

Chapter 3

Inorganic Binding Materials



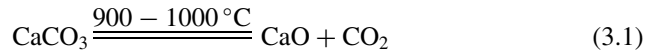
Binding materials can be classified into organic and inorganic materials according to their chemical composition. Organic binding materials in civil engineering mainly include asphalt and resin. Inorganic binding or cementing materials in civil engineering are mainly inorganic powder materials that can be mixed with water or the aqueous solution to form the slurry and gradually get hardened to form the artificial stone with strength after a series of physical and chemical changes. Inorganic binding materials can be classified into water-hardening and air-hardening binding materials according to their hardening conditions. Lime and gypsum are air-hardening materials which only harden in the air and cannot harden in the water; whereas Portland cement and aluminate cement, etc. are water-hardening materials that can harden in both air and water.

3.1 Lime

3.1.1 Production

Lime has been used in building construction since at least 10,000 years ago because of its wide range of raw materials, low cost, and simple process. Egyptians used mortar made with a binder obtained by gypsum calcination in the pyramids of Giza more than 4000 years ago. Chinese started using lime in building foundations and roofs around 3000 years ago, and the mix of lime, clay, and sand slurry more than 2000 years ago. In 1812, the French established hydraulic lime manufacture from synthetic mixtures of limestone and clay.

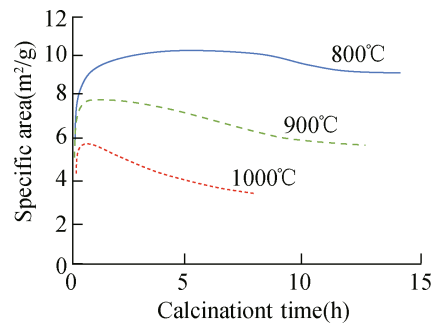
Lime is produced from limestone, dolomite, or other natural materials mainly containing calcium and/or magnesium carbonate. By calcining the above materials, the calcium carbonate breaks down into calcium oxide and forms the quicklime. The quicklime is in block shape, also known as block ash. Its structure is crisp and is ground into quicklime powder for construction. This process is shown in Eq. (3.1). The calcination temperature is often controlled at 900–1000 °C to accelerate the decomposition of limestone with varying density, size, and impurity.



The quicklime contains a specific amount of magnesium oxide from the magnesium carbonate in the limestone or the dolomite. The quicklime with less than 5% magnesium oxide is calcitic lime, while the quicklime with more than 5% magnesium oxide is dolomite lime. Compared with calcitic lime, dolomite lime is slower to mature but slightly stronger after hardening.

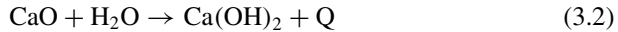
The quicklime may contain under-burnt lime and over-burnt lime. The under-burnt lime contains a specific amount of raw calcium carbonate or limestone residual in the lime due to the low calcination temperature. It generates less mortar when mixed with water and the quality is poor. The over-burnt lime contains over calcined particles which are less hydraulically reactive. Because of the high calcination temperature or longer calcination time, the surface of over-burnt lime particles is melted, forming a relatively dense structure and covering the lime particles with a layer of brown melt. The over-calcined particles harden slower than lime and may cause volume expansion, leading to uplifting and cracking of the hardened lime paste. Figure 3.1 (Huang et al., 2020) shows the specific surface area of lime at different calcination temperatures and times.

Fig. 3.1 The specific surface area of lime at different calcination temperatures and times, redrafted after Huang et al. (2020)



3.1.2 Slaking and Hardening

To eliminate the damage caused by the over-burnt lime, lime putty is produced by slaking or hydrating quicklime with an excess of water for several weeks until a creamy texture is produced. Lime releases a lot of heat when slaking which is nine times that of Portland cement, and the volume increases 1–2.5 times. The slaking process is the reaction of calcium and water to form calcium hydroxide, as shown in Eq. (3.2). Slaked lime has to be immersed in water to avoid being carbonized.

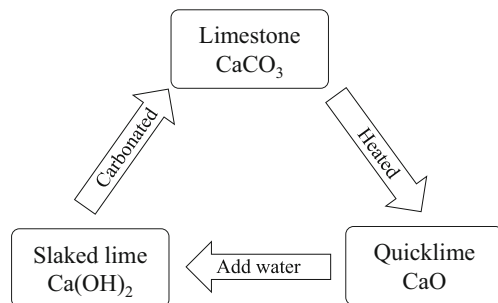


Hardening of lime slurry includes crystallization and carbonization. The crystallization is when lime mortar dries, the calcium hydroxide solution is over-saturated and precipitates crystals. This process contributes very low strength. In addition, during the drying process of lime slurry, the pore structure is formed. Due to the surface tension, the free water left in the pore forms a concave meniscus, generating capillary pressure and making the lime particles more compacted and obtaining strength. This strength is similar to the strength gained by clay after water loss but it may lose this strength when it encounters water again.

Carbonation is the process that lime reacts with carbon dioxide and water, which gradually reconverts calcium oxide back to calcium carbonate, as shown in Eq. (3.3). If the water content is too low, the carbonation process will stop. If the water content is too high and the pores are filled with water, the carbonation will only occur on the surface where the lime can contact carbon dioxide. When the calcium carbonate formed on the surface reaches a specific thickness, it prevents the infiltration of water and carbon dioxide, and the carbonation becomes extremely slow. Figure 3.2 shows the life cycle of lime, in which limestone (calcium carbonate) is converted to quicklime by heating, then to slaked lime by hydration, and naturally reverts to calcium carbonate by carbonation.



Fig. 3.2 The life cycle of lime



3.1.3 Properties

Lime mortar or cement lime mortar prepared with limestone plaster or hydrated lime powder are among the most commonly used materials in buildings. Lime, cement, and slag stabilized mixtures are used as pavement base or subbase. Lime stabilized soil and lime piles are used in the foundation. Lime has the following properties, making it a suitable material for many applications in civil engineering.

- (1) Good plasticity and water retention: Due to a large number of fine calcium hydroxide particles and the large specific surface area, a large amount of water can be absorbed on the surface of particles, making the mortar with good plasticity and water retention.
- (2) Slow setting and low strength: Because of the low content of carbon dioxide in the air and the insulation of calcium carbonate formed on the surface, the formation of strength is very slow. The hardened lime has low strength, and its 28-day strength is 0.2–0.5 MPa. Lime mortar can take a long time to achieve its full strength, which can be many months depending on the conditions of moisture and temperature.
- (3) Large shrinkage: A large amount of water evaporates during hardening, and the volume shrinks greatly due to the loss of capillary water. The porous structure also contributes to the large shrinkage, which may cause cracking.
- (4) Poor water resistance: In a humid environment, the moisture in the lime can not evaporate, and the hardening stops. That's why lime is an air-hardening material. The hardened lime has poor resistance to water mainly because it is porous and the calcium hydroxide is soluble in water.

The quality of quicklime is mainly determined by the content of effective calcium oxide, magnesium oxide, over-burnt lime, and other impurities in lime. The carbon dioxide content can be tested based on the amount of calcium carbonate. The content of residues can be determined based on the residues on the 5 mm round hole sieve in slaked lime. The specifications JC/T 479-2013 (2013) and JC/T 481-2013 (2013) classify quicklime and slaked lime used in buildings into three grades, as shown in Tables 3.1 and 3.2 respectively. Table 3.3 shows the three grades of lime used in pavements according to specification JTG/T F20-2015 (2015).

