CEMENT-BOOK

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Heidelberg Cement

Lime stone

- Limestone is a sedimentary rock such as greater than 50% **calcium** carbonate (**calcite** – $CaCO_3$). ...
- Colour: It can be yellow, white, or gray.
- **Chemical** Composition: **Calcite**.
- Texture – Clastic or Non-Clastic.
- Grain size – Variable, can consist of clasts of all sizes.
- Hardness – Generally hard.
- Lime Stone Total Carbonate Min = 75.0%

Note

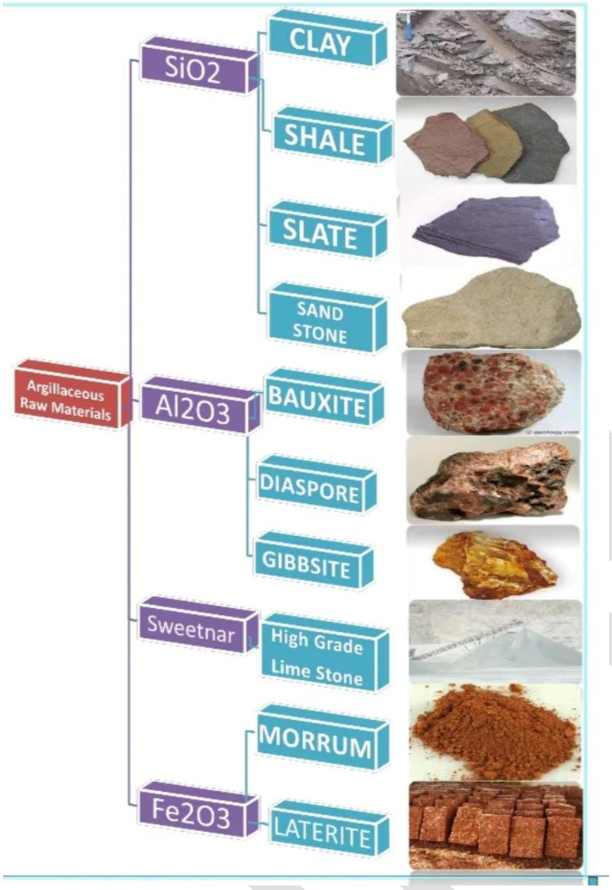
- IS 1760:1991 (Part- I to V) Methods of Chemical Analysis of Limestone.
- 1760 (Part 3):1992 Methods of chemical analysis of limestone, dolomite and allied material
- IS 2109 :1982 Methods of sampling of Dolomite, LS, iron, Etc
    - LOI – 1760 (P-1)
    - Silica – 1760(P-2)
    - Calcium Oxide -1760- (P-3)
    - Magnesium Oxide – 1760- (P-3) ,

Reaction

$$CaCo3 \xrightarrow[\Delta]{} CaO + Co2$$

100 56.0774 44.01

$$CaO + H2O \longrightarrow Ca(OH)2 \ (74.093)$$



*What is cement grade limestone?

Chemical **grade limestone** is used mainly in chemical industries producing quick lime and hydrated lime. White **Cement Grade Limestone** (+91% $CaCO_3$, Low Silica < 4.0%, Fe_2O_3 <0.1%) It is also used in White **Cement** and Grey **Cement** plants.

Lime stone- Geochemical analysis results

LOI- (29.94% to 40.64%) ,

$SiO2$ (6.14% to 27.18%),

CaO (37.93% to 50.78%),

$Al2O$- (0.49% to 2.27%)

$Fe2O3$ (0.28% to 2.4%).

IMP

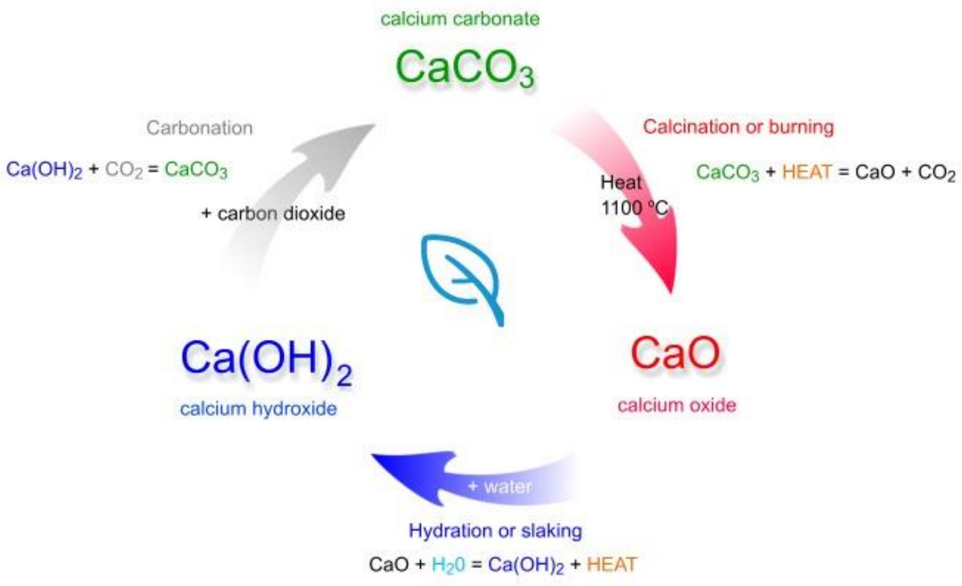
CC = TC – MC
CaO = CC / 1.786
MgO = MC / 2.0 9

