Tray Efficiency in the Air-Water-Solid System without Reaction and Its Application

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Abstract−This work deals with the investigation of tray efficiency with the gas-liquid-solid three phases and its application in the suspension catalytic distillation (SCD) process. The experimental data of tray efficiency under different conditions were measured by utilizing the desorption of oxygen dissolved in water by air. A method of estimation of tray efficiency for the gas-liquid-solid three phases was put forward and its suitability was also verified. This method was developed on the basis of the AIChE method for gas-liquid two phases. As an application example of this modified AIChE method, the SCD process for synthesizing cumene, based on equilibrium stage (EQ) model incorporating tray efficiency, was simulated.

Key words: Suspension Catalytic Distillation (SCD), Tray Efficiency, Gas-Liquid-Solid, Process Simulation

INTRODUCTION

With the development of chemical engineering, the process of distillation coupled with reaction, namely reactive distillation (RD), has been devised for many years. The RD process has been applied in the industry, e.g. the manufacturing of methyl acetate and methyl tert-butyl ether [Zhang and Xu, 1992; Smith and Huddleston, 1982; Subawalla and Fair, 1999; Solokhin and Blagov, 1996]. The RD process is commonly divided into two categories: homogeneous and heterogeneous catalytic distillation. Moreover, heterogeneous catalytic distillation is a more recent development that has attracted researchers' attention because the difficulty in separation between products and catalyst is easy to overcome. In recent years, a new type of heterogeneous catalytic distillation, called suspension catalysis distillation (SCD), has been put forward by Wen and Min [2000]. However, work on the SCD process is very scarce. In the SCD process, tiny solid particles are not used as packing in the column but blended with liquid phase. Compared to the conventional RD process, it has the following unique advantages:

1. No need for structured catalytic-packing

2. No need for shutting down the unit to replace the deactivated catalyst

3. Mass and heat transfers in the fine catalyst particles and interphase are quicker than those in the structured catalytic-packing.

It is believed that the packed column is not suitable for the SCD process. When solid particles are added to the liquid phase, the packed column is prone to be jammed. From this viewpoint, tray columns are more suitable for the SCD process. Among the tray columns (i.e., sieve tray, valve tray and slant-hole tray), sieve tray is the best candidate because the gas-liquid-solid three phases can flow more smoothly in this tray.

Both equilibrium stage (EQ) and non-equilibrium stage (NEQ) models [Taylor and Krishna, 2000; Sundmacher and Hoffmann, 1996; Tanskanen and Pohjola, 2000; Ko and Na, 2002; Kim and Choi, 2003] can be adopted to simulate the RD process. However, developing an NEQ model for an RD process is not as straightforward as it is for the EQ stage model in which we need to simply add a term to take account of the effect of reaction on the mass balances. As we know, the NEQ model is more complicated than the EQ model. In the NEQ model, the design information on the column configuration must first be specified so that mass transfer coefficients, interfacial areas, liquid hold-ups, etc. can be calculated. Therefore, for any newly invented configuration of the column, many experiments have to be done in advance to obtain the necessary model parameters. Evidently, it is too tedious, and much time will be spent on the design of the SCD process. Fortunately, as Lee and Dudukovic [1998] point out, close agreement between the predictions of EQ and NEQ models can be found if the tray efficiency is accurately predicted for the EQ model.

Now, an SCD process to produce cumene with benzene and propylene is exemplified. To design the SCD process, a mathematical model is required. In this work the EQ model for the SCD process to produce cumene is adopted, but it is combined with the tray efficiency used for calculating real stages in the tray column. In the beginning, an experimental study was carried out to measure the tray efficiency.

EXPERIMENTAL

1. Experimental Apparatus and Method

Two systems, air-water and air-water-solid particles, were adopted for measuring tray efficiency. Silica gel particles were selected as the solid phase, and it was based on the consideration that silica gel was used as the supporter of the catalyst used in the SCD process for producing cumene. The particle diameter distribution was obtained by screening method, and is listed in Table 1.

