Indira Gandhi National
Open University
School of Sciences

BCHET-141
ANALYTICAL METHODS IN CHEMISTRY

Block

2

CHROMATOGRAPHY AND ION EXCHANGE

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BLOCK 2: CHROMATOGRAPHY AND ION EXCHANGE

In this Block, we will discuss the general aspects of chromatography and ion exchange methods. Unit 5 will begin with a discussion on classification of chromatographic methods. In this unit, we will also explain basic principle of paper chromatography and its applications.

In Unit 6, the principle and efficiency of the column chromatography technique will be described. This will be followed by a brief account of the mechanism of separation operating in this technique. The important features of adsorption and partition chromatographic techniques will then be compared.

Unit 7 on 'Ion Exchange Chromatography' covers the classification of ion exchangers. The details of mechanism of exchangers ion exchangers and applications of ion exchange chromatography have also been explained.

Expected Learning Outcomes

After studying this block, you should be able to:

- discuss the classification of various chromatographic techniques according to the different criteria;
- state the importance of paper chromatography, column chromatography and ion exchange chromatography;
- explain the principle of paper chromatography, adsorption chromatography and ion exchange chromatography, and
- discuss the applications of paper chromatography, adsorption chromatography and ion exchange chromatography.

UNIT 5

GENERAL ASPECTS OF CHROMATOGRAPHY

Structure

5.1 INTRODUCTION

This is the first unit of this Block on 'Chromatography and Ion exchange'. Here, we will cover the general aspects of chromatography. The unit will begin with a discussion on classification of chromatographic methods. This will be really an eye opener for you to realise that what a vast variation of this technique is possible .A wide variety of options has resulted in this technique with time as newer developments kept on taking place and increasing need of separation of various types of substances was felt.

We will also explain the technique of paper chromatography which is classified as partition chromatography because the *mechanism* of the separation involved in this technique is partition. Under this technique, we will focus on the principle of separation as well as the efficiency of this technique. The mechanism involved will also be discussed and the development of the chromatograms will be explained. We will also discuss the characteristics of the mobile phase and the concept of R_f value.

Expected Learning Outcomes

After studying this unit you should be able to:

- discuss the classification of various chromatographic techniques according to the different criteria;

- ❖ state the importance of paper chromatography and explain the various terms involved in it;
- ❖ explain the principle of paper chromatography;
- ❖ describe the mechanism of separation in partition chromatography;
- ❖ explain how the chromatogram is developed in paper chromatography; and
- ❖ discuss the applications of paper chromatography.

5.2 CLASSIFICATION OF CHROMATOGRAPHIC METHODS

Mikhail Tswett (1872-1919)

'Chromatography' was introduced by a Russian Botanist, Mikhail Tswett in 1906. It is now a journey of more than a century and a lot of interesting developments have been introduced in using such techniques .A lot of variations with regard to the use of equipment and materials used have taken place from time to time.

Chromatography was used by Tswett to separate the plant pigments using a simple glass column packed with finely divided calcium carbonate. He used petroleum ether to separate various pigments which appeared as bands at different heights in the column after separation. You will also study about *column chromatography* in Unit 6.

Thus, the technique of chromatography involves the separation of the components of a mixture by their *distribution between two phases*- one being the ***stationary phase*** *of large surface area* and the other being the ***mobile phase.*** Here, the mobile phase moves on the stationary phase in a definite direction. Depending upon the ***type of stationary support, nature of mobile phase*** and ***the mechanism involved in the separation***; several types of chromatographic methods are possible, see Fig.5.1.

The technique of chromatography is a physical method of separation. It is based on molecular weight of a substance and its adsorption or partition coefficient. Thus, substances with lesser molecular masses diffuse more quickly than those with higher molecular masses.

Let us see the following possible variations when each of the above mentioned aspects are taken into account:

(i) Depending upon the **type of stationary support** or more accurately the ***shape*** of the stationary support, chromatographic technique can be ***two-dimensional*** or ***three-dimensional.***

The techniques of *paper chromatography* and *thin layer chromatography* are two further subdivisions of *two-dimensional chromatography* because the stationary phases used in these two types of chromatography are, respectively *paper* and a *solid support* coated on a plate *of glass, plastic* or *metal***.** The shapes of all these supports are *two-dimensional*; hence, these two techniques are classified under two-dimensional chromatography.

The technique of *column chromatography* is considered under the *three-dimensional chromatography. Column chromatography* involves the stationary support present in a *column* which is *three-dimensional in shape.*

(ii) According to the **nature of mobile phase**-whether the mobile phase is gas, liquid or supercritical fluid, the respective chromatographic techniques are called *gas chromatography*, *liquid chromatography* or *supercritical fluid chromatography.*

(iii) We can also classify chromatographic techniques into different classes according to the **mechanism involved in separation**. Accordingly, we can name these techniques as *partition, adsorption, ion exchange* or *size exclusion chromatography.*

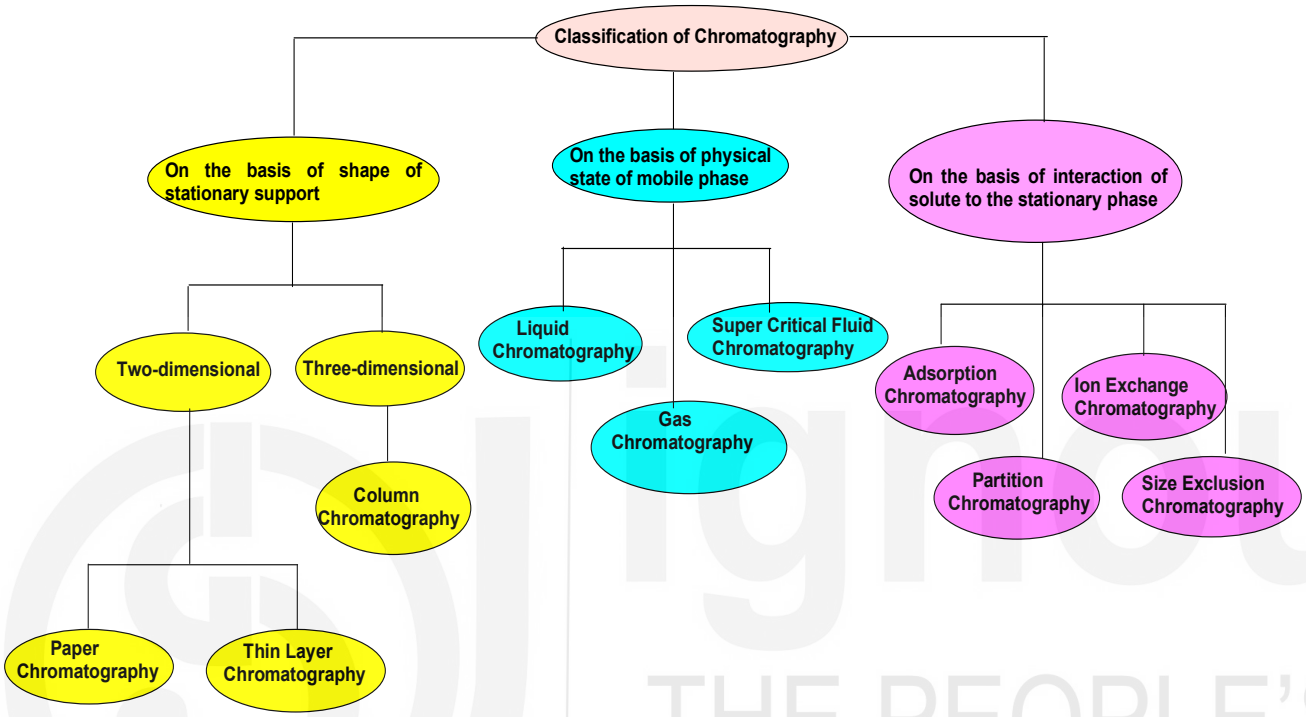


Fig. 5.1: Classification of Chromatography

We will be dealing with *partition chromatography* i.e. *paper chromatography* in this unit itself. The *adsorption chromatography* using *column chromatography* will be dealt in Unit 6 while *ion exchange chromatography* will be described in Unit 7. The *size exclusion chromatography*, however, is *not* a part of this course and will not be dealt here.

Thus, all the available chromatographic techniques cannot be classified using a single criterion, as the different combinations of stationary and mobile phases can be used and the mechanism operating in separations may also differ. Hence, it is the ***nature of mobile phase*** which is taken as main criterion to classify the technique. The other factors mentioned above are discussed as *details* or *sub-criteria* under this main class.

Thus, when the *nature of mobile phase is taken as the main criterion*, then the ***gas, liquid*** and ***supercritical fluid*** chromatographies are considered. The *gas* and *supercritical fluid chromatography* are beyond the scope of this course. Therefore, we will keep our discussion focused to the *liquid chromatography only*.

When the *mobile phase* is ***liquid,*** the *stationary phase* can be either a ***liquid supported on a solid*** or ***a solid*** and this gives rise to the **liquid-liquid chromatography** or **liquid-solid** chromatography. In *liquid-liquid chromatography*, the distribution of components of mixture takes place by their

partitioning of the components of the mixture in the two liquid phases, as was the case in the solvent extraction.

