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Performance and applications of flow-guided sieve trays for distillation of highly viscous mixtures

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Abstract–With a particular focus on the distillation of highly viscous or self-polymerized mixtures, this study reports the hydrodynamic and mass transfer performance of two flow-guided sieve trays, including their pressure drop, entrainment, weeping and tray efficiency, obtained experimentally with an air-water/oxygen system in a Φ600 mm plexiglass column. The results show that the 8 mm hole flow-guided sieve tray tested shows better characteristics than the 7 mm flow-guided hole tray in terms of pressure drop and mass transfer. Then we present practical industrial examples of applications of the flow guided trays for distillation of viscous mixtures, i.e., the separation of vinyl acetate (VAC) from a polyvinyl acetate (PVAC) polymer solution with dynamic viscosity μ=50,000 mPas, the separation of highly unsaturated C5 mixtures by extractive distillation, and the distillation of thick, condensed and highly viscous fermentation mixtures made from fermented mash. It is demonstrated that flow-guided sieve trays with relatively large holes are an excellent candidate for distillation of mixtures with suspended solids, or concentrated/self-polymerized polymer solutions.

Key words: Flow-guided Sieve Tray, Viscous Mixture, Distillation, Hydrodynamic Performance, Mass Transfer

INTRODUCTION

Distillation technologies are frequently encountered in the chemical, petrochemical, and environmental technology industries. Investigations and experimental work aimed at further improving the performance of various distillation columns have been carried out by many researchers for many years, accompanied by ongoing advances and improvements in this technology [1-6]. The current situation with respect to their design and scale up seems closer to maturity from a technological point of view. Yet, still there are challenges here for us process engineers.

One unresolved problem is the distillation of highly viscous mixtures [7-10], for example, products with suspended solids, or concentrated/self-polymerized polymer solutions. Mahiout and Vogelpohl [7] conducted hydrodynamic experiments on a sieve tray using viscous glycerol-water mixtures with viscosities between 10-150 mPas, and they found deteriorated mass transfer performance on the tray with increasing viscosity. Bocker and Ronge [8] studied the effect of viscosity on the mass transfer performance of a structured packing column with polymer solution viscosity up to 260 mPas and drew the same conclusion as above. Compared to the conventional liquid mixtures having a viscosity ranging from 1 to 10 mPas for distillation, the solutions studied by Mahiout and Vogelpohl [7] and Bocker and Ronge [8] have viscosities 2-3 orders higher. However, that is not the whole story.

The removal of monomers from viscous polymer solutions by distillation plays an important role in the polymer industry. In practice, we encountered a distillation task for removal of vinyl acetate (VAC) from a polyvinyl acetate (PVAC) polymer solution with dynamic viscosity μ=50,000 mPas, density ρ=852 kg/m^3; and containing 36 wt% PVAC solid particles, 32 wt% VAC, 31 wt% solvent methanol, with remaining 0.3-0.6 wt% acetone and aldehyde. The current process for distillating such a feedstock was based upon a sieve tray process. However, the trays were easy to jam or flood. This happened 1-2 times every month. Under such circumstances, applicable solutions to distillation column design other than the conventional ones are needed to meet particular individual demands. To this end, we designed large hole (say 7-25 mm) flow-guided sieve trays and succeeded in applying them to various distillation tasks of highly viscous mixtures.

As one type of column internal, the flow-guided sieve trays have the advantage of low pressure drop, high efficiency, throughput capacity, and simplified configuration. Such trays are suitable for low-pressure rectification and separation on a large scale [11] and have been used successfully in industry [12]. In the past, both laboratory and plant-scale data from cold model or distillation tests on such trays were reported now and then [10,11], but their hydrodynamic and mass transfer performance is not yet well understood.

This paper presents the results of an air-water cold model test on two flow-guided trays at different operation conditions, including their basic hydrodynamic and mass transfer properties. Then we present relevant industrial examples of applications of the flow-guided trays for distillation of viscous mixtures. It is hoped that our work can provide a basic understanding of the flow-guided trays and extend the range of applications of such trays in distillation technologies.