TC – Total Carbonate

MC - Magnesium Carbonate

CC – Calcium Carbonate

Lime Cycle



Lime -Stone Low Grade

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	P_2O_5	Mn_2O_3	LSF	SM	AM
	16.0	2.6	1.3	43.0	1.1	0.1	0.0	0.9	0.1	0.2	0.2	88.3	4.1	1.9
	15.2	3.5	2.3	41.1	1.6	0.3	0.1	1.2	0.4	0.2	0.2	85.3	2.6	1.5
	15.7	3.2	2.0	41.5	1.5	0.1	0.0	1.2	0.2	0.2	0.2	84.6	3.1	1.6
	17.9	3.4	1.7	41.3	1.3	0.2	0.0	1.2	0.2	0.2	0.2	75.0	3.5	2.0
	17.4	3.1	1.7	41.3	1.3	0.1	0.1	1.1	0.2	0.2	0.1	77.1	3.6	1.9
	16.2	3.3	1.6	41.6	1.5	0.1	0.1	1.2	0.1	0.2	0.2	82.6	3.3	2.1
	17.4	3.0	1.6	41.6	1.6	0.1	0.1	0.9	0.1	0.2	0.2	78.1	3.8	1.9
	16.2	3.5	1.8	41.9	1.8	0.2	0.1	1.2	0.2	0.1	0.2	82.6	3.1	2.0
	14.7	3.5	2.3	41.0	1.8	0.2	0.1	0.9	0.4	0.2	0.1	88.0	2.5	1.5
	15.6	2.7	1.5	42.1	2.1	0.1	0.0	1.0	0.1	0.1	0.2	88.1	3.7	1.9
	16.0	3.3	1.4	41.7	1.6	0.0	0.0	1.0	0.1	0.1	0.2	84.4	3.5	2.3
AVG	16.2	3.2	1.7	41.6	1.6	0.1	0.0	1.1	0.2	0.2	0.2	83.1	3.4	1.9
MIN	14.7	2.6	1.3	41.0	1.1	0.0	0.0	0.9	0.1	0.1	0.1	75.0	2.5	1.5
MAX	17.9	3.5	2.3	43.0	2.1	0.3	0.1	1.2	0.4	0.2	0.2	88.3	4.1	2.3
STD	1.0	0.3	0.3	0.6	0.3	0.1	0.0	0.1	0.1	0.0	0.0	4.6	0.5	0.2

Lime-Stone High Grade

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	Mn_2O_3	LSF	SM	AM	CC	$MgCO_3$	TC
3.34	0.61	0.26	51.5	0.81	0.1	0.07	0.09	0.14	0.03	503.49	3.83	2.32	92	1.7	94
2.49	0.89	0.28	51.26	1.07	0.02	0.07	0.12	0.07	0.03	622.99	2.13	3.15	91.5	2.2	94.2
5.66	1.27	0.4	50.89	1.15	0.04	-0.08	0.29	0.17	0.03	288.74	3.38	3.16	90.9	2.4	93.8
1.91	0.48	0.17	52.93	1.43	0.07	-0.06	0.08	0.11	0.02	876.64	2.94	2.82	94.5	3	98.1