Further, two-dimensional and three-dimensional chromatographies are possible as shown in Fig. 5.2, according to the shape of the stationary phase taken. Each of these variations may further involve *upward or downward* direction of movement of the mobile phase, thereby widening the options to *ascending* and *descending* modes of operation. This is illustrated below in Fig. 5.2.

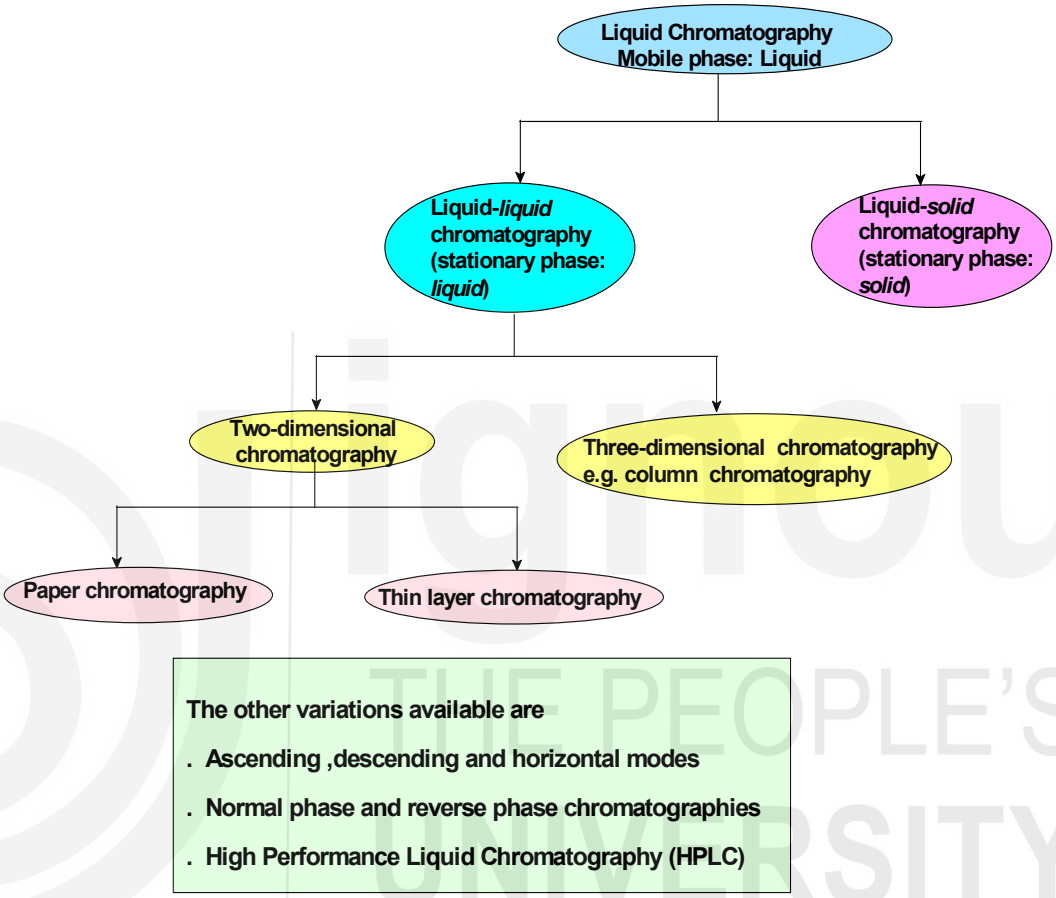


Fig. 5.2: Types of liquid chromatography

Normally, *polar support is taken as the stationary phase* and *non-polar mobile phase* is chosen. Such a combination is referred to as ***normal-phase*** chromatography. But, if the polarities of the two phases are reversed, then it is called ***reverse-phase*** chromatography.

Various forms of chromatographies explained above can also be performed under high pressure leading to **high performance** or **high pressure liquid chromatography (HPLC).**

Let us now focus our attention on paper chromatography which involves partition mechanism.

SAQ 1

Name two types of two-dimensional chromatography.

5.3 PARTITION CHROMATOGRAPHY: PAPER CHROMATOGRAPHY

The foundation of the paper chromatography dates back to 1944 when R. Consden, A.H. Gordon and A.P.J Martin reported the separation of amino acids using paper. The amino acids present in only 200 μg of wool could have been separated using paper chromatography. The importance of chromatography was recognised when Nobel prize was awarded in 1952.

The technique of the paper chromatography is very simple and useful. Let us now understand its principle.

5.3.1 Principle

Paper chromatography is a kind of liquid-liquid partition chromatography in which the substances are distributed between two liquids-the stationary phase(usually water that is held in the fibres of the paper, also called water –cellulose complex containing 20% water) and the moving liquid or the mobile phase ,also called the developing solvent.

The paper chromatography involves the use of paper which is made up of highly pure cellulose .The examples of such a paper are - Whatman papers with no. 1,2,3,3 MM, 31 ET etc. These papers are having uniform physical characteristics and are very low in organic and inorganic impurities. Some more types of modified papers are also used about which you will study later.

Here, it would be interesting to know that paper chromatography can be done in *ascending mode*, *descending mode* or in *circular fashion*. The simplest one is ascending mode paper chromatography for which the set up is shown below in Fig. 5.3.

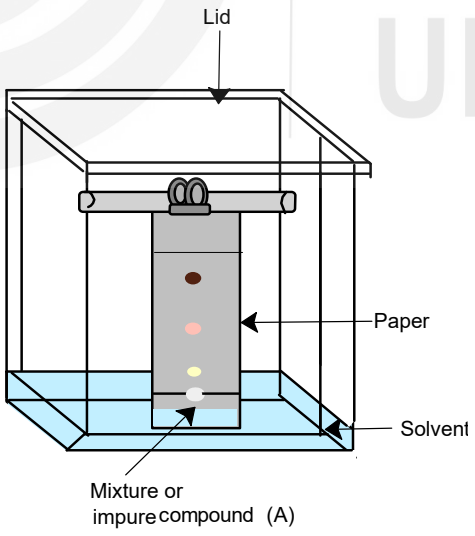


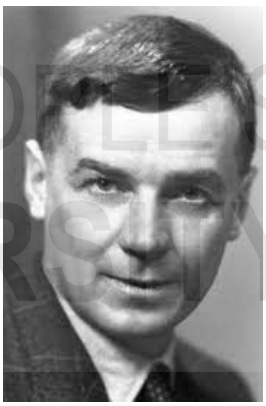
Fig. 5.3: Set up for the ascending mode paper chromatography

The sample containing the mixture of compounds to be separated is taken in a suitable solvent and using a capillary, a fine small spot is put on the line drawn on the paper. Let this spot be marked as A. The sample could also be an impure compound which could be tested by paper chromatography for how many impurities are present.

Archer John Porter Martin

(1st March 1910-28th July 2002)

He shared Nobel Prize in Chemistry for year 1952 with the Richard Synge for the invention of *partition chromatography*.



Richard Laurence Millington Synge

(28th Oct 1914-18th August 1994)

He shared Nobel Prize in chemistry for the year 1952 with A.J.P. Martin for the invention of *partition chromatography.*

The paper is then kept in the development chamber as shown in Fig. 5.3.The solvent which is the mobile phase is then allowed to rise by the capillary action over the spot of mixture of compounds . After some time, the solvent takes along with it, the different components of the mixture (of spot A) to different heights on the paper leading to their separation.

Similarly, we can have *descending paper chromatography* and *circular paper chromatography* .The experimental set ups for these two methods are shown in Fig. 5.4. a) and b) below.

