Characterization of the properties of cement

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Introduction

Cements are currently the most widely used binder in the construction industry. Cement is a finely ground inorganic substance which, when mixed with water, forms a slurry. It belongs to hydraulic binders. Once hardened, it retains its strength and stability in water. Hydraulic cement hardening results from the hydration of calcium silicates and aluminates. Thus, the effective hydraulic components of the cements are compounds of calcium oxide (CaO), silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and ferric oxide (Fe_2O_3).

Cements consist of various mineralogical components such as calcium silicates, aluminates and iron aluminates. Depending on the chemical aspect, the predominant active ingredients, cements can be divided into three groups:

- silicate cements, consisting predominantly of calcium silicate – Portland cement and composite cements;
- aluminate cements, consisting predominantly of calcium aluminate;
- special cements – other cements, e.g. ferrite, barium, etc.

Cements are formed by crushing, grinding and homogenization of raw materials of suitable composition (limestone, marlstone, other materials used for chemical correction such as clay) and subsequent heating above the sintering limit (heating temperature is approximately 1450 °C). This produces a clinker, which, after cooling and mating, is ground with ingredients and admixtures (gypsum, slag, fly ash, …) to fine flour of the final product – cement.

There are a number of different classes of common cement, which are described in European standard EN 197-1. Cement class are indicated by CEM followed by a number by Roman numerals. This may be followed by a forward slash with the letters A, B or C referring to the clinker. Then indication may be followed by a horizontal slash and a capital letter to indicate which component is used in addition to the clinker. A number by Arabic numerals shows the strength classes of the cement. The last capital letter identify the speed of development of the initial strength.

CEM II / A–M (S–V–L) 32.5 R

Portland-composite cement containing a total amount of blast furnace slag (S), silica fly ash (V) and limestone (L) between 6% and 20% by weight with strength classes of 32.5 (the minimal compressive strength is 32.5 MPa after 28 days) with the fast development of the initial strength (R).

Cement types are:

- CEM I – Portland cement with a maximum of 5% other materials;

- CEM II – Portland-composite cement – Portland cement with a maximum of 35% other materials, which are artificial or natural pozzolans such as fly ash, slag, silica fume, slate, calcined clays…
- CEM III – Blast furnace cement – Portland cement with higher percentages of slag;
- CEM IV – Pozzolanic cement – Portland cement and up to 55% of pozzolanic constituents;
- CEM V – Composite cements – mixtures of Portland cement, slag or fly ash and pozzolans.

The total content of active CaO and active SiO_2 must be at least 50% by weight in all CEM cement. The additives are inorganic compound in minimal amount of 5% by weight of all used main and minor additional constituent. Minor additional constituent need not be specified but it must be under amount of 5% by weight of all used constituent.

The maximum amount of additives reflected in the name in may be:

- A – between 6 and 20% by weight;
- B – between 21 and 35% by weight;
- C – between 81 and 95% by weight, this C is classify only for CEM III.

The additives hidden under capital letter are:

- S – blast furnace slag;
- D – silica fume;
- P – natural pozzolans;
- Q – natural calcined pozzolans;
- V – silica fly ashes;
- W – calcium fly ashes;
- T – calcined slate;
- LL, L – limestone;
- M – mixture of additives, which are necessary be specified in brackets.

The minimal strength of the cement in MPa after 28 days may be:

- 32.5;
- 42.5;
- 52.5.

This classification is based on the compressive strength after 28 days on mortar prisms of the standard ratio of components – cement, water and sand. Within each strength class, the speed of development of initial strengths is defined, which can be:

- N – normal;
- R – fast;
- L – slow, which is classify only for CEM III.

In the production of cements, it is necessary to perform prescribed determinations of the properties of cements. These tests guarantees the quality of the cement. Over time, the system of evaluation of the properties of cements has stabilized on a set of determinations that are included in standards and national legislation.

These methods of testing cement include:

- determination of strength – EN 196-1;
- chemical analysis of cement – EN 196-2;
- determination of setting time and soundness – EN 196-3;
- pozzolanicity test for pozzolanic cements – EN 196-5;
- determination of fineness – EN 196-6;
- heat of hydration – Solution method EN 196-8;
- heat of hydration – Semi-adiabatic method EN 196-9.

In our laboratory exercise, we will measure according the standard EN 196-6, which include:

- determination of cement density by the pycnometric method;
- determination of fineness by measuring the specific surface area of the cement by permeable method.

Determination of density

A density is defined by mass of a unit volume of a material substance, expressed as kilograms per cubic metre. To determine the density of the cement (specific gravity), volume measurement is replaced by weighing the material in the pycnometer. In the pycnometer is cement and a liquid that does not react with cement. The specific gravity of the cement is defined as the dry mass of non-porous and void-free cement expressed as kilograms per cubic metre.

Testing procedure

1) Weigh a clean and dry, empty pycnometer with a stopper. Three for the determination of a non-reactive liquid density (kerosene) and five for the determination of the cement density.

