Recovery of isopropyl alcohol from waste solvent of a semiconductor plant

Sheng H. Lin*, Chuen S. Wang

Department of Chemical Engineering, Yuan Ze University, Chungli 320, Taiwan, ROC

Abstract

An important waste solvent generated in the semiconductor manufacturing process was characterized by high isopropyl alcohol (IPA) concentration over 65%, other organic pollutants and strong color. Because of these characteristics, IPA recovery was deemed as a logic choice for tackling this waste solvent. In the present work, an integrated method consisting of air stripping in conjunction with condensation and packed activated carbon fiber (ACF) adsorption for dealing with this waste solvent. The air stripping with proper stripping temperature control was employed to remove IPA from the waste solvent and the IPA vapor in the gas mixture was condensed out in a side condenser. The residual IPA remaining in the gas mixture exiting the side condenser was efficiently removed in a packed ACF column. The air stripping with condensation was able to recover up to 93% of total IPA in the initial waste solvent. The residual IPA in the gas mixture, representing less than 3% of the initial IPA, was efficiently captured in the packed ACF column. Experimental tests were conducted to examine the performances of each unit and to identify the optimum operating conditions. Theoretical modeling of the experimental IPA breakthrough curves was also undertaken using a macroscopic model. The verified breakthrough model significantly facilitates the adsorption column design. The recovered IPA was found to be of high purity and could be considered for reuse.
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1. Introduction

Isopropyl alcohol (IPA) is an important solvent widely used as a cleaning and dehydrating agent in the electronic and precision machinery industries. In a semiconductor manufacturing process, IPA is used extensively in various stages of wafer surface washing and cleaning [1–4]. For a new generation semiconductor plant of 0.13 μm or smaller line width, over a thousand ton of high-purity IPA per year is consumed and the IPA after washing and cleaning ends up as waste solvent.

To tackle this waste solvent, incineration might be employed because of sufficient amount of organic materials it contains. However, incineration would lead to loss of recoverable resource (IPA). Due to its relatively low normal boiling point (82.4 °C), IPA can be separated from the waste solvent by distillation [5] or pervaporation [6,7]. Distillation is widely utilized in the chemical or petrochem-

ical industrial processes for separation and purification of volatile organic compounds. Although effective, it is an energy intensive and relatively costly process. Hence it is not generally considered as an appropriate technique for IPA recovery from the waste solvent. For the past decade, pervaporation has been becoming a popular method for dehydration using hydrophilic membrane. In spite of being less energy consuming, it is a complex process and is not easy to operate. Therefore, these methods are not ideal for effective IPA recovery from the waste solvent.

The objective of this study is to utilize air stripping in conjunction with condensation and activated carbon fiber (ACF) adsorption for IPA recovery. Air stripping has been recognized as a good method for ammonia stripping or removal of odorous gases and volatile organic compounds from aqueous solution [8]. In the present work, air stripping was adopted to remove IPA from the waste solvent. Although this technique is not as sophisticated as distillation, its apparatus is relatively simple, low cost and easy to operate. The IPA vapor in the gas mixture from the air stripping column was condensed in a water-cooled side condenser. The residual IPA in the gas mixture exiting the condenser

* Corresponding author. Fax: +886-3-455-9373.
E-mail address: ceshlin@saturn.yzu.edu.tw (S.H. Lin).

was finally adsorbed in a packed ACF column. Experimental tests were conducted to examine the performances of the air stripping system and the ACF adsorption column and to identify the optimum conditions of these units. The combined system of air stripping and ACF adsoption proposed here can offer an attractive alternative for IPA recovery.

2. Materials and methods

The raw waste solvent samples were obtained from a large semiconductor manufacturer in northern Taiwan. According to the information supplied by the manufacture, the waste solvent contained, in addition to IPA, many proprietary chemical compounds employed in the manufacturing process, as will be elaborated later. Hence, the quality parameters of the waste solvent were broadly quantified in terms of the chemical oxygen demand (COD), suspended solids (SS) and conductivity that were measured using the standard methods [9]. The color of the waste solvent was determined in terms of the American Dye Manufacturer Institute or ADMI using a GBC 916 UV-Vis spectrophotometer (GBC Scientific Equipment Ltd., Victoria, Australia) [9]. Its IPA concentration was measured using a HP 5890 II gas chromatograph (Hewlett-Packard Corp., Denver, CO, USA) equipped with a flame ion detector (FID) and a Restek RTX-5 capillary column (0.53 mm i.d. and 30 m long).

