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Improvement in Stage Efficiency and Reduction in Energy Consumption of Distillation Through Artificial Irrigation

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ABSTRACT

In past few years, distillation remains the widely used separation technique in chemical industries. Requirement of heat energy in terms of steam consumption is the key requirement in distillation. Distillation is perhaps the most energy consuming operation in all the separation processes. The paper in detail focuses on the reduction in energy consumption in distillation through the application of novel technology. The proposed technology offers various advantages over conventional distillation operation including increased stage efficiency, reduced space and height requirement. Additionally this technology brings the considerable reduction in fixed cost in terms of capital cost. The technology offers these advantages at the slightly increased operating cost in terms of artificial irrigation. An attempt is made to improve the stage efficiency, by using an external re-circulating pump. In a reported work, distillation of Methanol-Water binary system is studied. The liquid mixture on the stage is re-circulated back at same stage, by using an external pump. The stage efficiency was estimated by analyzing the liquid on the stage with and without pump and for different reflux ratios. The obtained results are satisfactory and show considerable improvement in stage efficiency.

1. Introduction

In rapidly prospering process industries, raw materials undergo series of unit operations and processes thereby resulting in an intended product. In such processes, often the solvent is used for the extraction of intermediate or final product. These solvents are then recovered by using a distillation. Since these industries are more cautious about hygieneity, perhaps distillation is the only operation that offers desired separation satisfying all the requirements of products. Humphrey et al [1] reported that distillation is a most widely used separation technique in chemical and allied process industries, and approximately account for 90–95% of all the separations.

Conventionally distillation is carried out either using packed column or plate column. Particularly in pharmaceutical industries, the manufacturing of products may change as per the seasonal requirements. Therefore different solvents may have to be used, at different stages and for different products. Further, it is impractical to use different separating columns for every system, since columns consume lot of space for its installation. Also the capital investments in terms of fixed cost would be high [2]. Therefore it's quite practicable to separate various binary systems in a single column. Thus it is clear that solvent recovery from any particular mixture needs to be achieved by changing the operating parameters rather than adding new columns.

In conventional distillation, for single columns, minimum energy requirements generally corresponds to minimum reflux and/or boil-up ratios and an infinite number of equilibrium stages so that the column just performs the desired separation [3]. High purity of distillate corresponds to higher reflux ratio which in turn needs subsequent increase in reboiler duty. On the other hand, minimum reflux ratio requires minimum thermal energy in reboiler duty resulting in a poor quality of product [4]. The main reason behind delivering poor quality product is lower reflux flow rate, which may not wet all the packing's in a column; thereby resulting in poor mass transfer [5-7]. To overcome this deficiency, a new system with artificial irrigation resulting in a sufficient wetness in a column is developed. In this novel technique, the liquid on the stage will be recirculated back to same stage by artificial irrigation through sparger using external pump. The new arrangement results in reduction of reflux ratio which ultimately reduce the reboiler duty.

This reduction in reboiler duty causes subsequent reduction in steam consumption thereby resulting in reduced energy consumption. As compared to conventional distillation, the energy requirement of the newly developed process for same separation is comparatively low. Also the purity of the product can be altered just by opting for artificial irrigation. The re-circulating liquid was irrigated on so called plate by using sparger. The stage efficiency is calculated by analysing the liquid on the stage with and without pump and for different reflux ratios. Stage efficiency shows considerable improvement in presence of pump. Murphree plate efficiency was calculated for each case. It was observed that better separation can be achieved by means of irrigation. Patil et al [8] also discussed the other possibilities of reduction in steam consumption by exploring the performance of tray and concluded that movable valve trays are most suitable for various types of distillation applications.

2. Experimental Methods

2.1 Design Aspects

Binary distillation columns are generally designed by using McCabe Thiele method. The method is based on material balance equations, which assumes constant molar overflow. The proposed technology is developed by using same methodology incorporating practical corrections to existing ones. Practical corrections include non-ideal flow pattern (such as cross-flow on plate), bypassing, channelling etc. All these non-idealities are related to a single factor known as Murphree efficiency, therefore improvement in Murphree efficiency is the prime objective of this development and hence the performance is evaluated in terms of Murphree efficiency. Artificial irrigation is expected to allow use of each stage in any direction either vertical or horizontal.

