_g the sensitivity of ϵ_g towards gas velocity is minimal. In general, this behavior is noted for bubble columns which operate either in the homogeneous flow regime or in the transition regime between homogeneous and heterogeneous flow. For the annular shear sparger in the homogeneous bubble flow regime, we found that gas hold up is adequately represented by $\epsilon_g = 9.33u_g^{1.03}$, if u_g is expressed in m/s [6]. At the other extreme for the heterogeneous flow regime [7] the gas hold up is given by $\epsilon_g = u_g / (0.25 + 0.9(gD_c u_g)^{1/3})$. The two equations are plotted alongside the experimental data in Fig. 3. This figure shows that in the regime of operation, <0.03 m/s in superficial nitrogen velocity, the flow was near the homogenous regime. Typical bubble diameters in the column were observed to be in the range of 2–4 mm, and are in agreement with earlier studies from our work on lab scale columns using the same annular shear sparger. Using the average bubble size of 3 mm, and the maximum value of $\epsilon_g = 0.12$ observed in the experiments, a value of a_v equal to 240 m^{-1} was calculated. This is a large value, and is more common to the heterogeneous regime [6]. Thus, the use of the annular shear sparger makes it possible to operate near the homogenous flow regime with a high gas/water interfacial area.

3.2. Removal efficiency in the two-phase continuous mode

Fig. 4 is the plot of the steady state removal of naphthalene from the refining process water as a function of the gas to liquid flow rate (G/L) ratio. Different liquid flow rates, L were

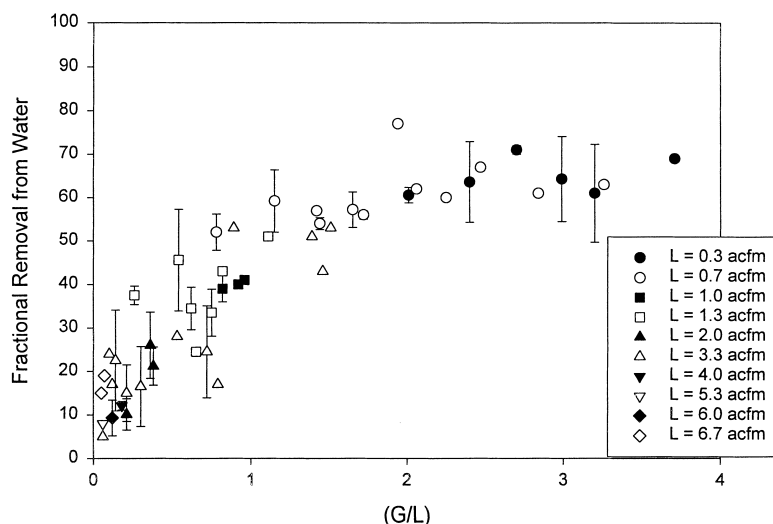


Fig. 4. Steady state fractional removals of naphthalene from process water as a function of the nitrogen-to-water flow rate in the two-phase continuous mode. The organic solvent was stagnant.

chosen and G was varied to obtain ranges of G/L ratios. In these experiments, the nitrogen and aqueous streams were in countercurrent flow. The organic solvent was stagnant and had a constant thickness of 24 in. within the upper reservoir. Maximum removal of 71% for naphthalene was obtained at a G/L value of 3.7. The removal increased linearly with G/L up to a value of this ratio of 1 and, thereafter increased only minimally with increasing G/L . The analysis of the oil layer (organic solvent) showed >99% of the naphthalene was captured in the organic solvent. This was corroborated by periodic measurements of naphthalene in the overhead gas stream. For example, in a typical run the total mass of naphthalene removed from the water was estimated as 3.42 lb, while the total mass of naphthalene lost to the overhead nitrogen gas stream was 0.0053 lb. The calculations showed that 99.84% of naphthalene was captured in the oil layer.