3.2 Production of Cement

Cement is a fundamental material in civil engineering. 2000 years ago, Romans started using lime and volcanic ash or pozzolan, which is a natural form of cement. In 1824, English inventor Joseph Aspdin invented Portland cement, which has remained the dominant form of cement used in concrete. In 1889, China built the first cement

Table 3.1 Grades of quicklime used in buildings (JC/T 479-2013, 2013)

Items		Calcitic quicklime			Dolomite quicklime	
		CL90	CL85	CL75	ML85	ML80
Min. CaO and MgO content (%)		90	85	75	85	80
MgO content (%)		≤ 5			> 5	
Max. CO ₂ content (%)		4	7	12	7	7
Max. SO ₃ content (%)		2				
Min. volume of slurry (L/kg)		2.6			-	
Fineness	Max. retained on 0.2 mm sieve (%)	2				7
	Max. retained on 0.09 mm sieve (%)	7				2

Note CL means CaO lime; ML means MgO lime

Table 3.2 Grades of slaked lime used in buildings (JC/T 481-2013, 2013)

Items		Calcitic slaked lime			Dolomite slaked lime	
		HCL90	HCL85	HCL75	HML85	HML80
Min. CaO and MgO content (%)		90	85	75	85	80
MgO content (%)		≤ 5			> 5	
Max. SO ₃ content (%)		2				
Max. free water content (%)		2				
Volume stability		Qualified				
Fineness	Max. retained on 0.2 mm sieve (%)	2				
	Max. retained on 0.09 mm sieve (%)	7				

Note HCL means hydrated CaO lime; HML means hydrated MgO lime

plant in Tangshan. Since 1985, China has been producing the majority of the cement in the world. In 2019, the world produced 4.4 billion tons of cement. China produced and consumed about 60% of the world's cement. The famous three Gorges Dam used 16 million tons of cement. The main application of Portland cement is to make Portland cement concrete. It can also be used for other purposes, such as stabilizing soil and aggregate base for highway construction. Understanding the production, chemical composition, hydration, and properties of Portland cement are critical for the selection, design, and quality control of cement concrete.

3.2.1 Classification

Based on the content of supplementary materials and the purposes, the specifications in China generally classify cement into four types:

Table 3.3 Grades of lime used in pavement engineering (JTG/T F20-2015, 2015)

Items		Calcitic quicklime			Dolomite quicklime			Calcitic slaked lime			Dolomite slaked lime		
		I	II	III	I	II	III	I	II	III	I	II	III
Min. effective CaO and MgO content (%)		85	80	70	80	75	65	65	60	55	60	55	50
Max. impurity (Residuals on 5 mm sieve) content (%)		7	11	17	10	14	20	–	–	–	–	–	–
Max. water content (%)		–	–	–	–	–	–	4	4	4	4	4	4
Fineness	Max. retained on 0.6 mm sieve (%)	–	–	–	–	–	–	0	1	1	0	1	1
	Max. retained on 0.15 mm sieve (%)	–	–	–	–	–	–	13	20	–	13	20	–
Content of CaO and MgO (%)		≤ 5			> 5			≤ 4			> 4		

- (1) Portland cement is a hydraulic binding material mainly consisting of Portland cement clinker, 0–5% limestone or granulated blast furnace slag, and a limited amount of gypsum. According to specification GB 175-2007 (2007), Portland cement is further classified into two types: Type I Portland cement, coded as P-I, does not contain supplementary materials; Type II Portland cement, coded as P-II, contains ≤ 5% ground limestone or granulated blast furnace slag.
- (2) The ordinary Portland cement contains 5–20% supplementary materials.
- (3) The specific or blended Portland cement which contains a higher percentage of supplementary materials includes fly ash cement, slag cement, and pozzolanic cement. The highest strength grade for ordinary Portland cement and blended Portland cement is 52.5, while that for Portland cement is 62.5.
- (4) The special cement is the cement with special functions, such as oil well cement, pavement cement, quick-set cement, and expansive cement.

3.2.2 Production

As shown in Fig. 3.3, the Portland cement production process begins with mixing and grinding the limestone and clay at specific proportions and then heating to a sintering temperature in a kiln to produce clinker which are rounded nodules between 1 and 25 mm. Then, the clinker is cooled and ground with gypsum (CaSO_4) into a fine powder in a mill to create cement.

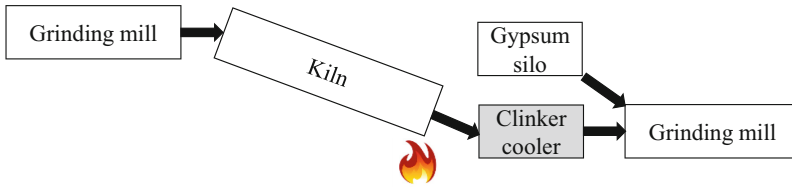


Fig. 3.3 The production of Portland cement

The raw materials for producing Portland cement include lime, silica, alumina, and iron oxide. The limestone, chalk, or oyster shells provide calcium oxide. The clay, shale, and blast furnace slag provide silica, alumina, and ferrite oxides. These raw materials are crushed, stored in silos, and then ground in the desired proportions using either a wet or dry process. The ground material is stored and then heated and sent to the kiln, where these raw materials interact to form complex chemical compounds. The final process is to grind the clinker into a fine powder and a small amount of gypsum is added during grinding to regulate the setting time of the cement.

Different calcine temperatures lead to different mineral composition of cement. At 800 °C, dicalcium silicate (C_2S) starts to be generated. At 900–1100 °C, tricalcium aluminate (C_3A) and tetracalcium aluminoferrite (C_4AF) are generated. At 1100–1200 °C, a large number of C_2S , C_3A , and C_4AF are generated. At 1200–1300 °C, the liquid phase is formed. At 1300–1450 °C, C_2S synthesizes C_3S by absorbing calcium oxide (CaO), which is the key to the production of cement.

3.2.3 Composition

Cement has four main compounds including C_3S , C_2S , C_3A , and C_4AF . Table 3.4 summarizes the formula, abbreviate, content, reaction rate, reaction heat, and strength contribution of the four main compounds. Generally, C_3S accounts for 36–60% of cement and has a moderate reaction rate and reaction heat. C_2S accounts for 15–37% of cement and has a slow reaction rate and low reaction heat. C_3A accounts for 7–15% of cement and has a fast reaction rate and very high reaction heat. C_4AF accounts for 10–18% of cement and has a moderate reaction rate and reaction heat. C_3S is the most abundant compound in cement clinker, and it hydrates more rapidly than C_2S contributing to the final setting time and early strength gain of the cement paste.

The properties of cement vary based on the proportions of the main compounds. C_3S provides both early and long-term strength because of its moderate reaction rate and large amount. C_2S only provides long-term strength due to the slow reaction rate. C_3A and C_4AF provide low strength due to the limited amount, although the reaction

Table 3.4 Properties of main compounds of Portland cement

Compound	Formula	Abbreviation	Content (%)	Reaction rate	Reaction heat	Strength contribution
Tricalcium Silicate	$3\text{CaO}\cdot\text{SiO}_2$	C_3S	36–60	Moderate	Moderate	Early and long-term
Dicalcium Silicate	$2\text{CaO}\cdot\text{SiO}_2$	C_2S	15–37	Slow	Low	Long-term
Tricalcium Aluminate	$3\text{CaO}\cdot\text{Al}_2\text{O}_3$	C_3A	7–15	Fast	Very high	Low
Tetracalcium Aluminoferrite	$4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$	C_4AF	10–18	Moderate	Moderate	Bending

rate of C_3A is fast. C_4AF mainly contributes to the bending strength of cement because its hydration product is a needle-shaped crystal that acts as reinforcement in the hardened cement.

In addition to these four main compounds, Portland cement includes several minor compounds, including free calcium oxide (f-CaO), free magnesium oxide (f-MgO), and alkaline minerals such as sodium oxide (Na_2O) and potassium oxide (K_2O), etc. These minor compounds represent a few percentages by weight of cement. The term minor compounds refer to their quantity and not to their importance. The alkaline minerals can react with the active silica in some aggregates causing the disintegration of concrete and their content should be controlled at a low or safe level.

