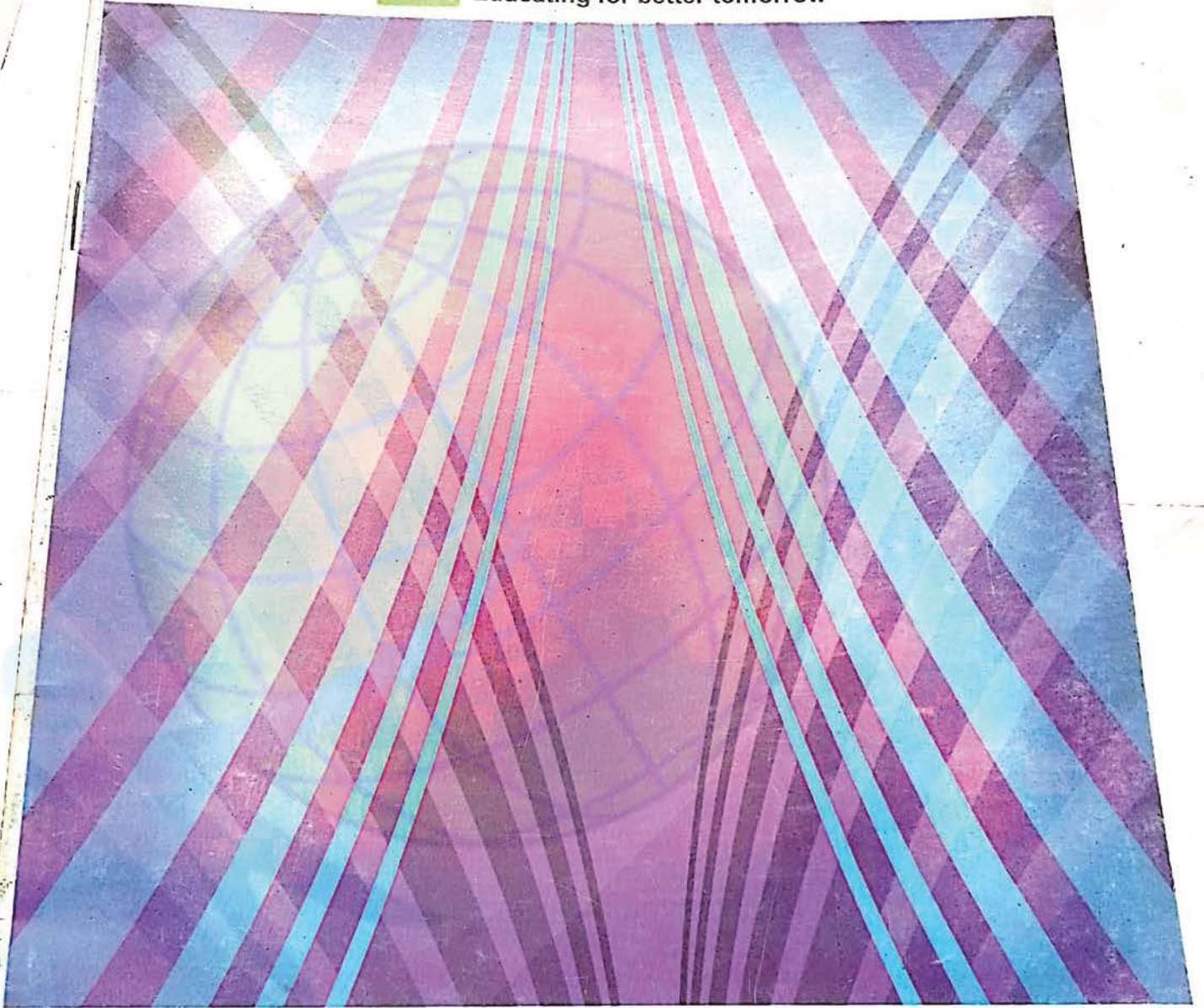




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# CHEMISTRY

Target : JEE (Main)

METALLURGY



## METALLURGY

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## JEE (MAIN) SYLLABUS

**General Principles and Processes of Isolation of Metals**

Modes of occurrence of elements in nature, minerals, ores; steps involved in the extraction of metals- concentration, reduction (chemical and electrolytic methods) and refining with special reference to the extraction of Al, Cu, Zn and Fe ; Thermodynamic and electrochemical principles involved in the extraction of metals.