COLD MODEL RESULTS AND DISCUSSION

In this section, we discuss experimental data of process variables known to have effects on the tray performance, including the pressure drop, entrainment, weeping and tray efficiency. The data

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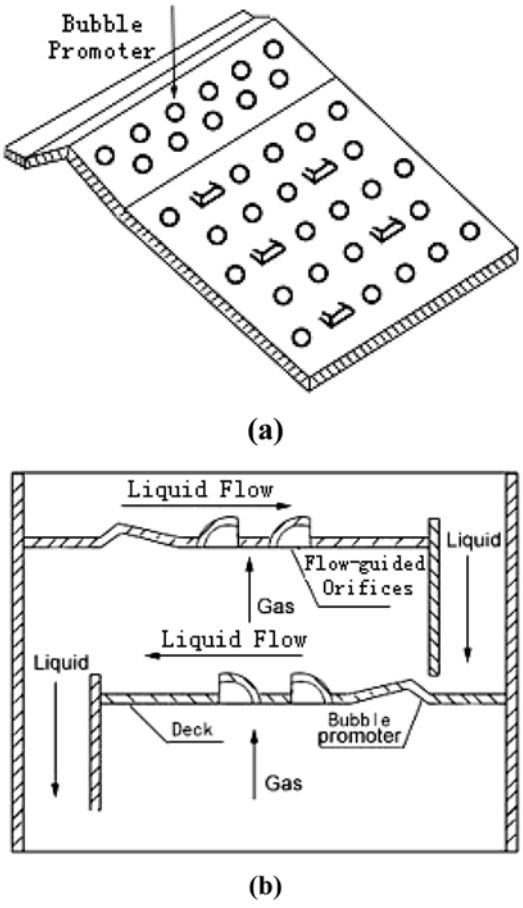

Fig. 1. Schematic representation of flow-guided sieve trays and column.

were obtained on a cold model setup made of a 0.6 m-diameter plexiglass column. Two flow-guided sieve trays were tested. The main features of the two trays are shown in Fig. 1 and Table 1, where the open area percentage of the trays was calculated by using open holes based on the overall area of the column. Due to experimental difficulties presented by a high viscous medium, the conventional air-water system was adopted for the hydrodynamic experiments and air-water-oxygen system for the mass transfer experiments. Therefore, this part of work could be better viewed as showing certain basic features of the performance of the trays.

1. Pressure Drop

Comparison of pressure drop between the two trays is shown in Fig. 2 and 3. As expected, under the same operating conditions the pressure drop of the 8 mm hole flow-guided sieve tray is much smaller than that of the 7 mm one over the hole velocities investigated.

2. Entrainment

In Fig. 4, the relative entrainment, q, for the 8 mm tray is plotted vs.

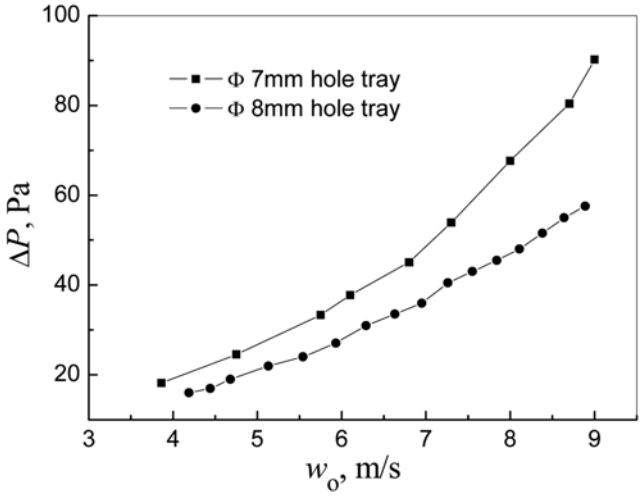

Fig. 2. Comparison of dry-plate pressure drop of the trays.

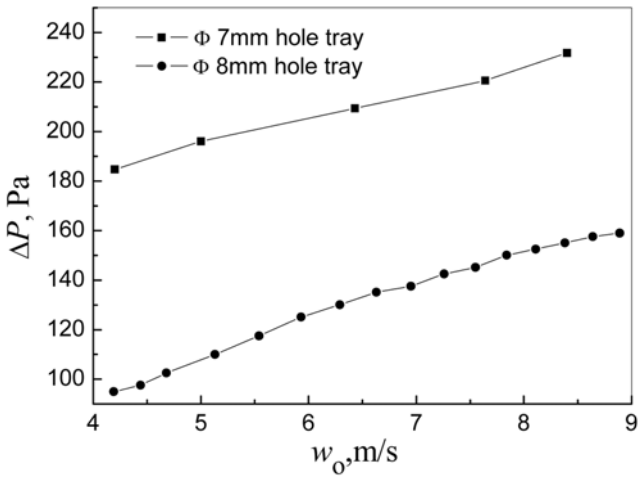

Fig. 3. Comparison of pressure drop of the gas across the liquid phase at L_w=2.7 m^3/(m h).

the superficial vapor velocity, w_G. As can be seen, entrainment increases with the velocity of vapor increased, and the values are no more than 0.1 kg liquid/kg vapor, which is the normal operation condition.

