

Simulated Heat Integration Study of Reactive Distillation Column for Ethanol Synthesis

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ABSTRACT: Ethanol is largely used as a solvent in the synthesis of varnishes and perfumes. It is also used as a preservative for biological products, fuel, and gasoline additives. An ethanol-water mixture, normally obtained by fractional distillation yields an azeotropic solution consisting of approximately 95 w% of ethanol. The synthesis of ethanol in a reactive distillation column with the azeotropic feed at 20°C with ethylene oxide as another reactant feed for the reaction with water to produce ethylene glycol was simulated in Aspenplus. Simulated results show 98.82 mol% purity of ethanol in the reactive distillation column. In order to reduce the energy consumption in reactive distillation, a Reactive Heat Integrated Distillation Column (R-HIDiC) is proposed for the same purity of ethanol product. From the simulated results, it has been found that the overall energy utilization in the proposed distillation column is reduced by 65% as compared to the conventional reactive distillation column. The pressure profile, temperature profile, and concentration profile of the reactive distillation column and the column proposed are also compared.

KEYWORDS: Reactive heat-integrated distillation column; Simulation; Ethylene oxide; Ethylene glycol; Ethanol-water.

INTRODUCTION

There are various distillation techniques such as simple distillation, flash distillation, rectification, pressure swing distillation, azeotropic distillation, and Reactive Distillation (RD). Among these techniques, RD is an important method for some conventional processes, especially the equilibrium-limited reactions such as esterifications [1]. Numerous relevant studies [2-6] have discussed steady-state simulations and binary close-boiling mixtures. Glavit [7] found that the depleting resources of nonrenewable fuels compel the engineers to reduce fuel and electricity consumption together

with materials requirements. The author suggested that pinch analysis with optimization and modeling of chemical processes should be mandatory for every plant. Multicomponent distillation is well presented by Hourichi and Iwakabe in 2006 [8, 9]. Antonio and Jorge [10] proposed that the separation of the ethanol/water system can be carried out using an Internally Heat Integrated Pressure Swing Distillation (IHIPSD) scheme, which is able to attain high ethanol purity and recovery. Moreover, energy can also be saved with this scheme because of internal heat integration between the different sections.

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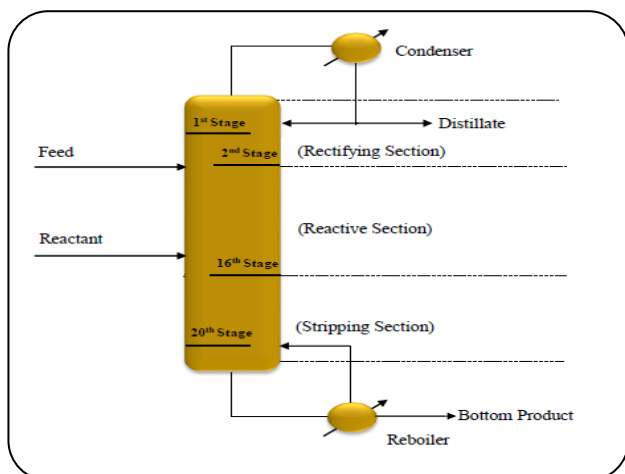


Fig. 1: Process flow diagram of Ethanol Separation from water in conventional reactive distillation

Zhu *et al.* [11] also suggested that heat integrated Pressure Swing Distillation (PSD) is more economical than conventional PSD. Li *et al.* [12] suggested that even a partially heat integrated process is more energy saving than that without heat integration. They showed a reduction in energy consumption by 19.79%. Hernández *et al.* [13] performed energy efficiency on indirect thermally coupled distillation sequence and suggested that the remixing of the intermediate component observed in the conventional distillation sequences gets reduced remarkably upon optimizing the interconnecting stream flow rate exiting from the second column.

Reactive distillation consists of both reaction and distillation in a single column, as shown in Fig. 1, in which reactants get converted to products, which are separated out in the same column [14-17]. This process has the advantages over the conventional one such as energy efficient, solvent consumption, the capital cost (because of less number of equipment's are required) and removal of hot spot problem by liquid evaporation [17]. The application of reactive distillation is constrained by operating conditions *viz.* temperature and pressure and difficulties in proper residence time characteristics [16]. Reactive distillation shows good energy savings for the systems in which the reaction is fast and the reaction temperature is suitable for separation [18]. Tavan and Hosseini [19] have done a parametric study on a reactive distillation column to break the azeotrope of the ethanol (EtOH)/water system.