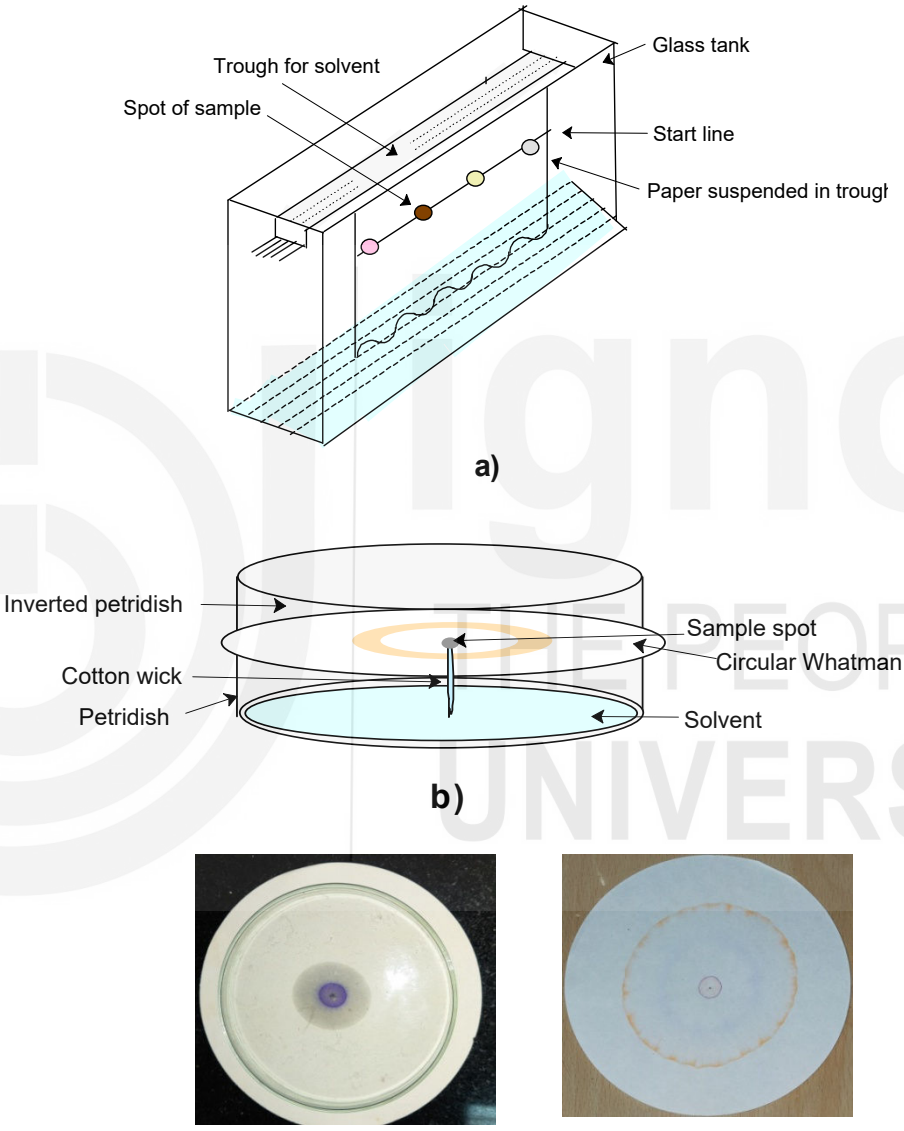


Fig. 5.4: a) Descending paper chromatography

b) Circular paper chromatography

c) Separated compounds by circular chromatography

In **descending paper chromatography**, the solvent tank contains the solvent which moves from *upside to downward* direction on the paper. The spots of the sample are put on the paper and the same is placed in the development chamber as shown in Fig. 5.4 a).

In circular paper chromatography, as shown in Fig. 5.4 b), the spots of the sample are placed on the paper in circular fashion. Then, the moving phase or the mobile phase which is a liquid (i.e. a solvent or a mixture of solvent) is taken in a Petri dish. At the centre of the paper, a very small hole is made using a pin. A wick is then placed in this hole whose other end dips in the mobile phase.

The solvent rises to the centre of the paper through the cotton wick .The mobile phase then carries along it, different components of the sample in a circular fashion. Thus the different separated components are obtained as rings on the Whatman paper and not as the spots.

Having understood the basic principle of paper chromatography, let us now study about its efficiency.

5.3.2 Efficiency of the Technique

As discussed above, the techniue of paper chromatography is not only simple and easy to perform but it is also efficient in separating the components of a mixure or in obtaining the pure compound from an impure sample.

For obtaining the pure components of a mixture or for getting a pure compound from its impure sample, *quantitative paper chromatography* has to be performed. A 30 cm x 30 cm sheet of paper is taken and several spots of the sample are put in a line near the base of the paper. The paper is then kept in the development chamber as shown in Fig. 5.5. The mobile phase is then allowed to rise by the capillary action over the spots of sample.

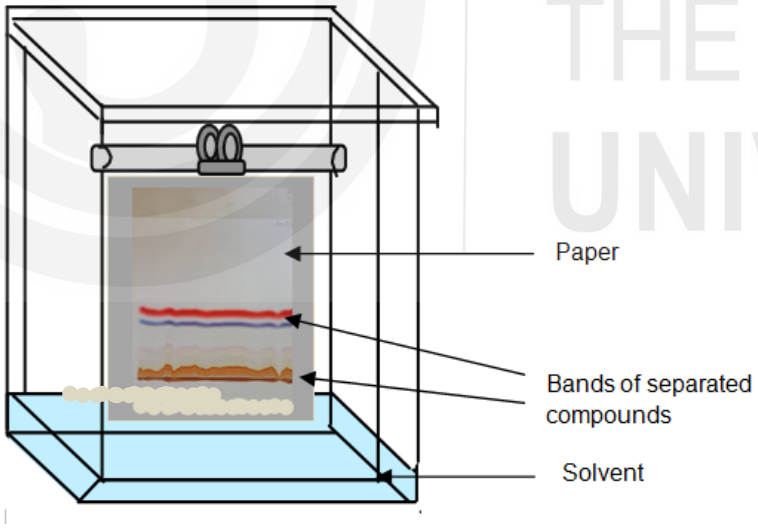


Fig.5.5: Quantitative paper chromatography

The separated components appear as separate bands at different heights on the paper. The relevant portions of the paper are cut for obtaining different components in pure form. Each of the component is then separataly extracted from the paper by dissolving it in a suitable solvent. The removal of the mobile phase or the solvent yields the pure components.

The technique of paper chromatography has been efficiently used for the *qualtative analysis* of several types of naturally occurring and synthetically obtained reaction mixtues. The separation can be effectively carried out and

the identification of different components can be done by putting the spot(s) of the known standard sample(s) alongwith the sample to be tested. The spots of the same compound (whether present in a mixture and as a pure sample) will appear at the same level on the paper after the separation has occurred. Hence,the componenets of the sample can be correlated with the standard compounds.

Paper chromatography is a very efficient technique and it can be used to separate very closely related compounds such as isomers, homologues and species with different valency or oxidation states etc.

Some examples of separations using paper chromatography are as follows:

(i) Identification of metal ions such as Ni^{2+}, Co^{3+}, Zn^{2+} and Mn^{2+}

(ii) Separation of mixture of pesticides containing heptachlor, BHC, aldrin etc. into its pure components

(iii) Checking of foods and drinks for adulterants using the standard samples of pure components and adulterents.

After understanding the efficiency of paper chromatography ,let us now study the mechanism by which such separations take place.

5.3.3 Mechanism of Separation

In the beginign of this section, we have mentioned that cellulose paper is used as solid support in paper chromatography and the mobile phase is a suitable solvent which passes over the sample carrying along with it, the different components of the sample to different extents. But how does this happen?

Which mechanism is operating in such a separation?Let us find out.

So far we have not deliberated upon the water present in the cellulose paper.But, this has the *actual role* in deciding the mechanism of separation. In fact, it is this water which is acting as the stationary phase. Here, the mobile phase is also a liquid. Thus, the components of the mixture will be separated between two liquid phaes and hence,paper chromatography is a *liquid-liquid chromatography .*

Therefore, the mechanism involved here is the ***partition*** of the componens in two different liquid phases. Every time the mobile phase moves, there is an equilibrium between the two liquid phases and the components partition themselves in the two phases.

The components which are held strongly by the stationary phase will be slow in moving alongwith the mobile phase while those which are weakly held by the stationary phase will be moving faster along with the mobile phase.

Thus,*different components of the mixture will move with different rates along with the mobile phase*. This eventually leads to their separation in due course of time.

However, this simple mechanism of partitioning is not taken as the only mode of separation in paper chromatography. It is now believed that hydrogen

bonding, interactions between solutes and cellulose support also play an important role in separation.

It is also possible to use **modified cellulose papers** in paper chromatography. The chemically modified papers such as carboxylated and acetylated papers are available. The carboxylated papers have increased carboxyl content and are suitable for the separation of amines and amino acids. The aceylated paper is more hdyrophilic and can be used for reverse phase chromatography.

The paper to be used in paper chromatography can also be loaded with cation or anion exchange resins. In such a case, the mechanism of separation would be via *excange of ions.*

It is also possible to use papers impregnated with silica or alumina, in which case, the mechanism of separation would involve adsorption of the components of the mixture on paper to different extents leading to their separation.

Hydrophilic papers can be obtained by treatment with methanol, glycerol, glycol etc.

Having understood the mechanism of separation,try to answer the following **SAQs.**

SAQ 2

What are modified cellulose papers?Give examples.

SAQ 3

What is the general mechanism of separation in paper chromatography? How does it get modified in case of papers impregnated with silica gel?

5.3.4 Development of Chromatograms

We have discussed above about the ascending, descending and circular forms of paper chromatography. Whatever form we choose from these, before starting the separation using paper chromatography ,the general procedure involves the saturation of the development tank with the vapours of the solvent used. The development tank containing the mobile phase is covered with the lid/cover and kept undisturbed for sometime to allow the vapours of the mobile phase to saturate the tank .

The substance to be analysed is dissolved in a suitable solvent and its spot of is put on the paper. Let the position of this spot be represented by the line at point A. The paper is then cap in the development chamber and it is then allowed to come in contact with the mobile phase. The system is again left undisturbed for sometime.

The components present in the substance are then carried along by the mobile phase. Their rates of movement will be different as their inteactions with the stationary and mobile phases will be different.

Here, it is also important to note that the nature of solvent used as the *mobile phase has an important role in the development of paper chromatogram.*

The solvent or the mobile phase should be pure, free from impurities and dry. The following other criteria should also be followed while chosing the mobile phase.

- The solvent system should not react chemically with the components of the substance to be analysed.
- The composition of the solvent system should not change during the course of separation. So, as far as voltility is concerned, we should prefer non-volatile solvents.
- The solvent system or the mobile phase should be so selected that it is able to separate different components present in the sample. The polarity of the solvent system can be changed for getting the good separation.