The experimental flowsheet is shown in Fig. 1. Air was driven by a compressor, into the measuring column from the bottom. Water (or the mixture of water and solid particles) was first blended with oxygen and then transported by a centrifugal pump into the top of the measuring column. So the gas from the bottom to the top and

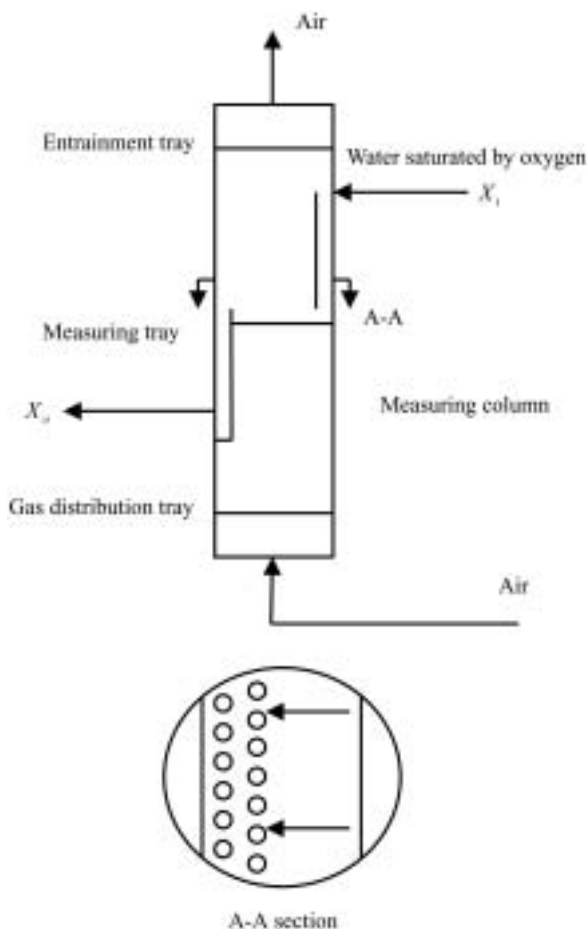†To whom correspondence should be addressed.
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Table 1. Diameter distribution of solid particles

Diameter/mm	Below 0.097	0.097-0.105	0.105-0.125	0.125-0.150	0.150-0.200	0.200-0.300
wt/%	1.99	2.96	25.77	20.28	44.57	4.44

the liquid from the top to the bottom contact countercurrently in the column.