As the bubbles traverse the oil layer, they release their naphthalene content to the oil because of the highly favorable partitioning towards the organic solvent. The organic solvent effectively acts as an infinite sink to the organic compound. The release of the contaminant back to the water from the oil layer is very slow and is overwhelmed by the unidirectional transport by the bubbles into the organic solvent layer. Without the oil layer such a sink is not available and the removal is incomplete. In order to demonstrate this, a series of gas stripping experiments was performed without any oil floating at the top of the aqueous phase. The results of the run are shown in Table 2. At a process water flow rate, L of 0.83 ACFM and at a nitrogen flow rate, G of 4.8 ACFM, i.e. at a G/L ratio of 4.8, the percent removal of naphthalene obtained was 16.4, which when compared to the percent removal of 71 in the presence of the oil layer, clearly demonstrates the effectiveness of the organic solvent layer in enhancing the removal. Measured overhead gas concentrations of naphthalene in the conventional stripping experiments (without the oil layer) were >80 ppmv, whereas those during the sublation experiments (with the oil layer) were substantially less (<1 ppmv).

Table 2
Naphthalene removal by gas stripping without floating oil layer

L (ACFM)	G (ACFM)	G/L	Removal at steady state (%)
0.83	4.1	4.8	16.4 ± 1.6
0.96	4.1	4.3	20

During the gas stripping experiments, we also observed the presence of naphthalene crystals on the dome of the reservoir at the top of the column. As the naphthalene was removed from the water by the gas bubbles, it crystallized and plated out on the carbon steel interior of the reservoir column. In the presence of the organic solvent, this behavior was non-existent and the dome did not need any cleaning to remove naphthalene. All these observations clearly demonstrated the efficacy of the oil layer in reducing the release of naphthalene to the overhead gas space. This fact has also been demonstrated by other work in the literature on laboratory scale columns [9,10]. Concentrations in the oil layer reached upwards of 1/2 wt.% for naphthalene in some of the solvent sublimation runs. The effective capacity of the oil for naphthalene at a temperature of 140°F was determined to be 15 wt.%, and hence as long as this capacity was not exceeded the oil layer was deemed useful and need not be recharged if solvent sublimation is conducted in the two-phase continuous mode. There was, however, one drawback that was noticed when the column was operated in this mode. This involved the accumulation of a “rag” layer at the solvent–water interface, which affected the movement of gas bubbles across the interface. The “rag” layer was analyzed and found to be made up of high molecular weight polymers that are produced within the plant. With a stagnant solvent layer, this rag layer tended to affect the nature of the oil/water interface.

In the experiments conducted we observed no oil in the effluent water, indicating little or no dissolution of mineral oil in the process water. We expect this for two reasons. First and foremost, the oil is essentially immiscible with water; its solubility in water is extremely low. Secondly, if the gas flow is in the homogeneous regime, any oil droplets that might enter the water column near the oil/water interface are quickly returned to the overlying oil layer by the gas bubbles which are in continuous transit across the solvent–water interface. However, under certain unusual conditions, there was entrainment of oil in water. For example, when the water temperature at the outlet was lowered to 110–112°F, the water column appeared milky and a thin sheen of oil was visible in the outlet water samples. We also observed that when the oil was initially cold, the density difference between the aerated water and oil was small, and hence oil appeared within the water column. In another instance, a dispersant that was present in the process water caused the oil to disperse in the water. Therefore, the oil was maintained at a high temperature and dispersants in the water were avoided during the course of the pilot scale test. We believe that these are essential issues that need to be addressed during the scale-up of the process.

3.3. Removal efficiency in the three-phase continuous mode

Table 3 gives the steady state naphthalene removal efficiency in the three-phase continuous mode. Different values of aqueous flow rate, L , oil flow rate, L_o and gas flow rate, G were chosen for the experiments. Three different L values (5, 10 and 15 gal/min) were chosen.

