

Muzaffarpur Institute of Technology , Muzaffarpur
B.Tech (Leather Technology) VI semester
Mid semester examination-2019

Subject: Chemical Engineering - III Code: LT-071614

Max .mark:20

Time :2Hours

Note: Question No-1 is compulsory. Attempt any other three questions. All question have equal marks. Assume any missing data.

Q.1 Choose the correct answer for the following objective question-

(I) In crystallization which process is occurred?

- (a) heat transfer (b) mass transfer (c) a and b both (d) none of these

(II) What is the effect on solubility due to temperature?

- (a) Increases with increasing temperature (b) Decreases with increasing temperature.
(c) No effect of temperature. (d) Firstly increases and then decreases

(III) If X is the moisture content of wet solid on dry basis, then the moisture content of the solid on wet basis will be equal to

- (a) X (b) $X/(1-X)$ (C) $X/(1+X)$ (d) $(1-X)/(1+X)$

(IV) In the drying of solids, the constant rate of period is absent if the initial moisture content of the solid is

- (a) less than the critical moisture content of the solid
(b) more than the critical moisture content of the solid
(c) more than the equilibrium moisture content of the solid
(d) are lying between the equilibrium and the critical moisture content of the solid.

(v) The critical moisture content for a given type of solid

- (a) is independent of drying rate and thickenss of solid
(b) increases with increased drying rate and thickness of solid
(c) increases with increased drying rate and decreases with increased thickness of solid
(d) decreases with increased drying rate and increases with increased thickness of solid

- Q.2 Discuss the working of plate and frame filtration equipment with diagram.
- Q. 3 State the manufacturing process of basic chromium sulphate.
- Q.4 Explain the working of one extraction equipment with proper diagram.
- Q.5 Explain the method to find out the number of theoretical plate of distillation column for separation of binary mixture by Mc. Cebé thiel method.

Model answer

Ans. 1

i. a

ii. a

III. C

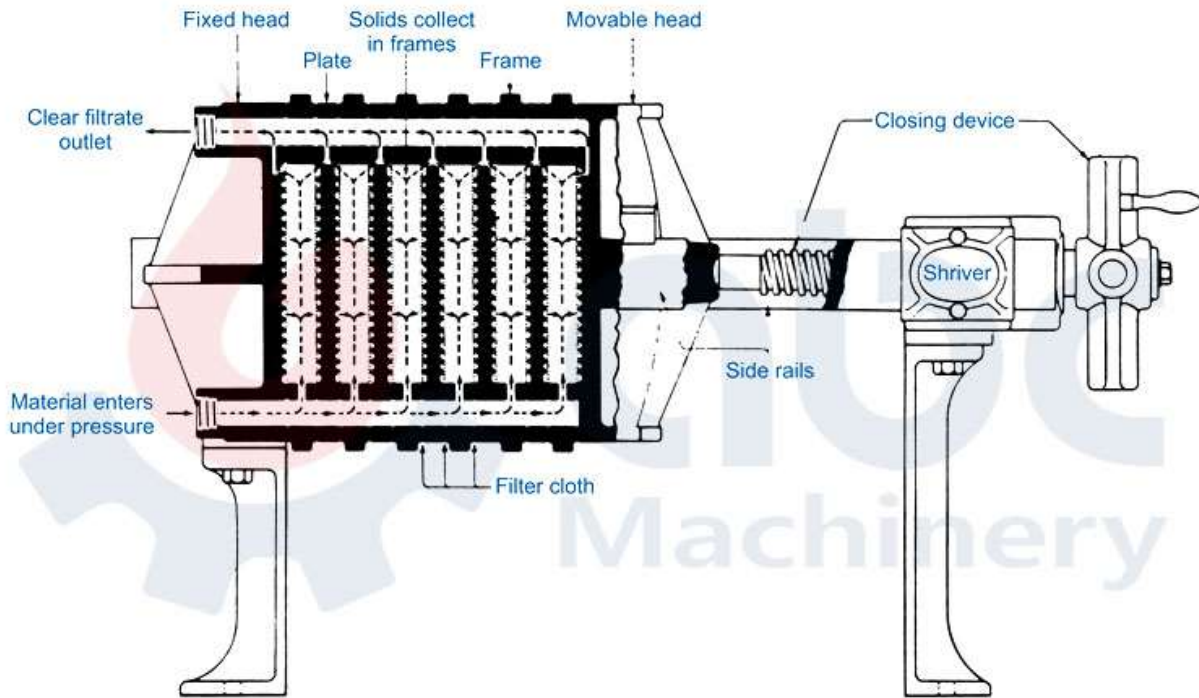
IV. A

V. B

ANS. 2 **Plate and frame filter press**[\[edit\]](#)