For IPA removal from the waste solvent, a packed stripping column was employed, as shown in Fig. 1. The apparatus consisted of a pyrex column of 6 cm i.d. and 100 cm long. The column was randomly packed with ceramic Raschig rings (8 mm o.d. and 10 mm long) to a height of 45 cm. The Raschig ring packing served to improve the liquid/gas contact during the air stripping period and to facilitate the IPA
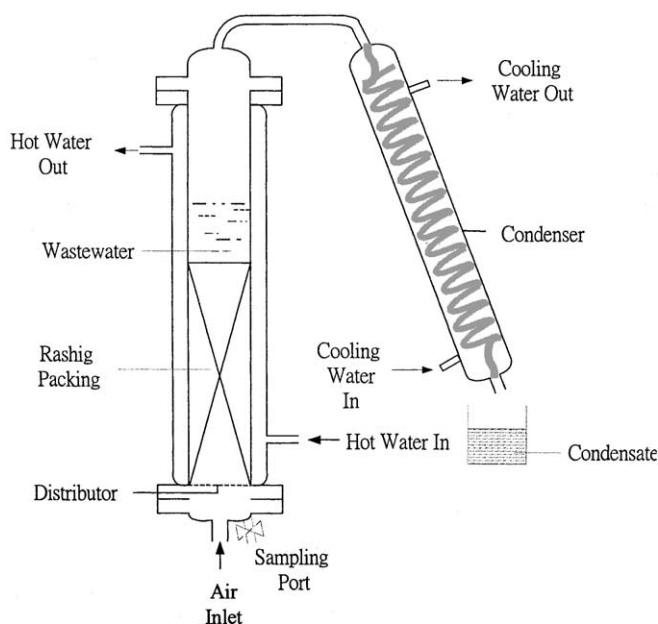

Fig. 1. Schematic diagram of air stripping with side condensation.

removal. The void of the packed column as determined by the water displacement method was 0.48. The pyrex column was equipped with an external water jacket for temperature control and during the test runs, the desired column temperature chosen between 40 and 70 °C was maintained by a constant temperature bath to an accuracy of ± 2 °C. Choice of 70 °C maximum air stripping temperature was made due to low normal boiling point of IPA (82.4 °C) and to avoid excessive energy consumption. A side condenser with an effective length of 50 cm was attached to the packed stripping column. During a test run, cooling water at a desired temperature between −5 and −15 °C was circulated through the condenser to condense out the IPA vapor in the gas mixture from the stripping column.

At the beginning of a test run, 1 l of the waste solvent was placed in the stripping column. The waste solvent reached a height of 55 cm in the stripping column that was 10 cm above the packing. The waste solvent was maintained at a desired constant temperature (between 40 and 70 °C) as controlled by a constant water bath. It usually took approximately 20–40 min for the waste solvent to reach a steady operating temperature. Air was then let in through the bottom gas distributor. The air flow rate was selected within the range between 1 and 3 l/min. The gas mixture leaving the stripping column entered the side condenser that was maintained at a desired temperature chosen between 5 and −15 °C by the cooling water and the condensate was collected. During a test run, samples were taken periodically from the bottom sampling port of the stripping column and the collection bottle of the side condenser for IPA measurements using the HP 5890II GC. The amount of condensate was also registered. An air stripping run was terminated when hardly any condensate came out of the condenser and this stripping time was usually reached in no more than 4 h except for a few cases with low stripping temperature at 40 °C or below. Note that the air stripping was conducted in batch mode for convenience of determining appropriate air flow rate, operating temperature and stripping time for optimum stripping performances.

Complete IPA removal from the gas mixture in the present simple side condenser was not possible. A residual IPA concentration up to 40 mg/l was detected in the gas mixture exiting the side condenser. To remove the residual IPA from the gas mixture, a packed ACF column was adopted in the present study. The adsorption apparatus, as shown in Fig. 2, primarily consisted of a pyrex column that was 2.08 cm in i.d. and 30 cm high. The IPA saturator containing 500 ml of GR grade IPA was employed for controlling the column inlet IPA concentration by properly adjusting the fractional air flow rate through the saturator. The total air flow rate was chosen between 1 and 3 l/min. The inlet IPA concentrations of gas mixture entering the stripping column for all test runs were kept below 40 mg/l in line with that in the gas mixture exiting the condenser of the air stripping apparatus. The activated carbon fiber employed was obtained from Neolite Technical Co., Taiwan. It was made of
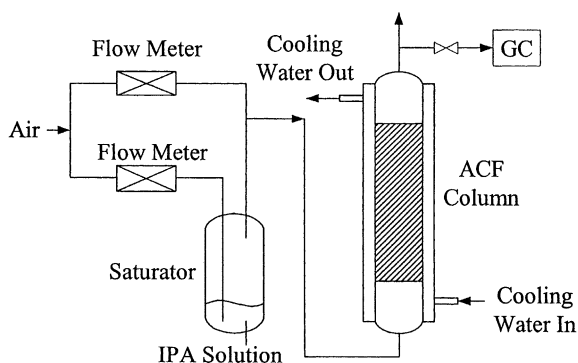
Fig. 2. Schematic diagram of packed activated carbon fiber adsorption of IPA.

