Warsaw University of Technology

Faculty of Civil Engineering

Department of Building Materials Engineering

BUILDING MATERIALS

LABORATORY TASK

Hydraulic building binders: Cement binders

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1. Aim of the task

Determination of the common cement class, determination of the w/c ratio at which cement paste is characterized by standard consistence and determination of the pH value of the paste.

2. Theoretical background

2.1. Definitions according to standard PN-EN 197-1:2012

- **main constituent** – specially selected inorganic material, of content that exceeds 5% of the cement mass (in relation to the sum of all the main and minor additional constituents),
- **minor additional constituent** – specially selected inorganic material, of content that do not exceeds 5% of the cement mass,
- **common cement** – one of 27 cements of composition as described in standard PN-EN 197-1 (table 1),
- **strength class of cement** – compressive strength class of cement
- **hydration heat** – the amount of heat released during cement hydration over a set period of time.

2.2. Introduction

Cement – according to definition given in the standard PN-EN 197-1 is a hydraulic binder. i.e. a finely ground inorganic material, which – when mixed with water – gives a cement paste, setting and hardening as a result of hydration reactions and processes, and after hardening remains strong and durable, also under water.

The standard PN-EN 197-1 mentions **types and composition of common cements** – 5 types of cement marked by roman numbers as following:

- CEM I – Portland cement,
- CEM II – Portland-composite cement,
- CEM III – blast furnace cement,
- CEM IV – pozzolanic cement,
- CEM V – composite cement.

Above-mentioned cements vary in terms of composition – the content of Portland clinker and other **main constituents**, i.e. specially selected inorganic materials, of content that exceeds 5% of the cement mass (in relation to the sum of all the main and minor additional constituents).

According to the standard PN-EN 197-1 **main constituents** of cement include:

- **Portland clinker (K)**

Portland clinker is a hydraulic material consisting of calcium silicates as well as calcium aluminates and calcium aluminate ferrates. It is produced by sintering raw materials containing: calcium oxide, silicon dioxide, aluminum oxide, iron oxide and small amounts of other materials.

- **Pozzolana (P, Q)**

  Pozzolana is a material of natural or industrial origin – it is silicate or aluminosilicate or a combination of both. Pozzolana does not harden itself, but finely ground and in the presence of water reacts at normal ambient temperature with dissolved calcium hydroxide, forming compounds of calcium silicates and calcium aluminates with increasing strength. Pozzolanas are similar to the compounds that form during the hardening of hydraulic materials. Pozzolanas contain reactive silicon dioxide and alumina. The residue contains iron oxide and other oxides. Two types of pozzolana are used in cements:

  - **natural pozzolana** (P),
  - **natural calcined pozzolana** (Q).

- **Fly ashes (V, W)**

  Fly ash are obtained by electrostatic or mechanical deposition of dusty particulate matter from furnaces fired with coal dust or coal dust co-combusted with other materials. Fly ash obtained by other methods should not be used in cement. Two types of fly ash are used in cements:

  - **siliceous fly ash (V),**
  - **calcareous fly ash (W).**

- **Shale (T)**

  Burnt shale is produced in a special furnace at a temperature of about 800°C. Due to the composition of the natural material and the production process burnt shale contains clinker phases, mainly dicalcium silicate and monocalcium aluminate. It also contains small amounts of free calcium oxide and calcium sulfate as well as oxides of increased pozzolanic reactivity, especially silicon dioxide. Consequently, in a finely ground state, shale exhibits pronounced hydraulic properties and, in addition, pozzolanic properties.

- **Limestone (L, LL)**

  Limestone should consist of at least 75% of calcium carbonate; the clay minerals content should not exceed 1.2 g / 100 g of limestone. The total organic carbon content (TOC) should not exceed:

  - 0,50% of total mass in case of **limestone L**,
  - 0,20% of total mass in case of **limestone LL**.

The degree of grinding of limestone should be approx. 5000 cm^2/g (Blaine apparatus).

