A HIGH-EFFICIENCY DISTILLATION SYSTEM FOR BATCH OR SEMI-BATCH CHEMICAL REACTORS

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A batch distillation system was improved by installing a newly invented stirrer into the kettle-type reboiler. It has been confirmed by the heat transfer observation that the F-factor can be kept constantly high over the whole of batch distillation.

KEYWORDS: new stirrer, batch distillation, high-efficiency reboiler, energy saving, evaporative heat transfer

INTRODUCTION

Organic chemical industries dealing with batch or semi-batch reaction processes usually need various separation processes to purify reaction products. In particular, a multi-purpose high-efficiency distillation system is very useful after or during a reaction process. For example, it is of practically great importance to rectify solvent mixtures contaminated with reaction byproducts and/or reactant substances. A new rotating stirrer named "Wall Wetter" (Yamaji et al. 2001 & 2002, Noda et al. 2005) is installed into the kettle-type reboiler of a batch distillation system. The Wall Wetter reboiler can keep the rate of evaporation constantly high during the entire distillation process (Yamaji et al. 2002). A batch distillation system accompanied with the Wall Wetter reboiler is very useful as the rectifier not only for the recovery of various solvents but also for the separation of unreacted substances, monomers and byproducts from the main reaction products. The Wall Wetter evaporator can also be utilized as the batch reactor vessel. Even during the term of batch reaction process, such a distillation column equipped with a vacuum controlling system can be employed to remove volatile byproducts or inhibitive components from the reacting solution.

This study deals with the characteristics of an experimental distillation system equipped with the Wall Wetter reboiler.

EXPERIMENTAL APPARATUS AND METHOD

Figure 1 shows a schematic picture of experimental setup. The reboiler with a steam jacket (total area for heat transfer $S = 0.68\ m^2$) has an inside diameter 400 mm. The distillation column (inside diameter = 200 mm) has seven stages with no downcomers and the stage-to-stage spacing is 400 mm.

The overhead condenser has a very large area for heat transfer $2\,m^2$ only for experiment.

As shown in Figure 2, the new stirrer "Wall Wetter" consists of two inclined pipes or half-cut pipes. If the Wall Wetter stirrer is installed into a jacketed tank (kettle-type)

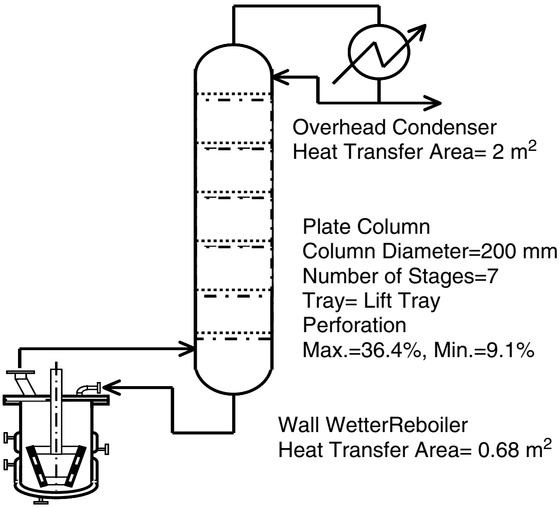


Figure 1. Experimental setup for batch distillation

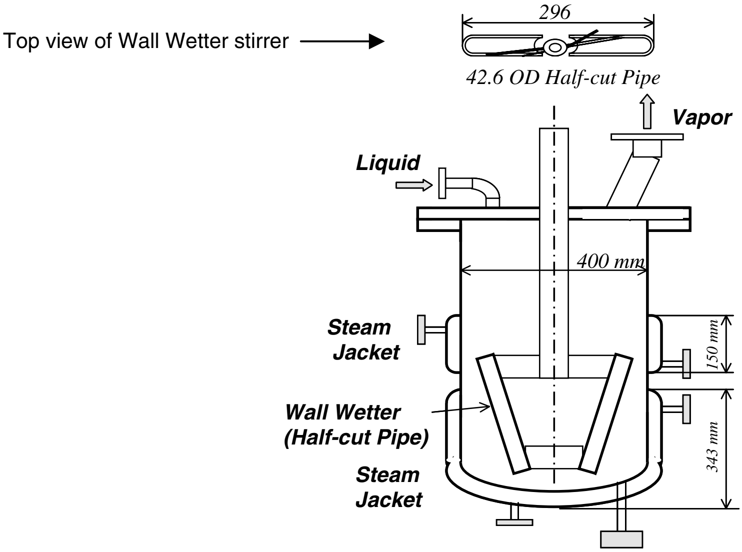
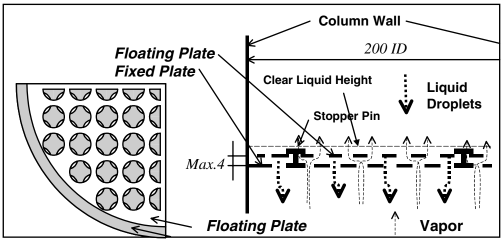