3.3 Hydration of Cement

3.3.1 Hydration Products

Hydration is the chemical reaction between cement particles and water. This reaction includes the change of matter and molecule energy level. When the cement particles contact water, the minerals in the cement clinker immediately hydrate with water and generate new hydration products and release heat. The main hydration products include calcium silicate hydrate ($3\text{CaO}\cdot 2\text{SiO}_2\cdot 3\text{H}_2\text{O}$ or CSH), calcium hydroxide ($\text{Ca}(\text{OH})_2$ or CH), calcium aluminate hydrate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 6\text{H}_2\text{O}$ or CAH), calcium ferrite hydrate ($\text{CaO}\cdot\text{Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$ or CFH), and calcium sulfoaluminate hydrate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 31\text{H}_2\text{O}$ or CASH). Below are the chemical reactions during hydration and the abbreviated form. In summary, C_3S and C_2S react with water to form CSH and CH. C_3A reacts with water to form CAH. C_4AF reacts with water to form CAH and CFH. CAH then reacts with water and calcium sulfate (CS) to form CASH.

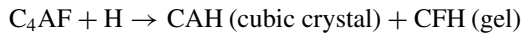
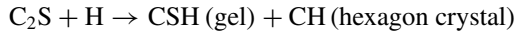
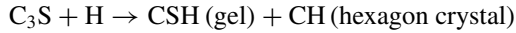
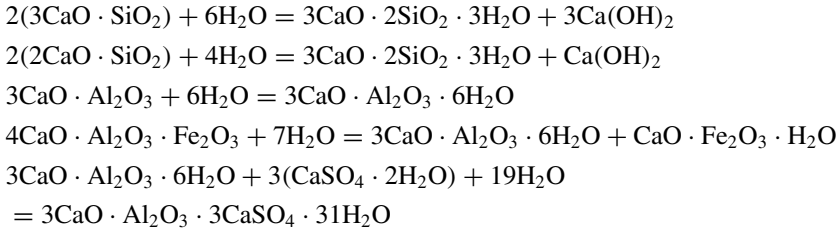


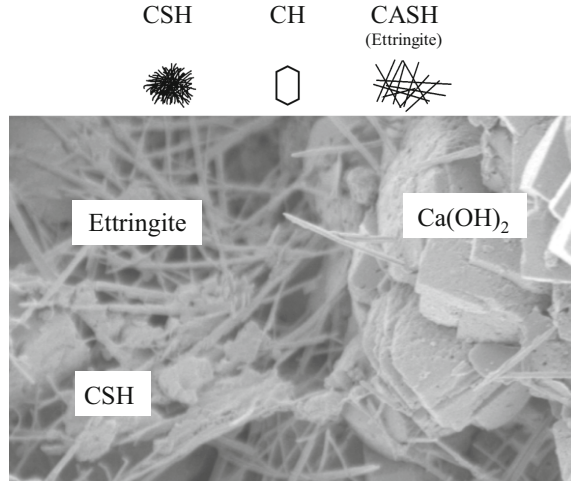
Table 3.5 summarizes the properties of the main hydration products. Figure 3.4 shows the microscopic image of the main hydration products. The main hydration product is CSH. It is an insoluble gel-like poorly crystalline material, accounts for 70% of the hydration products, and is primarily responsible for the strength of concrete. CSH is not a well-defined compound since the calcium-to-silicate ratio varies between 1.5 and 2, and the structurally combined water content is more variable. The rate of hydration is accelerated by sulfate ions in the solution. Thus, a secondary effect of the addition of gypsum to cement is to increase the rate of development of the CSH. Complete hydration of C_3S produces 61% CSH and 39% CH; hydration of C_2S results in 82% CSH and 18% CH.

CH is a hexagonal crystal accounting for 20% of the hydration products. It is easily soluble in water and therefore the lime concentration of the solution quickly reaches the saturation state. The hydration of cement is mainly carried out in the saturated solution of lime.

Table 3.5 The properties of the main hydration products

Product	Abbreviation	Form	Solubility	Proportion
Calcium silicate hydrate	CSH	Gel	Insoluble	70%
Calcium hydroxide	CH	Hexagon	Soluble	20%
Calcium aluminate hydrate	CAH	Cubic	Soluble	–
Calcium ferrite hydrate	CFH	Gel	Insoluble	–
Calcium sulfoaluminate hydrate (Ettringite)	CASH	Needle	Slightly soluble	7%

Fig. 3.4 The microscopic image of the main hydration products



The reaction of C_3A is the fastest and releases large amounts of heat. CS can react with C_3A and slow down the rate of aluminate hydration. CAH is a soluble cubic crystal and CFH is an insoluble gel-like material. CASH, also called ettringite, is a needle-shaped crystal slightly soluble in water, and accounts for 7% of the hydration products.

The aluminate hydrates much faster than the silicate. The reaction of C_3A with water is immediate and releases large amounts of heat. CS is used to slow down the rate of aluminate hydration. The CS dissolves into the solution quickly, producing sulfate ions that suppress the solubility of the aluminate. The balance of aluminate to sulfate is critical for the rate of setting.

3.3.2 Hydration Process

Hydration is the reaction of the cement with water to produce a hydrate, which brings about chemical, physical and mechanical changes in the system. There are two mechanisms for the hydration of Portland cement, the through-solution reaction and topochemical hydration. The through-solution reaction dominates the early stages of hydration and involves the dissolution of anhydrous compounds into the solution, the formation of hydrate products in the solution, and the precipitation of hydrate products from the supersaturated solution. The topochemical hydration is a solid-state chemical reaction occurring at the surface of the cement particles. Some studies found that low water/solid ratios lead to a topochemical reaction, while the through-solution reaction is more important at high water/solid ratios, and both reactions can occur simultaneously.

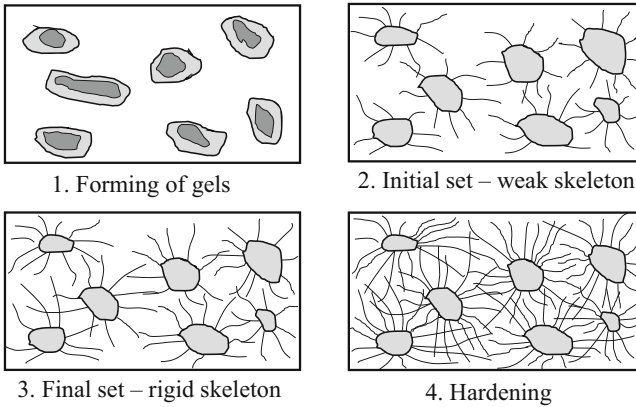


Fig. 3.5 Hydration process, redrafted after Hover and Phillco (1990)

After mixing cement with water, the cement paste has plasticity but will gradually lose plasticity, which is called setting. Then, the cement paste becomes hard and gains strength, which is called hardening. Cement setting and hardening is a continuous and complex process of physical and chemical changes, which determines a series of technical properties of cement. Scholars have researched this process for more than one hundred years and still hold different opinions. Usually, the initial setting is the time when cement can be molded in any desired shape without losing its strength. The final setting is the time when cement completely loses its plasticity and becomes hard. Hardening means cement paste gains strength through hydration of silicate compounds. The mechanism of setting and hardening can be explained with the sequential development of the structure in a cement paste during hydration as summarized in Fig. 3.5, which generally includes four stages.

- (1) During the early stages of hydration, weak bonds can form, particularly from the gel of hydrated C_3A .
- (2) The initial set occurs when further hydration stiffens the mix and begins locking the structure of the material in place.
- (3) The final set occurs when the CSH has developed a rigid structure, and all components of the paste are locked into place.
- (4) The space between the cement grains is filled with hydration products. The cement paste continues hardening and gains strength as hydration continues.

3.3.3 Influencing Factors

The cement setting and hardening process are not only affected by the compounds of the cement, but also by many other factors.