As in the case of paper chromatography, here also the water present in the cellulose acts as the stationary phase.Hence, here in paper chromatography , the starionary phase is polar in nature.Therefore.we can use a relatively less polar solvent such as ethanol, acetone, formamide, amines or a suitable mixture of such solvents as the mobile phase.

Some typical examples of such mobile phases are as follows:

- Isopropanol-Ammonia-Water (9:1:2 v/v)
- *n*-Butanol-Acetic acid-Water (4:1:5 v/v)
- Water-Phenol
- Formamide-Benzene

For the separation of cations, some of the commonly used mobile phases are as follows:

- Methanol
- Methyl ethyl ketone containing 30% (v/v) water and 1% (w/v) potassium thiocyanate
- Pyridine containing 10% (v/v) water
- Acetone containing 5% (v/v) water and 8% (v/v) hydro
- chloric acid.

The components of the substance get separated after leaving the paper in the development chamber for some time. The paper is then taken out from the development chamber and a line is marked on it with a pencil at the level to which the mobile phase has travelled. Let this line be represented by B. The solvent is then allowed to dry. Remember that the position of the original spot was represented as point A and a line can be drawn at this level, see Fig. 5.7.

If the separated components of the mixture are coloured, their spots will be clearly visible on the paper, see Fig. 5.6. Otherwise, a suitable locating agent is sprayed on the paper to detect the components.,

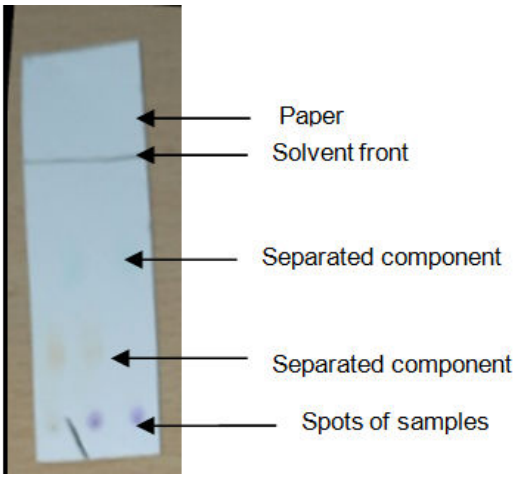


Fig. 5.6: Chromatogram

The examples of some locating agents are as follows:

- Dimethyl glyoxime (for Ni)
- Dithizone
- Potassium chromate
- Ammonium sulphide
- Iodine vapours (for organic compounds)
- Starch solution (for carbohydrates)
- Ninhydrin solution (for amino acids)

Sometimes, the spots of the components are visible under UV light.

Once the spots of the separated components are located, these then need to be characterised. One such way is to calculate their R_f values and match these values them with the R_f values of their standard or pure samples which are usually run simultaneously on the same paper under identical conditions.

For this purpose, let us first understand what is an R_f value.

The R_f value

It is the retardation factor and is defined as the following ratio:

$$R_f = \frac{\text{Distance travelled by a solute}}{\text{Distance travelled by the solvent}} \quad \ldots (5.1)$$

If we now look at the chromatogram shown in Fig. 5.6, then, we can calculate the R_f values for the separated components.

Let us understand this the calculation of the R_f values with the help of a hypothetical chromatogram shown in Fig. 5.7. Let there be two components 1 and 2 which have been separated by using the technique of paper chromatography (or TLC). Let the distance travelled by component 1 is AY and that travelled by component 2 is AX.

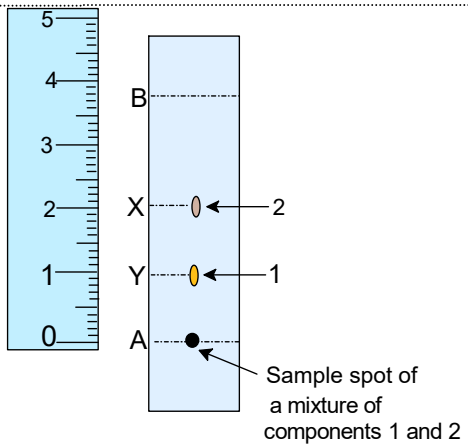


Fig. 5.7: Calculation of R_f value from chromatogram

Therefore, their R_f values can be calculated as given in the following equations:

For component 1, $$R_f = \frac{\text{Distance travelled by component 1}}{\text{Distance travelled by mobile phase}} = \frac{AY}{AB} \ldots (5.2\ a)$$

For component 2, $$R_f = \frac{\text{Distance travelled by component 2}}{\text{Distance travelled by mobile phase}} = \frac{AX}{AB} \ldots (5.2\ b)$$

It must be taken care that the distances AY and AX are measured between the line A and the centre points of spots Y and X, respectively.

Many times, the sample of the available standard substances are placed along with the mixture (or unknown substance). Then the chromatogram is obtained. The separated components of the mixture are then matched with the spots of the standard samples and the components are characterised, see Fig. 5.8.

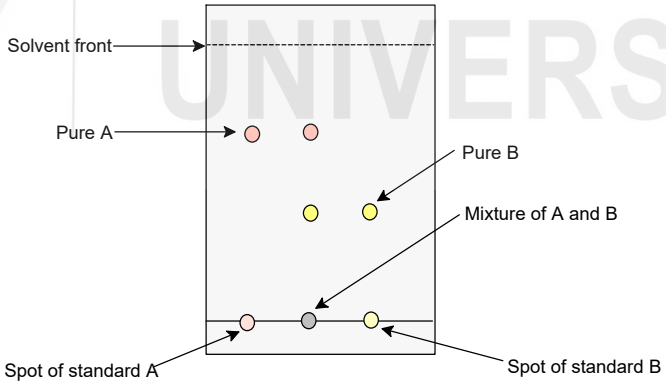


Fig. 5.8: Development of Chromatogram along with the use of standard samples

The R_f values are characteristic of the solute under a given set of conditions. They may change with the change in the solvent system, temperature, presence of impurities and sometimes with the development time. Hence, these need to be carefully compared.

Hence, we can say that paper chromatography can be used for the qualitative analysis of different samples and identification of components present in a mixture.

Now you can answer the following questions.

SAQ 4

Which component shown in Fig. 5.7 has smaller R_f value?

SAQ 5

Give any two characteristics of a mobile phase.

Let us now sum up, what we have learnt in this Unit.

5.4 SUMMARY

In this unit, we have learnt the following main aspects of chromatography:

- The chromatographic techniques can be classified in various ways according to the shape of the stationary support, nature of the mobile phase and the mechanism of separation.
- A variety of chromatographic techniques have evolved with variations in their operational aspects over the time.
- Paper chromatography is a very simple and useful chromatographic technique.
- Various modes of paper chromatographic techniques are available- ascending, descending and circular.
- The mechanism of separation using paper chromatography is mainly partition. However, other variations, i.e. adsorption and ion exchange using the modified papers, are also possible.
- The development of chromatograms is an important skill and several aspects related to placement of spots of samples, use of suitable mobile phases and proper handling of chromatogram are to be taken care of to achieve the good separation of the components. The spots of the sample and the reference substances should not dip in the solvent.
- The R_f values are characteristic of the substances but several factors may affect these values.

5.5 TERMINAL QUESTIONS

1 Name different types of mechanisms operating in the chromatographic techniques.

2. What are the polarities are two phases in reverse-phase chromatography?

3. Differentiate between ascending and descending modes of paper chromatography.

UNIT 6

ADSORPTION CHROMATOGRAPHY

Structure

6.1 INTRODUCTION

In Unit 5, you have studied about the classification of chromatographic methods and about *partition chromatography*, i.e. paper chromatography in detail.

In continuation to that, in this unit, you will study about one more type of chromatography, i.e. *column chromatography* in which the separation of the components of a mixture takes place by the *adsorption mechanism*.

We will begin the discussion of this unit by recapitulation of the classification of chromatographic methods dealt in Unit 5. Here , we will describe the principle and efficiency of the column chromatography technique. This will be followed by a brief account of the mechanism of separation operating in this technique. The important features of adsorption and partition chromatographic techniques will then be compared.

Finally, we will explain the development of chromatograms by *frontal analyses*, *elution* and *displacement* methods.

Expected Learning Outcomes

After studying this unit you should be able to:

- explain the classification of chromatographic techniques;

4. Give some examples of areas of application of paper chromatography.
5. List the factors which can affect the R_f value.

5.6 ANSWERS

Self Assessment Questions

1. Paper chromatography and thin layer chromatography.
2. Papers can be modified chemically to yield carboxylated and acetylated papers.
3. It is partition mechanism in paper chromatography when different components partition themselves between the two phases. In case of papers impregnated with silica gel, the mechanism involves adsorption of the components on the stationary phase to different extents.
4. Component 1.
5. It should be pure and should not react with the substance to be separated.

Terminal Questions

1. Partition, adsorption, ion exchange and size exclusion.
2. In reverse-phase chromatography, *non-polar support is* taken as the *stationary phase* and a *polar mobile phase* is taken.
3. Refer to Sec. 5.3.1.
4. Paper chromatography has applications in the following areas:
   - In the identifiction of metal ions
   - In the analysis of mixtures of organic compounds
   - To assess the purity of a given sample-organic and inorganic –both types
   - In the analysis of foods, drinks, pharmaceuticals, pollutants, fertilizers, drugs etc.
5. (i) Temperature change, (ii) change of solvent and (iii) the presence of impurities can change the R_f value.