polyacrylonitrile (PAN). Using a Micromeritics porosimeter (model ASAP 2000, Micromeritics Instrument Co., Norcros, GA, USA), the PAN-based ACF was found to have a BET surface area of 1200 m²/g, a pore volume of 0.55 ml/g and an average pore size of 7.5 Å. The ACF was cut into 1 cm square and 6 g of the ACF squares were packed in the adsorption column. In all test runs of column adsorption, a constant column temperature of 25 °C was maintained. By properly varying the fractional air flow rate through the IPA saturator, the inlet IPA concentration to the packed column was controlled at 13.5, 22.1 and 31.2 mg/l. A test run started with a desired air flow rate and inlet IPA concentration. The outlet IPA concentration of the gas mixture from the adsorption column was periodically measured by the HP 5890II GC. A test run was discontinued when the exit IPA concentration became close to the inlet one. The registered exit IPA concentration data were employed to establish the breakthrough characteristics of the IPA adsorption.

3. Discussion of results

3.1. Characteristics of waste solvent

The original solvent employed in the semiconductor manufacturing contained only IPA and was clear aqueous solution. However, the waste solvent samples, as obtained from a large semiconductor plant, were contaminated with organic and inorganic compounds. Their contents were rather complex because of unknown organic and inorganic components, in addition to IPA, in these solvents. According to the information supplied by the technical personnel of the semiconductor plant, the waste solvents could have small amounts of surfactants, photoresists (consisting of complex polymers, additives, sensitizers, etc.), developing agents (tetra-methyl ammonium hydroxide, mono-ethanol amine, glycol ether, etc.), stripping agents (2-propanol amine, diethylene glycol mono-methyl ether, etc.), cleaning agents (N-methyl-2-pyrrolidinone, etc.) and other organics (methanol, alkylene glycol, dyes, etc.). Over 1 year period of the present study, the raw waste solvent samples had a

pH of 9.2 ± 0.2 and a dark orange color with an ADMI of 10,773 ± 640, indicating the presence of dissolved, colored organic compounds. The samples' COD exceeded 1,000,000 mg/l and SS and conductivity were below 10 mg/l and 65 ± 21 μm h/cm, respectively. The IPA concentration was found to be quite high at 675.4 ± 28.7 g/l, as anticipated. Such a high IPA concentration prompted the adoption of air stripping and ACF adsorption for IPA recovery.

3.2. IPA recovery by air stripping

For optimum operation of the IPA recovery system, the stripping temperature, air flow rate and stripping time need to be properly identified. Fig. 3 displays the effect of stripping temperature on the IPA recovery during the stripping period. The IPA recovery is defined as

$$\text{Recovery} = \frac{W}{W_0} \qquad (1)$$

in which that W and W_0 are the amounts of IPA in the accumulated condensate at time t and in the initial waste solvent, respectively. Fig. 3 reveals that at 70 °C, the IPA recovery increases rapidly and reaches a maximum in 153.1 min. The rapid increase of the IPA recovery is proportionally slowed down as the stripping temperature decreases from 70 to 60 and 50 °C. At 40 °C, the IPA recovery was considerably reduced and it took 2265 min to reach its maximum. The maximum IPA recovery and the corresponding stripping time to reach this maximum are depicted in Fig. 4 as a function of the stripping temperature. It is apparent in this figure that for 40 °C, it takes rather long (2265 min) to reach a maximum IPA recovery of 62.1%. As the stripping temperature is raised to 50 °C, the stripping time and the maximum IPA recovery are drastically improved to 384.4 min and 92.4%, respectively. As the stripping temperature is further increased
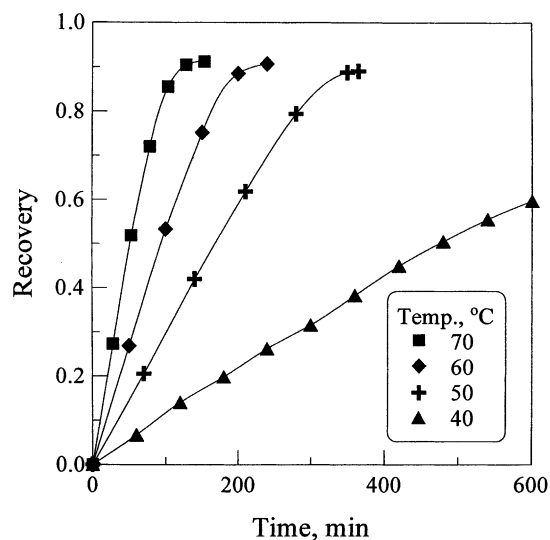