Figure 2. Wall Wetter evaporator for reboiler



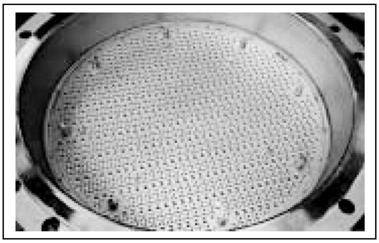


Figure 3. Dual flow tray "Lift Tray" and its photograph of a large tray for a 900 mm ID column

reboiler, a large volume of liquid can be pumped up through these gutters due to its centrifugal effect from the liquid pool and sprayed over the upper jacket surface above the liquid pool level. The upper heat transfer area is covered with a falling liquid film, so that the entire jacket surface can be utilized effectively over the whole term of batch distillation.

Original dual flow trays named "Lift Tray" are installed. As shown in Figure 3, this tray is a set of two perforated plates: a fixed perforated plate covered from upside with a floating perforated plate which can move up and down depending on the vapor flow rate. Therefore this tray has a great advantage of keeping the pressure drop almost constant over a wide range of F-factor by automatically changing the perforation per cent (max. = 36.4%, min. = 9.1%) with the height of the floating plate.

The batch distillation experiment was started by supplying a binary mixture of methanol and water (30 mole%, 35 Liter) into the reboiler. Heat was supplied by condensation of steam through the wall of the jacket. The heat transfer resistance on the side of steam jacket can be assumed negligibly small. Two types of batch operations were conducted in order without supplying any additional feed. One is steady state operation under the total reflux condition. The other is batch distillation operated at a constant reflux ratio with no feed rate and no bottom rate. The liquid volume in the reboiler was determined as an average between the beginning and end. The rotation number of the Wall Wetter stirrer was varied depending on the liquid pool depth to keep the pumped-up liquid flow rate constant. The operation pressure was kept 1 atm at the top. The experimental conditions are listed in Table 1.

When the Wall Wetter is rotated, the entire wall of the steam jacket is used effectively as the active heat transfer area. On the other hand, when the Wall Wetter stirrer is at rest, the F-factor, i.e. the reflux flow rate decreases because only the lower part of the heat transfer surface in contact with the liquid pool is used for evaporation.

EXPERIMENTAL RESULT AND DISCUSSION

HEAT TRANSFER CHARACTERISTICS OF REBOILER

The vapor of methanol in the Wall Wetter reboiler is supplied into the bottom of the test column shown in Figure 4 shows variation of heat transfer rate with the liquid pool volume

Table 1. Experimental condition

Run	Condition	Wall Wetter (rpm)	Liq. volume (L)	Reflux rate (L/h)	Reflux ratio	Steam temp. (C)	Reboiler temp. (C)
1	Total Reflux	150	30.1	133.6	Infinity	126.6	87.8
	Total Reflux	0	30.1	130.4	Infinity	128.9	84.5
2	Batch Distil.	150	28.0 ~ 21.3	111.8	5.4	129.4	87.2
	Total Reflux	150	21.3	128.4	Infinity	131.6	89.4
	Total Reflux	0	21.3	124.2	Infinity	154.2	92.7
3	Batch Distil.	175	21.3 ~ 17.74	104.2	5.03	132.8	92.8
	Total Reflux	175	17.74	131.9	Infinity	138.0	99.0
	Total Reflux	0	17.74	64.0	Infinity	155.8	100.4
4	Batch Distil	200	17.74 ~ 14.88	120.0	5.80	138.3	100.0
	Total Reflux	200	14.88	110.0	Infinity	138.9	100.5
	Total Reflux	0	14.88	41.0	Infinity	156.2	100.6
5	Batch Distil	225	14.88 ~ 10.6	68.4	3.35	138.2	100.6
	Total Reflux	0	10.6	28.1	Infinity	156.3	100.4

of the reboiler. As far as the Wall Wetter stirrer is rotated at an appropriate speed, the heat transfer rate can be kept at a high constant value around 35 kW. The heat transfer rate decreases directly with the effective heat transfer area decreasing unless the Wall Wetter stirrer is rotated.