- **Silica fume (D)** – consists of very fine spherical particles with an amorphous silica content of at least 85%. It is formed during the reduction of high purity quartz in the presence of carbon in electric arc furnaces in the production of silicon or ferrosilicon alloys.

Minor additional constituents are selected inorganic mineral materials remaining from the clinker production process or the substances considered as main components (fly ash, pozzolana, granulated blast furnace slag, burnt slate, limestone, silica dust) if their content in the cement composition is less than 5% (by mass).

Due to the share of the main constituents, any type of cement can occur in variant **A, B or C**. Taking that in consideration the standard PN-EN 197-1 mentions 27 common cements altogether (table1).

Table 1. Composition of 27 common cements according to standard PN-EN 197-1

Main types	Notation		Composition (percentage by mass [a])										
			Main constituents										Minor additional constituents
			Clinker	Blast furnace slag	Silica fume	Pozzolana		Fly ash		Burnt shale	Limestone		
						natural	natural calcined	siliceous	calcareous				
			K	S	D[b]	P	Q	V	W	T	L	LL	
CEM I	Portland cement	CEM I	95-100	—	—	—	—	—	—	—	—	—	0-5
CEM II	Portland-slag cement	CEM II/A-S	80-94	6-20	—	—	—	—	—	—	—	—	
		CEM II/B-S	65-79	21-35	—	—	—	—	—	—	—	—	
	Portland-silica fume cement	CEM II/A-D	90-94	—	6-10	—	—	—	—	—	—	—	
	Portland-pozzolana cement	CEM II/A-P	80-94	—	—	6-20	—	—	—	—	—	—	
		CEM II/B-P	65-79	—	—	21-35	—	—	—	—	—	—	
		CEM II/A-Q	80-94	—	—	—	6-20	—	—	—	—	—	
		CEM II/B-Q	65-79	—	—	—	21-35	—	—	—	—	—	
	Portland-fly ash cement	CEM II/A-V	80-94	—	—	—	—	6-20	—	—	—	—	
		CEM II/B-V	65-79	—	—	—	—	21-35	—	—	—	—	
		CEM II/A-W	80-94	—	—	—	—	—	6-20	—	—	—	
		CEM II/B-W	65-79	—	—	—	—	—	21-35	—	—	—	
	Portland-burnt shale cement	CEM II/A-T	80-94	—	—	—	—	—	—	6-20	—	—	
		CEM II/B-T	65-79	—	—	—	—	—	—	21-35	—	—	
	Portland-limestone cement	CEM II/A-L	80-94	—	—	—	—	—	—	—	6-20	—	
		CEM II/B-L	65-79	—	—	—	—	—	—	—	21-35	—	
		CEM II/A-LL	80-94	—	—	—	—	—	—	—	—	6-20	
		CEM II/B-LL	65-79	—	—	—	—	—	—	—	—	21-35	
	Portland-composite cement [c]	CEM II/A-M	80-94	<——————12-20——————>									
		CEM II/B-M	65-79	<——————21-35——————>									
CEM III	Blast furnace cement	CEM III/A	35-64	36-65	—	—	—	—	—	—	—	—	
		CEM III/B	20-34	66-80	—	—	—	—	—	—	—	—	
		CEM III/C	5-19	81-95	—	—	—	—	—	—	—	—	
CEM IV	Pozzolanic cement [c]	CEM IV/A	65-89	—	11-35					—	—	—	
		CEM IV/B	45-64	—	36-55					—	—	—	
CEM V	Composite cement [c]	CEM V/A	40-64	18-30	—	18-30			—	—	—	—	
		CEM V/B	20-38	31-50	—	31-50			—	—	—	—	

a) the values in the table refer to the sum of the main and minor additional constituents, b) the proportion of silica fume is limited to 10%, c) in Portland-composite cements CEM II/A-M and CEM II/B-M, in pozzolanic cements CEM IV/A and CEM IV/B and in composite cements CEM V/A i CEM V/B the main constituents other than clinker shall be declared by designation of the cement.

Cement class is determined on the basis of the compressive strength test result carried out according to the procedure described in the standard PN-EN 196-1.

