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Study of Spinning Band Distillation Column

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Abstract: *The separation of close boiling mixture cannot be achieved by conventional distillation methods as it possesses various properties such as, the constituents of mixture boils at same temperature by forming azeotrope. Spinning band distillation is one of the methods to separate close boiling mixtures. Spinning band distillation can be effective as it does not require any solvent or other component for the separation. The separation close boiling mixtures can be achieved by this with good efficiency. Spinning band distillation creates intimate contact between vapors and condensate in a dynamic process the helical pumping action of band forces refluxing liquid down with an intimate contact of the vapor going up the column which results in better efficiency in short distillation column. In the present study spinning band distillation column has been fabricated and used for the study on separation of acetic acid-water mixture. After experimental work got results as 91% water found in distillate and 64% water found in bottoms from the equal volume feed mixture.*

Keywords :*close boiling components, spinning band,rotating band, spinning band distillation.*

1. Introduction:

Spinning band distillation is a technique that uses a rotating helical band to create a high number of theoretical plates. Spinning band column can be made with metal or Teflon bands. The metal spinning band column is good for preparing samples of relatively broad boiling point ranges. The Teflon spinning column consists of glass tube containing solid Teflon rod around which is wound a helix of Teflon. The band is tightly fitted to bore of glass tube and is rapidly rotated. The helical pumping action of band forces the refluxing liquid down with an intimate contact of vapor going up the column. Because of this downward pumping action of liquid, flooding of column is easily avoided, and because of the open path between the solid rod glass tube, hold up and pressure drop are minimized. In the spinning band column the vapor going up is in close proximity of liquid going down the very narrow annular space between the walls of the column.[1]

Teflon spinning bands are used for distillations below 225 °C.Metal bands are used for higher temperature distillations where Teflon would become soft. The high number of theoretical plates makes spinning band distillation ideal for distillations requiring very good separation of components. Applications such as distillation of essential oils, crude oil and solvent recycling often use spinning band distillation to get the high purity fractions required.[1]

Zuiderwe (1951) worked on Performance of a Vigreux column and a spinning band column over wide range pressures and reported that the Pressure can be the major concept in distillation process that can affect the performance. Performance evaluation of a spinning band distillation column and Vigreux column over wide range of pressure was carried out. In order to obtain mass transfer mechanism analysis of separate vapor and liquid phase resistances were made.11 mm diameter Vigreux column and a 6 mm diameter spinning band column over a pressure range of about 0.13 kN/m^2-101.5 kN/m^2(about 1-760 mm Hg) were used.

In the case of the Vigreux column it was found that liquid phase resistance was about half the total resistance at atmospheric pressure. A reduction in pressure causes the liquid phase resistance to be increased by increasing liquid viscosity on the one hand and to be decreased by the higher vapour rates on the other hand. These two compensating factors cause & total efficiency loss of the Vigreux column of about 25% when the pressure is reduced from atmospheric value to about 0.13 kN/m^2 (1 mm Hg). At this top pressure the maximum pressure drop along the Vigreux section is about 0.3 kN/m^2(3-4 mm Hg) per meter.[6]

In a spinning band column the main resistance to material transfer is offered by the vapor phase. This resistance is reduced by creating turbulence through ration of the spinning band. The present study disclosed the existence of two limiting values of rotational speed: a lower limit below which rotation does not yet cause turbulence, and a higher limit above which back mixing in the liquid phase causes the separating power to be independent of-or to decrease with-speed of rotation.[6]

Katusz et al., (1981) had reported recovery of HPLC-grade acetonitrile by spinning-band distillation. In that they stated the recovery of previously used HPLC-grade acetonitrile by distillation without chemical pretreatment on a thirty-plate PTFE annular spinning- band column. Using static, preconcentration, and dynamic (on-column solvent enrichment) evaluation, spectrophotometric and chromatographic data indicate 85-95 % recovery of acetonitrile with purity at or near the specifications of commercially available HPLC-grade solvents. They uses previously used mobile phase mixtures

were refluxed for 24 h to ensure equilibrium, followed by distillation at a band speed of 2500 rpm and take-off rate of 125 ml/h. The first 20 ml of distillate at the appropriate boiling range (ambient barometric pressure) were discarded. Acetonitrile was distilled without chemical pre-treatment as the pure solvent or as a binary Azeotrope acetonitrile-water. [7]