3. Weeping

Fig. 5 shows, for different operating conditions, the relationship between the relative weeping rate w and the hole flow factor F_h, for the 8 mm tray. It can be seen that an increase in hole flow factor reduces the weeping. In particular, in the lower range of hole flow factor, the fall of weeping is rapid.

4. Tray Efficiency

Comparison of the tray efficiency is shown in Fig. 6: the tray effi-

Table 1. Dimensions and geometry of the flow-guided sieve trays tested

Hole diameter, mm	Hole separation, mm	Plate thickness, mm	Fraction of hole area φ, %	Flow-guided holes		Bubble promoter	
				No.	Fractional area φ', %	Height, mm	Inclination tanθ
8	12	3	31	24	5.6	5	0.1
7	15	3.5	15	24	3.6	10	0.1

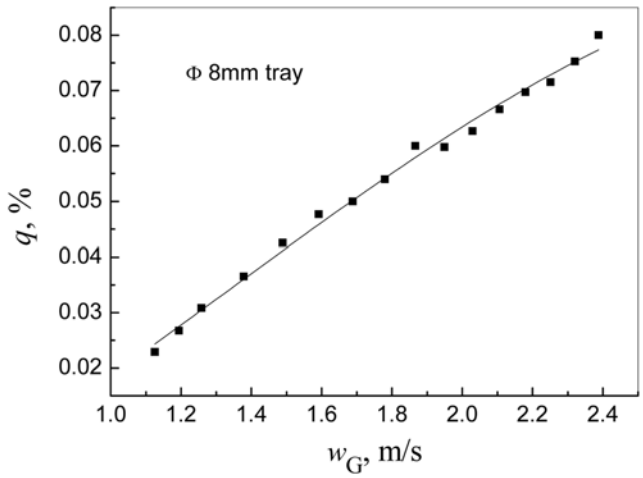

Fig. 4. Relationship between entrainment and w_G at L_w=2.7 m³/(m h).

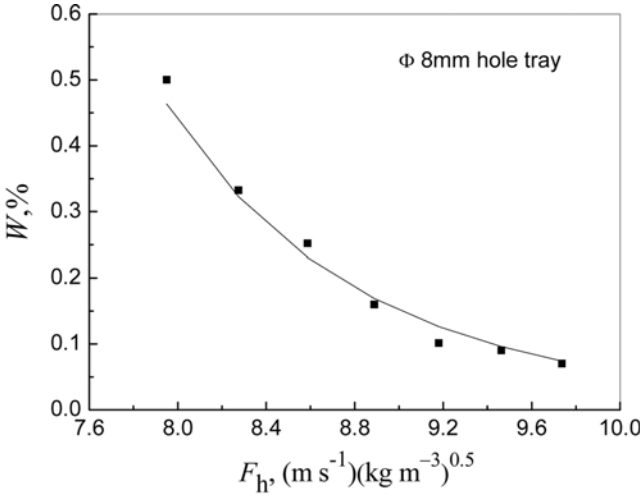

Fig. 5. Relationship between weeping rate and hole flow factor at L_w=8.1 m³/(m h).

ciency of the 8 mm hole flow-guided sieve tray is evidently higher than that of the 7 mm flow-guided sieve tray under the same conditions.

From the above, it could be seen that the 8 mm hole flow-guided sieve tray performed better compared with the 7 mm hole flow-guided sieve tray. Besides, all the experimental data have been correlated for predicting pressure drop, entrainment and lower limiting vapor velocity, which can be used for a preliminary design of flow-guided sieve trays.

