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Overview of Distillation Techniques and Process in Recovery and Reuse of Volatile Liquids

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Abstract: Distillation is a process in which miscible liquids are separated based on their physical properties, specifically, relative volatilities. A liquid can be classified as volatile when it is readily vaporized at a relatively low temperature. The boiling of the more volatile components of the mixture drives the distillation process. When the vapor is cooled, the more volatile material condenses in a greater proportion than the less volatile material. It is a unit operation in which constituents of liquid mixture are separated by using thermal energy depends upon the relative volatility of constituents by partial vaporization and condensation. Distillation with rectification or Fractional distillation gives almost pure product. The product removed from top is called the distillate or overhead product and that removed from the bottom is called the bottom product. This unit operation is also termed as fractional distillation or fractionation.

I. INTRODUCTION

Distillation is a widely used operation in the petroleum, chemical, petrochemical, beverage and pharmaceutical industries. It is important not only for the development of new products, but also for the recovery and reuse of volatile liquids. For example, pharmaceutical manufacturers use large quantities of solvents, most of which can be recovered by distillation with substantial savings in cost and pollution reduction. While distillation is one of the most important unit operations, it is also one of the most energy intensive operations. It is easily the largest consumer of energy in petroleum and petrochemical processing, and so, must be approached with conservation in mind. Distillation is a specialized technology, and the correct design of equipment is not always a simple task. There are many types of distillations based on separation technique used. Here we follow the simple distillation. Distillation is the combination of two unit operations, evaporation and condensation. Evaporation takes place in the column and condensation occurs in condenser.

A. Main Components Of Distillation Columns

Distillation columns are made up of several components, each of which is used either to transfer heat energy or enhance material transfer.

A typical distillation contains several major components

1) A vertical shell where the separation of liquid components is carried out Column internals such as trays/plates and/or packing's which are used to enhance component separations.
2) A reboiler provide the necessary vaporization for the distillation process
3) A condenser to cool and condense the vapour leaving the top of the column.
4) A reflux drum to hold the condensed vapour from the top of the column so that liquid (reflux) can be recycled back to the column.
5) The vertical shell houses the column internals and together with the condenser and reboiler, constitutes a distillation column.

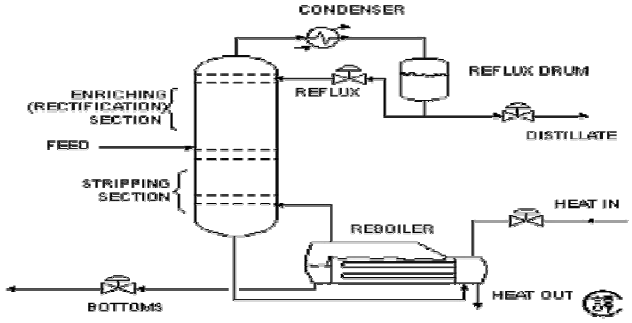

Fig -1.1 A schematic of a typical distillation unit with a single feed and two product stream

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B. Basic Operation And Terminology

The liquid mixture that is to be processed is known as the feed and this is introduced usually somewhere near the middle of the column to a tray known as the feed tray. The feed tray divides the column into a top (enriching or rectification) section and a bottom (stripping) section. The feed flows down the column where it is collected at the bottom in the reboiler.

Heat is supplied to the reboiler to generate vapour. The source of heat input can be any suitable fluid, although in most chemical plants this is normally steam. In refineries, the heating source may be the output streams of other columns. The vapour raised in the reboiler is re-introduced into the unit at the bottom of the column. The liquid removed from the reboiler is known as the bottoms product or simply, bottoms

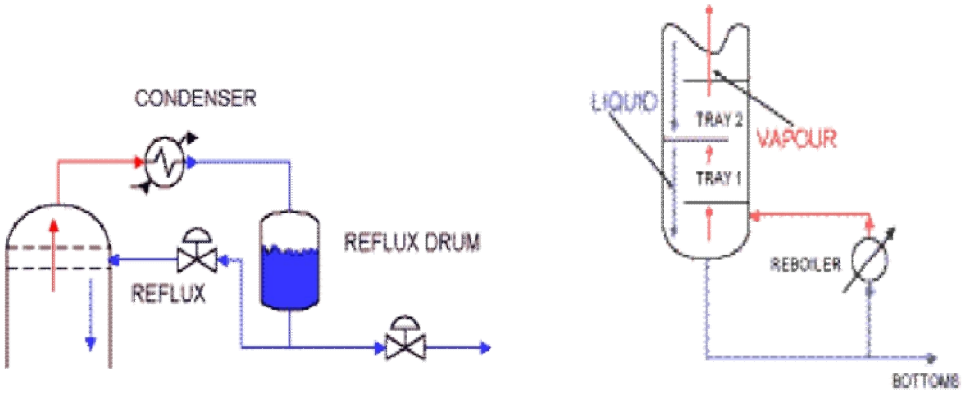


Fig-1.2 schematic diagram of column

The vapour moves up the column, and as it exits the top of the unit, it is cooled by a condenser. The condensed liquid is stored in a holding vessel known as the reflux drum. Some of this liquid is recycled back to the top of the column.