- ❖ discuss the principle of column chromatography which is a form of liquid-solid chromatography;
- ❖ describe the efficiency of the column chromatography technique;
- ❖ explain the mechanism of adsorption involved in the separation of components of a mixture using column chromatography;
- ❖ compare the important aspect of adsorption and partition chromatographic techniques; and
- ❖ describe different methods of development of chromatograms.

6.2 CLASSIFICATION

You may remember from Unit 5, Sec. 5.2 that according to the ***shape of the stationary support*****,** chromatography can be classified as two-dimensional or three-dimensional chromatography. A portion of Fig 5.1 is shown below in Fig.6.1.

Both thin layer chromatography and column chromatography are the **liquid-solid chromatography techniques** as these involve the *liquid mobile phase* and *a solid stationary phase.*

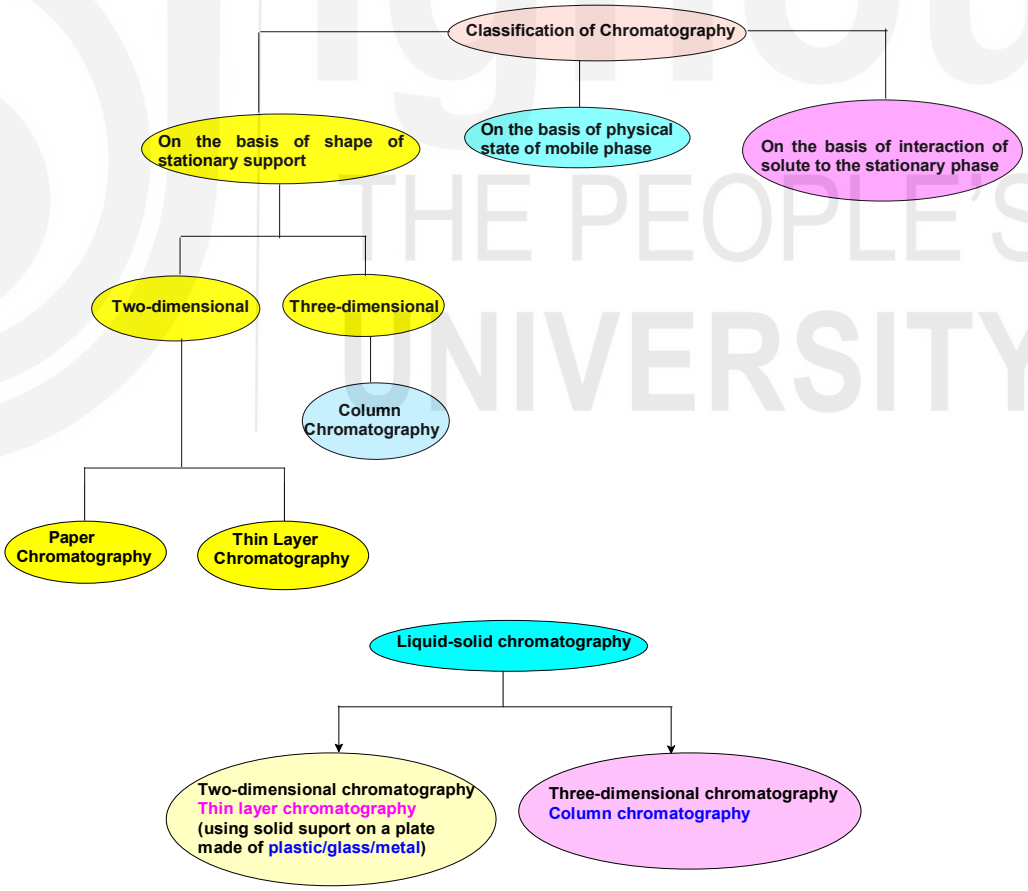


Fig. 6.1: Types of Liquid-solid Chromatography

The technique of thin layer chromatography is classified as the *two-dimensional chromatographic* technique but its discussion is beyond the scope of this course. Hence, in this unit, we will be dealing mainly with the *column chromatography* which is a *three-dimensional* chromatographic technique.

6.3 PRINCIPLE

The *column chromatography* involves the use of liquid mobile phase and a solid stationary phase; hence, it is a type of **liquid-solid chromatography** as mentioned above.

A solid stationary phase is used in *liquid-solid chromatography***.** Several stationary supports such as silica gel, activated alumina, magnesium carbonate, hydrated calcium silicate, talc, fuller's earth, sucrose and powdered cellulose can be used in liquid-solid chromatography. Thus, the availability of a large number of materials offer a wide range of flexibility in choosing the desired stationary phase in terms of the *surface area*, *particle size* and *type of adsorbent.*

As shown in Fig. 6.2 a column which is a long tube which is filled with the solid support. The mixture to be separated is first dissolved in a suitable solvent and its slurry is prepared which is then placed at the top of column. Different components of the mixture get adsorbed on the stationary phase to different extents.

When the mobile phase is allowed to pass over stationary phase, the component of the mixture which is least adsorbed on the stationary phase gets carried along the mobile phase at a faster rate than those components which are strongly adsorbed.

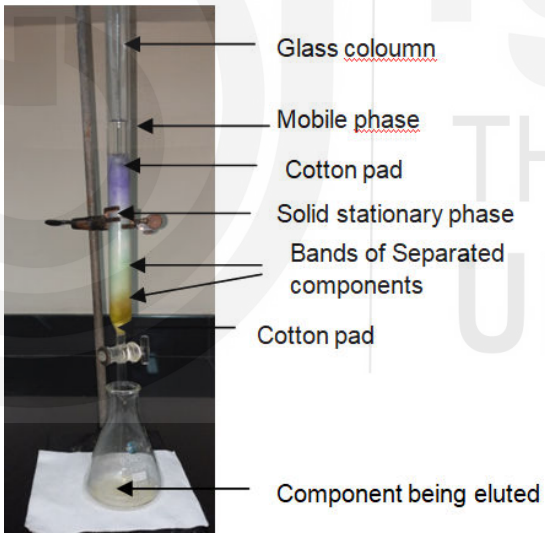


Fig. 6.2: Set up for the Column Chromatography and column showing different separated components.

The mobile phase is continuously added from the top of the column and allowed to move out of the bottom coloumn by opening the stop cock. The different components of the mixture keep moving downwards as bands in the column. These are then collected as different fractions in the flask placed at the bottom of the column. The different components are then obtained from the *eluent* by removal of the solvent.

SAQ 1

Name any two stationary phases which can be used in column chromatography.

6.4 EFFICIENCY OF THE TECHNIQUE

Column chromatography is a very useful and convenient technique, both for the effective separation of components of a mixture and for the purification of an impure compound. Virtually, any type of mixture – occurring naturally (in plants, animals or in any other source) or obtained synthetically from chemical reactions, can be separated into its components using column chromatography. The sample size needed for such a separation could vary from a few milligrams to grams.

Since a lot of stationary phase materials and different mobile phases-whether single solvents or suitable mixtures, can be used in column chromatography, it has the applications in many areas and many different types of compounds can be separated in pure form from their mixtures or impure samples.

The following points are to be kept in mind for this technique.

1. Type of Adsorbent or the Stationary Phase

Different types of compounds are adsorbed to different extents on different stationary phases.

Unsaturated, aromatic and polar compounds such as alcohols, acids and amines are adsorbed on polar adsorbents such as metal oxides and magnesium silicate.

Silica gel and magnesium silicate are *acidic* adsorbents and they adsorb bases. Alumina adsorbs both acidic and basic substances.

Kieselguhr is a weak adsorbent and shows no selectivity for adsorption of polar compounds. Similarly, sucrose is the weakest adsorbent while charcoal is the strongest adsorbent.

The adsorbents can be activated by heating when these lose water and other adsorbed molecules. Silica gel can be activated by heating from 100^{o}C to 120^{o}C for one hour while alumina is to be heated at 400^{o}C.

The following other characteristics of the adsorbent are also important:

- It should be colourless, easily available and inexpensive.
- It should not react with the sample.

2. Particle size of the Stationary Phase

Its particles should be of uniform size. The finely divided nature of stationary phase leads to better separations. Particle size from74 to149μ is suggested with the mesh size of 100 to 200.With still smaller size, the column packing may not be efficient and it may cause irregular zones. Also, it will be difficult for the solvent to pass through such a packed column.

3. Surface Area of the Stationary Phase

The larger the surface area available, the larger will the possibility of interaction of molecules adsorbed on the stationary phase with the mobile phase; hence, better will be the separation.

Also, the smaller size will provide more surface area. But, the size has to be optimally chosen as too small particle size also slows down the mobile phase and can have cracks in the column, as mentioned above.

4. Length of the Column

The longer the column, the better will be the separation. The ideal length to width ratio for the column is 20:1 or 30:1.

5. Sample to Adsorbent Ratio

Ideally, the sample to the adsorbent ratio is chosen as 1:20 to 1:50. Otherwise, more concentrated samples may not lead to clear separations.

6. Choice of Mobile Phase

The mobile phase should be appropriately selected. It should not be viscous and it should not react with the components. The flow rate of the solvent or the mobile phase should be uniform and not very fast. This will give better separation of bands of different components of the mixture without much tailing.