TC = CC + MC
CC = CaO * 1.78
MC = MgO * 2.09

$\Updownarrow$

CaO = CC / 1.786
MgO = MC / 2.0 9

TC – Total Carbonate

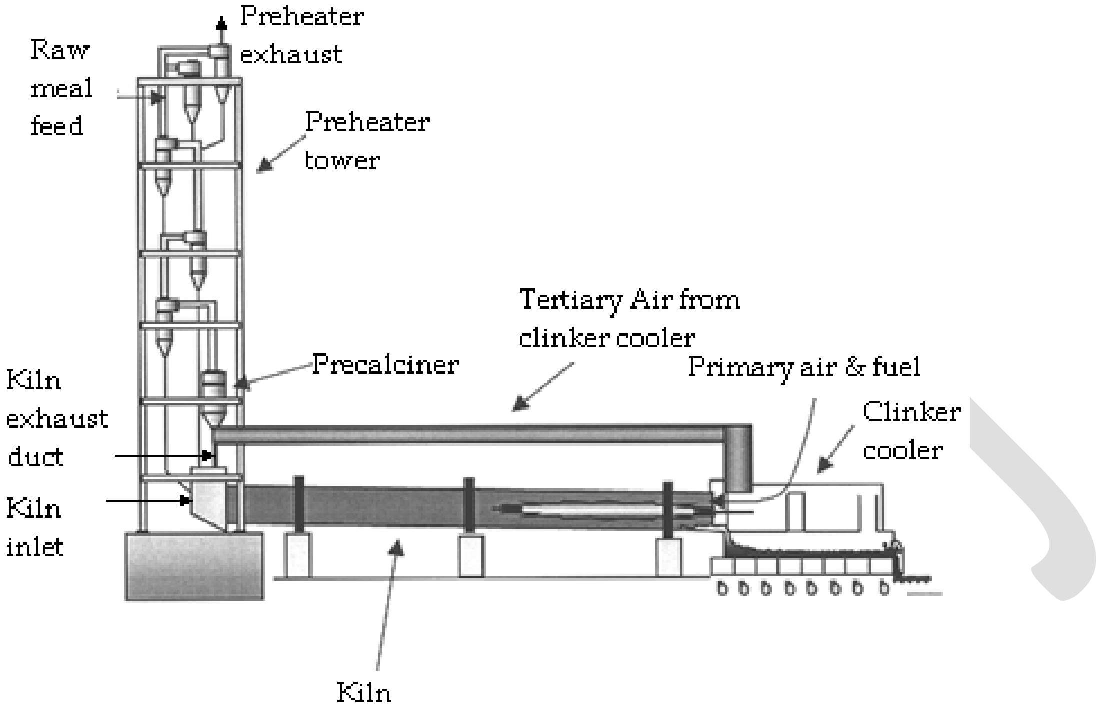
MC - Magnesium Carbonate

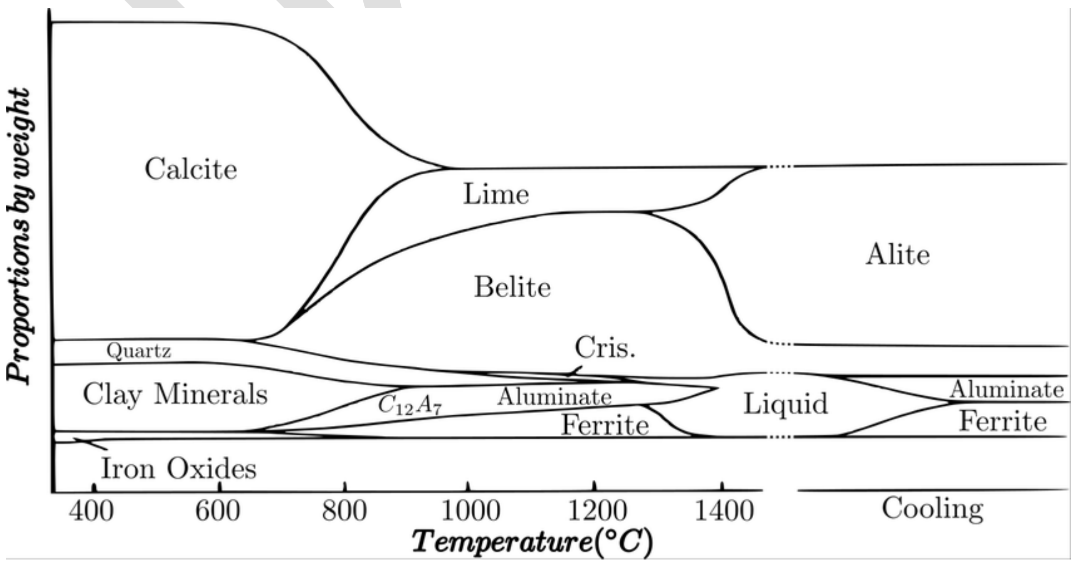
CC – Calcium Carbonate

Cement Manufacturing Technologies:

 Wet Process
 Dry Suspension (SP) Process
 Dry Pre calciner (PC) Process (Present time use)

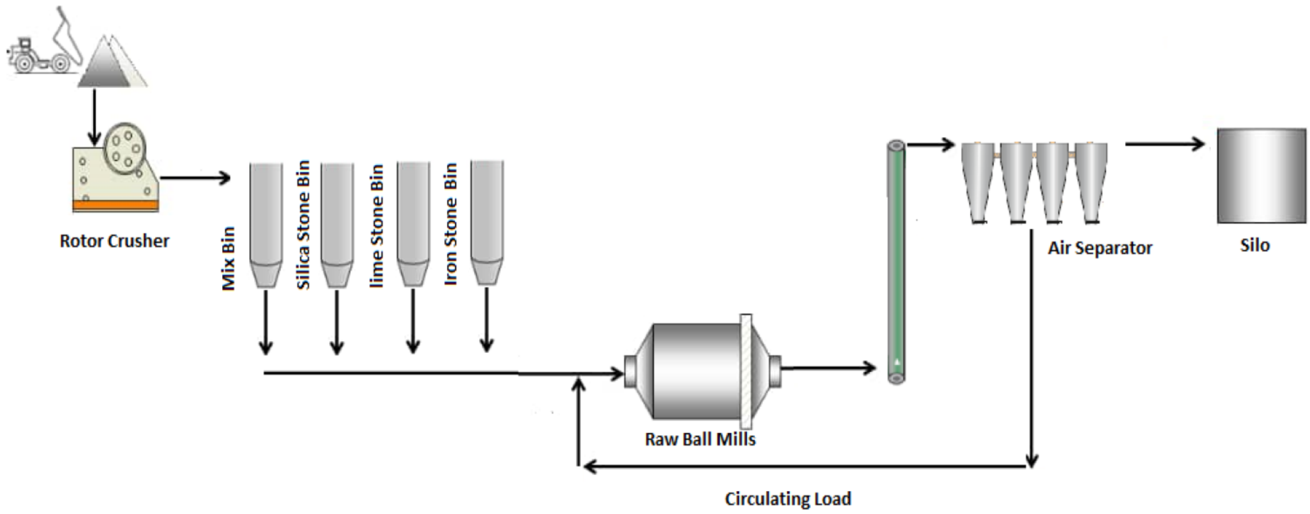
Kiln preheater process





Note ⇨ Kiln Inlet Tem –
⇨ Kiln Out-let Tem

Raw- Mix(Mill)



<u>Use Raw-Material</u>

Limestone ($CaCo_3+SiO_2$)

Clay, Sand Stone – SiO_2

Flux , Laterite ($Fe_2O_3+Al_2O_3$) LOI—(12-15) %

Silica Ratio :(SM) - SM = $SiO_2 / Al_2O_3 + Fe_2O_3$

Typical Range: 1.8 to 2.7

Effects: - SM High -1. Result in hard burning,
2. High fuel consumption,
3. Difficulty in coating formation,
4. Radiation from shell is high,
5. Deteriorates the kiln lining
6. C3S-Low and C2S- High

Alumina modulus :-(AM) AM = Al_2O_3/Fe_2O_3

Typical Range: 1.0 to 1.7

Effects: - AM High – 1.Impacts harder burning,
2 high fuel consumption,
3.Increases C3A decreases C4AF,
4 reduces liquid phase & kiln output,
Silica, clinker sticking and balling is too high.
5. C3S-Low, C2S- High, C3A high and
Liquid High

Lime saturation Factor :(Lime stone):- LSF = CaO X 100 2.8 / SiO2 + 1.2Al2O3 +0.65Fe2O3

Effects: - LSF High –1 Make it difficult to burn raw mix,
2 increases C3S,
3 reduces C2S,
4 deteriorates refractory lining,
5 increases radiation from shell,
6 increases kiln exit gas Tem.

Raw Mill Result

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Cl	LOI	LSF	SM	AM
13-15%	3-4 %	1.9-2.7%	40-42%	1.5-3%	0 .1-.4%	0.1-.5%	0.5-2%	.05	33.-35	90-95	2.3-2.8	1.1-2%

Effects:-

MgO : - 1. A higher MgO- Favours dissociation of C2S and CaO and lets C3S form quickly,
2. Tends the balling easy in the burning zone and affects kiln operation.