Table 3
Steady state fractional removals of naphthalene in the three-phase continuous mode^a

L (gal/min)	L_o (gal/h)	G (ACFM)	G/L	F_R
5 (=0.7 ACFM)	0.2	1.5	2.1	51
	1.0	1.4	2.0	55
	4.1	1.5	2.1	52 ± 7
	7.1	1.4	2.0	52 ± 2
10 (=1.3 ACFM)	0.2	1.5	1.1	34 ± 2
	4.1	1.5	1.1	38 ± 5
15 (=2.0 ACFM)	2.0	1.2	0.57	26 ± 4
	4.1	1.1	0.54	29 ± 1

^a G/L is the volumetric ratio, where both G and L are expressed in ACFM. Also note that whereas L is in gal/min, L_o is in gal/h.

At a constant G value of 1.5 ACFM and a constant L_o of 4.1 gal/h, the fractional removal of naphthalene decreased from 52 ± 7 at an L value of 5 gal/min to 29 ± 1 at an L value of 15 gal/min. The same decreasing trend of F_R with increasing L at a G of 1.5 ACFM and L_o of 0.2 gal/h was also observed. However, at a constant G/L value of 2.0, and a constant L value of 5 gal/min, increasing L_o from 0.2 to 7.1 gal/h had no effect on the fractional removal. The same trend was also observed at L values of 10 and 15 gal/min. Note also that the recoveries for the three-phase continuous mode are identical to those obtained in the two-phase continuous mode at similar G/L values (see Fig. 4). This indicates that the efficiency of solvent sublation is independent of the mineral oil flow rate as long as the concentrations of the solute do not approach saturation. This is not a surprising result and has been observed in other work involving solvent sublation of polynuclear aromatic hydrocarbons using small laboratory scale equipment [8]. It has been shown earlier that for compounds with $K_{ow} > 700$, the solvent flow rate does not influence the efficiency of solvent sublation [2,6]. In fact, since most hydrophobic organic compounds possess large partition coefficients, the amount of solvent needed for sublation is only that which ensures a steady state. Herein lies an important advantage for solvent sublation over conventional solvent extraction.

Whether a stagnant oil layer or a flowing oil layer is more appropriate depends to a large extent on the subsequent separation process for naphthalene from the oil layer. Typically, since the solvent requirement is very small, the equipment for solute recovery is a small unit. If one runs the solvent recovery unit as a batch operation, then the solvent can be either flowing at a very low rate or even stagnant in the sublation unit. When enough solvent is collected, the solvent recovery unit can be started up. These aspects are important in the design of a full scale unit for solvent sublation.

4. Conclusions

Solvent sublation, an adsorptive bubble separation process, was carried out on a pilot scale to separate dilute concentrations (30–100 ppmw) of naphthalene from a process water stream at a temperature of 140°F. The test was conducted at the Borden Chemicals and Plastics

(BCP) acetylene plant site located in Geismar, Louisiana. A carbon steel column of 6' i.d. and 17' high was constructed. White mineral oil supplied by Texaco Inc., was used as the organic solvent for solvent sublation. A special annular shear sparger supplied by Mott Metallurgical Corporation was used for nitrogen gas sparging into the vessel. The process was conducted in two-phase continuous and three-phase continuous modes. The two-phase continuous operation involved continuous countercurrent flow of water and nitrogen but a stagnant mineral oil layer, while the three-phase continuous mode involved all three streams in continuous flow. Various nitrogen-to-water flow rate ratios were investigated for both modes of operation. In the three-phase continuous mode, a removal efficiency of $55 \pm 5\%$ was achieved with a small nitrogen-to-water flow rate ratio (G/L) of 2.3 and a small oil-to-water flow rate ratio (L_o/L) of 0.013. The naphthalene recovery from the process water was independent of the oil flow rate, but depended on the ratio G/L . An efficiency of 71% at a G/L ratio of 3.7 was obtained in the two-phase continuous mode. The target efficiency of naphthalene recovery from the process water stream was $\geq 50\%$ for the scale-up of the process. The release of naphthalene to the overhead gas space during the solvent sublation process was substantially less than that during conventional gas stripping. The improved performance of solvent sublation over both conventional gas stripping and solvent extraction operations were apparent.

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