In other configurations, two individual columns or heat integration of various sections are used to design

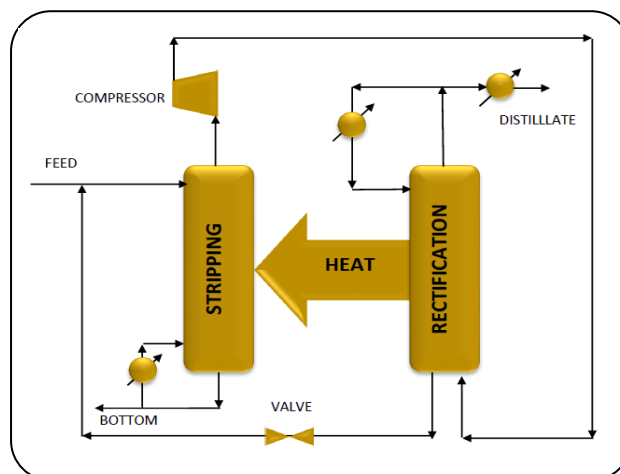


Fig. 2: Schematic structure of HIDiC.

more energy efficient columns. In a distillation column with internal heat integration, heat from hot stages is exchanged to the cold stages. In this configuration, the column is separated into two sections as shown in Fig. 2: low temperature/pressure section and a high temperature/pressure section. Such a column is designed to enhance the heat exchange between high pressure to the low pressure section. This type of column is typically termed as Heat Integrated Distillation Column (HIDiC). Gadalla [4] analyzed the HIDiC design. Olujić [20] carried out an economic analysis of HIDiC. Several authors [2, 3, 21-24] studied the basic concepts of HIDiC such as energy saving and thermodynamic efficiency. In these studies, the authors showed that there is no need for condenser and reboiler to operate the binary HIDiC. Most of the research articles have carried out a study on binary HIDiC, however, some studies have focused on multi-component systems. The applicability of reactive distillation for a certain reaction depends on several factors *viz.* volatilities of products and reactants in addition to the operating temperature [14-16]. The column's internal pieces are fabricated in such a way that they permit heat exchange between the stripping section and the rectifying section, which makes the vapor condensed in the rectifying section and liquid vaporized in the bottom section. HIDiC is a potential research subject in the study of reactive distillation. The heat of reaction can enhance the HIDiC performance. In fact, in the stripping section, the liquid is mostly vaporized due to heat of exothermic reactions, however, in the rectifying section, the vapor is condensed due to heat utilized

in the endothermic reactions, which makes more reflux of liquid in the column. This configuration leads to better energy efficient scheme; such type of configuration is known as the Reactive Heat Integrated Distillation Column (R-HIDiC).

The authors did not find any paper on the reactive heat integration distillation column for ethanol synthesis. In this paper, a simulation study has been carried out for heat integration of a reactive distillation column for the production of ethanol in Aspen v8.8. The energy consumption has been computed and temperature profile, composition profile, and pressure profile have been compared for both conventional RD column and R-HIDiC.

THEORITICAL SECTION

Process description

HIDiC column combines the benefits of Vapor Recompression Cycle (VRC) and adiabatic operations to lower the energy requirements [25]. In the rectifying section of HIDiC, the temperature is raised by making the arrangement such that the rectifying section is in direct contact with the stripping section. This arrangement provides the required energy that is generally added or withdrawn from the column. Vanaki and Eslamloueyan [26] proposed a model and the solution algorithm for the *tert*-amyl methyl ether (TAME) production process and found that the overall energy utilization in the R-HIDiC proposed is approximately 22% less as compared to that in the conventional reactive distillation column. For designing the HIDiC configuration, the conventional distillation column sections are separated but are made concentric to facilitate the heat exchange between the sections [4].