INDUSTRIAL APPLICATIONS

One revamp application of the large hole trays was the separation of vinyl acetate (VAC) from a polyvinyl acetate (PVAC) polymer solution with dynamic viscosity μ=50,000 mPas, density ρ=852 kg/m³. The feed mixture contained 36 wt% PVAC solid particles, 32 wt% VAC, 31 wt% methanol, with remaining 0.3-0.6 wt% acetone and aldehyde. The current process for the separation was based on a 2.0 m diameter column and 50 sieve trays process. However, the trays were easy to jam or flood. Such events could happen 1-2 times every month. In the bad cases, this could lead to explosion of the upstream polymerization kettle due to overpressure errors. To solve the problem, we revamped the valve trays with the 8 mm hole flow-guided trays. The results before and after technological revamping are presented in Table 2. It is noted that after the revamp the content of VAC at the top of the column increased from 50 wt% to 60 wt%, and the kettle composition of VAC decreased from 0.26 wt% to around 0.05 wt%. Meanwhile, the normal operation period of the column was largely prolonged and the reflux ratio was reduced by a factor around 2, even though an increased throughput capacity was employed. Obviously, the improvements are substantial. Both the flow-guided holes and the bubble enhancement equipment on the trays serve to promote the viscous media flowing unblocked, helping to form a *zero gradient* liquid mixture level and *slug-like* liquid mixture flow. In this way, an effective mass transfer between phases and less pronounced liquid back mixing are guaranteed, which leads to a superior separation efficiency and capacity. To support this argument further, a field experiment was performed to measure the liquid mixture levels on the flow-guided trays of a commercial-scale 1.4 m I.D. column using the real feedstock, the PVAC polymer solution. The results are presented in Table 3 over a wide range of test conditions. As can be seen, the differences in liquid level over the trays are all below 7 mm, indicating that the gradient on the trays is very small and hence the suspension flow is unblocked.

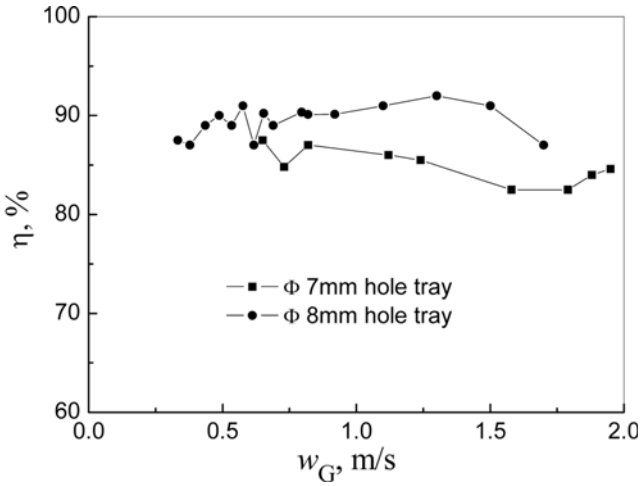

Fig. 6. Comparison of tray efficiency of the trays.

Table 2. Technological indexes before and after renovation of the PAVC column

	Feed rate, kg·h^{-1}	Normal production span, month	Top composition VAC, wt%	Kettle composition VAC, wt%	Reflux ratio
Before revamp	6,500	0.5-1	50	0.26	1.3
After revamp	9,000	≥18	60	0.04-0.06	0.7

Table 3. Liquid level difference ($\triangle^{\#}$) on trays in the 1.40 m I.D. column at varying gas velocities through the holes (W_G) and liquid weir loadings (L_W)

L_W=17.5 $m^3/m\cdot h$		L_W=22.9 $m^3/m\cdot h$		L_W=28.6 $m^3/m\cdot h$	
W_G, m/s	$\triangle$, mm	W_G, m/s	$\triangle$, mm	W_G, m/s	$\triangle$, mm
1.33	3	1.34	5	1.4	5
1.56	−2	1.59	2	1.68	3
1.7	6	1.77	−5	1.87	−6
1.88	−5	1.9	−5.5	1.98	−6.3
2.02	4	2.15	−6.4	2.13	−6.8
2.25	−7	2.3	−7		

Note: # the liquid level difference is defined as (level at the upstream inlet-level at the weir) along the tray.

A further typical use of the large hole trays was implemented for the separation of C5 mixtures by extractive distillation. Here we encountered a mixture containing the bulk of isoprene and cyclopentadiene, and trace of piperyene. The current process for the separation of isoprene was based on a 1.0 m diameter column and 50 float valve tray process. It is known that the highly unsaturated isoprene is prone to self polymerization and formation of viscous micro-aggregates, causing frequent jam or flooding during the extractive distillation. Indeed, this was the situation for the existing process. We designed a special revamp scheme to deal with this case. The existing 160 valve trays were completely replaced with our large hole flow-guided trays, among which 10 flow-guided trays having 25 mm holes were used as the feeding part of column, 5 flow guided trays having 12 mm holes as the transitional part of column, and 145 flow guided trays having 8 mm holes as the main body of column. The results before and after technological revamping are presented in Table 4. From the results, similar remarks to those made for the previous example could be drawn. In particular, for the present case, one major target is to minimize the cyclopentadiene content at the column top for meeting the isoprene product standard. Before the revamp, the top composition of cyclopentadiene fluctuated between 0.5-1.5 ppm, while after the revamp the composition was well below 0.6 ppm steadily.