When solid particles are added into water, more attention should be paid to preventing them from sinking down. Otherwise, the local concentration of solid particles in the liquid would be changed again and again. To avoid this, the water tank was kept constantly stirred. In the pipe and column trays, solid particles were found to have no deposition because particles are too small and suspended by the flowing liquid.
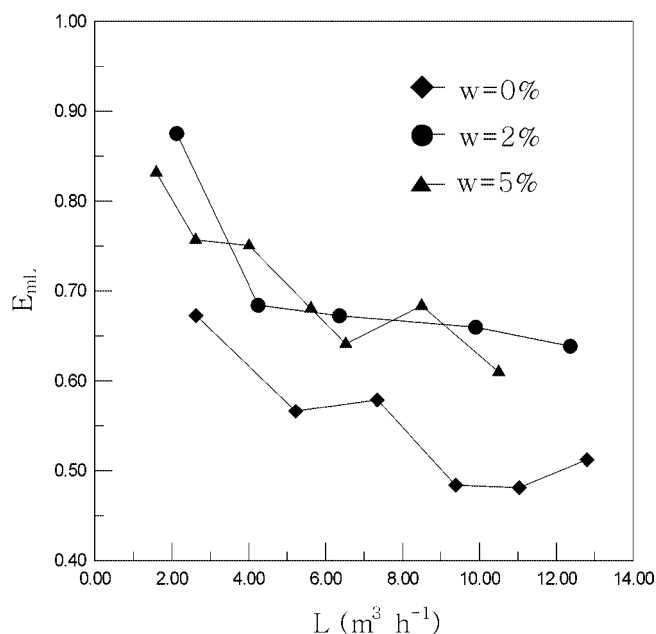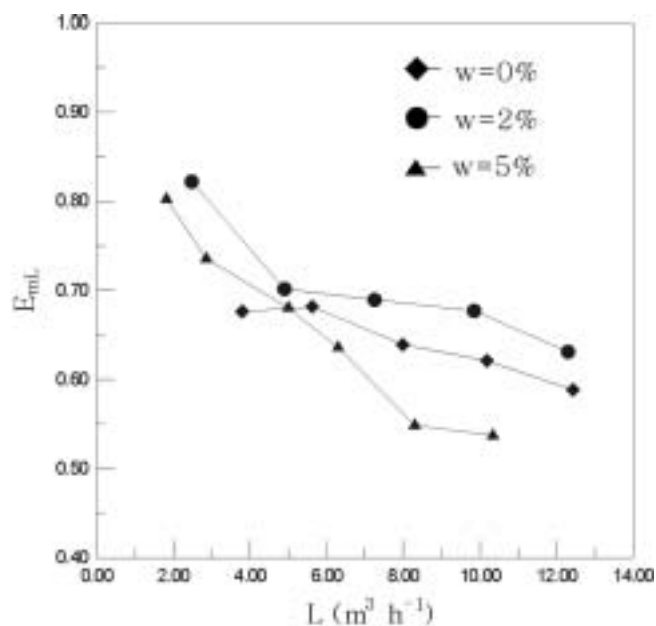
The column having three trays, i.e., entrainment tray, measuring tray and gas distribution tray, was made up of organic glass and had 500 mm I.D., 15 mm thickness, tray distance 300 mm. In the measuring tray the big sieve hole with diameter 10 mm, as well as weir length 336 mm, downcomer area 0.0164 m^2, and sieve-hole area ratio 5.52%, was adopted in this investigation because it was relatively more difficult to be crowded by solid particles in these trays as compared to valve, bubble and small-hole sieve trays. The sieve holes on the tray were arranged according to the equilateral triangle manner. The section A-A of measuring tray is also shown in Fig. 1. The tray efficiency E_{mL} is expressed by the following equation:

$$E_{mL} = \frac{X_i - X_o}{X_i - X_o^*} \times 100\% \qquad (1)$$

where the oxygen concentrations, X_i and X_o are the oxygen concentrations in the liquid phase at the inlet and the outlet, respectively, and X_o^* is the oxygen concentration in the water tank equi-

Fig. 2. Effect of liquid flowrate L on tray efficiency E_{mL} (V=359.74 m^3·h^{-1}, h$_w$=40 mm).

Fig. 3. Effect of liquid flowrate L on tray efficiency E_{mL} (V=440.59 m^3·h^{-1}, h$_w$=40 mm).

Fig. 1. Configuration of the measuring column and trays.

librium with the atmosphere (or the equilibrium oxygen concentrations in the liquid phase with the air leaving the measuring tray). They were determined by oxygen gauge (type JPSJ-605). The flowrates of gas and liquid were, respectively, obtained by orifice-plate flowmeter and rotary flowmeter, both of which had been calibrated beforehand. It took only a few of minutes to achieve the steady state for each run. In the beginning, some preliminary experiments under the same conditions were made to ensure that the data were repeatable.
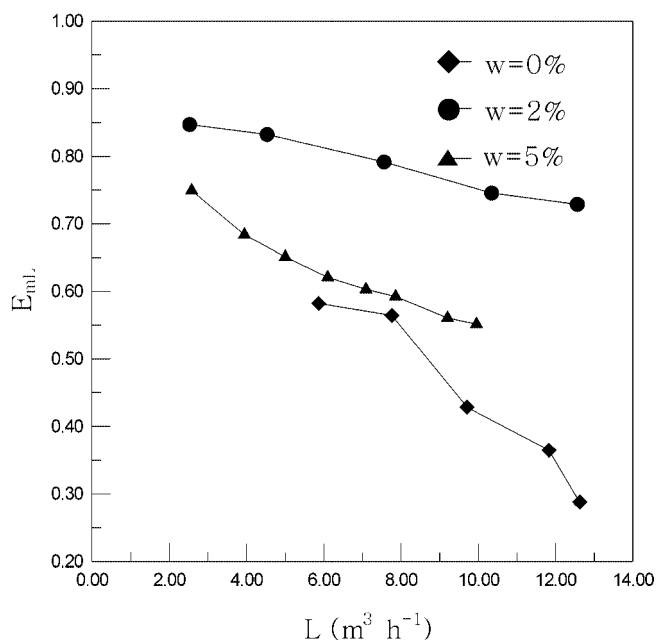
2. Experimental Results