Thus, entire distillation column can be spread horizontally in sections. Purity of end products is greatly decided by column hydraulics [9], which requires much higher refluxes in practice. Artificial irrigation is expected to remove these non-idealities. This facility allows the use of single column

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for multiple pairs of solvents. In a developed methodology, a stage consists of a liquid holding horizontal glass vessel. The liquid in a stage is re-circulated using an external micro-axial flow pump since these pumps are capable of delivering higher flow at low head. The pump may be used for any number of stages, however for evaluation of performance, only one stage is considered. Vapours to stage are fed in cross-flow manner.

The liquid holding capacity of stage can be adjusted according to requirement. Since the liquid is continuously re-circulated, it can be considered as completely mixed phase and hence the requirements of packing or plate are eliminated. Additionally, due to application of sparger, the possibilities of channelling, bypassing (as in case of packed columns) and on stage in-homogeneity (as in case of plate columns) are eliminated. The performance is evaluated by varying the reflux ratio. In present work, the evaluation of the developed technology is checked by testing it with continuous distillation of binary Methanol-Water mixture. The distillation is carried with and without running the irrigation pump. The stage efficiency in each case was calculated and compared.

2.2 Experimental Setup

Newly developed distillation set up mainly consists of:

1. Reboiler
2. Horizontal Stage Assembly
3. Condenser.

2.2.1 Reboiler

Reboiler is a 4 necked flask with 5 L capacity. Out of these four necks, largest diameter neck connects the reboiler to horizontal stage through 1 inch glass tube carrying vapours. The second neck is connected to horizontal stage through 0.5 inch glass tube carrying liquid. The third neck is provided for the removal of sample whereas fourth neck is provided for thermowell to facilitate RTD to measure the temperature of reboiler liquid. The reboiler assembly is as shown in Fig. 1. The complete removal of reboiler bottom is accomplished by using siphon facility. Reboiler mixture is heated using heating coils (Capacity – 2 kW).

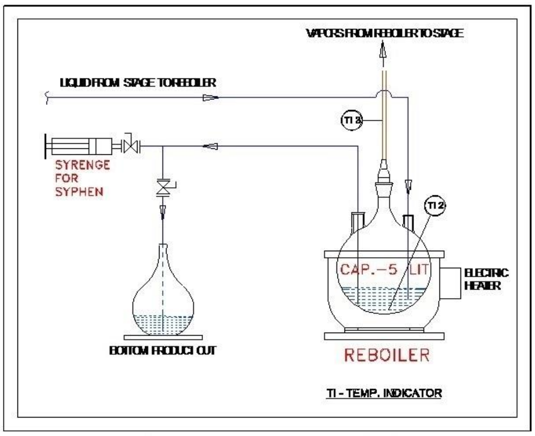

Fig. 1. Schematic diagram of Reboiler

2.2.2 Horizontal Stage Assembly

The horizontal stage assembly is as shown in Fig. 2. It consists of a horizontal cylindrical glass vessel, 4 inches in diameter and 12 inches in length with vapour inlet from re-boiler and exit to the condenser. The horizontal stage assembly has five nozzles thereby facilitating in and out flow of liquid and vapour. The three nozzles with 1 inch size are utilized for injection of feed, vapour in flow from reboiler and vapour out flow to condenser. The two 0.5 inch nozzles are used for the facilitation of transfer of stage liquid to reboiler and to pump for recirculation back to stage. The liquid on stage is then re-circulated and irrigated to same stage through external self-priming pump. Pump has a discharge capacity of 0.5 litre/sec and head of 21 meter (In practice 1 meter head is enough). Irrigation is achieved through sparger assembly as shown in figure. Sparger is a brass tube having 4 holes of 1.5 mm diameter. The bottom flow rate to reboiler is controlled by screw clamp as shown in figure and the flow rate is measured manually. The entire assembly behaves and treated like a single stage/plate in distillation column.

