Cement tests

1. Fineness test

The fineness of cement has an important bearing on the rate of hydration and hence on the rate of gain of strength and also on the rate of evolution of heat. Finer cement offers a greater surface area for hydration and hence faster the development of strength, fig. below. Different cements are ground to different fineness. The disadvantages of fine grinding is that it is susceptible to air and early hydration. Increase in fineness of cement is also found to increase the drying shrinkage of concrete.

Fineness is necessary to cover surfaces of the fine aggregate particles at better manner – leading to better adhesion and cohesion between cement mortar constituents.

An increase in the fineness of cement, both consistency of cement and workability of concrete increase. Increase in consistency means increase in water demand to get cement paste of standard consistency while increase in workability means increase in flow ability at the same water/cement ratio. In other words decrease in water demand to get the same workable concrete.
The disadvantage of high fineness, include:
- The cost of grinding to a higher fineness is considerable.
- The finer the cement the more rapidly it deteriorates on exposure to the atmosphere during bad storage.
- Finer cement increases the surface area of its alkalis – leads to stronger reaction with alkali-reactive aggregate – cracks and deterioration of concrete.
- Finer cement exhibits a higher shrinkage and a greater proneness to cracking.
- An increase in fineness increases the amount of gypsum required for proper retardation because, in finer cement, more C₃A is available for early hydration (due to the increase of its surface area).

Fineness of cement is tested in two ways:

(a) By sieving.
(b) By determination of specific surface (total surface area of all the particles in one gram of cement) by air-permeability apparatus. Expressed as cm²/gm or m²/kg. Generally Blaine Air permeability apparatus is used.

a) By sieving:

Weigh correctly 100 grams of cement and take it on a standard IS Sieve No. 9 (90 microns). Break down the air-set lumps in the sample with fingers. Continuously sieve the sample giving circular and vertical motion for a period of 15 minutes. Mechanical sieving devices may also be used. Weigh the residue left on the sieve. This weight shall not exceed 10% for ordinary cement. Sieve test is rarely used due to clogging sieves opens with cements particles.

b) By Blaine test

Principle of this method is in observing the time taken for a fixed quantity of air to flow through compacted cement bed of specified dimension and porosity.

Volume of cement bed in the cell can be found by it with mercury before and after putting cement on it then determine the difference between two weights, this difference divided by mercury density in room temperature to obtain volume of cement bed. Repeat this for twice at least and use the average value of volume in following equations, also the weight of cement used in cell must be with porosity equal to 0.5 ± 0.0005.

\[ W = 3.15 \times V \times (1 - e) \]

Which: 3.15 = specific gravity of cement
W = weight of cement
V = volume of cement bed
e = porosity of cement bed
Place the perforated disc on the ledge at the bottom of the cell and place on it a new filter paper disc. Place the sample of standard cement weighted (W) with knowing surface area, in the cell. Place a second new filter paper disc on the levelled cement. Insert the plunger and press it gently but firmly until the lower face of the cap is in contact with the cell.

Then air pumped inside the tube raise the level of the manometer liquid to that of the highest etched line. Close the stopcock and the manometer liquid will begins to flow. Start the timer as the liquid reaches the second etched line and stop it when the liquid reaches the third etched line. Record the time, t . The procedure repeats three times for standard sample and tested one. The surface area then can be calculated by:

\[ S = \frac{S_s \sqrt{T}}{\sqrt{T_s}} \]

Which:
S = specific area of tested cement sample \( ( \text{cm}^2/\text{g}) \)
\( S_s \) = specific area of standard cement \( ( \text{cm}^2/\text{g}) \)
T = average time for tested sample
\( T_s \) = average time for standard sample.
2. Standard Consistency Test

For finding out initial setting time, final setting time and soundness of cement, and strength a parameter known as standard consistency has to be used. The standard consistency of a cement paste is defined as that consistency which will permit a Vicat plunger having 10 mm diameter and 50 mm length to penetrate to a depth of 33-35 mm from the top of the mould. The apparatus is called Vicat Apparatus.