Fig. 4. Effect of liquid flowrate L on tray efficiency E_{mL} (V=359.74 $m^3 \cdot h^{-1}$, h_w=50 mm).

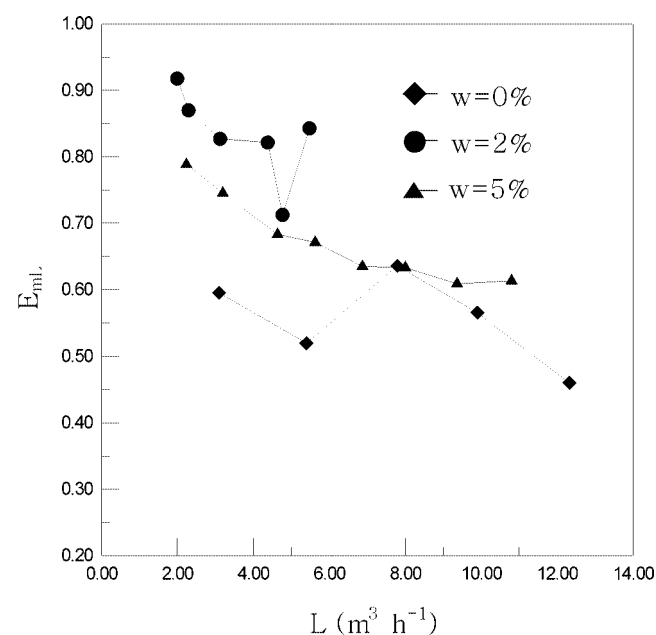Fig. 5. Effect of liquid flowrate L on tray efficiency E_{mL} (V=440.59 $m^3 \cdot h^{-1}$, h_w=50 mm).

The curves describing the relations of liquid flowrate with tray efficiency E_{mL} at different conditions are shown from Fig. 2 through Fig. 5 where three weight concentrations of the solid particles in the liquid phase, w=0%, 2% and 5%, were employed. These concentrations covered the range of the catalyst concentration used in the SCD process, below 5 wt%. If the solid particles are in higher concentrations, problems such as sedimentation, jamming and so on may arise.

From the above figures, it can be found:

(1) For a given solid concentration, tray efficiency decreases with increasing liquid flowrate. This is attributed to the decreasing amount of oxygen stripped from the liquid phase under a constant gas flowrate. That is, in Eq. (1) X_i and X_o^* remain constant, but X_o increases, which results in the decrease of tray efficiency. But at some points, there is a slight fluctuation to this tendency. This is may be due to the unstable flow driven by centrifugal pump and thus setting a buffer tank in the outlet of centrifugal pump is advisable.

(2) With raising the solid concentration, tray efficiency first increases and then decreases over a wide range of liquid flowrate. The reason may be that at low concentration the added solid particles promote the disturbance of interface between gas-liquid phases, and thus decrease the liquid film thickness. A thinner liquid film thickness means a higher liquid-side mass transfer coefficient. Since the systems discussed in this work are just governed by liquid film, the resistance to mass transfer is mainly in the liquid film. So in terms of double-film theory, the tray efficiency will increase as the solid concentration arises. On the other hand, the presence of particles close to the interface may hold back the mass transfer of liquid phase and reduce the contact area between gas and liquid phases. At higher solid concentration, this effect will be dominant. Consequently, the overall result is that tray efficiency no longer increases and even seems go down. This trend is consistent with the conclusion about the effect of solid particles on mass transfer coefficients, reported by Alper [1980] and Beenackers [1993].