The subject of the test is **standard mortar** prepared according to the procedure described in the abovementioned standard, using:

- cement previously sieved through a sieve with a mesh of 1 mm in amount of 450 ± 2 g;
- standard sand in the amount of 1350 ± 5 g;
- tap water at a temperature of 20 ± 1°C in an amount of 225 ± 1 g (ml).

For the preparation of the mortar, the following standardized automatic mixer with the possibility of speed control is used, molds guaranteeing obtaining specimens in the shape of "beams" measuring 40 mm x 40 mm x 160 mm, a vibrator, a tub for storing fresh mortars with molds in air conditions (guaranteeing maintaining a temperature of 20 ± 1.0°C and relative humidity of air ≥ 90%) and a tub for storing specimens in water. Specimens, after proper curing, are subjected to strength tests.

Cement standard strength is compressive strength determined after 28 days of curing. According to the standard PN-EN 197-1, **3 classes of standard strength** (compressive strength expressed in N/mm^2, i.e. in MPa) are distinguished:

- **class 32.5**,
- **class 42.5**,
- **class 52.5**.

The requirements for individual classes are given in table 2.

Cement early strength is compressive strength determined after 2 days of curing or after 7 days of curing. According to standard PN-EN 197-1, for each standard strength three additional early strength classes are distinguished:

- **class L** – low early strength (concerns only blast furnace cements, CEM III),
- **class N** – normal early strength,
- **class R** – high early strength.

The requirements for individual classes are given in w table 2.

Table 2. Mechanical and physical properties of common cements PN-EN 197-1

Cement compressive strength class	Compressive strength, MPa				Start setting time, min	Volume stability, mm
	Early strength		Standard strength			
	after 2 days	after 7 days	after 28 days			
32.5L*	-	≥ 12.5	≥ 32.5	≤ 52.5	≥ 75	< 10
32.5N	-	≥ 16.0				
32.5R	≥ 16.0	-				
42.5L*	-	≥ 16.0	≥ 42.5	≤ 52.5	≥ 65	
42.5N	≥ 10.0	-				
42.5R	≥ 20.0	-				
52.5L*	≥ 10.0	-	≥ 52.5	-	≥ 45	
52.5N	≥ 20.0	-				
52.5R	≥ 30.0	-				

*) class L concerns only the blast furnace cements (CEM III)

3. Practical tasks:

3.1. Determination of cement compressive strength class according to standard PN-EN 196-6:2016

3.1.1. Materials and equipment

- common cement,
- tap water,
- standard sand,
- anti-adhesion agent,
- automatic laboratory scale,
- 400 ml laboratory glass beaker,
- plastic bowl,
- automatic standard mixer,
- molds for set of 3 beams of dimensions 40 mm x 40 mm x 160 mm,
- laboratory vibrator,
- storage tub (air condition),
- storage tub (water condition).

3.1.2. Task completion

The test consists in preparations the proper amounts of individual components of the standard mortar, i.e. cement being the subject of the test, tap water (meeting the requirements formulated in the standard PN-EN 1008) and standard aggregate (standard sand meeting the requirements formulated in the standard PN-EN 196-1), then putting them in a standard mixer following the standard mortar specimens preparation procedure:

- Weigh 450.0 g of cement specimen in a plastic bowl.
- Measure 225 g of cold tap water in a 400 ml beaker.
- Check the tightness of the packaging, and then unseal the standard sand bag, place it in a special container in the automatic standard mixer.
- Unlock the standard mixer bowl and moisten its interior; moisten the mixer agitator.
- Place the water and then the cement in the standard mixer bowl and immediately start the mixer programmed for the standard mortar mixing procedure.
- The mixing procedure is as follows (see Fig. 1):
  - 30 s of low speed mixing,
  - 30 s of low speed mixing – during this time, standard sand is applied to the paste,
  - 30 s of high speed mixing,
  - 90 s break – during the first 15 s unlock the bowl and scrap the mortar from the walls and bottom to the center of the bowl,
  - 60 s of high speed mixing.