SAQ 2

How does particle size of stationary phase affect the separation in column chromatography?

6.5 MECHANISM OF SEPARATION

The separation of components of mixture using adsorption chromatography (such as by use of column chromatography) involves their *adsorption* on the stationary support. The mobile phase then displaces the different components selectively one by one.

The following aspects, thus, need to be considered for the separation process:

- How strongly the component is adsorbed to the stationary phase?
- How much is the surface area of the stationary phase?
- What is the binding strength of the mobile phase is there to get adsorbed on the stationary phase and how much it can displace the adsorbed components(s)?

So, as the process of *adsorption* is involved in such separations, the different forces involved are hydrogen bonding, van der Waals forces and dipole-dipole interactions.

Hence, the *polarity of the mobile phase* will also affect how effectively the separations can be carried out. The separations will also be affected by the nature of the stationary phase used. For example silica gel which is polar in nature; polar solvents may have better interactions with it as compared to the non-polar ones.

Having understood the details of the adsorption chromatography, let us now compare the two chromatography methods which have studied in this unit and the previous unit.

6.6 ADSORPTION AND PARTITION CHROMATOGRAPHY

So far we have studied that both the adsorption and partition chromatographic techniques have the following commons points:

- These chromatographic techniques are useful in separating the components of a mixture or in the purification of compounds.
- Both these techniques involve stationary and mobile phases and the mobile phases in both these techniques are liquids.

However, the main differences between these techniques are as follows:

- In the adsorption chromatography, the separation of components occurs on the basis of their adsorption while in partition chromatography, the separation of components takes place by their partitioning between the stationary and the mobile phases.
- Adsorption chromatography is a *liquid-solid* chromatography while partition chromatography is a *liquid-liquid* chromatography.
- Adsorption chromatography requires more amount of solvent and more time than partition chromatography.

Needless to mention here that suitable mobile phase should be used and other factors need to be taken care of while separations using these chromatographic techniques are meaningfully achieved.

So far, we have learnt about many important aspects of adsorption chromatography. But, we are yet to know more about the development of the chromatogram, i.e. how to obtain the separated components of the mixture in the column.

Let us learn about it in detail. But before that answer the following SAQ.

SAQ 3

List the similarities between adsorption and partition chromatographic techniques.

6.7 DEVELOPMENT OF CHROMATOGRAMS: FRONTAL ANALYSES, DISPLACEMNET AND ELUTION METHODS

The development of chromatograms is a very important aspect of separations using column chromatography. The development of chromatograms can be done in different ways. It would be very interesting to

know about the difference between them and understand which method will be more useful in which situation. Let us study each one of these ways is more detail.

(i) Frontal Analysis

Let a mixture containing three compounds X, Y and Z, is to be separated by using frontal analysis. See Fig. 6.3 a) in which such a mixture is shown in the column as the top layer. The three compounds X, Y and Z are shown by red, green and blue colours.

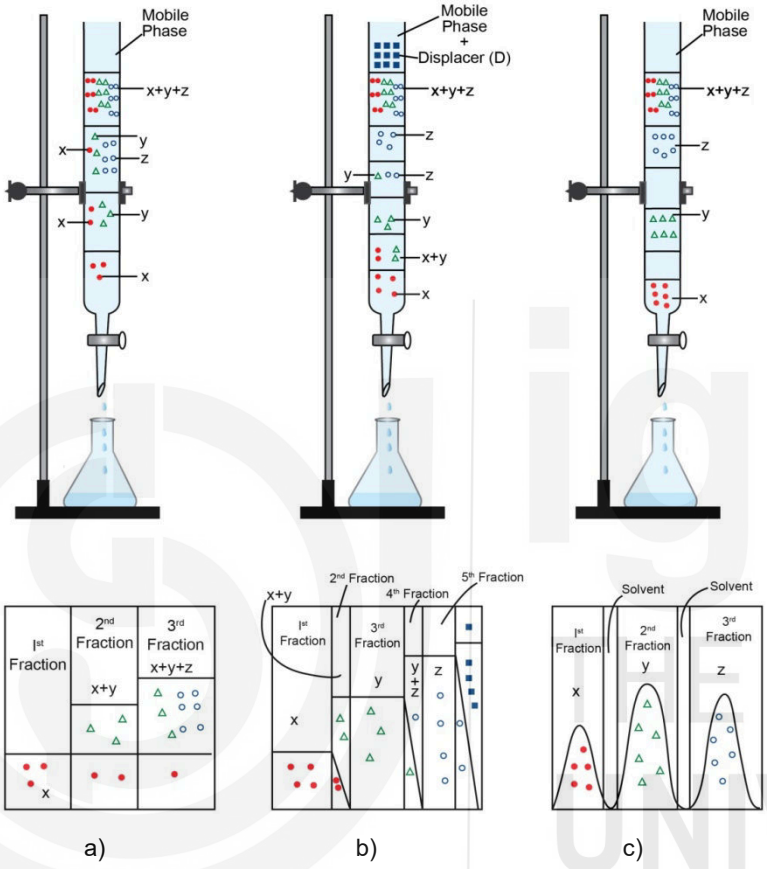


Fig. 6.3: a) Frontal analyses, b) Displacement and c) elution methods with their graphical representations.

- Note that before placing the mixture (X+Y+Z) on the column, the column has been saturated with the mobile phase. A large amount of the sample of X+Y+Z is introduced from the top. Then the mobile phase is continuously added from the top and the components start passing down the column along with the mobile phase. The least adsorbed component X (represented as▪) will reach the bottom and will be eluted first.
- Next to it, in the second fraction, a mixture of X+Y will be eluted.
- Finally, the third fraction will contain X+Y+Z, i.e. all the three components. Such a separation is shown in graphical mode also in Fig.6.3 below a).

Thus, in such a mode of separation, *only one component has been obtained in the pure form.*

Such separations were used long back when chromatography methods were *not* that much advanced.

Also, this mode of development of chromatogram was useful when only one component which was the desired component , was to be obtained in pure form and it could not be separated from the mixture by other methods.

But, otherwise such a method is not very useful.

(ii) Displacement Method

Here, again we start with the same mixture but this time we *add a displacing agent with the mobile phase*. Let all the components X, Y and Z are strongly adsorbed to the stationary phase. Here, the displacing agent is so taken that it is more strongly adsorbed than any of the components X, Y and Z. Thus, the displacing agent will compete with X, Y and Z for the adsorption sites and displace X, Y and Z into the mobile phase.

The order in which X, Y and Z are displaced will be according to the fact that the least out of the three will be desorbed first and hence, eluted first. Hence, will be desorbed first and eluted first. Let us say that it is the X which is eluted first.

This will be followed by another fraction in which both X and Y will be eluted together, i.e. Y will also start getting displaced along with X. After that, pure Y will get eluted.

Next, a mixture of Y and Z will start coming out in the eluent. Lastly, pure Z will get eluted. Then, at the end, the displacing agent will be passing out of the column.

Here also, this method is not perfect for separation as the quantitative amounts of pure X, Y and Z are *not obtained* at the end of the procedure. The graphical representation of such a separation is shown in the lower part in b) in Fig. 6.3.

Let us now study what happens in elution method.

(iii) Elution Method

Let us start with the same mixture of X+Y+Z taken at the top of the column. Here, there are two options of elution-*isocratic elution* and *gradient elution*.

In **isocratic elution**, the solvent or the mobile phase having the same composition (if a mixture of two or more solvents is taken) is used throughout the separation process. Different fractions of components start appearing as different bands in the column depending upon which components get eluted first. Again, the one which is least adsorbed will come out first from the column. Let this be X, here also.

But, if the other components e.g. Y and Z are adsorbed to quite different extents; then, the use of a single solvent, i.e. mobile phase of the single composition many not be able to move Y and Z down the column along with the mobile phase. Here, the *gradient elution* is used .The solvents of different polarities, i.e. with increasing order polarities are used to elute the different

components Y and Z in the pure form with a reasonable gap or difference between their bands in the column.

Thus, all the three components X, Y and Z could be separated in the pure form quantitatively using elution method.

The graphical representation in c) in Fig. 6.3 clearly shows this.

Here, we can conclude that elution method, especially the gradient elution is the most useful method for separating the components of a mixture using column chromatography.

SAQ 4

Why is gradient elution more useful than the other methods of development of chromatograms?

6.8 SUMMARY

In this unit, we have learnt that

- Adsorption chromatography is classified as the three-dimensional chromatographic technique.
- Adsorption chromatography is based on the phenomenon of adsorption of various components of a mixture on the stationary phase.
- The mobile phase on passing on to the stationary phase, carries along with it, the least adsorbed component at a faster rate than those which are strongly adsorbed on the stationary phase.
- The technique of column chromatography is very efficient method for the separation of a wide range of mixtures.
- A variety of adsorbents and mobile phases are used in column chromatography for separating different mixtures.
- Adsorption and partition chromatographic techniques have similarities as well as differences and these are useful as per the requirement of the separation.
- The gradient elution is more useful than the frontal analysis and the displacement methods.

6.9 TERMINAL QUESTIONS

1. What factors need to be taken care of with respect to the mobile phase to achieve better separations in column chromatography?
2. Discuss the activation of adsorbents.
3. Why is frontal analysis not a good method of the development of chromatogram?