1. Fineness

As shown in Fig. 3.6, finer cement particles increase surface specific area and therefore increase the hydration activity of cement. When the particle size is larger than $45\ \mu\text{m}$, the hydration is difficult, and when the particle size is larger than $75\ \mu\text{m}$, the hydration nearly stops.

2. Gypsum

As shown in Fig. 3.7, gypsum (CaSO_4 or CS) can react with CAH quickly to generate CASH which deposits and forms a protection film on the cement particles to hinder the hydration of C_3A and delay the setting time of cement. When there is sufficient CS, we have calcium tri-sulfoaluminate hydrate, also called Aft and its natural form is ettringite. When CS content is low, we have calcium mono-sulfoaluminate hydrate, also called Afm. Here, A means alumina, f means ferric oxide, t means tri, and m means mono.

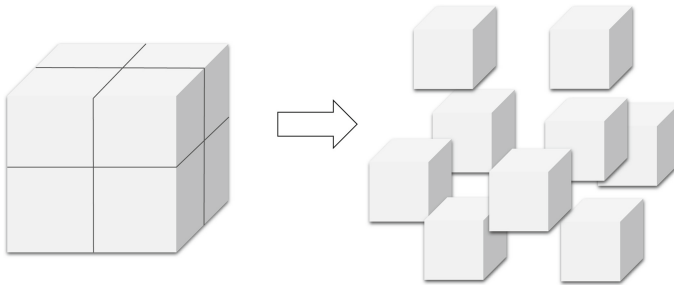


Fig. 3.6 Increased specific surface area for finer cement particles

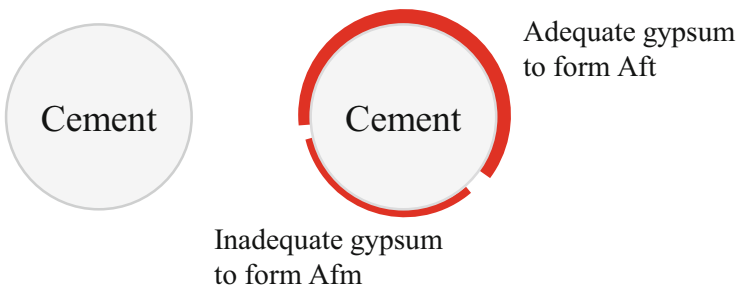


Fig. 3.7 Effects of gypsum in cement hydration

3. Curing time

The amount of hydration products increases with the increase of time and therefore strength increases. The curing time can be classified into setting and hardening. After setting, the amount of hydration products increases rapidly in the first 28 days and the 28-day strength is usually tested for determining cement and concrete strength.

4. Water-cement ratio

Figure 3.8 shows that with the increase of water-cement ratio, the proportional volume of cement hydrates increases first and then decreases. When the water-cement ratio is lower than 0.38, there is not sufficient water to hydrate all cement particles and unhydrated cement remains. However, if the water-cement ratio is too high, there will be a large number of capillaries in the hardened cement paste, increasing porosity and reducing strength.

5. Temperature

As shown in Fig. 3.9, high temperature accelerates hydration and improves the 28-day strength. However, if the temperature is too high (>40 °C), the hydration rate of cement will accelerate, and a large number of hydrates will form around the unhydrated cement particles, which will hinder the further hydration of cement. Low temperature causes the reduction of strength. When water is frozen, hydration nearly stops.

Fig. 3.8 The relationship between proportional volume and water-cement ratio, redrafted after Torben (1986)

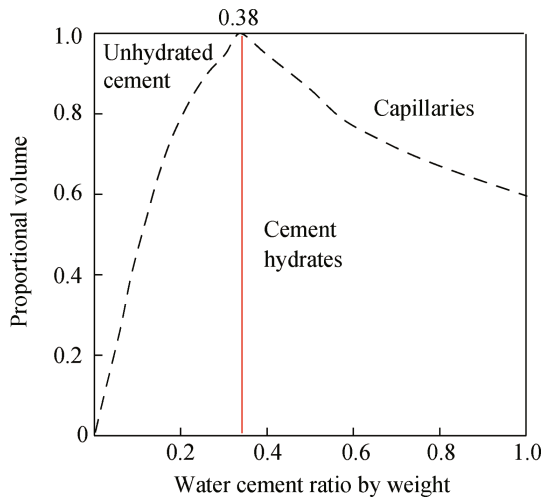
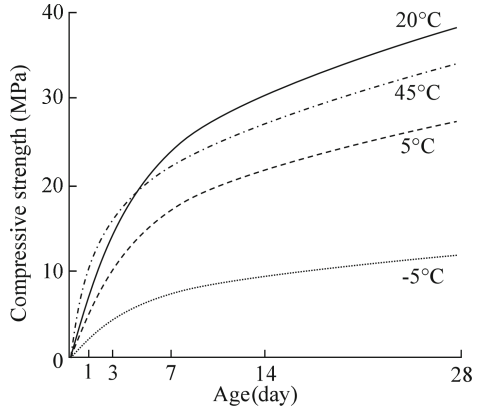


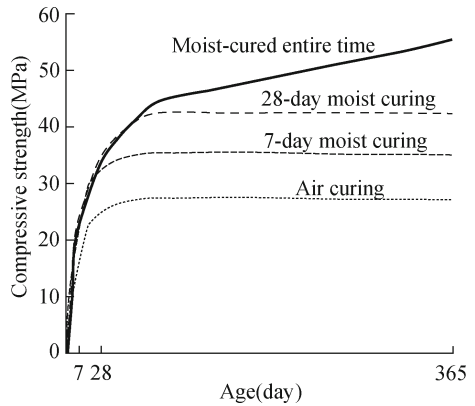
Fig. 3.9 Compressive strength at different curing temperatures, redrafted after Huang et al. (2020)



6. Moisture

Hydration needs sufficient water. The curves in Fig. 3.10 show the compressive strength of cement under different moist curing times. The longer the moisture curing times, the higher the strength. A dry environment causes fast evaporation. When water evaporates, the hydration stops, causing shrinkage cracks and low strength.

Fig. 3.10 Compressive strength at different curing moisture levels, redrafted after Gonnerman and Shuman (1928)



3.4 Properties of Cement

The proper hydration of Portland cement is a fundamental quality of cement, whereas the quality of the concrete is also influenced by mix proportion, characteristics of aggregates, and quality control. Properties of the hydrated cement are evaluated with either cement paste which is the mix of water and cement, or mortar which is the mix of water, cement, and sand. The specification GB 175-2007 (2007) provides the requirements of the chemical and physical properties of cement. In addition to the properties such as fineness, setting time, soundness, and strength, it also has requirements of the amount of insoluble impurities, the loss during sintering, the content of SO_3 , MgO , and chloride ion, and fineness.

3.4.1 Density

The density and unit weight of cement are often needed in the calculation of the proportions in concrete, storing, and transporting. The density of Portland cement is generally $3\text{--}3.15\text{ g/cm}^3$. The unit weight mainly depends on the packing of cement, in addition to the mineral composition and fineness. The unit weight of slack cement is around $1\text{--}1.1\text{ g/cm}^3$, while that of compact cement is around 1.6 g/cm^3 .

3.4.2 Fineness

Fineness refers to the particle size of cement. The fineness of cement particles is an important property that must be carefully controlled. The finer the cement particle, the larger the surface area and the faster the hydration. Therefore, finer material results in faster strength development and greater initial heat of hydration. Cement particles larger than 0.045 mm will have hydration difficulties. $85\text{--}95\%$ of the cement particles are smaller than 0.045 mm , and the average diameter is 0.01 mm . Figure 3.11 shows the irregular shapes of cement particles at the micro-scale.