Alkalies(K_2O+Na_2O):- A high alkali-Improves burn ability at lower temperature & deteriorates at higher, increase liquid content and coating formation, lowers the Solubility of CaO in melt, breaks down alite & belite phases, creates Operational problem due to external & internal cycle

Sulphur compound(S):- A higher Sulphur compound Acts as an effective mineraliser and modifier of alkali cycle by forming less volatiles,

Chlorides (Cl):- Higher Cl forms more volatiles % causes operational problem due to its complete volatilization in burning zone, increases liquid formation & melting point of the absorbed phase is drastically change.

CLINKER

CLINKER :-

$CaCO_3 \rightarrow CaO + Co_2 \uparrow \rightarrow$ (900ºC)

$MgCO_3 \rightarrow MgO + Co_2 \uparrow$

$3CaO + Al_2O_3 \rightarrow 3CaO.Al_2O_3$ (C3A)

$4CaO + Al_2O_3 + Fe_2O_3 \rightarrow 4Cao.Al_2O_3.Fe_2O_3$ (C4AF)

$2CaO + SiO_2 \rightarrow 2CaO.SiO_2$ (C2S) $\rightarrow$ (≤1200ºC)

$2Cao.SiO_2 + CaO \rightarrow 3CaO.SiO_2$ (C3S) $\rightarrow$ (1200ºC-1400 ºC)

Bogue Compound :-

(1) C_3S
(2) C_2S
(3) C_3A
(4) C_4AF

(1) C3S:-

→ Chemical Crystal Name – Alite
→ Chemical Name - Tricalcium Silicate
→ Molecular weight - 228 g/g mol
→ Formula -

$$C_3S = 4.071\,CaO - (7.602\,SiO_2 + 6.718\,Al_2O_3 + 1.43\,Fe_2O_3)$$ Note: CaO = CaO - F/CaO

→ It is responsible for - Early Strength of cement
→ First 7 Days Strength is due to C3S
→ It produces more Heat of hydration (500Joule/gm)
→ Higher C3S –
- Higher Early Strength
- Higher heat generation
- It is responsible for initial Strength of cement
- Hence if in any engineering Construction early strength is Required proportion of C3S is increased
  Ex. Road, Cold Weather

(2) C2S :-

→ Chemical Crystal Name – Belite
→ Chemical Name - Dicalcium Silicate
→ Molecular weight - 172 g/g mol
→ Formula

$$C_2S = 2.867\,SiO_2 - 0.7544\,C_3S$$

→ It is responsible for Late Strength of cement
→ C2S Hydrates and Hardens slowly
→ C2S hydrates after 7 days. Hence it gives Strength after 7 days
→ If produces less heat hydration (260Joule/gm)
→ Higher C2S –
- lower Early Strength
- Lower Heat generation.

(3) C3A

→ Chemical Crystal Name –Aluminate
→ Chemical Name - Tricalcium Aluminate
→ Molecular weight - 270 g/g mol
→ Formula

$$\boxed{C3A = 2.65\ Al_2O_3 - 1.692\ Fe_2O_3}$$

→ Contributes to early strength,(1Day)
→ Help faster setting,
→ Liberates more heat in concrete
→ The reaction of C3A with Water is very Fast
→ Hydration –Fast

(4) C4A.F.

→ Chemical Crystal Name – Ferrite
→ Molecular weight - 486 g/g mol
→ Chemical Name – Tetracalcium Aluminoferrite
→ Formula

$$\boxed{C4AF = 3.043\ Fe_2O_3}$$

→ It Hydrates very rapidly
→ Also responsible for grey colour of OPC Cement

Note

Percentage by Mass: - **C3S > C2S > C3A > C4AF**

Formation in Kiln: - **C4AF > C3A > C2S > C3S**

3 Days Strength: - **C3S > C3A > C2S > C4AF**

28 Days Strength:- **C3S > C2S > C3A > C4AF**

Heat of Hydration:- **C3A > C3S > C4AF > C2S**

Reaction With Water **C3A > C4AF > C3S > C2S**

Reaction Completed **C4AF > C3A > C2S > C3S**

X-Ray :-

XRF full form--X-ray Fluorescence spectroscopy

IS Code = 12803:1989 Methods of analysis of hydraulic cement by X-ray fluorescence Spectrometer

IS Code = 3535: 1986 Title Methods of sampling hydraulic cement

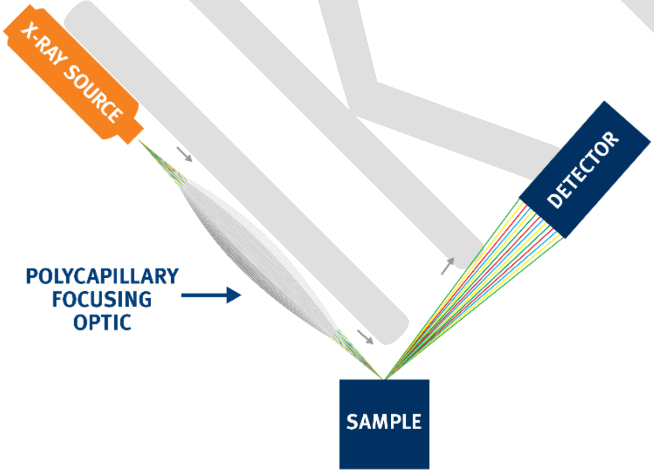
X-Ray Fluorescence (XRF)

What is XRF

X-ray fluorescence (XRF) is a powerful quantitative and qualitative analytical tool for elemental analysis of materials. It is ideally suited to the measurement of film thickness and composition, determination of elemental concentration by weight of solids and solutions, and identification of specific and trace elements in complex sample matrices. XRF analysis is used extensively in many industries including semiconductors, telecommunications, microelectronics, metal finishing and refining, food, pharmaceuticals, cosmetics, agriculture, plastics, rubbers, textiles, fuels, chemicals, and environmental analysis. The method is fast, accurate, non-destructive, and usually requires only minimal sample preparation.