## JEE(ADVANCED) SYLLABUS

**Extractive metallurgy:** Chemical principles and reactions only (industrial details excluded); Carbon reduction method (iron and tin); Self reduction method (copper and lead); Electrolytic reduction method (magnesium and aluminium); Cyanide process (silver and gold).

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# Metallurgy

## Introduction :

The compound of a metal found in nature is called a mineral. The minerals from which metal can be economically and conveniently extracted are called ores. An ore is usually contaminated with earthy or undesired materials known as gangue. So all minerals are not ores but all ores are minerals. Ores may be classified mainly into following four classes.

- (a) **Native ores** : They contain the metal in free state. Silver, gold, platinum etc, occur as native ores.
- (b) **Oxidised ores** : These ores consist of oxides or oxysalts (e.g. carbonates, phosphates, sulphates and silicates) of metals.
- (c) **Sulphurised ores** : These ores consist of sulphides of metals like iron, lead, zinc, mercury etc.
- (d) **Halide ores** : These ores consist of halides of metals.

## Important ore :

### 1. Oxide ore :

- (i) Bauxite  $\rightarrow \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$  (Major ore of Al)
- (ii) Haematite  $\rightarrow \text{Fe}_2\text{O}_3$
- (iii) Zincite = ZnO
- \*\*\* (iv) Cassiterite or tin stone  $\rightarrow \text{SnO}_2$  (Black Tin = 60 - 70%  $\text{SnO}_2$ )

### 2. Carbonate ore :

- (i) Lime stone  $\rightarrow \text{CaCO}_3$
- (ii) Dolomite  $\rightarrow \text{CaCO}_3 \cdot \text{MgCO}_3$
- (iii) Siderite  $\rightarrow \text{FeCO}_3$
- (iv) Malachite  $\rightarrow \text{CuCO}_3 \cdot \text{Cu(OH)}_2$
- (v) Azurite  $\rightarrow 2\text{CuCO}_3 \cdot \text{Cu(OH)}_2$
- \*\*\* (vi) Calamine  $\rightarrow \text{ZnCO}_3$
- \*\*\* (vii) Cerussite  $\rightarrow \text{PbCO}_3$

### 3. Sulphate ore :

Epsom salt  $\rightarrow \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$

### 4. Silicate ore :

Feldspar  $\rightarrow \text{KAISi}_3\text{O}_8$

### 5. Sulphurised ore :

- Consist of sulphides of metal like Iron, lead, zinc & mercury.
- (i) Iron pyrites  $\rightarrow \text{FeS}_2$
  - (ii) Copper glance  $\rightarrow \text{Cu}_2\text{S}$
  - (iii) Copper pyrite or chalcopyrite  $\rightarrow \text{CuFeS}_2$
  - (iv) Zinc Blende  $\rightarrow \text{ZnS}$
  - (v) Galena  $\rightarrow \text{PbS}$
  - (vi) Cinnabar  $\rightarrow \text{HgS}$

### 6. Halide ore :

- (i) Cryolite  $\rightarrow \text{Na}_3\text{AlF}_6$
- (ii) Sylvine  $\rightarrow \text{KCl}$
- \*\* (iii) Carnallite  $\rightarrow \text{KCl} \cdot \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$
- (iv) Fluorspar  $\rightarrow \text{CaF}_2$
- (v) Horn silver  $\rightarrow \text{AgCl}$

Note : Mg obtained from both sea water & earth crust.

## Solved Examples

### Example-1

Which metals are supposed to occur in the native state in nature ?

### Solution

Elements below hydrogen in the electrochemical series like Cu, Ag, Au etc, exist native ores.

### Example-2

Match the ores listed in Column-I with their correct chemical formula listed in Column-II.