According to Nakaiwa *et al.* [2], equal numbers of trays are thermally coupled to both the sections of the column keeping heat transfer required between the stages as fixed. They formulated a Mixed Integer NonLinear Programming (MINLP) problem to design HIDiC with a finite number of heat transfer stages. Therefore, a novel approach for process intensification was evolved by adopting reactive distillation and heat integration principles, resulting in the heat integrated reactive distillation column (R-HIDiC) [27]. The proposed configuration of R-HIDiC is shown in Fig. 3.

Heat integration of a RD column consists of the utilization of hot process streams to transfer heat with the cold process streams. In this type of configuration,

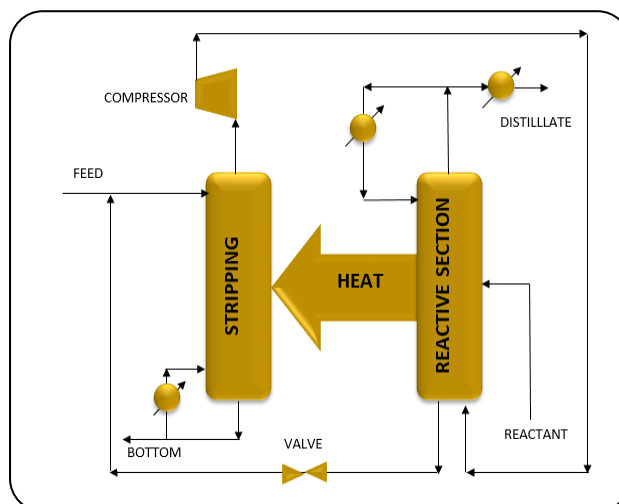


Fig. 3: A schematic diagram of R-HIDiC configuration.

the heat exchange from the rectifying section to the stripping section makes the rectifying section operable at higher pressure and temperature. The heat required to vaporize the liquid in the stripping section is extracted from the rectifying section, which reduces the energy charge on the reboiler.

In the reactive zone of R-HIDiC, ethylene oxide (EO) reacts with water and form ethylene glycol (EG). The reaction kinetics [28] of EG synthesis is as follows:



$$r_{\text{EG}} = 3.15 \exp\left(\frac{-9547}{T}\right) x_{\text{EO}} x_{\text{water}}$$

Where r_{EG} is the ethylene glycol reaction rate in mole/cm³s.

Where, x_{EO} and x_{water} is the liquid mole fraction of EO and water, respectively.

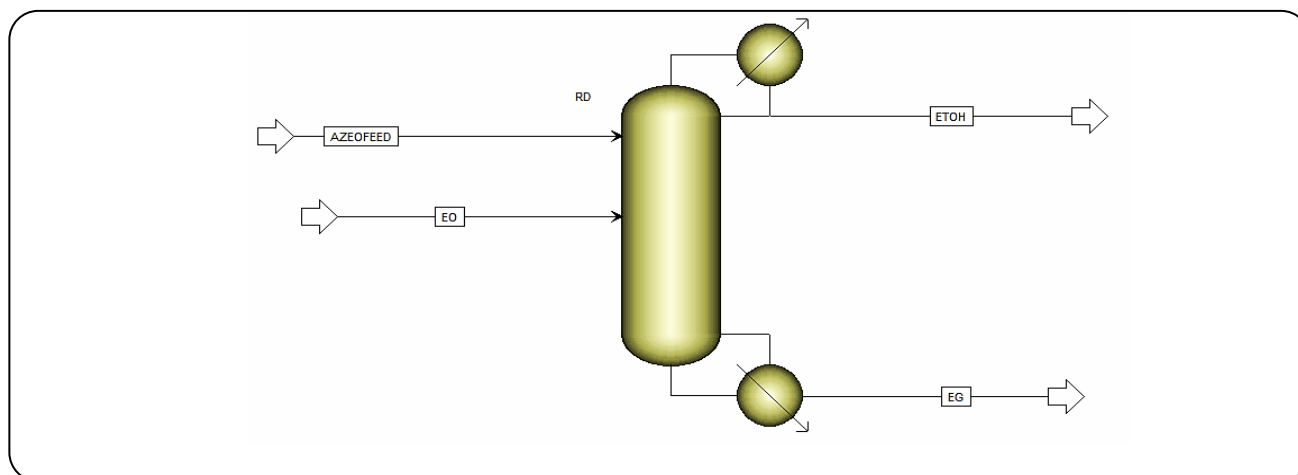
Ethylene oxide can further react with ethylene glycol to form the undesired diethylene glycol as a by-product. Both reactions are reversible and exothermic, which occur in the range of moderate temperatures. The rate of the by-product formation is much lower than that produced by the main reaction and, therefore, the effect of the side reaction can be ignored on the component profiles. This system can operate in the atmospheric as well as high pressures.