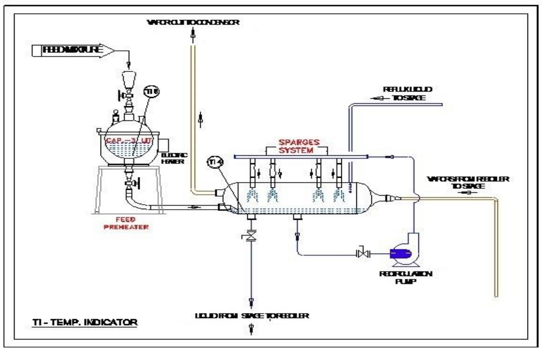

Fig. 2 Schematic diagram for Stage Assembly

2.2.3 Condenser

Condenser is used for the condensation of hot vapours from horizontal stage. This condensation is accomplished by supplying cooling water at the flowrate of 100 mL/s. Condenser is provided with a tap water supply for cooling. Tap water was pumped to the condenser from a pot of capacity 50 litres. Water level in the pot was maintained using tap water make up. It eliminates the possibility of escape of uncondensed vapour, due to the fluctuations in tap water flow. The condenser assembly is as shown in Fig. 3. A round bottom flask is provided at the bottom of condenser in such a way that all the condensate is collected in it. Round bottom flask is provided with liquid discharge at the bottom. This discharge is further divided into the distillate and reflux. The required reflux ratio was maintained by adjusting the screw clamp. Both flow rates could be measured to estimate the reflux ratio.

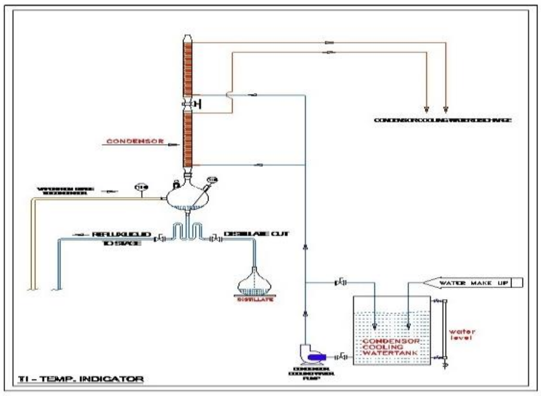

Fig. 3 Schematic diagram for Condenser

The entire experimental set up including reboiler, horizontal stage assembly and condenser is as shown in Fig. 4. To prevent the heat losses and hence to avoid the bias in results complete assembly inclusive of all the liquid and vapour lines is insulated by asbestos ropes. Valves are provided at proper places to ensure the smooth control of process.

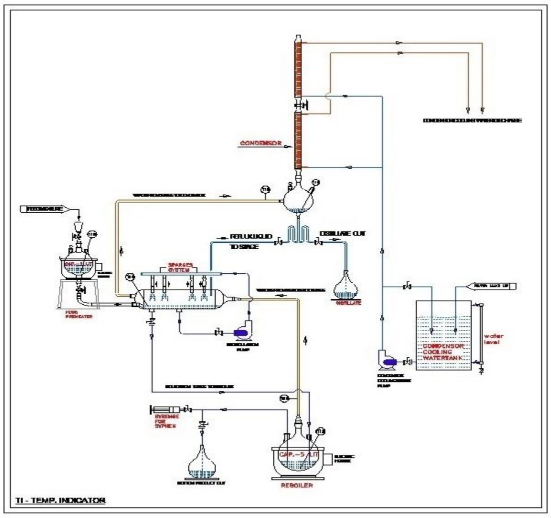

Fig. 4 Experimental Setup including reboiler, horizontal stage assembly and condenser