Fig. 3. IPA recovery as a function of time for various air stripping temperatures with 697 g/l initial IPA, 1.5 l/min air flow rate and −10 °C condenser temperature.
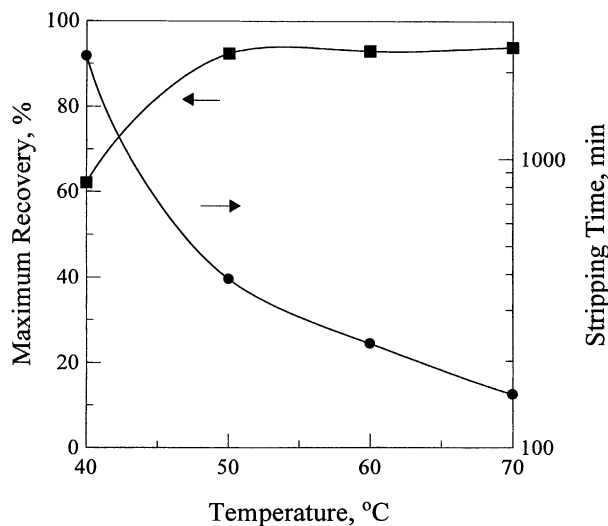
Fig. 4. Maximum IPA recovery and the corresponding stripping time required as a function of stripping temperature with 697 g/l initial IPA, 1.6 l/min air flow rate and −10 °C condenser temperature.

Fig. 5. IPA recovery as a function of time for various air flow rates with 697 g/l initial IPA, 60 °C stripping temperature and −10 °C condenser temperature.

to 70 °C, there is only a small improvement in the maximum IPA recovery from 92.4 to 93.9%, but the stripping time to reach the maximum recovery is more than cut in half to 153.1 min from 384.4 min. High stripping temperature of 60 or 70 °C has the advantage of short stripping time, but it has the disadvantage of high energy consumption. For the case shown in Fig. 4, an increase in the stripping temperature from 50 to 60 °C represents an increase in energy consumption by about 20%, but the stripping time is reduced by about 40% from 384.4 to 229.6 min. Hence, tradeoff between these two contradictory factors must be carefully evaluated in order to arrive at a proper stripping temperature.

The effects of air flow rate on the IPA recovery and on the maximum IPA recovery and the stripping time to reach the maximum recovery are demonstrated in Figs. 5 and 6, respectively, for a fixed stripping temperature of 60 °C. Both figures clearly show that the effect of air flow rate on the maximum recovery is rather small, varying from 92.6% for 1 l/min to 93.4% for 2.5 l/min. However, its effect on the stripping time to reach the maximum recovery is still significant, although not drastic as that of stripping temperature. In spite of the improved stripping time, a high air flow rate may not necessarily be a good choice for the air stripping operation. It is due to that the IPA condensation in the side condenser will be adversely affected by an increase in the air flow rate because of reduced residence time. Furthermore, a high air flow rate would entail a larger air pumping facility also. This will also negatively impact the performance of the activated carbon fiber adsorption column. In view of this, an air flow rate of 1.5 l/min may be considered as sufficient for the present stripping operation.