The following procedures are adopted to find out standard consistency. Take about 500 gm of cement and prepare a paste with a weighed quantity of water (say 24 per cent by weight of cement) for the first trial. The paste must be prepared in a standard manner and filled into the Vicat mould within 3-5 minutes. After completely filling the mould, shake the mould to expel air. A standard plunger is attached and brought down to touch the surface of the paste in the test block and quickly released allowing it to sink into the paste by its own weight. Take the of penetration of the plunger. Conduct a 2nd trial (say with 25 per cent of water) and find out the depth of penetration of plunger. Similarly, conduct trials with higher and higher water/cement ratios till such time the plunger penetrates for a depth of 33-35 mm from the top. That particular percentage of water which allows the plunger to penetrate only to a depth of 33-35 mm from the top is known as the percentage of water required to produce a cement paste of standard consistency.

3. Setting Time Test

Prepare a cement paste by mixing (400 g) of cement with water required to give a paste of standard consistency. Start a stop-watch, the moment water is added to the cement. Then fill the Vicat mould completely with the cement paste, the mould resting on a non-porous plate and smooth off the surface of the paste making it level with the top of the mould.

a) Initial setting time: Place the test block under the rod bearing the needle. Lower the needle gently in order to make contact with the surface of the cement paste and release quickly, allowing it to penetrate the test block. Repeat the procedure till the needle fails to penetrate the test block to a point 5.0 ± 0.5mm measured from the bottom of the mould. The time period elapsing between the time, water is added to the cement and the time, the needle fails to penetrate the test block by 5.0 ± 0.5mm measured from the bottom of the mould, is the initial setting time.

b) Final setting time: Replace the above needle by the one with an annular attachment. The cement should be considered as finally set when, upon applying the needle gently to the surface of the test block, the needle makes an impression therein, while the attachment fails to do so. The period elapsing between the time, water is added to the cement and the time, the needle makes an impression on the surface of the test block, while the attachment fails to do so, is the final setting time.
4. Soundness of cement

Soundness of cement is determined by Le-Chatelier method, it consists of a small split cylinder of spring brass or other suitable metal. It is 30 mm in diameter and 30 mm high. On either side of the split are attached two indicator arms 165 mm long with pointed ends. Mixing (200 gm) of cement with suitable amount of water to make standard consistency cement paste. Filled into the mould kept on a glass plate. The mould is covered on the top with another glass plate, place a small weight on this covering glass sheet and immediately submerge the whole assembly in water at a temperature of 19 ± 1 C° and keep it there for 24hrs. Measure the distance separating the indicator points to the nearest 0.5mm (say d1 ). then Submerge the mould again in water at the temperature prescribed above. Bring the water to boiling point in 25 to 30 minutes and keep it boiling for one hour. Remove the mould from the water, allow it to cool and measure the distance between the indicator points (say d2 ). (d2 – d1 ) represents the expansion of cement it should be not more than 10 mm.
5. Compression strength test
The compressive strength of hardened cement is the most important of all the properties. Therefore, it is not surprising that the cement is always tested for its strength at the laboratory before the cement is used in important works. Strength tests are not made on neat cement paste because of difficulties of excessive shrinkage and subsequent cracking of neat cement. Strength of cement is indirectly found on cement sand mortar in specific proportions. The standard sand is used for finding the strength of cement. Take 600 gms of standard sand, 200 gms of cement (i.e., ratio of cement to sand is 1:3) mix them with a trowel for one minute, then add 80 gm of water of mix the three ingredients thoroughly until the mixture is of uniform color. The time of mixing should not be less than 3 minutes nor more than 4 minutes. Immediately after mixing, the mortar is filled into a cube mould of size 7.07 cm. compact the mortar either by hand compaction in a standard specified manner or on the vibrating equipment (12000 RPM) for 2 minutes. Keep the compacted cube in the mould at a temperature of 19°C ± 1°C and at least 90 per cent relative humidity for 24 hours. Where the facility of standard temperature and humidity room is not available, the cube may be kept under wet gunny bag to simulate 90 per cent relative humidity.
After 24 hours the cubes are removed from the mould and immersed in clean fresh water until taken out for testing. Three cubes are tested for compressive strength at the periods mentioned. The compressive strength shall be the average of the strengths of the three cubes for each period.