A plate and frame filter press is the most fundamental design, and many now refer it as a "membrane filter plate". This type of filter press consists of many plates and frames assembled alternately with the supports of a pair of rails. The presence of a centrifuge pump ensures the remaining [suspended solids](#) do not settle in the system, and its main function is to deliver the suspension into each of the separating chambers in the plate and frame filter. For each of the individual separating chambers, there is one hollow filter frame separated from two filter plates by filter cloths. The introduced [slurry](#) flows through a port in each individual frame, and the filter cakes are accumulated in each hollow frame. As the filter cake becomes thicker, the filter resistance increases as well. So when the separating chamber is full, the filtration process is stopped as the optimum pressure difference is reached. The [filtrate](#) that passes through filter cloth is collected through collection pipes and stored in the filter tank. Filter cake (suspended solid) accumulation occurs at the hollow plate frame, then being separated at the filter plates by pulling the plate and frame filter press apart. The cakes then fall off from those plates and are discharged to the final collection point.^[5]

Cake discharge can be done in many ways. For example: Shaking the plates while they are being opened or shaking the cloths. A scraper can also be used, by moving from one chamber to another and scraping the cake off the cloth. At the end of each run, the cloths are cleaned using wash liquid and are ready to start the next cycle.¹



Ans. 3 The invention relates to the preparation of chromium sulphate from residual solutions containing hexavalent chromium compounds.

The extraction of chromium from its ores by wet method processes for the preparation of various compounds such as chromium sulphate or basic chromium sulphate, chromic acid, chromates or bichromates, always results in residual solutions containing large amounts of chromium derivatives, and notably hexavalent chromium derivatives. Similarly, electroplating installations dispose of large amounts of solutions which have become unusable but which still have high chromium contents.

All said residual solutions have the characteristic of being charged with all sorts of impurities, and notably metal impurities, which prevent their being reused directly. For many years, manufacturers preferred to discharge these solutions into rivers rather than purify them or recover the chromium contained in these solutions. Yet chromium compounds, and especially hexavalent chromium compounds, are redoubtable pollutants as in addition to the toxicity of chromium it has a high oxidizing capacity. Regulations now in force limit the concentration of hexavalent chromium in liquid effluents discharged into watercourses to a level of 0.1 to 1 mg per liter, according to the country, whereas up to 2 mg per liter of trivalent chromium is tolerated.

Various processes for the treatment of liquid effluents containing hexavalent chromium have already been suggested or put into effect. It is thus possible to reduce hexavalent chromium and precipitate chromic hydroxide $\text{Cr}(\text{OH})_3$. Many reducing agents are known, one of the least costly being sulphur dioxide. Precipitation involves neutralization of the solution with a cheap reagent such as lime. This process has two drawbacks. Firstly, the $\text{Cr}(\text{OH})_3$ precipitate is difficult to filter; it is very gelatinous and needs to be treated with a filter press technique. In addition as it is very impure, it cannot be reused economically: it then forms a solid waste, the disposal of which constitutes a further risk of pollution.

Another process consists in taking advantage of the properties of ion exchange resins to extract hexavalent chromium from liquid effluents. As said chromium is in the form of anions, it can be extracted selectively with a basic anionic exchange resin. Elution is effected by washing the resin with a soda solution which takes up sufficient pure sodium chromate to be reused in the preparation of various compounds.

When the preparation of chromium sulphate is envisaged, such a process has the drawback of resulting in a considerable consumption of soda and sulphuric acid, as the sodium chromate solution must be neutralized with sulphuric acid then, after reduction, be reacidified with the same acid.

In this connection, an elution-reduction process for an anionic exchange resin on which hexavalent chromium had previously been fixed has also been described, a process according to which a sulphuric solution of sodium bisulfite NaHSO_3 is used. According to this process, an anion is therefore adsorbed on an ion exchange resin and the said resin is treated with a chemical agent capable of converting the anion into a cation, the said cation then being desorbed from the resin, which is therefore regenerated.