The fineness of cement can be evaluated by the specific surface area, which is the total surface area per unit weight of cement particles, and can be measured with the Blaine air permeability apparatus. In specification GB 175-2007 (2007), the specific surface area of Portland cement and ordinary Portland cement must be greater than $300\text{ m}^2/\text{kg}$. For the blended cement including slag cement, fly ash cement, Pozzolanic cement, etc., the percentage retained at 0.08 and 0.045 mm sieve should be no more than 10% and 30% , respectively.

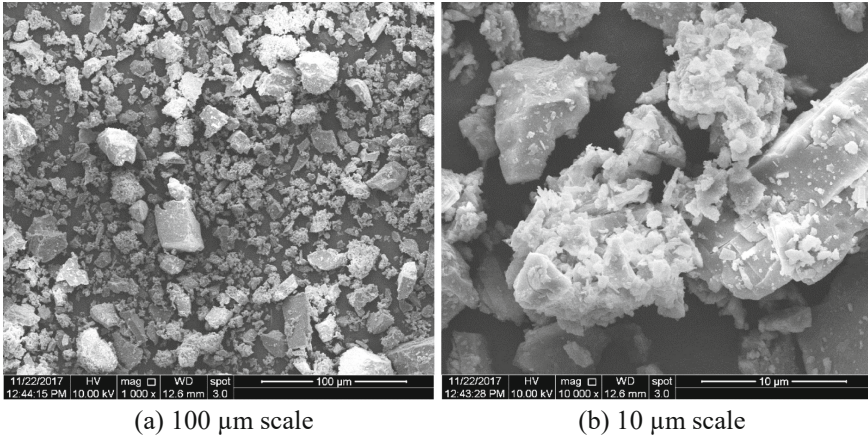


Fig. 3.11 Scanning electron microscope (SEM) image of cement particles

3.4.3 Consistency

Water content for standard consistency refers to the amount of water, expressed as a percentage of the weight of cement, required to mix cement into a specific plastic state, which is called the standard consistency or normal consistency. The properties of the cement paste are tested at the standard consistency so that the results can be compared. In specification GB/T 1346-2011 (2011), the standard consistency allows a standard plunger of 10 mm diameter to penetrate to 6 ± 1 mm above the bottom of the Vicat mold (Fig. 3.12). During the test, the 1 mm diameter needle is allowed to penetrate the paste for 30 s and the amount of penetration is measured. The water content at the standard consistency of Portland cement is generally between 24 and 30%, mainly determined by the mineral composition and fineness.

3.4.4 Setting Time

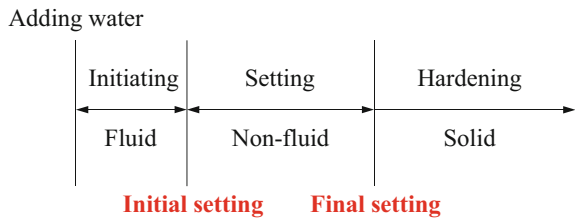
When we add water to cement, the cement paste changes from a fluid to a plastic solid and then a hardened solid. Setting refers to the stiffening of the cement paste or the change from a plastic state to a solid state and is classified into the initial setting and final setting. The initial setting is the time that elapses from the time water is added until the paste ceases to be fluid and plastic. The final setting is the time required for the paste to acquire a certain degree of hardness, lose all plasticity/flowability and start to gain strength. The setting time of cement is important for construction. The initial setting time should not be too fast to have enough time for mixing, transportation, and casting; whereas, after the completion of the casting, the concrete should harden quickly.

Fig. 3.12 Consistency tester



Figure 3.13 shows the definition of setting time. The setting time of cement is tested with the cement paste of standard consistency under specified temperature and humidity using the consistency tester. In specification GB/T 1346-2011 (2011), the initial setting time should be tested from the time when cement is added into water to the time when the needle sinks to 4 ± 1 mm from the bottom plate in the neat paste. The final setting time should be tested from the time when cement is added into the water to the time when the needle sinks into the neat paste 0.5 mm without a ring mark. According to specification GB 175-2007 (2007), for Portland cement, the initial setting time should be no less than 45 min, and the final setting time should be no more than 390 min. For other types of cement, the initial setting time should be no less than 45 min.

Fig. 3.13 Definition of setting time



3.4.5 Soundness

The soundness of the cement paste refers to its ability to retain its volume after setting. Expansion after setting is mainly caused by delayed or slow hydration or other reactions, causing expansive cracks and poor quality of the structure. According to specification GB/T 1346-2011 (2011), the soundness can be evaluated by the pat test and the Le Chatelier test. The pat test is to prepare a cement cake with cement paste of standard consistency, boil it for 3 h, and then observe it with naked eyes. If there is no crack and no bending by ruler inspection, it is called qualified soundness. The Le Chatelier test is to measure the expansion value after the cement paste is boiled and get hardened on Le Chatelier needles. If the expansion value is within the required value (< 5 mm), its stability is qualified. If there is a contradiction between the results of the two tests, the Le Chaterlier test shall prevail.

Too much f-CaO and f-MgO are the main reasons for poor volume stability. Excessive CS can also lead to volume expansion, because the sulfur trioxide ions in CS will react with CAH to form CASH which leads to excess volume expansion. The poor stability caused by f-CaO is tested by boiling curing. The poor stability caused by f-MgO should be tested by the pressure steam curing, because the hydration of f-MgO is slower than that of f-CaO. Generally, the content of f-CaO, f-MgO, and SO_3 ions must be strictly restricted.

3.4.6 Strength

Strength is the key technical performance for selecting cement. The strength mainly depends on the mineral composition and fineness of the cement. Since the strength increases gradually in the hardening process, the strength is evaluated at different curing ages. The specification GB/T 17671-2021 (2021) introduces a method for testing the three-point flexural strength of the cement mortar beam and the compressive strength of each fractured part. The cement, standard sand, and water are mixed at a ratio of 1:3:0.5 to prepare the $40 \times 40 \times 160$ mm cement mortar beams which will be cured under the standard condition of 20 ± 1 °C and 90% humidity for 3 and 28 days.

The loading rate for the flexural test is 50 N/s and the flexural strength is calculated by Eq. (3.4). The final flexural strength is the average of three specimens. If the result of any specimen is not within $\pm 10\%$ of the average of the three, that result should be dropped, and the final result is the average of the rest two specimens. If any of the two values is not within $\pm 10\%$ of the average value, the test result is invalid.

$$R_f = \frac{3F_f L}{2bh^2} = 0.00234F_f \quad (3.4)$$

Where

R_f = flexural strength (MPa);

- F_f = failure load (N);
- L = distance between two supporting points (100 mm),
- b = width of the beam (40 mm);
- h = height of the beam (40 mm).

The fractured specimens from the flexural tests should be tested immediately for compressive strength. The top and bottom compression surfaces are the two lateral sides of the cement mortar beam. The loading rate is 2400 N/s and the compressive strength is calculated by Eq. (3.5). The final compressive strength is the average of the six specimens. If any of the specimens is not within $\pm 10\%$ of the average of the six, that result shall be dropped, and the final result is the average of the rest five specimens. If any of the five values is not within $\pm 10\%$ of the average value, the test result is invalid.

$$R_c = \frac{F_c}{A} \tag{3.5}$$

Where

- R_c = compressive strength (MPa);
- F_c = failure load (N);
- A = area of compression surface, $40 \times 40 \times 160\text{mm}^2$.

According to specification GB 175-2007 (2007), the Portland cement can be classified into three strength grades including 42.5, 52.5, and 62.5, according to the 3-day and 28-day flexural strength and compressive strength. If the cement has higher flexural strength and compressive strength at an early age, it is called early-strength cement, which comes with a serial number “R”. The strength at different ages of cement of each strength grade should not be lower than the values in Table 3.6.