6. Tensile strength test
The proportions of the standard mortar shall be 1 part cement to 3 parts standard sand by Weight. The percentage of water 8% of total weight of cement and sand. Before being filled, thinly cover the molds with a film of mineral oil. Molds are like (8) shape with certain dimensions with metal or glass base. Mix dry cement and sand with a trowel for one minute, then add water and continue mixing for 4 minutes. Filled molds with mortar and shuffle their surface, Place them in moist room for 24 hr. after that remove the molds and immerse specimens to time of test.
Hydration of Cement

It is the reaction (series of chemical reactions) of cement with water to form the binding material. In other words, in the presence of water, the silicates (C\textsubscript{3}S and C\textsubscript{2}S) and aluminates (C\textsubscript{3}A and C\textsubscript{4}AF) form products of hydration which in time produce a firm and hard mass.

There are two ways in which compounds of the type present in cement can react with water. In the first, a direct addition of some molecules of water takes place, this being a true reaction of hydration. The second type of reaction with water is hydrolysis, in which its nature can be illustrated using the C\textsubscript{3}S hydration equation

\[ 3\text{CaO.SiO}_2 + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 + \text{xCaO.ySiO}_2\text{.aq.} \text{ (Calcium silicate hydrate)} \]

\[ 2 \text{C}_3\text{S} + 6\text{H} \rightarrow 3 \text{Ca(OH)}_2 + \text{C}_3\text{S}_2\text{H}_3 \]

The reaction of C\textsubscript{3}S with water continue even when the solution is saturated with lime and the resulted amounts of lime precipitate in crystals form Ca(OH)\textsubscript{2}.

**Calcium silicate hydrate** → remains stable when it is in contact with the solution saturated with lime, and it hydrolyzed when being in water – some of lime form, and the process continues until the water saturate with lime.

The rates of the chemical reactions of the main compounds are different:

**Aluminates** - React with the water in the beginning

- Affect the route of the chemical reactions at early periods of hydration.

**Silicates** – Affect the later stage reactions.

**The main hydrates of the hydration process are:**
- Calcium silicates hydrate
- Tricalcium aluminate hydrate
- C\textsubscript{4}AF which hydrates to tricalcium aluminate hydrate and calcium ferrite CaO.Fe\textsubscript{2}O\textsubscript{3} in amorphous form.

**Since calcium silicates (C\textsubscript{3}S and C\textsubscript{2}S)** – are the main cement compounds (occupies about 75% of cement weight) – they are responsible for the final strength of the hardened cement paste.

**With time:**
- The rate of hydration decreases continuously.
- The size of unhydrated cement particles decrease.
This is due to:

1. Accumulation of hydration products around the unhydrated cement grains which lead to prevent water from channeling to them.
2. Reduction of the amount of water either due to chemical reaction or evaporation.
3. Reduction of the amount of cement due to reaction.

**Tricalcium aluminate hydrate and the action of gypsum**

The amount of C₃A present in most cement is comparatively small but its behavior and structural relationship with the other phases in cement make it of interest. The tricalcium aluminate hydrate forms a prismatic dark interstitial material.

The reaction of pure C₃A with water is very violent with evolution of large amount of heat, forming calcium aluminates hydrate in the form of leaf hexagonal crystals. In Portland cement, this reaction leads to immediate stiffening known as "flash setting".

Gypsum, added to the clinker through grinding process cause delaying the reaction of C₃A with water by its reaction with C₃A to form insoluble calcium sulfoaluminate (3CaO.Al₂O₃.3CaSO₃.30-32H₂O) - ettringite - around C₃A particles, which give enough time for the hydration of C₃S that its reaction is slower than C₃A. But eventually tricalcium aluminate hydrate is formed, although this is preceded by a metastable 3CaO.Al₂O₃. CaSO₃.12H₂O, produced at the expense of the original high-sulfate calcium sulfoaluminate.