It should be noted that large amounts of reagents necessarily have to be used in this process, as will be seen in cases (1) for the production of a basic chromium sulphate solution (basicity 33) and (2), for the production of a neutral chromium sulphate solution.

1. In the case where it is desired to obtain a basic chromium sulphate, of the formula $\text{Cr}(\text{OH})_2\text{SO}_4$

generally with a basicity of 33° Schorlemmer, notably for use in tanning, the following reactions should be effected, wherein R is the anionic exchange resin on which the hexavalent chromium has been fixed: $\text{Cr}(\text{OH})_2\text{SO}_4$

Therefore, for 2 moles $\text{Cr}(\text{OH})_2\text{SO}_4$, 3 moles NaOH , 3 moles SO_2 and 1.5 mole H_2SO_4 are required.

2. In the case where neutral chromium sulphate $\text{Cr}_2(\text{SO}_4)_3$ is desired, it is necessary to conduct the following reactions to obtain said compound:



It follows that, for 1 mole $\text{Cr}_2(\text{SO}_4)_3$, 3 moles NaOH, 3 moles SO_2 and 2.5 moles $\text{H}_2 \text{SO}_4$ should be used. It is also known to extract hexavalent chromium compounds dissolved in an aqueous solution by a water-insoluble organic liquid, but the application of this practice to the treatment of industrial waste waters has not been possible up to now owing to the lack of economical processes for reextracting hexavalent chromium from the organic solution. In order for such a process to be economical it should directly provide a commercial chromium compound, that is to say, salts such as sodium bichromate. Then again, the solvent used to effect extraction should not only possess the usual properties required for this type of treatment but it should also have an excellent resistance to oxidizing agents, even in a highly acid medium. Indeed, this situation is frequently met with, as the waste solutions obtained from electroplating installations or residual solutions from the production of chromic acid are highly sulphuric solutions of chromic acid.

The object of the present invention is a process which avoids stream pollution caused by solutions containing hexavalent chromium compounds, with the accompanying production of chromium sulphate. The process according to the invention is based on the recovery of hexavalent chromium from solutions containing the same, which hexavalent chromium has previously been fixed on ion exchange resins or extracted with a water-insoluble organic solvent, by more economical means than those previously applied or envisaged, the trivalent chromium sulphate being obtained directly, and without excessive consumption of reagent, from the hexavalent chromium ions thus fixed or extracted.

The process according to the invention consists in that hexavalent chromium in the residual solution is extracted and fixed by known means, notably by contacting the said solution either with an ion exchange resin or with a water-insoluble organic solvent; then the hexavalent chromium thus fixed by the resin or organic solvent is reextracted by contacting the fixing agent with an aqueous solution, and it is characterized in that said reextraction is effected by the use of an aqueous solution of sulphur dioxide in the presence of sulphuric acid to directly recover a chromium sulphate solution.

The residual solutions to which the invention is applied can have very varied compositions. Notably, apart from hexavalent chromium compositions, they may contain a large number of metal impurities, such as alkaline and alkaline-earth metals, iron and metals of the family of iron,

aluminium, magnesium, zinc, titanium, etc. They may also contain trivalent chromium. The exact compositions vary with the origin or said solutions. There should notably be mentioned the solutions discarded by installations treating chromium ore, or by electroplating installations.