#### Column I

- (A) Cassiterite
- (B) Siderite
- (C) Cerussite
- (D) Anglesite

#### Column II

- (p)  $\text{FeCO}_3$
- (q)  $\text{SnO}_2$
- (r)  $\text{PbSO}_4$
- (s)  $\text{PbCO}_3$

### Solution

$\text{SnO}_2$  is called as cassiterite or tin stone,  $\text{FeCO}_3$  is called as siderite,  $\text{PbCO}_3$  is called as cerussite and  $\text{PbSO}_4$  is called anglesite. So correct match is (A)  $\rightarrow$  (q), (B)  $\rightarrow$  (p), (C)  $\rightarrow$  (s) and (D)  $\rightarrow$  (r).



Metallurgy

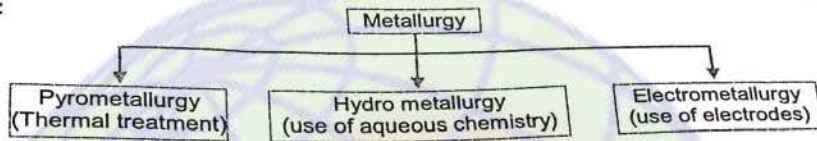
**Metallurgy :**

The scientific and technological process used for the extraction/isolation of the metal from its ore is called as metallurgy.

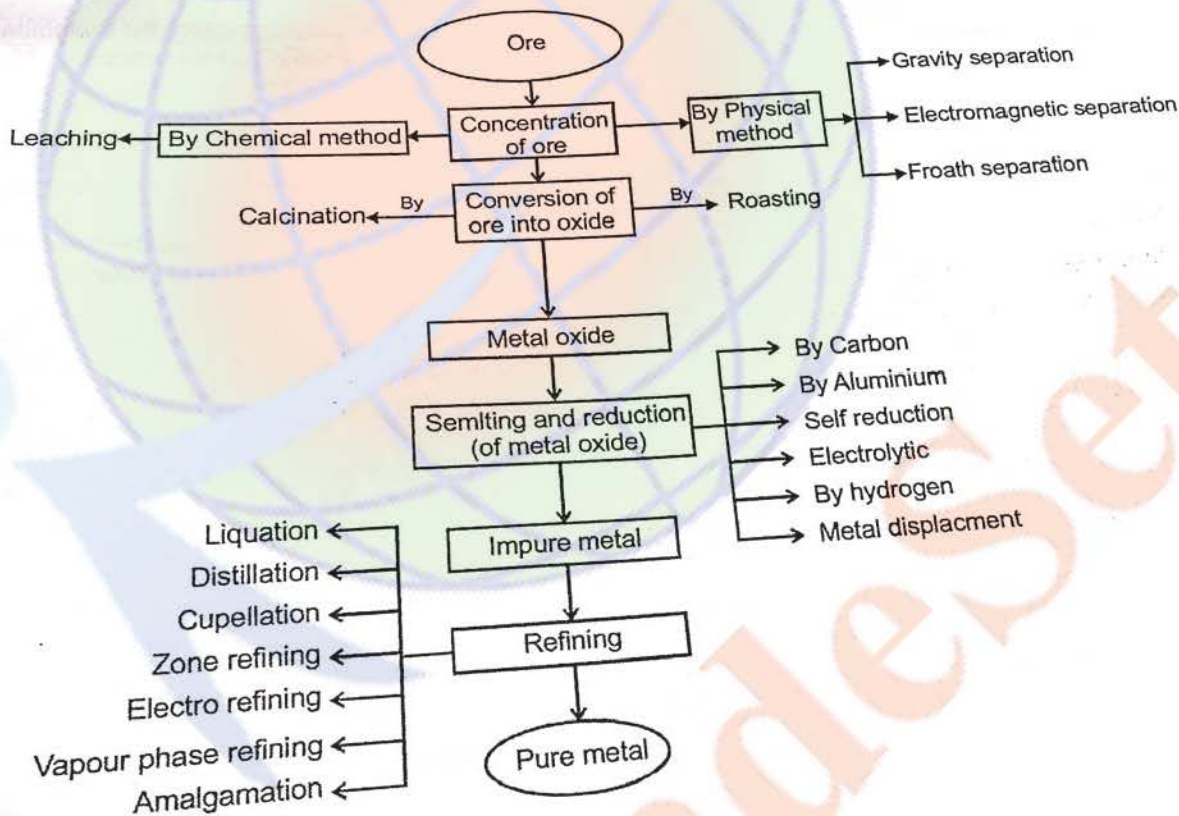
The isolation and extraction of metals from their ores involve the following major steps:

- (A) Crushing of the ore.
- (B) Dressing or concentration of the ore.
- (C) Isolation of the crude metal from its ore
- (D) Purification or refining of the metal.