Table 3.6 Strength grades of different cement (GB 175-2007, 2007)

Types	Grades	Minimum compressive strength (MPa)		Minimum flexural strength (MPa)	
		3-day	28-day	3-day	28-day
Portland cement	42.5/42.5R	17/22	42.5	3.5/4	6.5
	52.5/52.5R	23/27	52.5	4/5	7
	62.5/62.5R	28/32	62.5	5/5.5	8
Ordinary Portland cement	42.5/42.5R	17/22	42.5	3.5/4	6.5
	52.5/52.5R	23/27	52.5	4/5	7
Slag, pozzolanic, fly ash, blended cement	32.5/32.5R	10/15	32.5	2.5/3.5	5.5
	42.5/42.5R	15/19	42.5	3.5/4	6.5
	52.5/52.5R	21/23	52.5	4/5	7

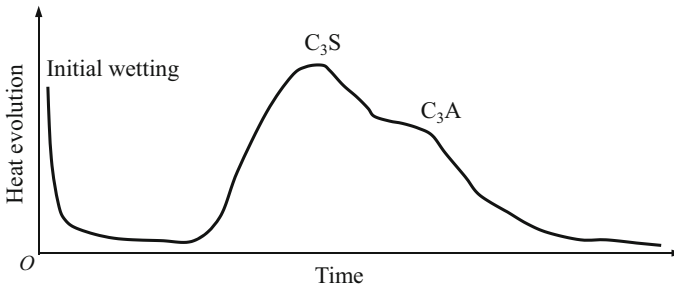


Fig. 3.14 Cement hydration heat over curing time, redrafted after Wilson and Paul (2021)

3.4.7 Hydration Heat

Hydration heat refers to the heat released in the hydration process of cement. Most of the hydration heat is released at the initial stage within 7 days and then decreases gradually. Heat develops rapidly during setting and initial hardening and gradually declines and finally stabilizes as hydration slows. Figure 3.14 shows the hydration heat over curing time, which includes three peaks. The first peak is during the initial wetting, mainly caused by the formation of Aft of C_3A . The second peak is during the hydration of C_3S , the third peak is during the transformation from Aft to Afm, and the hydration of C_3A . The hydration heat is determined by the mineral composition and fineness of the cement. In winter construction, the hydration heat is beneficial for the cement hardening. But for mass concrete structures, too much hydration heat is harmful. The hydration heat accumulated in the interior is not easy to release and the internal temperature can reach 70–90 °C, resulting in temperature stress and cracks.

3.4.8 Voids

Due to the random growth of the crystals and the different types of crystals, voids are left in the paste structure as cement hydrates and have a great influence on the strength, durability, and volume stability of concrete. Two types of voids are formed during hydration: the interlayer hydration space and capillary voids. Interlayer hydration space is between the layers in the CSH. The space thickness is 0.5–2.5 nm, which is too small to influence the strength but contributes 28% to the porosity. Water in the interlayer hydration space is held by hydrogen bonds, but can be removed when humidity is less than 11%, causing shrinkage. Capillary voids are mainly determined by the spaces and hydration of cement particles. Capillary voids greater than 50 nm decrease strength and increase permeability. Removal of water from capillary voids greater than 50 nm does not cause shrinkage, whereas removal of water from the smaller voids causes shrinkage. In addition to the interlayer hydration space and capillary voids, the cement paste contains trapped air which reduces strength

and increases permeability. However, well-distributed small air bubbles can greatly increase the durability of the cement paste.

3.5 Corrosion

Portland cement has good durability in a normal environment after hardening. The compressive strength of cement concrete after 30 to 50 years can be 30% higher than that of 28 days due to the continuous hydration. However, corrosion may occur when the cement hydration products react with other chemicals in the environment resulting in surface chalking, discoloration, cracking, steel rusting, and other distress. Generally, corrosion is mainly caused by either the formation of expansive products or the loss of materials due to the dissolution of hydration products in water or the reaction of hydration products with acids or ions.

3.5.1 *Soft Water Corrosion*

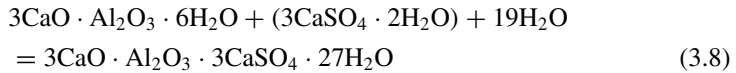
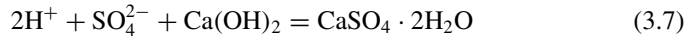
Soft water refers to water with small temporary hardness, such as rainwater, snow water, river water, and lake water containing less bicarbonate. Hard water is formed when water percolates through deposits of limestone, chalk, or gypsum which are largely made up of calcium and magnesium carbonate, bicarbonate, and sulfate. Temporary hardness is calculated as the bicarbonate content per liter of water.

When the cement mortar contacts soft water for a long time, some of the hydration products will be gradually dissolved in water. CH has the greatest solubility about 1.2 g/L at 25 °C. In the case of still water or with no water pressure, the surrounding water is rapidly saturated with the dissolved CH. The dissolution effect is quickly terminated. Therefore the dissolution is limited to the surface and has little effect. But in flowing water, especially water pressure, water will continuously dissolve CH and carry it away. Other hydration products, such as CAH, CAH, etc., may also dissolve in water, which will reduce the strength, and even cause the failure of the structure. The higher the temporary hardness of water, the weaker the corrosion of cement mortar. Because the bicarbonate in water can react with CH in cement to form CaCO₃ which is insoluble in water, as shown in Eq. (3.6). The CaCO₃ accumulates in the pores of the cement mortar, forming a dense protective layer, and preventing the infiltration of water.



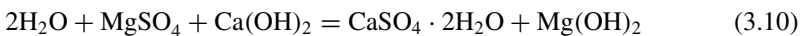
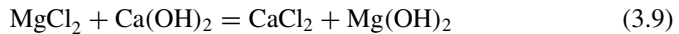
3.5.2 Sulfate Attack

The seawater, groundwater, and some industrial sewage may contain sulfate, which has a corrosive effect on cement mortar. Sulfate reacts with calcium hydrates to form CS and then reacts with CAH to form CASH, as shown in Eqs. (3.7) and (3.8). CASH contains a large amount of crystal water, and its volume increases by 1.5 times. CASH is often called “cement bacillus” because it is a needle-shaped crystal. This reaction has a destructive effect on cement concrete. Adding the CS to the cement clinker to regulate the setting time can also produce CASH, but its content is strictly restricted and will not cause damage.



3.5.3 Magnesium Corrosion

Seawater and groundwater often contain a large number of magnesium salts, mainly magnesium sulfate and magnesium chloride. They usually have a replacement reaction with CH. Magnesium salts react with calcium hydroxide to form calcium dichloride (CaCl_2) and calcium sulfate (CaSO_4 , i.e. CS), as shown in Eqs. (3.9) and (3.10). Because magnesium hydroxide is soft and unbonded, and calcium chloride is soluble in water, magnesium corrosion can seriously damage the structure, especially if there is also sulfate in the water. CS can react with CAH to form CASH. The magnesium corrosion is shown in Fig. 3.15.



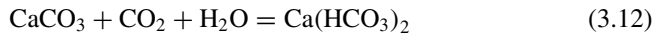
3.5.4 Carbonation

In most natural water, there is usually some free carbon dioxide and its salts. If the free carbon dioxide is too much, it will be destructive. Calcium hydroxide reacts with carbon dioxide and water to form calcium carbonate which is low bonding, or calcium hydrogen carbonate which is soluble, as shown in Eqs. (3.11) and (3.12).

Fig. 3.15 Typical magnesium corrosion of cement concrete

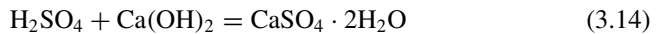
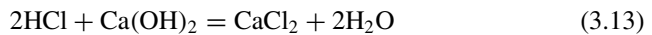


Calcium hydroxide in cement mortar is dissolved by transforming into soluble calcium hydrogen carbonate, leading to the decomposition of other hydrates, and enhancing the corrosion effect.