2.3 Experimental Procedure

The entire setup is established as shown in Fig. 4. The experiment is performed by using a binary methanol-water system. The reboiler of 5 litres capacity is charged with 30 % Methanol, by mol. The mixture is continuously heated using heating mantle. The stage is charged with 500 mL condensate through reflux. Once the stage was filled, the liquid flow from stage to reboiler was started. The temperatures of re-boiler, stage and condensate were measured and recorded. The steady state was ensured when all these temperatures remain constant at least for 15 minutes. Samples of the liquid flowing from condenser to stage and stage to re-boiler were withdrawn at a time of interval 15 min. The concentration of methanol in distillate is predicted. The re-circulating pump is then started for irrigation in stage. Again after ensuring the steady state operation for at least 15 minutes, liquid samples at same locations are collected and analyzed. Finally the concentration of methanol in distillate is predicted. Then the efficiency of distillation in presence and absence of irrigation is calculated.

2.4 Technical and Economic Aspects

As far as technical and economic aspects are considered, high energy consumption is perhaps the only weakness in distillation. Besides this, distillation remains the most applied separation technology in chemical and allied process industries. Particularly the separation of ethanol-water binary system is the most studied topic in the arena of distillation. Over the years, lot of work was published on energy saving in distillation but all the work done is either on reduction in energy consumption within a single distillation column or on column to column heat integration in a whole process.

Low thermodynamic efficiency, approximately 5–20% is also the serious concern in distillation [10]. Humphrey et al [1] further define the thermodynamic efficiency of separation process as the ratio of the minimum amount of thermodynamic work required for the desired separation to the minimum amount of energy required for the said separation. It demands the significant efforts to increase the efficiency of column either thermodynamic or stage (Murphree). Thermodynamic efficiency in distillation could be significantly enhanced by heat integration. The heat integration could either be internal or external. The internal heat integration in a column is achieved either through direct vapour recompression system [11-13] or through HIDiC (Heat Integrated Distillation Column) [14, 15]. The external heat integration could be achieved through column to column coupling in a process.

For distillation, it is assumed that vapours leaving each tray are in equilibrium with liquid leaving same tray. This analysis assumes that each tray is operating at 100% efficiency. However due to insufficient contact time and degree of mixing, in actual practice trays are not perform at 100% efficiency. It means, in actual operation, vapours and liquid leaving the same tray are not in equilibrium. Therefore in order to achieve desired separation, additional trays need to be incorporated in a distillation column. The actual number of trays needs to be added is calculated by using trays efficiency. As far as tray efficiencies are concerned, two efficiencies namely overall efficiency and Murphree efficiency are considered. Overall tray efficiency is defined as the ratio of number of theoretical trays to number of actual trays [16, 17]. The overall efficiency can be calculated by Eq. 1. The operating and equilibrium lines for binary distillation are as shown in Fig. 5.

$$E_o = \left(\frac{\text{No.of therotical trays}}{\text{No.of actual trays}}\right) \text{X } 100 \tag{1}$$

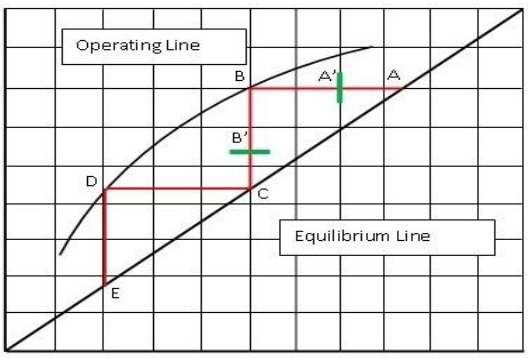

Fig. 5 Operating and equilibrium lines in binary distillation

Whereas, Murphree efficiency is based on semi-theoretical model which assumes that vapour between the trays are well mixed (uniform in composition), liquid in the downcomer is well mixed (uniform in composition), liquid on the tray is also well mixed (uniform composition) and is of the same composition of liquid leaving the tray. Murphree efficiency can be based either on liquid phase or vapour phase and can be calculated as follows.