It is known that factors influencing tray efficiency are very complicated and involve physical properties of the systems, tray configuration, hydrodynamics, etc. The American Institute of Chemical Engineers (AIChE) has even organized many researchers to study tray efficiency. Ultimately, a set of methods recommended for estimation of tray efficiency of gas-liquid two-phase flow, which is now called the AIChE method, was put forward [Committee of chemical engineering handbook, 1996; Richardson et al., 2002]. Herein, this method is tested with the data obtained in this study for an air-water system and then modified to predict tray efficiency of an air-water-solid particles system.

First, this method is used for predicting tray efficiency of the air-water system. It is found that the average relative deviation (ARD) of tray efficiency is 15.7% between this investigation and the values estimated by AIChE method for 21 points, and can satisfy the general engineering requirement. At the same time, it indicates that the obtained experimental data are reliable. The ARD is defined as

$$ARD = \sum_{i=1}^{n} \left| \frac{(E_{mL}^{exp} - E_{mL}^{cal})}{E_{mL}^{exp}} \right| / n \qquad (2)$$

where n is the number of data points.

Then, this method is extended to estimate the tray efficiency of the air-water-solid particle system by means of replacing the liquid

physical properties with slurry density ρ_m and viscosity μ_m which can be calculated from the following equations [Tong, 1996]:

$$\rho_m = w\rho_s + (1-w)\rho_L \tag{3}$$

$$\mu_m = \mu_L(1+4.5\phi) \tag{4}$$

where ρ_L and μ_L are given by

$$\rho_L = \sum_{i=1}^{c} x_i\rho_i \tag{5}$$

$$\mu_L = \sum_{i=1}^{c} x_i\mu_i \tag{6}$$

It is found that in this case, the ARD of tray efficiency is 13.0% between this investigation and the modified AIChE method for 50 points. It shows that the modified AIChE method can also be employed to estimate the tray efficiency of air-water-solid particles system.

Beenackers and Swaaaij [1993] have studied the influence of solid particles on gas-liquid mass transfer and reported the method of calculating the volumetric liquid-side mass transfer coefficients at the gas-liquid interface $k_L a$, from which tray efficiency can be deduced [Committee of Chemical Engineering Handbook, 1996], for gas-liquid-solid particles system. $k_L a$ can be obtained according to

$$k_L a/(k_L a)_0 = (\mu_m/\mu_L)^{-0.42} \tag{7}$$

with index 0 indication of no solids present.

We select a three phases system, i.e., propylene(gas)-benzene (liquid)-Silica gel(solid particles), to test the modified AIChE method. The calculated values of tray efficiency from the method of using $k_L a$ are compared with those from the modified AIChE method under the same conditions. The results are listed in Table 2 where h_w= 40 mm, and good agreement is obtained. It is shown that the modified AIChE method is suitable for either air-water-solid particle or organic gas-organic liquid-solid particle systems.

PROCESS SIMULATION OF THE SCD PROCESS

The alkylation of benzene with propylene to produce cumene is

Table 2. Comparison of tray efficiency between the modified AIChE method and the method of using $k_L a$