It is noted that the gas mixture from the stripping column contained primarily air and IPA and water vapor. Thermodynamic principle dictates that a side condenser employed in the present work would be inadequate to completely remove
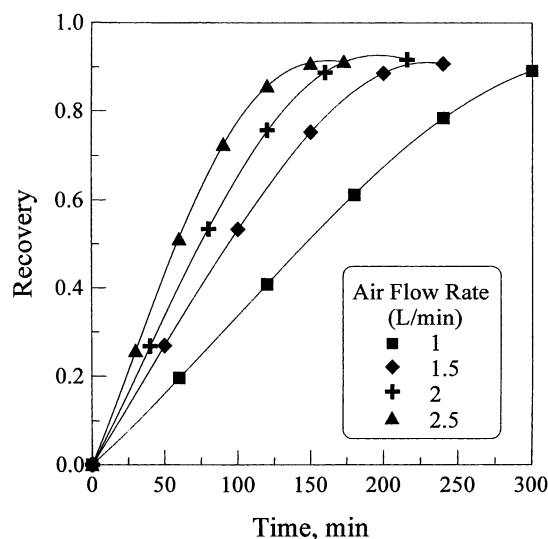
IPA and water vapor from the gas mixture unless a cryogenic condensation system is utilized. However, it would be of interest to know to what extent the condenser temperature affects the IPA recovery. Fig. 7 demonstrates the effect of this condenser temperature on the maximum IPA recovery and the stripping time to reach the maximum recovery. A decrease in the condenser temperature from 5 to −15 °C increases the maximum IPA recovery from 85.3 to 94.9% and shortens the stripping time from 234.1 to 227.3 min. The improvements in both the maximum IPA recovery and the stripping temperature may not be sufficient to compensate for the increased energy consumption. Hence, considering the overall energy requirement, a condenser temperature of
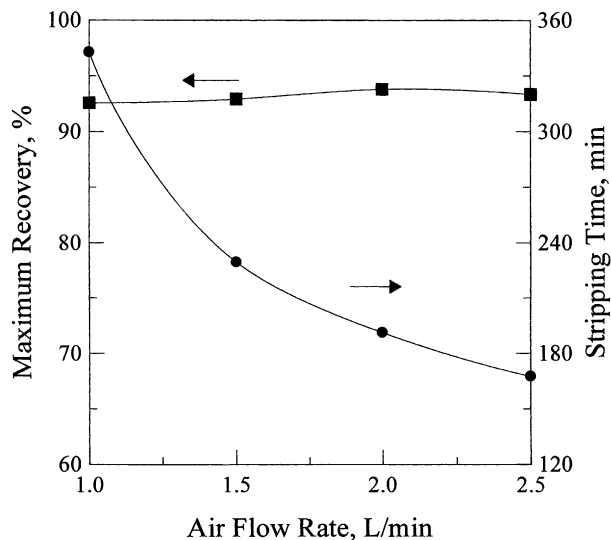

Fig. 6. Maximum IPA recovery and the corresponding stripping time required as a function of air flow rate with 697 g/l initial IPA, 60 °C stripping temperature and −10 °C condenser temperature.
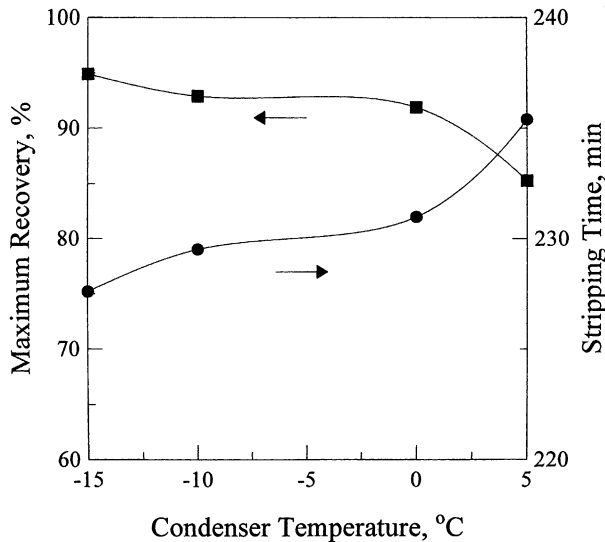
Fig. 7. Effect of condenser temperature on the maximum IPA recovery and the corresponding stripping time required with 697 g/l initial IPA, 60 °C stripping temperature and 1.6 l/min air flow rate and 60 °C stripping temperature.

−10 °C or higher would be adequate for the present IPA stripping operation.

3.3. IPA adsorption in the packed ACF column

The results of air stripping, as shown in the above figures, clearly indicate that the present air stripping system equipped with a side condenser achieve a maximum IPA recovery of close to 93%. Part of the un-recovered IPA remained in the aqueous solution in the stripping column and the rest left the side condenser in the gas mixture. In the experimental tests, an IPA concentration up to 40 mg/l was detected in the gas mixture leaving the side condenser and this IPA accounted for up to 3% of the original IPA amount in the waste solvent. Removal of this IPA serves to recover the resource and also to avoid any negative atmospheric environmental impact. These objectives can be fulfilled by IPA adsorption in a packed ACF column.