6.10 ANSWERS

Self Assessment Questions

1. Silica gel, alumina or any other.

2. Particle size of 74-149 μ is recommended.

   The smaller the particle size, the better is the separation. But too small particles may lead to irregularities in the packing of the column. Hence, the particle size needs be carefully chosen.

3. These chromatographic techniques are useful in separating the component of a mixture or in the purification of compounds.

   Both these techniques involve stationary and mobile phases and the mobile phase in both these techniques are liquids.

4. The gradient elution is more useful than the other methods of development of chromatograms because by using elution method, all the components of a mixture could be separated in the pure form quantitatively.

Terminal Questions

1. The mobile phase should not be viscous and should not react with the components of the mixture. Further, the flow rate should not be fast and should be uniform.

2. The adsorbents can be activated by heating when these lose water and other adsorbed molecules. Silica gel can be activated by heating from 100^{o}C to 120^{o}C for one hour while alumina is to be heated at 400^{o}C.

3. This is so because in such a mode of separation, only one component has been obtained in the pure form.

UNIT 7

ION EXCHANGE CHROMATOGRAPHY

Structure

7.1 INTRODUCTION

You have studied about the charomatographic techniques based on partition and adsoprtion respectively, in Units 5 and 6 of this Block.In this unit ,you will study about one more chromatographic technique called **ion exchange chromatography** based on ion exchange .

Ion exchange has the popular application in water softening. The analytical applications of ion exchange chromatography include the analysis of sugars, amino acids, proteins, nucleotides, pharamaceuticals,clinical samples etc.

Ion exchange is also important in the recovery of metals from industrial wastes, separation of rare earths, decontamination of cooling water from nuclear reactors. Glen T. Seaborg also utilised this technique to identify elements of the 5*f* series which were obtained in a sequence similar to that for 4*f* elements.

We will begin the disussion of this unit by describing the types of materials which are used for ion exchange. Then, the principle of ion exchange chromatography will be explained alongwith the ion exchange capacity.

Finally, we will focus our attention on the mechanism of ion exchange.

Expected Learning Outcomes

After studying this unit, you should be able to:

- explain the importance of ion exchange chromatography;

- ❖ give examples of natural and synthetic ion exchangers;
- ❖ classify the exchangers as weak and strong ion exchanges;
- ❖ discuss the principle of ion exchange chromatography;
- ❖ define ion exchange capacity of a resin;
- ❖ explain the mechanism of ion exchange; and
- ❖ describe different factors affecting the retention time of ions on the resin and hence, affecting the efficiency of separation.

7.2 ION EXCHANGE MATERIALS

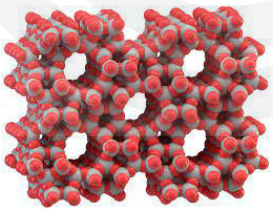
The phenomenon of ion exchange was observed by two agricultural chemists Thomson and Way in 1850 who observed the exchange of ammonium ions with calcium ions in the soil. The use of clay minerals for ion exchange was thus realised and their use for water softening was attempted.

Synthetic ion exchange resins have found widespread laboratory and industrial applications for water softening, water deionisation, purification of solutions and separation of ions.

Ion exchange is a process in which there is an exchange of ions of like sign between a solution and an insoluble solid in contact with the solution. Clearly, the solid must contain ions of its own to exchange, in addition, it must have a permeable structure of large in addition, it must have a permeable structure of large specific surface area so that the solvent and solute ions can readily come in contact with the solid surface.

During the course of time, more such materials were synthesised. The first synthetic ion exchanger was prepared by two German chemists – Harm and Rumpler in 1903. Meanwhile, permutits were also tried. However, in 1935, Adams and Holmes – two English chemists observed that crushed phonograph records exhibited ion-exchange properties.

Before taking up the detailed description of synthetic ion exchange materials, let us first know about some natural ion exchangers.



Zeolites are aluminosilicate minerals that contain alkali and alkaline-earth metals. They are three-dimensional, microporous, crystalline solids with cavities and channels in which cations, water or small molecules can reside. They are often called molecular sieves.

Natural ion exchangers: These include *zeolites* having cation exchange properties. Their examples being the following minerals:

(i) *Analcite* – $Na\,[SiAlO_6]_2.\,H_2O$

(ii) *Chabazite* – $(CaNa)\,[SiAlO_6]_2.\,6\,H_2O$

(iii) *Naturalite* – $Na_2\,[Si_2Al_2O_{10}].\,2\,H_2O$

These have three dimensional networks with negatively charged lattice. The alkali and alkaline earth cations which move freely, balance the negative charge. These behave as *counter ions* and can be exchanged with other counter ions.

The exchange of ^{-}OH ions for Cl^{-}, SO_4^{-} and PO_4^{3-} ions has been reported in *montmorillonite*, *kaolinite* and *feldspar* of sodalite and camerinite groups.

Zeolites have poor mechanical strength, less abrasive resistance and are decomposed partially by acids and alkalis.

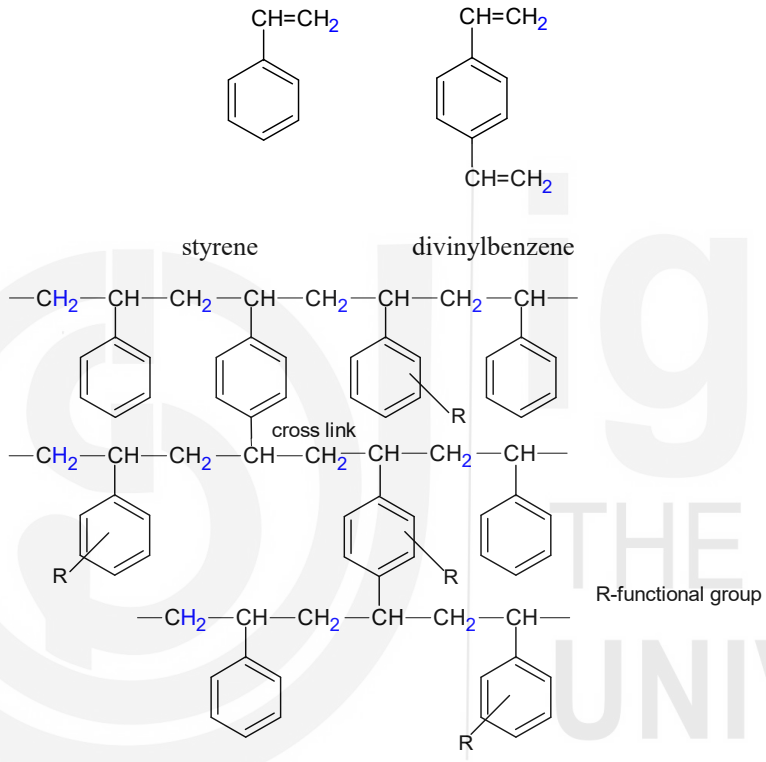
Some types of coals are also used as cation exchangers after stabilising with metal ion solutions. These contain weak acidic and carboxylic groups and are thus, weak cation exchangers. But, these can be converted to strong cation exchangers by sulphonation by treatment with strong sulphuric acid.

Let us now explore more about synthetic ion exchangers.

Ca^{2+} or Mg^{2+} forms a coating on the inner walls of a boiler and cannot be easily removed. This is known as scale formation. Then, zeolites are used for removing Ca^{2+} and Mg^{2+} ions from hard water before water is put in the boiler. These zeolites are known as molecular sieves.

Synthetic Ion Exchangers

High molecular weight copolymer beads made by copolymerisation of styrene with divinylbenzene are used. The use of ~80% divinylbenzene leads to cross linking of the polymer to give it mechanical stability. Acidic and basic functional groups are then bonded to this polymeric structure to convert it into weak or strong cation as well as anion exchangers as shown below in Fig. 7.1.



Exchange Reaction

Na_2Z (solid) + Ca^{2+} (solution) ⇄ CaZ (solid) + 2 Na^+ (solution)

Regeneration

CaZ + 2NaCl (10% solution) → Na_2Z (solution) + $CaCl_2$ (solution)

Fig. 7.1: Copolymer of styrene-divinylbenzene with R functionalised chain to convert it into cation or anion exchanger.

Table 7.1 shows different R groups corresponding to strong and weak cation and anion exchangers.

Table 7.1: Strong and Weak Cation and Anion Exchangers

Type of Ion Exchanger	Functional Group, R	Example
Weak cation exchanger	Carboxylic group	$-COO^-$, $-CH_2COO^-$
Strong cation exchanger	Sulphonic group	SO_3^-, $-CH_2CH_2SO_3^-$
Weak anion exchanger	Amine	$-\overset{+}{N}H_3$, $-CH_2CH_2\overset{+}{N}H(CH_2CH_3)$
Strong anion exchanger	Quaternary amine	$-CH_2\overset{+}{N}(CH_3)_3$, $-CH_2CH_2-\overset{+}{N}(CH_2CH_3)_3$

SAQ 1

Give examples of two natural ion exchangers.

SAQ 2

What is the role of divinylbenzene in the copolymer with styrene?

7.3 PRINCIPLE OF ION EXCHANGE

Ion exchange resins are porous and insoluble in aqueous medium. These contain many ionic functional groups per molecule as shown above in Fig. 7.1.