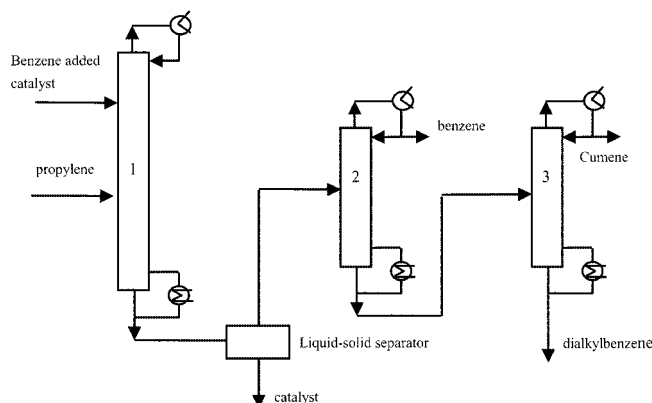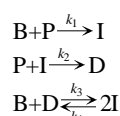| W	V [m³ h⁻¹]	L [m³ h⁻¹]	E_{mL}	
			Modified AIChE method	Method of using $k_L a$
0.02	440.59	2.48	0.8233	0.8278
0.02	440.59	4.90	0.6236	0.6293
0.02	440.59	7.25	0.5069	0.5123
0.02	440.59	9.85	0.4258	0.4308
0.02	440.59	12.30	0.3746	0.3792
0.05	440.59	1.82	0.8929	0.9010
0.05	440.59	2.86	0.7866	0.7984
0.05	440.59	5.00	0.6187	0.6324
0.05	440.59	6.30	0.5487	0.5623
0.05	440.59	8.30	0.4709	0.4837
0.05	440.59	10.34	0.4152	0.4271

Fig. 6. Flowsheet to produce cumene by the SCD process. 1-suspension catalytic distillation column; 2-benzene column; 3-cumene column.

an important reaction in the industry [Nie et al., 2000; Shoemakers and Jones, 1987]. An SCD process, based on the solid acid catalyst for this reaction, is simulated. An EQ model incorporating the tray efficiency model (i.e., the modified AIChE method) is adopted.

The involved reaction network includes alkylation and transalkylation reactions and is expressed as:

$$B+P \xrightarrow{k_1} I$$
$$P+I \xrightarrow{k_2} D$$
$$B+D \underset{k_4}{\overset{k_3}{\rightleftharpoons}} 2I$$

where B, P, I and D represent benzene, propylene, cumene and dialkylbenzene, respectively. The flowsheet of the SCD process is illustrated in Fig. 6.

As shown in Fig. 6, benzene and propylene are fed into the SCD column 1, but at different sites due to their boiling point difference. Benzene is into the top and propylene into the middle. A stream leaving the SCD column from the bottom goes through a liquid-solid separator where the catalyst is separated and then sent outside for regeneration and recycle. The resultant liquid is dealt with in column 2 and column 3 to remove the unconverted benzene and dialkylbenzene (it is a side product). In column 1, the solid acid catalyst is blended with benzene and fed into the top of the column. Along the whole column, the catalyst is evenly distributed.

Provided that the theoretical stages are determined, EQ model can be established to simulate the SCD process. The real stages are deduced from tray efficiency. The equations that model equilibrium stages are known as the MESH equations [Lei et al., 2003] into which the reaction terms including reaction rate equations and reaction heat equations are incorporated. MESH is an acronym referring to the different types of equations. The M equations are the mass balance, E the phase equilibrium relations, S the summation equations with respect to mole fractions, and H the enthalpy balance. Under steady-state conditions all of the derivatives with respect with time in the MESH equations are equal to zero. The modified relaxation method, where the MESH equations written in unsteady-state form are integrated numerically until the steady-state solution has been found, is used to solve the MESH equations.

With the model, herein, an illustrative example is given. It is as-

sumed that the SCD column has 21 theoretical stages with the condenser stage 1 and the reboiler stage 21. The reaction rate equations for the catalyst measured in the range of 80-150 °C, are adopted as follows [Nie et al., 2000].

$$r_1 = k_1 C_B^{0.9} C_P^{1.0} \tag{8}$$

$$r_2 = k_2 C_I^{0.5} C_P^{0.9} \tag{9}$$

where $k_1 = 3.74 \times 10^4 \exp(-7.39 \times 10^3 / T)$, $k_2 = 3.68 \times 10^7 \exp(-1.00 \times 10^4 / T)$.