How XRF works

When elements in a sample are exposed to a source of high intensity X-rays, fluorescent X-rays will be emitted from the sample at energy levels unique to those elements.



The basic concept for all XRF spectrometers is a source, a sample, and a detection system. The source irradiates the sample and a detector measures the fluorescence radiation emitted from the sample. In most cases for XRF, the source is an X-ray tube. Alternatives are a radioactive source or a synchrotron.

There are two main types of XRF instruments:

(1) Energy Dispersive X-ray fluorescence (EDXRF)and

(2) Wavelength Dispersive X-ray Fluorescence (WDXRF).

X-ray optics can be used to enhance both types of XRF instrumentation. For conventional XRF instrumentation, typical focal spot sizes at the sample surface range in diameter from several hundred micrometers up to several millimeters. Polycapillary focusing optics collect X-rays from the divergent X-ray source and direct them to a small focused beam at the sample surface with diameters as small as tens of micrometers. The resulting increased intensity, delivered to the sample in a small focal spot, allows for enhanced spatial resolution for small feature analysis and enhanced performance for measurement of trace elements for Micro X-ray Fluorescence applications. Doubly curved crystal optics direct an intense micron-sized monochromatic X-ray beam to the sample surface for enhanced elemental analysis.

X-Ray

↓ Medical → ↓ Radiographic

↓ Analysis → ↓ **XRF** (↓ **WD-XRF**, ↓ **ED-XRF**), ↓ **XRD**

Note:-

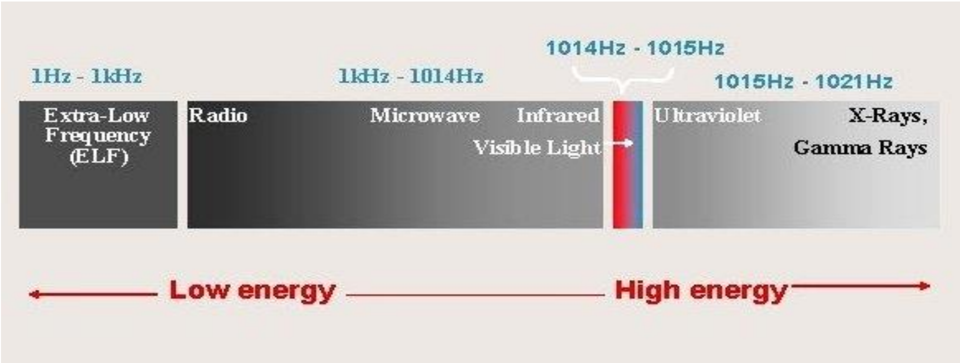
→ **WD-XRF** **-** Wavelength Dispersive X-Ray Fluorescence (WDXRF)
→ **ED-XRF** - Energy Dispersive X-ray Fluorescence (EDXRF)
→ XRD - **X**-Ray Diffraction

XRF mainly follows the Bragg principle**.**

$$n \lambda = 2d \sin\Theta$$

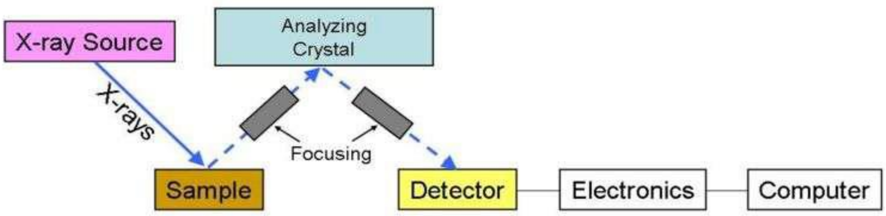
- where n (an integer) is the "order" of reflection, λ is the wavelength of the incident X-rays, d is the interplanar spacing of the crystal and Θ is the angle of incidence.
- Wavelengths of X-ray 0.1 to 10nm
- Energy in range from 0.125 to 125KeV

<u>Electromagnetic Radiation</u>:-

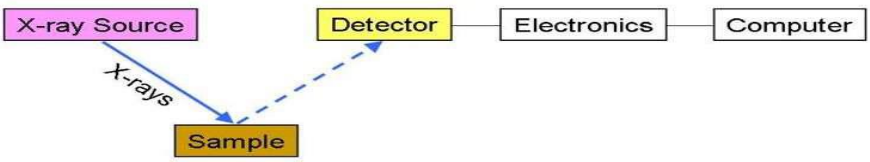


XRF Spectrometers

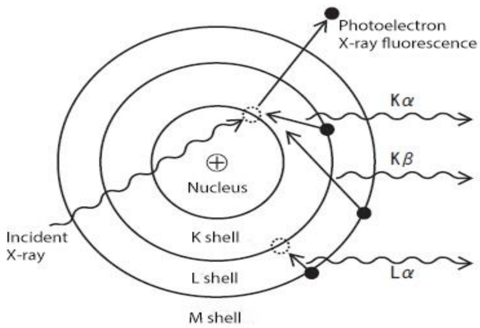
(1) - Wavelength Dispersive WDXRF Spectrometer :-