Smith and Mathews (1976) Annular Teflon Spinning-Band Distillation Column to Determine Practical Liquid-Vapor Equilibrium Data for Close-Boiling Systems worked on and describe use of an Anuular Teflon Spinning Band Distillation Column to determine practical liquid- vapor equilibrium data for close boiling liquid system. An Annular Teflon Distillation Column was used to magnify the difference in the liquid and vapor compositions in carbon tetra chloride – benzene system. The data clearly shows that there is no azeoptrope formed in this system. It was further shown that useful liquid – vapor equilibrium data could be calculated from the distillation column result. [8]

Freeman et al., (1959) worked on Macro Spinning-Band Distillation Column and stated that the comparison of a macro spinning band distillation column by using the different shape and material of bands for the same operation. The system used in operation was n-heptane - 2, 2, 4-trimethylpentane system. A comparison of performance of these macro spinning band columns shows, Teflon bands have lowest HETP values. These lowest HETP values were obtained with four bladed Teflon band. It shows both band design and use of Teflon increases the efficiency of macro spinning band distillation columns. In contrast to metal bands with two blades, the Teflon band feature the large band core and score more point of contact with reflux on column wall. Although the six bladed band has more point of contact, the four bladed has large core and high efficiency. [9]

2. Materials &Method:

Materials: Binary mixture of acetic acid and water is used, acetic acid with water are selected for operation. Sodium hydroxide (NaOH) is used for titration of distillate and bottoms after operation and Phenolphthalein used as indicator. Standardization of NaOH will be done by oxalic acid.

Method: Take equal volume mixture of acetic acid and water in three-necked flask. Start heating and reflux the distillate obtained from top (Operate the column under total reflux). Continue this till steady state is reached (i.e. temp at the top and still is same). At steady state collect sample from still and top and titrate it against 2N NaOH From the analysis, calculate mole fractions at steady state for distillate and still under total reflux conditions by obtaining mole fraction used this mole fraction for calculating number of by using average relative volatility data over range of compositions, we can use Fenskey equation to calculate number of stages (Analytically) Measure the total height and using this, calculate the HETP. Change speed and repeat the same procedure and find HETP

Experimental Setup: Table 2.1 shows The List of components that has been use to Fabricate the Experimental Setup with their specification and functions. The schematic diagram of experimental setup is shown in figure 2.1.

Figure 2.1: Schematic Diagream of Spinning band Distillation Column. [10]