It is known that the transalkylation reaction is reversible, and in this work it is supposed to reach chemical equilibrium in each stage. The chemical equilibrium constant K is written below [Lei et al., 2004]:

$$K = 6.52 \times 10^{-3} \exp(27240/RT) \tag{10}$$

where R is the universal gas constant (8.314 J mol^{-1} K^{-1}).

The operating parameters of the SCD column are listed in Table 3.

The MESH equations are solved for this condition and the com-

Table 3. Operating parameters of the SCD column

Feeding propylene		Feeding benzene	
Rate, kmol h^{-1}	50	Rate, kmol h^{-1}	100
Temperature, K	323.15	Temperature, K	323.15
Stage No.	13	Stage No.	2
Operating pressure, kPa	700	Total catalyst weight, kg	22000
Reflux rate, kmol h^{-1}	960	Product of the bottom, kmol h^{-1}	100

Table 4. The composition of product on the bottom of the SCD column

	Benzene	Propylene	Cumene	Dialkylbenzene
mole%	47.72	1.25×10^{-3}	46.13	6.14

Table 5. Tray parameters of the SCD column

	Tray number	
	2-12	13-24
Column diameter (m)	2.0	2.0
Tray spacing (m)	0.5	0.5
Sieve-hole area ratio (%)	10.0	10.0
Overflow type	Single overflow	Single overflow
Weir length (m)	1.6	1.6
Weir height (m)	0.03	0.03
Downcomer clearance (m)	0.03	0.03
Downcomer area (m^2)	0.447	0.447
Overflow intensity (m^3·m^{-1}·h^{-1})	47.98	47.36
Vapor superficial velocity (m·s^{-1})	0.32	0.32
Hole F-factor	12.63	12.61
Liquid height in the tray (mm)	67.50	67.17
Tray pressure drop (Pa)	521.10	518.94
Downcomer pressure drop (Pa)	296.16	288.61
Liquid height in the downcomer (mm)	150.81	149.49
Residence time (s)	8.39	8.50
Vapor entrainment	0.0020	0.0021

position of the product on the bottom of the SCD column is given in Table 4, from which it can be seen that the SCD process is very effective for the reaction with final propylene conversion approximately 100% and cumene selectivity 92.26%.

A big-hole sieve tray is chosen for the design of the SCD column. In terms of the vapor and liquid loads of the SCD column, the tray parameters are obtained by means of tray software [Lei et al., 2003] and listed in Table 5, where it is shown that the difference of tray parameters between the rectifying section and striping section is not apparent. The reason may be that the vapor and liquid loads along the column change slowly, and thus constant molar flowrate is approximately valid. These parameters are reasonable because their values are within the range of normal operation conditions.

Under the operating condition listed in Table 3, tray efficiency on most trays is predicted to be about 0.82 according to the modified AIChE method developed in this work for gas-liquid-solid three phases. This value is close to the tray efficiency, 0.83, deduced by using $k_L a$. Thus, the real stages of 25 (including condenser and reboiler) are required for such a synthesis of cumene. The corresponding feeding site for propylene is at the 12th stage. Therefore, the necessary information on the design of the SCD column is known.

However, it is expected that the actual tray efficiency is greater than the calculated values coming from both the modified AIChE method and the method of using $k_L a$. It is known that $k_L a$ is the product of k_L and a, in which a is not changed whether or not a chemical reaction takes place, but k_L will increase if a chemical reaction in the liquid phase is considered. The reason is that the mass transfer is enhanced due to rapid chemical reaction, which can be described by enhancement factor E_A. E_A is defined as the ratio of J_A with particles, to J_A with the same but inert particles, in which J_A is evaluated at the same overall driving force. Inert means that neither the particles nor components produced from the particles participate in the reaction as a reactant or a catalyst. Further, inert particles do not adsorb the gas phase component transported towards the bulk of the liquid phase, nor any other reactant or reaction product. The SCD process discussed above is just the case. Even so, it seems that the designed result is safe and conservative because we take the tray efficiency smaller than actual into consideration.