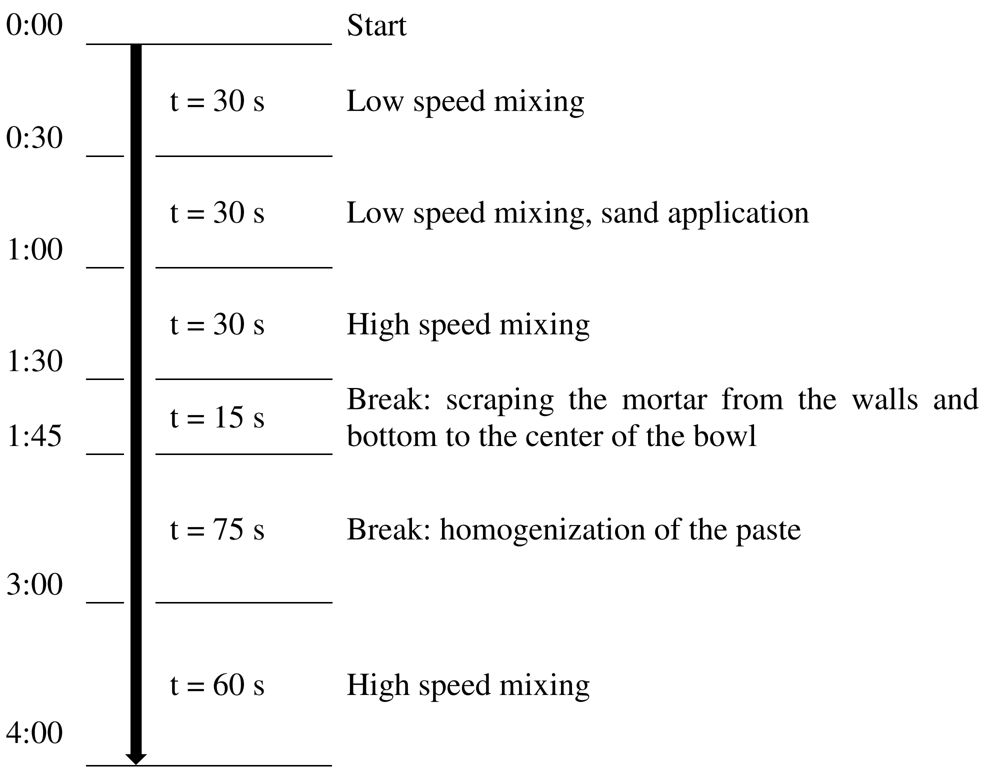

Fig.1. The standard mortar mixing procedure (according to standard PN-EN 196-1)

- Apply the mortar to the prepared (covered with anti-adhesion agent) mold placed on the vibrator stand - place the first of two layers of mortar in each mold compartment.

- Thicken the first layer of mortar with 60 shakes.
- Apply a second coat of mortar.
- Thicken the second layer with 60 shakes.
- Remove excess mortar with a knife.
- Place a smooth glass, metal or plastic plate on the mold.
- Place molds in an air-conditioned room.
- After 20 to 24 hours demold the specimens and place them on stainless steel grates in a container with water at temperature 18 ÷ 20°C in such a way that water has free access to all surfaces of the specimen.
- • After 27 days, determine the flexural strength and compressive strength of the mortar, and evaluate the results in light of the standard requirements (table 2).

Flexural strength of standard mortar is determined on three specimens. The specimen is placed in a testing machine on supports whose spacing is 100 mm, and then it is loaded with concentrated force in the middle of the span until destruction. The flexural strength of the mortar is calculated in MPa with an accuracy of 0.1 MPa according to the formula:

$$f_b = 2{,}34 \cdot P \quad (2.1)$$

where:
f_b – flexural strength, MPa,
P – bending force, kN.

The result is the arithmetic mean of three values of flexural strength, which was determined on a set of three specimens (beams).