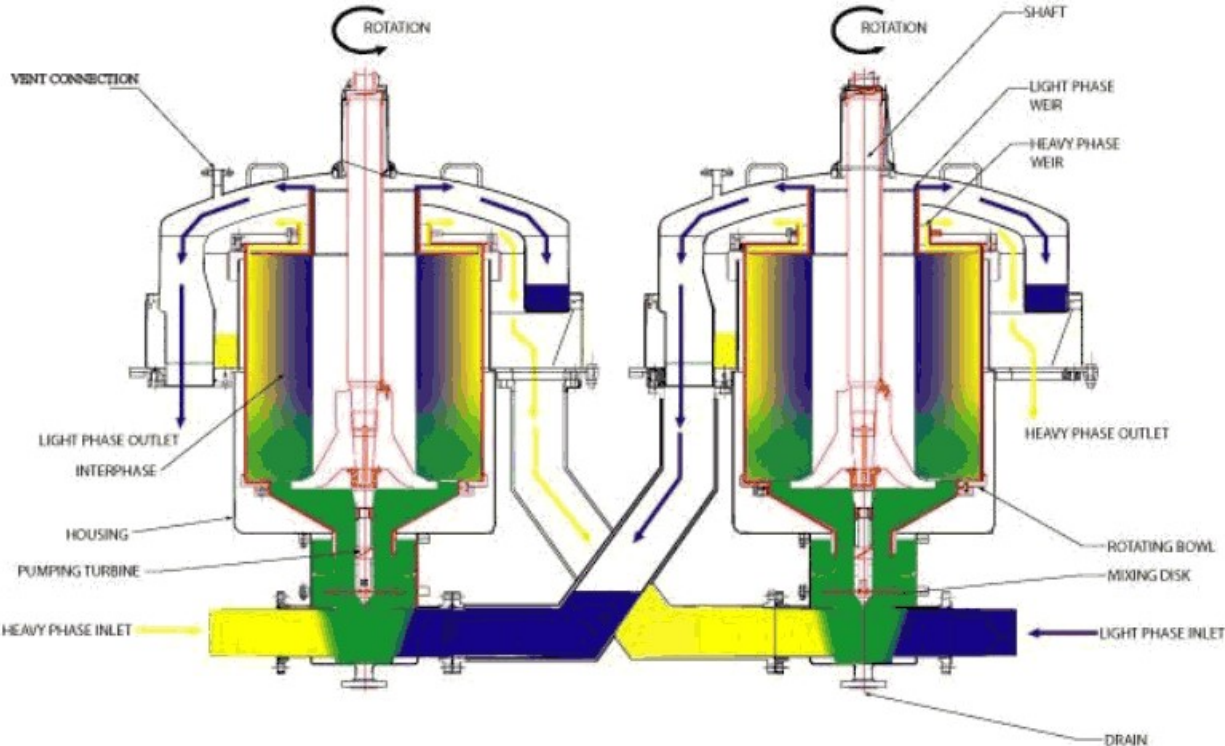
The concentration of hexavalent chromium compounds in said solutions can vary in a large range. It is normally in the range of 0.01 to 200 g/l, expressed as CrO_3 and preferably between 0.1 and 30 g/l.

To obtain satisfactory operation of the process in the case where the hexavalent chromium in the solution to be treated is fixed with an ion exchange resin, the pH of the residual solutions should be in the range of 0 to 6 and preferably from 2 to 3. When they contain a large amount of free acid which is very frequently the case, they should therefore, as has been said, be previously neutralized until their pH is within the above mentioned limits, said neutralization can be carried out with a cheap base such as lime. Conversely, when extraction of the hexavalent chromium from the initial residual solution is effected by liquid-liquid contact of the solution with a water-insoluble organic solvent, said initial solution can be used as it is, without preliminary neutralization, even if it is highly acid. This is an important advantage of said second method with respect to the first as, when neutralization has to be effected, by means of lime, for example, as was mentioned hereinabove, the calcium sulphate precipitated must be separated by filtration, with the difficulties this involves, and washed to deplete it of the initial solution and the chromium compounds with which it may be impregnated.

Ans. 4 Extractions are a way to separate a desired [substance](#) when it is mixed with others. The [mixture](#) is brought into contact with a solvent in which the substance of interest is soluble, but the other substances present are insoluble.

Extractions use two immiscible phases (these are phases that do not mix, like oil and [water](#)) to separate the substance from one phase into the other.

MONO-STAGE CENTRIFUGAL EXTRACTORS:



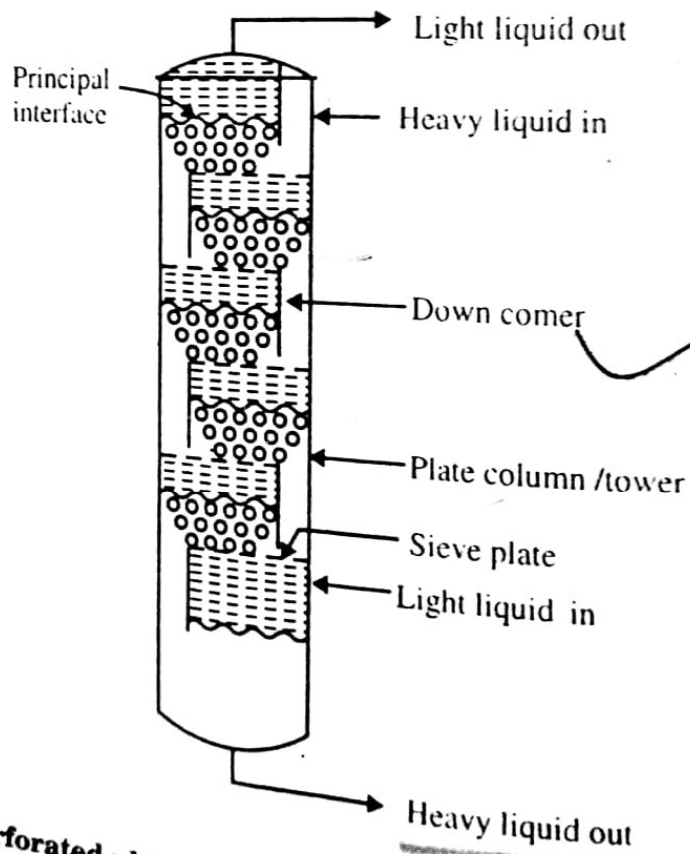
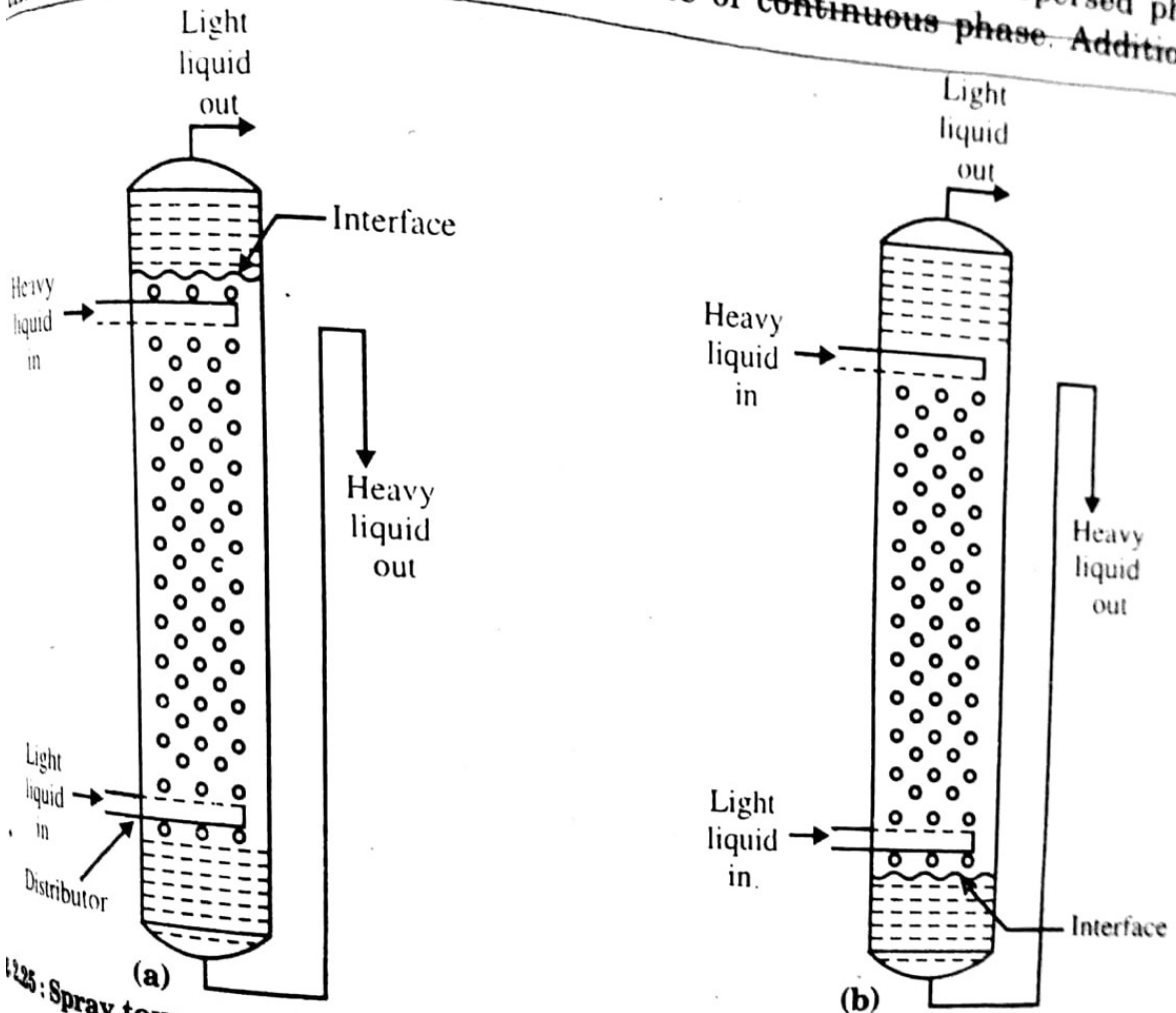