One typical equation of expressing E_A for rapid first-order reaction, constant gas phase concentration and no liquid through-flow of bulk [Wimmers and Fortuin, 1988] is:

$$E_A = \frac{Ha_f}{\sinh(Ha_f)} \left\{ \cosh(Ha_f) - \cfrac{1}{\cosh(Ha_f) + \left[\cfrac{k_r C_{si} \sinh(Ha_f)}{k_L a \cdot Ha_f} \right]} \right\} \tag{11}$$

where C_{si} is the actual solids concentration in the gas-liquid film for mass transfer, k_r is the pseudo-homogeneous first-order reaction rate constant, and Ha_f is the modified Hatta number defined by the following equation:

$$Ha_f = \frac{\sqrt{k_r C_{si} D_A}}{k_L} \tag{12}$$

CONCLUSION

Due to the unique advantages of the SCD process, it is interest-

ing to explore this new subject. Tray efficiency for the sieve tray of gas-liquid-solid three phases is experimentally studied. It is demonstrated that if such physical properties as the density and viscosity of the liquid phase, ρ_L and μ_L, are replaced with those of the liquid-solid mixture, ρ_m and μ_m, the AIChE method is still suitable for no air-water-solid systems. This work contributes to extend the application range of the AIChE method.

On the basis of tray efficiency, the EQ model is adopted to simulate the SCD process. It is verified by process simulation that the SCD process is valid for the alkylation of benzene with propylene to produce cumene. Although the same conversion and selectivity may be reached in a conventional RD process, the reuse of catalyst is tedious and very difficult, especially for such reactions as synthesizing alkylbenzene with longer carbon chains. In a conventional RD process, the catalyst is quick to be deactivated and should be replaced by fresh catalyst from time to time. But the SCD process does not have this problem and is convenient to operate, which has been verified in our pilot plant. By combining the EQ model and tray efficiency model, much real information on the SCD column may be derived.

ACKNOWLEDGMENTS

NOMENCLATURE

a : gas-liquid contact area [m^2 m^{-3}]

C : mole concentration [mol l^{-1}]

C_{si} : actual solids concentration in the gas-liquid film for mass transfer [mol l^{-1}]

c : number of components [-]

D_A : molecular diffusion coefficient for component A [m^2 s^{-1}]

E_A : enhancement factor for component A [-]

E_{mL} : tray efficiency relative to the liquid phase [-]

Ha_f : modified Hatta number defined by Eq. (10) [-]

h_w : weir height [mm]

J_A : molar flux of component [kmol m^2 s^{-1}]

K : chemical equilibrium constant [-]

k : reaction rate constant [-]

k_L : liquid-side mass transfer coefficient [m s^{-1}]

$k_L a$: volumetric liquid-side mass transfer coefficient [s^{-1}]

k_r : pseudo-homogeneous first-order reaction rate constant [-]

L : liquid flowrate [m^3 h^{-1}]

R : the universal gas constant [J mol^{-1} K^{-1}]

r_i : reaction rate of component i [mol s^{-1} kg^{-1}]

T : temperature [K]

V : vapor flowrate [m^3 h^{-1}]

w : weight fraction of solid phase in the liquid [-]

X_i : oxygen concentration in the liquid at the inlet [kg m^{-3}]

X_o : oxygen concentration in the liquid at the outlet [kg m^{-3}]

X_o^* : oxygen concentration in the equilibrium [kg m^{-3}]

x : mole fraction in the liquid phase [-]

ρ_L : liquid density [kg m^{-3}]

ρ_m : mixture density [kg m^{-3}]

ρ_s : solid density [kg m^{-3}]

μ_L : liquid viscosity [Pa s^{-1}]

μ_m : mixture viscosity [Pa s^{-1}]

ϕ : solid volume fraction [-]

Superscripts

cal : calculated value

exp : experimental value

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