The reaction of gypsum with C₃A continues until one of them exhausted, while C3S continue in hydration.

- **If C₃A exhausted before gypsum**
  - The surplus gypsum → expand → become an agent assist the disruption and deterioration of cement paste.
- **If gypsum exhausted before C₃A**
  - The remaining C3A begins in hydration:
    \[ C₃A + 6H \rightarrow C₃AH₆ \]
    
    C₃AH₆ is stable –cubical crystals- with high sulfate resistance.

The presence of C₃A in cement is undesirable: it contributes little to the strength of cement except at early ages (1-3 days) and, when hardened cement paste is attacked by sulfates, expansion due to the formation of calcium sulfoaluminate from C₃A may result in a disruption of the hardened paste.

But it is useful in the cement industry – work as flux material – reduce the temperature needed to form the clinker. Also it facilitates the combination of lime with silica.
**C₄AF compound**

Gypsum reacts with C₄AF to form calcium sulfoaluminates and calcium sulfoferrite. C₄AF – work as flux material and also it accelerates the hydration of silicates.

*Using the optimum percentage of gypsum is very important because:*

- It regulates the speed of the chemical reactions in the early ages.
- Prevent the local concentration of the hydration products.

*The necessary gypsum content increase with the increase of:*

- C₃A content in the cement.
- Alkalis content in the cement.
- Fineness of cement.

Iraqi specification No. 5 limits the maximum gypsum content (expressed as the mass of SO₃ present) to be not more than 2.5% when C₃A ≤ 7% and 3% when C₃A >7%.

**Calcium silicates hydrate**

*C₃S*

C₃S + water → - lime and silica ions in the solution.

- Ca(OH)₂ crystals
- Calcium silicate hydrate gel.

This initial gel form an external layer over C₃S causing the delay of the reaction. After few hours, this initial C-S-H undergo hydrolysis to form the second product of the gel CSH I then form the stable C-S-H II form.

Hydration of C₃S takes about one year or more. The full hydration of C₃S can be expressed approximately by the following equation:

\[
2(3\text{CaO.}\text{SiO}_2) + 6\text{H}_2\text{O} \rightarrow 3\text{CaO.}2\text{SiO}_2.3\text{H}_2\text{O} + 3\text{Ca(OH)2}
\]

*C₂S*

There are three main crystal forms of C₂S (α, β, γ) but the β-form is the only one occurred in the Portland cement and it react slowly with water.

- Its reaction is slower than C₃S.
- The amount of Ca(OH)₂ from its hydration is less.
Its formed gel is similar to that produced from $\text{C}_3\text{S}$, but there is difference in the route of the chemical reactions between the two compounds.

Hydration of $\text{C}_2\text{S}$ – takes more than 4 years. The full hydration of $\text{C}_2\text{S}$ can be expressed approximately by the following equation:

$$2(2\text{CaO.SiO}_2) + 4\text{H}_2\text{O} \rightarrow 3\text{CaO.2SiO}_2.3\text{H}_2\text{O} + \text{Ca(OH)}_2$$

The rates of hydration of the two compounds are different as shown in the figure below:

The gel formed after the completion of hydration of the two compounds is $\rightarrow \text{C}_3\text{S}_2\text{H}_3$ – Tobermorite.

$\text{C}_3\text{S}$ and $\text{C}_2\text{S}$ – require approximately the same amount of water for hydration, but $\text{C}_3\text{S}$ produces more than twice as much $\text{Ca(OH)}_2$ as is formed by the hydration of $\text{C}_2\text{S}$, as shown in the equations below:

For $\text{C}_3\text{S}$ hydration
$$2 \text{C}_3\text{S} + 6\text{H} \rightarrow \text{C}_3\text{S}_2\text{H}_3 + 3\text{Ca(OH)}_2$$

For $\text{C}_2\text{S}$ hydration
$$2 \text{C}_2\text{S} + 4\text{H} \rightarrow \text{C}_3\text{S}_2\text{H}_3 + \text{Ca(OH)}_2$$