**Chart1:**



**Chart2: Steps involved in metallurgy.**



**1. Physical Method :**

- (A) **Crushing and Grinding :** The ore is first crushed by jaw crushers and ground to a powder (pulverisation of the ore) in equipments like ball mills and stamp mills.
- (B) **Concentration or benefaction of ore.** The removal of unwanted useless impurities from the ore is called dressing, concentration. It involves several steps and selection of these steps depends upon the difference in physical properties of the compound of metal and that of gangue. Some of the important procedures are described below.



Metallurgy

**(i) Hydraulic washing or Gravity separation or Levigation method :** It is based on the difference in the densities of the gangue and ore particles. In this, the powdered ore is agitated with water or washed with an upward stream of running water, the lighter particles of sand, clay etc are washed away leaving behind heavier ore particles. For this either hydraulic classifier or Wilfley table is used. This method is generally used for the concentration of oxide and native ores.

**(ii) Electromagnetic separation :** It is based on differences in magnetic properties of the ore components.

It is used when either the ore or the impurities associated with it are magnetic in nature. A magnetic separator consists of a belt (of leather or brass) moving over two rollers, one of which is magnetic. When the powdered ore is dropped on the belt at the other end, magnetic component of the ore is attracted by the magnetic roller and falls nearer to the roller while the non-magnetic impurities fall away from it.

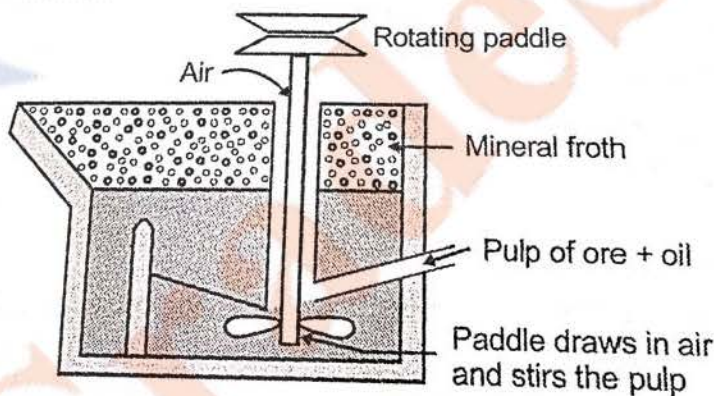
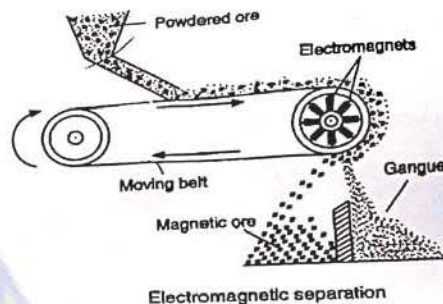
**Examples :** Chromite ore ( $FeO \cdot Cr_2O_3$ ) is separated from non-magnetic silicious impurities and cassiterite ore ( $SnO_2$ ) is separated from magnetic Wolframite ( $FeWO_4 + MnWO_4$ ).

**(iii) Froth floatation process :** This method is commonly used for the concentration of the low grade sulphide ores like galena,  $PbS$  (ore of  $Pb$ ) ; copper pyrites  $Cu_2S \cdot Fe_2S_3$  or  $CuFeS_2$  (ore of copper) ; zinc blende,  $ZnS$  (ore of zinc) etc., and is based on the fact that gangue and ore particles have different degree of wettability with water and pine oil; the gangue particles are preferentially wetted by water while the ore particles are wetted by oil. In this process one or more chemical frothing agents are added.