2) Fill three empty pycnometer of known volume with the kerosene. Since the volume of fluids varies with temperature, the pycnometer must be tempered in a thermostat bath. In our case, the pycnometer going to be tempered at 20 ° C about 30 minutes. After removing the pycnometer from the thermostat bath, refill the pycnometer with kerosene and replace the stopper carefully. Allow excess liquid to escape through the hole in the stopper. Make sure there are no bubbles. Dry outside and weight. Count the density of kerosene. Express the result as the arithmetic mean of three values.

$$\rho_{kerosene} = \frac{m_{kerosene}}{V_{pycnometer}} \qquad [kg \cdot m^{-3}]$$

3) Fill five empty pycnometer of known volume about 1/3 full of the sample and weigh again. Add the kerosene to the sample. Fill pycnometer about 2/3 full. Then place the pycnometer in a desiccator and remove air bubbles using a membrane pump. Then after 30 minutes of tempering in the thermostat bath at 20 ° C, refill the pycnometer with the kerosene. Replace stopper carefully and allow excess liquid to escape through the hole in the stopper. Make sure there are no bubbles. Dry outside and weight. For each pycnometer count the density of cement. Express the result in kg · m $^{-3}$ as the arithmetic mean with accuracy of 0.001 g · cm $^{-3}$.

$$\rho_{sample} = \frac{m_{cement} \cdot \rho_{kerosene, 20\ °C}}{m_{sample} + m_{pycnometer+kerosene} - m_{pycnometer+kerosene+sample}} \qquad [kg \cdot m^{-3}]$$

Determination of fineness

For measuring the fineness of cement two methods of measurement are described:

- sieving method;
- air permeability method (Blaine method).

Sieving method

The fineness of cement is measured by sieving it on standard sieves. This method serves only to demonstrate the presence of coarse cement particles. It is determined the proportion of cement of which the grain sizes are larger than the specified mesh size by weigh of the individual mass on the sieves. It is not possible to obtain information about the content of the finest cement particles or the composition of individual fractions.

This method is primarily suited to checking and controlling production process.

Air permeability Blaine method

The fineness of cement is measured as the specific surface area, calculated from the time required for the fixed quantity of air to flow through the compacted cement bed of specified dimension and porosity. The number and size range of pores in a given cement bed are determined by the size and distribution of the cement grains which also determined the time for the specified airflow. The higher the specific surface is the finer cement will be.

Figure 1 – Blaine apparatus

The measurement is performed on a Blaine apparatus (Figure 2). It consists of two parts: a cylindrical permeability cell with the plunger and a manometer in the U-tube form with etched lines. The permeability cell has a removable perforated disc on which a compacted cement bed is formed. The permeability cell is placed on a manometer filled with paraffin oil.

This method is a comparative; a reference material of known specific surface is required to determinate the apparatus constant.

Testing procedure

1) At first, determine the density of sample by the pycnometric method before measurement.
2) Calculate the amount of cement needed to prepare the cement bed from
$$m_{sample} = V_{cement\ bed} \cdot (1 - e) \cdot \rho_{sample} \qquad [g]$$
where: V............. volume of the cement bed – in our case is 1.855 cm^3;
e .... exact porosity – in our case is 0.500;
ρ_{sample} ... density of sample measured by the pycnometer method [$g \cdot cm^{-3}$].
3) Place the perforated disc on the ledge at the bottom of the cell and place on it a new filter paper disc. Place the weighed amount of cement in the cell. Place a second new filter paper disc on the levelled cement. Insert carefully the plunger and press it. Pull the plunger by 5 mm, rotate it through 90 °, and press once again. The cement bed is now compacted and ready for the permeability test. Pull the plunger out slowly.
4) Insert the conical part of the cell into the conical socket at the top of the manometer.
5) Open the stopcock and raise the level of the liquid to the highest etched line. Close the stopcock. The liquid level will begins to flow. Start the timer as the liquid reaches the second etched line and stop the timer when the liquid reaches the third one. Record the time and the temperature. Repeat the measurement 5 times on the same cement bed.
6) Measure a second cement bed of the same sample according to the same procedure.
7) For each measured time, calculate the specific surface area of sample from
$$S = \frac{K}{\rho} \cdot \frac{\sqrt{e^3}}{(1 - e)} \cdot \frac{\sqrt{t}}{\sqrt{10 \cdot \eta}} \; [cm^2 \cdot g^{-1}]$$
where: S............. specific surface area of sample [$cm^2 \cdot g^{-1}$];
K .... apparatus constant – in our case is 24.844 $\sqrt{Pa \cdot cm^{-1}}$;
e .... exact porosity – in our case is 0.500;
t .... measured time [s];
ρ .... density of sample measured by pycnometer method [$g \cdot cm^{-3}$];
η .... viscosity of air at tested temperature [Pa·s].

Table 1 – Viscosity of air according to standard EN 196-6. Linear interpolation can be used for the determination of the intermediate values.

temperature [°C]	18	19	20	21	22	23	24
air viscosity [$\times 10^{-6}$ Pa·s]	18.10	18.15	18.19	18.24	18.29	18.34	18.39

7) Express the result in $m^2 \cdot kg^{-1}$ as the arithmetic mean with accuracy of 10 $cm^2 \cdot g^{-1}$.

The protocol of the work must contain

- name and date of the work
- the aim of the work
- materials and method
- tables of raw, measured and calculated values with standard deviations
- a correctly calculated density in $kg \cdot m^{-3}$ with accuracy of $0.001\ g \cdot cm^{-3}$
- a correctly calculated specific surface area in $m^2 \cdot kg^{-1}$ with accuracy of $10\ cm^2 \cdot g^{-1}$
- conclusion of the work with summarized results

Otherwise the protocol will not be accepted!