The performances of the packed-bed IPA adsorption are governed by a number of characteristic operating and system parameters [10]. An important process performance parameter of the IPA adsorption is the exit column IPA concentration. Theoretical prediction of this exit concentration is not easy. Usually it involves rigorous solution of a set of complex nonlinear partial differential equations governing the packed-bed fluid flow and mass transfer by sophisticated numerical schemes and with proper identification of a number of system parameters. This approach is much complicated. As an alternative, a macroscopic version was developed from the original microscopic model by Thomas [11] and Hutchins [12] for describing the solute breakthrough behavior in a column adsorber. The advantages of this macroscopic model are its simplicity and reasonable accuracy in predict-

ing the breakthrough curves (times) under various operating conditions. The macroscopic model is represented by

$$\ln\left(\frac{C}{C_0 - C}\right) = k_1 C_0 \left(t - \frac{Q_e M}{C_0 F}\right) \quad (2)$$

in which C is the exit IPA concentration of the stripping column at time t, C_0 the inlet IPA concentration, k_1 the rate constant of adsorption, Q_e the equilibrium solid-phase (ACF) IPA concentration, M the mass of ACF and F the gas flow rate. According to Eq. (2), the left-hand quantity, $\ln[C/(C_0 - C)]$ is a linear function of time (t) for a given set of system and operating conditions. Eq. (2) is, in fact, the same as the simplified logistic function that describes the biological or population growth and distribution [13]:

$$\ln\left(\frac{C}{C_0 - C}\right) = k(t - t_0) \quad (3)$$

Comparison of Eqs. (2) and (3) reveals that $k = k_1 C_0$ and $t_0 = Q_e M/C_0 F$. In many practical cases, the relation between $\ln[C/(C_0 - C)]$ and t may not be linear. For these instances, a general logistic function [13], as represented by the following equation, is needed:

$$\ln\left(\frac{C}{C_0 - C}\right) = a_0 + a_1 t + a_2 t^2 + \cdots + a_n t^n \quad (4)$$

where a_0, a_1, a_2, ..., a_n are the constant parameters. The number of polynomial term required on the right-hand side of Eq. (4) for a particular case of IPA adsorption and the corresponding constant parameters can be determined by nonlinear curve fitting of the above equation to the observed data.

To determine the constant parameters of the above breakthrough model, plots of $\ln[C/(C_0 - C)]$ versus t using the observed data are demonstrated in Figs. 8 and 9 for different inlet IPA concentrations and air flow rates, respectively. It is apparent in these figures that linear relationship
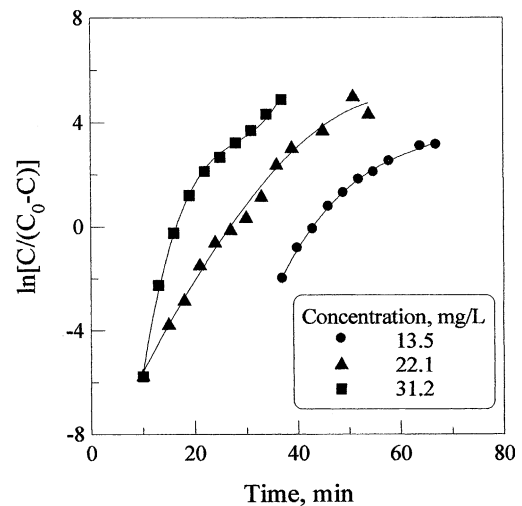

Fig. 8. Plots of $\ln[C/(C_0 - C)]$ vs. time (*t*) of IPA adsorption for various inlet IPA concentrations with 1.6 l/min air flow rate and 24 °C adsorption temperature.
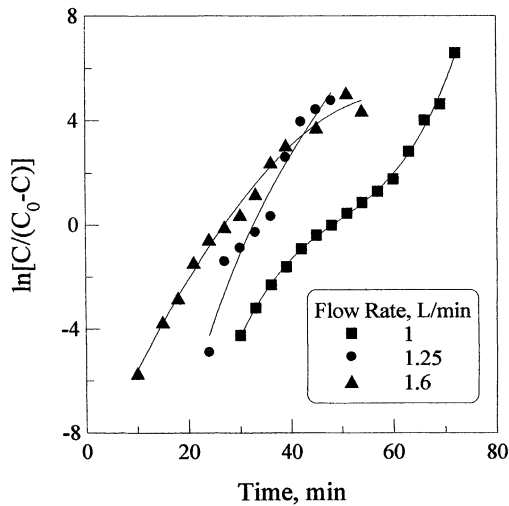
Fig. 9. Plots of $\ln[C/(C_0 - C)]$ vs. time (t) of IPA adsorption for various air flow rates with 22 mg/l inlet IPA concentration and 24 °C adsorption temperature.
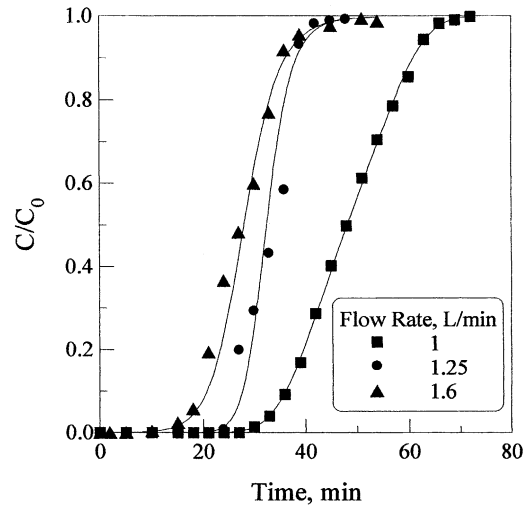