**(a) Frothers :** These form stable froth which rises to the top of the floatation cell. Oils like pine oil, camphor oil etc., are used as frothers. These are added in small quantity. The stabiliser are added to the frothers so that the froth can last for longer period.

**(b) Collectors :** Potassium or sodium ethyl xanthate is used as a collector. These get attached with the particles of the sulphide ore and thus make them water-repellant. Consequently the ore particles pass on into the froth. Collectors are always added in small quantity.

**(c) Activating and depressing agents :** When a mineral contains other minerals as impurities. The addition of these agents activates or depresses the floatation property of other minerals present as impurities and thus helps in separating the impurities. For example galena ( $PbS$ ) usually contains the minerals namely zinc blende ( $ZnS$ ) and pyrites ( $FeS_2$ ) as impurities. Flotation is carried out by using potassium ethyl xanthate (used as a collector) along with  $NaCN$  and  $Na_2CO_3$  (used as depressing agent). The addition of  $NaCN$  and  $Na_2CO_3$  depresses the floatation property of  $ZnS$  and  $FeS_2$  grains, so mainly  $PbS$  passes into the froth when air is blown in. After  $PbS$  has been collected with the froth, the process is repeated by adding  $CuSO_4$  (activator) which activates the floatation property of  $ZnS$  grains which are now removed with the froth. The acidification of the remaining material left in the floatation cell leads to the floatation of  $FeS_2$ .



Froth floatation process



**Solved Examples**

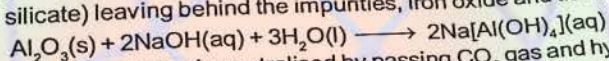
- Example-3** How does NaCN act as a depressant in preventing ZnS from forming the froth?  
**Solution** NaCN reacts with ZnS and forms a layer of  $\text{Na}_2[\text{Zn}(\text{CN})_4]$  complex on the surface of ZnS and thus prevents it from the formation of froth.
- Example-4** What is the role of stabiliser in froth floatation process?  
**Solution** Froth can last for a longer period in presence of stabiliser.

**2. Chemical Method :**

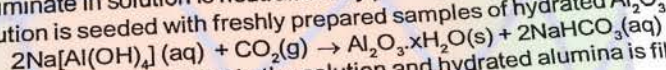
(iv) **Leaching** : Leaching is often used if the ore is soluble in some suitable solvent, e.g, acids, bases and suitable chemical reagents.  
 e.g. Al ore, Ag ore, Au ore, low grade copper ore.

**Leaching of alumina from bauxite :**

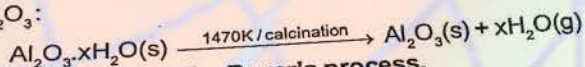
The principal ore of aluminium, bauxite, usually contains  $\text{SiO}_2$ , iron oxide and titanium oxide ( $\text{TiO}_2$ ) as impurities. Concentration is carried out by digesting the powdered ore with a concentrated solution of NaOH at 473-523 K and 35-36 bar pressure. This way,  $\text{Al}_2\text{O}_3$  is leached out as sodium aluminate (and also  $\text{SiO}_2$  as sodium silicate) leaving behind the impurities, iron oxide and titanium oxide.



The aluminate in solution is neutralised by passing  $\text{CO}_2$  gas and hydrated  $\text{Al}_2\text{O}_3$  is precipitated. At this stage, the solution is seeded with freshly prepared samples of hydrated  $\text{Al}_2\text{O}_3$  which induces the precipitation.



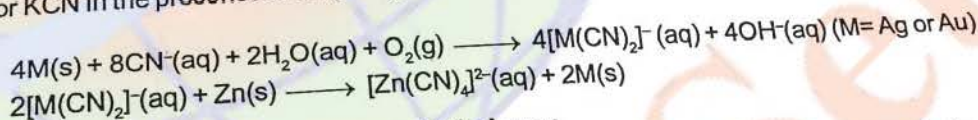
The sodium silicate remains in the solution and hydrated alumina is filtered, dried and heated to give back pure  $\text{Al}_2\text{O}_3$  :



These steps comprises the **Bayer's process**.