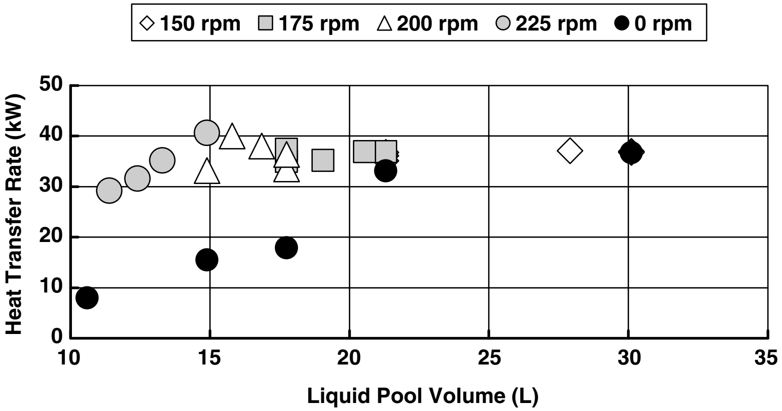


Figure 4. Variation of heat transfer rate with liquid pool volume

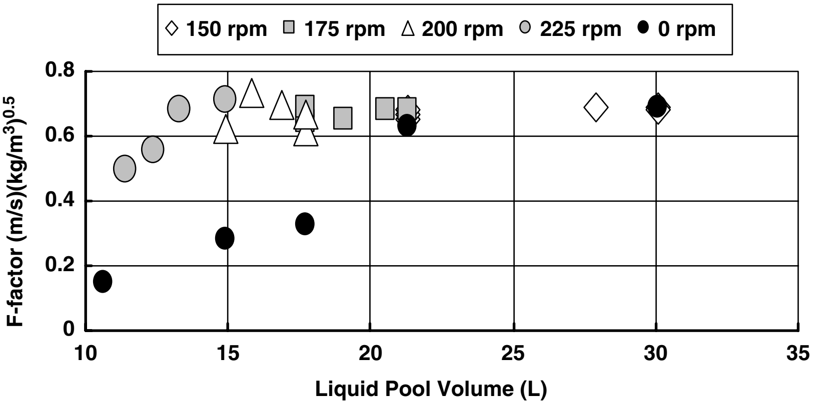


Figure 5. Effect of Wall Wetter rotation on the relation of F-factor with liquid volume

Figure 5 shows correlation of the pool volume V of liquid contained in the reboiler with the vapor flow rate expressed in terms of F-factor. As far as the Wall Wetter stirrer is rotated at an appropriate speed, the F-factor can always be kept at a high constant value until $V = 12.5$ liter irrespective of the liquid volume contained in the reboiler. If the lower edge of the Wall Wetter stirrer is lowered further close to the bottom, the F-factor can be kept at the high value even when the liquid pool volume becomes much smaller with time for the case of batch distillation. This result suggests the advantage of the Wall Wetter reboiler. When the Wall Wetter stirrer is at rest (i.e. 0 rpm), the F-factor decreases depending on the decreasing effective heat transfer area. The corresponding result of heat transfer coefficient is shown in Figure 6, where the heat transfer

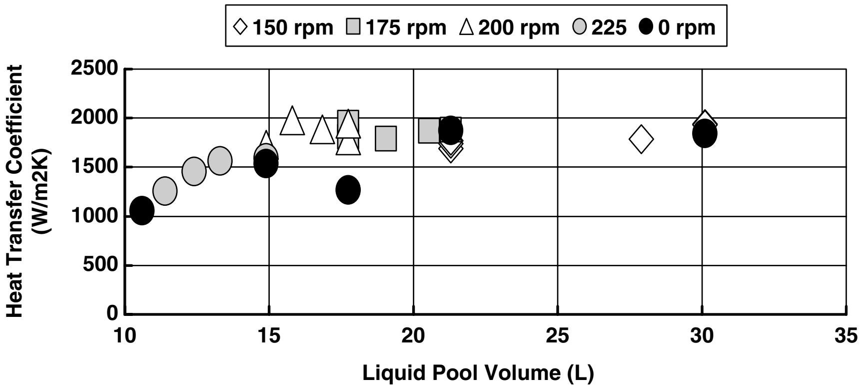


Figure 6. Heat transfer coefficient on the inner wall of the reboiler

resistance on the outer side of the jacket wall is assumed negligibly small because of the great heat transfer coefficients of steam condensation.