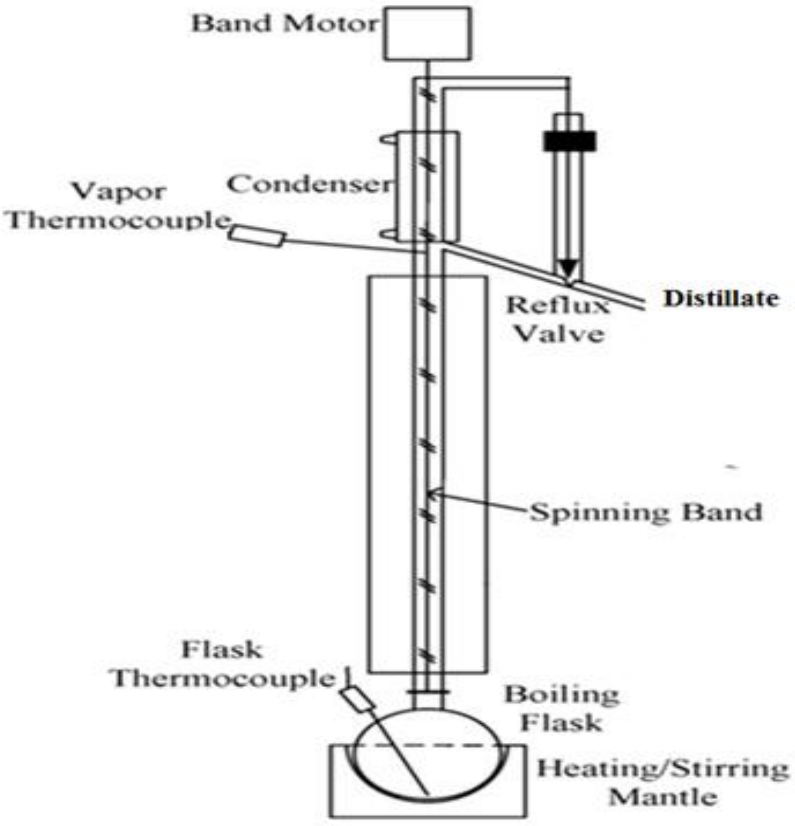

Table 2.1:Components of spinning band distillation column.

Sr.No.	Components	Specification
1.	Heating Mantle	Capacity:3 lit
2.	Motor	RPM: Up to 3000rpm
3.	Thermometer	Mercury Thermometer
4.	Condenser	Double Surface Condenser
5.	Connecting shaft	Diameter: 0.5-0.6mm,Length; 1.5m
6.	Column	Height: 1m,OD: 48mm,ID:42mm,Thickness 3:mm
7.	Teflon Disk	Clearance between column wall and disk: 1 mm, Diameter =40 mm, Distance between disc=34mm.
8.	3 necked Bottom Flask	Capacity: 1 lit

Experimental Study: The performance evaluation of a experimental setup can be determined by calculating the number minimum number of theoretical stages required, H.E.T.P. and Number of transfer unit. Number of theoretical stage scan be determined by using fenskey equation

$$N_m + 1 = \frac{log \frac{x_D}{1-x_D} \frac{1-x_W}{x_W}}{log \alpha_{avg}} (1)$$

Where, X_D is mole fraction of distillate, X_W is mole fraction of residue; α_{avg} is average relative volatility. The average relative volatility (α_{avg}) can be represented by evaluating the relative volatility of volatile component at various temperatures. VLE data of acetic acid-water system is given in Appendix I

$$\frac{\alpha x}{1-(\alpha-1)x} = y \qquad (2)$$

By using Equation (2) relative volatility of component can by calculate. During distillation, due to gas liquid contact, vapor gets enriched in the more volatile component and the liquid becomes leaner in more volatile component. As a result, the gas and liquid phase compositions change with height. Thus, over a certain height of a column, the gas and the liquid phases leaving that section will be in thermodynamic equilibrium with one another. Thus, this section would correspond to one theoretical stage, and the height of section corresponding to this one theoretical section is called 'height equivalent to a theoretical plate' (HETP). Clearly, smaller the HETP value better is the gas – liquid contact. The total packed height required can be calculated as = H.E.T.P. □ (No. of theoretical stages required for a certain operation. Since the column provides continuous contact, number of transfer units (NTU) can also calculated.

$$\int_{Xw}^{Xd} \frac{dy}{Y*-Y} = \text{Area Under The curve of } \frac{1}{Y*-Y} \text{ vs Y} \qquad (3)$$

3. Result and Discussion

The fabrication of experimental setup is done. The dimensions of column are calculated according to the literature review. The 1 meter glass column of 42mm inside diameter and 48mm outside diameter is used. A rotating spinning band is incorporated inside the column and coupled to the motor at the top for rotating the shaft. A spinning band is made up by putting a 41mm diameter and 15mm thick Teflon disk on the stainless steel rod. The length of rod is 1.5 meter having Teflon tubing in between the disk to prevent the falling of the disk. The Teflon cap is connected at the bottom of the rod with having perforations on it. The 2lit bottom flask and a double surface condenser are connected at the bottom and top respectively. Condenser is connected to the column with a reflux arrangement for operating column under total reflux.