**Other examples :**

In the metallurgy of **silver** and that of **gold**, the respective metal/ore is leached with a dilute solution of NaCN or KCN in the presence of air (or  $\text{O}_2$ ) from which the metal is obtained later by displacement with zinc scrap.

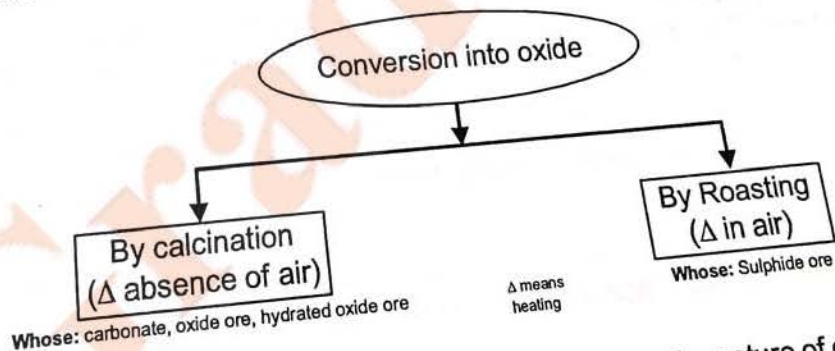


(C) **Extraction of crude metal from concentrated ore :**

The concentrated ore must be converted into a form which is suitable for reduction. Usually the sulphide ore is converted to oxide before reduction. Oxides are easier to reduce. Thus isolation of metals from concentrated ore involves two major steps as given below.

- (i) Conversion to oxide
- (ii) Reduction of the oxide to metal.

**(i) Conversion to oxide :**



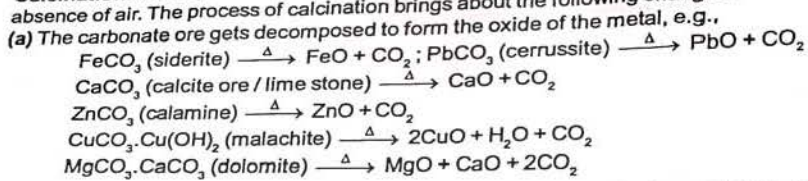
Conversion of ore into oxide is carried out in two ways depending upon the nature of ore.



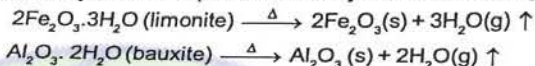


**Metallurgy**

**Calcination.** It is a process of heating the concentrated ore strongly in a limited supply of air or in the absence of air. The process of calcination brings about the following changes :



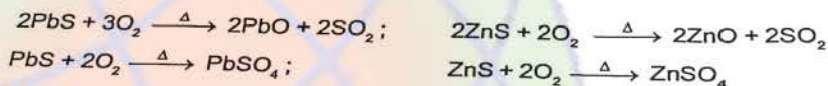
(b) Water of crystallisation present in the hydrated oxide ore gets lost as moisture, e.g.,



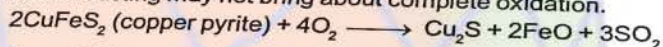
(c) Organic matter, if present in the ore, gets expelled and the ore becomes porous. Volatile impurities are removed.

**Roasting.** It is a process of heating the concentrated ore (generally sulphide ore) strongly in the excess of air or  $\text{O}_2$  below its melting point. Roasting is an exothermic process once started it does not require additional heating. The process of roasting does the following things :

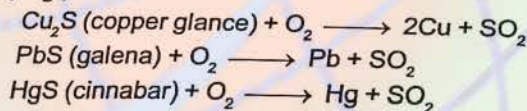
(a) Roasting at moderate temperature. Some portion of the sulphide ores like galena ( $\text{PbS}$ ), Zinc blende ( $\text{ZnS}$ ) is converted into metallic oxide. If the temperature is fairly low (about  $500^\circ\text{C}$ ) and the concentration of  $\text{SO}_2$  in the gaseous environment is more, sulphate may be produced that are stable, and high temperature is needed to decompose them.