Fig. 2.24 : Perforated plate/tower for continuous countercurrent extraction (light-liquid dispersed)

... dispersed phase
... of continuous phase. Additional t



1.25: Spray towers (a) for light liquid dispersed and (b) for heavy liquid dispersed

Consider any plate n in the rectifying section.

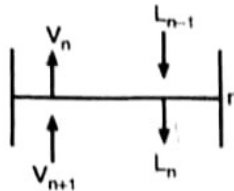


Fig. 1.13

A heat balance across plate n is

$$L_{n-1} H_{L_{n-1}} + V_{n+1} H_{V_{n+1}} = L_n H_{L_n} + V_n H_{V_n} + \text{heat losses} + \text{heat of mixing}$$

Heat losses are negligible for good insulation.

Heat of mixing is zero for an ideal system.

$$\therefore L_{n-1} H_{L_{n-1}} + V_{n+1} H_{V_{n+1}} = L_n H_{L_n} + V_n H_{V_n}$$

H_L and H_V - enthalpies of liquid and vapour per mol.

If the molar heat of vaporisation is constant (independent of the composition) then one mol of vapour V_{n+1} on condensing releases sufficient heat to liberate one mol of vapour V_n . It follows that $V_n = V_{n+1}$. Therefore, the molar flow of vapour up the column is constant. As the temperature change from one plate to the next is small, H_{L_n} can be taken equal to $H_{L_{n-1}}$.

$$\therefore L_{n-1} H_{L_{n-1}} + V_{n+1} H_{V_{n+1}} = V_{n+1} H_{V_{n+1}} + L_n H_{L_{n-1}}$$

$$\therefore L_{n-1} = L_n$$

So the moles of liquid reflux in this section are also constant.

Therefore, L_n and V_{n+1} are constant in the rectifying section and V_{m+1} and L_m are constant in the stripping section.

i.e. molar flow rates of vapour and liquid are constant in each section of the column.

$$L_n = L_{n-1} = L, \quad L_m = L_{m+1} = L'$$

$$V_n = V_{n+1} = V, \quad V_{m+1} = V_m = V'$$

[The above cited assumptions lead to a concept of constant molal vapour flow and constant molal liquid flow in any section of the column. In other words, for one mole of vapour condensed, one mole of liquid is vaporised. Thus $V_n = V_{n+1}$, $L_n = L_{n+1}$... etc.]

The subscripts n , $n + 1$, m , $m + 1$... etc. may be dropped and the operating lines then plotted on the equilibrium diagram are straight.

The operating line of the rectifying section becomes :

$$y = \frac{L}{L+D} x + \frac{D x_D}{L+D} \quad \dots (1.59)$$

As $R = \frac{L}{D}$, the above equation becomes :