Compressive strength of standard mortar is determined on six halves of specimens remining after the flexural strength test. The specimen is placed in a testing machine between two metal 40 x 40 mm (1600 mm^2) pads and then loaded until damaged. The device for determining the compressive strength should provide a load increase rate of 2400 ± 200 N/s. The compressive strength of the mortar is calculated in MPa with an accuracy of 0.1 MPa according to the formula:

$$f_c = P/A \quad (2.2)$$

where:
f_c – compressive strength, MPa,
A – compressed area (area of the metal pad), mm^2,
P – compressive force, N.

The result is the arithmetic average of six values of compressive strength, which was determined on halves of three specimens (beams). If one of the six strength values differs from the mean value by more than 10%, this value should be rejected and the average of the remaining five results recalculated. If one of the other five values differs from the new average by more than 10%, then the entire study should be rejected.

3.1.3. Results

The average compressive strength value of the standard mortar evaluated according to the requirements for standard strength (table 2.) is the basis to indicate the class of tested cement.

3.1.4. Elaboration of results

The results should be presented in the form of a table (table 3). To assess mortar homogeneity, in addition to the average value, standard deviation (SD) and coefficient of variance (CV) should also be calculated.

Table 3. Test results for flexural strength and compressive strength of standard mortar and cement class determination

Flexural strength				Compressive strength					
No	*P* [kN]	f_b [MPa]	$f_{b,av}$ [MPa]	No	*P* [kN]	f_c [MPa]	$f_{c,av}$ [MPa]	*SD* [MPa]	*CV* [%]
1				1.1					
				1.2					
2				2.1					
				2.2					
3				3.1					
				3.2					

3.2. Standard consistence of cement paste

3.2.1. Materials and equipment

- common cement,
- tap water,
- anti-adhesion agent,
- automatic laboratory scale,
- 400 ml laboratory glass beaker,
- plastic bowl,
- automatic standard mixer,
- Vicat apparatus (with bolt).

3.2.2. Task completion

The determination of the standard consistence is carried out according to the procedure described in the standard PN-EN 196-3. It is necessary to prepare the paste from the tested cement with different w/c (water/cement) ratio, and then assess their consistence in a Vicat apparatus with a stainless bolt (the result of the consistence test is the depth of immersion of the bolt in the grout). Based on the results obtained, indicate the composition of the paste (taking into account the percentage of water in the paste) at which the depth of immersion corresponds to the so-called standard consistence (i.e. when the bolt distance from the bottom of the ring in the Vicat apparatus is 6 mm ± 2 mm).

Procedure:

- Weigh 500.0 g of cement specimen in a plastic bowl.
- Measure 125 g of cold tap water in a 400 ml beaker.
- Unlock the standard mixer bowl and moisten its interior; moisten the mixer agitator.
- Place the water and then the cement in the standard mixer bowl and immediately start mixing (attention: this moment is “zero time” of the test).
- The mixing procedure is as follows (see Fig. 2):
- 90 s of low speed mixing.
- 30 s break – unlock the bowl and scrap the paste from the walls and bottom to the center of the bowl,
- 90 s of low speed mixing.

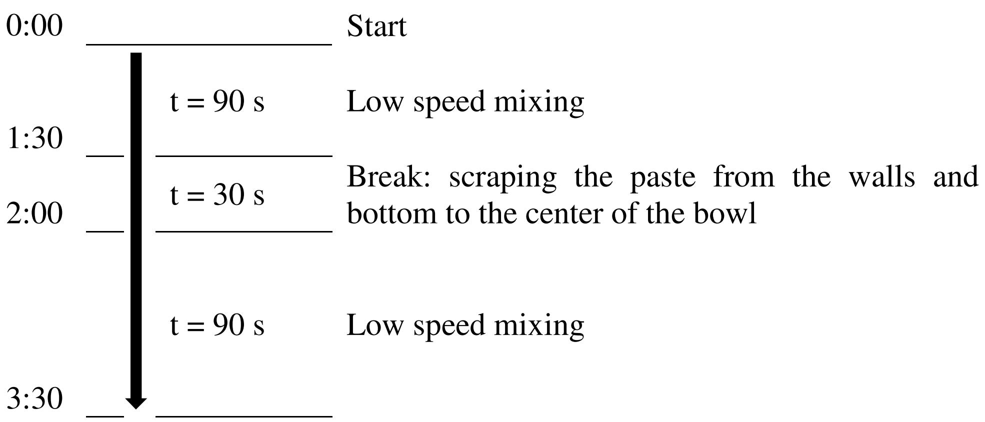

Fig.2. The paste mixing procedure (according to standard PN-EN 196-3)