Based on vapour phase $$E_{mv} = \left(\frac{CB'}{CB}\right) \text{X } 100 \tag{2}$$

Based on liquid phase $$E_{ml} = \frac{X_n - X_{n-1}}{X_n^* - X_{n-1}} \tag{3}$$

$$E_{ml} = \left(\frac{AA'}{AB}\right) \text{X } 100 \tag{4}$$

In a present study, Murphree efficiency based on liquid phase is considered since sampling and analysis of liquid phase is much easy as compared to that of the vapour phase. Alternatively Murphree efficiency can also be calculated graphically using Eq. 2 and 4 from Fig. 5.

3. Result and Discussion

The experimental setup as shown in Fig. 4 is established and binary distillation of methanol-water system is carried out. Experiments are carried out for continuous distillation and with variable reflux ratios. The performance of distillation in terms concentration of more volatile component in distillate is evaluated. The efficiency of the distillation with variable reflux ratios, and in presence and absence of irrigation is calculated. Details of distillate analysis to determine composition of MVC (methanol) is tabulated in Table 1, whereas details of the liquid sample analysis for the cases of total reflux, variable reflux are tabularized in Table 2. From Table 1, it is observed that irrigation in stage by external re-circulation pump offers significant enhancement in rectification and is clearly observed in terms of increased MVC (methanol) concentration in distillate.

Table 1 Distillation under total reflux

Sample No.	Sample Location	Pump Condition	Composition of MVC (Methanol) in Distillate (W/W)
1	Condensate Flask	Off	85.8%
2	Condensate Flask	On	89.3%

Table 2 Results of sample analysis for estimation of stage efficiency

Distillation Condition	Composition of Methanol In/On (Mole Fraction)				Murphree Efficiency
	Feed	Re-boiler	Stage	Condenser	
Total Reflux Pump Off	0.4112	0.168	0.5183	0.6324	0.29
Total Reflux Pump On	0.4112	0.158	0.50	0.65	0.37
Continuous Distillation D = 0.072 mL/s, B = 0.1 mL/s	0.4112	0.170	0.49	0.68	0.50
Continuous Distillation D = 0.175 mL/s, B = 0.23 mL/s	0.4112	0.168	0.47	0.68	0.54
Continuous Distillation D = 0.43 mL/s, B = 0.32 mL/s	0.4112	0.160	0.46	0.67	0.56
Total Reflux Pump Off	0.29	0.200	0.61	0.70	0.22
Total Reflux Pump On	0.29	0.180	0.59	0.71	0.30
Total Reflux Pump Off	0.20	0.175	0.60	0.69	0.23
Total Reflux Pump On	0.20	0.16	0.55	0.68	0.34
Continuous Distillation D = 0.2 mL/s, B = 0.15 mL/s	0.20	0.12	0.49	0.55	0.14
Pump On	0.20	0.15	0.50	0.66	0.41
Continuous Distillation D = 0.1 mL/s, B = 0.08 mL/s	0.20	0.12	0.50	0.55	0.16
Pump On	0.20	0.16	0.40	0.52	0.30

4. Conclusion

In the present work a newly developed distillation technique is constructed and tested using a binary methanol-water system. Experiments are carried out for continuous distillation with variable reflux ratios and in presence and absence of irrigation. The efficiency of distillation in both the cases is calculated. It is observed that irrigation in stage by external re-circulation pump offers significant enhancement in rectification irrespective of liquid flow of previous upper stage. This

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enhancement in rectification is clearly observed in terms of increased concentration of MVC (methanol) in distillate resulting in reduced energy consumption.

Abbreviation

MVC -More Volatile Component

Notation

E_o - Overall efficiency
E_{mv} - Murphree Efficiency based on vapour phase
E_{ml} - Murphree Efficiency based on liquid phase
X_n - Actual composition of liquid on n^{th} stage
X_{n^*} - Equilibrium liquid composition of n^{th} stage
X_{n-1} - Actual composition of liquid from n-1^{th} stage.

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