3.5.5 Acid Corrosion

Various acids are often found in industrial waste water, groundwater, and marsh water which have a corrosion effect on cement mortar. The acids react with calcium hydroxide to form compounds that either dissolve in water or expand in volume. Typical acid corrosion includes hydrochloric and sulfuric acid corrosion. Hydrochloric acid reacts with calcium hydroxide to form calcium chloride which is soluble in water, as shown in Eq. (3.13). Sulfuric acid (H_2SO_4) reacts with CH to form CS, as shown in Eq. (3.14). The CS either leads to volume expansion or reacts with CAH to form CASH causing cracking. Generally, the greater the concentration of hydrogen ions in water, the smaller the pH, the more severe the corrosion.



Generally, the corrosion discussed above can be summarized into three types.

- (1) Dissolution corrosion: Some media gradually dissolve components of cement mortar, resulting in dissolution damage.

- (2) Ion exchange: Ion exchange reaction occurs between corrosion materials and cement components, and forms dissolution products or unbonded products which can damage the original structure.
- (3) Forming expansive compounds: The salt crystals formed by corrosion reactions increase in volume and generate harmful internal stress, causing cracking or other expansion failures.

3.5.6 Measures

According to the different corrosion causes, the following prevention measures can be taken to reduce the risk of corrosion:

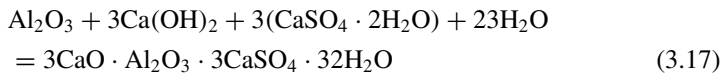
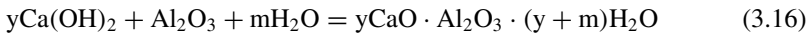
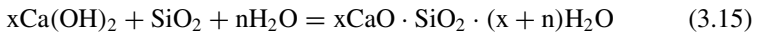
- (1) Choosing proper cement according to the environment: If the cement mortar suffers from soft water corrosion, it is recommended to select the cement with less calcium hydroxide content. If the cement mortar suffers sulfate corrosion, it is recommended to select sulfate-resistant cement with low content of tricalcium aluminate. Additionally, there are some types of Portland cement clinker added with artificial or natural mineral materials (mixed materials), which have better corrosion resistance.
- (2) Reducing permeability: Improving the impermeability of cement mortar is an effective measure to prevent corrosion. The greater the density of the cement mortar, the stronger the impermeability, the more difficult for the corrosive media to enter. Reducing permeability also can resist soft water corrosion.
- (3) Using protection sealing: When taking the above measures is still difficult to prevent corrosion, it is recommended to apply a waterproof layer with strong corrosion resistance on the surface of cement products. Common coatings include silica sealing coat, acid-resistant stone, acid-resistant ceramics, glass, plastics, asphalt, etc.

To control cement quality, there are some properties of cement should be tested. Insoluble residues are to evaluate the insoluble residues in HCl or NaCO₃ solutions to check the impurities in cement. Loss on ignition is to check the weight loss of cement heated at 950 °C for 15 min. As the main cause of steel corrosion, the chloride ion content must be tested. According to specification GB 175-2007 (2007), the chloride ion in cement should be less than 0.06%. To control the content of f-MgO and SO₃ in cement, the soundness should be also tested.

3.6 Supplementary Materials and Blended Cement

3.6.1 Supplementary Materials

Since the 1970s, some byproducts of other industries have been used in cement as supplementary cementitious materials. These supplementary cementitious materials mainly include fly ash, slag, and pozzolan. Since these materials are cementitious, they can be used in addition to or as a partial replacement for Portland cement. They are added as part of the total cementitious system to adjust the strength, improve cement performance and reduce the cost. The materials usually contain active SiO_2 and Al_2O_3 , which can react with CH to form CSH or CAH, and CAH can further react with gypsum to form CASH, as shown in Eqs. (3.15)–(3.17). This is called the pozzolanic property or secondary hydration because it utilizes and relies on the CH which is generated during cement hydration. The key to the pozzolanic property is that the structure of the silica must be in a glassy or amorphous form with a disordered structure, which can be formed by rapid cooling from a molten state. Intermolecular bonds in the structure are not at their preferred low energy orientation and are easy to break and link with calcium hydroxide.



1. Slag

Slag, also called ground granulated blast furnace slag (GGBFS), is a glass material formed by molten slag produced in the blast furnace as an industrial byproduct of the production of iron. The slag is developed in a molten condition simultaneously with iron in a blast furnace. The molten slag is rapidly chilled by quenching in water to form a glassy, sand-like granulated material. The material is then ground to less than $45 \mu\text{m}$ to be the GGBFS which contains CaO , MgO , Al_2O_3 , SiO_2 , Fe_2O_3 , and other oxides and a small amount of sulfate. Generally, the CaO , SiO_2 , and Al_2O_3 account for more than 90% of slag. Its chemical composition is similar to that of Portland cement. The quality of GGBFS is evaluated by the quality coefficient (K) as shown in Eq. (3.18). The quality coefficient is the weight ratio of active compounds over the less and inactive compounds in the slag. The higher the quality coefficient, the higher the activity of slag. The specification GB 175-2007 (2007) requires that the quality coefficient of GGBFS used for cement shall not be less than 1.2.

$$K = \frac{\text{CaO} + \text{MgO} + \text{Al}_2\text{O}_3}{\text{SiO}_2 + \text{MnO} + \text{TiO}_2} \quad (3.18)$$

2. Fly ash

Fly ash is the most commonly used supplementary material in cement. It is a byproduct of burning pulverized coal in electric power generating plants. China relies heavily on coal-fired power and generates around 600 million tons of fly ash in 2020. Combusting pulverized coal in an electric power plant burns off the carbon and most volatile materials. However, the carbon content of common coal ranges from 70 to 100%. The impurities such as clay, feldspar, quartz, and shale fuse as they pass through the combustion chamber. When those impurities fuse in suspension and are carried away from the combustion chamber by the exhaust gas, they cool and solidify into 1–50 μm solid and hollow spherical glassy fly ash. The particle diameters of fly ash range from 1 μm to more than 0.1 mm, with an average of 0.015–0.02 mm, and are 70–90% smaller than 45 μm . Fly ash is primarily silica glass composed of CaO, SiO₂, Al₂O₃, and Fe₂O₃. Fly ash can be classified into two types: Class F and Class C, depending on the content of CaO. Class F fly ash usually has less than 5% CaO but may contain up to 10%. Class C fly ash has 15–30% CaO. According to specification GB/T 1596-2017 (2017), the fly ash used in cement should meet the requirements in Table 3.7.

3. Pozzolan

Pozzolan is a siliceous and aluminous material with little or no cementitious effect but in the presence of moisture, it can react with CH in a finely divided form to form compounds possessing cementitious properties. Natural pozzolan includes volcanic ash, tuff, pumice, zeolite, diatomite, etc. Artificial pozzolan includes calcining shale and clay, cinder, siliceous slag, etc.

Table 3.7 Technical requirements for fly ash used in cement (GB/T 1596-2017, 2017)

Items	Types of fly ash	Requirements
Max. loss on ignition(%)	Class F and Class C	8.0
Max. water content (%)	Class F and Class C	1.0
Max. SO ₃ content (%)	Class F and Class C	3.5
Max. f-CaO content (%)	Class F	1.0
	Class C	4.0
Max. soundness (mm)	Class C	5.0
Min. strength activity index (%)	Class F and Class C	70.0

3.6.2 Blended Cement

Supplementary materials can be added to Portland cement to replace part of cement to produce blended cement. The properties of Portland cement, Portland ordinary cement, Portland slag cement, Portland fly ash cement, and Portland pozzolanic cement, are shown in Table 3.8.