- Pour the mixed paste into the ring on the Vicat apparatus that was previously placed on a lightly greased glass plate. Remove excess paste with a knife in such a way that the cement paste in the ring has a smooth surface.

- Carefully place the ring in the axis of the bolt, place the bolt on the surface of the paste, adjust to zero the scale.
- Release the bolt block so that it submerges in the cement paste (note: this should be done 4 minutes after zero time - when the paste ingredients are mixed).
- When the bolt stops immersing in the cement paste, but no later than 30 s after releasing the blockage, read the immersion depth in mm; it is the distance of the bolt from the bottom of the ring, i.e. the glass plate. Note the value together with the information on the value of the w/c ratio and the percentage (calculated by mass) of water content in the paste.
- Repeat the cement paste test with different water contents until the gap between the bolt and the glass plate reaches 6 mm ± 2 mm. Note the percentage of water content in such paste with an accuracy of 0.5%.

3.2.3. Results

Based on the obtained test results, indicate the ranges of the percentage of water and the w/c ratio corresponding to the limits of the immersion depth range corresponding to the standard consistence of the paste prepared from the tested cement.

3.2.4. Elaboration of results

The results should be presented in the form of a table (table 4):

Table 4. Immersion depth of Vicat apparatus indenter in cement pastes of various water content (and various w/c ratio)

No.	Cement [g]	Water [g]	Water [%]	*w/c*	Immersion depth [mm]
1	500	125	20,0	0,25	
2	500				
..	500				
n	500				

Based on the obtained results, diagrams of immersion depth (in mm) should be prepared as a function of:

a) percentage water content in the paste (%, by mass),
b) w/c ratio (g/g),

with a range of 6 mm ± 2 mm as the depth range corresponding to the standard consistence of the paste prepared using the tested cement.

3.3. Determination of cement paste pH

3.3.1. Materials and equipment

- common cement,
- tap water,
- universal paper pH indicator.

3.3.2. Task completion

For each cement past prepared as part of the standard consistence determination, immediately before filling the Vicat ring, check the pH value by the approximate method (universal paper) and indicate if the cement paste is an acid, base or presents neutral pH.

3.3.3. Results

When formulating the conclusion regarding the pH of the cement paste, reference should be made to the theoretical information (including the reasons for the high alkalinity of the cement paste). Moreover, the values obtained for cement paste should be compared with the pH of gypsum paste and/or lime paste determined as part of the practical task on air binders.

3.3.4. Elaboration of results

The results should be presented in the form of a table (table 5):

Table 5. Values of pH determined for cement pastes with various w/c ratio

No.	Cement [g]	Water [g]	w/c	pH	Acid/neutral/base
1	500	125	0,25		
2	500				
…	500				
n	500				

The pH value obtained for cement paste with the highest w/c ratio should be compared with the pH values of gypsum paste and/or lime paste obtained as part of a practical task on air binders. The results should be presented in the form of a table (table 6).

Table 6. Values of pH determined for pastes with various binders

	Gypsum paste	Calcium paste	Cement paste*
w/s			
pH			
acid/neutral/base			

*) the value for the highest water/cement ratio obtained in the test

4. Laboratory report

The laboratory report should include:

I. Subject, aim and scope of research (containing basic information about tested materials/products, test methods, requirements),

II. Tests results with proper units (results obtained in the laboratory prepared in the indicated manner, e.g. put in the proper tables),

III. Conclusions (bulleted statements formulated based on the results obtained),

IV. Bibliography (list of references to the literature or www used to prepare the report).