This is called the reflux. The condensed liquid that is removed from the system is known as the distillate or top product.

Thus, there are internal flows of vapour and liquid within the column as well as external flows of feeds and product streams, into and out of the column.

Formulae Used and Definitions:

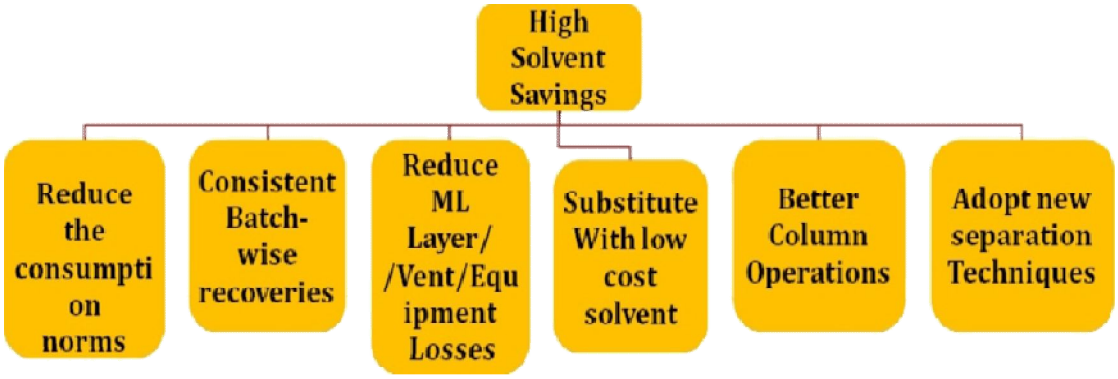
1) Solvent Added: it is the solvent input to the reactor for a particular process. Its unit is kg (for batch processes) or kg/s (continuous processes).

2) Solvent Recovered: it is the solvent recovered after the distillation. Solvent must have the permissible purity percentage for reusing the solvent. Units are same as above.

3) Solvent Consumed: it is the difference between the input solvent and recovered solvent. Mostly it goes in the waste. Ideally, solvent should not be consumed because it only provides the medium for reaction b/w constituents.

4) Consumption Coefficient (CC): the ratio of solvent consumed to the product produced for same reference of time (for the continuous process) or for the same batch (for batch process).

$$CC = \frac{SolventConsumed}{ProductProduced}$$



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5. Total Consumption of solvent over a year: it is the solvent consumed in a financial year. We calculate it by given formula
   = CC × total product produced in financial year
6. Total solvent consumption in a year in rupees:
   = total consumption × budgeted price

C. Quality Control Analysis Report

Mainly Aurobindo Pharmautilises one the most advanced and quick Instrumentation like High Performance Liquid Chromatography(HPLC),Gas Chromatography(GC) and Wet Lab Analysis where they believe Quality is their Prior Motto
Here are some of the results of GC and Wet Lab Analysis of our Designed Distillation Column Samples.

D. Gas Chromatography Results

Here at different Retardation Time the peaks are being obtained which means their levels in the sample are displayed. The more the peak height more is the purity level corresponding to the Retardation Time

PEAK	RETARDATION TIME(IN MINUTES)	COMPOUND NAME	PURITY
1	1.706	ETHANOL	0.05
2	1.997	ACETONE	0.29
3	2.192	IPA	99.61
4	13.621	N-N-DIMETHYL FORAMIDE	0.05

E. Wet Lab Analysis Report

The Wet lab mainly focus on the moisture content and the Assay Content(i.e the solvent level) present in the given sample

SPECIFICATION	COMPOUND	RESULT
IPA	>=99.5%	IPA=99.61
WET CONTENT	<1%	WATER=0.03

F. Process Economics

The main Costs involved in this process are

1) INSTALLATION COST

a) Column Cost – Packing Cost= $\frac{\pi}{4} * (0.4)^2$* column Height* Cost of Packing for $1m^3$

$=\frac{\pi}{4} * (0.4)^2$* 10.71 * 5,00,000

=Rs6.72 Lakhs

For $1m^3$of packing=Rs.5,00,000

b) Steel Cost: Here the material cost is Low Carbon Steeel where the Cost of Steel is=Rs.400.