A final, but not the last example, was the distillation of thick, condensed and highly viscous fermentation mixtures made from fermented mash of grain like corn and sweet potato. In such case, edible alcohol is to be separated from the mushy mixtures by distillation. The current process for the separation of alcohol was based on a 1.8 m diameter column and 26 bubble-cap plates process. On such plates the highly viscous fermentation mixture was difficult to flow smoothly and easy to jam and block. Our solution to this problem was to revamp all the bubble-cap plates with our 8 mm flow-guided trays. The results before and after technological revamping are presented in Table 5. Again, the excellent performance of the large hole trays is fully displayed.

CONCLUSIONS

The hydrodynamic and mass transfer performance of two test flow-guided sieve trays and the relevant industrial examples of applications of the flow guided trays for distillation of highly viscous mixtures has been presented. Based on the work performed and the discussions presented, the following major conclusions can be drawn:

1. The 8 mm hole flow-guided sieve tray tested shows better characteristics than the 7 mm hole tray, i.e., a decreased pressure drop by 25% and an increased efficiency by 5% can be obtained with use of the 8 mm hole tray.

2. Flow-guided sieve trays with relatively large holes are an excellent candidate for distillation of highly viscous mixtures.

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NOMENCLATURE

q : vapor entrainment (based on the vapor flow rate) [%]
E : tray efficiency [%]
F_h : hole flow factor $[(m\ s^{-1})\cdot(kg\ m^{-3})^{0.5}]$
ΔP : pressure drop [Pa]
L_w : liquid weir loadings $[m^3/(m\ h)]$
W : liquid weeping based on the liquid flow rate [%]
w_o : hole velocity [m/s]
w_G : calculated velocity of vapor based on cross-section of the column [m/s]

Greek Letters

φ : fractional area of holes
φ' : fractional area of flow-guided holes

REFERENCES

1. S. Pradhan and A. Kannan, *Korean J. Chem. Eng.*, **22**, 441 (2005).

Table 4. Technological indexes before and after renovation of the C5 extractive distillation column

	Feed rate, $kg\cdot h^{-1}$	Normal production span, day	Top composition cyclopentadiene, ppm	Solvent rate, m^3/h
Before revamp	650	15-25	0.5-1.5	5.6
After revamp	1,050	120	0.5-0.6	5.0

Table 5. Technological indexes before and after renovation of the alcohol column

	Feed rate, $kg\cdot h^{-1}$	Normal production span, month	Top composition alcohol, wt%	Kettle composition alcohol, wt%
Before revamp	22,400	4-6	50	0.08
After revamp	35,700	18	55	0.01

2. W. Lianying, H. Yangdong and W. Ya, *Korean J. Chem. Eng.*, **24**, 556 (2007).
3. J. G. Segovia-Hernandez, A. Bonilla-Petriciolet and L. I. Salcedo-Estrada, *Korean J. Chem. Eng.*, **23**, 689 (2006).
4. Y. H. Kim, K. S. Hwang and M. Nakaiwa, *Korean J. Chem. Eng.*, **21**, 1098 (2004).
5. J. Cho and D. M. Kim, *Korean J. Chem. Eng.*, **24**, 438 (2007).
6. J. Cho and J. K. Jeon, *Korean J. Chem. Eng.*, **23**, 1 (2006).
7. S. Mahiout and A. Vogelpohl, *Ger. Chem. Eng.*, **8**, 152 (1985).
8. S. Bocker and G. Ronge, *Chem. Eng. Technol.*, **28**, 25 (2005).
9. Z. G. Lei, B. H. Chen and Z. W. Ding, *Special distillation processes*, Elsevier B.V., Amsterdam (2005).
10. Y. Pi, Q. S. Li, Z. D. Liang and H. Y. Liu, *Advanced Materials Research*, **11-12**, 717 (2006).
11. Y. F. Zhou, J. F. Shi, X. M. Wang and Y. H. Ye, *Int. Chem. Eng.*, **30**, 642 (1980).
12. D. W. Weiler, F. W. Bonnet and F. W. Leavitt, *Chem. Engrg. Progr*, **67**, 86 (1971).