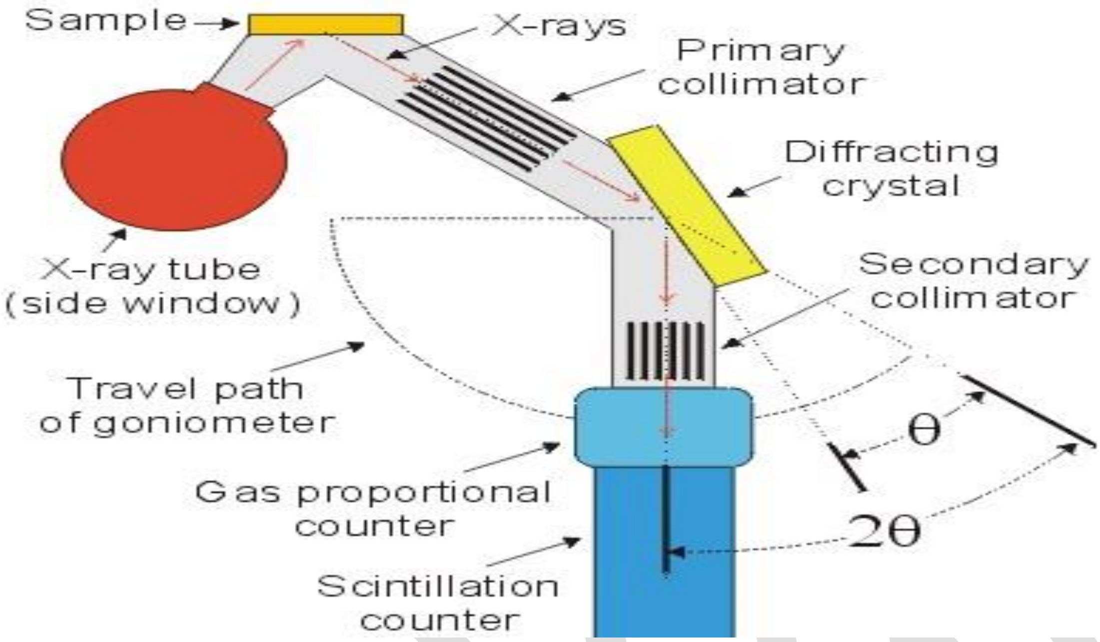
(2) - Energy Dispersive EDXRF Spectrometer :-



<u>XRF sample preparation</u>:-



<u>XRF- Principal</u>:-



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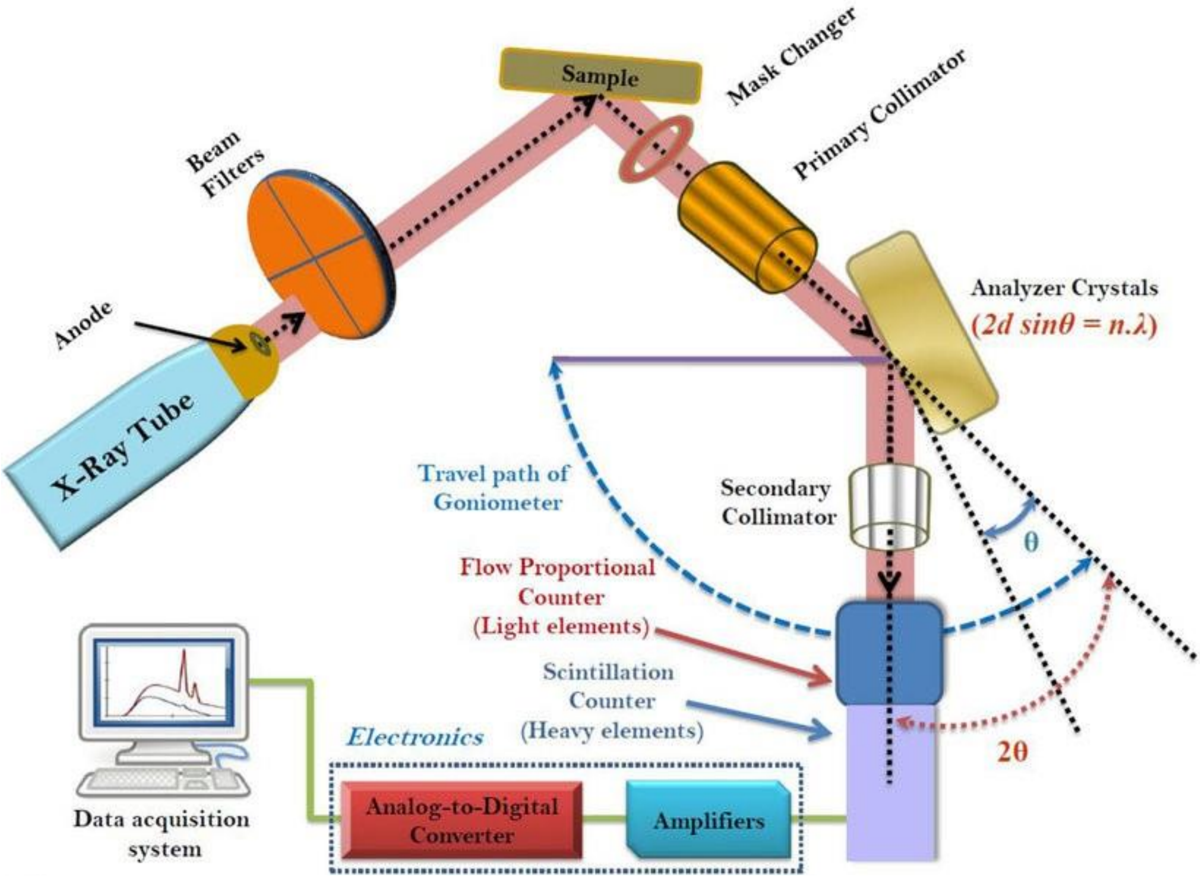
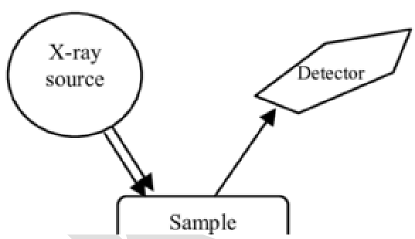
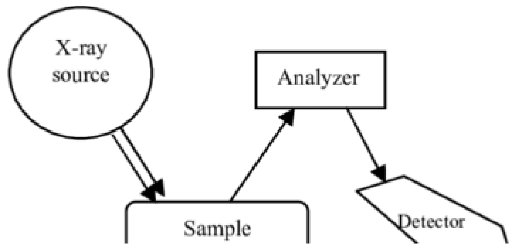
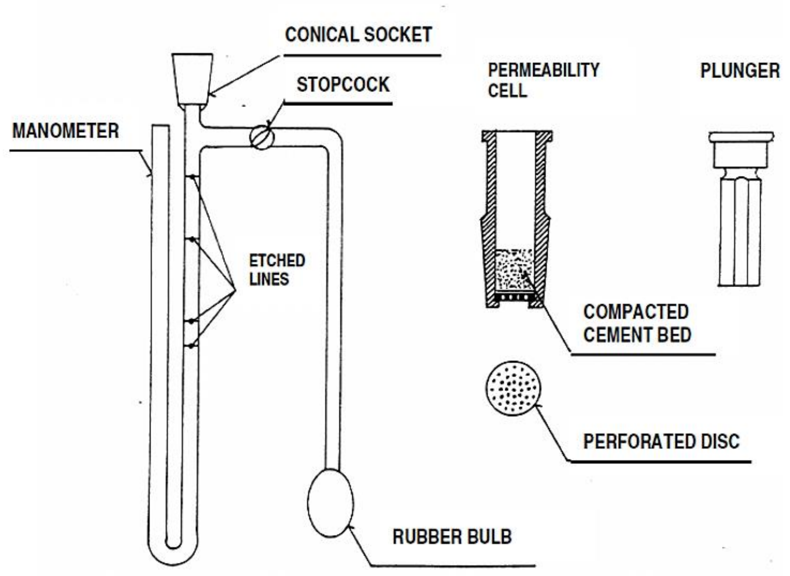