=3.14* D*L*Thickness of Steel * Density of Steel* Cost of Steel

=3.14 * 0.4* 10.71 * 0.008 * 800 *400

= Rs 3.44 Lakhs

c) Reboiler Cost: Rs. 2 Lakhs

d) Condenser Cost: For a Primary Condenser of Area Between 30-40 m^2–Rs.6 Lakhs.

-For a Secondary Condenser of Area of greater than 40 m^2– Rs 3 Lakhs

e) Cooling Tower Cost:

For 250 TR cooling tower cost-Rs 2.5 Lakhs
For 190.47 TR cooling tower cost= (190.47 * 40)/ 250 = Rs. 1.90 Lakhs

f) Pump Cost:
For a 250 m3 Pump Capacity the motor required is of 40 Hp motor is required
For 190.47 m3 Pump= Capacity of motor=(190.47 * 40)/250 = 30.47 Hp of Motor
Cost of 40 Hp motor=Rs.75000
Cost of 30.47 Hp motor= (75000* 30.47)/40 = Rs.58000

g) Pipeline And Other Costs= Rs 5 Lakhs
h) Storage Tanks Cost-
For 20KL capacity tank , steel required=2.5 tons
For 5KL capacity tank, steel required = 1.5 tons
Cost for 20KL tank=2500 * 50(Cost of Mild Steel/ Kg)= Rs 1.25 Lakhs
For 2 Storage Tanks of 20KL capacity= 1.25 * 2= Rs 2.5 Lakhs
Cost for 5 KL tank= 1500 * 50=Rs 75000
For 2 Storage Tanks of 5KL capacity=75000 * 2 =Rs 1.5 Lakhs
Total Investment Required for overall setup =
Column Cost+ Reboiler Cost+ Condenser Cost+ Cooling Tower Cost + Pump Cost +Pipeline and Other Cost+ Storage Tanks Cost = Rs 33 Lakhs .

2) Operating Cost
Operating Cost = 10% of Total Investment
= 10 % of 33 Lakhs = Rs 3.3 Lakhs

3) Manpower Cost
Manpower Cost= 20% of Total Investment
= 20% of 33 Lakhs = Rs 6.6 Lakhs
Total Fabrication and Structural Cost = Rs 10 Lakhs (approx..)
Therefore, Total Investment for the process= Rs 43 Lakhs.

a) Payback Period: Operating Cost for the treating of IPA column= Rs 4(steam cost= Rs 1.5/Kg ,Power Cost = Rs .1.5 , Manpower Cost from Employee side= Rs 1)
In a month the column is being Operated for 28 Days and 2 Days for Maintainence
So on an Average = 15 Tons of Solvent Collection is being done
=15 Tons * 28 =420 Tons * Rs 10 (per feed)
= Rs 42 Lakhs

So the payback period could be for 28-30 Days and this is feasible.
It also shows that the distillation curve of the IPA/Water mixture with the addition of $MgSO_4$ as a drying agent is similar to the distillation curve of the conventional distillation. The water content of the residue was 0.14 mass%. When the $MgSO_4$ salt was filtered prior to the distillation, the curve in the distillation line is more pronounced. The water content of the residue in this case is 0.02 mass%. The IPA recovery is thus 75 – 70 %.

II. SAFETY ASPECTS

Technical advancement and the risk factor both move almost in the same proportion. The risk factor if not eliminated, can be controlled to a great extent and can be kept in the safety zone if we adhere to the certain safety measures. Right from the time the raw materials enters the factory, it involves unloading, storage, transporting and the participates in the reaction. Different types of hazards are hidden at different operation, if in general instructions of safety are followed strictly we can achieve “accident free work environment” which is the preamble of safety policy.

A. Some of the safety measures which we should strictly follow

1) While charging the reactants through the manhole one should always wear goggles, face shield to avoid any splashing in the eyes.

2) After charging the materials are must to taken to be to see that all the bolts of manhole are tightened fully to avoid any flying of the manhole cover in the case of some pressure which might built in the reactor.

3) Most of the values used in the industry are ball valves, so care is to be taken to see that all these are closed slowly. Abrupt closing of these values causing the danger of line getting damaged or burst at any point due to liquid hammer which may go up to abnormally high pressure.

4) Abrupt heating and closing of the glass-lined reactor should always be avoid otherwise it may get a crack in lining due to thermal shock.

5) Whenever temperature scans are fixed and maintain, temperature in the reactor is important, it should checked locally by mercury thermometer.

6) While discharging solvent in drums check that earthen wire is connected properly.