Simulation study

NRTL activity coefficient and equilibrium stage model were considered in Aspen plus V8.8 software

Table 1: Physical property parameters of NRTL model.

Component i	Water	Water	Water	Ethanol
Component j	Ethanol	Ethyl-01	Ethyl-02	Ethyl-02
Temperature units	°C	°C	°C	°C
Property parameters				
A_{ij}	3.4578	0	0.3479	14.8422
A_{ji}	-0.8009	0	-0.0567	-0.1115
B_{ij}	-586.0809	188.6878	34.8234	-4664.4058
B_{ji}	246.18	868.971	-147.1373	157.5937
C_{ij}	0.3	0.3	0.3	0.47
D_{ij}	0	0	0	0
E_{ij}	0	0	0	0
E_{ji}	0	0	0	0
F_{ij}	0	0	0	0
F_{ji}	0	0	0	0
T_{LOWER}	24.99	5	30.4	50
T_{UPPER}	100	50	196.7	190

**Fig.4: Flow sheet of Reactive Distillation Column (RD).**

in order to have a comparison between the RD column and the suggested R-HIDiC column. All the properties of the NRTL model are given in Table 1. In this simulation study, the configurations were designed to produce 0.051 kmol/s of ethanol from an initial mixture of ethanol–water consisting of 84 mol% ethanol. The azeotropic feed and EO at the atmospheric pressure and 20 °C were considered for simulation. For both the processes, the feed rate of EO was 9.5×10^{-3} kmol/s.

The RD column as shown in Fig. 4 used for ethanol manufacture has twenty equilibrium trays; one tray in the rectifying zone (stage 1), fifteen trays in the reaction zone (stages 2–16) and four trays in the stripping zone (stages 17–20). Azeotropic mixture of ethanol and water is feed at the 2nd stage and EO is fed at the 16th stage. The column specifications include a distillate product rate of 0.051 kmol/s and a reflux ratio of 10. Column operates at 1.0×10^5 Pa pressure. The feed and product conditions of the stream are given in Table 2.

Table 2: Specifications of Feed and Product streams for RD configuration.

Stream ID	Units	Azeo feed	EG	EO	ETOH
Temperature	°C	20.0	197.1	20.0	78.4
Pressure	Pa	1.013×10^5	1.013×10^5	1.013×10^5	1.013×10^5
Vapor Frac		0.000	0.000	1.000	0.000
Mole Flow	kmol/s	0.06	9.0×10^{-3}	9.3×10^{-3}	0.05
Mass Flow	kg/s	2.494	0.559	2.345	2.494
Volume Flow	m ³ /s	3.05×10^{-3}	5.9×10^{-4}	3.19×10^{-3}	3.05×10^{-3}
Enthalpy	kcal/s	-4.0×10^3	-9.25×10^3	-117.5	-3.3×10^3
Mole Flow	kmol/s				
Water		9.6×10^{-3}	trace		3.1×10^{-4}
Ethanol		0.0504	trace		0.0504
Ethyl-01 (EO)			trace	9.3×10^{-3}	1.08×10^{-3}
Ethyl-03 (EG)			9×10^{-3}		2.78×10^{-4}