ED-XRF And WD-XRF Diff.

Equipment	**ED-XRF**	**WD-XRF**
X-rays source	K- Lines of elements	K- Lines of elements
Signal to noise ratio	Very good and dependent on crystal	Limited and dependent on detector and electronics
	Analysis of elements from Beryllium (Be) to Uranium(U)	Analysis of elements from Beryllium (Be) to Uranium(U)

Equipment	EDXRF	WDXRF
X-rays source	K lines of elements or X-rays or Gamma-ray sources	K lines of elements
Collimator	Not required	Collimators which are necessary to guarantee good spectral resolution are thin tubes or lamella (soller slits).
Analyzer Crystal	Not required	For spectral separation of the fluorescence radiation Bragg diffraction at monocrystals or multilayer are used.
Detector	Si(Li) or HPGe detector	Proportional Counter and Scintillation counters
Geometry schematic diagram	EDXRF 	WDXRF 

Blaine(fineness):-



→ Fineness Unit - **cm^2/g or m^2/kg**

→ IS-4031- PART-2

→ Liquid Use - Di-n-butyl phthalate ($C_{16}H_{22}O_4$)

→ Di-n-butyl phthalate properties

- non-volatile
- non-hygroscopic
- low viscosity
- low density

Increasing fineness causes an:-

- High strength, and
- High heat generation

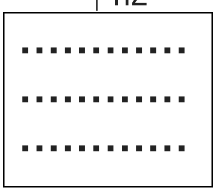
→ Blaine = **$\sqrt{\text{Time}}$ * Factor**

Factor = STD Blaine / $\sqrt{\text{Time}}$

Type Of Cement	Surfacr Area (Cm2/gm
1. OPC	2250
2. PPC	3000
3. PSC	2250
4. W.C	2250

***WC = White Cement**

→ ↑ h2



↑ h1

→ Hl = h1 – h2 α Specific, Area surface

- ➢ Air Permeability Apparatus is conforming of IS :- 5516 :1996
- ➢ Permeability cell is conforming of IS :- 6911 :1972
- ➢ Plunger is conforming of IS :- 6911 :1972
- ➢ Perforated metal disc is conforming of IS : 6911 :1972

Cement

Cement Type

1. OPC
2. PPC
3. PSC
4. White Cement

TYPE OF CEMENT	LOI	MgO	I R	SO_3	Fineness (M^2/Kg)	Soundness Lechate - Auto Clave	Setting Time IST- FST	Compressive Strength Days 3 Days	7 Days	28 Days
OPC-33	5% MAX	6% MAX	5% MAX	3.5% MAX	>225	10-0.8	30-600	16	22	33
OPC-43	5% MAX	6% MAX	5% MAX	3.5% MAX	>225	10-0.8	30-600	23	33	43
OPC-53	4% MAX	6% MAX	5% MAX	3.5% MAX	>225	10-0.8	30-600	27	37	53
PPC	5% MAX	6% MAX	ULA	3.5% MAX	>300	10-0.8	30-600	16	22	33
PSC	5% MAX	10% MAX	4% MAX	3.5% MAX	>225	10-0.8	30-600	16	22	33
.										

Note

- OPC-33 Max Strength – 48
- OPC-43 Max Strength – 58
- Compressive Strength Unit—Mpa (Megapascal)
- All OPC 33,43,53 - IS Code - 269 : 2015
- PPC IS Code - 1489 (P-1): 1991 (Fly ash Based) , 1489 (P-1): 1991 (Calcined Clay Based)
- PSC IS Code – 455 : 1989

Chemical Reaction

Cement water reaction:-

1.0 Portland Pozzolana cement (PPC)

Cement (OPC) + Water(H_2O) $\Longrightarrow$ C-S-H gel + $Ca(OH)_2$
(Primary Gel)

$Ca(OH)_2$ + SiO_2 (Fly Ash) $\Longrightarrow$ C-S-H gel + $Ca(OH)_2$
(secondary Gel)

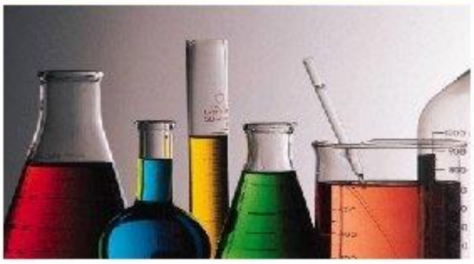
2.0 Portland Slag Cement (PSC)

Cement (PSC) + Water(H_2O) $\Longrightarrow$ C-S-H gel + $Ca(OH)_2$
(Primary Gel)