Fig. 10. Comparison of predicted (solid lines) and observed (solid symbols) IPA adsorption breakthrough curves for various inlet IPA concentrations with 1.6 l/min air flow rate and 24 °C adsorption temperature.

of $\ln[C/(C_0 - C)]$ versus t, as represented by Eq. (2) or (3), is inadequate for the IPA adsorption by ACF. Instead, a third-order polynomial of Eq. (4) represents the relation reasonably well. The constant parameters obtained from the model fit of these two figures are listed in Table 1. Using the estimated parameters, the breakthrough curves for different inlet IPA concentrations and air flow rates were reconstructed for the IPA adsorption. Figs. 10 and 11 compare the theoretical and observed breakthrough curves for different inlet IPA concentrations and gas flow rates, respectively. The model predictions compare fairly well with the observed data. Therefore, Eq. (4) can be adopted for representing the IPA adsorption by ACF.

A practical application of the experimental breakthrough curve is the determination of the breakthrough time. The breakthrough time can help establish the optimum operating conditions of the IPA adsorption process. Unfortunately, accurate estimation of the breakthrough time from the experimental curves is not easy, especially at low breakthrough IPA concentration. This difficulty can be resolved with Eq. (4). Currently, there is no official IPA discharge standard in Taiwan. In the present work, 5% of the inlet IPA concentration was selected as the breakthrough point

while the equilibrium (saturation) IPA adsorption point was reached when $C/C_0 = 1$. Based on these definitions, the breakthrough adsorption time can be readily calculated from Eq. (4) and 24 h is chosen as the equilibrium adsorption time. The breakthrough and equilibrium IPA adsorption capacities of ACF are then computed in terms of different inlet IPA concentrations and gas flow rates from the following equation:

$$\text{IPA adsorption capacity} = \frac{FC_{\text{eff}}}{M} \int_0^{t_{\text{eff}}} \left(1 - \frac{C}{C_0} \right) dt \quad (5)$$

in which t_{eff} is the time to reach the IPA breakthrough or saturation point and C_{eff} the IPA concentration corresponding to t_{eff}. The breakthrough and equilibrium IPA adsorption capacities were computed from Eq. (5) using the breakthrough
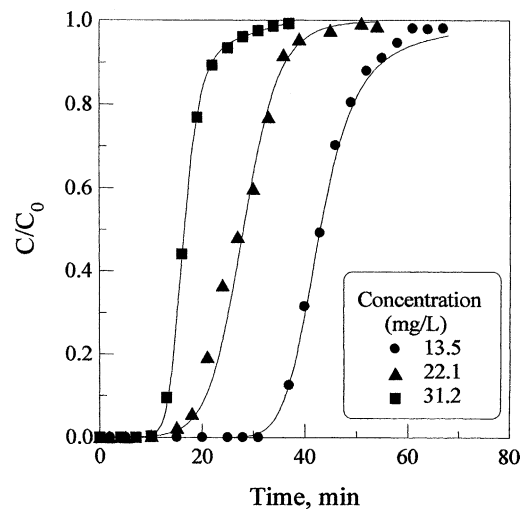

Fig. 11. Comparison of predicted (solid lines) and observed (solid symbols) IPA adsorption breakthrough curves for or various air flow rates with 22 mg/l inlet IPA concentration and 24 °C adsorption temperature.

Table 1
Model parameters of breakthrough curves

	a_0	a_1	a_2	a_3
Inlet IPA concentration (mg/l)				
13.4	−36.38	1.64×10^{-0}	2.36×10^{-2}	1.18×10^{-4}
22.1	−9.61	4.12×10^{-1}	-1.99×10^{-3}	-1.70×10^{-6}
31.2	−24.62	2.70×10^{-0}	-9.09×10^{-2}	1.06×10^{-3}
Gas flow rates (l/min)				
1	−37.61	2.03×10^{-0}	-3.87×10^{-2}	2.63×10^{-4}
1.25	−28.52	1.53×10^{-0}	-2.60×10^{-2}	1.81×10^{-4}
1.6	−9.61	4.12×10^{-1}	-1.99×10^{-3}	-1.70×10^{-6}