\* Some times roasting may not bring about complete oxidation.

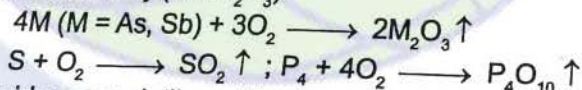


(b) Roasting at high temperature. The sulphide ores of some of the metals like Cu, Pb, Hg, Sb etc., when heated strongly in the free supply of air or  $\text{O}_2$  are reduced directly to the metal rather than to the metallic oxides, e.g.,



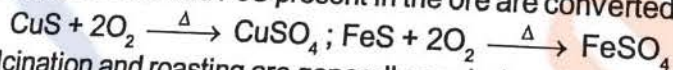
The reduction of the sulphide ore directly into metal by heating it in air or  $\text{O}_2$  is called by various names like self-reduction, auto-reduction, air-reduction etc. The  $\text{SO}_2$  produced is utilised for manufacturing of  $\text{H}_2\text{SO}_4$ .

(c) It removes easily oxidisable volatile impurities like arsenic (as  $\text{As}_2\text{O}_3$ ) sulphur (as  $\text{SO}_2$ ), phosphorus (as  $\text{P}_4\text{O}_{10}$ ) and antimony (as  $\text{Sb}_2\text{O}_3$ ).



These oxides are volatile and hence escape as gases through the chimney.

(d) When the concentrated tin stone ore  $\text{SnO}_2$  (ore of Sn) is heated strongly in a free supply of air (roasting), the impurities of  $\text{CuS}$  and  $\text{FeS}$  present in the ore are converted into  $\text{CuSO}_4$  and  $\text{FeSO}_4$  respectively.



Both calcination and roasting are generally carried out in a reverberatory furnace. In case of roasting, the air holes are kept open while they are partially or completely closed during calcination.

**Smelting :**

**Slag formation :** In many extraction processes, an oxide is added deliberately to combine with other impurities and form a stable molten phase immiscible with molten metal called a slag. The process is termed smelting.

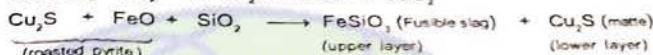


Metallurgy

The principle of slag formation is essentially the following :

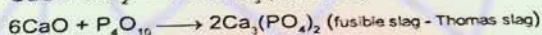
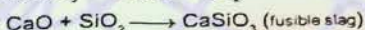
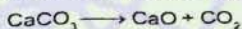
Nonmetal oxide (acidic oxide) + Metal oxide (basic oxide)  $\longrightarrow$  Fusible (easily melted) slag

Removal of unwanted basic and acidic oxides: For example, FeO is the impurity in extraction of Cu from copper pyrite.



Matte also contains a very small amount of iron(II) sulphide.

To remove unwanted acidic impurities like sand and  $\text{P}_2\text{O}_5$ , smelting is done in the presence of limestone.



**Properties of a slag :**

- (i) Slag is a fusible mass.
- (ii) It has low melting point.
- (iii) It is lighter than and immiscible with the molten metal. It is due to these impurities that the slag floats as a separate layer on the molten metal and can thus be easily separated from the metal. The layer of the slag on the molten metal prevents the metal from being oxidised.

**Type of flux :** Fluxes are of two types viz., acidic flux and basic flux.

(a) **Acidic flux :** It is an acidic oxide (oxide of a non-metal) like  $\text{SiO}_2$ ,  $\text{P}_2\text{O}_5$ ,  $\text{B}_2\text{O}_3$  (from borax). It is used to remove the basic impurity like CaO, FeO, MgO etc. The acidic flux combines with the basic impurity and forms a slag.

(b) **Basic flux :** It is a basic oxide (i.e., oxide of a metal) like CaO (obtained from lime stone,  $\text{CaCO}_3$ ), MgO (from magnesite,  $\text{MgCO}_3$ ), haematite ( $\text{Fe}_2\text{O}_3$ ) etc. It is used to remove the acidic impurity like  $\text{SiO}_2$ ,  $\text{P}_2\text{O}_5$  etc. The basic flux combines with the acidic impurity and forms a slag.