Table 3.1 For 500ml water & 500ml Acetic Acid operated at total reflux

Sr. No	RPM	Xd	Xw	Nm+1	Nm	HETP(m)	NTU
1	300	0.912	0.721	2.669	1.669	0.59	3.28
2	700	0.912	0.719	2.737	1.737	0.575	3.17
3	900	0.913	0.721	222.742	1.742	0.573	3.28

Table 3.2 for 700ml water & 300ml Acetic Acid operated at total reflux

Sr. No	RPM	Xd	Xw	Nm+1	Nm	HETP(m)	NTU
1	300	0.94	0.86	1.77	0.77	1.282	3.49
2	900	0.95	0.87	2.023	1.023	0.97	4.1

Table 3.3 for 500ml water & 500ml Acetic Acid operated for continious output

Sr. No	RPM	Time	Xd	Xw	Nm+1	Nm	HETP(m)	NTU
1	600	00	0.910	0.716	2.811	1.811	0.532	3.17
2	600	15	0.906	0.707	2.712	1.712	0.584	2.35
3	600	30	0.901	0.710	2.556	1.556	0.642	2.24
4	600	45	0.910	0.644	3.699	2.699	0.37	4.29
5	600	60	0.903	0.646	3.177	2.177	0.46	3.86

Sample calculations:
1. at **600 RPM**
For, **Feed**
N1V1=N2V2

$$N1 = \frac{N2V2}{V1}$$

$N1 = \frac{23.5*2}{5}$ =9.4N
No. of Moles of Acetic Acid= 9.4*0.005=0.047 moles
Mass of Acetic Acid= 0.047*60=2.82gm
Considering Density of Mix q_{mix}= 1 gm/cc
Mass of H2O=5-2.82=2.18gm
Moles of H2O= $\frac{2.18}{18}$=0.121 moles
$X_{H2O=}$ $\frac{0.121}{0.121+0.047}$=0.72
X_{AA}=0.28

For, **Distillate**
N1V1=N2V2

$$N1 = \frac{N2V2}{V1}$$

$N1 = \frac{9.9*2}{5}$ =3.96N
No. of Moles of Acetic Acid=3.96*0.005=0.0198moles
Mass of Acetic Acid= 0.0198*60=1.188 gm
Considering Density of Mix q_{mix}= 1 gm/cc
Mass of H_2O=5-1.188 =3.812gm
Moles of H2O= $\frac{3.812}{18}$=0.211 moles
$X_{H2O=}$ $\frac{0.211}{0.211+0.0198}$= 0.9144
X_{AA}=0.0856

For, **Bottom Product**
C.B.R: 26.9 ml
N1V1=N2V2

$$N1 = \frac{N2V2}{V1}$$

$N1 = \frac{26.9*2}{5}$ = 10.76N
No. of Moles of Acetic Acid= 10.76*0.005=0.0538 moles
Mass of Acetic Acid= 0.0538 *60= 3.228gm
Considering Density of Mix q_{mix}= 1 gm/cc
Mass of H2O=5-3.228=1.772gm
Moles of H2O= $\frac{1.772}{18}$=0.09844 moles
$X_{H2O=}$ $\frac{0.09844}{0.09844+0.0538}$= 0.6466
X_{AA}=0.3534
α can be calculate by,

$$y = \frac{\alpha x}{1+(\alpha-1)x}$$

$$0.3063 = \frac{\alpha * 0.1881}{1+(\alpha-1)0.1881}$$

$$\alpha = 1.904$$

The VLE data shown in Table 4.4 is used for evaluating the relative volatility of Acetic acid-Water mixture at various temperatures. It is also used for calculating number of transferring unit shown in figure 4.3

Table 3.4: VLE data for acetic acid-water mixture

Sr. No.	X	Y	1/Y*-y	Temperature
1.	0.0000	0.0000	00	118.3
2.	0.1881	0.3063	8.46	110.6
3.	0.3084	0.4467	7.23	107.8
4.	0.4498	0.5973	6.77	105.2
5.	0.5195	0.6580	7.22	104.9
6.	0.5824	0.7112	7.76	103.5
7.	0.6750	0.7797	9.55	102.8
8.	0.7261	0.8239	10.22	102.1
9.	0.7951	0.8671	13.88	101.5
10.	0.8556	0.9042	20.57	100.8
11.	0.8787	0.9186	25.00	100.8
12.	0.9134	0.9409	36.36	100.5
13.	0.9578	0.9708	76.92	100.2
14.	1.000	1.000	0	100.0