Except when V = 18 liter with 0 rpm, the heat transfer coefficients have a high constant value around 1800 W/m^2K irrespective of Wall Wetter rotation. This suggests that the heat transfer coefficient in the liquid pool zone is almost as large as that in the falling liquid film zone. This means that the main factor controlling the heat transfer in a reboiler of this kind should be the effective area for heat transfer in contact with the liquid to be vaporized.

EFFECT OF WALL WETTER STIRRER ON DISTILLATION CHARACTERISTICS

Figure 7 shows the temperature and methanol concentration in vapor observed when the batch distillation is conducted at steady state under the total reflux condition with/without Wall Wetter rotation. It has been found that water vapor is flowing upward at 100°C from the 7th stage i.e. the bottom whereas the almost pure methanol vapor is leaving the column at its boiling point. When the Wall Wetter stirrer is rotated at 175 rpm, the rectification of methanol vapor is promoted due to an increase in the internal reflux. This suggests that the reflux ration can be kept at a large value by means of the of the Wall Wetter stirrer withdrawing the overhead product even when the liquid pool in the reboiler is reduced to a very small volume. When the rotation number was changed from 175 rpm to 0 rpm, however, the internal reflux at the top dropped from 131.9 L/h to 62.9 L/h.

PRACTICAL APPLICATION TEST FOR SOLVENT RECOVERY

This batch distillation system can be utilized for various purposes of practical processes. As an example, it was tested for recovery of solvents form a practical waste solvent

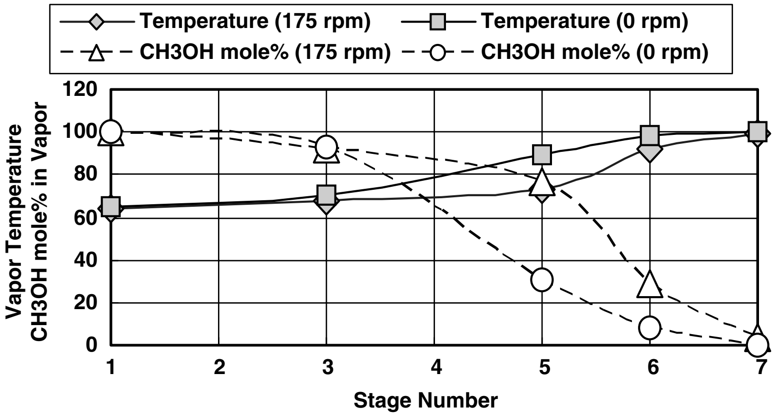


Figure 7. Distribution of vapor temperature and methanol concentration in vapor in the test column operated under total reflux condition

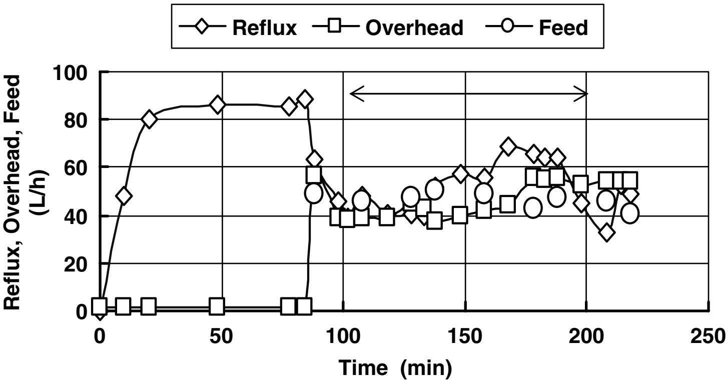


Figure 8. Temporal variation of reflux, overhead product and feed rates (Operation Record) ↔ Quasi-steady operation term

mixture used for washing a reactor. The composition of feed mixture had 97.8 wt% of ethyl acetate, 2 wt% of n-butyl acetate and traces of toluene with traces of other less-volatile components and resin residua.