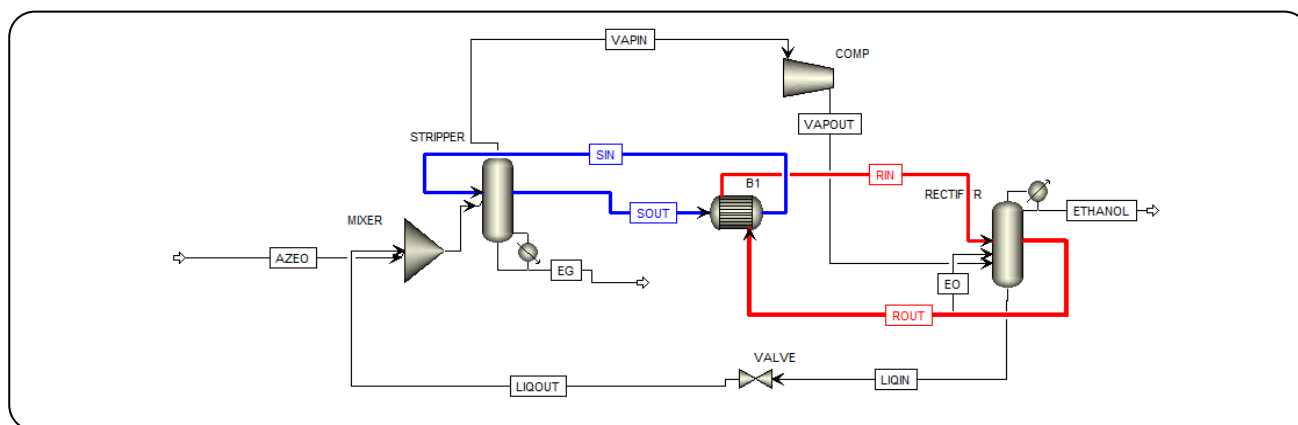


Fig.5: Flow sheet of Reactive Heat-Integrated Distillation Column (R-HIDiC).

The proposed R-HIDiC as shown in Figure 5 comprises two sections: the low-pressure rectifying zone containing the first fifteen trays, and the high-pressure stripping zone containing five trays from tray 16 to 20. The reaction occurs in the rectifying zone from the 3rd stage to the 14th stage. The rectifying and stripping zones are operated at 2.5 and 1.0×10^5 Pa, respectively.

For simulation purpose, rectification section was assumed to be a column with no reboiler having distillate product flow rate at 0.051 kmol/s and the stripping section was assumed to be a column with no condenser having bottom product rate at 9.2×10^{-3} kmol/s. The vapor stream coming out from the stripping section is first compressed and then transported to the last tray

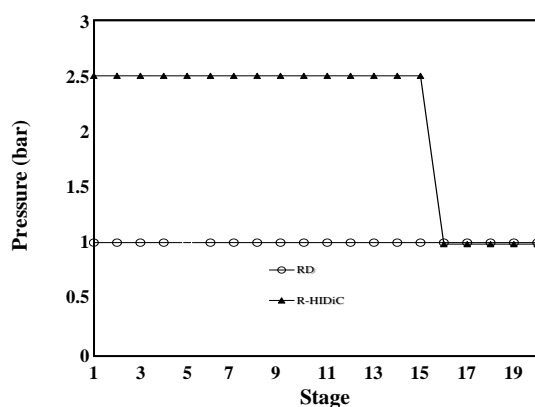
of the rectifying section. The compressor was specified to increase the pressure up to 2.5×10^5 Pa. Before entering into the top section of the stripping section, the liquid exiting from the rectifying section is depressurized. The valve was specified to have an outlet pressure of 1.0×10^5 bar. The second stage of the stripping section at a temperature of about 82 °C (Stream SOUT) was thermally coupled with the 11th stage of the rectifying section at a temperature of about 103 °C (Stream ROUT). The column feeds and product stream conditions are given in Table 3.

RESULTS AND DISCUSSION

In the RD column, the pressure is 1.0×10^5 Pa throughout the column without any internal heat integration among

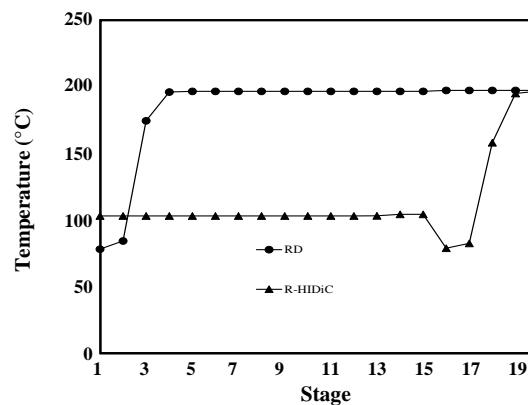
Table 3: Specifications of Feed and Product streams for R-HIDiC configuration.