Table 2
Breakthrough and equilibrium IPA adsorption capacities

	Breakthrough time (min)	Breakthrough capacity (mg/g)	Equilibrium capacity (mg/g)
Inlet IPA concentration(mg/l)			
13.4	34.5	127.7	0.173.7
22.1	18.4	−112.4	0.158.1
31.2	112.5	1107.0	0.146.9
Gas flow rates (l/min)			
1	27.8	172.0	0.298.5
1.25	26.3	−162.3	0.233.1
1.6	118.4	1112.4	0.158.1

time and the equilibrium IPA adsorption time and are listed in Table 2. This table reveals that the ratio of breakthrough to equilibrium IPA adsorption capacities varies within a narrow range between 0.55 and 0.75 for all inlet IPA concentrations and gas flow rates.

Figs. 10 and 11 demonstrated the effectiveness of IPA adsorption by ACF. However, for continuous operation of the IPA adsorption system, regeneration of exhausted ACF plays a crucial role due to the need of repeated use of ACF. Regeneration of the exhausted ACF was achieved by thermal regeneration. Preliminary test runs revealed that heating of the exhausted ACF column at 150 °C for 60 min yields good results. Fig. 12 demonstrates the breakthrough curves for the virgin ACF and repeated runs using regenerated ACF. The breakthrough time calculated on the basis of 5% of inlet IPA concentration decreased slightly from 18.1 min for virgin ACF to 17.6 min for the third repeated run. In view of the simplicity of thermal regeneration employed here, the decrease in the breakthrough time was considered acceptable for practical purposes.
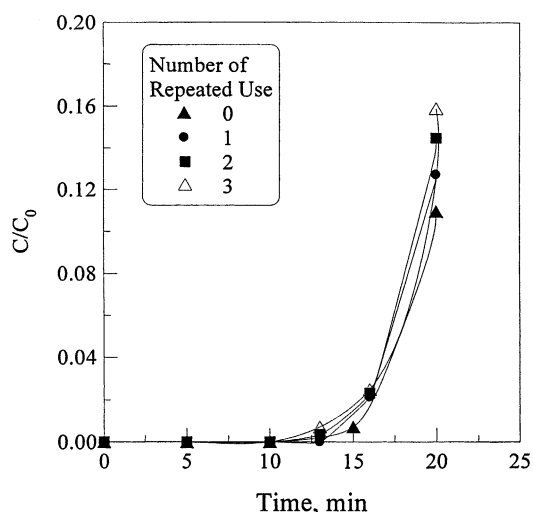

Fig. 12. IPA adsorption breakthrough curves of virgin ACF and these thermally regenerated at 150 °C for 60 min and with IPA saturation operation using 22 mg/l inlet IPA concentration, 1.6 l/min air flow rate, 60 °C adsorption temperature and 24 h adsorption time.

4. Conclusions

Recovery of IPA from waste solvent of semiconductor fabrication is investigated. The waste solvent was characterized by a high isopropyl alcohol content over 65%, other organic pollutants and strong color. These characteristics defy the traditional treatment methods. In this study, air stripping in conjunction with condensation and activated carbon fiber adsorption was employed to recover the IPA for reuse. Experimental tests were conducted to investigate the performances of air stripping, condensation and ACF adsorption and to identify the optimum operating conditions for these units. Based on the test results, the following conclusions can be drawn.

(1) The waste solvent obtained from a semiconductor plant contained 674 g/l of initial IPA and some other organic compounds and had a dark orange color. Recovery of IPA offers better alternative than direct incineration of the waste solvent disposal.

(2) Air stripping using a packed column with a simple side condensation system was found efficient in removing (IPA) from the waste solvent. Stripping operating at 70 °C, 1 l/min air flow rate with −10 °C condenser cooling and for 180 min was able to recover about 93% of IPA originally present in the waste solvent. The recovered IPA was of high purity and can be recycled for reuse.

(3) The packed ACF adsorber compliments the air stripping/condensation combination quite well in removing the small amount of IPA that remained in the gas mixture. Up to 3% of IPA can be recovered from the gas mixture exiting the side condenser.

(4) A general logic function was adopted to model the breakthrough curves of the IPA adsorption process. The model with parameters properly estimated using the observed data offers reasonably accurate prediction of the experimental breakthrough curves. The model can then be conveniently used for estimating the breakthrough time and capacity that in turn facilitate the design of the packed adsorption column.

(5) A simple thermal process was tested for regenerating the exhausted ACF. Heating of the exhausted ACF column at 150 °C for 60 min was found to yield good results and the regenerated ACF can be reused with minimum of IPA adsorption efficiency.

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