When such an ion exchanger is immersed in an aqueous solution, containing the ions to be exchanged, an equilibrium gets established.

Let us first understand this for *cation exchangers*. Let the resin, i.e., cation exchanger is immersed in a solution of metal ions, M^{n+} which are to be exchanged. We can write the equilibrium for both the weak and strong cation exchangers as follows:

$$n\,RzCOO^-H^+ + M^{n+}_{(aq)} \rightleftharpoons (RzCOO)_n M + nH^+_{(aq)} \qquad \ldots (7.1)$$

Weak cation exchanger

$$n\,RzSO_3^-H^+ + M^{n+}_{(aq)} \rightleftharpoons (RzSO_3^-)_n M^{n+} + nH^+_{(aq)} \qquad \ldots (7.2)$$

(solid) (solid)

Strong cation exchanger

Here, Rz denotes the resin.

The equilibrium constant, *K* or the *selectivity coefficient*, can be written for the reaction shown in Eq. 7.2 as follows:

$$K = \frac{[RzSO_3)_n M]\,[H^+]^n_{(aq)}}{[RzSO_3^-H^+]^n\;[M^{n+}]_{(aq)}} \qquad \ldots (7.3)$$

Hence, terms in the square brackets indicate the surface concentrations (or more strictly the *activities*).

Eq. 7.3 can be rearranged, in terms of the distribution ratio as follows:

$$R = \frac{\text{Amount of M in stationary phase}}{\text{Amount of } M^{n+} \text{ in aqueous phase}}$$

$$\frac{[RzSO_3)_n M]}{[M^{n+}]_{(aq)}} = \frac{[RzSO_3^-H^+]^n}{[H^+]^n_{(aq)}} \qquad \ldots (7.4)$$

Thus, the equilibrium in Eqs. 7.1 and 7.2 can be shifted to left or right by increasing $[H^+]$ or $[M^{n+}]$.

Weak cation exchangers are used in the pH range 5-14 while strong cation exchangers can be used in the pH range 1-14. Weak cation exchangers hold the protons tightly in the low pH range and hence, exchange will not occur.

Weak acid cation exchangers will not completely exchange cations of very weak bases. These are exchanged by strong cation exchangers only.

Weak acid cation exchangers, however, separate strongly basic substances e.g., peptides or proteins and multifunctional ionic compounds.

The strong cation exchangers are mostly used for separating the complex mixtures.

Let us now see what happens with the *anion exchangers*. The *anion exchange equilibrium* for the strong and weak anion exchangers can be represented as given below:

$$n\,RzNR_3^+\overline{O}H + A^{n-} \rightleftharpoons (RzNR_3)_n A + n\,OH^-$$

Strong anion exchange

and

$$n\,RzNH_3^+\overline{O}H + A^{n-} \rightleftharpoons (RzNH_3)_n A + n\,OH^-$$

Weak anion exchanger

Here, the ^-OH ions are exchanged for the anion A^{n-}. The strong anion exchangers work in the range of pH from 1-12 while weak anion exchangers work in the pH range 0-9.

The weak anion exchangers are used for strong acids.

Having understood the basic concepts of anion and cation exchangers, it is important to understand about their *capacity*. Let us understand it in more detail.

7.3.1 Ion Exchange Capacity

We have studied above that the counter ions of a resin are exchanged in an exchange process. The *counter ion content* in the given amount of an exchanger, is the fixed charges which are balanced by counter ion charges.

The *capacity* of an ion exchanger is the *number of ion equivalents in the specific amount of the material*. But, the context of its usage needs to be specified.

We define the *total capacity* of the resin as the number of ionic (or potentially ionic) sites per unit volume or weight of the resin.

The *dry weight total capacity* is usually expressed in milliequivalents per gram of *anhydrous* resin. It is expressed as meq/g dry H^+ or Cl^- form.

The *wet volume capacity* is number of sites per unit volume of the water swollen resin. It is the maximum theoretical capacity; however, the performance of the resin is generally based on its volume. It may be expressed as milliequivalents per millilitre.

The *operating capacity* of a resin in a particular cycle is the net number of sites utilised in a given volume in that cycle. It is expressed in the same terms as total capacity or as a percent of total capacity.

One more capacity is *dynamic* or *breakthrough capacity* which is used in column operation and depends upon the operating conditions.

The exchange capacity affects the retention of the solute. *The higher capacity, the higher retention and improved will be the resolution.*

Besides the *capacity* of the resin, the *particle size* of resin particles is also important. The preferred effective size is of diameter 0.4-0.6 mm. The corresponding particle size distribution is between 20-50 mesh screens. Note that hydrated particles (i.e., swollen resin) are present in the aqueous medium.

The decrease in the particle size, decreases the time required to attain the equilibrium; hence, the efficiency for a given volume of resin increases. In other words, *lesser volume will be required with smaller particle size*.

SAQ 3

Write the expression for the equilibrium constant for a weak anion exchange resin.

7.4 MECHANISM OF ION EXHCNAGE

The stationary phase in the ion exchange chromatography is the matrix containing the ionisable functional groups with fixed ions and carrying oppositely charged displaceable ions.

The mobile phase consists of an aqueous buffer system with small amount of organic solvent. Note that exchange of cations or anions occurs with the counter ions of the resin, as the case may be, if we are separating a mixture of cations or anions, respectively.

The *selectivity coefficient* determines the affinity of the resin for the particular ion. The large value of selectivity coefficient indicates strong tendency for the resin to retain the ion and *vice-versa*.

For a typical strong cation exchange resin, the decreasing order of selectivity coefficients is

$Al^{3+} > Ba^{2+} > Pb^{2+} > Ca^{2+} > Ni^{2+} > Cd^{2+} > Cu^{2+} > Co^{2+} > Zn^{2+} > Mg^{2+} > Ag^{+} > K^{+} > NH_4^{+} > Na^{+} > H^{+} > Li^{+}$

This sequence indicates that the *highly charged ions bind more strongly as compared to ions with lower charge. For the ions having similar charge, the ions with smaller hydrated radii or those which are more polarisable bind more strongly to the resin.*

Similarly, the order for strong anion exchangers is as follows:

$$SO_4^{2-} > I^{-} > HSO_4^{-} > NO_3^{-} > Br^{-} > NO_2^{-} > Cl^{-} > HCO_3^{-} > CH_3COO^{-} > {}^{-}OH > F^{-}$$

Here again, *the highly charged ions and those with smaller hydrated radii bind more strongly to the resin*.

With regard to the aqueous buffer mobile phase, the pH and the ionic composition are related to the retention time. Gradient elution involving the change in pH or ionic strength, is used for eluting different cations. Here, if dil. HCl is used as the mobile phase, then the increase in the concentration of HCl increases the elution rate as more H^+ ions are now available to compete successfully for the ion-exchange sites.

SAQ 4

Why is the selectivity coefficient important?

7.5 SUMMARY

In this Unit, you learnt that

- Ion exchange materials are useful materials for separating a variety of substances containing complex mixtures.
- Ion exchange materials could be natural or synthetic ones. These are categorised as cation exchange materials or anion exchange materials. Further, these could be weak or strong ion exchangers.
- For cation exchange, there is a choice between strong acid groups (RSO_3H) or weak acid resins containing carboxylic acid (RCOOH) groups. The former has wider applications. Anion exchange resins contain basic functional groups attached o the polymer molecule. These are generally amines; strong base exchanges are obtained with tertiary amines and (quaternary ammonium salts) $[RN(CH_3)_3^+OH^-]$ and weak types with primary and secondary amines.
- Synthetic ion exchange resins are high molecular weight polymeric materials containing large number of ionic functional groups per molecule.
- Ion exchange capacity of an exchanger can be defined in different ways.
- The particle size of the resin plays an important role in the retention and hence, separation of materials. The other features being the temperature and the pH of the mobile phase.
- The mechanism of ion exchange involves exchange of counter ions of the resin. The selectivity coefficient determines the affinity of the ion for the resin.
- Aqueous buffers along with some organic solvent is used in elution.
- Gradient elution using change of pH and ionic strength can separate a mixture into its components.

7.6 TERMINAL QUESTIONS

1. What are the limitations of zeolites as ion exchangers?
2. What functional groups are present in a strong cation exchanger and a weak anion exchanger?

3. Differentiate between dry weight total capacity and wet volume capacity of a resin.

7.7 ANSWERS

Self-Assessment Questions

1. Analcite, chabazite or any other suitable example.

2. It acts as a cross linking agent.

3. $$K = \frac{[(RzNH_3)_n A]\,[^{-}OH]^n}{[RzNH_3^{+}\ ^{-}OH]^n\,[A^{n-1}]}$$

4. The *selectivity coefficient* determines the affinity of the resin for the particular ion. The large value of selectivity coefficient indicates strong tendency for the resin to retain the ion and *vice-versa*.

Terminal Questions

1. Zeolites have poor mechanical strength, less abrasive resistance and are partially decomposed by acids and alkalis.

2. Strong cation exchangers have $-SO_3^{-}\ ^{+}H$ group and weak anion exchangers have $-\overset{+}{N}H_3$ group.

3. The dry weight total capacity is total capacity of anhydrous resin whereas wet volume capacity is the number of sites per unit volume of water- swollen resin.