$$y = \frac{R}{R+1} x + \frac{x_D}{R+1} \quad \dots (1.60)$$

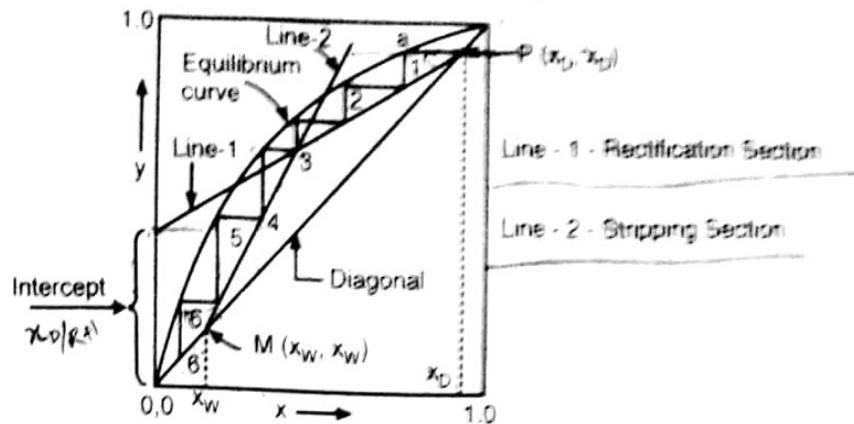


Fig. 1.14 : Determination of theoretical plates by McCabe - Thiele Method

6. Proceed in this way, that is constructing the triangles (representing the stages) between the equilibrium curve and the operating line of rectifying section till we are above the point of intersection of two operating lines. Once we cross this point of intersection, construct the triangles between the equilibrium curve and the operating line of the stripping section i.e. dropping the verticals on the operating line of the stripping section.
 7. Proceed in the same manner till we reach exactly or cover the point M (x_W, x_W).
 8. Count the triangles constructed between x_D and x_W . Each triangle on $x - y$ diagram represents a theoretical plate.
9. If the number of triangles are 'n', then 'n' represents the theoretical number of plates including reboiler and $n - 1$ represents the number of theoretical plates in a column to achieve a desired degree of separation.

Limitations of McCabe - Thiele Method :

1. It is not used when the relative volatility is less than 1.3 or greater than 5.
2. It is not used when more than 25 theoretical stages/plates are required and
3. It is not used when the operating reflux ratio is less than 1.1 times the minimum reflux ratio.

Lewis - Sorel Method of Determination of Number of Theoretical Stages/Plates :

The point of intersection of the operating line for the rectifying section and 45° diagonal ($x = y$) obtained as,

$$y = \frac{R}{R+1} x + \frac{x_D}{R+1}$$

$$y = x$$

Putting

$$x = \frac{R}{R+1} x + \frac{x_D}{R+1}$$

∴

$$(R+1)x = Rx + x_D$$

$$x = x_D$$

and

$$y = x_D$$

Thus, the operating line of the rectifying section is to be drawn on the equilibrium diagram through the point (x_D, x_D) on the 45° diagonal and with a slope equal to $R/(R+1)$. intercept on y-axis equal to $x_D/(R+1)$.

The equation of the operating line of the stripping section becomes :

$$y = \frac{L'}{L' - W} x - \frac{W x_w}{L' - W} \quad \dots (1.6)$$

The point of intersection of the operating line of the stripping section and 45° diagonal is obtained as follows :

$$y = \frac{L'}{L' - W} x - \frac{W x_w}{L' - W}$$

Diagonal :

$$y = x$$

∴

$$x = \frac{L'}{L' - W} x - \frac{W x_w}{L' - W}$$

∴

$$L' \cdot x - W \cdot x = L' \cdot x - W x_w$$

∴

$$x = x_w$$

and

$$y = x_w$$

Thus, the operating line of the stripping section is to be drawn through the point (x_w, x_w) on the diagonal and with slope equal to $\frac{L'}{L' - W}$.

Step-wise procedure for obtaining theoretical plates :

1. By material balances, evaluate the terms D , W , L etc.
2. Draw the equilibrium curve and the diagonal with the help of $x - y$ data given. If relative volatility is given, generate the $x - y$ data.
3. Draw the operating line of the rectifying section through the point $P (x_D, x_D)$ on the diagonal and with an intercept equal to $x_D/(R+1)$ or $Dx_D/(L+D)$ or slope equal to $R/(R+1)$.
4. Draw the operating line of the stripping section through the point $M (x_w, x_w)$ on the diagonal and with a slope equal to $\frac{L'}{L' - W}$.
5. Starting from the point (x_D, x_D) on the diagonal, draw a horizontal line to meet the equilibrium curve at point 'a'. Drop a vertical from point 'a' to meet the operating line at point 1.