The specific gravity of slag cement ranges from 2.85 to 2.95. The rough and angular-shaped ground slag in the presence of water and an activator, NaOH or $\text{Ca}(\text{OH})_2$, both supplied by Portland cement, hydrates, and sets like Portland cement. Fly ash increases the workability of the fresh concrete because of its sphere particle shape. Fly ash extends the hydration process, allowing greater strength development and reduced porosity. Studies have shown that concrete containing more than 20% fly ash has a much smaller pore size distribution. Pozzolanic cement and slag cement have a lot in common in properties. It mainly reacts with the CH produced during the hydration of C_3S and C_2S . Table 3.9 summarizes the properties of blended cement and the details are discussed below.

Table 3.8 Types of blended cement

Abbreviations	Names	Blended materials	Content (%)
P-I, P-II	Portland cement	Supplementary materials	< 5
P-O	Portland ordinary cement	Supplementary materials	6–15
P-S	Portland slag cement	Slag	20–70
P-F	Portland fly ash cement	Fly ash	20–40
P-P	Portland pozzolanic cement	Pozzolan	20–50

Table 3.9 Properties of blended cement

Properties	Portland cement	Portland ordinary cement	Portland slag cement	Portland fly ash cement	Portland pozzolanic cement
Content (%)	< 5	6–15	20–70	20–40	20–50
Workability	Moderate	Moderate	Moderate	Good	Moderate
Hydration	Very fast	Fast	Slow	Slow	Slow
Hydration heat	High	High	Low	Low	Low
Early strength	Very high	High	Low	Low	Low
Impermeability	Moderate	Moderate	Good	Good	Good
Corrosion resistance	Moderate	Moderate	Good	Good	Good
Freeze–thaw resistance	Good	Good	Low	Low	Low
Shrinkage	High	High	High	Low	High

- (1) Workability: The workability of fly ash cement is better because of its spherical particles with dense surfaces.
- (2) Hydration heat: Generally, the hydration of blended cement is slower than that of Portland cement or ordinary Portland cement, and therefore the early age strength and hydration heat are also lower (Fig. 3.16).
- (3) Strength: As shown in Fig. 3.17, although the early age strength of slag and fly ash cement are lower, strength development after 28 days can exceed that of Portland cement. Usually, the higher the content of supplementary materials, the lower the early strength, but the higher the long-term strength. Therefore, it should be cured in a moist condition for a longer time.

Fig. 3.16 Concrete temperature of blended cement, redrafted after Bamforth (1980)

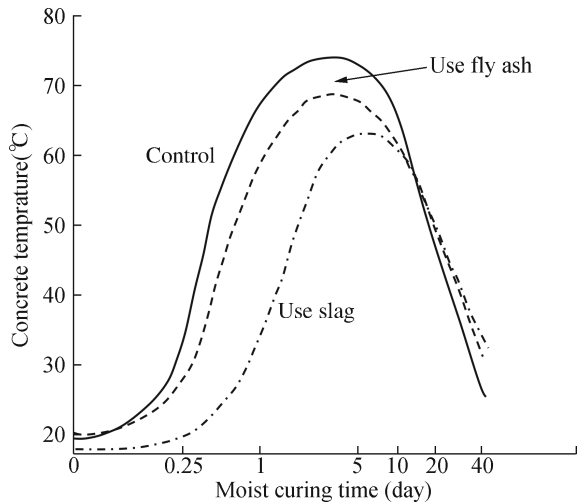
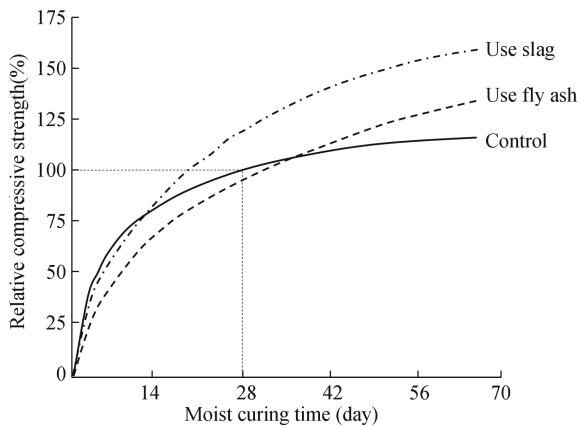


Fig. 3.17 Strength curves of blended cement, redrafted after Bamforth (1980)



- (4) **Impermeability:** Blended cement has good impermeability and corrosion resistance because the secondary hydration generates more CSH gels and crystals filling the pores. Figure 3.18 shows that using fly ash and slag reduces both the accessible pore volume and the effective pore diameter. Generally, the permeability and absorption of hardened cement paste are reduced.
- (5) **Corrosion resistance:** As shown in Fig. 3.19, the volume expansion of blended cement during sulfate attack and alkali-aggregate reaction is also reduced. One reason is the improved impermeability of hardened cement paste. Another reason is CH and CAH are all consumed during the secondary hydration and therefore the risks of sulfate, salt, and acid corrosion are greatly reduced.
- (6) **Freeze–thaw resistance:** Because blended cement has fewer micro air voids in hydrated cement mortar and can not provide space for the volume expansion of water during freezing. The freeze–thaw resistance of blended cement is lower than that of Portland cement.
- (7) **Shrinkage:** Low content of supplementary materials usually have little effect on shrinkage. High content of slag may increase shrinkage, while fly ash tends to reduce the dry shrinkage. As the surface of fly ash is dense and spherical, its

Fig. 3.18 The relationship between pore volume and pore diameter, redrafted after Jan (1996)

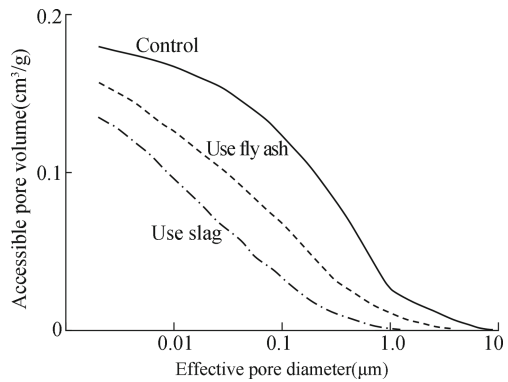


Fig. 3.19 Expansion under sulfate attack or alkali-aggregate reaction, redrafted after Freeman and Carrasquillo (1991)

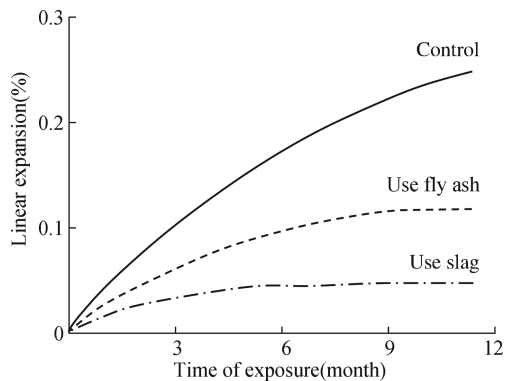


Table 3.10 Strength test results

Ultimate compressive load (kN)	80.9	86.5	95.2	91.3	81.4	92.5
Ultimate flexural load (kN)	2.79		2.81		2.78	

water absorption is low. The water content at the standard consistency of fly ash cement is low, and the dry shrinkage is also small.

Questions

1. What are reactions during the slaking and hardening of lime?
2. What are the primary chemical reactions during the hydration of Portland cement?
3. What are the four main chemical compounds in Portland cement?
4. Why gypsum is added in Portland cement?
5. What are the main hydration products of Portland cement?
6. Briefly discuss the initial setting and final setting of Portland cement.
7. Why is the early strength of slag cement lower while the long-term strength is higher than the same grade of ordinary cement?
8. What are the two main reactions of the magnesium corrosion?
9. Discuss the measures to prevent cement corrosion.
10. A batch of 42.5 grade Portland cement has been stored in the factory for a very long time. The 28-day strength of samples are tested and the results are shown in Table 3.10. Do the test results of this cement reach the requirements of the original strength grade? Can we determine the strength grade of the cement only by this test result?

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