$$Nm + 1 = \frac{ln\frac{Xd\,(1-Xw)}{Xw\,(1-Xd)}}{\ln \alpha\, avg}$$

For,
Xd=0.9144
Xw=0. 64

$$Nm + 1 = \frac{ln\frac{0.9144\,(1-0.6466)}{0.6466\,(1-0.9144)}}{\ln 1.67}$$

Nm+1=3.699
Nm=2.699

HETP (Height equivalent to one theoretical plate)
It can be calculated by,
The total packed height = H.E.T.P. x (No. of theoretical stages required for a certain operation)
1 = H.E.T.P. X 2.699
H.E.T.P. = 0.37m
NTU (Number of transfer Unit):
Data is taken table 4.4

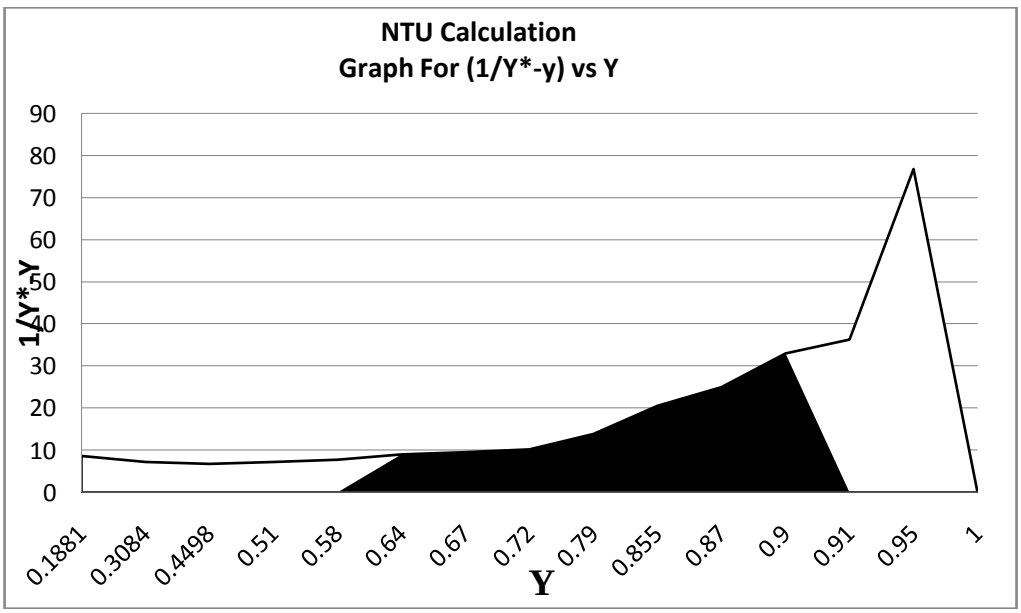

Figure 3.2: Graph For (1/Y*-y) vs Y

Area Under the curve: $\int_{Xw}^{Xd} \frac{dy}{Y*-Y}$ =4.29

In table 4.1, 4.2 & 4.3 the result obtain with spinning band distillation column are shown. The result shows 91% water content on mole basis in the top product, also the height equivalent to theoretical plate gets decreased with increase in the speed of spinning band. The results shown in table 4.3 are obtained by operating column under total reflux for one hour and then taking continuous distillate. The feed mixture has 71% of water, after the distillation the amount of water in bottoms is decreased up to 64%. The HETP of column has been also varying with the time as the distillation proceeds the HETP decreased. By plotting the equilibrium curve using VLE data is used to evaluate NTU. The NTU can give the transfer units for distillation. As the distillation proceeds the NTU gets increased.

4. Conclusion

Fabrication of experimental setup has been completed. Performance evaluation of experimental setup is carried out by using the binary mixture of acetic acid and water. From the analysis 91% efficiency was found in distillate. The minimum number of theoretical stages required is evaluated by using fensky's equation. The H.E.T.P. and NTU is also calculated to evaluate the performance of experimental setup.

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