Stream ID	Unit	Azeo feed	Bottom	Distillate	EO
Temperature	°C	20.0	196.5	103.1	20.0
Pressure	Pa	1.0×10^5	1.0×10^5	2.5×10^5	2.5×10^5
Vapor Frac		0.000	0.000	0.000	0.000
Mole Flow	kmol/s	0.06	9.21×10^{-3}	0.05	9.3×10^{-3}
Mass Flow	kg/s	2.490	0.572	2.334	0.409
Volume Flow	m^3/s	3.05×10^{-3}	6.02×10^{-4}	3.33×10^{-3}	4.68×10^{-4}
Enthalpy	kcal/s	-4.0×10^3	-9.46×10^2	-3.24×10^3	-1.73×10^2
Mole Flow	kmol/s				
Water		9.6×10^{-3}	1.944×10^{-6}	3.108×10^{-4}	
Ethanol		0.0504	<0.001	0.0504	
Ethyl-01 (EO)			trace	1.08×10^{-5}	9.3×10^{-3}
Ethyl-03 (EG)			33.167	2.783×10^{-4}	

**Fig. 6: Pressure profiles for RD and R-HIDiC configuration.**

the trays. Fig. 6 illustrates the difference between the pressure profiles of the R-HIDiC proposed and the conventional RD column. The proposed R-HIDiC operates at 1.5×10^5 Pa up to 15th equilibrium tray, and the pressure is decreased to 1.0×10^5 Pa suddenly after the stage 15th so that the temperature of the rectification section is maintained higher than stripping section. The pressure is suddenly reduced to 1.0×10^5 Pa after the stage, 15th to make the liquid entering the stripping section to be at the same pressure as that of the stripping section.

Temperature profiles for RD and R-HIDiC are shown in Fig. 7. The temperature profile of conventional RD shows that rectifying section has 78.37 °C after then the temperature gradually increases to about 200 °C

**Fig. 7: Temperature profiles for RD and R-HIDiC configuration.**

in the reactive section and then it becomes constant throughout the stripping section.

The temperature in R-HIDiC is constant (100 °C) throughout the rectifying section. In the stripping section, the initial temperature is low in comparison to the rectifying section because of the azeotropic feed and the liquid from the rectifying section are introduced at the 16th stage. After the 17th stage, the temperature increases as we move down the column because of the reboiler which is heating the bottom liquid. The difference in the temperature of R-HIDiC's top stages of the stripping column and rectification column is mainly due to pressure difference. Moreover, the exothermic reaction in the rectifying section also contributes to this difference

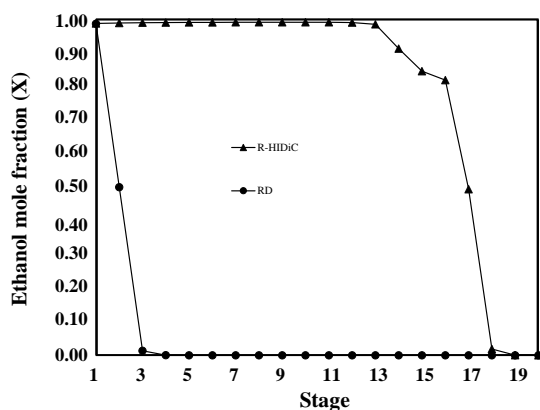


Fig. 8: Ethanol concentration profile for RD and R-HIDiC in liquid.

in temperature. Since the difference is about 23°C, stages 11th and 17th are thermally coupled to the heat exchanger. The simulation shows that the reflux ratio required in the R-HIDiC is 3.25, however, it is 10 in RD and therefore a reduction of 68.16% in reboiler heat duty is achieved in R-HIDiC.

Fig. 8 shows the comparison between ethanol concentration for the R-HIDiC proposed and the conventional reactive distillation (RD). It shows that ethanol concentration in product distillate and the bottom is almost the same for both RD and R-HIDiC.

Fig. 9 shows the concentration variation of ethanol, water, EO and EG in the RD column. It is observed that ethanol concentration increases towards the top while EG concentration increases towards the bottom due to more volatility of ethanol. Water and EO are less in concentration throughout the column as they react to form EG.