7) Activated carbon or any other fine dust material should not be charged free through the man hole to avoid the dust explosion. It should always be kept wet with water or with media in which the carbon treatment is given and charged.

III. CONCLUSION

The recovery of solvent Iso propyl alcohol is recovered from Iso propyl alcohol-water mixture. Solvent is recovered by distillation and also taking care and precaution and also safety devices are used while product manufacturing in plant.

The waste effluent which is coming out from the industry is also well treated and disposed effectively and gases coming out from the condensate is also treates well and converted into byproduct.

The above project is also feasible when compared to the Economics involved in it and by using Heteroazeotropic Distillation Process maximum separation could be done by using Cyclohexane as an entrainner.

But, since the process is being developed in laboratory scale, the scope for future study has more priority in this Solvent Recovery Field.

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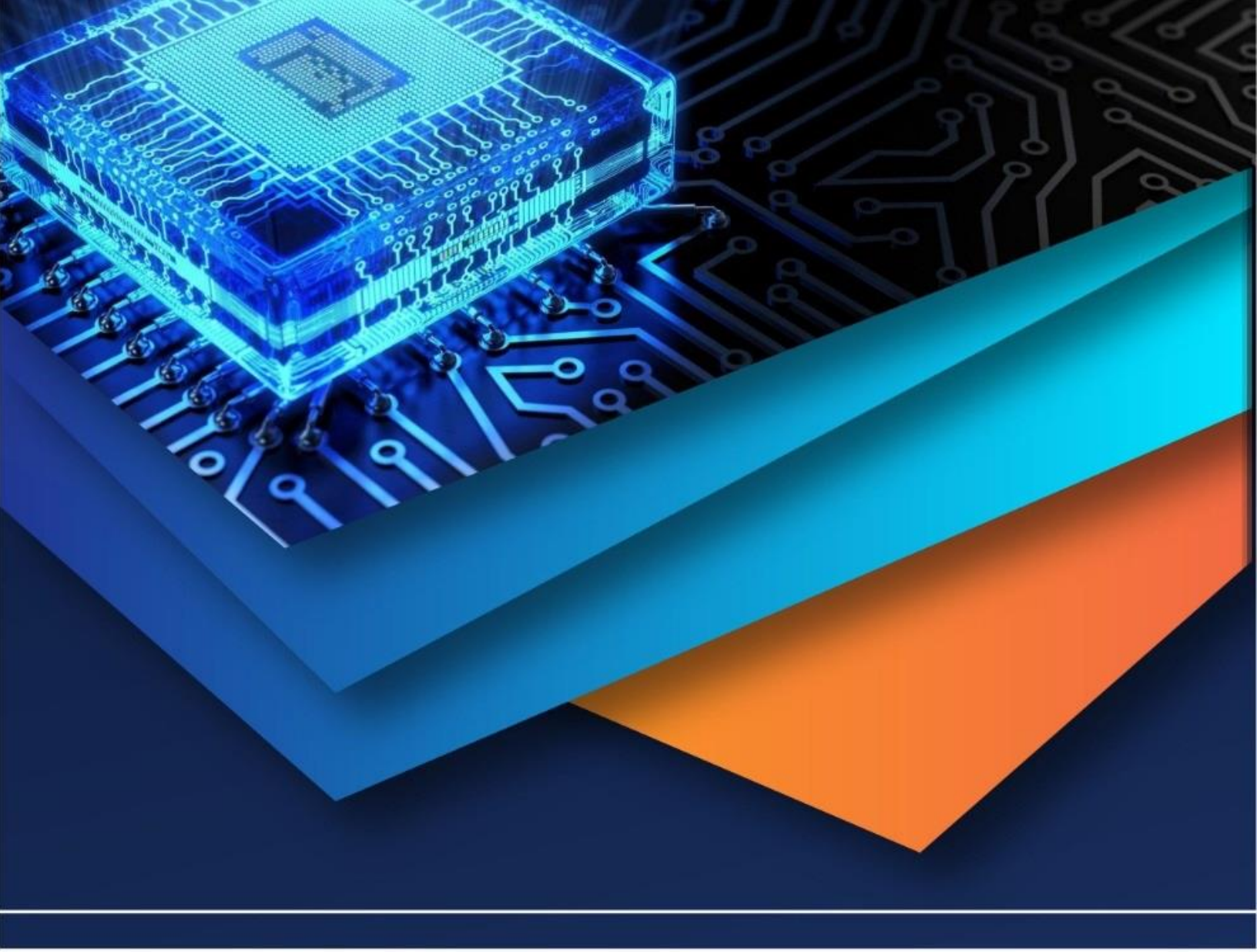

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