Figures 8 and 9 show some experimental results obtained by operating the same plate column at normal pressure. Figure 8 shows an operation record of the reflux flow, overhead product and feed. In the first term (until 83 min) after supplying 31.5 kg of a waste solvent mixture into the reboiler, the test column was operated under the total reflux condition with no feed. For about the next 2 hr, a quasi-steady

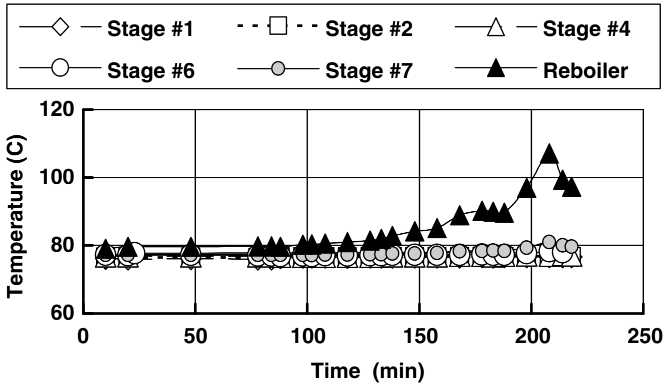


Figure 9. Vapor temperature trajectory of each stage in the quasi-steady operation period

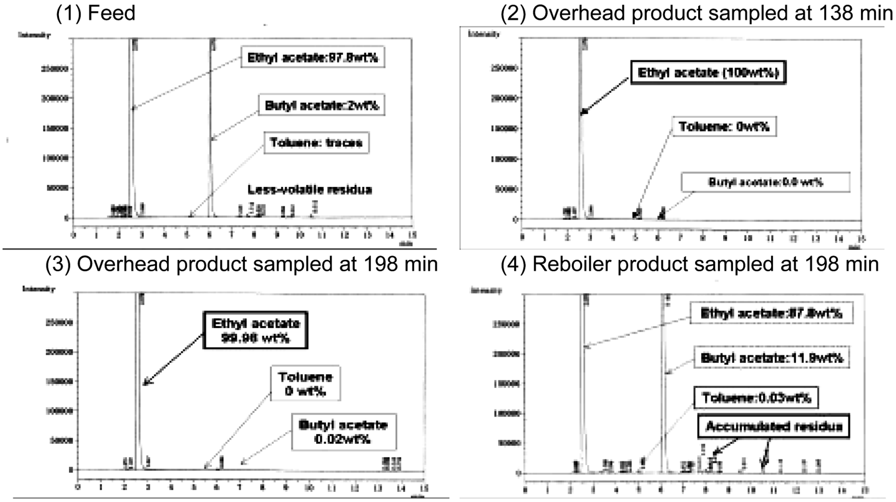


Figure 10. Gas chromatograms of composition of overhead product and reboiler solution in the quasi-steady operation term with the feed composition

operation was performed keeping the feed rate into the reboiler equal to the discharge rate of the overhead product under a condition of constant reflux ration (reflux ration = 1.0). One of the practical purposes of the solvent recovery is how to make the residuum volume as small as possible. Therefore no bottom product was discharged from the reboiler in the quasi-steady operation, where less-volatile components including toluene and some residuary waste substances such as resin, surfactant and pigment were accumulated. The pure solvent to be reused was successfully recovered from the overhead product.

The gradual rise of the reboiler temperature indicated in Figure 9 suggests the same accumulation of less-volatile components. Figure 10 shows gas chromatograms of the overhead product and the reboiler solution. It has also been well confirmed that in the quasi-steady operation (83 $\sim$ 208 min), the majority of pure ethyl acetate was successfully recovered as the overhead product while less-volatile components such as toluene and butyl acetate were accumulated with non-volatile substances in the reboiler. It is another great advantage that this reboiler can be easily washed after the batch distillation because the head transfer surface is always kept wet and does not have any dried-up substances.

CONCLUSION

The heat transfer performance of the kettle-type reboiler of batch distillation system was improved by installing the Wall Wetter stirrer. Batch distillation processes can be conducted keeping the F-factor at a high constant value even when the liquid solution contained in the reboiler becomes a small volume owing to evaporation. This leads to great saving of energy and shortening of distillation time. It has successfully been demonstrated that this system can be effectually utilized for recovery of reuseable solvent from waste solvent mixtures.

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