Thus, slag can be defined as a fusible mass, which is obtained when a flux reacts with an infusible acidic or basic impurity present in the oxide ore.

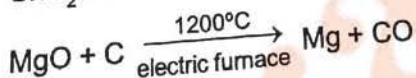
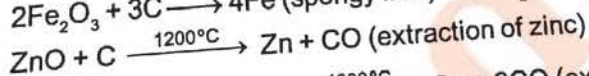
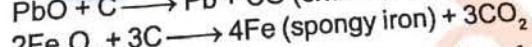
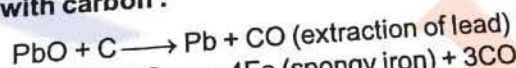
**(ii) Reduction of a metal oxide :**

The free metal is obtained by reduction of a compound, using either a chemical reducing agent or electrolysis.

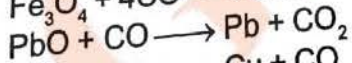
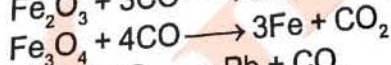
**1. Chemical reduction method :**

A large number of commercial processes come under this category. Carbon can be used to reduce a number of oxides and other compounds, and because of the low cost and availability of coke this method is widely used. The disadvantages are that a high temperature is needed, which is expensive and necessitates the use of blast furnace.

**(A) Reduction with carbon :**



**(B) Reduction with CO :** In some cases CO produced in the furnace itself is used as a reducing agent.





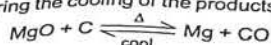


**Metallurgy**

Carbon or carbon monoxide reduction process is usually carried out in blast furnace. There are some disadvantages of using carbon as reducing agents e.g.  
 (a) Some metallic oxides like CaO give metallic carbides instead of metals.

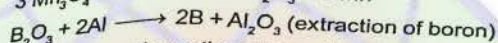
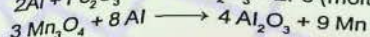


(b) During the cooling of the products, in many cases, reformation of the oxide and carbon may take place.

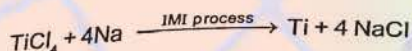
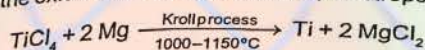


**2. Reduction by other metals :**

If the temperature needed for carbon to reduce an oxide is too high, for economical or practical purposes, reduction by other metals is done. Also, certain metallic oxides cannot be reduced by carbon because the affinity of oxygen for the metal is greater than its affinity for carbon. Such metallic oxides (Cr and Mn) can be reduced by a highly electropositive metal such as aluminium that liberates a large amount of energy (1675 kJ/mol) on oxidation to  $\text{Al}_2\text{O}_3$ . The process is known as Goldschmidt or aluminothermic process and the reaction is known as thermite reaction.



As it is a strongly exothermic reaction, it proceeds with explosive violence and only initial heating is needed. **Magnesium reduction method :** Magnesium is used in similar way to reduce oxides. In certain cases where the oxide is too stable to reduce, electropositive metals are used to reduce halides.

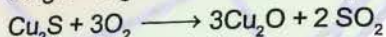


Advantages of using Na and Mg as reducing agents are the higher reducing power of the metals and solubility of their halides in water so that the reduced metals can be washed free from impurities.

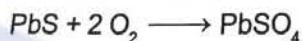
**3. Self-reduction method :**

This method is also called auto-reduction method or air reduction method. If the sulphide ore of some of the less electropositive metals like Hg, Cu, Pb, Sb, etc. are heated in air, a part of these is changed into oxide or sulphate then that reacts with the remaining part of the sulphide ore to give its metal and  $\text{SO}_2$ .

Examples :



The extraction of Pb by heating its sulphide ore ( $\text{PbS}$ ) in air can also be represented as



**4. Electrolytic reduction :**

It presents the most powerful method of reduction and gives a very pure product. As it is an expensive method compared to chemical methods, it is used either for very reactive metals such as magnesium or aluminium or for production of samples of high purity. Electrolytic reduction of copper has the additional advantage of allowing the recover of valuable minor contaminants such as silver.



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