The energy consumptions in the conventional reactive distillation column and R-HIDiC proposed have been compared as shown in Table 4. Total energy saved by the proposed R-HIDiC is about 65.2% as compared to that in the conventional reactive distillation. The energy consumption in the reboiler is 68.16% and that in the condenser is 65.40% less than those in the conventional reactive distillation column. Some part of the energy needed in the proposed column is the work done in the compressor that is about 4.37% of the overall energy consumed in the R-HIDiC. Therefore, the energy consumed in the compressor is not a significant part of that in R-HIDiC.

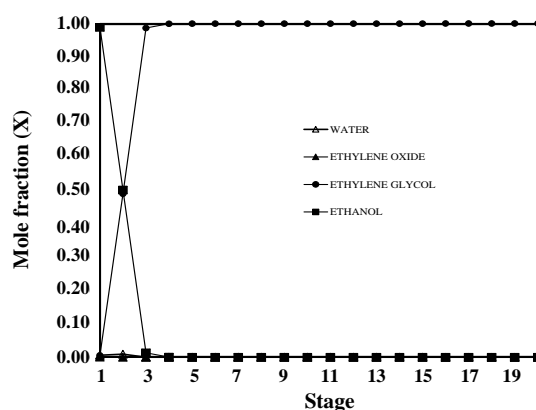


Fig. 9: Concentration profiles of water, ethylene oxide (EO), ethylene glycol (EG) and ethanol for RD column in liquid.

SNOWBALL EFFECT

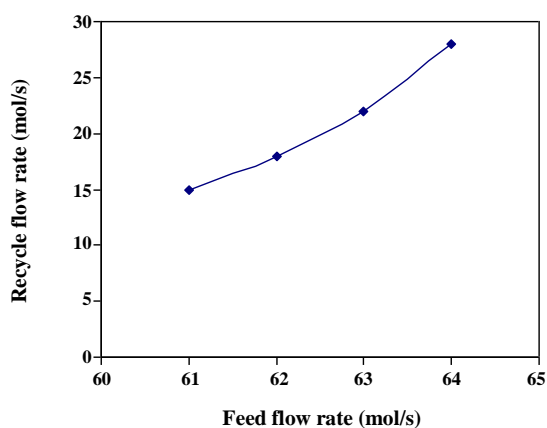
The processes with recycle flow show a high sensitivity of the recycle flow rate to small changes in input flow, known as the snowball effect. It is mainly a steady state effect and continuous operation around the snowball region can lead to mal-operation and eventually plant upset [29]. Therefore, while designing these processes, the snowball effects must be analysed. For the system of the present the snowball effect has been analyzed as shown in Fig. 10. It is clear that on increasing feed flow rate, the required recycle rate increases exponentially, which may lead to varying the high value of recycling rate requirement for the desired purity.

CONCLUSIONS

In the R-HIDiC configuration proposed, the simulated results show that the overall energy utilization is about 65.2% less than that in the case of the conventional reactive distillation column. It was found that 68.16 % and 65.4% energy is saved in the reboiler and condenser, respectively for the R-HIDiC configuration. In addition, 1.658×10^2 kcal/s energy is also consumed by the compressor in the R-HIDiC proposed. The energy saved in the R-HIDiC is because of heat integration and change in the pressure level in the rectifying and stripping sections, i.e., 1.5×10^5 Pa. Simulation results show that ethanol purity is 98.8 mol% in both R-HIDiC and RD. Also, the reflux flow rate required in the R-HIDiC is 67.5% less than that in the conventional reactive distillation column for the desired purity, because of the higher temperature in the rectifying section. The R-HIDiC

Table 4: Energy consumption of the accessories in the R-HIDiC and Conventional distillation column.

	R-HIDiC	Conventional RD	Reduction %
Condenser Duty (kcal/s)	1.852×10^3	5.354×10^3	65.40
Reboiler Duty (kcal/s)	1.669×10^3	5.241×10^3	68.16
Compressor Work (kcal/s)	1.658×10^2	-----	-----
Total (kcal/s)	3.686×10^3	10.596×10^3	65.2

**Fig. 10: Snowball Effect**

proposed is more energy efficient in comparison to the conventional reactive distillation, still, its controllability and operation performance can be more challenging than the conventional reactive column. The snowball effect has been analyzed for this system.

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