$Ca(OH)_2$ + SiO_2 (Silicates) $\Longrightarrow$ C-S-H gel + $Ca(OH)_2$
(secondary Gel)

Clinker

Burning Process : Chemical Reactions in Rotary Klin Zones



- Calcination Zone :

$$CaCO_3 \longrightarrow CaO + CO_2 \uparrow$$

- Clinkering Zone :

$$2CaO + SiO_2 \longrightarrow Ca_2SiO_4 \text{ (Dicalcium silicate – } C_2S \text{)}$$

$$3CaO + SiO_2 \longrightarrow Ca_3SiO_5 \text{ (Tricalcium silicate – } C_3S \text{)}$$

$$3CaO + Al_2O_3 \longrightarrow Ca_3Al_2O_6 \text{ (Tricalcium aluminate – } C_3A \text{)}$$

$$4CaO + Al_2O_3 + Fe_2O_3 \longrightarrow Ca_4Al_2Fe_2O_{10} \text{ (Tricalcium aluminoferrite – } C_4AF\text{)}$$

<u>Bogus' formula for Clinker Constituent (if Alumina modulus >0.64)</u>

- C3S = 4.071 CaO – (7.602 SiO2 + 6.718 Al2O3 +1.43Fe2O3) Note: CaO = CaO - F/CaO
- C2S = 2.867 SiO2 - 0.7544 C3S
- C3A = 2.65 Al2O3 - 1.692 Fe2O3
- C4AF = 3.043 Fe2O3
- LV= 1.13 C3A +1.35 C4AF + MgO +Alkalise

<u>Chemical Req</u> :-

- CLK IS Code –16353 : 2015
- C3S Percentage by mass Min – 35.0
- C3A Percentage by mass – 3 -12
- C3S+C2S % By mass Min – 70
- SO3 Max - 3.0
- Cl^- Max - 0.1
- LOI Max - 1.0
- MgO Max - 6.0
- IR – 1.0 (Max)

COAL:-

Types of Coal

- Sub bituminous
- Lignite
- Bituminous Coal
- Anthracite Coal

Coal Analysis:

NCV = 8455 – 114 (M% + Ash %) Cal/gm

UHV = 8900 – 138 (M % + Ash %) Cal/gm

GCV = PC **X** 86.5 – (60*M %)

PC = 100- (1.1*Ash + M %)

$$CV = \frac{\%C.X\,8000}{100} + \frac{\%H\,X\,32000}{100}$$

Coal Consumption = Coal feed **X** 100 /Clinker Production

Coal

IS 1350:1984 (Part-I) Methods of test Proximate analysis

IS 1350:1970 (Part-II) Methods of test Calorific value

IS 1350:1969 (Part-III) Methods of test Sulphur analysis

IS 1350:1974 (Part-IV) Methods of test Ultimate analysis.

IS 1350:1979 (Part-V) Methods of test Special Impurity.

Important Formula Use in Cement Analysis.

Hydraulic Modulus: $HM = \frac{CaO}{SiO_2 + Al_2O_3 + Fe_2O_3}$

Silica Ratio: $SM = \frac{SiO_2}{Al_2O_3 + Fe_2O_3}$

Alumina Ratio $AM = \frac{Al_2O_3}{Fe_2O_3}$

Liquid Value: $LV = 1.13\ C_3A + 1.35\ C_4AF + MgO + \text{Alkalies}$

Burnability Index: $BI = \frac{C_3S}{C_4AF + C_3A}$

Burnability Factor: $BF = LSF + 10\ SM - 3(MgO + \text{Alkalies})$

Coal Analysis:

$NCV = 8455 - 114\ (M\% + Ash\ \%)\ Cal/gm$

$UHV = 8900 - 138\ (M\ \% + Ash\ \%)\ Cal/gm$

$GCV = PC\ X\ 86.5 - (60 * M\ \%)$

$PC = 100 - (1.1 * Ash + M\ \%)$

$CV = \frac{\%C * 8000}{100} + \frac{\%H * 32000}{100}$

Coal Consumption: $= \frac{\text{Coal feed} * 100}{\text{Clinker Production}}$

Ash absorption $= \frac{\%\ \text{of ash in fuel}\ \mathbf{X}\ \text{coal consumption}}{100}$

Raw meal to clinker factor $= \frac{100 - \text{ash absorption}}{100 - LOI}$

Specific Heat $= \frac{NCV\ \mathbf{X}\ \%\ \text{of coal Consumption}}{100}$

Insoluble Residue $IR\ (\max\ \%) = \frac{X + 4\ (100 - X)}{100}$ (Note: X= % of Fly ash)

CYCLONE LOSS: $= \frac{100\ (\text{KF loss} - \text{Cyclone loss})}{(100 - \text{Cyclone loss})\ X\ \text{KF loss}} * 100$

Clinker to cement factor $= \frac{\text{Clink.} + \text{Fly ash/Slag} + \text{additives (kg)}}{\text{Clinker consumed (kg)}}$

References

▪IS book-4031,4032,1350,16353,1760,269,1489
▪Cement data book -Duda_III edition
▪Copyright XOS ®
▪Reno, M.L.G.,Silva, R.J., Melo. M.L.N.M., Boas, S.B.V. (2016) “An overview of industrial wastes as fuel and mineralizer in the cement industry”,Latin American Applied Research, 46(2), pp 43-50.
▪Significance of Curing of Concrete for Durability of Structures S.B. Kulkarni AVP, Technical Services and Clinton Pereira Dy. Manager Technical-UltraTech Cement Ltd, Mumbai
▪Bogues compounds are responsible for initial setting of the cement grout. These compounds have been researched extensively by an American chemist named Rober Herman Bogue (R.H. Bogue) who is well known for his books on The Chemistry of Portland Cement and the Chemistry and Technology of Gelatin and Glue.
▪ https://sites.google.com/site/strengtheningofmasonrywalls/

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